

2.01

Pyrroles and their Benzo Derivatives: Structure

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2.01.1 INTRODUCTION

The first isolation of pyrrole occurred at the end of the nineteenth century and since then the physical and chemical properties of the pyrrolic heterocycles have been the subject of intensive research in all of the diverse fields of chemistry and biology. Pyrroles and indoles particularly have been the subject of great interest owing to their frequent appearances in the structures of complex, biologically active compounds. The unsubstituted compounds and their respective numbering schemes are depicted in Figure 1. Numerous reviews and books are available on each individual compound in Figure 1.

Since the publication of *Comprehensive Heterocyclic Chemistry, First Edition, (CHEC-I)*, a major

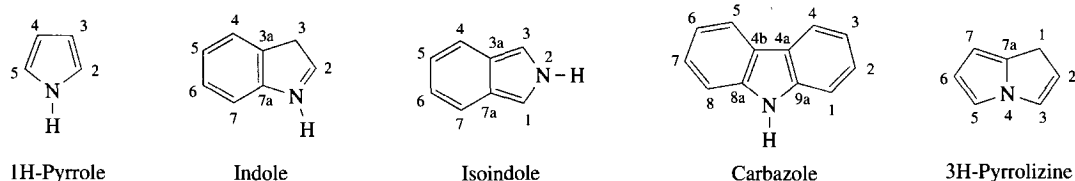


Figure 1 Numbering schemes of pyrrolic heterocycles.

update on the chemistry of pyrrole has been published <B-90M1 201-01>, and the carbazole alkaloids have seen several reviews <87FOR159, 91FOR71>. Pyrrolizines and isoindoles have both been excellently covered in the *Advances in Heterocyclic Chemistry* series <81AHC342, 84AHC(37)1>. Additionally, the *Progress in Heterocyclic Chemistry* series has provided excellent yearly updates on the chemistry of many of the compounds included in this chapter.

2.01.2 THEORETICAL METHODS

Since the mid 1980s there have been a number of improvements in the ability to predict both geometry and reactivity using computational methods. Even the less rigorous molecular mechanics type calculations have achieved a remarkable amount of refinement. It is not uncommon to see computer-simulated spectra used to aid in assignments of experimental spectra. The refinement of computer simulations has given much insight into geometrical questions such as protein folding and drug-receptor interactions, as well as a number of electronic effects including internal rotational barriers, electrostatic potential mapping, and chemistry at the transition state level.

The *ab initio* STO-3G basis set has been used (after MNDO) to calculate electron densities, Mulliken π -overlap populations, and ionization potentials for pyrrole <85JCS(P2)97>. As expected, the nitrogen has the highest electron density at 7.31, with the C-3 at 6.10 only slightly higher than the C-2 at 5.98. Mulliken π -overlap populations for the bonds are as follows: N—C(2)(5) 0.060, C(2)(5)—C(3)(4) 0.139, and C(3)—C(4) 0.077. The ionization potential calculations are somewhat surprising in that the simpler MNDO field more accurately predicts the ionization potentials than does the more complex STO-3G field. These results are included in Table 1. *Ab initio* calculations have also been used to reassign the fundamental vibrations of pyrrole <93KGS651>.

Table 1 Ionization potentials (eV) for pyrrole.

	MNDO	STO-3G	Experimental (UPS)
1a ₂	8.57	6.28	8.22
2b ₁	9.32	7.52	9.22
6a ₁	13.09	12.41	12.70
4b ₂	13.51	13.14	12.95

Additionally, *ab initio* calculations have been employed on pyrrole and other five-membered heterocycles to argue the importance of spin coupling rather than orbital delocalization in stability derived from aromaticity <89JCS(P2)263>. It was found that five of the six π orbitals did indeed show significant localization, while one orbital was delocalized over the nitrogen and carbons 2 and 5. Calculations of the pyrrole ionization potentials by this method were also found to be in good agreement with experimental data. A study on a variety of 2-oxo pyrrole tautomers using MNDO and MINDO/3 SCF-MO methods has been reported <87JHC457>. It was found in comparison with x-ray data on similar compounds that MNDO calculations always gave exaggerated bond lengths between carbon and nitrogen and between carbon and oxygen atoms. While MINDO/3 is more accurate for carbon–nitrogen bond lengths, it is less so for carbon–oxygen bonds. MINDO/3 also gave the N—C(2)—O and O—C(2)—C(3) bond angles about 5° more accurately than MNDO. Apparently, either method is sufficient for calculations of the internal ring angles. It was noted, however, that the MINDO/3 calculations are slightly more accurate for predicting either charge-controlled or orbital-controlled reactivity.

A study comparing the bond orders and π -localization of pyrrole, indole, and isoindole has been performed <84KGS1497>. The calculated bond orders are summarized in Tables 2, 3 (SCF method), and 4 (CNDO/2 method), and the calculated atomic charges are listed in Tables 5 and 6.

Table 2 SCF calculated π bond orders for pyrrole.

Bond	Ground state	Excited state
1—2	0.47	0.33
2—3	0.77	0.50
3—4	0.57	0.35

Table 3 SCF calculated π bond orders.

Bond (atom—atom)	Indole		Isoindole	
	Ground state	Excited state	Ground state	Excited state
1—2	0.41	0.39	0.51	0.34
2—3	0.83	0.64	0.51	0.34
3—3a	0.45	0.51	0.55	0.59
3a—4	0.58	0.52	0.49	0.54
4—5	0.72	0.55	0.78	0.56
5—6	0.62	0.56	0.55	0.68
6—7	0.71	0.59	0.78	0.56
7—7a	0.59	0.52	0.49	0.54
7a—1	0.38	0.36	0.55	0.59
3a—7a	0.59	0.45	0.49	0.49

Table 4 π Bond orders calculated by the CNDO/2 method.

Bond	π Bond order	
	Indole	Isoindole
1—2	0.42	0.54
2—3	0.85	0.54
3—3a	0.39	0.68
3a—4	0.59	0.48
4—5	0.71	0.78
5—6	0.62	0.53
6—7	0.71	0.78
7—7a	0.59	0.48
7a—1	0.38	0.68
3a—7a	0.61	0.46

Table 5 π Charges for each atom (SCF method).

Atom	Pyrrole		Indole		Isoindole	
	Ground	Excited	Ground	Excited	Ground	Excited
1	0.36	0.50	0.29	0.37	−0.18	−0.15
2	−0.16	−0.34	−0.13	−0.16	0.41	0.26
3	−0.02	0.09	0.02	0.03	−0.18	−0.15
3a			−0.01	0.00	−0.01	−0.01
4	−0.02	0.09	0.00	−0.04	−0.01	0.03
5	−0.16	−0.34	0.00	−0.02	−0.01	0.00
6			−0.01	−0.01	−0.01	0.00
7			0.00	0.02	−0.01	0.03
7a			−0.12	−0.16	−0.01	−0.01

The calculated bond orders are clearly more uniform for indole than for isoindole in the ground state, and even more so in the first excited state. Also, the calculated HOMO of isoindole is substantially higher than that of indole (−204.55 versus −134.11, respectively) which correlates well with the experimentally observed higher stability of indole derivatives. The localization of π -charge on the nitrogen atoms can also be used to explain the higher acidity of the isoindole N—H. One surprising result is the uniformity of the total atomic charges calculated by the CNDO/2 method for isoindole, with all of the carbon atoms carrying a charge of either 0.01 or −0.01, and the nitrogen carrying a 0.02 charge. Upon excitation, the electron density of each system shifts drastically. In pyrrole electron density is transferred both from the C-3(C-4) and the nitrogen atom to the C-2(C-5), while in indole, electron flow goes from the C-3 to the C-2 and from the nitrogen to both C-7a and C-4. Conversely, in isoindole, electron density all flows to the nitrogen atom from

Table 6 $\sigma + \pi$ Atomic charges calculated by the CNDO/2 method.

Atom	Atomic charge	
	Indole	Isoindole
1	-0.14	-0.01
2	0.09	0.02
3	0.09	-0.01
3a	0.01	0.01
4	0.00	-0.01
5	-0.02	-0.01
6	0.01	-0.01
7	0.03	-0.01
7a	0.10	0.01

C-1(C-3) and C-4(C-7). It has been suggested that viewing the π system of indole as three weakly interacting systems (benzene, N lone pair, and 2—3 double bond) while taking isoindole as a true 10 π system explains the differences in stability and reactivity between indole and isoindole <84KGS1497>.

A scaled AM1 field has been used successfully to help identify the fundamental vibrational bands of indole <88JCP(88)7295>. Pyrrole and benzene were used to generate a set of scale factors which were then used to simulate a complete vibrational spectrum (included in Table 40, see Section 2.01.3.5) which in turn was used to identify the fundamental vibrations of indole, and to calculate the derived force constants.

AM1 studies for pyrrole, indole, and carbazole, and their *N*-deprotonated analogues have been reported <90JCS(P2)65, 90JCS(P2)1881>, and the calculated bond distances for indole are summarized in Table 7. Of note are the geometrical changes brought about by anion formation. In all three systems, deprotonation brings about a decrease in the C—N—C bond angle of the pyrrole ring of about 3°. For pyrrole, the N—C(2) bond and the C(3)—C(4) bond are shortened by 1.8 and 1.6 pm, respectively, while the C(2)—C(3) bond is lengthened by 2.2 pm. For indole, the changes in the pyrrolic ring are more pronounced, with shortening of the majority of the bonds (N—C(2) by 2.9 pm; C(3)—C(3a) by 2.1 pm; C(3a)—C(4) by 0.1 pm; C(4)—C(5) by 0.4 pm; C(5)—C(6) by 0.3 pm; C(6)—C(7) by 0.4 pm; and C(7a)—N by 0.9 pm), and lengthening of the α - β (to nitrogen) bonds (C(2)—C(3) by 3 pm; C(3a)—C(7a) by 2.6 pm; and C(7)—C(7a) by 0.8 pm). Similar geometric changes were found for carbazole as well, with the relatively large changes taking place in the pyrrolic ring.

Table 7 AM1 derived bond lengths for indole.

Bond	Length (pm)
1—2	139.9
2—3	139.1
3—3a	144.5
3a—4	139.9
4—5	138.8
5—6	140.6
6—7	138.8
7—7a	139.9
3a—7a	144.8
7a—1	139.6

A variety of reported calculations for bond lengths and bond angles of pyrrole are collected in Table 8 <88JCP1739, 89KGS1321, 93JA11906>. Of particular note is the refinement achieved by Allinger and co-workers <93JA11906> using his MM3 molecular mechanics field. MM3 predicts the bond lengths more accurately than all of the other fields, and is roughly equal to the 3-21G, 4-21G, and 6-31G fields for predicting bond angles.

Table 9 summarizes the experimental and MM3 calculated heats of formation for several pyrrole-containing heterocycles, and the accuracy is impressive indeed. Table 10 shows the good agreement

Table 8 Comparison of computational methods for bond lengths (pm) and angles (deg) of pyrrole.

	MM3	MP2	PPP	QCFF/ PI	MINDO/ 3	MNDO	AM1	STO-3G	3-21G	4-21G	6-31G	Experi- mental
Bond length												
N—C(2)	138.0	137.5	139.0	134.4	136.8	139.8	139.1	138.9	137.7	137.9	137.3	137.0
C(2)—C(3)	138.2	139.1	135.8	138.5	139.1	139.4	140.1	135.1	136.0	135.7	136.3	138.2
C(3)—C(4)	141.7	142.4	144.0	143.3	143.3	143.7	143.6	143.2	143.3	143.2	143.0	141.7
Angle (deg)												
C(5)—N—C(2)	110.1	110.3		110.1				108.7	109.2	109.1	109.4	109.8
N—C(2)—C(3)	107.1	107.4		108.9				107.9	108.0	108.0	108.0	107.7
C(2)—C(3)—C(4)	107.8	107.4		106.1				107.8	107.4	107.5	107.3	107.4

References: MM3 <93JA11906>, MP2 <88JCP1739>, PPP <69JA796>, QCFF/PI <81JA4664>, MINDO/3 <75JA1302>, MNDO <77JA4907, 81JHC1189>, AM1 <85JA3902>, STO-3G <88JST(163)173>, STO-3G, 3-21G and 6-31G <86JPC5597>, 4-21G <81JST(85)163>.

found in calculations of the dipole moments by this method, which for pyrrole are more accurate than MNDO, MINDO/3, AM1, 6-31G*/6-31G, or π -SCF calculations. Additionally, the vibrational frequencies are efficiently estimated by this method. The *ab initio* MP2 method also accurately predicts both geometrical parameters and vibrational frequencies <88JCP1739>.

Table 9 Heats of formation (kcal mol⁻¹ (kJ mol⁻¹)).

Compound	Experimental	Calculated (MM3)
Pyrrole	25.88 (108.28)	24.99 (104.56)
N-Methylpyrrole	24.64 (103.09)	24.64 (103.09)
2,5-Dimethylpyrrole	9.51 (39.79)	9.51 (39.79)
Indole	37.41 (156.52)	38.56 (161.34)
Carbazole	50.10 (209.62)	49.92 (208.87)

Table 10 MM3 calculated and experimental dipole moments (Debye units)^a.

Compound	Experimental	Calculated
Pyrrole	1.74	1.75
N-Methylpyrrole	1.92	1.93
2-Methylpyrrole	1.89	1.78
2,4-Dimethylpyrrole	1.75	1.66
2,5-Dimethylpyrrole	2.08	1.88
Indole	2.07	1.65
Carbazole	2.10	1.41

^a 1 Debye = 3.336×10^{-30} C m.

2.01.3 MOLECULAR SPECTROSCOPY

2.01.3.1 X-Ray Crystallography

Several x-ray structures of isoindole compounds have been reported <84KGS1497, 84JCS(P2)833>. Bond lengths and angles for N-methylisoindole are summarized in Table 11 <84KGS1497>. These parameters are very nearly the same as those in 1-formyl-3-phenylisoindole, suggesting that substituents on the pyrrole ring do not significantly alter the solid-state geometry. The geometry of the pyrrolic ring is not significantly altered from that of pyrrole or indole. However, the bond lengths in the carbocyclic ring are alternately shorter and longer than their more uniform indole counterparts,

suggesting that significant localization of electron density is occurring in the six-membered ring of the isoindole nucleus.

Table 11 X-ray crystallographic bond lengths and bond angles for *N*-methylisoindole.

Bond length (pm)		Bond angle (deg)	
C(1)—N	136.2	C(1)—N—C(3)	110.6
N—C(3)	136.0	N—C(3)—C(3a)	107.6
C(3)—C(3a)	138.2	C(3)—C(3a)—C(7a)	107.1
C(3a)—C(4)	143.2	C(3a)—C(7a)—C(1)	107.0
C(4)—C(5)	135.7	C(7a)—C(1)—N	107.7
C(5)—C(6)	141.6	C(7a)—C(3a)—C(4)	119.0
C(6)—C(7)	134.7	C(3a)—C(4)—C(5)	118.7
C(7)—C(7a)	142.0	C(4)—C(5)—C(6)	121.5
C(7a)—C(1)	138.0	C(5)—C(6)—C(7)	121.7
C(3a)—C(7a)	142.9	C(6)—C(7)—C(7a)	119.0
		C(7)—C(7a)—C(3a)	120.0

Solid-state structures of *N*-lithio- and *N*-potassioindole–TMEDA complexes have been reported (<90OM1485>). As expected, the presence of TMEDA allows only for aggregation as dimers, and indeed that is how they behave in the solid state. Interestingly, the lithioindole crystallizes as a *syn* dimer, while the potassioindole forms the *anti* dimer as shown in Figure 2. MNDO calculations show little energetic preference for either conformation, so NMR studies were also performed on both systems. It was subsequently found that no definitive preference for either conformation exists in solution.

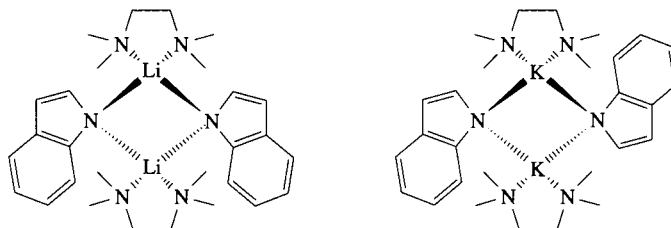


Figure 2 Orientations of indole anions in the solid state.

Interestingly, the solid-state structures of carbazole anions have been shown to be largely determined by the counterion (<89AG(E)1224>). For example, the *N*-caesiocarbazole–PMTDA complex shows some interesting deviations from the expected simple dimeric species. The caesium atoms show the normal σ -bonding to the nitrogen atom, but also have significant π interactions with all five atoms comprising the pyrrole ring. These π interactions are actually closer in the solid state than the σ bond in methylcaesium. Conversely, the *N*-potassiocarbazole–PMTDA complex shows a much smaller interaction between the metal ion and the π cloud, interacting only with carbons 8a and 9a, whereas the *N*-lithioindole shows interaction only between the lithium and the C-2 (<90OM1485>). Similarly, the *N*-lithiocarbazole–THF dimer has two different orientations of the cation: one σ interaction between the lithium and the in-plane nitrogen lone pair, and the other a perpendicular interaction between a second lithium and the $N\pi$ orbital (<87CB1533>).

2.01.3.2 Microwave Spectroscopy

The microwave rotational transitions for pyrrole, indole, and carbazole are given in Tables 12 (<88JSP(127)200>), 13 (<88JSP(127)472, 90JST(240)253>), and 14 (<88JST(190)135>), respectively. Of particular note are the accuracies of the predicted rotational transition frequencies compared with those found experimentally. It has been suggested that a good model for such predictions in indole and carbazole systems is to begin with a basis set of benzene and pyrrole (<88JST(190)135>). The data suggest that the ionic character of the N—H bond increases in the order carbazole > indole > pyrrole, and the π -bonding of the nitrogen lone pair follows an opposite trend. Additionally, the negative charge on the nitrogen atom increases as carbazole > indole > pyrrole. These results taken together suggest

that the nitrogen is moving from sp^2 to sp^3 as more “benzene” rings are added, and that the carbazole framework would prefer nonplanarity but the two benzene rings prohibit this geometry. The ^{14}N quadrupole constants suggest that there is little delocalization of electron density from benzene ring to benzene ring, which also explains the predicative ability of a basis set from pyrrole and benzene. Microwave spectra and rotational assignments for pyrrole-2-nitrile and pyrrole-3-nitrile have also been reported <91JSP(147)229>.

Microwave spectroscopy is especially useful in the indole system where the crystal structure is composed of two different molecular orientations with different bond lengths and angles. The values given by microwave spectroscopy are generally considered to be more reliable. Microwave-derived molecular coordinates for pyrrole <69JST(3)491> and indole <90JST(240)253> (with both sets of x-ray coordinates <75AX(B)1559>) are summarized in Tables 15 and 16, respectively. The lengths and angles for pyrrole have been found to correspond well to x-ray crystallographic data <85CJC2683>.

2.01.3.3 Nuclear Magnetic Resonance Spectroscopy

2.01.3.3.1 Proton NMR

For a basic treatment of the ^1H NMR spectra of these compounds, the reader should consult CHEC-I <84CHEC-I(3)155>.

(i) Pyrrolizines

The ^1H NMR spectra of these compounds has been reviewed <84AHC(37)1>. The protons of the 3*H*-pyrrolizine nucleus occur (neat) at 6.54 (5-H), 6.2 (1-H), 6.08 (6-H), 5.77 (7-H), 5.63 (2-H), and 3.75 ppm (3-H). In opposition to a number of the systems discussed in this chapter, substitution of 3*H*-pyrrolizines usually affects all of the other ring protons noticeably. The common 3*H*-pyrrolizin-3-one has chemical shifts of 7.1 (1-H), 6.83 (5-H), 5.97 (6-H and 7-H), and 5.61 ppm (2-H). The signals for 1*H*-pyrrolizin-1-one are 7.55 (3-H), 6.75 (5-H), 6.62 (7-H), 6.25 (6-H), and 5.47 ppm (2-H).

(ii) Pyrroles

The ring protons of 2*H*-pyrroles are somewhat predictable <B-90MI 201-02>. The chemical shift of the 3-proton is remarkably insensitive to substituents at the 2 position and usually appears around 7.25 ppm. However, 4-substitution has a drastic effect on the 3-proton. For example, a 4-methyl ester substituent shifts the 3-proton to around 7.9 ppm. The 4-proton generally falls between 5.7 and 6.5 ppm. Substitution at 3 or 5 positions can cause downfield shifts to 7 ppm (3,5- Ph_2) or even 7.3 ppm (3- CO_2Me , 5- Ph). Lastly, the 5-proton usually appears at lowest field in the range 7.7–8 ppm, and is slightly sensitive to conjugative substituents at the 4 position.

The proton spectra of 3*H*-pyrroles are similar to those of the 2*H* compounds <B-90MI 201-02>. There are few reported examples of 2-unsubstituted 3*H*-pyrroles, with the 2-proton appearing near 7.9 ppm. Again, the 4-proton appears at highest field in the range of 5.5–6.5 ppm with little variance due to 3-substituents. The 5-proton appears in the polyalkyl-substituted example at 6.64 ppm but is somewhat sensitive to 2-substitution, moving downfield in the 2-phenyl-3,3-dimethyl case to 7 ppm, and appearing as high as 7.6 ppm in some cases.

An overview of the proton spectra of 1*H* pyrroles is included in Table 17. The large range of reported values for the *N*-proton is due to both concentration and solvent dependence, usually resulting in a very broad peak. A study on *N*-aryl-2-formylpyrroles has illuminated a number of interesting points about transmission of electronic effects through the ring <89KGS180>. These compounds show that, despite widely varying electron density in the attached aryl group, very little effect is observed in the protons of the pyrrole ring, suggesting that only slight interaction exists between the attached aryl group and the pyrrole nucleus. A separate study on the tautomeric behavior of 3-hydroxypyrroles has also been reported <91JCS(P2)1999>. As keto–enol tautomerization is largely solvent dependent, these results show that the hydroxy compounds are indeed stable in solution, with the chemical shift differences between the hydroxy and methoxy compounds more attributable to solvent (the 3-hydroxy spectra were taken in $d_6\text{DMSO}$, while the 3-methoxy spectra

Table 12 Rotational transitions of pyrrole.

<i>To</i>			<i>From</i>			<i>Experimental</i> (MHz)	<i>Exp. – Calc.</i> (kHz)	
<i>J</i>	<i>K_p</i>	<i>K_o</i>	<i>J</i>	<i>K_p</i>	<i>K_o</i>			
10	6	4	9	6	3	149	613.221	8
10	7	4	9	7	3	149	580.662	32
10	9	2	9	7	3	204	288.589	–37
11	5	6	10	3	7	258	338.508	176
11	6	6	10	6	5	149	571.077	54
11	7	5	10	6	4	158	645.807	–4
12	5	8	11	5	7	149	565.239	–55
12	6	7	11	6	6	158	632.314	12
12	10	2	11	10	1	205	649.041	41
13	3	10	12	3	9	149	562.293	–46
13	4	9	12	4	8	158	627.946	–8
13	9	4	12	9	3	204	139.806	–27
13	10	4	12	10	3	203	969.658	35
13	12	2	12	10	3	259	222.283	–14
14	3	12	13	3	11	149	559.986	–44
14	4	11	13	4	10	158	625.310	–3
15	1	14	14	1	13	149	557.785	–34
15	3	13	14	3	12	158	623.110	–9
15	8	8	14	8	7	203	951.027	–28
15	13	2	14	13	1	259	460.955	42
16	0	16	15	0	15	149	555.442	–22
16	1	15	15	1	14	158	620.968	6
16	7	10	15	7	9	203	945.242	–46
16	12	4	15	12	3	258	900.621	20
17	0	17	16	0	16	158	618.637	7
17	5	12	16	5	11	203	942.213	–2
17	11	6	16	11	5	258	375.868	–14
17	12	6	16	12	5	258	374.484	–30
18	4	14	17	4	13	203	940.093	8
18	11	8	17	11	7	258	335.703	–51
18	1	17	18	1	18	158	656.789	30
19	4	16	18	4	15	203	938.316	22
19	10	10	18	10	9	258	322.893	–18
19	2	17	19	2	18	158	649.415	28
20	2	18	19	2	17	203	936.575	36
20	8	12	19	8	11	258	317.196	10
20	3	17	20	3	18	158	641.508	15
21	1	20	20	1	19	203	934.677	50
21	8	14	20	8	13	258	314.104	10
21	4	17	21	4	18	158	633.036	–19
22	0	22	21	0	21	203	932.461	57
22	6	16	21	6	15	258	312.185	12
22	5	17	22	5	18	158	624.067	13
23	5	18	22	5	17	258	310.812	9
24	5	20	23	5	19	258	309.651	–2
24	7	17	24	7	18	158	604.268	2
25	4	22	24	4	21	258	308.499	–3
25	8	17	25	8	18	158	593.442	11
26	2	24	25	2	23	258	307.149	–32
26	9	17	26	9	18	158	581.926	–9
27	1	26	26	1	25	258	305.541	–3
27	10	17	27	10	18	158	569.761	10
28	0	28	27	0	27	258	303.443	–12
28	11	17	28	11	18	158	556.856	5
29	12	17	29	12	18	158	543.220	13
30	13	17	30	13	18	158	528.797	9
31	14	17	31	14	18	158	513.568	3
32	15	17	32	15	18	158	497.484	–20
33	16	17	33	16	18	158	480.594	20
34	17	17	34	17	18	158	462.715	–26
35	13	22	35	13	23	203	806.087	–10
35	18	17	35	18	18	158	443.997	28
36	14	22	36	14	23	203	787.240	–41
36	19	17	36	19	18	158	424.220	–2
37	15	22	37	15	23	203	767.738	10
37	20	17	37	20	18	158	403.482	18
38	16	22	38	16	23	203	747.384	–35

Table 12 (continued)

To			From			Experimental (MHz)	Exp. – Calc. (kHz)
<i>J</i>	<i>K_p</i>	<i>K_o</i>	<i>J</i>	<i>K_p</i>	<i>K_o</i>		
38	21	17	38	21	18	158	381.670
39	17	22	39	17	23	203	726.355
39	22	17	39	22	18	158	358.784
40	18	22	40	18	23	203	704.398
40	23	17	40	23	18	158	334.716
41	19	22	41	19	23	203	681.831
41	24	17	41	24	18	158	309.576
42	20	22	42	20	23	203	658.209
42	25	17	42	25	18	158	283.115
43	21	22	43	21	23	203	633.945
44	22	22	44	22	23	203	608.607
45	23	22	45	23	23	203	582.619
46	24	22	46	24	23	203	555.524
47	25	22	47	25	23	203	527.483
48	26	22	48	26	23	203	498.656
49	27	22	49	27	23	203	468.689
50	28	22	50	28	23	203	437.808
51	29	22	51	29	23	203	405.870
52	30	22	52	30	23	203	372.921
53	21	22	53	31	23	203	338.849
54	32	22	54	32	23	203	303.682
55	33	22	55	33	23	203	267.407
56	34	22	56	34	23	203	229.985
57	35	22	57	35	23	203	191.352
58	36	22	58	36	23	203	151.525

were taken in CDCl₃) than to tautomerization. A number of *N*-triisopropylsilylpyrroles have been characterized <90JOC6317>. Again, some conclusions on substituent effects can be drawn. Of the compounds in this study, only the 2-trifluoromethyl ketone showed any truly noticeable effect at the proton at the 5 position. Both the 2- and 4-protons were only very slightly affected by 3-substitution. However, when the 3-substituent was strongly electron withdrawing (cf. NO₂ and COCX₃), the 2-proton was shifted downfield by up to 1 ppm, while the 4-proton was shifted downfield as well, albeit only by 0.6 ppm.

(iii) Indoles

An overview of the proton spectra of indole derivatives is included in Table 18. A thorough study on the effects of 5-substitution has been reported <89MRC1039>. Large downfield shifts of all protons are observed when the 5-substituent is an electron-withdrawing conjugated group such as an ester, nitrile, or nitro group. The most drastic effects are seen, as expected, at the *ortho* protons 4-H and 6-H with downfield shifts of almost 1 ppm for 5-nitroindole. Of the three 5-haloindoles studied, the fluoro derivative is the only one to show major effects, again at the 4- and 6-protons with upfield shifts of about 0.3 ppm and 0.2 ppm, respectively. The electron-donating methoxy and dimethyl-amino groups show large upfield shifts of both 4-H and 6-H, with the change in the shift of 4-H being more pronounced at about 0.5–0.6 ppm. Full assignment of the protons of many indoles is somewhat problematic given that the carbocyclic ring protons have an unfortunate tendency to overlap, especially in the cases of less-substituted indoles <89T5955>. For purposes of structure elucidation, ¹³C NMR is a much more commonly used and reliable tool (see 2.01.3.3.2).

(iv) Isoindoles

Proton spectral data for several *N*-substituted isoindoles has been reported <87KGS1629> and are included in Table 19. *N*-Substitution has a drastic effect on the α-protons. Aryl substituents seem to have the largest effect on protons 1-H and 3-H, shifting them almost 1.5 ppm downfield, with

Table 13 Hyperfine-free transition frequencies for indole.

Transition $J'_{K-K+} - J''_{K-K+}$	Frequency (MHz)	Obs. - Calc. (MHz)
3 _{2,2} -2 _{2,1}	8360.836	0.0017
3 _{1,2} -2 _{1,1}	9040.757	-0.0022
4 _{1,4} -3 _{0,3}	11384.813	0.0014
5 _{1,5} -4 _{1,4}	12473.005	-0.0007
2 _{2,1} -1 _{1,0}	12784.407	-0.0007
5 _{0,5} -4 _{0,4}	12826.609	-0.0014
2 _{2,0} -1 _{1,1}	13340.107	0.0004
5 _{1,5} -4 _{0,4}	13340.898	0.0007
13 _{12,2} -12 _{12,1}	36566.65	0.19
13 _{10,4} -12 _{10,3}	36648.00	0.16
13 _{9,5} -12 _{9,4}	36711.00	0.05
13 _{8,6} -12 _{8,5}	36800.43	0.08
13 _{8,5} -12 _{8,4}	36800.43	-0.04
13 _{4,10} -12 _{4,9}	36872.12	0.04
13 _{7,7} -12 _{7,6}	36932.81	0.21
13 _{7,6} -12 _{7,5}	36935.90	-0.11
13 _{6,8} -12 _{6,7}	37122.61	0.12
13 _{6,7} -12 _{6,6}	37182.68	-0.06
13 _{5,9} -12 _{5,8}	37263.50	0.11
14 _{3,12} -13 _{3,11}	37887.53	-0.01
15 _{1,14} -14 _{2,13}	37970.32	-0.01
16 _{0,16} -15 _{0,15}	37980.10	-0.22
16 _{1,16} -15 _{1,15}	37980.10	-0.02
15 _{2,14} -14 _{2,13}	37988.22	-0.01
15 _{1,14} -14 _{1,13}	38006.50	-0.04
15 _{2,14} -14 _{1,13}	38024.40	-0.04
14 _{2,12} -13 _{2,11}	38342.93	0.05
14 _{1,14} -13 _{1,13}	39454.60	0.10
14 _{10,5} -13 _{10,4}	39512.59	0.09
14 _{9,6} -13 _{9,5}	39591.70	0.03
14 _{7,8} -13 _{7,7}	39869.86	0.37
14 _{7,7} -13 _{7,6}	39878.75	-0.17
14 _{6,9} -13 _{6,8}	40089.00	-0.23
14 _{5,10} -13 _{5,9}	40164.95	-0.02
14 _{6,8} -13 _{6,7}	40224.22	0.14
17 _{0,17} -16 _{0,16}	40281.75	-0.09
17 _{1,17} -16 _{1,16}	40281.75	0.01
16 _{2,15} -15 _{2,14}	40291.20	-0.04
14 _{5,9} -13 _{5,8}	41184.84	0.06
21 _{9,13} -20 _{9,12}	60191.32	0.09
21 _{9,12} -20 _{9,11}	60210.47	-0.05
21 _{8,14} -20 _{8,13}	60549.72	-0.07
25 _{1,24} -24 _{1,23}	61000.17	0.18
22 _{21,1} -21 _{21,0}	61858.40	-0.07
22 _{20,2} -21 _{20,1}	61888.18	0.03
22 _{19,3} -21 _{19,2}	61922.62	-0.02
22 _{18,4} -21 _{18,3}	61963.05	-0.02
22 _{17,5} -21 _{17,4}	62010.91	-0.02
21 _{7,14} -20 _{7,13}	62057.60	-0.07
22 _{16,6} -21 _{16,5}	62068.20	0.03
22 _{15,8} -21 _{15,7}	62137.50	0.02
22 _{14,9} -21 _{14,8}	62222.64	0.02

about 0.1 ppm downfield shifts observed for the remaining four isoindole protons, where simple alkyl substitution shifts 1-H and 3-H downfield by only about 0.9 ppm, with almost no effect on the protons of the carbocyclic ring.

(v) Carbazoles

Very little has been published on the full assignment of protons in carbazole-containing systems because of the difficulties in full assignment of the often overlapping signals. The basic chemical shifts are as follows: 1-H (8-H) 7.49 ppm; 2-H (7-H) 7.36 ppm; 3-H (6-H) 7.16 ppm; 4-H (5-H) 8.08

Table 14 Rotational transition frequencies for carbazole.

Transition $J'_{K'-,K'+}-J''_{K''-,K''-}$	$F'-F''$	Frequency (MHz)	Obs. - Calc. (kHz)
$3_{2,2}-2_{1,1}$		8170.649 ^a	3
	2-1	8170.179	-2
	4-3	8170.518	2.7
	3-2	8171.117	0.0
$3_{2,1}-2_{1,2}$		8575.410 ^a	1
	3-2	8574.298	0.4
	4-3	8575.735	1.1
	2-1	8576.498	1.0
$4_{2,3}-3_{1,2}$		9048.526 ^a	3
	3-2	9048.113	0.9
	5-4	9048.340	0.9
	4-3	9049.067	0.1
$9_{1,9}-8_{0,8}$		9308.165 ^a	-4
	9-8	9308.380	-3
$5_{2,4}-4_{1,3}$		9864.612 ^a	5
	4-3	9864.209	-1.5
	5-4	9864.393	0.9
	4-3	9865.194	-2.9
$4_{2,2}-3_{1,3}$		9890.189 ^a	-3
	4-3	9889.052	-1.1
	6-5	9890.567	-0.8
	5-4	9891.098	0.8
		11795.650 ^a	0
$3_{3,1}-2_{2,0}$		11795.560	3.6
	3-2	11795.609	-2.1
	2-1	11795.709	-0.7
	4-3	11802.556 ^a	4
$3_{3,0}-2_{2,1}$		11802.409	-1
	3-2	11802.570	0.7
	2-1	11802.628	-1.3
	4-3	11905.866 ^a	0
$8_{4,4}-8_{3,5}$		11905.818	-1.6
	8-8	12033.367 ^a	-4
$4_{4,0}-4_{3,1}$		12032.856	1.1
	4-4	12033.552	-1.2
	5-5	12033.731	-1.8
	3-3	12034.939 ^a	-1
$4_{4,1}-4_{3,2}$		12034.419	-2.5
	4-4	12035.128	0.8
	5-5	12035.308	-0.7
	3-3	12881.141 ^a	0
$4_{3,1}-3_{2,2}$		12880.915	-1.8
	4-3	12881.220	-2.5
	6-5	12881.360	4.8
	5-4	13976.251 ^a	-1
$5_{3,2}-4_{2,3}$		13976.014	-0.3
	5-4	13976.338	0.5
	6-5	13976.410	-9.2
	4-3		

^aHypothetical unsplit center point of the transition.**Table 15** Microwave-derived bond lengths and angles for pyrrole.

Bond Length (pm)		Bond Angle (deg)	
N—C(2)	137.0	N—C(2)—C(3)	107.7
C(2)—C(3)	138.2	C(2)—C(3)—C(4)	107.4
C(3)—C(4)	141.7	C(2)—N—C(5)	109.8

Table 16 X-ray and microwave derived bond lengths and bond angles for indole.

	Bond length (pm)				Bond angle (deg)		
	Microwave	X-ray 1	X-ray 2		Microwave	X-ray 1	X-ray 2
N—C(2)	137.0	140.6	139.2	N—C(2)—C(3)	109.1	108.0	108.7
C(2)—C(3)	138.2	140.7	139.9	C(2)—C(3)—C(3a)		108.4	107.8
C(3)—C(3a)		140.1	140.1	C(3)—C(3a)—C(7a)		108.4	107.8
C(3a)—C(4)	142.5	138.8	141.0	C(3a)—C(7a)—N	108.7	107.9	108.1
C(4)—C(5)	138.2	139.0	138.6	C(7a)—C(3a)—C-4	121.4	119.7	119.6
C(5)—C(6)	—	139.9	139.4	C(3a)—C(4)—C(5)	117.2	120.6	120.3
C(6)—C(7)	138.2	138.2	140.3	C(4)—C(5)—C(6)		119.8	120.2
C(7)—C(7a)	142.5	140.7	139.8	C(5)—C(6)—C(7)		120.5	120.1
C(7a)—C(3a)	138.2	140.4	140.8	C(6)—C(7)—C(7a)	117.2	119.8	120.2
C(7a)—N	137.0	140.2	139.1	C(7)—C(7a)—C(3a)	121.4	119.8	119.6
				C(7)—N—C(2)	109.1	108.1	107.9

ppm; and the *N*-H 10.3 ppm (solvent and concentration dependent) (B-89MI 201-01). Again, for structural elucidation, ^{13}C NMR is much more widely used and easily correlatable with the wealth of literature on these compounds.

2.01.3.3.2 Carbon-13 NMR

(i) Pyrroles

Representative ^{13}C data for 2*H*- and 3*H*-pyrroles is summarized in Tables 20 and 21 (for a more comprehensive survey, see (B-90MI 201-02)). The C=N resonance for the 3*H*-compounds is generally at 10–15 ppm lower field than the corresponding 2*H*-compounds (174–191 versus 161–185 ppm). Conversely, the tetrahedral carbon resonance for the 3*H* compounds is usually at 20 ppm higher field than that for the 2*H* compound (typically 55–65 versus 78–85 ppm). For the 2*H*-pyrroles, the C-2 resonance usually occurs in the range 78–85 ppm, the C-3 at 157–167 ppm, the C-4 at 120–137 ppm, and the C-5 at 165–185 ppm. The C-2 resonance of the 2*H*-pyrroles is largely insensitive to other ring substituents owing to its nonconjugated nature. The C-3 shows mild effects (3–4 ppm) due to substituents at the 2-position, moderate effects due to substituents at C-5 (~6 ppm), and much larger effects (~10 ppm) for some substituents at the 4 position. The C-4 resonance is most strongly influenced by the C-2 substituents (up to 14 ppm), and is almost totally insensitive to the substituents at C-3 and C-5. For the 3*H*-pyrroles, typical chemical shifts for the C-2 are 175–185 ppm, for the C-3 are 55–65 ppm, for the C-4 are 118–128 ppm, and for the C-5 are 149–155 ppm. Surprisingly, substitution at C-5 has almost no effect on any of the other ring carbons. The C-2 chemical shift is moderately affected by C-3 substituents (6 ppm), while the C-4 resonance is not.

A cross-section of ^{13}C data for 1*H*-pyrroles is collected in Table 22. *N*-Substitution can have varying effects on the ring carbons. Alkyl or benzyl substituents have a small effect on the C-2 and C-5 (3 ppm downfield), and a negligible effect on the C-3 and C-4. In contrast, both the *N*-acetyl and *N*-ethylcarboxylate show very little effect on the C-2 and C-5 (1–2 ppm), with a more pronounced conjugative effect through the ring to the C-3 and C-4 (5 ppm). *N*-Arylpyrroles show similar effects, with negligible changes at the 2 and 5 positions, and (relatively) larger shifts of the C-3 and C-4. More metallic substituents such as the tributyltin group cause moderate changes at the 2 and 5 positions (~7 ppm), and smaller changes at the 3 and 4 positions. 2-Substituents seem to disperse electronic effects through the ring more effectively than *N*-substituents. For example, the 2-(*N*-propyl)amide causes shifts of the other ring carbons of about 3 ppm. The 2-methyl ester gives more pronounced differences in the C-3 and C-5 chemical shifts of approximately 7 ppm, and the more electron-deficient 2-trichloroacetyl group shifts the C-3 signal by 14 ppm, and the C-5 signal by 9 ppm. 1,2-Disubstituted pyrroles show roughly additive substituent effects of the monosubstituted compounds (89KGS146). For the 2-carbonyl-containing *N*-aryl compounds, mild effects at the 4 position are common. Large effects are reported for the 3 positions (up to 18 ppm) and the 5 positions (~13 ppm). Carbon-13 NMR has also been used in concert with ^1H NMR to study tautomerization at the 3 position (91JCS(P)21999). The similarity of the ^{13}C chemical shifts of 3-hydroxypyrroles to those of the derived 3-methoxypyrroles suggests that in $d_6\text{DMSO}$ solution

Table 17 Proton NMR data (δ , ppm) for 1-*H*-pyrroles.

Substituent(s)	<i>N-H</i>	2- <i>H</i>	3- <i>H</i>	4- <i>H</i>	5- <i>H</i>	Ref.
None	7–12	6.62	6.05	6.05	6.62	B-89MI 201-01
1-Ph		6.97	6.26	6.97	6.26	89MRC187
1,2,5-Ph ₃			6.47	6.47		89MRC187
1-Me, 2,5-Ph ₂			7.33	7.33		89MRC187
1-Bu ^a , 3-OH		6.26		5.50	6.49	91JCS(P2)1999
1-Bu ^a , 3-MeO		6.37		5.80	6.56	91JCS(P2)1999
1-Ph, 3-OH		6.74		5.85	7.06	91JCS(P2)1999
1-Ph, 3-MeO		6.73		6.14	6.95	91JCS(P2)1999
1-Et, 2-Me, 3-OH				5.52	6.30	91JCS(P2)1999
1-Et, 2-Me, 3-MeO				5.86	6.37	91JCS(P2)1999
1-Me, 2-Ph, 3-OH				5.72	6.51	91JCS(P2)1999
1-Me, 2-Ph, 3-MeO				6.07	6.55	91JCS(P2)1999
1-Ph, 2-Me, 3-OH				5.78	6.56	91JCS(P2)1999
1-Ph, 2-Me, 3-MeO				6.18	6.68	91JCS(P2)1999
1-Ph, 2-Ph, 3-OH				5.97	6.78	91JCS(P2)1999
1-Ph, 2-Ph, 3-MeO				6.19	6.73	91JCS(P2)1999
1-(<i>p</i> -NO ₂)Ph, 2-CHO			7.14	6.34	7.31	89KGS180
1-(<i>m</i> -NO ₂)Ph, 2-CHO			7.22	6.44	7.47	89KGS180
1-(<i>p</i> -CO ₂ Et)Ph, 2-CHO			7.33	6.55	7.50	89KGS180
1-(<i>p</i> -Cl)Ph, 2-CHO			7.00	6.38	7.11	89KGS180
1-(<i>m</i> -Cl)Ph, 2-CHO			7.02	6.39	7.11	89KGS180
1-(<i>o</i> -Cl)Ph, 2-CHO			7.22	6.47	**	89KGS180
1-(<i>p</i> -Br)Ph, 2-CHO			7.00	6.38	7.10	89KGS180
1-(<i>p</i> -SO ₂ NH ₂)Ph, 2-CHO			7.29	6.55	*	89KGS180
1-(<i>p</i> -Me)Ph, 2-CHO			7.15	6.41	*	89KGS180
1-(<i>p</i> -MeO)Ph, 2-CHO			*	6.40	*	89KGS180
1-SEM		6.9	6.2	6.2	6.9	86T3723
1-SEM, 2-COPh			7.2–7.9	6.25	6.78	86T3723
1-SEM, 2-CO ₂ Me			7.0	6.2	7.0	86T3723
1-SEM, 2-TMS			6.45	6.18	6.92	86T3723
2-CO ₂ Me	9.0–9.6		7.0	7.0	6.25	86T3723
1-SEM, 2-COCH ₃			7.30	6.30	7.30	84JOC203
1-SEM, 2-CHO			7.20	6.40	7.10	84JOC203
1-tips ^b		6.80	6.32	6.32	6.80	90JOC6317
1-tips, 3-Br		6.70		6.26	6.65	90JOC6317
1-tips, 2-Br			6.27	6.21	6.81	90JOC6317
3-Br	Weak	6.68		6.15	6.62	90JOC6317
1-tips, 3,4-Br ₂		6.72			6.72	90JOC6317
1-tips, 2,3-Br ₂				6.31	6.80	90JOC6317
1-tips, 3-I		6.79		6.36	6.66	90JOC6317
1-tips, 3,4-I ₂		6.79			6.79	90JOC6317
1-tips, 3-NO ₂		7.60		6.83	6.65	90JOC6317
1-tips, 3-COCCl ₃		7.80		6.95	6.75	90JOC6317
1-tips, 3-COCF ₃		7.61		6.85	6.79	90JOC6317
1-tips, 2-COCF ₃			7.44	6.45	7.44	90JOC6317
1-tips, 3-Me		6.55		6.16	6.71	90JOC6317
1-tips, 3-TMS		6.90		6.40	6.83	90JOC6317
1-tips, 3-CO ₂ H		7.50		6.75	6.73	90JOC6317
1-tips, 3-CHO		7.41		6.75	6.78	90JOC6317
1-tips, 3-CH ₂ OH		6.75		6.33	6.75	90JOC6317
1-tips, 3-COMe		7.39		6.72	6.73	90JOC6317
1-tips, 2-Me			6.01	6.15	6.73	90JOC6317

^aUnassignable owing to *N*-aryl protons. ^btips = 1,1,3,3-tetraisopropylidisiloxyl.

minimal ketonization is occurring, and the compounds have enough stability to be synthetically useful. Both 3-alkoxy- and 3-hydroxypyrroles show characteristic upfield shifts of about 18 ppm for C-2 and 6–10 ppm for C-4.

(ii) Indoles

In comparison with the carbocyclic analogue indene, one can see the effect of the heteroatom most strongly in the β -carbons C-3, C-3a, and C-7 with upfield shifts of 29.6, 16.7, and 12.4 ppm, respectively. Only moderate effects (0.1–9.5 ppm) are seen at the other carbons <87MRC377>. Relative

Table 18 Proton NMR data (δ , ppm) for indoles.

Substituent(s)	N—H	H-2	H-3	H-4	H-5	H-6	H-7	Ref.
None	7.74	7.00	6.51	7.64	7.12	7.18	7.24	88H(27)377
1-Me		6.94	6.48	7.60	7.08	7.19	7.25	88MRC552
2-Me	7.35		6.15	7.49	7.04	7.08	7.23	88MRC552
3-Me	7.46	6.76		7.56	7.10	7.16	7.21	88MRC552
1,2-Me ₂			6.20	7.49	7.04	7.11	7.18	88MRC552
1,3-Me ₂		6.71		7.54	7.08	7.18	7.21	88MRC552
2,3-Me ₂	7.20			7.44	7.05	7.08	7.10	88MRC552
1,2,3-Me ₃				7.46	7.05	7.11	7.16	88MRC552
5-NO ₂	8.63	7.38	6.74	8.62		8.12	7.44	89MRC1039
5-CN	9.08	7.33	6.60	7.97		7.39	7.48	89MRC1039
5-CO ₂ Me	8.98	7.20	6.60	8.43		7.89	7.35	89MRC1039
5-Br	7.97	7.08	6.44	7.74		7.24	7.13	89MRC1039
5-Cl	7.99	7.11	6.45	7.59		7.11	7.19	89MRC1039
5-F	7.99	7.14	6.48	7.27		6.92	7.20	89MRC1039
5-OAc	8.25	6.94	6.41	7.28		6.80	7.06	89MRC1039
5-Me	7.65	6.96	6.42	7.41		6.99	7.13	89MRC1039
5-MeO	7.94	7.03	6.44	7.09		6.85	7.14	89MRC1039
5-N(Me) ₂	8.11	6.96	6.39	7.05		6.88	7.11	89MRC1039
1-SEM, 2-TMS			6.80	7.64	7.14	7.26	7.51	86T3723
1-tips, 4-CO ₂ Et		7.40	7.33		7.93	7.20	7.71	88T7325
1-tips, 4-TMS		7.25–7.3	6.75		7.25–7.3	7.13	7.54	88T7325
1-tips, 4-Sn(Me) ₃		7.4–7.75	6.60		7.0–7.3	7.0–7.3	7.4–7.75	88T7325
1-tips, 3-MOM, 4-TMS		7.3–7.45			7.3–7.45	7.12	7.55	88T7325
1-tips, 3-MOM, 4-I		7.30			7.45	6.80	7.60	88T7325
4-PhS	8.1–8.3	7.1–7.3	6.54		7.1–7.3	7.1–7.3	7.35	88T7325
1-Me, 2-TMS, 7-CO ₂ Et			6.75	7.5–7.8	7.05	7.5–7.8		89T5955
1-Me, 7-CO ₂ Et		7.0	6.55	7.6–7.9	7.1	7.6–7.9		89T5955
1-MOM, 7-TMS		7.2	6.6	7.66	7.12	7.44		89T5955

Table 19 Proton spectral data (δ , ppm) for isoindoles.

Substituent(s)	H-1 (H-3)	H-4 (H-7)	H-5 (H-6)	Solvent	Ref.
None (–40°C)	6.28	7.5	6.8	(CD ₃) ₂ CO	73JCS(P1)1432
N-Me	7.19	7.43	6.78	d ₆ DMSO	87KGS1629
N-Ph	7.80	7.52	6.88	d ₆ DMSO	87KGS1629
N-(p-Me)Ph	7.76	7.52	6.88	d ₆ DMSO	87KGS1629
N-(p-Br)Ph	7.82	7.52	6.89	d ₆ DMSO	87KGS1629

Table 20 Carbon-13 NMR data (δ , ppm) for 2H-pyrroles.

R ¹	R ²	R ³	R ⁴	R ⁵	C-2	C-3	C-4	C-5	Solvent	Ref.
PhCH ₂	PhCH ₂	H	H	H	86.0	157.7	136.8	163.4	CDCl ₃	87JCS(P1)2027
Me	Me	Me	H	Me	78.1	170.1	123.0	172.0	CDCl ₃	B-90MI 201-02
Me	Et	Me	H	Me	81.2	170.4	124.4	170.9	CDCl ₃	B-90MI 201-02
PhCH ₂	PhCH ₂	Me	Me	H	86.2	158.8	133.3	167.1	CDCl ₃	87JCS(P1)2027
Me	Me	H	H	Ph	79.5	162.6	123.1	169.6	CDCl ₃	81BCJ2779
Me	Me	H	Ph	H	80.3	153.6	138.1	161.3	CDCl ₃	81BCJ2779
Me	Ph	Me	H	Me	83.3	172.5	124.1	172.1	CDCl ₃	B-90MI 201-02
Me	Me	Me	H	Ph	77.9	168.5	118.8	171.8	CDCl ₃	B-90MI 201-02
Me	Ph	Me	H	Ph	83.8	171.2	120.4	173.3	CDCl ₃	79T2285
Me	Me	Ph	H	Ph	79.1	168.2	120.0	172.0	CDCl ₃	79T2285
Me	CO ₂ Et	Me	H	Me	85.1	166.9	126.4	175.1	CDCl ₃	B-90MI 201-02
Me	CO ₂ Et	Me	H	Bu ^t	84.6	165.8	123.7	185.4	CDCl ₃	B-90MI 201-02
Me	CO ₂ Et	Me	Me	Me	83.6	157.4	132.4	176.4	CDCl ₃	B-90MI 201-02
Me	CO ₂ Et	Me	H	Ph	85.8	167.4	123.4	173.8	CDCl ₃	B-90MI 201-02
Me	Me	CN	H	Ph	82.0	143.8	133.7	167.5	CCl ₄	76HCA1018
CO ₂ Et	CO ₂ Et	Ph	H	Ph	92.3	163.0	125.7	176.7	CDCl ₃	87JCR(S)258
Me	Me	Ph	Ph	NH ₂	73.5	163.0	130.8	166.3	CDCl ₃	79T2285
Me	OH	NH ₂	COMe	Me	94.6	182.0	105.5	168.9	d ₆ DMSO	87JCS(P1)1959
Me	OH	NH ₂	CO ₂ Et	Me	93.5	180.5	95.7	169.0	d ₆ DMSO	87JCS(P1)1959
Ph	OH	NH ₂	CO ₂ Et	Me	94.4	181.0	98.0	172.1	d ₆ DMSO	87JCS(P1)1959
Cl	Cl	Cl	Cl	Cl	98.3	151.1	125.3	164.0	CDCl ₃	80JOC435

Table 21 Carbon-13 NMR data (δ , ppm) for 3*H*-pyrroles.

<i>R</i> ¹	<i>R</i> ²	<i>R</i> ³	<i>R</i> ⁴	<i>R</i> ⁵	C-2	C-3	C-4	C-5
Me	Me	Me	H	Me	188.3	56.5	128.6	149.4
H	Me	Me	H	Ph	181.0	57.6	128.0	152.1
Ph	Me	Me	H	Me	188.6	57.2	129.7	151.4
Me	Me	Me	H	Ph	188.1	56.9	127.2	151.2
Me	Me	Ph	H	Ph	187.3	63.9	127.3	151.9
Me	Me	CO ₂ Et	H	Me	182.3	66.8	122.9	153.7
Me	Me	CO ₂ Et	H	Bu ^t	181.4	66.1	118.9	167.2
Bu ^t	Me	CO ₂ Et	H	Me	191.3	66.5	123.6	153.4
Me	Me	CO ₂ Et	Me	Me	179.2	68.1	129.9	147.1
Ph	Me	CO ₂ Et	H	Me	179.3	64.9	124.8	154.5
Ph	Me	CO ₂ Et	H	Bu ^t	178.4	64.4	120.8	167.9
Me	Me	CO ₂ Et	H	Ph	182.4	67.4	121.3	155.2
CO ₂ Et	Me	CO ₂ Et	H	Ph	173.0	67.1	125.7	155.7
Me	Me	CO ₂ Et	Me	Ph	179.0	69.8	127.5	149.2
Ph	Me	CO ₂ Et	H	Ph	179.1	65.6	122.7	155.8
Ph	Me	CN	H	Me	175.3	49.5	121.4	155.7
Ph	Me	CN	H	Bu ^t	174.5	49.1	118.1	168.9
Ph	Me	CN	H	Ph	175.1	50.1	119.3	156.4
NH ₂	Ph	Ph	CO ₂ Me	H	183.5	67.6	124.9	154.6

to pyrrole, C-2 is shifted downfield by almost 6 ppm while the C-3 is shifted upfield by the same amount. The chemical shifts of carbons 3a and 7a more closely resemble those of naphthalene, suggesting a higher degree of aromaticity than for the fused carbons of isoindole. A selection of ¹³C spectral data is collected in Table 23. Studies on the *N*-formyl indoles have revealed several interesting features <85JJC(B)266, 86JJC(B)1081>. Both 3-phenyl- and 3-ethyl-1-formylindole exist as both *E* and *Z* conformers at ambient temperature, with coalescence occurring at about 100°C. However, 2-substituted *N*-formyl indoles (see Table 23) apparently do not exist as rotamers owing to unfavorable interactions of the formyl group with the 2-substituent. Similar studies on corresponding 2-substituted *N*-acetylindoles gave similar results. In contrast, the *N*-acetyl-2-unsubstituted indoles exist solely as the *E* conformer <85JJC(B)654>.

Fortunately, the ¹³C shifts for indoles have almost no solvent dependence, allowing facile comparison even between spectra obtained in different solvents. Substitution on the nitrogen has varying effects on the ring carbons. *N*-Deprotonation causes extremely large downfield shifts of the C-2 and C-3a, with slight upfield shifts of C-5, C-6, and C-7a. On the other hand, replacement of the *N*-proton with a phenyl group does not noticeably affect any of the ring carbons. Alkyl substitution seems to only affect the chemical shift of the C-2, moving its signal downfield by about 5 ppm. Carbonyl-containing substituents attached to nitrogen show similar behavior to their pyrrole analogues in that the 2 and 7a positions are unaffected while the conjugated 3 and 3a positions experience downfield shifts of 6 and 3 ppm, respectively. Additionally, the C-5, C-6, and C-7 are all affected by about 3 ppm. When the *N*-substituent is a trimethylsilyl group, the expected shift of the C-2 is accompanied by moderate (3 ppm) downfield shifts of the C-3, C-3a, C-7, and C-7a. In contrast, the *N*-tributyltin group only significantly affects the C-2 and C-7a (8 ppm downfield), with a minor shift also seen for C-3a. 2-Substitution by alkyl or aryl groups seems to only affect the C-3 and C-7, while conjugative substituents such as a carboxylic acid moiety show roughly equal effects on C-5, C-6, C-7, and C-7a (an approximately 2 ppm downfield shift), and larger downfield shifts on C-3. Simple 3-substitution seemingly only has a noticeable effect on the C-7a. Conjugative substituents at the 3 position have a much different effect. Large (10–15 ppm) downfield shifts for the C-2 resonance are observed, along with slight upfield shifts for the C-3a carbon, and slight downfield shifts for the C-5, C-6, C-7, and C-7a. A study detailing the effects of 5-substitution on ¹³C chemical shifts has been reported <89MRC1039>. Surprisingly, none of the 5-substituted indoles showed more than a slight effect at the C-3, C-3a, and C-7. In all these compounds, the C-2 was shifted 1–3 ppm downfield from indole itself. Upfield shifts for carbons 4, 6, and 7a were noted for the electron-rich 5-methoxy- and 5-dimethylaminindoles, while downfield shifts of these same carbons were seen for the electron-deficient 5-nitro-, 5-cyano-, and 5-methoxycarbonylindoles. A separate study detailing the effects of nitro substituents at different positions on the carbocyclic ring has been published <89MRC134>. Carbons *ortho* to the nitro group are shifted downfield as expected. When not *ortho* (or *ipso*) to a nitro group, C-3a, C-4, C-5, and C-6 all experience upfield shifts of

Table 22 Carbon-13 NMR data (δ , ppm) for pyrroles.

Substituent(s)	C-2	C-3	C-4	C-5	Ref.
None	118.0	107.7	107.7	118.0	B-89MI 201-01
1-Me	121.0	107.7	107.7	121.0	78JCS(P2)99
1-CH ₂ Ph	121.0	108.3	108.3	121.0	88MRC134
1-COMe	119.0	112.9	112.9	119.0	78JCS(P2)99
1-CO ₂ Me	120.2	112.6	112.6	120.2	88MRC134
1-SnBu ₃	125.2	109.4	109.4	125.2	88MRC134
2-CO ₂ Me, 3-Ph, 4-Me, 5-Et	116.0	132.1	116.3	135.6	86MRC827
2-CO ₂ Et, 3-Ph, 4-Me, 5-Et	116.2	132.0	116.4	135.4	86MRC827
2-CO ₂ Me, 3-Ph, 4-Me, 5-NEt ₂	112.1	132.6	111.3	140.3	86MRC827
2-CO ₂ Et, 3-Ph, 4-Me, 5-NEt ₂	112.6	132.4	111.3	140.2	86MRC827
2-CO ₂ Me, 3-Ph, 4-Ph, 5-Et	116.9	131.1	123.8	136.1	86MRC827
2-CO ₂ Me, 3-Ph, 4-H, 5-Ph	118.4	133.7	110.1	135.7	86MRC827
2-CO ₂ Me, 3-Ph, 4-Me, 5-Ph	117.8	132.4	118.2	132.9	86MRC827
2-CO ₂ Me, 3-Ph, 4-COMe, 5-Me	117.6	132.7	123.8	138.7	86MRC827
2-CO ₂ Me, 3-Ph, 4-CO ₂ Et, 5-Me	117.8	133.7	114.1	138.9	86MRC827
2-CO ₂ Me, 3-CO ₂ Me, 4-Ph, 5-Et	118.5	121.8	123.0	136.3	86MRC827
2-CO ₂ Me, 3-CO ₂ Me, 4-H, 5-Ph	121.6	122.8	110.7	135.2	86MRC827
2-CO ₂ Me, 3-CO ₂ Me, 4-CO ₂ Et, 5-Me	117.9	124.4	112.4	139.3	86MRC827
2-CONMe ₂ , 3-Ph, 4-Ph, 5-Et	120.2	125.4	120.4	134.3	86MRC827
1-Ph	119.3	110.4	110.4	119.3	89MRC187
1-Me, 2,5-Ph ₂	136.9	108.7	108.7	136.9	89MRC187
1,2,5-Ph ₃	135.8	109.9	109.9	135.8	89MRC187
2-CONHPr	126.0	109.3	109.3	121.6	88MRC1037
2-CONHPr, 4-Br	126.2	110.8	96.8	121.7	88MRC1037
2,3-Br ₂ , 5-CONHPr	104.9	99.2	112.8	127.6	88MRC1037
1-Me, 2,3-Br ₂ , 5-CONHPr	111.3	97.9	113.3	128.0	88MRC1037
2-COCCl ₃	122.8	121.5	111.9	127.8	88MRC1037
2-COCCl ₃ , 4-Br	123.0	122.2	99.4	126.9	88MRC1037
2,3-Br ₂ , 5-COCCl ₃	113.2	102.4	123.1	124.0	88MRC1037
1-Me, 2,3-Br ₂ , 5-COCCl ₃	119.7	100.3	124.7	123.4	88MRC1037
2-CO ₂ Me	122.4	115.4	110.2	123.3	88MRC1037
2-CO ₂ Me, 4-Br	122.8	117.1	97.7	123.2	88MRC1037
2,3-Br ₂ , 5-CO ₂ Me	107.4	100.7	118.1	123.8	88MRC1037
1-Me, 2,3-Br ₂ , 5-CO ₂ Me	113.7	98.9	119.4	123.9	88MRC1037
3,4-Bu ₂	116.5	131.5	131.5	116.5	90TL4473
1-(<i>p</i> -NO ₂)Ph	119.3	112.2	112.2	119.3	89KGS146
1-(<i>p</i> -NO ₂)Ph, 2-CHO	131.7	125.1	111.4	131.9	89KGS146
1-(<i>p</i> -NO ₂)Ph, 2-CO ₂ H	124.21	120.1	110.1	129.9	89KGS146
1-(<i>p</i> -NO ₂)Ph, 2-CO ₂ Me	122.7	120.1	110.4	130.5	89KGS146
1-(<i>p</i> -NO ₂)Ph, 2-CON(Et) ₂	127.0	112.8	109.8	124.0	89KGS146
1-(<i>m</i> -NO ₂)Ph	119.1	111.2	111.2	119.1	89KGS146
1-(<i>m</i> -NO ₂)Ph, 2-CHO	131.2	125.4	111.4	132.0	89KGS146
1-(<i>m</i> -NO ₂)Ph, 2-CO ₂ H	124.0	120.3	110.5	130.7	89KGS146
1-(<i>m</i> -NO ₂)Ph, 2-CO ₂ Me	122.4	120.6	109.6	130.2	89KGS146
1-(<i>m</i> -NO ₂)Ph, 2-CON(Et) ₂	126.9	111.8	109.3	123.9	89KGS146
1-CH ₂ OH	120.7	108.8	108.8	120.7	88MRC129
2-CH ₂ OH	132.5	106.1	107.4	117.5	88MRC129
1,2-(CH ₂ OH) ₂	132.3	106.4	108.4	121.5	88MRC129
2,5-(CH ₂ OH) ₂	132.2	105.6	105.6	132.2	88MRC129
1,2,5-(CH ₂ OH) ₃	133.3	106.9	106.9	133.3	88MRC129
2,5-Ph ₂ , 3-N ₂ ⁺	142.7	76.5	106.4	157.2	90SA(A)995
2,5-Ph ₂ , 3-N ₂ ⁺ , 4-CN	150.6	81.5	88.2	158.8	90SA(A)995
2,5-Ph ₂ , 3-N ₂ ⁺ , 4-CO ₂ Et	149.3	79.9	110.6	159.0	90SA(A)995
2-Ph, 3-N ₂ ⁺ , 4-CO ₂ Et, 5-Me	149.9	91.7	110.6	158.7	90SA(A)995
1-Bu ^t , 3-OH	101.2	143.4	97.6	114.2	91JCS(P2)1999
1-Bu ^t , 3-MeO	99.8	148.4	95.8	115.0	91JCS(P2)1999
1-Ph, 3-OH	101.5	146.1	102.2	116.0	91JCS(P2)1999
1-Ph, 3-MeO	100.9	150.8	100.3	117.0	91JCS(P2)1999
1-Et, 2-Me, 3-OH	109.9	139.4	97.7	114.2	91JCS(P2)1999
1-Et, 2-Me, 3-MeO	112.1	144.1	95.2	114.4	91JCS(P2)1999
1-Me, 2-Ph, 3-OH	116.5	141.6	98.1	120.3	91JCS(P2)1999
1-Me, 2-Ph, 3-MeO	118.4	145.5	95.5	119.5	91JCS(P2)1999
1-Ph, 2-Me, 3-OH	110.5	141.4	100.5	116.4	91JCS(P2)1999
1-Ph, 2-Me, 3-MeO	112.9	145.4	97.2	116.8	91JCS(P2)1999
1-Ph, 2-Ph, 3-OH	115.0	144.0	101.1	121.5	91JCS(P2)1999
1-Ph, 2-Ph, 3-MeO	116.9	146.9	97.3	120.4	91JCS(P2)1999

Table 23 Carbon-13 NMR data (δ , ppm) for indoles.

Substituent(s)	C-2	C-3	C-3a	C-4	C-5	C-6	C-7	C-7a	Solvent	Ref.
None	123.7	101.8	127.0	119.9	119.0	121.1	110.4	134.8	CDCl ₃	87MRC377
	125.1	101.1	127.7	120.0	118.8	120.9	111.4	135.9	d ₆ DMSO	
	124.9	102.7	128.2	121.0	120.1	122.2	111.7	136.1	CCl ₄	
	124.3	102.5	128.0	120.8	119.9	122.0	111.2	135.8	dioxane	
	137.0	101.8	148.9	120.5	117.0	118.2	114.8	132.6	d ₈ toluene	90OM1485
1-Li (TMEDA complex) <i>anti</i> dimer (−81 °C)										
1-Li (TMEDA complex) <i>syn</i> dimer (−81 °C)	136.7	101.8	149.0	120.5	117.0	118.3	115.3	132.6	d ₈ toluene	90OM1485
1-Na (TMEDA complex)	137.1	100.6	147.6	120.4	117.0	118.3	114.3	132.2	d ₈ toluene	90OM1485
2-Me	135.7	100.4	129.9	120.0	119.9	121.1	110.9	137.1	dioxane	87MRC377
2-Bu ^t	148.9	97.1	128.7	120.1	119.7	121.2	110.7	136.0	CDCl ₃	87MRC377
2-CO ₂ H	126.2	106.8	127.7	119.3	121.2	123.5	111.9	136.5	d ₆ DMSO	87MRC377
2-CO ₂ Et	127.8	107.8	127.2	122.1	120.3	124.7	112.8	137.8	CDCl ₃ – d ₆ DMSO	87MRC377
2-Ph	137.4	98.5	128.2	119.7	119.1	121.2	110.9	136.8	CDCl ₃	87MRC377
3-Me	121.6	110.9	128.0	118.6	118.9	121.6	110.9	136.0	CDCl ₃	87MRC377
3-CH ₂ CN	123.1	103.9	125.9	120.0	117.9	122.5	111.2	136.3	CDCl ₃	87MRC377
3,3-CH ₂ NMe ₂	124.0	111.7	127.7	118.5	118.9	121.0	111.3	136.3	CDCl ₃ – d ₆ DMSO	87MRC377
3-CH ₂ CO ₂ H	124.8	108.7	128.2	119.5	119.5	122.0	112.4	137.2	d ₆ DMSO	87MRC377
3-CH ₂ CO ₂ Me	123.5	107.6	127.1	118.6	119.5	121.9	111.4	136.2	CDCl ₃	87MRC377
3-CH ₂ CH ₂ NH ₂ (tryptamine)	122.6	112.6	127.4	118.3	118.1	120.8	111.3	136.4	d ₆ DMSO	87MRC377
3-CH ₂ CH ₂ NHMe	122.2	113.3	127.3	118.7	119.0	121.7	111.2	136.5	CDCl ₃	87MRC377
3-CH ₂ CH ₂ NMe ₂	121.2	113.5	126.9	118.2	118.5	121.2	110.7	135.9	CDCl ₃	87MRC377
3-CH ₂ CH ₂ N(CHO)Me	122.1	111.7	126.8	117.9	118.8	121.4	111.2	136.0	CDCl ₃	87MRC377
3-(CH ₃) ₃ CO ₂ H	122.0	114.3	127.3	118.4	118.4	121.1	111.4	136.5	CDCl ₃ – d ₆ DMSO	87MRC377
3-C=C–CO ₂ H	130.4	112.0	125.1	120.8	119.7	122.4	112.3	137.4	CDCl ₃ – d ₆ DMSO	87MRC377
3-CHO	138.1	118.2	124.2	120.8	122.0	123.3	112.3	137.1	d ₆ DMSO	87MRC377
3-COMe	133.4	116.2	124.4	120.9	120.9	122.0	111.4	135.9	d ₆ DMSO	87MRC377
3-OCOMe	121.6	119.8	129.2	114.4	116.9	118.8	111.8	133.3	d ₆ DMSO	87MRC377
3-SMe	127.7	108.0	128.7	119.2	120.3	122.7	111.6	136.3	CDCl ₃ – d ₆ DMSO	87MRC377
2,3-Me ₂	130.7	106.8	129.5	117.9	119.0	120.8	110.2	135.3	CDCl ₃	87MRC377
2-Me, 3-Pr	131.0	111.7	128.9	118.9	118.2	120.5	110.5	135.4	CDCl ₃	87MRC377
2-Me, 3-Ph	134.0	108.6	130.0	119.5	119.0	122.2	110.8	135.9	CDCl ₃	87MRC377
2-Me, 3-SMe	139.1	104.0	130.0	118.5	120.2	121.7	110.7	135.1	CDCl ₃ – d ₆ DMSO	87MRC377
1-Me, 3-CHO	139.8	117.6	124.9	123.7	122.6	121.5	109.9	137.7	CDCl ₃	87MRC377
1-Ph, 3-OCOPh	136.4	117.3	127.5	123.1	124.2	122.8	110.8	138.3	CDCl ₃	87MRC377
1-CHCH ₂ , 2,3-Me ₂	131.5	100.9	129.8	117.8	120.0	121.6	108.8	135.1	CDCl ₃	87MRC377
3-CH ₂ NH ₂	122.0	116.4	126.2	118.2	119.1	121.8	111.5	136.5	CDCl ₃	86MRC829
3-CH ₂ NHMe	123.2	113.8	127.1	118.6	119.4	121.9	111.5	136.5	CDCl ₃	86MRC829
3-CHNMe	129.0	115.0	125.5	120.9	121.2	123.1	111.8	137.2	CDCl ₃	86MRC829
3-CH ₂ OH	123.2	115.5	126.4	118.8	119.5	122.0	111.3	137.0	CDCl ₃	86MRC829
3-CO ₂ H	133.2	120.2	127.4	121.9	122.2	123.4	112.7	138.0	CDCl ₃ – CD ₃ OD	86MRC829
6-Br	124.6	102.6	126.5	121.7	122.8	115.2	113.7	136.3	CDCl ₃	86MRC829
3-CH ₂ NMe ₂ , 6-Br	125.0	111.3	126.5	119.9	122.5	115.2	114.1	136.8	CDCl ₃	86MRC829
1-TMS	129.5	104.6	131.6	120.8	119.8	121.3	112.7	140.1	CDCl ₃	86MRC829
1-Me	128.6	100.8	128.4	119.1	120.7	121.3	109.0	136.6	CDCl ₃	87MRC377
1-CH ₂ CH ₂ NEt ₂	128.0	100.9	128.6	120.8	119.0	121.2	109.2	135.9	CDCl ₃	87MRC377
1-CH ₂ CH ₂ CH ₂ NMe ₂	127.7	100.8	128.5	120.7	119.0	121.1	109.7	136.5	CDCl ₃	87MRC377
1-Ph	123.7	103.1	128.8	120.5	119.9	121.8	110.0	135.2	CDCl ₃	87MRC377
1-COCH ₃	125.0	108.7	130.2	120.6	123.4	124.8	116.0	135.2	CDCl ₃	87MRC377
1,2-Me ₂	138.1	100.0	128.9	120.0	119.6	120.7	109.2	137.2	dioxane	87MRC377
1-Me, 2-CO ₂ H	128.6	110.3	125.7	122.2	120.3	124.6	109.8	139.4	CDCl ₃ – d ₆ DMSO	87MRC377
1,3-Me ₂	127.0	108.6	128.2	118.4	118.1	120.9	109.3	136.6	d ₆ DMSO	88MRC552
1,2,3-Me ₃	132.8	104.9	128.0	117.4	118.1	120.0	108.6	136.2	d ₆ DMSO	88MRC552
2,3-Me ₂ , 4-NO ₂	137.4	104.4	141.1	115.7	118.2	116.2	119.9	137.5	d ₆ DMSO	89MRC134
2,3-Me ₂ , 5-NO ₂	135.4	107.9	113.9	138.2	115.3	110.6	127.9	139.7	d ₆ DMSO	89MRC134
2,3-Me ₂ , 6-NO ₂	133.3	107.0	116.7	113.3	140.5	106.6	133.0	139.6	d ₆ DMSO	89MRC134
2,3-Me ₂ , 7-NO ₂	132.9	107.0	125.4	117.6	116.9	131.4	127.5	135.3	d ₆ DMSO	89MRC134
2-Me, 3-Ph, 4-NO ₂	137.3	115.9	140.7	112.0	119.1	116.3	118.0	138.2	d ₆ DMSO	89MRC134
2-Me, 3-Ph, 5-NO ₂	135.9	115.7	115.2	138.0	114.5	110.6	125.9	140.3	d ₆ DMSO	89MRC134
2-Me, 3-Ph, 6-NO ₂	132.3	116.2	118.1	113.0	101.3	105.4	127.1	133.6	d ₆ DMSO	89MRC134
2-Me, 3-Ph, 7-NO ₂	131.9	116.2	120.4	117.3	116.5	130.2	127.8	136.1	d ₆ DMSO	89MRC134

Table 23 (continued)

Substituent(s)	C-2	C-3	C-3a	C-4	C-5	C-6	C-7	C-7a	Solvent	Ref.
2-CHO, 3-Me, 4-NO ₂	142.1	122.3	144.1	116.3	123.2	118.7	118.9	139.8	d ₆ DMSO	89MRC134
2-CHO, 3-Me, 5-NO ₂	139.9	125.2	118.6	134.6	120.8	113.4	126.4	140.8	d ₆ DMSO	89MRC134
2-CHO, 3-Me, 6-NO ₂	138.2	124.8	121.9	110.0	146.0	109.3	131.7	141.3	d ₆ DMSO	89MRC134
2-CHO, 3-Me, 7-NO ₂	137.7	124.5	130.3	113.8	122.2	134.2	125.9	136.5	d ₆ DMSO	89MRC134
2-CHO, 3-Ph, 5-NO ₂	140.3	133.0	120.0	134.8	120.2	113.3	124.5	142.1	d ₆ DMSO	89MRC134
2-CHO, 3-Ph, 6-NO ₂	137.0	133.8	122.5	109.0	106.9	108.1	125.7	135.0	d ₆ DMSO	89MRC134
2-CHO, 3-Ph, 7-NO ₂	136.6	133.8	125.0	113.9	122.4	133.1	126.2	138.0	d ₆ DMSO	89MRC134
2-Ph, 3-Me, 4-NO ₂	139.0	104.3	141.0	114.9	118.5	116.4	117.9	137.2	d ₆ DMSO	89MRC134
2-Ph, 3-Me, 5-NO ₂	137.0	109.1	115.1	138.7	116.6	110.9	128.3	140.0	d ₆ DMSO	89MRC134
2-Ph, 3-Me, 6-NO ₂	133.6	108.1	118.3	113.6	141.6	107.2	133.8	140.3	d ₆ DMSO	89MRC134
2-Ph, 3-Me, 7-NO ₂	137.3	108.6	126.6	118.4	118.4	132.1	128.2	133.2	d ₆ DMSO	89MRC134
2-Ph, 3-Me, 5-NO ₂	151.5	128.1	110.1	142.0	120.0	112.8	125.2	140.4	d ₆ DMSO	89MRC134
2-Ph, 3-Me, 6-NO ₂	148.5	127.1	113.2	116.8	145.0	108.9	130.8	140.8	d ₆ DMSO	89MRC134
2-Ph, 3-Me, 7-NO ₂	152.0	127.6	121.8	121.4	121.6	133.8	125.0	133.6	d ₆ DMSO	89MRC134
5-NO ₂	127.3	105.1	127.2	118.0	142.0	17.7	111.0	138.8	CDCl ₃	89MRC1039
5-CN	126.7	102.9	127.5	126.2	101.9	124.4	112.1	137.5	CDCl ₃	89MRC1039
5-CO ₂ Me	125.9	103.7	127.5	123.7	121.5	123.1	110.9	138.6	CDCl ₃	89MRC1039
5-Br	125.4	102.1	129.5	123.1	112.9	124.7	112.5	134.3	CDCl ₃	89MRC1039
5-Cl	125.6	102.3	128.9	120.0	125.3	122.2	112.0	134.1	CDCl ₃	89MRC1039
5-F	126.0	102.7	128.2	105.4	158.0	110.3	111.6	132.3	CDCl ₃	89MRC1039
5-OAc	125.8	102.3	128.0	112.3	144.1	115.7	111.6	133.7	CDCl ₃	89MRC1039
5-Me	124.3	101.9	128.0	120.3	128.9	123.5	110.7	134.0	CDCl ₃	89MRC1039
5-MeO	125.0	102.2	128.2	102.3	154.1	112.2	111.8	131.0	CDCl ₃	89MRC1039
5-Me ₂ N	124.8	101.7	128.5	105.1	145.9	113.2	111.5	130.4	CDCl ₃	89MRC1039
1,5-Me ₂ , 2,3-Ph ₂	137.2	114.2	129.3	123.6	127.2	119.1	109.2	135.8	CDCl ₃	85MRC218
2,3-Ph ₂ , 7-MeO	135.2	115.3	126.4	120.6	112.4	102.5	145.8	128.9	CDCl ₃	85MRC218
2,3-Ph ₂ , 4-Me, 7-MeO	136.7	116.7	126.1	127.9	121.2	102.1	weak	129.4	CDCl ₃	85MRC218
3-CH ₂ NEt ₂	123.9	112.0	127.9	118.9	118.9	121.3	111.0	135.9	CDCl ₃	87MRC911
3-CH(CN)CO ₂ Me	123.8	104.4	124.8	118.4	120.5	122.9	111.6	136.1	CDCl ₃	87MRC911
1-CO ₂ Me, 3-CH ₂ CN	123.5	110.3	128.2	118.1	123.0	125.2	115.1	135.2	CDCl ₃	87MRC911
1-CO ₂ Me, 3-CH(CN)CO ₂ Me	125.0	110.4	127.2	118.9	123.5	125.5	115.3	135.4	CDCl ₃	87MRC911
3-CH ₂ NMe ₂ , 5-MeO	125.1	111.6	128.0	100.6	153.5	111.6	111.8	131.4	CDCl ₃	87MRC911
3-CH ₂ CN, 5-MeO	123.7	103.4	126.1	99.6	153.9	112.4	112.3	131.2	CDCl ₃	87MRC911
1-CH ₂ Ph	128.1	101.6	128.6	119.4	120.8	121.6	109.6	136.2		88MRC134
1-(<i>p</i> -NO ₂)Ph	128.1	105.7	129.8	121.2	121.2	123.1	110.7	134.5		88MRC134
1-CO ₂ Me	125.3	107.9	130.3	120.8	122.8	124.3	114.9	135.1		88MRC134
1-OMe	122.8	97.9	124.1	119.8	121.0	122.1	108.0	131.7		81H(16)1523
1-SnBu ₃	132.1	102.9	130.5	118.4	120.3	120.3	112.5	142.6		87MRC134
1-CHO, 2,3-Me ₂	131.2	115.5	129.9	123.9	123.9	124.2	117.9	134.5	CDCl ₃	85IJC(B)266
1-CHO, 2-Ph, 3-Me	134.4	116.0	129.6	118.6	124.3	125.2	117.1	134.6	CDCl ₃	85IJC(B)266
1-CHO, 2-Ph, 3-Et	134.1	123.3	129.5	118.8	124.2	125.0	116.0	134.5	CDCl ₃	85IJC(B)266
1-CHO, 2,3-Ph ₂	134.7	122.7	129.3	119.6	124.7	125.5	116.1	134.7	CDCl ₃	85IJC(B)266
1-CHO, 2,3-Ph ₂ , 5-Me	134.2	121.8	129.6	119.3	132.5	126.5	115.5	134.6	CDCl ₃	85IJC(B)266
1-COCH ₃ , 3-Me	125.1	118.1	131.4	118.7	122.1	123.2	116.5	135.9	CDCl ₃	85IJC(B)654
1-COCH ₃ , 3-Et	124.8	124.8	130.4	118.6	120.8	123.0	116.3	135.7	CDCl ₃	85IJC(B)654
1-COCH ₃ , 3-Ph	125.2	123.6	128.6	119.6	121.8	123.6	116.5	136.0	CDCl ₃	85IJC(B)654
1-COCH ₃ , 2,3-Me ₂	132.4	115.2	131.1	118.0	122.7	123.6	114.9	135.5	CDCl ₃	85IJC(B)654
1-COCH ₃ , 2-Ph, 3-Me	136.8	118.0	130.2	118.5	123.3	125.2	116.2	134.8	CDCl ₃	85IJC(B)654
1-COCH ₃ , 2-Ph, 3-Et	136.9	124.2	129.2	118.6	123.3	125.1	116.5	134.3	CDCl ₃	85IJC(B)654
1-COCH ₃ , 2,3-Ph ₂	136.8	123.4	129.3	119.5	123.7	125.4	116.2	133.0	CDCl ₃	85IJC(B)654
1,2,3-Ph ₃	135.1	113.0	127.8	119.2	118.2	121.5	111.1	135.8	d ₆ DMSO	88JHC1059
2,3-Ph ₂	135.0	113.1	127.7	119.4	118.3	121.7	111.2	135.9	d ₆ DMSO	88JHC1059
2,3-Ph ₂ , 5-Me	134.3	112.0	128.0	117.9	125.7	123.4	111.0	135.3	d ₆ DMSO	88JHC1059
2,3-Ph ₂ , 6-Me	134.3	112.9	125.7	118.5	120.5	131.7	111.2	137.2	d ₆ DMSO	88JHC1059
2,3-Ph ₂ , 7-Me	134.6	113.4	127.3	117.0	118.7	121.7	120.3	137.2	d ₆ DMSO	88JHC1059
2,3-Ph ₂ , 4,7-Me ₂	135.1	114.0	126.9	126.3	120.9	122.0	118.3	137.3	d ₆ DMSO	88JHC1059
2,3-Ph ₂ , 6-NO ₂	137.0	113.2	130.5	117.9	112.5	141.4	107.3	139.3	d ₆ DMSO	88JHC1059
2,3-Ph ₂ , 5-NO ₂ , 6-Me	138.2	116.0	124.9	113.8	138.6	127.2	112.0	142.9	d ₆ DMSO	88JHC1059
2-Ph, 5-Me	138.7	99.4	130.4	124.2	129.1	120.7	111.6	136.7	(CD ₃) ₂ CO	85IZV312
2-Ph, 5-Cl	140.4	99.4	131.1	122.5	125.5	120.1	113.2	136.6	(CD ₃) ₂ CO	85IZV312
2-Ph, 5-MeO	139.2	99.8	130.5	113.0	155.2	102.6	112.6	133.5	(CD ₃) ₂ CO	85IZV312
2-Ph, 5-NH ₂	139.2	99.7	130.4	116.4	145.4	110.3	112.0	135.2	(CD ₃) ₂ CO	85IZV312
2-Ph, 5-NO ₂	141.2	101.7	129.2	117.8	142.5	117.8	112.1	142.3	(CD ₃) ₂ CO	85IZV312
2-Ph, 5-CN	141.4	100.3	130.4	126.2	103.4	125.1	113.1	140.0	(CD ₃) ₂ CO	85IZV312
2-Cl	123.2	100.5	128.8	119.7	120.4	122.0	110.3	134.8	CDCl ₃	86H(24)2879
3-Cl	120.3	106.3	125.2	118.1	120.7	123.0	111.4	134.8	CDCl ₃	86H(24)2879
2,3-Cl ₂	119.9	103.7	125.4	117.7	121.0	123.3	110.7	133.1	CDCl ₃	86H(24)2879
2-Br	108.6	104.7	128.6	119.5	120.3	122.1	110.2	136.3	CDCl ₃	86H(24)2879
3-Br	122.9	91.4	126.7	118.9	120.5	123.2	111.2	135.1	CDCl ₃	86H(24)2879

Table 23 (continued)

Substituent(s)	C-2	C-3	C-3a	C-4	C-5	C-6	C-7	C-7a	Solvent	Ref.
2,3-Br ₂	109.8	94.3	127.4	118.7	121.1	123.3	110.6	135.4	CDCl ₃	86H(24)2879
2-Br, 3-Cl	107.1	107.4	125.6	117.7	120.9	123.3	110.7	134.8	CDCl ₃	86H(24)2879
2-Cl, 3-Br	122.3	90.2	127.1	118.7	121.2	123.4	110.6	133.8	CDCl ₃	86H(24)2879
3-I	120.8	57.3	129.5	128.3	120.8	123.0	111.2	135.4	CDCl ₃	86H(24)2879

varying degrees, while C-7 and C-7a are shifted downfield, with C-3a and C-7 usually experiencing the largest changes.

(iii) Isoindoles

The ¹³C NMR spectrum of *N*-methylisoindole has been reported (88MRC134). It was found that the C-1(C-3) resonates at 111.3 ppm, the C-4(C-7) at 119.1 ppm, the C-5(C-6) at 120.2 ppm, and the C-3a(C-7a) at 124.3 ppm. By comparison, the C-1 and C-3 are shifted upfield by approximately 7 ppm relative to their pyrrole counterparts.

(iv) Carbazoles

Carbon-13 NMR has established itself as an indispensable tool for structure determination in the area of carbazole-containing natural products, and the reader is encouraged to consult the articles by Chakraborty (87FOR159, 91FOR71) for a more exhaustive treatment of carbazole alkaloids. A shorter overview of the typical ¹³C resonances for carbazoles is included in Table 24.

In the same vein as indole/indene, comparison of carbazole with the carbocyclic fluorene shows the heteroatom effect to be strongest at the β -carbons 1(8) and 4a(5a) with upfield shifts of 14.2 and 18.4 ppm, respectively. As in the indole series, only small effects are seen in the other ring carbons. The carbazole ¹³C chemical shifts are very similar to those of the indole nucleus with two exceptions: the C-2 is shifted almost 5 ppm downfield from its indole counterpart, and the C-8a is shifted downfield by 6 ppm. *N*-Substitution has a varying effect on the chemical shifts of the ring carbons. Simple *N*-alkyl or *N*-aryl substitution shows noticeable effects only at the C-4a and C-4b in the form of a 3 ppm downfield shift, while the other carbons are unaffected. *N*-Silylated carbazole again shows the largest changes in the chemical shifts of C-4a and C-4b, shifting them almost 7 ppm downfield, while shifting the C-8a and C-9a carbons downfield by 4 ppm, and the *peri* carbons 1 and 9 downfield by over 2 ppm. The *N*-carbonyl derivatives (*N*-formyl, *N*-acetyl, *N*-trifluoroacetyl, and *N*-methylcarboxylate) give more insight into conjugative effects through the ring system. A very pronounced shift of the C-4a and C-4b of about 7 ppm downfield is the most indicative feature of these compounds, along with upfield shifts of C-8a and C-9a of 2–3 ppm. C-1 and C-9 both experience downfield shifts of about 6 ppm, while the C-3 and C-6 are shifted downfield by up to 8 ppm in the case of *N*-trifluoroacetylcarbazole. Most substituents on the carbocyclic rings affect changes only to the chemical shifts of the *ipso* carbon and the *ortho* carbons on either side of it. Interestingly, the proposition that there is little interaction between the two 6-membered rings (88JST(190)135) is strongly supported by the available ¹³C data. In the cases of only A-ring substitution (such as carbazomycins A and B, and koenoline), little or no effect is seen in the chemical shifts of the C-ring carbons, suggesting only minimal transmission of electronic effects through the pyrrolic ring into the third ring.

(v) Pyrrolizidines

A large number of naturally occurring pyrrolizidine compounds are known, and the ¹³C spectroscopy of these compounds has been extensively reviewed (83H(20)863, 90P11). Typical chemical shifts for these types of alkaloids are around 137 ppm for C-1, 125 ppm for C-2, 60 ppm for C-3, 55 ppm for C-5, 25–35 ppm for C-6, and 75 ppm for C-7a. C-7 is often oxygenated with chemical shifts around 75 ppm, but some nonoxygenated derivatives have been reported with chemical shifts for C-7 of around 30 ppm.

Table 24 Carbon-13 NMR data (δ , ppm) for carbazoles.

Substituent(s)	C-1	C-2	C-3	C-4	C-4a	C-4b	C-5	C-6	C-7	C-8	C-8a	C-9a	Ref.
None	110.5	125.8	119.4	120.3	119.2	119.2	120.3	119.4	125.8	110.5	140.5	140.5	91FOR71
N-Me	108.1	125.2	118.3	119.7	122.1	122.1	119.7	118.3	125.2	108.1	140.4	140.4	87KGS956
N-Ph	109.5	125.7	119.7	119.9	122.8	122.8	119.9	119.7	125.7	109.5	140.3	140.3	87KGS956
N-CHO	116.5	127.2	124.2	120.1	125.7	125.7	120.1	124.2	127.2	116.5	137.1	137.1	87KGS956
N-COCF ₃	116.1	125.4	127.7	119.8	126.8	126.8	119.8	127.7	125.4	116.1	137.4	137.4	87KGS956
N-CH ₂ Ph	108.8	125.7	119.1	120.2	122.9	122.9	120.2	119.1	125.7	108.8	140.5	140.5	88MRC134
N-p-NO ₂ Ph	109.8	126.5	120.6	121.0	123.3	123.3	121.0	120.6	126.5	109.8	139.1	139.1	88MRC134
N-COMe	115.8	126.9	123.2	119.4	126.0	126.0	119.4	123.2	126.9	115.8	138.3	138.3	88MRC134
N-CO ₂ Me	116.0	127.0	123.1	119.4	125.7	125.7	119.4	123.1	127.0	116.0	138.0	138.0	88MRC134
N-NH ₂	108.3	125.9	119.5	120.2	120.9	120.9	120.2	119.5	125.9	108.3	141.5	141.5	88MRC134
N-TMS	112.9	125.2	119.9	119.4	126.1	126.1	119.4	119.9	125.2	112.9	144.1	144.1	88MRC134
1-NO ₂	129.3	120.3	122.7	129.3	127.3	124.4	122.7	120.3	127.3	114.1	141.8	135.4	88IJC(B)402
3-NO ₂	113.1	122.7	138.8	118.3	128.9	122.9	122.4	122.0	128.9	112.2	138.8	152.3	88IJC(B)402
1-MeO, 3-Me (Murrayafoline A)	145.2	107.7	125.4	128.0	110.9	123.4	110.9	120.3	124.3	112.5	139.5	129.2	91FOR71
1-OH, 3-CHO (<i>O</i> -demethylmurrayanine)	146.0	108.4	118.0	127.3	109.5	116.0	121.0	120.1	126.0	112.7	145.2	131.1	91FOR71
1,2-Me ₂ , 3,4,6-(MeO) ₃ (carbazomycin D)	114.5	129.0	144.5	146.6	115.0	123.8	105.9	154.4	114.5	112.0	135.9	138.7	91FOR71
1-CHO, 2-Me, 3,6-(MeO) ₂ , 4-OH (carbazomycin F)	110.3	135.0	139.0	151.9	111.7	123.3	106.5	154.8	115.4	112.4	140.1	139.9	91FOR71
1-CHO, 2-Me, 3-MeO, 4-OH (carbazomycin E)	111.2	134.8	139.6	154.4	111.9	122.6	123.1	120.5	121.6	111.9	140.7	139.6	91FOR71
1,2-Me ₂ , 3,6-(MeO) ₂ , 4-OH (carbazomycin C)	109.8	127.9	139.0	143.6	110.8	124.7	106.2	154.0	113.9	111.5	135.6	138.9	91FOR71
3,6-Cl ₂	112.5	125.9	123.0	120.1	122.6	122.6	120.1	123.0	125.9	112.5	138.5	138.5	88MRC1109
3,6-Br ₂	112.9	128.5	110.8	123.0	123.2	123.2	123.0	110.8	128.5	112.9	138.6	138.6	88MRC1109
3-Cl	113.0	126.2	123.4	120.4	122.8	122.9	119.8	117.9	125.6	110.7	140.0	138.2	88MRC1109
2-OH, 3-CHO (mukonal)	96.3	160.0	115.7	124.9	116.9	122.9	125.5	119.8	119.9	111.1	140.7	145.8	87FOR159
2-OH, 3-Me	96.0	154.7	116.2	121.2	114.9	120.9	120.7	118.2	123.0	110.3	139.7	139.5	87FOR159
3-Me, 6-MeO (glycozoline)	110.6	119.8	126.5	126.6	122.6	122.6	114.5	152.8	102.9	111.4	134.9	138.7	87FOR159
1,2-Me ₂ , 3-MeO, 4-MeO (carbazomycin A)	114.4	128.7	144.4	145.9	113.5	122.8	122.5	119.4	125.0	110.3	139.4	136.4	87FOR159
1,2-Me ₂ , 3-MeO, 4-OH (carbazomycin B)	110.0	127.0	142.0	138.5	109.3	123.3	122.7	119.5	124.7	111.0	139.3	136.8	87FOR159
1-MeO, 3-CH ₂ OH (koenoline)	146.5	105.5	132.6	111.5	123.8	123.3	120.3	119.2	125.5	110.9	139.3	129.2	87FOR159
1,4-Me ₂ , 6-MeO	117.4	125.6	119.5	129.6	120.5	123.8	105.5	152.9	113.4	111.3	134.8	139.9	88H(27)1395
1,4-Me ₂ , 2-CH ₂ CN	115.9	125.7	120.9	129.9	120.3	123.1	122.0	118.7	125.0	110.9	140.1	139.6	90MRC635
1-Me, 2-CH ₂ CN	118.6	126.1	119.6	117.9	121.8	122.5	120.2	118.7	125.6	111.1	140.2	139.6	90MRC635
1,4-Me ₂	116.9	126.1	120.8	130.8	124.4	121.3	122.6	119.4	125.0	110.4	139.3	138.7	90MRC635

2.01.3.3.3 Nitrogen-15 NMR

A study on the protonation effect of azoles has been reported <93MRC791>. ^{15}N NMR spectra were taken for carbazole (−264.1 ppm), *N*-methylcarbazole (−272.7 ppm), and *N*-methylindole (−253.6 ppm) in both d_6DMSO and acidic media ($\text{CF}_3\text{CO}_2\text{H}$ or H_2SO_4) relative to external CH_3NO_2 . The large shifts (35–45 ppm) for carbazoles are much more pronounced than the anilines, probably owing to disruption of the carbazole aromaticity. However, the indole shifts approximately 94 ppm downfield upon protonation. This shift, combined with the couplings, suggests the formation of a 3*H*-indole cation rather than the *N*-protonated species. This data agrees well with protonation sites calculated by AM1 <90JCS(P2)65>.

A ^{15}N NMR study on 5-substituted indoles has been reported, and is summarized in Table 25 <93MRC238>. As expected, electron-donating substituents induce upfield shifts of up to 3.1 ppm, whereas electron-withdrawing substituents shift the signal downfield by up to 6.8 ppm. In comparison to structurally similar acetanilides, substituent transmission is more efficient through the indole system, possibly owing to the higher degree of rigidity of the indole nucleus. The use of $\text{Cr}(\text{acac})_3$ has been advocated as a relaxation agent to shorten the acquisition time for ^{15}N NMR spectroscopy of a variety of carbolines. For carbazole, the relaxation agent induced a change in the ^{15}N NMR signal of only 1.3 ppm, while cutting the acquisition time from approximately 10 hours to a mere 3 hours, and reducing the relaxation delay significantly <93MRC694>.

Table 25 Nitrogen-15 NMR data for 5-substituted indoles^a.

<i>R</i>	$\delta^{15}\text{N}$ (ppm)	^{15}N SCS	<i>J</i> (<i>N</i> , <i>H</i>)	δ (<i>C</i> -2) ^b	δ (<i>C</i> -7a) ^b
H	−244.5	0.0	97.5	163.6	156.2
CN	−238.9	5.6	98.1	167.2	161.7
CO ₂ Me	−240.7	4.2	95.7	165.6	161.7
Br	−242.9	1.6	95.3	166.6	157.9
Cl	−243.0	1.5	98.0	167.0	156.4
F	−244.3	0.2	97.5	168.0	153.9
AcO	−244.2	0.3	97.7	164.8	155.6
NO ₂	−237.7	6.8	96.5	168.8	165.5
Me	−245.5	−1.0	96.5	164.7	154.2
MeO	−246.0	−1.5	95.1	165.9	151.9
Me ₂ N	−247.6	−3.1	95.7	166.1	150.9

^aFrom external neat MeNO_2 . ^bIn d_6DMSO .

2.01.3.4 Ultraviolet Spectroscopy

(i) Pyrroles

The parent 1*H*-pyrrole nucleus (Table 26) shows a characteristic band at about 210 nm (EtOH), with alkyl substitution causing only minor shifts (cf. 3,4-dimethylpyrrole at 203 nm) (for exhaustive treatment, see <B-90MI 201-02>). Table 27 shows the effects of disubstitution on the UV spectra, and Table 28 shows the effects of various solvents. Substitution with a conjugating electron-withdrawing substituent leads to bathochromic shifts of 30–60 nm and emergence of a second intense band at longer wavelength arising from electron transfer between the ring and the substituent. The position of this electron-transfer band moves to longer wavelengths as the degree of conjugation increases, and the band for the 2-substituted compounds is generally found at 15–20 nm longer wavelength than for the corresponding 3-substituted isomer.

The 3*H*-pyrroles generally absorb at approximately 20 nm longer wavelength than the 2*H* analogues (Tables 29 and 30). Both the 2*H*- and 3*H*-pyrrole nuclei show bathochromic shifts of 20 nm when acidified, accompanied in the 2*H* series by a substantial increase in log ϵ <B-90MI 201-02>.

(ii) Pyrrolizines

The absorption spectra of a number of 3*H*-pyrrolizines have been collected in Table 31 <84AHC(37)1>. The parent compound and some simple derivatives show two major bands at

Table 26 UV spectra for pyrrole and mono-substituted derivatives.

<i>Substituent</i>	$\lambda_{\max}$ (nm) (<i>log</i> ϵ)	<i>Solvent</i>	<i>Ref.</i>
None	210 (4.2)	EtOH	71PMH74
1-Me	210 (3.76)	H ₂ O	71PMH74
2-Me	208 (3.85)	H ₂ O	B-74MI 201-01
3-Me	208 (3.77)	H ₂ O	B-74MI 201-01
1-CHO	236 (4.05), 241 sh (3.98), 263 sh (3.18)	C ₆ H ₁₄	B-60MI 201-01
2-CHO	251 (3.49), 287 (4.12)	EtOH	71PMH74
3-CHO	243 (3.97), 270 (3.66)	EtOH	84CHEC-I(3)155
1-COMe	238.5 (4.03), 288 sh (2.88)	EtOH	71PMH74
2-COMe	250 sh, 287 (4.20)	EtOH	71PMH74
3-COMe	243 (3.97), 270 sh (3.66)	MeOH	71T245
2-COCCl ₃	312 (4.13)	EtOH	B-60MI 201-01
2-COCF ₃	260 sh (3.7), 303 (4.21)	EtOH	B-60MI 201-01
1-COPh	220 (4.1), 250 (4.1)	hexane	B-60MI 201-01
2-CHNOH	270 (4.3), 278 (4.25)	dioxane	B-60MI 201-01
3-CH ₂ NOH	243 (4.03)	dioxane	B-60MI 201-01
1-CO ₂ H	224 (3.68)	EtOH	B-74MI 201-01
2-CO ₂ H	228 (3.66), 258 (4.10)	EtOH	B-74MI 201-01
3-CO ₂ H	223 (3.89), 245 (3.71)	EtOH	B-74MI 201-01
1-CO ₂ Me	228 (3.85)	EtOH	71PMH74
2-CO ₂ Me	238 sh (3.63), 263 (4.14)	EtOH	71PMH74
3-CO ₂ Me	224 (3.90), 247 (3.73)	EtOH	71PMH74
2-COCl	288 (4.23)	CHCl ₃	B-60MI 201-01
1-Ph	253 (4.13)	EtOH	B-74MI 201-01
2-Ph	228 sh (3.90), 287 (4.31)	EtOH	B-74MI 201-01
2-NO ₂	231 (3.61), 335 (4.23)	MeOH	71T245
3-NO ₂	268 (3.86), 315 (3.73)	MeOH	71T245
3-Cl	246 (3.18)	H ₂ O	B-60MI 201-01

210–220 and 285–295 nm of roughly equal intensity. Conjugating substituents have a maximum effect when placed at positions 1 or 2, and cause a shift of the long-wavelength band as far as 375–391 nm. Similar conjugating substituents on the pyrrole portion have little effect on the wavelength of absorption maxima. The absorptions for some 3*H*-pyrrolizin-3-ones are summarized in Table 32. Characteristically, there are bands at 220–240, 290–300, and 410–440 nm. Conjugative substituents have widely varying effects. Spectra for the 1*H*-pyrrolizin-1-ones (Table 33) are remarkably similar to those of the 3*H*-pyrrolizin-3-ones. Of note are the halogenated examples 2-chloro-, 2-bromo-, and 2,5-dichloropyrrolizinones, which give strong bathochromic shifts of the long-wavelength bands, in one case as high as 544 nm in the presence of either Lewis acids or strong protic acids, presumably a consequence of altered polarization.

(iii) Indoles

Representative UV spectral data for indoles are included in Table 34 <64JA3796>. Indole itself has six distinct bands with the most intense at 216 nm. This band appears in alkyl indoles around 220–225 nm, and is almost always the most intense band, followed by three other bands of roughly equal intensity near 270, 280, and 290 nm. The UV spectra appear to be largely independent of the number or positioning of simple alkyl groups on the indole nucleus; however, conjugated electron-withdrawing substituents cause major changes in the range of the major bands.

(iv) Isoindoles

UV spectral data for a variety of isoindoles is collected in Table 35. Most of the reported isoindoles have at least two common absorption bands: the first around 320–370 nm, which arises owing to the orthoquinoid-like structure <87KGS1629>, and the second a more intense band anywhere from 210 to 230 nm.

Table 27 UV data for disubstituted pyrroles.

Substituent(s)	Solvent	$\lambda_{\max}$ (nm) ($\log \epsilon$)
2-CHO, 3-CN	H ₂ O	265 (3.62), 300 (4.17)
2-CHO, 4-CN	H ₂ O	281 (4.15)
2-CHO, 3-Me	H ₂ O	295 (4.21)
2-CHO, 4-Me	H ₂ O	258 (3.78), 300 (4.13)
2-CHO, 5-Me	H ₂ O	253 (3.65), 306 (4.32)
2, 3-Me ₂	H ₂ O	258 (3.97), 283 (3.81)
2-CHO, 3-MeO	H ₂ O	294 (4.30)
2-CHO, 5-MeO	H ₂ O	251 (3.38), 315 (4.36)
2-Et, 3-Me	hexane	213 (3.94)
2-CHO, 3-Ph	H ₂ O	299 (4.11)
2-Me, 3-Et	hexane	213 (3.95)
2-Me, 3-Pr	hexane	213 (3.90)
2-Pr, 3-Et	hexane	222 (3.87)
2,3-Ph ₂	hexane	202 (4.31), 242 (4.08), 248 (4.04), 297 (3.95)
2,5-Ph ₂	EtOH	208 (4.13), 232 (4.05), 327 (4.48)
2-COMe, 5-Me	Et ₂ O	290 (4.27)
2-Me, 3-COMe	EtOH	206 (4.17), 243 (3.96), 281 (3.82), 279 (3.78)
2-Me, 4-COMe	EtOH	205 (4.14), 245 (4.01), 280 (3.72)
2-CO ₂ Et, 5-CN	EtOH	268 (4.50), 275 sh (4.38)
3,4-Cl ₂	EtOH	208 (3.72)
2-CN, 3-Me	EtOH	231 sh (3.94), 248 (4.15)
2-CN, 4-Me	EtOH	224 (3.76), 256 (4.05)
2-CN, 5-Me	EtOH	220 (3.65), 257 (4.18)
2-Me, 3-CN	EtOH	238 (3.87)
3-COMe, 4-Me	Et ₂ O	239 (4.13), 272 sh (3.64)
2-COMe, 4-NO ₂	MeOH	247 (4.10), 303 (3.88)
2-COMe, 5-NO ₂	MeOH	241 (4.08), 330 (4.17)
2-NO ₂ , 4-COMe	MeOH	226 (4.06), 323 (3.95)
2-CO ₂ Me, 4-NO ₂	MeOH	236 (4.25), 301 (3.78)
2-CO ₂ Me, 5-NO ₂	MeOH	228 (4.10), 325 (4.10)
2-NO ₂ , 4-CO ₂ Me	MeOH	321 (4.08)
1-Me, 2-COMe	EtOH	247 (3.65), 287 (4.18)
2-CHO, 4-Br	EtOH	253 (3.85), 298 (4.10)
2-CHO, 4-Cl	EtOH	252 (3.84), 302 (4.16)
2-CHO, 3-I	EtOH	257 (3.95), 303 (4.07)
1-CHO, 2-CONH ₂	EtOH	249 (4.03), 258 (3.92), 310 (4.03), 335 sh (3.70)
1-Me, 2-CHO	hexane	246 (3.79), 280 (4.21)
1-Me, 3-CHO	EtOH	254 (4.08), 274 sh (3.93)
1-Me, 3-COMe	hexane	241 (3.93), 262 (3.72)
3,4-(CO ₂ H) ₂	MeOH	240 (3.90), 259 (3.88)
3-CO ₂ Me, 4-CO ₂ H	EtOH	245 (3.90), 260 (3.87)
2-Et, 5-Me	MeOH	216 (3.80)
3,4-(CO ₂ Me) ₂	EtOH	253 (3.90)
2-Me, 3-CO ₂ Et	EtOH	255 (3.87)
1-CHO, 2-Ph	EtOH	234 (4.13), 282 (3.95)
2-Me, 5-Ph	EtOH	298 (4.33)
2-CHO, 5-Ph	EtOH	318 (4.46)
2-CN, 3-Cl	H ₂ O	248 (4.15)
2,3-(CN) ₂	H ₂ O	233 (3.84), 240 (3.85), 267 (4.01)
2-CN, 3-MeO	H ₂ O	247 (4.28)
2-CN, 3-Ph	H ₂ O	265 (4.18)
2-SMe, 3-NO ₂	EtOH	222 (4.17), 274 (3.77), 382 (3.84)

(v) Carbazoles

Ultraviolet spectroscopy has been used extensively in the structure elucidation of carbazole-containing natural products owing to the characteristic spectroscopic features of substituents such as the formyl or methoxy groups; an overview of the UV spectra of carbazoles is given in Table 36. When both are present (such as murrayanine or indizoline), the spectrum resembles that of a formylcarbazole rather than that of a methoxycarbazole. It has also been reported that large solvent effects exist for the UV absorption of carbazole. Solvents capable of hydrogen bonding to the carbazole *N*-H show substantial red shifts <92SA(A)733>.

Table 28 Solvent dependence of the UV spectra of pyrroles.

Substituent	Solvent	λ_{max} (nm) ($\log \epsilon$)	Ref.
None	heptane	210 (3.88)	71CR(C)850
	dioxane	208 (3.71)	71CR(C)850
2-NO ₂	heptane	227 (3.51), 313 (4.07)	71CR(C)850
	MeOH	231 (3.61), 335 (4.23)	71T245
		230 (3.53), 336 (4.11)	72BCJ313
	dioxane	230 (3.08), 330 (3.93)	71CR(C)850
	hexane	194 (3.82), 231 (3.49), 316 (4.05)	72BCJ313
	THF	230 (3.53), 333 (4.15)	72BCJ313
	H ₂ O	233 (3.47), 352 (4.13)	72BCJ313
	MeCN	231 (3.47), 335 (4.12)	72BCJ313
3-NO ₂	hexane	199 (4.10), 246 (3.80), 287 (3.60)	72BCJ313
	heptane	245, 287	71CR(C)850
	MeOH	268 (3.86), 315 (3.73)	71T245
		222 (3.55), 269 (3.80), 313 (3.65)	72BCJ313
	H ₂ O	198 (4.03), 223 sh (3.53), 281 (3.79), 329 (3.65)	72BCJ313
	dioxane	262 (3.85), 307 (3.68)	71CR(C)850
	THF	222 sh (3.53), 265 (3.79), 310 (3.64)	72BCJ313
	MeCN	202 (4.04), 224 (3.54), 268 (3.81), 314 (3.65)	72BCJ313
2-CHO	heptane	245 (3.69), 279 (4.17)	71CR(C)850
	dioxane	294 (3.69), 321 (4.22)	71CR(C)850
	MeOH	252 sh (3.79), 289 (4.24)	71T245
3-CHO	heptane	230, 258	71CR(C)850
	dioxane	237 (3.70), 264 (3.51)	71CR(C)850
2-CH ₃	heptane	212 (3.89)	71CR(C)850
	dioxane	214 (3.86)	71CR(C)850
3-CH ₃	heptane	211 (3.81)	71CR(C)850
	dioxane	212 (3.90)	71CR(C)850

Table 29 UV spectra for 3*H*-pyrroles.

<i>R</i> ¹	<i>R</i> ²	<i>R</i> ³	<i>R</i> ⁴	<i>R</i> ⁵	λ_{max} (nm) ($\log \epsilon$)	Solvent	Ref.
Me	Me	Me	H	Me	253 (3.08)	EtOH	B-90MI 201-02
					271 (3.07)	EtOH-HCl	
Me	Me	Et	Me	H	254 (3.49)	EtOH	70CC142
					272 (3.50)	EtOH-HCl	
Me	Me	Me	Me	Me	262 (3.41)	EtOH	70CC142
					283 (3.42)	EtOH-HCl	
Me	Me	Et	Me	Me	264 (3.33)	EtOH	70CC142
					286 (3.35)	EtOH-HCl	
Ph	Me	Me	H	Me	215 (3.97), 250 sh (3.51), 303 (3.94)	EtOH	B-90MI 201-02
					214 sh (3.65), 260 (3.61), 330 (3.96)	EtOH-HCl	
Me	Me	Me	H	Ph	218 (4.28), 222 (4.28), 270 (3.85)	EtOH	B-90MI 201-02
					235 (4.27), 272 sh (3.54)	EtOH-HCl	
Me	Me	Ph	H	Ph	217 (4.37), 268 sh (3.89), 279 sh (3.73)	EtOH	B-90MI 201-02
					240 (4.29), 280 (3.91)	EtOH-HCl	
Bu ^t	Me	CO ₂ Et	H	Me	210 sh (3.23), 257 (3.38)	EtOH	B-90MI 201-02
Ph	Me	CO ₂ Et	H	Me	215 sh (3.82), 300 (3.83)	EtOH	B-90MI 201-02
Me	Me	CO ₂ Et	Me	Ph	225 (4.11), 273 (3.69)	EtOH	B-90MI 201-02
Ph	Me	CO ₂ Et	H	Ph	217 (4.18), 255 (4.56), 322 (3.78)	EtOH	B-90MI 201-02
Bu ₂ N	H	H	H	Bu ₂ N	269	EtOH	73IZV2722
NH ₂	PhCH ₂	PhCH ₂	CO ₂ Et	NHCH ₂ Ph	240 (4.41), 330 (3.99)	EtOH	72T4431

2.01.3.5 Infrared Spectroscopy

(i) Pyrrolizines

The most common features of pyrrolizine derivatives in the infrared are the carbonyl bands of 3*H*-pyrrolizin-3-ones at approximately 1740 cm⁻¹ and 1*H*-pyrrolizin-1-ones at 1690 cm⁻¹.

Table 30 UV spectra for 2*H*-pyrroles.

<i>R</i> ¹	<i>R</i> ²	<i>R</i> ³	<i>R</i> ⁴	<i>R</i> ⁵	λ_{max} (nm) (<i>log</i> ϵ)	Solvent	Ref.
Me	Me	H	Me	Me	236 (3.42)	EtOH	59JCS1587
					255 (3.48)	EtOH-HCl	
Me	Me	Me	H	Me	233 (3.38)	EtOH	59JCS1587
					250 (3.47)	EtOH-HCl	
Me	Et	Me	Me	H	248 (3.49)	EtOH	59JCS1587
					271 (3.58)	EtOH-HCl	
Me	Me	Me	Me	Me	243 (3.40)	EtOH	59JCS1587
					266 (3.45)	EtOH-HCl	
Me	Me	H	H	Ph	212.5 (4.08), 217.5 (4.08), 249 (4.0)	MeOH	81BCJ2779
Me	Me	H	Ph	H	224 (4.18), 268 (3.7), 287 sh, 296 sh	MeOH	81BCJ2779
Me	Me	Me	H	Ph	244 (4.19), 277 sh (3.5), 285 sh (3.3)	EtOH	76C453
					216 (3.83), 279 (4.35)	EtOH-HCl	B-90MI 201-02
Me	Me	Ph	Me	Et	252.5 (3.78)	EtOH	79T2285
Me	Ph	Me	H	Ph	215 (4.37), 245 (4.21)	EtOH	B-90MI 201-02
Me	Me	Ph	H	Ph	248 (4.25)	EtOH	79T2285
Me	Me	Ph	Me	Ph	240 (4.17)	EtOH	79T2285
Me	CHCl ₂	H	H	Me	233 (3.38)	EtOH	65G83
Me	CHCl ₂	Me	Me	Me	247 (3.45)	EtOH	65G83
Me	Me	H	H	CO ₂ Et	242 (3.41)	EtOH	85JCR(S)238
Me	Me	CN	H	Ph	237 (4.17), 276 sh(3.76)	EtOH	76HCA1018
Me	Me	Ph	CO ₂ Me	H	230 sh (3.55), 251 (3.60), 280 sh (3.59)	MeOH	82HCA1489
Me	Me	Ph	CO ₂ Me	CO ₂ Me	227 (3.58), 293 (3.57)	MeOH	82HCA1489
Me	Me	CO ₂ Me	CO ₂ Me	Ph	230.5 (3.87), 248 sh (3.74), 283 sh (3.19)	EtOH	76HCA2149
Me	Me	Ph	CO ₂ Et	Ph	222 (4.19), 245 sh (4.10), 287 sh (3.68)	EtOH	77HCA687
H	H	Me	Me	MeO	200 (3.82)	Bu'OH	81MC359
Bu ¹	OOH	H	H	Bu ¹	205 (3.60), 235 (3.08), 275 sh (2.51)	MeOH	72TL1337
Bu ¹	OOH	Bu ¹	H	Bu ¹	< 204 (3.53), 249 (3.440), 285 sh (2.92)	MeOH	72TL1337
Me	Me	MeO	H	Me	255 (3.65)		71KGS1530
Me	Me	Me ₂ N	H	Me	333 (4.04)		71KGS1530
H	H	OH	Ph	Me ₂ N	222 (4.08), 287 (4.30)	MeOH	69T5721
H	Me	OH	Me	Et ₂ N	228 (4.00), 289 (4.29)	MeOH	69T5721
Me	Me	OH	Me	Et ₂ N	226 (4.00), 287 (4.35)	EtOH	68CB308
Cl	Cl	Cl	Cl	Cl	220 (3.48), 282 (3.00)		77MI 201-01

Table 31 UV/visible spectra of 3*H*-pyrrolizines.

Substituent	Solvent	λ_{max} (nm) (<i>log</i> ϵ)	Ref.
H	EtOH	218 (3.4), 290 (3.78)	63AC(R)309, 66JOC870
H (anion)	THF	210 (4.3), 295 (3.98)	67T2941
1-Me	EtOH	287 (3.79)	66JOC870
2-Me		219 (3.56), 295 (3.88)	72JCS(P1)840
1,2-Me ₂	EtOH	210 (3.77), 285 (3.88)	73JOC1583
1-Me-5-C(CO ₂ Me)=CHCO ₂ Me	EtOH	215 sh, 240 (4.19), 247 sh, 377 (4.32)	73JOC1583
3,3-Me ₂ -5-C(CO ₂ Me)=CHCO ₂ Me	EtOH	209 (4.19), 287.5 (3.88), 391 (3.25)	72JCS(P1)2517
1,2-Me ₂ -3,5-(C(CO ₂ Me)=CHCO ₂ Me) ₂	EtOH	209.5 (4.24), 240.5 (4.27), 247 sh, 382 (4.21)	72JCS(P1)844
1-AcO	EtOH	220 (4.8), 240 (3.7), 320 (4.06)	63AC(R)309
2-Ac	EtOH	242 (3.31), 373 (4.07)	82JCS(P1)1123
6-Ac	EtOH	250 (3.31), 292 (2.45)	82JCS(P1)1123
5-CCl ₃ CO		358 (3.57)	71ACS2447
2-MeO ₂ C		354 (4.4)	77AP(310)186
2-EtO ₂ C		352 (4.4)	77AP(310)186
6-MeO ₂ C		223 (4.0 ⁻), 273 (4.4)	77AP(310)186
6-EtO ₂ C		232 (4.6), 273 (4.3), 355 (1.7)	68CB3843, 77AP(310)186
7-EtO ₂ C		233 (4.08), 258 (3.4), 268 (3.4), 305 (3.94)	82JCR(S)54
1-NH ₂ -2-CO ₂ Me-3-Ph	CH ₂ Cl ₂	252 (4.04), 262 sh (3.92), 332 (4.22)	82CB714
1-NH ₂ -2-CO ₂ Et-3-Ph	CH ₂ Cl ₂	252 (4.04), 262 sh (3.95), 331 (4.21)	82CB714, 80JCR(S)232
1-NH ₂ -2-CO ₂ -Bu ¹ -3-Ph	CH ₂ Cl ₂	250 (4.04), 260 sh (3.93), 331 (4.21)	82CB714
6-Br-2,3-(CO ₂ Me) ₂ -1-(<i>N</i> -pyrrolidinyl)	EtOH	235 (4.09), 281 (4.32), 349 (4.17)	74LA2110

Table 32 UV/visible spectra of substituted 3*H*-pyrrolizin-3-ones and related derivatives.

Substituent	3- <i>X</i>	Solvent	λ_{max} (nm) ($\log \epsilon$)	Ref.
H	O	EtOH	292 (5.0), 416 (3.7)	71CB2170
1-Me	O	EtOH	285 (3.9), 412 (2.8)	71CB2170
2-Ph	O	EtOH	256 (4.1), 293 (3.7), 440 (3.4)	71CB2170
1,2-Ph ₂	O		204 (4.58), 252 (4.31), 490 (3.5)	61AC(R)595
2-CO ₂ H	O	EtOH	230 (3.98), 302 (3.61), 352 (3.52), 442 (3.06)	80JCR(S)233
2-CO ₂ Et	O	EtOH	230 (4.13), 296 (3.71), 448 (3.16)	80JCR(S)233
2-CONEt ₂	O	EtOH	228 (3.83), 294 (3.94), 434 (3.26)	80JCR(S)233
1-Me ₂ N	O	EtOH	212 (4.13), 274 (4.18), 308 (4.1), 431 (3.32)	60JOC2049
5,7-Me ₂ -6-EtO ₂ C	O	cyclohexane	250 (4.2), 300 (3.78), 439 (3.22)	60JA2258
H	NOH	EtOH	293 (4.1), 303 (4.1), 372 (3.3)	68CB3843
2-CN-1-(<i>N</i> -piperidinyl)	NH	MeOH	239 (4.36), 287 (4.25), 317.5 (4.29), 445 (3.27)	74LA2110
2-CO-1-MeS	NH	MeOH	235 (4.0), 348 (3.92), 427 (4.0), 550 sh (2.67)	74LA2110
H	CPh ₂	EtOH	242 (4.11), 342 (4.23), 407 sh	67T2941
H	C(COPh)Ph	cyclohexane	228 sh (4.12), 248 (4.2), 333 (4.25)	67T2941
H	C(CO ₂ R)CH ₂ (CO ₂ R)	EtOH	209.5 (4.14), 230 sh, 322.5 (4.3), 420 (3.29)	73JOC1583
7-Me	C(CO ₂ R)CH ₂ (CO ₂ R)	EtOH	211.5 (4.18), 235 sh, 340 (4.31), 423 (3.44)	73JOC1583
1,2-Me ₂	C(CO ₂ R)CH ₂ (CO ₂ R)	EtOH	210 (4.1), 320 (4.09), 400 sh	73JOC1583

Table 33 UV/visible spectra of 1*H*-pyrrolizin-1-ones.

Substituent	Solvent	λ_{max} (nm) ($\log \epsilon$)	Ref.
H	CH ₂ Cl ₂	310 (3.9), 395 (2.4)	82CB706
2,3-Ph ₂		206 (4.5), 250 (4.24), 332 (4.08), 410 (2.95)	61AC(R)595
2-CO ₂ H	CH ₂ Cl ₂	240 (4.32), 330 (4.05), 422 (2.83)	82CB706
	TFA	330 (4.0), 453 (2.7)	82CB706
2-COCl	CH ₂ Cl ₂	258 (4.4), 330 (4.0), 420 (3.1)	82CB706
	TFA	312 (4.02), 330 sh (3.9), 454 (2.7)	82CB706
2-CO ₂ Et	CH ₂ Cl ₂	294 (3.87), 323 (3.95), 418 (2.79)	82CB706
	TFA	310 (3.94), 326 sh (3.92), 454 (2.58)	82CB706
2-Cl	MeOH	246 (3.3), 300 (3.97), 404 (2.5)	82CB706
	CH ₂ Cl ₂	240 (3.3), 315 (4.1), 418 (2.5)	82CB706
	TFA	322 (4.09), 430 (2.3)	82CB706
	CH ₂ Cl ₂ + SbCl ₅	544 (2.3)	82CB706
2-Br	CH ₂ Cl ₂	234 (3.3), 317 (4.1), 420 (2.6)	82CB706
2,5-Cl ₂	hexane	300 (3.93), 323 (3.95), 335 (3.75), 392 (2.61)	82CB706
	MeOH	310 (4.2), 400 (2.96)	82CB706
	CH ₂ Cl ₂	310 (3.9), 324 (3.94), 337 (3.75), 420 (2.55)	82CB706
	H ₂ SO ₄	340 (3.96), 354 (3.91), 372 (3.68), 500 (2.3)	82CB706
	CH ₂ Cl ₂ + SbCl ₅	354 (4.07), 376 (3.83), 500 (2.38)	82CB706
	TFA	332 (4.07), 426 sh (3.46), 442 (3.01)	82CB706

(ii) Pyrroles

For 2*H*-pyrroles, a band assigned to C=N generally falls in the range 1600–1665 cm⁻¹, with the corresponding C=C band falling between 1515 and 1575 cm⁻¹. Generally, these bands are between 90 and 125 cm⁻¹ apart. Additionally, a band between 1310 and 1335 cm⁻¹ has been assigned to the C—N absorption. Representative data for 2*H*-pyrroles are collected in Table 37. Similarly, the C=N band for 3*H*-pyrroles falls in the range 1610–1675 cm⁻¹, with the C=C band at 1545–1590 cm⁻¹. These bands are usually separated by anywhere from 20 to 70 cm⁻¹. Data for 3*H*-pyrroles is collected in Table 38.

An exhaustive assignment of the fundamental vibrations of pyrrole is found in Table 39. A number of computational papers have also appeared on predictions and reassignments of these vibrations <88JPC1739, 93KGS651>. However, many of these bands are indistinguishable in the conventional infrared spectrum. Pyrrole shows five major bands in the infrared at approximately 3600,

Table 34 Ultraviolet spectroscopic data for indole derivatives (in EtOH).

Substituent	λ_{max} (nm) ($\log \epsilon$)
None	216 (4.54), 266 (3.76), 270 (3.77), 276 (3.76), 278 (3.76), 287 (3.68)
1-Me	219 (4.54), 275 (3.77), 282 (3.78), 287 (3.74), 293 (3.66)
2-Me	220 (4.53), 272 (3.85), 277 (3.84), 281 (3.84), 289 (3.73)
3-Me	222 (4.50), 275 (3.73), 281 (3.78), 290 (3.69)
5-Me	219 (4.48), 269 (3.75), 277 (3.72), 284 (3.69), 295 (3.50)
3-CH ₂ CO ₂ H	222 (4.51), 273 (3.77), 279 (3.78), 294 (3.71)
5-NO ₂	254 (4.20), 264 (4.24), 322 (3.90)
6-NO ₂	247 (4.03), 324 (3.92), 358 (3.88)
1,2-Me ₂	223 (4.55), 276 (3.85), 282 (3.88), 291 (3.81)
1,3-Me ₂	225 (4.50), 278 (3.68), 288 (3.72)
2,3-Me ₂	227 (4.52), 276 (3.82), 282 (3.85), 290 (3.79)
2,5-Me ₂	221 (4.47), 272 (3.86), 276 (3.85), 282 (3.83), 293 (3.69)
1,2,3-Me ₃	230 (4.54), 279 (3.77), 286 (3.82), 293 (3.80)

Table 35 UV spectral data for isoindoles.

Substituent(s)	Solvent	λ_{max} (nm) ($\log \epsilon$)	Ref.
None	hexane	230, 263.5, 268.5, 275, 286.5, 294, 300, 306.5, 312.5, 320, 326.5, 335	72CC393, 73JCS(P1)1432
1-Ph	EtOH	272 (3.86), 282 (3.92), 314 (3.87), 325 (3.99), 357 (4.10)	66T1011
2-(<i>p</i> -Me)Ph	MeOH	242 (4.50), 288 (3.94), 298 (3.94), 337 (3.62)	64AG(E)639, 66AG(E)967
1,3,4,7-Me ₄	EtOH	249, 289, 298, 347	68JCS(C)3036
1-CN	EtOH	222 (4.52), 242 (4.08), 249 (4.05), 256 (4.04), 320 (3.98), 327 (4.04), 341 (3.89)	72AJC607
1-CO ₂ Me	EtOH	228 (4.32), 256 (4.17), 262 (4.23), 336 (4.22), 350 (4.19)	76G65
1,3-(CO ₂ Me) ₂	MeOH	223 (4.25), 248 (4.58), 345 (4.35), 362 (4.37)	70AC(R)765
<i>N</i> -Me	EtOH	227 (4.5), 250 (3.5), 277 (3.0), 322 (3.5)	87KGS1629
<i>N</i> -Ph	EtOH	213 (4.2), 244 (4.4), 294 (3.7), 345 (3.4)	87KGS1629
<i>N</i> -(<i>p</i> -Br)Ph	EtOH	213 (4.4), 256 (4.5), 294 (4.2), 345 (3.6)	87KGS1629

Table 36 UV spectra of carbazoles.

Substituent(s)	λ_{max} (nm) ($\log \epsilon$)	Ref.
None	233 (4.51), 257 (4.18), 293 (4.10), 322 (3.47), 336 (3.40)	77FOR299
<i>N</i> -Me	236 (4.67), 261 (4.37), 293 (4.25), 330 (3.57), 336 (3.49), 344 (3.60)	77FOR299
1-Me	214 (4.42), 235 (4.67), 259 (4.29), 298 (4.22), 322 (3.61), 336 (3.43)	77FOR299
2-Me	235 (4.57), 259 (4.18), 296 (4.12), 322 (3.55), 334 (3.40)	77FOR299
3-Me	236 (4.40), 260 (4.03), 296 (4.03), 328 (3.31), 342 (3.31)	77FOR299
1-MeO	240 (4.59), 252 (4.39), 286 (3.84), 320 (3.46)	77FOR299
2-MeO	235 (4.58), 257 (4.24), 303 (4.09)	77FOR299
3-MeO	228 (4.49), 251 (4.18), 262 (4.08), 300 (4.24), 314 (4.24), 329 (3.39), 342 (3.51)	77FOR299
4-MeO	224 (4.46), 243 (4.67), 286 (4.16), 327 (3.63), 332 (3.76)	77FOR299
3-MeO, 2-Me	232 (4.48), 263 (4.05), 303 (4.21), 322 (3.46), 336 (3.50)	77FOR299
1-MeO, 3-CHO (murrayanine)	238 (4.47), 247 (4.30), 274 (4.50), 289 (4.56), 335 (4.16)	77FOR299
2-MeO, 3-Me, 6-MeO (glycozolidine)	233 (4.47), 237 (4.47), 260 (4.08), 309 (4.20)	77FOR299
3-Me, 6-MeO (glycozoline)	227 (4.16), 252 (4.16), 264 (4.06), 304 (4.17)	77FOR299

1075, 1050, and 1025 cm⁻¹, and a broad band centered at 730 cm⁻¹. Additionally, several small bands are visible in the range 1400–1600 cm⁻¹. *N*-Substitution usually reduces the intensity of the 3600 cm⁻¹ band, while heightening the bands between 900 and 1500 cm⁻¹. The band at 730 cm⁻¹ is absent in tetrasubstituted pyrroles.

Table 37 IR data (cm^{-1}) for 2*H*-pyrroles.

R^1	R^2	R^3	R^4	R^5	C=N	C=C	Others	Medium	Ref.
Me	Pr ⁿ	H	H	Me	1615	1525		film	71TL3923
Me	Me	Me	H	Me	1640	1552	1320	film	B-90MI 201-02
Me	Me	Me	Me	Me	1665	1570		CCl ₄	70CC142
Me	Me	H	H	Ph	1610			CCl ₄	76C453
Me	Ph	Me	H	Me	1636	1557	1334	film	B-90MI 201-02
Me	Me	Me	H	Ph	1626	1534	1310	film	B-90MI 201-02
Me	CHCl ₂	H	H	Me	1622	1532	1349	film	67G685
Me	CHCl ₂	Me	Me	Me	1661	1573	1330	Nujol	67G685
Ph	CHCl ₂	Me	Me	Ph	1654	1545	1351	Nujol	67G685
CF ₃	CF ₃	H	H	Bu ^t	1610			film	76CB1984
CF ₃	CF ₃	H	H	Ph	1610			KBr	76CB1984
CF ₃	CF ₃	H	Ph	Bu ^t	1632			film	76CB1984
CF ₃	CF ₃	Ph	H	Bu ^t	1620			film	76CB1984
Me	Me	H	H	CO ₂ Me	1597	1516	1712	CHCl ₃	85JCR(S)238
Me	CO ₂ Et	Me	H	Me	1635	1555	1735	film	B-90MI 201-02
Me	CO ₂ Et	Me	Me	Me	1655	1565	1725	film	B-90MI 201-02
Me	CO ₂ Et	Me	H	Ph	1630	1530	1735	film	B-90MI 201-02
CF ₃	CF ₃	H	CO ₂ Me	H	1635		1735	film	87S924
CF ₃	CF ₃	CO ₂ Me	CO ₂ Me	H	1645		1740	film	87S924
Bu ^t	OOH	H	H	Bu ^t	1625	1550	3100	Nujol	72TL1337
Bu ^t	OOH	Bu ^t	H	Bu ^t	1630	1560	3100	Nujol	72TL1337
Ph	MeO	Ph	Ph	Ph	1630		2830	CHCl ₃	69BSF1667
Me	Me	MeO	H	Me	1660	1550			71KGS1530
Me	Me	Et ₂ N	H	Me	1610	1540			71KGS1530
Me	Me	Cl	H	Ph	1612			CCl ₄	76C453
H	H	OH	Ph	Me ₂ N	1603	1563	3205	KBr	69T5721
H	Me	OH	Me	Et ₂ N	1613	1553	3155	KBr	69T5721
Me	Me	OH	Me	Et ₂ N	1613	1538	3130	KBr	68CB308
Cl	Cl	Cl	Cl	Cl	1610	1520	1240	CCl ₄	80JOC435

Table 38 IR data (cm^{-1}) for 3*H*-pyrroles.

R^1	R^2	R^3	R^4	R^5	C=N	C=C	Other	Medium	Ref.
Me	Me	Me	H	Me	1630	1580		film	90JCS(P1)457
Me	Me	Et	Me	H	1615	1575		CCl ₄	70CC142
Me	Me	Me	Me	Me	1650	1580		CCl ₄	70CC142
Me	Me	Et	Me	Me	1650	1580		CCl ₄	70CC142
H	Me	Me	H	Ph	1612	1550		film	90JCS(P1)457
Ph	Me	Me	H	H	1675			film	85KGS1573
Me	Me	Me	H	Ph	1640			film	90JCS(P1)457
Bu ^t	Me	CO ₂ Et	H	Me	1635	1555	1735	film	B-90MI 201-02
Me	Me	CO ₂ Et	Me	Me	1650	1590	1730	film	B-90MI 201-02
Ph	Me	CO ₂ Et	H	Me	1620	1545	1735	film	B-90MI 201-02

(iii) *Indoles*

Extensive vibrational data for indole is included in Table 40 <88JCP(88)7295>. Again, some of these bands are indistinguishable in everyday laboratory spectroscopy. The major bands for indole are 3450 cm^{-1} , several bands between 1250 and 1500 cm^{-1} , and a broad band around 740 cm^{-1} . Indoles are very sensitive to crystalline modifications, giving different band shifts for the same batch of a given indole. The substitution pattern can be deduced from the bands between 665 and 910 cm^{-1} , with an unsubstituted pyrrolic ring giving a band in the $715\text{--}740\text{ cm}^{-1}$ range.

(iv) *Isoindoles*

Infrared data for several isoindole derivatives is included in Table 41. Probable assignments are as follows, the band near 800 cm^{-1} should be assigned to the out-of-plane vibrations of the C—H

Table 39 Computed and experimental vibrational spectra (cm^{-1}) for pyrroles.

Number	Symmetry	Assignment ^a	Computed				Experimental		
			ν^a	ν^a	I^a	ν^b	Raman (liquid ^c)	IR (liquid ^c)	IR (gas ^c)
1	B_2	N—H wag	611	474	153.7	474	568	560	474
2	A_2	ring torsion	608	605	0.0	613	618		
3	B_2	ring torsion	612	615	23.6	605			602
4	A_2	C—H wag	728	707	0.0	712	712		
5	B_2	C—H wag	735	716	104.6	722	735	733	720
6	B_2	C—H wag	865	832	3.7	830	838	834	826
7	B_1	ring deformation	885	868	2.5	877	868	865	863
8	A_2	C—H wag	897	869	0.0	840			
9	A_1	ring deformation	895	880	1.3	879	878	877	880
10	A_1	C—H deformation	1029	1021	41.8	1019	1014	1010	1018
11	B_1	C—H deformation	1050	1046	22.2	1066	1047	1046	1049
12	A_1	C—H deformation	1076	1071	4.0	1075	1076	1072	1074
13	B_1	N—H deformation	1156	1144	6.5	1144		1130	1134
14	A_1	C—C, C—N stretch	1163	1161	4.0	1148	1144	1140	1148
15	B_1	C—H deformation	1294	1284	0.6	1308	1284	1284	1287
16	A_1	C—H, C—N stretch	1382	1386	11.6	1391	1380	1383	1391
17	B_1	C—C, C—N stretch	1428	1416	3.6	1447	1415	1415	1424
18	A_1	C—C, C—N stretch	1470	1463	7.4	1474	1460	1467	1470
19	B_1	C—C, C—N stretch	1549	1551	13.3	1544	1545	1568	1548
20	B_1	C—H stretch	3131	3113	4.7	3134	3102	3100	3116
21	A_1	C—H stretch	3145	3126	5.4	3118	3110	3109	3125
22	B_1	C—H stretch	3160	3142	7.4	3161		3128	3140
23	A_1	C—H stretch	3171	3153	0.6	3148	3133		3148
24	A_1	N—H stretch	3620	3257	85.4	3529	3407	3400	3527

^a<86MP401>. ^b<86SA(A)919>. ^c<83AQ(A)557>.

bonds, and the 1000 cm^{-1} bands should be assigned to the breathing stretching vibrations of the isoindole rings <87KGS1629>. The N—H bond has a stretching band at $3400\text{--}3500\text{ cm}^{-1}$, and usually there are one or two bands in the 1600 cm^{-1} range.

(v) Carbazoles

Carbazoles show some characteristics of imino compounds, but the N—H band is dependent on phase, owing to stronger intermolecular associations in the solid phase. Carbazole itself shows two major bands at $\nu_{\text{max}} = 856$ and 915 cm^{-1} . Again, formylcarbazoles have distinctive spectra, with the formyl carbonyl at approximately 1680 cm^{-1} , and with chelation causing shifts to higher wavelength in many cases. A carbonyl band at $1630\text{--}1645\text{ cm}^{-1}$ is also considered indicative of a 3-formyl group chelated to a 2-hydroxy group, and is a common feature of many alkaloids <91FOR71>. Additionally, a study has been reported on the hydrogen bonding of carbazole to tertiary amides <90JST(219)239>. The binary mixtures of carbazole and *N*-methylpyrrolidinone, *N,N*-dimethylacetamide, or *N*-methylcaprolactam all show distinctive shifts of the carbazole N—H band from 3490 cm^{-1} (in tetrachloroethene) to the 3320 cm^{-1} range.

2.01.3.6 Mass Spectrometry

For a discussion of the basic mass spectral fragmentation of several of these classes of compounds, the reader is encouraged to consult the treatment by Chadwick <84CHEC-I(3)155>.

(i) Pyrroles

A number of *N*-aryl-2-formylpyrroles have been examined <89KGS180>. Typical fragmentation is depicted in Figure 3. For the majority of the compounds studied ($X = p\text{-NO}_2$, $m\text{-NO}_2$, $p\text{-Cl}$, $m\text{-Cl}$,

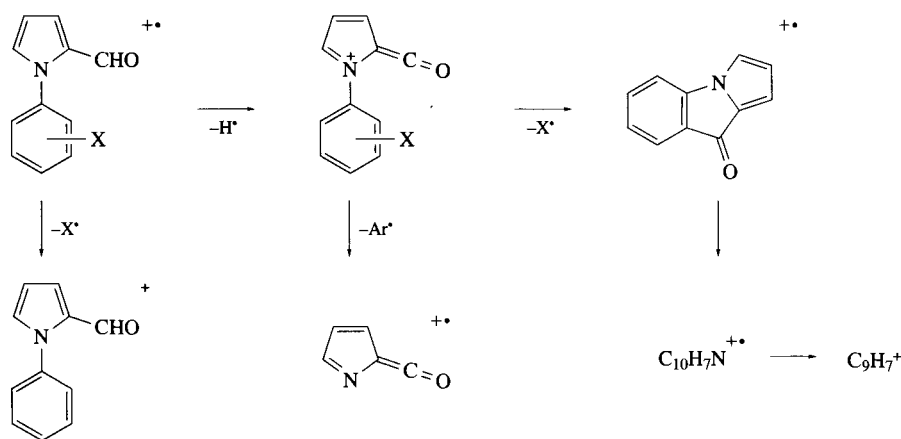
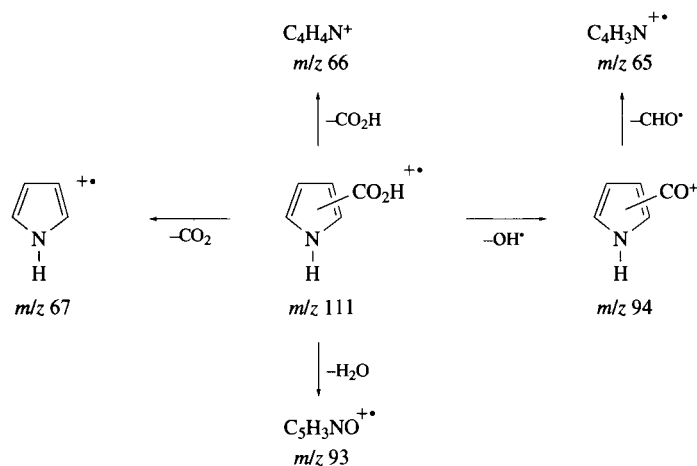
Table 40 Computed and experimental vibrational spectra (cm^{-1}) for indoles.

<i>Vapor IR</i>	<i>Liquid IR</i>	<i>Liquid Raman</i>	<i>AM1</i>	<i>Symmetry</i>
208	230	229	224	A''
240	254	257	231	A''
405	398		256	A''
		400	413	A'
	422	428	437	A''
	460–525	460		A'
	460–525	485		A'
	460–525	510		A'
	543	544	554	A'
	575	575	560	A''
	608		606	A'
625	620	610	620	A''
715	720	729	760	A''
		761	774	A'
738	740		782	A''
762	768		797	A''
800	795	795		A'
800	795		828	A''
		845		A'
	870	869	869	A'
900	895	897	884	A'
	860	860	892	A''
	930		922	A''
		968	935	A''
	1003	1005		A'
1015	1010	1011	1045	A'
1068	1062	1068	1060	A'
1082	1090	1095	1098	A'
1122	1120	1120	1139	A'
	1148	1150	1167	A'
	1190	1195		A'
1205		1205	1212	A'
1245	1245	1247	1235	A'
1275	1276	1279	1273	A'
	1300	1300	1310	A'
	1332	1335		A'
1348	1352	1353	1364	A'
	1365	1368		A'
1410	1410	1412	1402	A'
1458	1455	1455	1440	A'
	1488	1489	1483	A'
	1509	1510	1523	A'
	1576	1578	1566	A'
	1590			A'
1620	1615	1617	1620	A'
	3060	3068	3053	A'
	3060	3068	3061	A'
	3060	3068	3069	A'
	3080	3083	3077	A'
	3080	3083	3078	A'
3140	3150	3155	3132	A'
3520	3420	3400	3548	A'

and *p*-Br), the molecular ion was found to be the base peak. A comprehensive study on the electron impact fragmentations of pyrrole-mono-, -di-, and -tricarboxylic acids has been reported (91OMS(26)972). Both pyrrole 2- and 3-carboxylic acids behave similarly and lead to fragmentations as rationalized in Figure 4. Fragmentations of the pyrrole-2,3-, -3,4-, and -2,5-dicarboxylic acids are depicted in Figure 5, and those for pyrrole-2,3,5-tricarboxylic acid are summarized in Figure 6. As expected, the loss of CO_2 forms the base peak in all cases, with the other major fragments forming from loss of either water or hydroxide.

Table 41 Infrared data for isoindoles.

Substituent	$\nu_{\max}$ (cm ⁻¹)	Ref.
None	3350, 3110, 3070, 3030, 2900, 2850, 1460, 1360, 1230, 1110, 1065, 1020, 985, 740	73JCS(P1)1432
<i>N</i> -Bu	3120, 3060, 3030, 2960, 2935, 2875, 1660, 1550, 1366, 1330, 1158, 1140, 758	73JCS(P1)1432
<i>N</i> -Me	3110, 3060, 3030, 2940, 2850, 1640, 1625, 1545, 1520, 1477, 1413, 1365, 1335, 1237, 1203, 1023, 815, 760	87KGS1629
<i>N</i> -Ph	3130, 3060, 3030, 2910, 2860, 1630, 1595, 1540, 1505, 1460, 1435, 1365, 1340, 1220, 1200, 1050, 1030, 795, 755	87KGS1629
<i>N</i> -(<i>p</i> -Me)Ph	3120, 3060, 3030, 2910, 2860, 1630, 1617, 1560, 1520, 1485, 1440, 1367, 1345, 1218, 1200, 1055, 1022, 825, 760	87KGS1629
<i>N</i> -(<i>p</i> -Br)Ph	3130, 3065, 3030, 2910, 2860, 1630, 1590, 1545, 1503, 1440, 1415, 1365, 1345, 1214, 1200, 1053, 1013, 830, 765	87KGS1629

**Figure 3** Proposed mass spectral fragmentations of *N*-aryl pyrroles.**Figure 4** Proposed mass spectral fragmentations of pyrrole carboxylic acids.*(ii) Indoles*

Electron impact mass spectrometry has been used to prove the intermediacy of a 2-carbonyl-2*H*-indole intermediate in the fragmentation of indole-2-acid derivatives <89H(29)629>. The authors postulate that thermal elimination of RH from acid derivatives generates a ketene (*m/z* 143) as shown in Figure 7, which is then followed by dimerization (*m/z* 286) before electron impact.

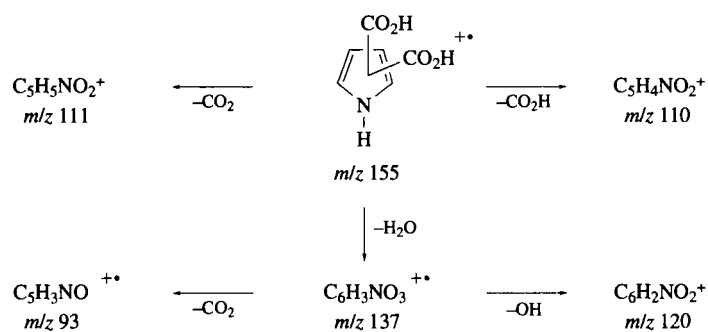


Figure 5 Proposed mass spectral fragmentations of pyrrole dicarboxylic acids.

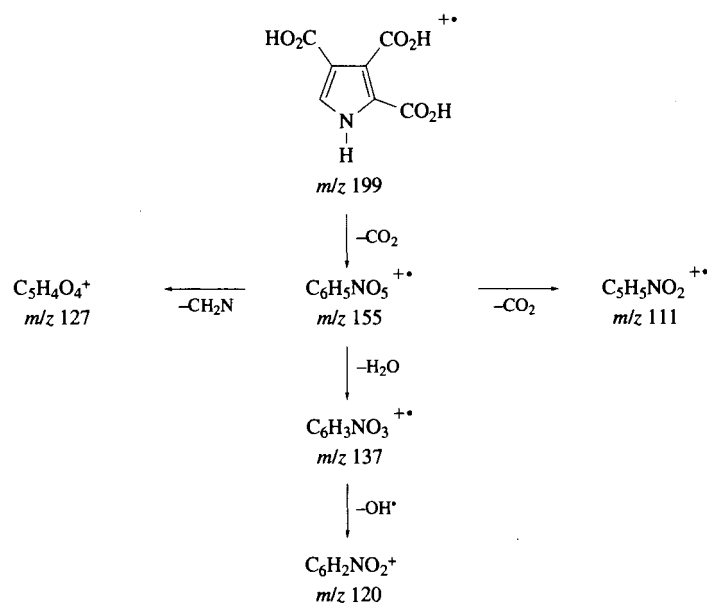


Figure 6 Proposed mass spectral fragmentations of 2,3,4-pyrrole tricarboxylic acid.

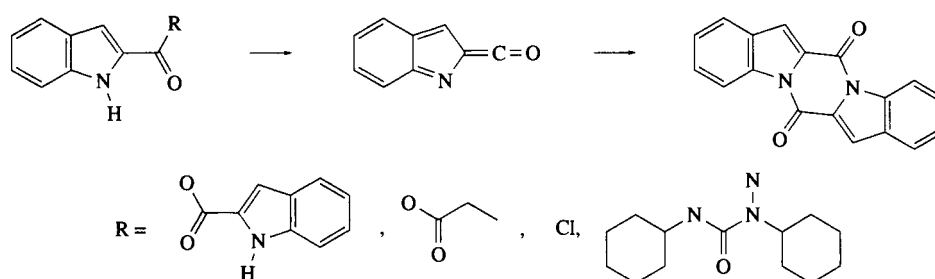


Figure 7 Dimerization of indole-2-acid derivatives.

(iii) Isoindoles

The principal features of the electron impact mass spectrum of isoindole are shown in Figure 8. The molecular ion is the base peak (m/z 117.0575), followed by HCN extrusion to give the benzyl radical cation (m/z 90.0467). Other fragments arise from the molecular ion in another path, beginning with hydrogen loss to give the *o*-cyanobenzyl cation (m/z 116), which fragments further by sequential loss of HCN followed by ethene <73JCS(P1)1432>.

A mass spectral study of several other isoindole derivatives has been reported <90KGS923>. Rationalizations of the fragmentation patterns of *N*-methyl- and *N*-phenylisoindole are included in

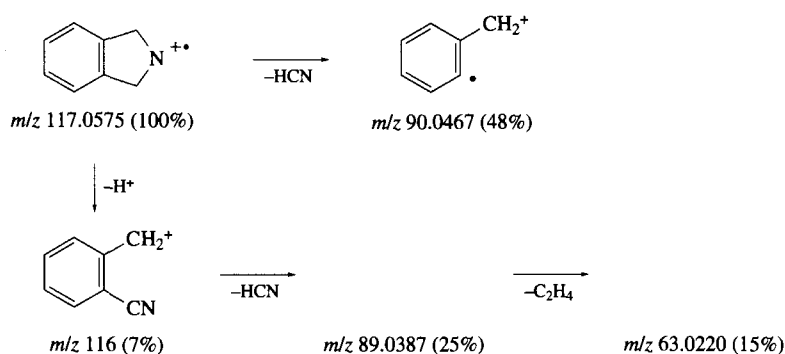


Figure 8 Proposed mass spectral fragmentations of isoindole.

Figures 9 and 10, respectively. The principal fragments of *N*-methylisoindole are those arising from loss of a methyl radical followed by loss of cyanide radical or hydrogen cyanide, or from loss of hydrogen radical to give an azafulvene-like cation, followed by rearrangement to the isoquinolinium cation and further fragmentation to the styrene cation and loss of ethene to a phenyl cation. The corresponding fragmentation pattern for *N*-phenylisoindole is much more complex and is summarized in Figure 10.

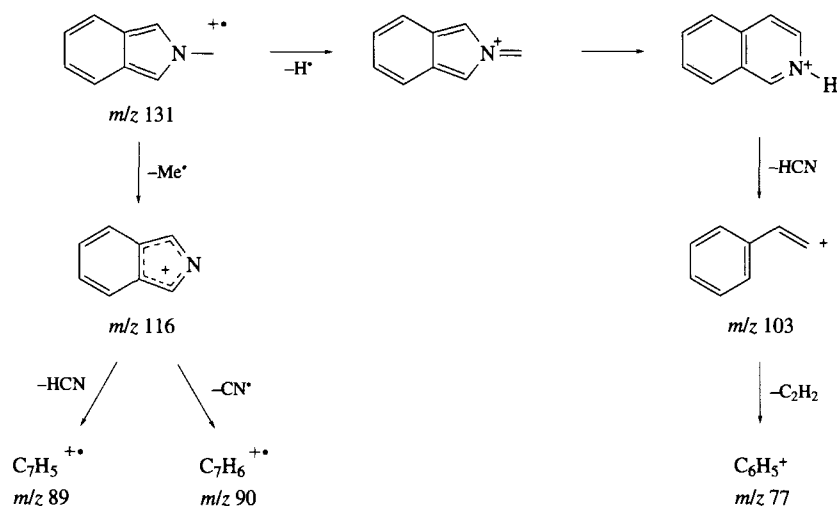


Figure 9 Proposed mass spectral fragmentations of *N*-methylisoindole.

(iv) Carbazoles

The electron impact mass spectra of a number of nitrocarbazoles have been analysed <87OMS(22)39>. Typical fragmentation is rationalized in Figure 11. Major fragmentation begins with the loss of NO_2 , which then brings fragmentation identical to that of the bare carbazole nucleus. Also observed are fragments derived from fission of the nitro group. *N*-Alkyl-1-nitrocarbazoles also show fragments arising from oxygen transfer from the nitro group to the *N*-substituent.

2.01.3.7 Other Structural Methods

Electronic transition energies, dipole strengths, and Faraday *B* values for carbazole are collected in Table 42 <87SA(A)1431>. The major bands were assigned, and compared with data calculated by

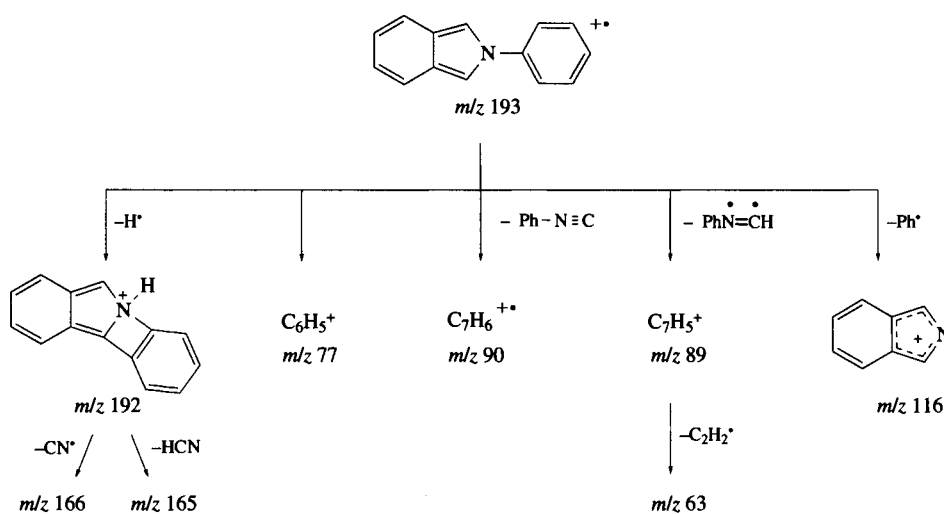


Figure 10 Proposed mass spectral fragmentations of *N*-phenylisoindole.

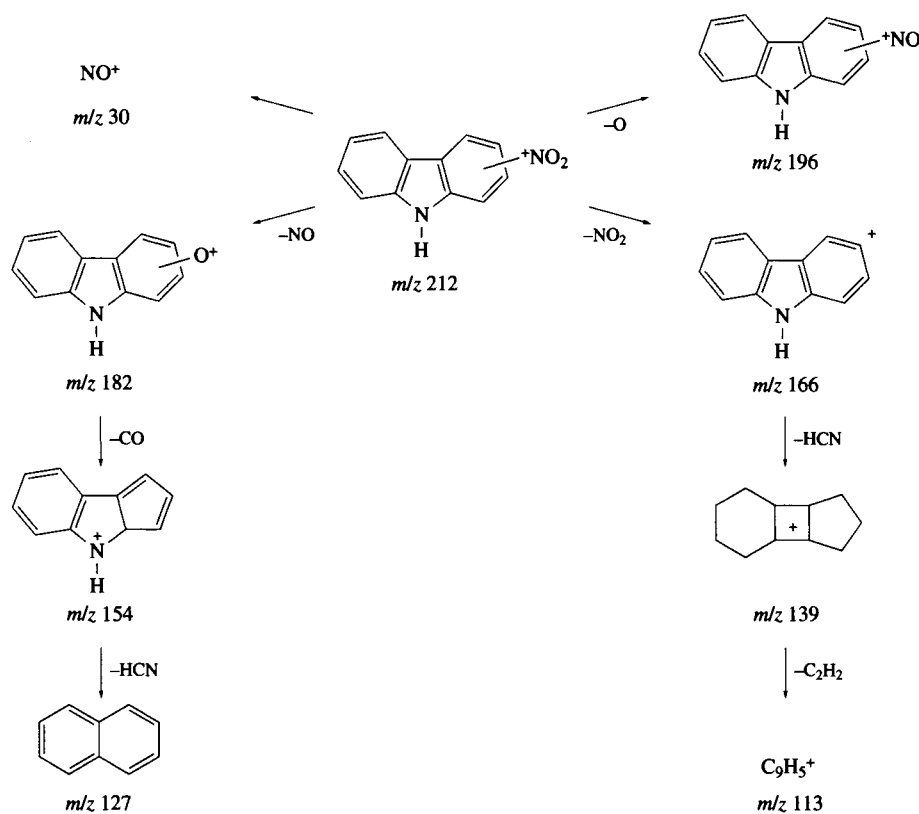


Figure 11 Proposed mass spectral fragmentations of nitrocarbazoles.

semiempirical SCF π -MO CI methods. The computational data showed very poor correlation with the experimental values for carbazole (as well as dibenzofuran and dibenzothiophene).

Photoelectron spectroscopy has been used to give the vertical ionization potentials for a number of pyrrole derivatives; these are included in Table 43 <B-90MI 201-01>. Additionally, experimental dipole moments for a number of substituted pyrroles are summarized in Table 44 <B-90MI 201-01>.

Table 42 Electronic spectral data for carbazoles.

Transition energy (10^3 cm^{-1})	Dipole strength (D^2) ^a	Faraday <i>B</i> value ($10^{-3} \mu_B \text{ D}^2 \text{ cm}^{-1}$) ^b
31.5	3.01	−0.238
34.4	10.7	0.133
39.2	24.8	−0.052
40.9	30.8	1.13
43.0	21.7	0.440
46.0	49.6	−0.104

^a $1 \text{ D} = 3.336 \times 10^{-30} \text{ C m}$. ^b $\mu_B = \text{Bohr magneton} = 9.274\,0154 \times 10^{-24} \text{ J T}^{-1}$.

Table 43 First and second ionization potentials for pyrrole derivatives.

Substituents					Ionization potential (eV)		Ref.
1	2	3	4	5	1st	2nd	
Me	H	H	H	H	8.09 ± 0.01	8.83 ± 0.03	70DOK(192)131
Me	H	H	H	H	7.95 ± 0.05	8.80 ± 0.05	70ANC1064
Bu ⁿ	H	H	H	H	7.87 ± 0.02		70DOK(192)131
H	Me	H	H	H	7.78 ± 0.01	8.88 ± 0.03	70DOK(192)131
H	Me	H	H	H	8.01		71JCS(B)1585
H	Et	H	H	H	7.97		71JCS(B)1585
H	Bu ^t	H	H	H	7.95		71JCS(B)1585
H	Me	H	Me	H	7.54 ± 0.02	8.52 ± 0.03	70DOK(192)131
H	H	Me	H	H	7.90 ± 0.02	8.62 ± 0.03	70DOK(192)131
H	CO ₂ Me	H	H	H	8.65		71JCS(B)1585
H	COMe	H	H	H	8.72		71JCS(B)1585
H	CHO	H	H	H	8.93		71JCS(B)1585
H	COCF ₃	H	H	H	9.18		71JCS(B)1585
H	NO ₂	H	H	H	9.30		71JCS(B)1585
H	Me	CO ₂ Et	Me	H	7.95	8.83	81JCS(P2)656
H	Me	CO ₂ Et	Me	Me	7.82	8.90	81JCS(P2)656
H	Me	CO ₂ Et	Me	COMe	8.23	9.19	81JCS(P2)656
H	Me	CO ₂ Et	Me	CO ₂ Et	8.15	9.03	81JCS(P2)656
H	Me	CO ₂ Et	Me	Cl	7.94	9.08	81JCS(P2)656
H	Me	CO ₂ Et	Me	NO ₂	8.78	9.62	81JCS(P2)656
H	CO ₂ Et	Me	H	Me	7.91	8.95	81JCS(P2)656
H	CO ₂ Et	Me	Me	Me	7.71	8.55	81JCS(P2)656
H	CO ₂ Et	Me	COMe	Me	8.26	9.13	81JCS(P2)656
H	CO ₂ Et	Me	CO ₂ Et	Me	8.15	9.03	81JCS(P2)656
H	CO ₂ Et	Me	Cl	Me	8.03	8.87	81JCS(P2)656
H	CO ₂ Et	Me	Br	Me	8.03	8.79	81JCS(P2)656
H	CO ₂ Et	Me	NO ₂	Me	8.76	9.67	81JCS(P2)656

2.01.4 THERMODYNAMIC ASPECTS

2.01.4.1 Aromaticity

The concept of “heteroaromaticity” has been the subject of much intensive research from the mid 1980s, less concerned with qualitative aromaticity and more concerned with quantitative aromaticity.

Katritzky *et al.* have published a comprehensive review of the various methods for quantifying heteroaromaticity and their respective problems reported before 1991 (91H(32)127). The classical methods for determining the resonance energy of benzene are hydrogenation and combustion. For heterocyclic compounds, hydrogenation suffers from poisoning of the catalyst by nitrogen-containing fragments. On the other hand, combustion analyses often suffer from large errors. Benzene has a heat of combustion of 36–37 kcal mol^{−1} (151–155 kJ mol^{−1}), while combustion of pyrrole leads to values ranging from 14 to 31 kcal mol^{−1} (56–130 kJ mol^{−1}), making true quantification by this method nearly impossible. A more reproducible resonance energy is found by comparing the basicity of the heteroaromatic with that of a similar nonaromatic compound. Basicity-based calculations of resonance energy are depicted in Figure 12. It was also noted that ring current

Table 44 Dipole moments (μ) of substituted pyrroles.

Substituent					$\mu(\text{D})^a$	Ref.
1	2	3	4	5		
Me	H	H	H	H	1.96	71JCS(B)1811
H	Me	H	H	H	1.95	71JCS(B)1811
H	Me	H	Me	H	1.75	52CA2360
H	Me	H	H	Me	2.03	52CA2360
Me	Me	H	H	Me	2.08	52CA2360
H	CHO	H	H	H	2.18	71JCS(B)1811
Me	CHO	H	H	H	2.84	71JCS(B)1811
H	H	CHO	H	H	4.96	72CR(C)462
H	COMe	H	H	H	1.82	71JCS(B)1811
H	H	COMe	H	H	5.03	71JCS(B)1811
H	COPh	H	H	H	1.81	71JCS(B)1811
H	CO ₂ Me	H	H	H	1.77	71JCS(B)1811
H	CONMe ₂	H	H	H	2.07	71JCS(B)1811
H	H	CO ₂ Me	H	H	3.60	71JCS(B)1811
H	NO ₂	H	H	H	4.33	71JCS(B)1811
H	I	I	I	I	2.54	71JCS(B)1811
H	CN	H	H	H	3.76	72CR(C)462
Me	CN	H	H	H	4.17	71JCS(B)1811
H	Ph	H	H	H	1.66	71JCS(B)1811
Ph	Ph	H	Ph	Ph	2.04	85ZOR203
H	2-Furyl	H	H	H	1.58	81JCS(P2)127
H	2-Thienyl	H	H	H	1.82	81JCS(P2)127

^a 1 D = 3.336×10^{-30} C m.

quantification of aromaticity yields such varying results as to be highly questionable. A number of the relevant aromaticity parameters (Jugl ring current (RC), Pozharskii indices (ΔN), Dewar resonance energies (both from experimental (DRE) and AM1 optimized geometries (DRE')), Hess and Schaad resonance energies (HSRE), heats of formation (ΔH_f experimental and ΔH_{fcal} calculated by AM1), molar magnetic susceptibility (χ_m), diamagnetic susceptibility (Λ), and ^{15}N chemical shifts are included in Table 45. In the model studies, it was found that the magnetic parameters had little predictive power, while the energetic parameters were of much higher utility.

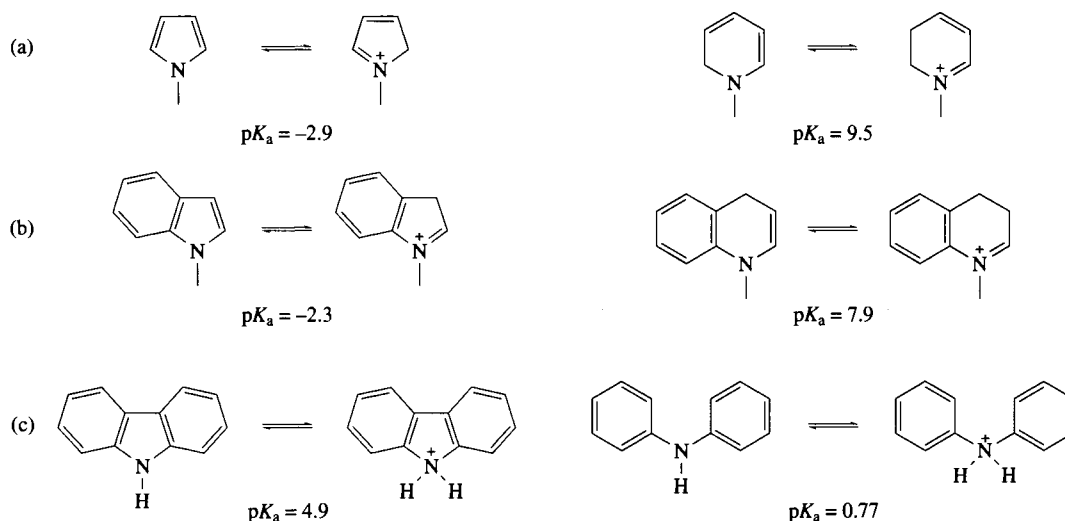


Figure 12 Basicity-based quantification of aromaticity. (a) $\Delta \text{p}K_a = 12.4$, $\Delta \Delta H^\circ = 1.1$ p K_a , delocalization energy = 14 kcal mol⁻¹ (58.6 kJ mol⁻¹). Resonance energy of 1-methyl-1,2-dihydropyridine is 6 kcal mol⁻¹ (25.1 kJ mol⁻¹), therefore pyrrole resonance energy is 6 + 14 = 20 kcal mol⁻¹ (83.7 kJ mol⁻¹). (b) $\Delta \text{p}K_a = 10.2$, $\Delta \Delta H^\circ = 11$ kcal mol⁻¹ (46.0 kJ mol⁻¹). Resonance energy of the hydroquinoline is 42 kcal mol⁻¹ (175.7 kJ mol⁻¹), therefore the resonance energy of 1-methylindole is 53 kcal mol⁻¹ (221.7 kJ mol⁻¹). (c) $\Delta \text{p}K_a = 4.1$, $\Delta \Delta H^\circ = 5$ kcal mol⁻¹ (20.9 kJ mol⁻¹). Resonance energy of diphenylamine is 78 kcal mol⁻¹ (326.4 kJ mol⁻¹), therefore the resonance energy of carbazole is 78 + 5 = 83 kcal mol⁻¹ (347.3 kJ mol⁻¹).

Table 45 Aromaticity data^a for selected heterocycles.

Compound	RC	ΔN	DRE <i>exp.</i>	DRE' AM1	HSRE	ΔH_f <i>exp.</i>	ΔH_{fcal} AM1	χ_m	Λ	$\delta^{15}N$ (ppm)
Benzene	1.774	0	3.77	3.84	0.0065	19.8	22.0	54.8	17.9	
Thiophene	1.533	0.25	1.08	0.096	0.032	27.6	40.1	57.4	17.8	
Furan	1.105	0.47	0.72	0.63	0.007	-8.3	3.0	43.1	13.9	
Pyrrole	1.344	0.24	1.40	2.15	0.039	25.9	40.1	47.6	14.5	-224.6
Benzo-furan	1.059	0.33	2.03	0.76	0.036	6.5	20.8	79.8	31.8	
Indole	1.311	0.23	2.38	1.47	0.047	44.6	55.2	85.0	33.1	-253.6
Isoindole	1.378	0.22	1.16	0.76	0.029	56.0	61.7			-218.0
Benzothiophene	1.515	0.18	2.48	1.18	0.044	40.0	53.9	93.3	34.9	

^aRC, Julg ring current; ΔN , Pozharskii index; DRE, Dewar resonance energy (experimental); DRE', Dewar resonance energy from AM1 optimized geometry; HSRE, Hess-Schaad resonance energy; ΔH_f , heat of formation (experimental); ΔH_{fcal} , heat of formation calculated by AM1; χ_m , molar magnetic susceptibility; Λ , diamagnetic susceptibility; $\delta^{15}N$, ^{15}N chemical shift.

Bird has introduced unified aromaticity indices for five- and six-membered heterocycles and fused systems, allowing for realistic direct comparisons of aromaticity (92T335). The original indices (85T1409) were scaled relative to the parent carbocycles (cyclopentadienyl anion and the indenyl anion for 5 and 6,5 systems, respectively, with benzene representing an I_A of 100) to yield a quantitative scale which holds well in a linear relationship with the dilution shift parameter (A) and, more importantly, in a linear relationship with experimental resonance energies. The aromaticity indices for a variety of heterocycles are summarized in Table 46. As can be seen, pyrrole is slightly more aromatic than thiophene and much more so than furan, while indole and isoindole are much more aromatic than their sulfur- and oxygen-containing analogues. Of particular note is the energetic basis of this scale, with one I_A unit corresponding to 0.02β .

Table 46 Unified aromaticity indices.

Compound	I_A
Furan	53
Thiophene	81.5
Pyrrole	85
Benzo[b]furan	94
Benzene	100
Indolizine	115
Benzo[b]thiophene	119
Indole	146
Isoindole	150

2.01.4.2 Tautomerism

For a basic discussion of tautomerism of pyrrolic heterocycles, the reader is encouraged to consult the first edition (84CHEC-I(3)155).

Equilibrium and rate constants for the keto-enol tautomerization of 3-hydroxy indoles and pyrroles are included in Table 47 (86TL3275). The pyrroles ketonize substantially (10^3 – 10^4 times) faster than their sulfur or oxygen analogues, and faster still than the benzo-fused systems, indole,

Table 47 Rate and equilibrium constants for ketonization of hydroxy-heterocyclic compounds at 25°C.

Compound	k_{H^+} (mol ⁻¹ s ⁻¹)	K_{enol} (H ₂ O)	K_{enol} (DMSO)
3-Hydroxypyrrole	2.38×10^4	0.13	$> 10^2$
1-Me-3-hydroxypyrrole	9.65×10^3	0.18	$> 10^2$
3-Hydroxyindole	3.44	0.086	28.3
1-Me-3-hydroxyindole	5.82	0.303	11.4
1-Hydroxyindene	9.03×10^2	1.85×10^{-8}	$< 2 \times 10^{-2}$

benzofuran, and benzothiophene. However, in contrast to the 3-hydroxyfuran, detectable amounts of the aromatic hydroxypyrrole and hydroxythiophene were found to be present at equilibrium where the furan system was completely ketonized. Attempts at preparing the corresponding 2-hydroxy heterocycles were unsuccessful. It was also noted that the solvent had a significant effect on the equilibrium constants $\langle 89JA5346 \rangle$.

2.02

Pyrroles and their Benzo Derivatives: Reactivity

DAVID St. C. BLACK

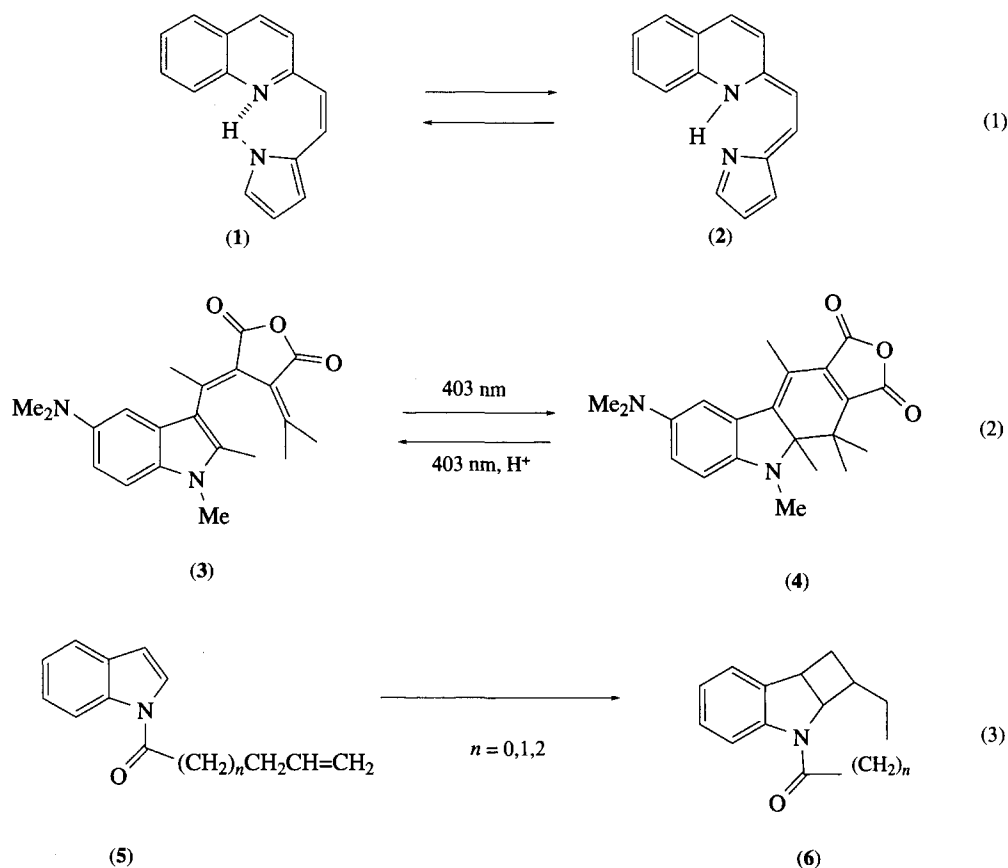
The University of New South Wales, Sydney, NSW, Australia

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2.02.1 REACTIVITY OF FULLY CONJUGATED RINGS

2.02.1.1 Thermal and Photochemical Reactions

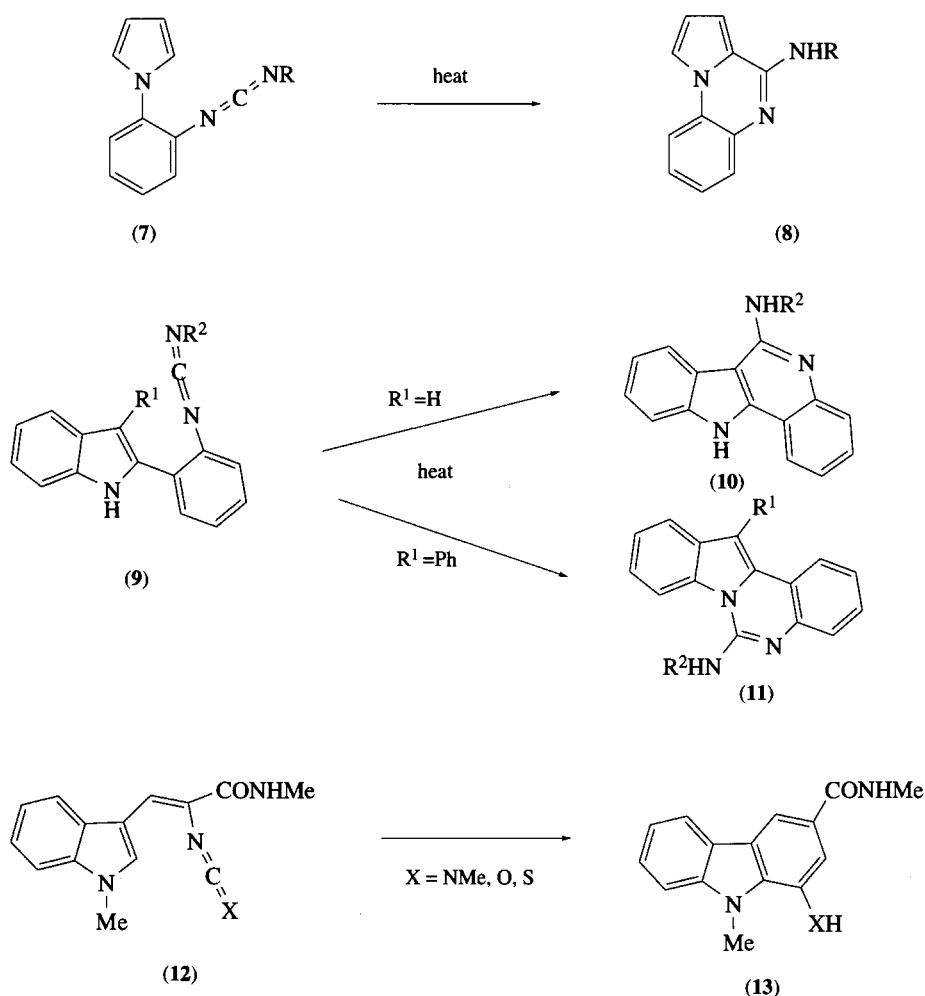
Hydrogen atom transfer occurs concurrently with intramolecular hydrogen bonding in the photo-induced tautomerism of *cis*-1-(2-pyrrolyl)-2-(2-quinolyl)ethene (**1**) to tautomer (**2**) <94JA3171> (Equation (1)). A similar phenomenon has been observed in a related pyridine-indole example <93CL691>. The 5-dimethylaminoindolylfulgide (**3**) undergoes photochemical rearrangement to the colored cyclic isomer (**4**), whose protonated form can be reconverted by photoirradiation to the protonated form of the ring-opened isomer (Equation (2)) <91CC1722>. Ultraviolet light irradiation of *N*-alkenoylindoles (**5**) gives intramolecular cycloadducts (**6**), which can be hydrolysed to generate different regioisomers from those obtained in intermolecular reactions (Equation (3)) <94JOC1333>. The aza-Wittig reaction has been exploited for the synthesis of 2-phenyl-1-pyrrolyl- and 2-phenyl-2-indolyl-heterocumulenes (**7**) and (**9**), respectively. These undergo thermal cyclization to pyrrolo-quinoxalines (**8**) and indoloquinazolines (**10**) or (**11**) in general and high-yielding processes <90TI063>. Similarly, the heterocumulenes (**12**) undergo cyclization to the β -carboline derivatives (**13**) (Scheme 1) <92TL4491>.



2.02.1.2 Electrophilic Attack at Nitrogen

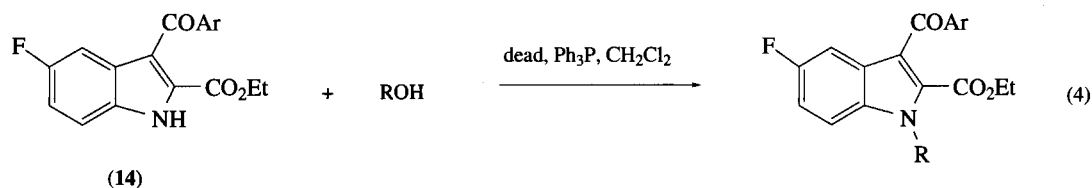
Several papers have dealt with the comparison of conditions required for *N*- and *C*-alkylation of the anions of pyrrole and indole. The results from the early 1990s, support the known observations that *N*-alkylation is favored by sodium and potassium cations, whereas *C*-alkylation is promoted by the more strongly coordinating lithium and magnesium cations <90JCS(P1)111>. *N*-Alkylation is also favored by polar solvents and the use of tosylates rather than iodides. Under phase transfer conditions, the preference for *N*-alkylation in indoles ranges from 1.5 : 1 for benzyl bromide to 10 : 1 for benzyl chloride and *n*-alkyl bromides, over 3-alkylation <90H(31)447>.

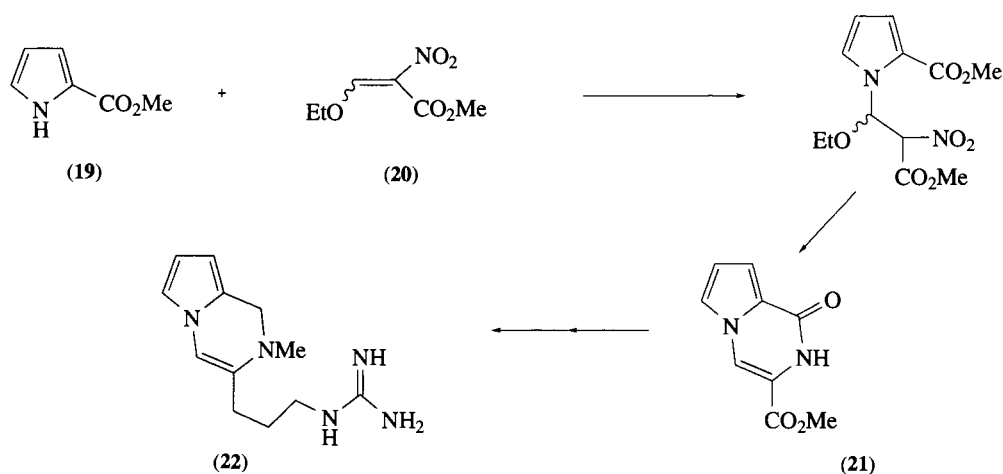
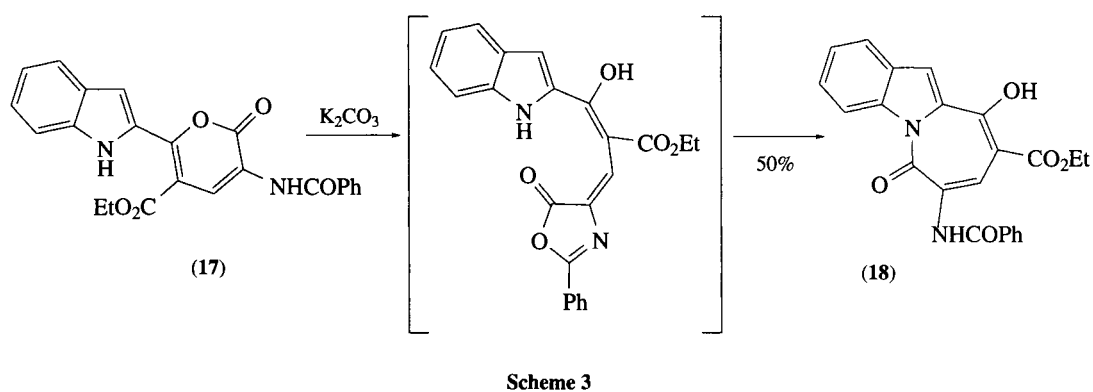
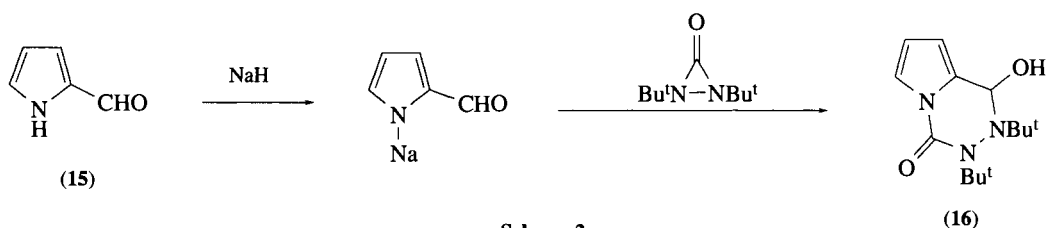
Methylation has been carried out on the pyrrole NH groups of an indolocarboline using sodamide



Scheme 1

as the base and methyl iodide: a mixture of mono- and dimethyl compounds is formed in moderate yields of each $\langle 88\text{JOC}4873 \rangle$. Reaction of pyrrole with propargyl chloride and KOH in DMSO yields the *N*-allenylpyrrole via isomerization of the initial product $\langle 93\text{S}571 \rangle$. *N*-Alkylation of 3-acylindoles can be effected in high yield by treatment with 3–5 equivalents of lithium cyanide and the alkyl halide $\langle 87\text{S}396 \rangle$. *N*-Cyanoindoles can be formed in excellent yields from the reaction of indoles with sodium hydride in DMF and phenyl cyanate: they are less reactive than *N*-acyl indoles towards electrophilic substitution $\langle 90\text{TL}3681 \rangle$. Relatively electron-deficient indoles such as (14) can be alkylated on nitrogen using Mitsunobu reaction conditions (Equation (4)) $\langle 94\text{TL}1847 \rangle$. The sodium salt of pyrrole-2-carbaldehyde (15) undergoes acylation by attack on *N,N'*-di-*t*-butyldiaziridinone, with concomitant cyclization to the pyrrolotriazinone (16) in good yield (Scheme 2) $\langle 93\text{JOC}6620 \rangle$. An intramolecular *N*-acylation occurs when the indolylpyrone (17) is treated with base: the product (18) contains a fused seven-membered ring (Scheme 3) $\langle 93\text{JCS}(\text{P}1)969 \rangle$. Michael addition of the anion of the pyrrole ester (19) to the nitroalkene (20) was followed by reductive cyclization to the lactam (21). This reaction provided the key sequence in the synthesis of the insect feeding deterrent peramine (22) (Scheme 4) $\langle 94\text{T}4887 \rangle$.



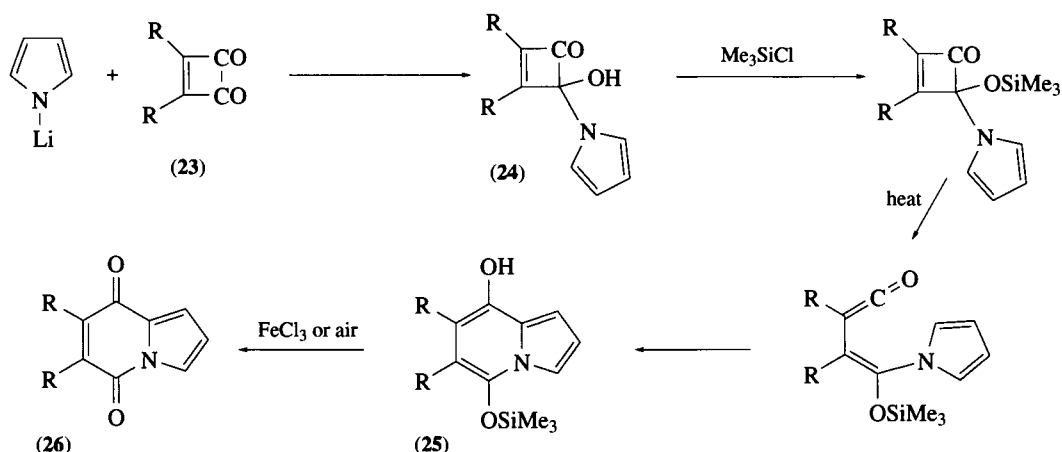


1-Lithiopyrrole reacts with a cyclobutenedione (**23**) to give an adduct (**24**): pyrolysis of the trimethylsilyl derivative generates a ketene, which allows intramolecular cyclization to the indolizine (**25**) and further easy oxidation to the dione (**26**) (Scheme 5) <92TL7811>. The *N*-nitrosation of 5-chloroindole is followed by a migration of the nitroso group from N to C-3, to give an indolenine-3-oxime (**27**): hydrolysis and recyclization leads to a benzopyrazole carbaldehyde (**28**) (Scheme 6) <86JA4115>.

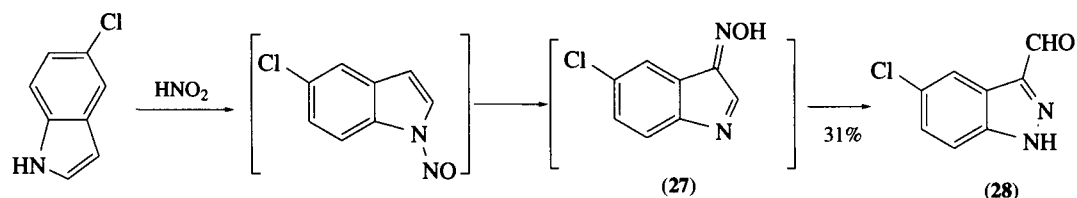
2.02.1.3 Electrophilic Attack at Carbon

2.02.1.3.1 Protonation

Partial rate factors and σ^+ values for detritiation have been calculated from rates of deuteriation for indole, 1-methylindole, 2-methylindole, 1,2-dimethylindole, and *N*-methylisindole and confirm the known reactivity patterns. The isindole is the most reactive, being approximately 10^4 times more reactive than indole <87JCS(P2)591>.



Scheme 5



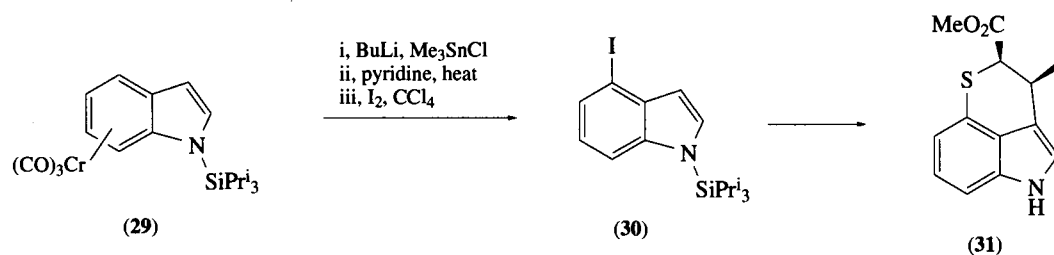
Scheme 6

2.02.1.3.2 Nitration

Thallation of 3-acyl indoles gives the 4-thallated products, which can be converted to both the 4-nitro and 4-azido derivatives in copper(II)-promoted processes $\langle 89H(29)643 \rangle$. The nitro compound is formed by heating the organothallium intermediate with sodium nitrite and copper sulfate in DMF at 100°C . This methodology has been used in a total synthesis of indolactam-V $\langle 90T6623 \rangle$.

2.02.1.3.3 Halogenation

Thallation also provides a route to 4-iodoindoles from the related 3-acyl compounds. Here the intermediate thallium compound can be treated with iodine and copper(I) iodide in DMF to effect the transformation $\langle 84H(22)797, 93JCS(P1)2561 \rangle$. The iodo compounds can be converted to the corresponding alkoxy derivatives in good yield by treatment with sodium alkoxides. More recently, in the mid-1980s, 4-iodo-1-methoxyindole-3-carbaldehyde has been prepared by this method, in which the iodine transfer can be effected by potassium iodide in 91% yield $\langle 86CPB677 \rangle$. The relatively reactive 1-methoxyindole can be iodinated directly at C-3 using iodine in morpholine $\langle 85CPB5147 \rangle$. 4-Iodination in indoles can also be achieved via the chromium tricarbonyl complexes, such as (29), after treatment with butyllithium followed by iodine $\langle 88T7325 \rangle$, or preferably with the insertion of a stannylation step as shown to give the iodo compound (30) $\langle 92JCS(P1)323 \rangle$. This methodology has been used in a synthesis of chuangxinmycin methyl ester (31) (Scheme 7).

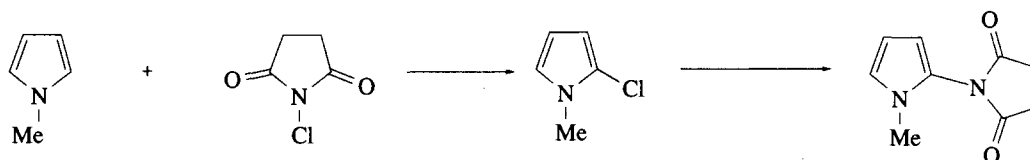


Scheme 7

Pyrroles with electron-withdrawing groups such as acyl or ethoxycarbonyl at C-3, undergo bromination using copper(II) bromide. If the 2- and 5- positions are blocked, reaction occurs at C-4 <90JHC1209, 90JHC1277>. Bromination of several 4,6-dimethoxyindoles has been shown to occur at C-7 <92T7601>. The related 7-iodo compounds can only be isolated for indoles stabilized with electron-withdrawing groups. Otherwise, the unstable 7-iodo compounds undergo oxidative coupling to give 7,7'-biindolyls <89CC111, 94T10497>. Attempted chlorination of 4,6-dimethoxy-2,3-diphenylindole using copper(II) chloride in acetonitrile gave a 5,5'-dichloro-7,7'-biindolyl in 80% yield.

Pyrroles containing an electron-withdrawing group at C-2 undergo fluorination at C-5 in 25–54% yield using xenon difluoride in acetonitrile; only α -fluorination is observed <94TL3679, 94T6181>. The reaction fails if the pyrroles contain no electron-withdrawing groups or two electron-withdrawing groups. Replacement of the solvent acetonitrile with dichloromethane results in chlorination instead of fluorination.

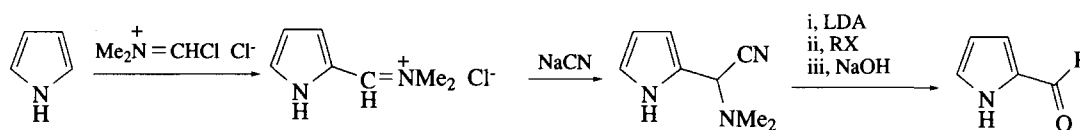
N-Methylpyrrole reacts with *N*-chloroimides to give a product in which the imidyl group is attached to C-2; the 2-chloro compound is proposed as an intermediate (Scheme 8) <89JOC5347, 93CC1757>.



Scheme 8

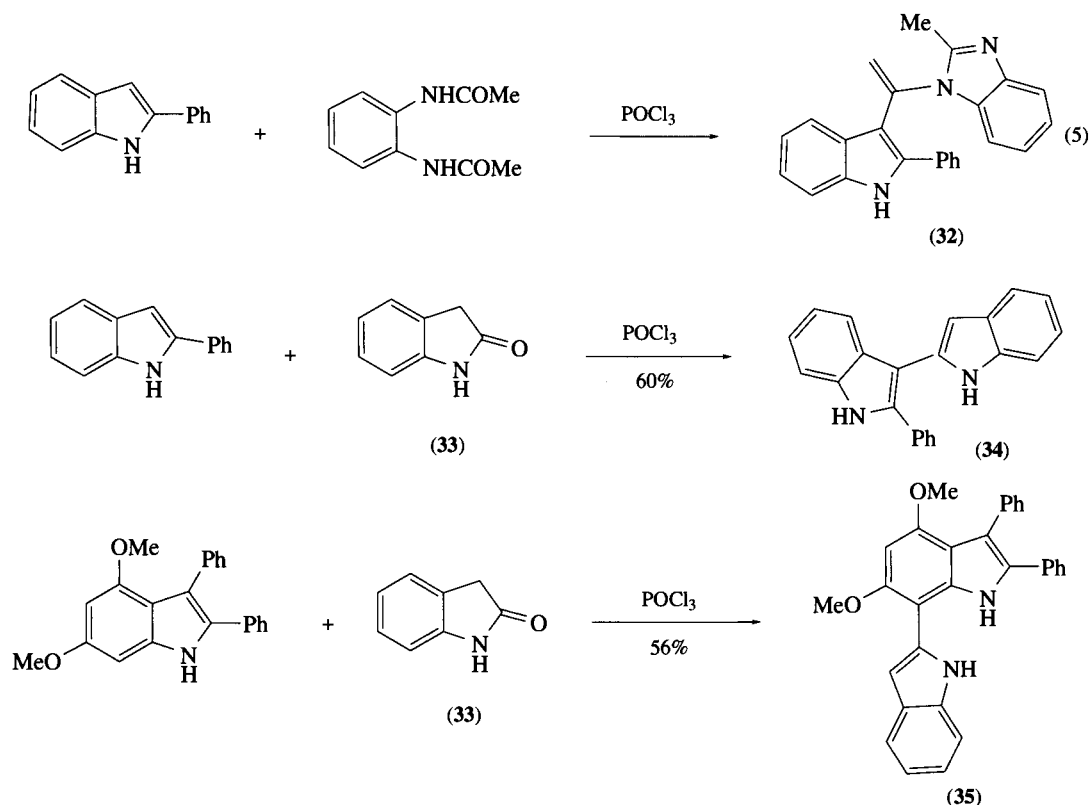
2.02.1.3.4 Acylation

N-Methylnitrilium fluoroborates, which are prepared from trimethyloxonium fluoroborate and nitriles, are very effective acylating agents. They react readily at low temperatures with indoles and pyrroles to give iminium salts, which can be isolated in high yields and hydrolysed to the corresponding acyl products if desired <85TL4649>. The iminium salts prepared from pyrroles and *N,N*-dimethylchloroformiminium chloride can be converted into acyl pyrroles by a sequence of reactions involving addition of cyanide ion, alkylation of the resulting intermediate, and hydrolysis. These transformations apply both to the normal 2-iminium salts and also to 3-iminium salts, the latter arising from reaction of the hindered *N*-(triisopropylsilyl)pyrrole (Scheme 9) <88JOC6115>.



Scheme 9

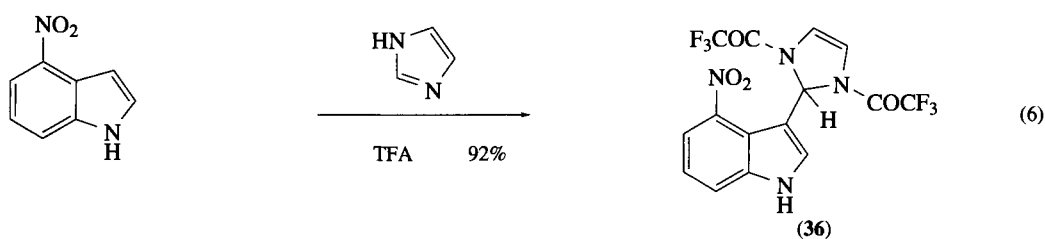
Diethoxycarbenium tetrafluoroborate, generated from the low-temperature reaction of triethyl orthoformate and boron trifluoride etherate, is an effective substitute for the Vilsmeier reagent and has been used to formylate indoles <86LA1621>. Variations on the Vilsmeier reaction have made use of secondary amides and lead to the formation of isolable imines <89JCS(P1)200>. Attempts to extend this reaction to include *N,N'*-diacetyl-*o*-phenylenediamine did not give a bisimine but instead yielded a benzimidazolylenamine (**32**) (Equation (5)). These reactions have been applied to 4,6-dimethoxyindoles to form 7-substituted products. Furthermore, the use of indolin-2-one (**33**) provides a very effective route to 2,3'- (**34**) and 2,7'-biindolyls (**35**) as a result of isomerization of the initially formed imines (Scheme 10) <84CC441>.



Scheme 10

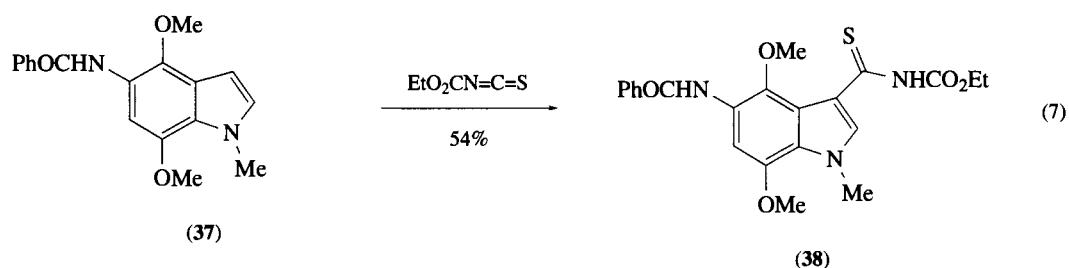
4-Nitroindole reacts readily with imidazole and TFA to give the 3-substituted indole (36), which can be converted subsequently into the 3-carbaldehyde (Equation (6)) <90T6085>.

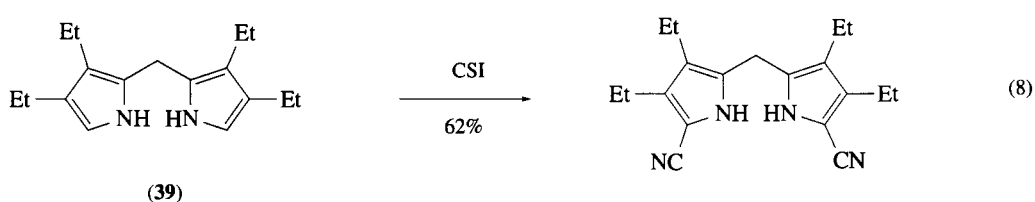
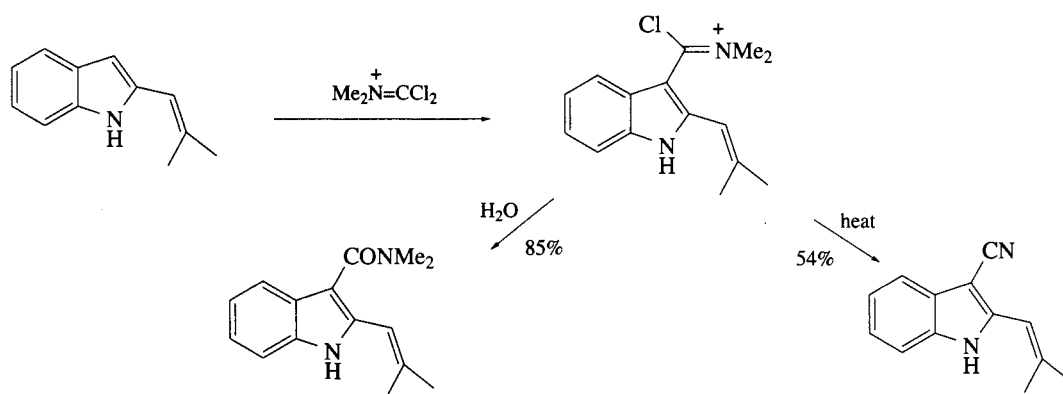
3-Carboxamido- and 3-cyanoindoles can be formed by the reaction of indoles with dimethylaminodichlorocarbonium salts, followed by hydrolysis and heating, respectively (Scheme 11) <86TL1939>.



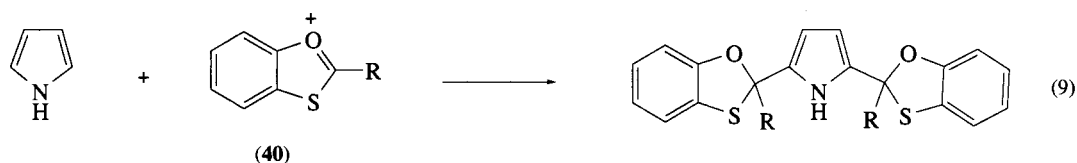
Thioacylation can be effected by the reaction of indoles such as compound (37) with ethoxycarbonyl isothiocyanate to give the 3-substituted ethoxycarbonyl thioamide (38) (Equation (7)) <93TL3797>.

A range of 1,19-dicyanodipyrromethanes have been formed from the analogous unsubstituted compounds (39) by treatment with chlorosulfurylisocyanate (Equation (8)) <94SL939>.



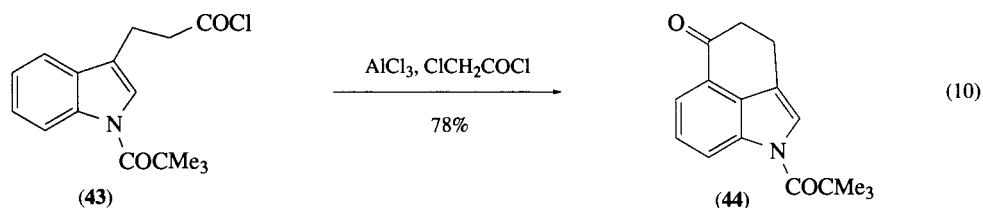
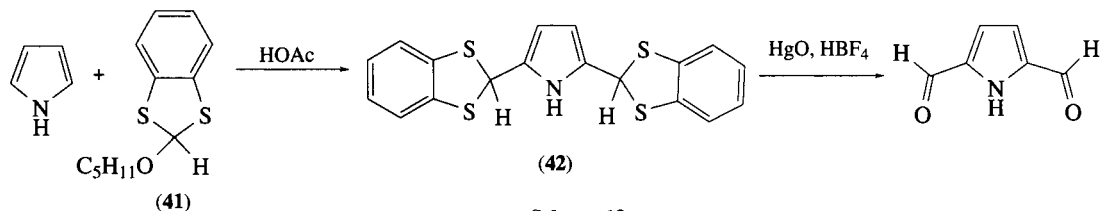


Although a 2-acyl pyrrole undergoes a second acylation at C-4, yields are variable. A more consistent route to 2,4-diacylpyrroles is by the acylation of a 3-acylpyrrole, which can in turn be obtained through the acylation of a 1-benzenesulfonylpyrrole $\langle 93\text{JCS(P1)273} \rangle$. In contrast, 2-substituted-1,3-benzoxathiolium ions (**40**) effect 2,5-disubstitution of pyrrole itself, though not of *N*-methylpyrrole (Equation (9)) $\langle 88\text{JOC2245} \rangle$. Indoles can also be acylated by these reagents $\langle 87\text{S311} \rangle$.

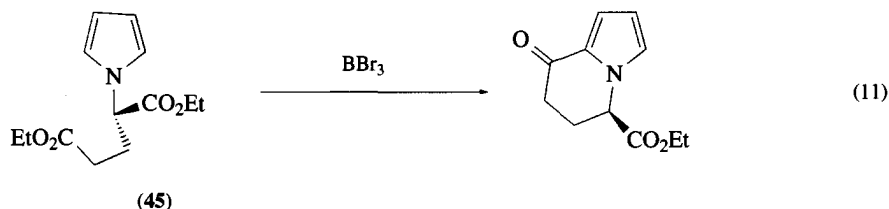


A related acylation of 2,5-unsubstituted pyrroles with 2-isopentyloxy-1,3-benzodithiole (**41**) gives the 2,5-disubstituted derivative (**42**), which can be converted by mercury-catalysed hydrolysis to the pyrrole-2,5-dicarbaldehyde (Scheme 12) $\langle 93\text{JCS(P1)2939} \rangle$.

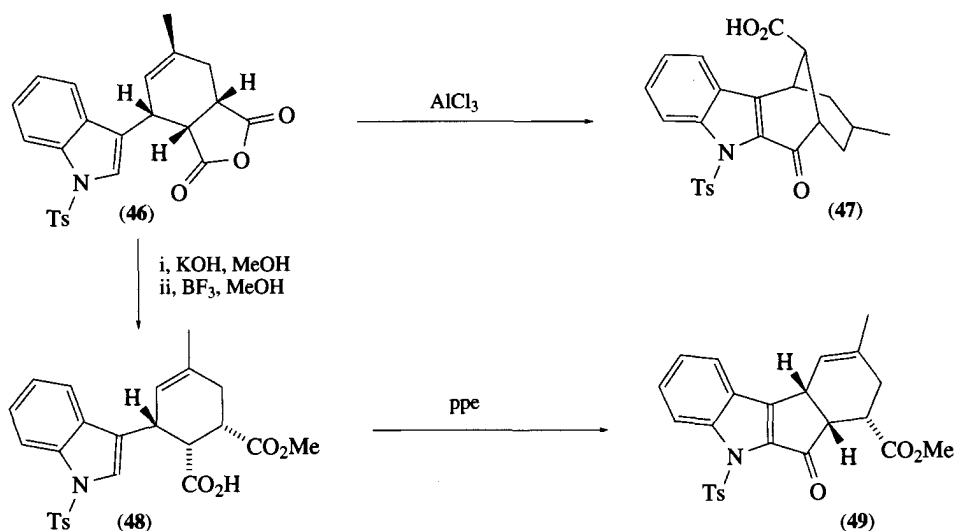
1-Acylindoles can be acylated regioselectively at C-6 by α -halogenoacyl chlorides and aluminum chloride in excellent yields. Simple alkanoyl chlorides give the usual C-3 derivatives $\langle 94\text{TL2699} \rangle$. A regioselective intramolecular acylation of the indolyl acid chloride (**43**) to the Uhle's ketone derivative (**44**) is facilitated by the *N*-pivaloyl group and the presence of a large excess of chloroacetyl chloride and aluminum chloride (Equation (10)) $\langle 94\text{TL8173} \rangle$.



Some routine intramolecular acylation reactions of pyrroles and indoles have provided useful synthetic possibilities, for example intramolecular acylation of the *N*-glutamyl-substituted pyrrole (45) occurred regioselectively, with complete retention of configuration, on brief treatment with methanolic hydrogen chloride <93JCS(P1)2241> or boron tribromide (Equation (11)) <94TL3905, 94TL4759>.



Intramolecular acylations of the Bischler–Napieralski type have been used as crucial steps in the synthesis of indole alkaloids of the yohimbine class <94JCS(P1)299, 94TL1071>. Also, the aluminum chloride-catalysed reaction of the indolosuccinic anhydride (46) gave the bridged cyclic product (47) rather than the cyclopentanone (49), presumably because the reaction involves initial acylation at C-3 and reaction via the less strained intermediate is favored. Formation of the cyclopentanone (49) can be achieved using the polyphosphate ester cyclodehydration of the modified intermediate (48) (Scheme 13) <90JCS(P1)2487>.

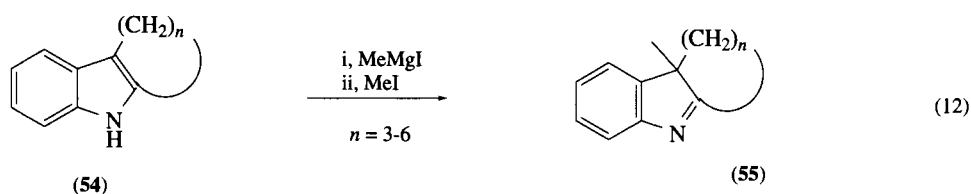
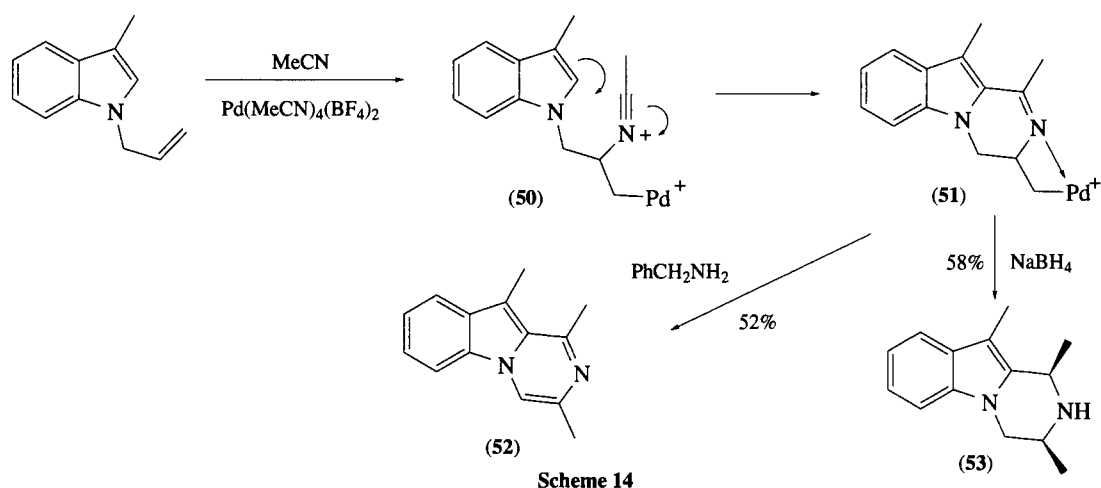


Scheme 13

In a new kind of process, *N*-allylskatole reacts with a nitrile and a palladium acetonitrile complex to give a palladium complex of proposed structure (51), presumably via intramolecular acylation of the nitrilium cation (50). The complex (51) can be converted into the pyrazinoindole (52) by reaction with benzylamine or the tetrahydro analog (53) with sodium borohydride (Scheme 14) <86JA6224>. Acylation has been effected at C-7 in some 4,6-dimethoxyindoles <94T10497>.

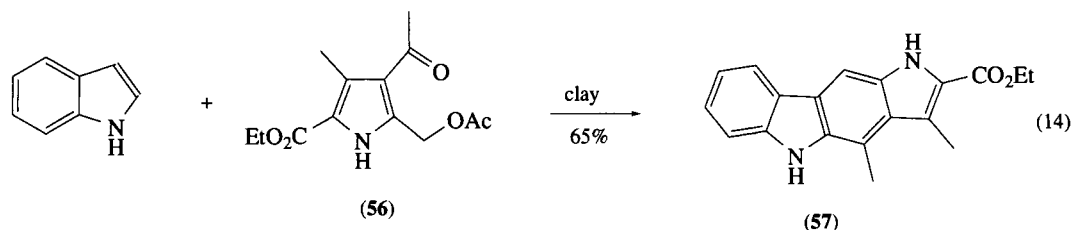
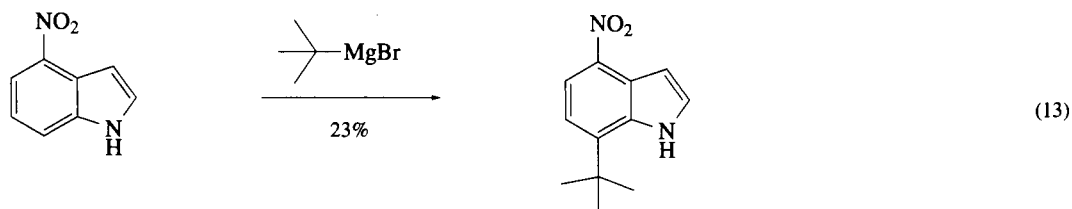
2.02.1.3.5 Alkylation

C-Alkylation of pyrroles and indoles can be mediated by alumina in association with the appropriate halide <92JCS(P1)2821>. A wide range of alkylating agents includes dithiolium cations <86LA1628>. 3-Indolyl sugar derivatives have been obtained by alkylation reactions of indolyl-magnesium bromide <91JOC5466>. This type of alkylation has been extended to ring-fused indoles (54) and yields 3*H*-indole derivatives (55) in good yield (Equation (12)) <91JHC1293>.

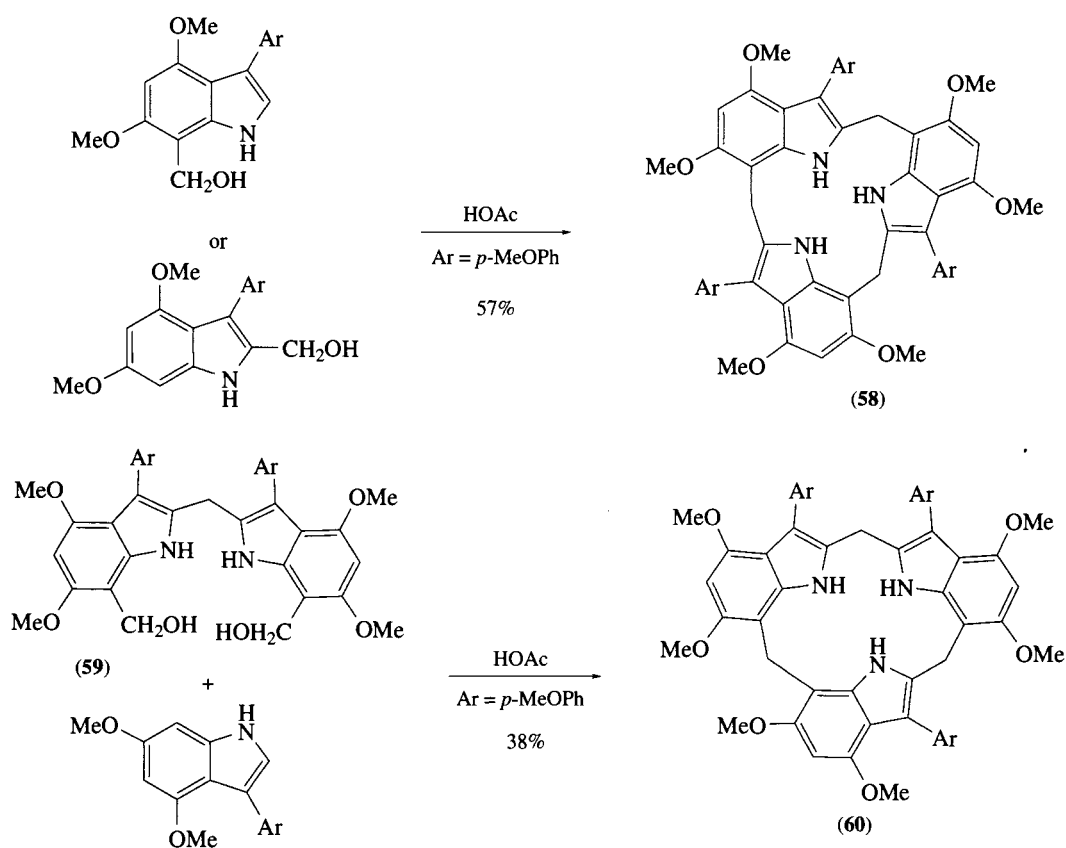


A novel alkylation occurs at C-7 in the reaction of 4-nitro-indole with alkylmagnesium halides. Up to the mid 1990s, the yields obtained are still rather low (Equation (13)) <94TL8553>.

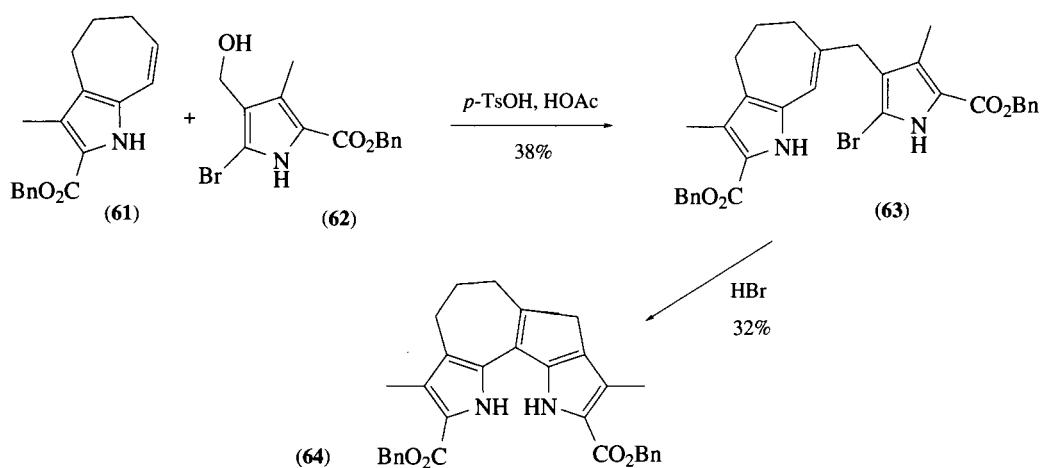
Allylation and benzylation continue to be exploited in a variety of situations. Indole undergoes a clay-catalyzed reaction with the acetoxymethylpyrrole (56) to give the pyrrolocarbazole (57). The reaction is not general and small yields of a variety of related products are obtained with substituted indoles (Equation (14)) <94JCS(P1)1765>.



The 2- or 7-carbinols of electron-rich indoles undergo acid-catalyzed self-condensation to give cyclic trimers (58). The related unsymmetrically linked trimer (60) can be formed by combination of the 2,2'-diindolylmethane dicarbinol (59) and the indole (Scheme 15) <93CC819>. This synthetic technique also allows the formation of 2,7'-diindolylmethanes. The bicyclic 2-vinylpyrrole (61) undergoes acid-catalyzed condensation with the 3-pyrrolocarbinol (62) to give the pyrrolylmethyl derivative (63), which can be cyclized further to give the cross-conjugated dipyrrole (64) (Scheme 16) <94SL905, 94SL909>.

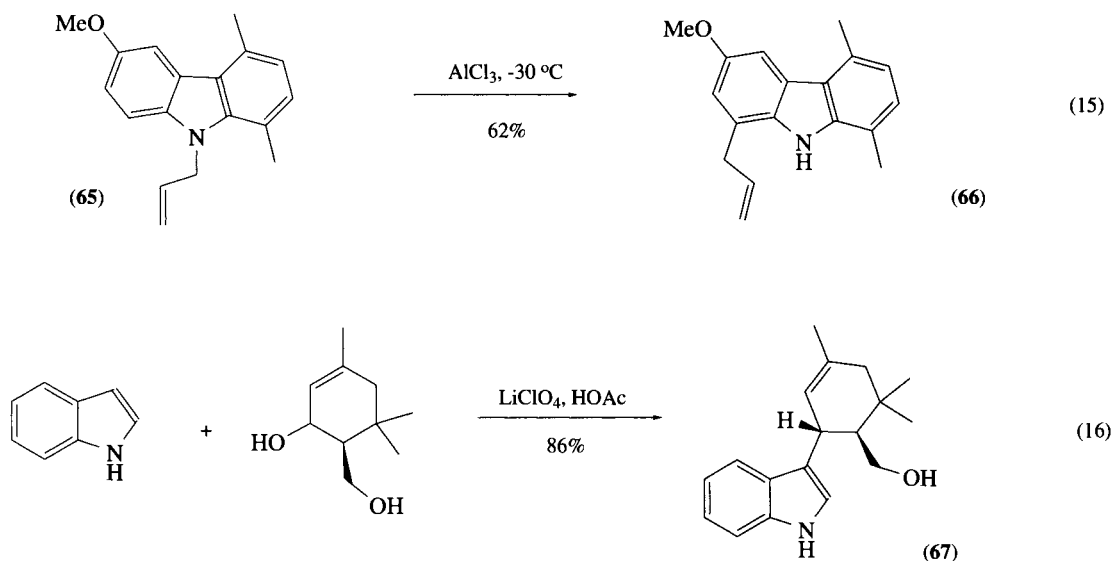


Scheme 15

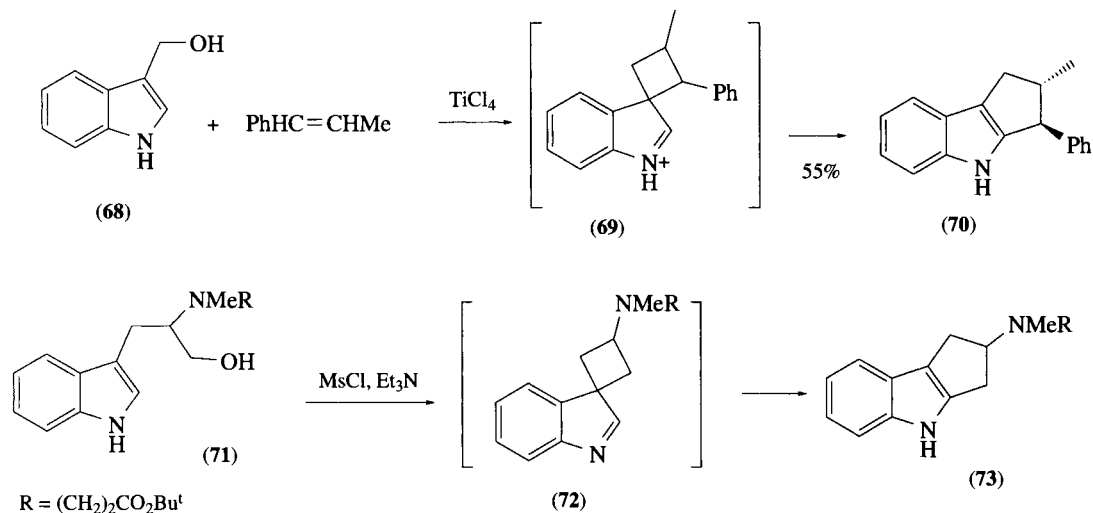


Scheme 16

Magnesium indolates and allyl alcohols undergo reaction in the presence of bis(triphenylphosphine)nickel chloride to give 3-allylindoles in moderate yields (86JOC2343). Effective migration of an allyl group from nitrogen to carbon can be effected by aluminum chloride at -30°C in the conversion of the *N*-allylcarbazole (65) to the *C*-allyl isomer (66) (Equation (15)) (88JCS(P1)2945). Indoles can be alkylated by allyl alcohols in the presence of lithium perchlorate and acetic acid: for example, products such as the precursor (67) to the anti-fertility alkaloid yuechukene can be produced (Equation (16)) (93CC510).



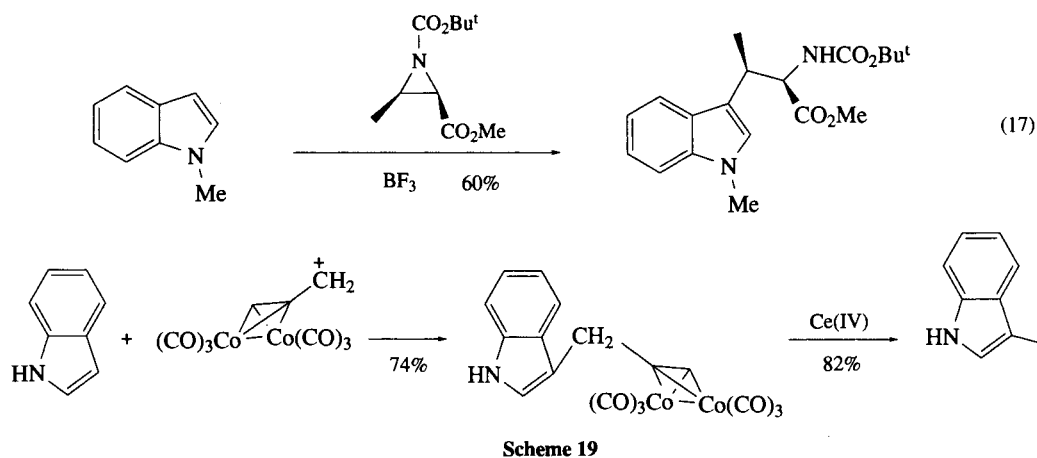
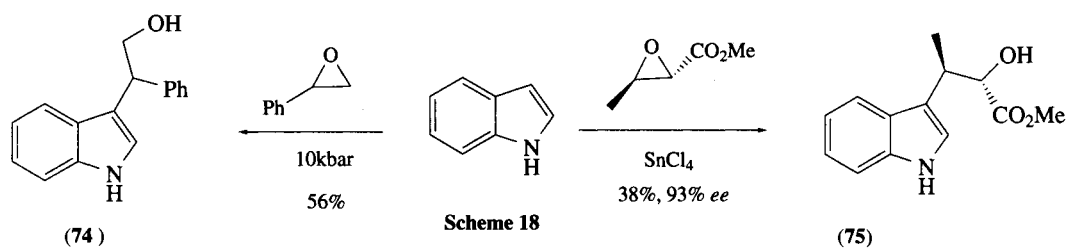
A similar reaction affords a simple route to cyclopent[b]indoles. Indole-3-carbinol (**68**) reacts with β -methylstyrene in the presence of titanium tetrachloride to give compound (**70**) <93TL8527>. The presumed four-membered spiroindolenine (**69**) in this sequence receives support from the observation that the conversion of the D-tryptophan derived compound (**71**) to its mesylate leads to the formation of racemic cyclopent[b]indole (**73**) via the achiral intermediate (**72**) (Scheme 17) <93TL439>.



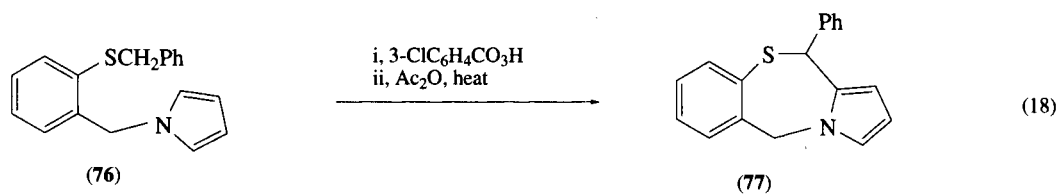
Scheme 17

Indole is also alkylated by styrene oxide, under conditions of high pressure but without catalysis, giving the hydroxylated 3-benzylindole (**74**) <90JOC2969>. The use of chiral epoxides can lead to products of high enantioselectivity such as the indolyl hydroxy ester (**75**) (Scheme 18) <90CPB323>. Related aziridines give the potential for enantioselective synthesis of amino acids (Equation (17)) <90CPB564>. Dicobalt hexacarbonyl stabilised propargyl cations effect alkylation of indoles at C-3 and on deprotection yield 3-propargylindoles: a significant amount of the 3,3-dipropargylindolenine is formed from 2-methylindole (Scheme 19) <90H(30)451, 93SL529>. Some N-alkylation can occur

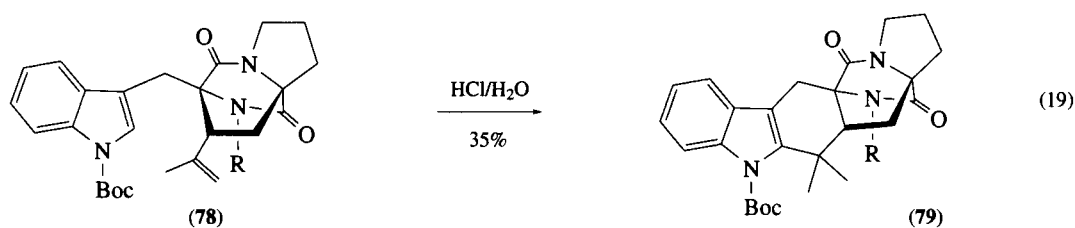
unless the indole nitrogen atom is substituted, and tryptamine derivatives give C-3 substitution together with cyclization.

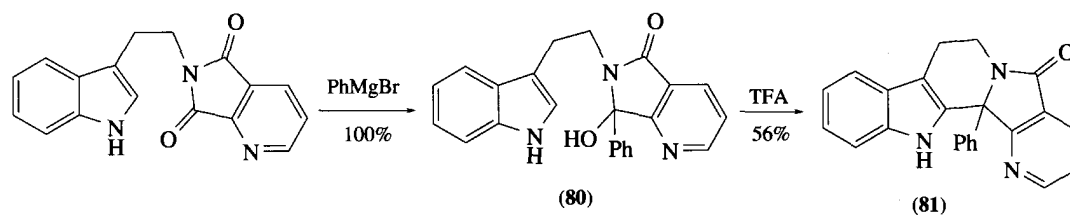


Intramolecular electrophilic cyclization of an *N*-benzylpyrrole (**76**) can be achieved by treatment of the sulfoxide with acetic anhydride <92H(34)51>; the product (**77**) is an interesting pyrrolo-benzothiazepine derivative (Equation (18)).



A variety of other intramolecular alkylation reactions have been utilized in the synthesis of indole alkaloids. The acid-catalyzed conversion of the alkene (**78**) to the cyclic product (**79**) is a key step in the synthesis of a brevianamide (Equation (19)) <93T8471>. Similar acid treatment of the benzylic carbinol (**80**) yields the β -carboline derivative (**81**) (Scheme 20) <88CC623>.

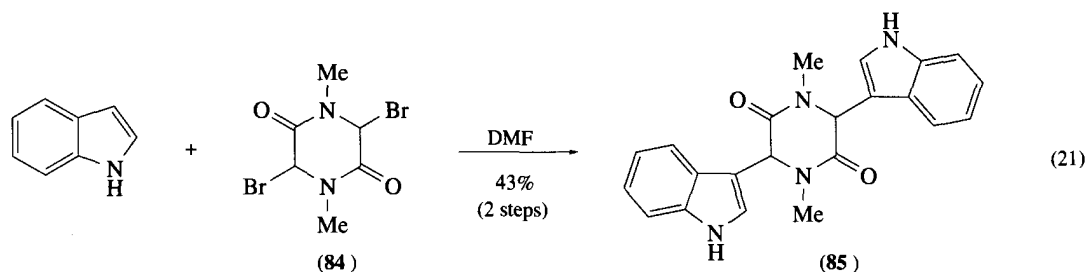
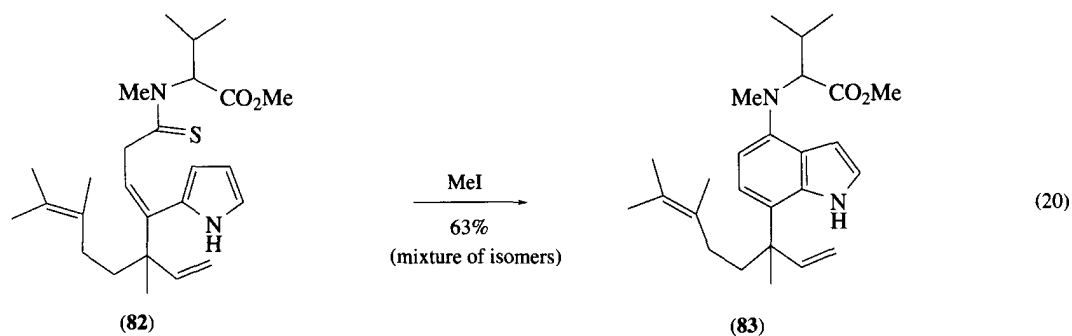




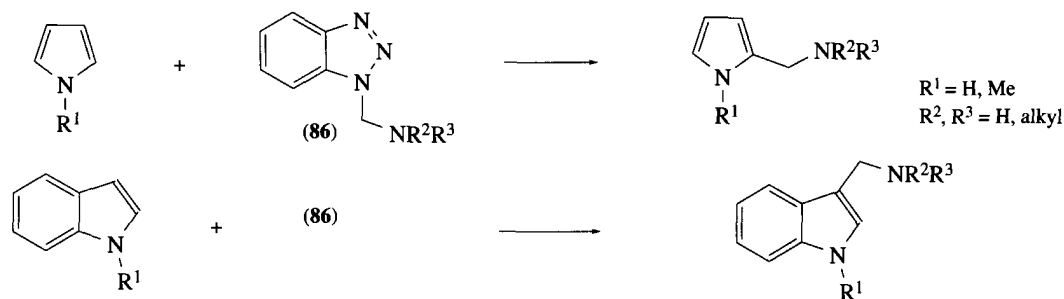
Scheme 20

The intramolecular alkylation of pyrroles can be used as a synthetic route to indoles, as illustrated in the conversion of the thioamide (**82**) to the teleocidin precursor (**83**) (Equation (20)) <88TL6267>.

Alkylation can also be effected by α -halocarbonyl compounds. Tryptophan analogs can be prepared by the combination of 2-substituted indoles with the oxime of ethyl bromopyruvate, followed by reduction of the resulting oxime <88S73>. The active electrophile in this type of reaction is evidently an unsaturated nitroso compound <83JCS(P1)1283>. Indole also reacts with the dibromo derivative of 1,4-dimethylpiperazine-2,5-dione (**84**) to generate a 2,5-bis(3'-indolyl)piperazine (**85**): the 6,6'-dibromo analog of the tetrahydropiperazine reduction product is drarmacidin B, a marine natural product (Equation (21)) <94TL371>.

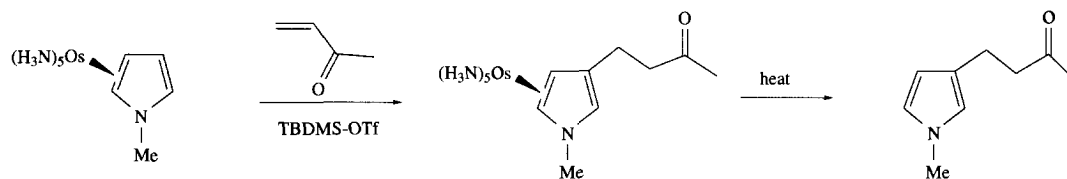
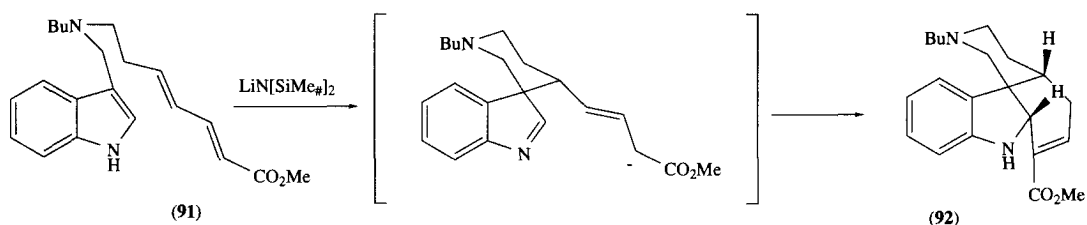
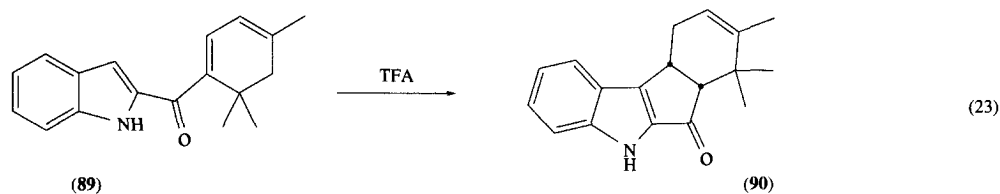
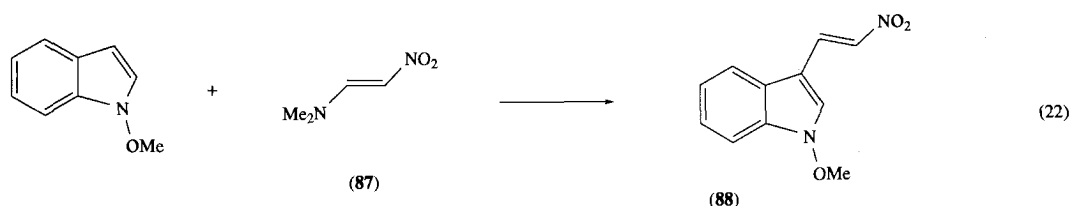


Aminoalkylation reactions of the Mannich type have seen some developments since the 1990s. Secondary and tertiary aminoalkylbenzotriazoles (**86**) react with pyrrole, indole, and their *N*-methyl analogs under mild conditions in the presence of a Lewis acid to afford selectively the corresponding secondary or tertiary aminoalkyl derivatives (Scheme 21) <92T4971>. *N,N*-Bis(alkoxymethyl)amines can also be used to give secondary alkylamines, which operate via reactive iminium salts formed by treatment with trimethylsilyl chloride <90TL4229>.



Scheme 21

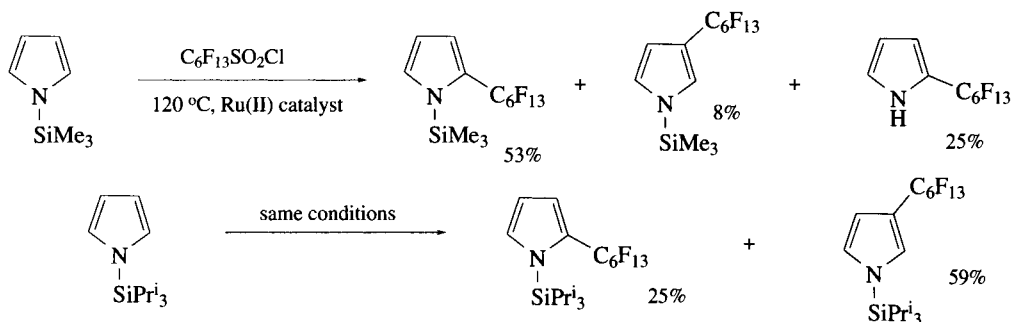
A useful method for the alkylation of pyrroles and indoles is their addition to electrophilic alkenes. These reactions can be catalyzed by acidic clays, which are often more effective than Lewis acids <88TL2577>. Indoles are alkylated as usual at C-3, and the alkylation of 3-substituted indoles has also been shown to take place at C-3 with subsequent migration occurring to C-2. *N*-Methoxyindole reacts with the nitrovinyl compound (87) to give the 3-nitrovinylindole (88) (Equation (22)) <85H(23)1101>. Intramolecular examples are features of the synthesis of some indole alkaloids. The 2-acylindole (89) undergoes cyclization to the yuehchukene precursor (90) on treatment with trifluoroacetic acid (Equation (23)) <88TL2993>. A tandem intramolecular Michael addition of the indole (91) is followed by cyclization under the influence of lithium bistrimethylsilyl amide. The stereochemistry of the ring junction in the product (92) is determined by the stereochemistry of the starting material <92TL4657> (Scheme 22). Pyrrole osmium complexes react with methyl vinyl ketone at C-3 in the presence of TBDMS triflate to give good yields of products after thermal removal of the osmium. In the absence of the catalyst, acrylonitrile and methyl acrylate prefer cycloaddition to give derivatives of a 7-azabicyclo[2.2.1]heptane (Scheme 23) <93JOC4788>.



1-Trimethylsilyl- and 1-triisopropylsilylpyrrole can be regioselectively perfluoroalkylated at their 2- and 3- positions, respectively, with perfluoroalkanesulfonyl chlorides in the presence of dichloro-tris(triphenylphosphine)rhodium catalyst (Scheme 24) <94JCS(P1)1339>.

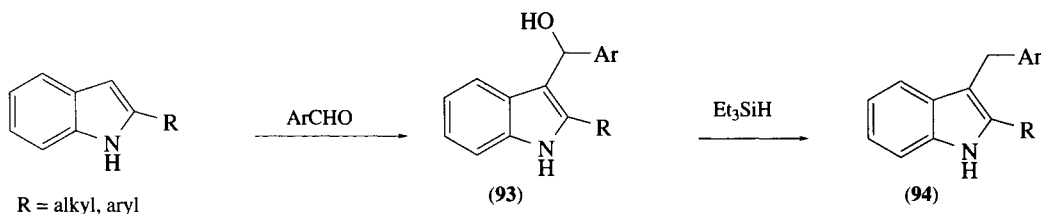
2.02.1.3.6 Reactions with aldehydes and ketones

Pyrroles and indoles react readily with aldehydes and ketones. The initially formed carbinol usually generates a reactive intermediate, which is attacked by another equivalent of substrate to yield a disubstituted methane. The reliable synthesis of arylidpyrromethanes from the direct



Scheme 24

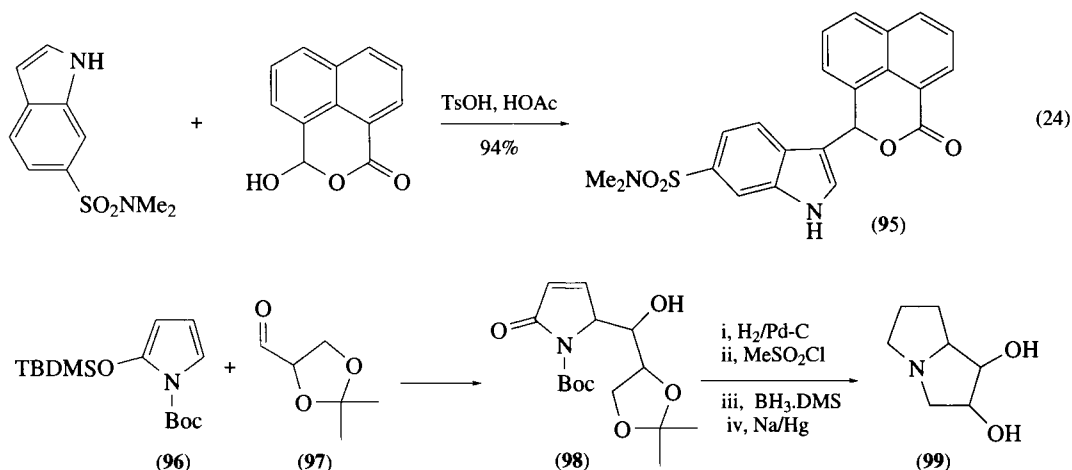
condensation of pyrroles with aryl aldehydes has been described: the yields range from 15% to 45% and are better for those aldehydes with electron-withdrawing groups <94TL2455>. New examples of diindolylmethanes have been reported in the early 1990s <90T2367, 91T9225>: these include 2,7' and 7,7'-diindolylmethanes formed from 7-activated indoles <93CC819, 94T10497>. The initial 3-carbinols (**93**) formed from 2-substituted indoles can be reduced *in situ* by triethylsilane to give 3-benzylindoles (**94**) (Scheme 25) <93TL1529>.



Scheme 25

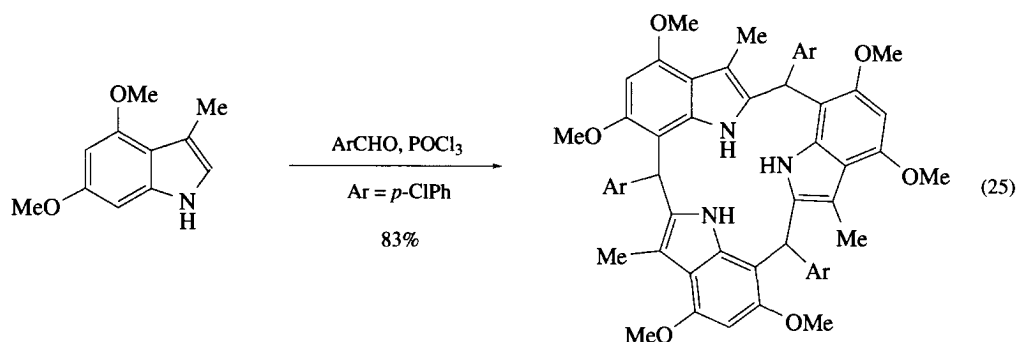
Related but more complex processes occur when indole is treated with *p*-toluenesulfonic acid <88JCS(P1)2377, 88JCS(P1)2387>. Indoles also undergo acid-catalyzed addition to lactols to form derivatives such as (**95**), which are precursors of a range of photographic dyes (Equation (24)) <91JOC4576>.

A reductive sequence of reactions has resulted in the synthesis of all four isomers of *cis*-1,2-dihydropyrrolizidine (**99**) from the carbinol (**98**), formed from the combination of the oxygenated pyrrole (**96**) with the various isomers of the protected glyceraldehyde (**97**) (Scheme 26) <94JOC2906>.

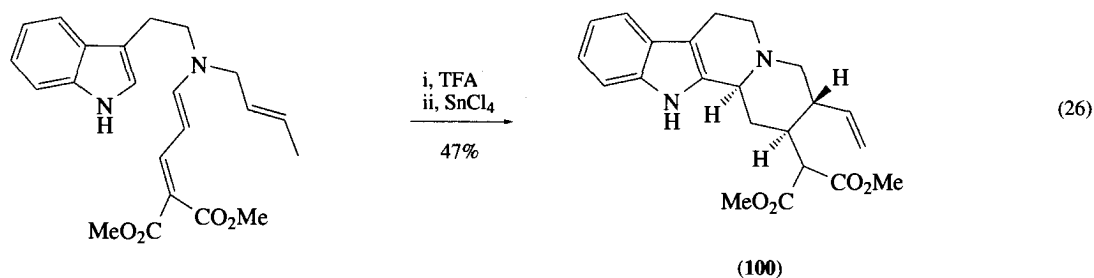


Scheme 26

3-Methyl-4,6-dimethoxyindole can be converted by treatment with aryl aldehydes and phosphoryl chloride in chloroform into macrocyclic triindolylmethanes, with the indole rings linked at C-2 and C-7 (Equation (25)) <89CC425>. This reaction presumably proceeds through the intermediate carbinol (see Section 2.02.1.3.5).

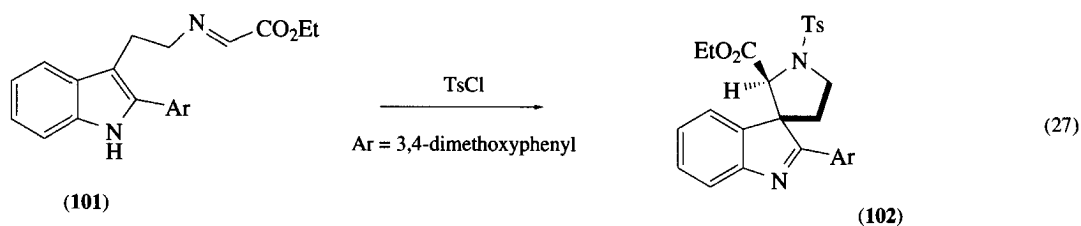


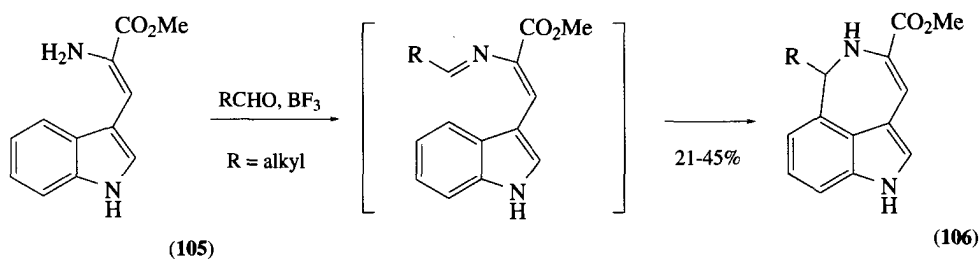
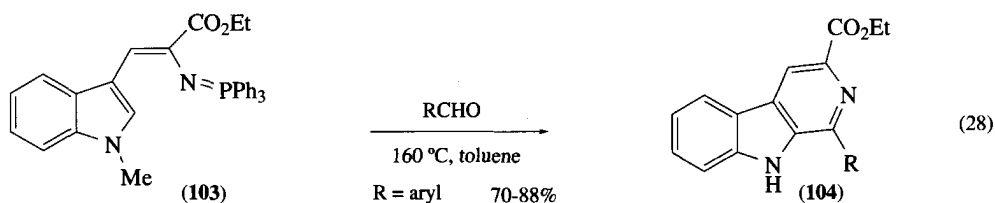
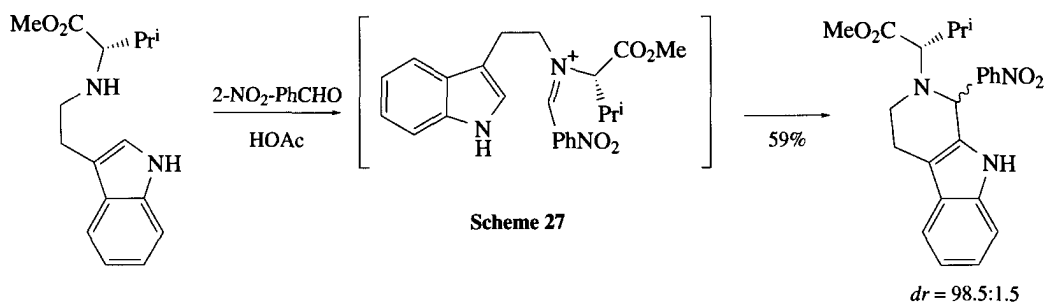
There have been many examples of the Pictet–Spengler reaction used for the synthesis of tetrahydro- β -carbolines. Some examples include the cyclization of imines derived from tryptamine and indole-2-carbaldehyde <91JCS(P1)1871>, an α -aminoaldehyde derived from L-glutamic acid <93T8589>, or cycloalkanones <93JHC373>. An enamine formed from an *N*-allyltryptamine has also been studied and combines a Pictet–Spengler cyclization with an ene reaction to give an indole alkaloid precursor (**100**) as a single diastereomer (Equation (26)) <92AG(E)1079>. The related *N*-hydroxy compounds have been generated from intermediate nitrones formed from *N*-hydroxytryptamine and aldehydes <88CC463, 90CPB59>.



The imine (**101**) derived from a 2-aryltryptamine and ethyl glyoxylate undergoes a diastereoselective spirocyclization to the indolenine (**102**) (Equation (27)). Replacement of the ethyl ester by chiral esters, such as the 8-phenylmenth-3-yl ester, leads to asymmetric induction at the spiro carbon atom <90HCA439>. Imines derived from tryptophan esters have also been studied as substrates for the Pictet–Spengler cyclization (e.g., <87TL1131>). Enantioselective cyclizations have made use of enantiomerically pure aldehydes and tryptophan esters <92H(34)517, 93JCS(P1)431, 93T8589>.

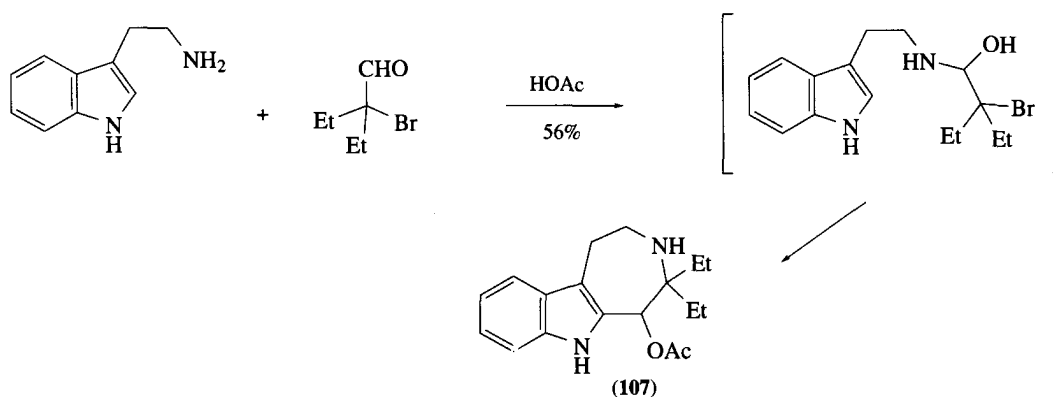
Another approach to this enantioselective cyclization involves the formation of imines from *N*- β -(3-indolyl)ethyl amino acid esters and a variety of aryl aldehydes. These imines give tetrahydro- β -carbolines with diastereomer ratios of up to 98.5 : 1.5 (Scheme 27) <94T11865>. The chiral auxiliary group can be removed after cyclisation. The iminophosphorane (**103**), derived from a dehydrotryptophan, reacts thermally with aldehydes to give the β -carbolines (**104**) (Equation (28)) <92TL2891>. In contrast, the dehydrotryptophan (**105**) itself interacts with aldehydes to form compounds (**106**) with the clavicipitic acid skeleton (Scheme 28) <86TL4757>.





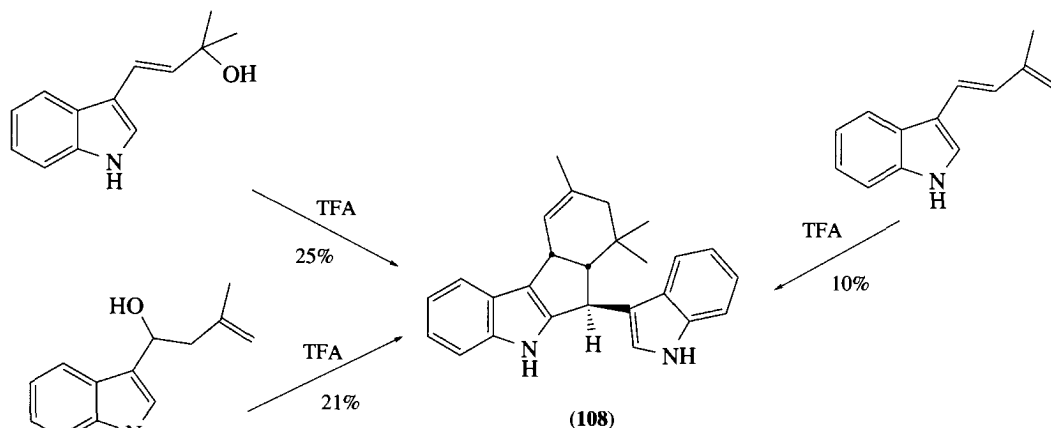
Scheme 28

A homo-Pictet–Spengler reaction has been developed. Tryptamine undergoes reaction with α -bromoaldehydes to yield hexahydroazepinoindoles such as **(107)** in reasonable yield (Scheme 29) <91TL619>.

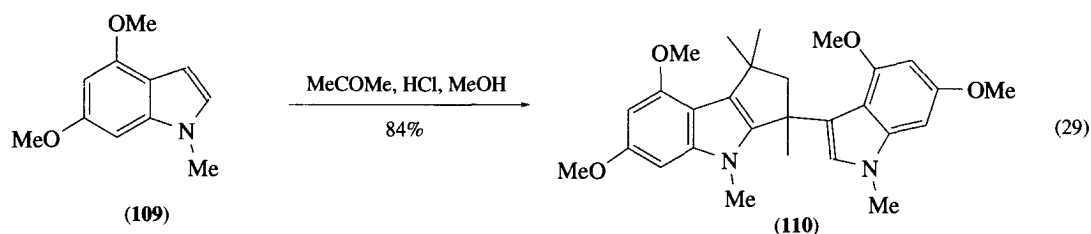


Scheme 29

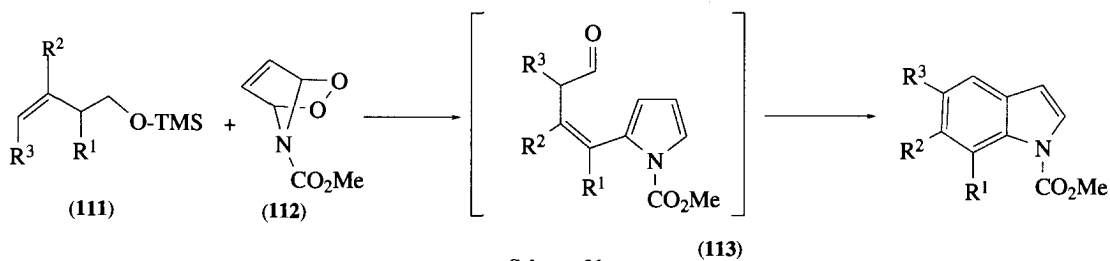
The synthesis of yuehchukene **(108)** depends on the addition of C-2 to a vinylogous indoleninium cation for the crucial formation of the cyclopentane ring (Scheme 30) <85CC48, 88JOC3170, 93JOC5784>. The rather low yields of these reactions can be improved by the use of activated indoles, as shown in the conversion of 4,6-dimethoxy-1-methylindole **(109)** to the simpler cyclopentanoindole **(110)** (Equation (29)) <91TL1587>. Similar products can be formed in acid-catalyzed reactions of indole with acetone <89T5549>.



Scheme 30

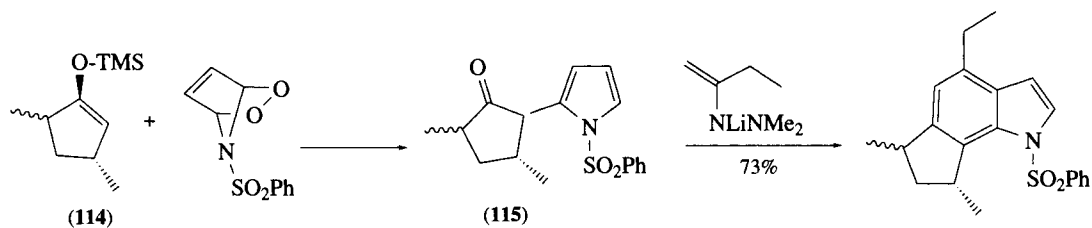


Pyrroles can undergo intramolecular addition to carbonyl groups in a process that has been developed into a synthesis of indoles containing alkyl substituents in the benzene ring. There is considerable variation in the construction of the crucial precursors. One approach is the addition of a trimethylsilyl ether (111) of an α,β -unsaturated aldehyde to the endoperoxide (112) of 1-methoxycarbonylpyrrole to give an isomeric mixture of the pyrrolylbutenone (113): cyclization is then effected by treatment with *p*-toluenesulfonic acid in benzene (Scheme 31) <89H(29)771>. Treatment of the endoperoxide with a simpler enol ether such as a cyclopentanone TMS ether (114) gives a ketone (115), which can be converted to the indole by addition of the lithium anion of 2-butanone *N,N*-dimethylhydrazone and cyclization (Scheme 32) <89TL5771, 90H(31)691, 90T5113>. This strategy has been used in the synthesis of teleocidins and trikentins. Another route to an appropriately substituted pyrrole involves the addition of 2-(2-dioxolanyl)ethylmagnesium bromide to an *N*-protected 2- or 3-formyl- or acylpyrrole: cyclization of a 2-substituted pyrrole occurs at C-3 and that of a 3-substituted pyrrole at C-2. In some cases pyrrole deprotection is required prior to cyclization <89H(29)783, 90H(31)683, 92CPB2358, 93TL4815>.

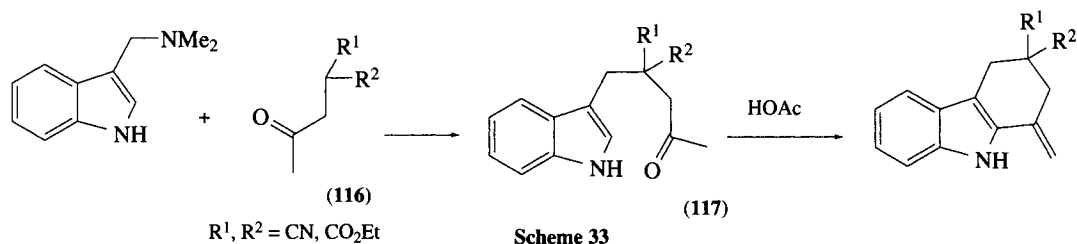


Scheme 31

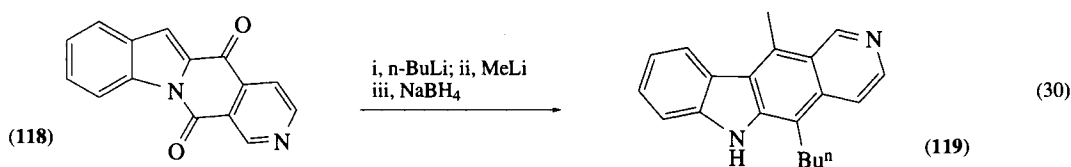
Carbazoles can be derived from the acid-catalyzed intramolecular addition of the ketones (117) derived from gramine and oxomalonate systems (116) <88TL6505> (Scheme 33). Treatment of the ketone (118) with one equivalent of *n*-butyllithium followed by one equivalent of methyllithium and then borohydride reduction of the intermediate diol gave the pyridocarbazole (119). The transformation involves intramolecular trapping of the intermediate aldehyde at C-3 (Equation (30)) <83TL3831>.



Scheme 32

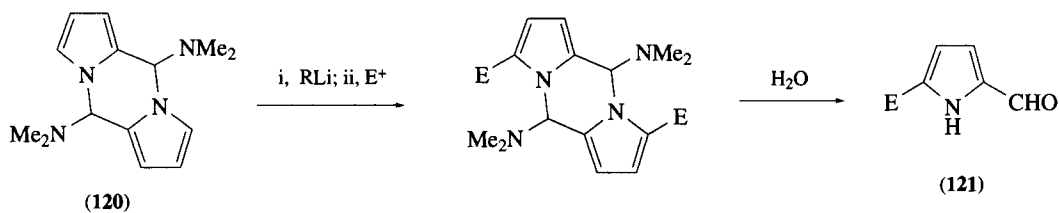


Scheme 33

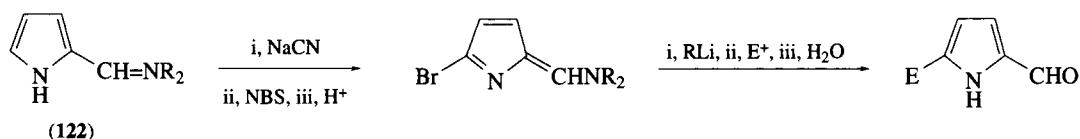


2.02.1.3.7 Reactions on C and N anionic species

Organometallic intermediates continue to be important in the synthetic methodology for substitution of pyrroles and indoles. Numerous lithiation techniques have been reported and provide a range of useful reaction conditions. The pyrrolyl dimer (120) (really a dimeric equivalent of pyrrole-2-carbaldehyde) can be lithiated at the 5- and 5'-positions. Electrophilic substitution then yields 5-substituted pyrrole-2-carbaldehydes (121) (Scheme 34) <88TL777>. The related monomeric aldiminium salts (122) yield similar products after anion formation, bromination, lithiation, and electrophilic attack (Scheme 35) <88HCA2053>. In a similar process, the dimer (120) can be brominated at C-4 and converted into 4-substituted pyrrole-2-carbaldehydes <88TL3215>.



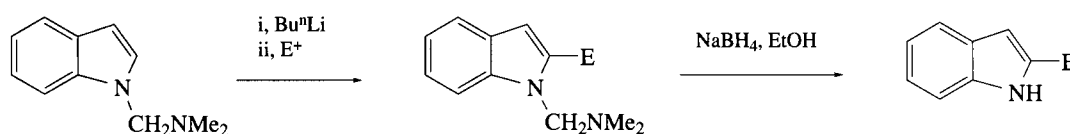
Scheme 34



Scheme 35

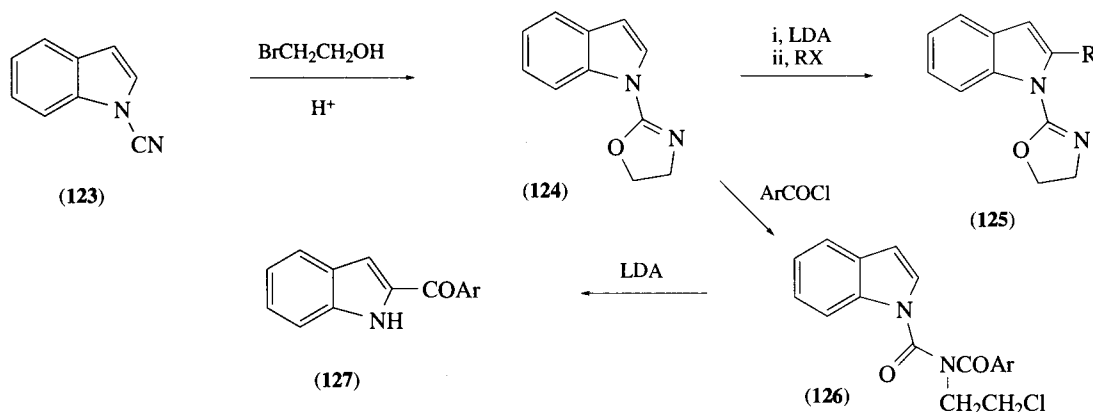
Lithiation can be influenced by a range of *N*-substituents. One of the most useful processes involves the *N*-lithiation, carbonation, and C-2 lithiation of indoles, which leads especially to 2-haloindoles in excellent yields <92JOC2495>. C-2 Lithiation is also promoted by the *N*-dimethyl-

aminomethyl group <89H(29)849, 90JOC3688>. After substitution, the *N*-substituent is removed by borohydride reduction rather than hydrolysis (Scheme 36).



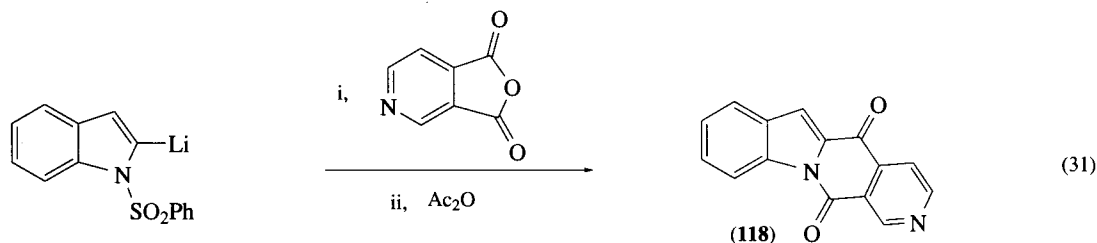
Scheme 36

The *N*-(2-oxazolynyl) group also directs lithiation to C-2. This group is introduced from the *N*-cyano compound (**123**) by reaction with 2-bromoethanol under acidic conditions. Treatment of the lithio derivative of the product (**124**) with alkyl halides gives a range of 2-substituted indoles (**125**) (Scheme 37) <92H(33)173, 93H(35)573>. The oxazoline ring of indole (**124**) can be opened by acylation to indole (**126**), which on lithiation results in intramolecular acyl transfer to C-2, thus providing a useful route to 2-acylindoles (**127**). *N*-(Methoxymethyl)indole is also readily lithiated at C-2 and the protecting group removed after substitution by hydrogenolysis <92H(34)1295>.

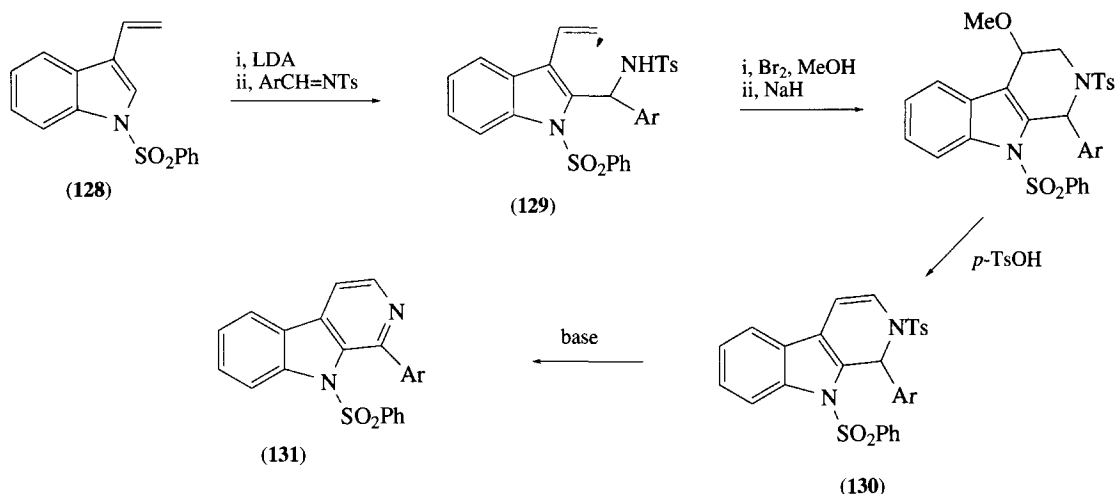


Scheme 37

Lithiation of the related 1-methoxymethylindole-2-carboxylic acid occurs at C-3 <89H(29)1661>. Similar C-3 lithiation occurs in 1-benzenesulfonyl-2-(1,3-dithian-2-yl)indole, but the indole then undergoes ring opening <88T443>. An *N*-benzenesulfonyl group directs lithiation to C-2, further acylation with pyridine-3,4-dicarboxylic acid anhydride gives the tetracyclic ketone (**118**), which serves as a precursor of ellipticine (Equation (31)) <92JOC5891>. The 1-benzenesulfonyl-3-vinylindole (**128**) can be lithiated at C-2 and then added to a tosyl imine to give the derivative (**129**). Addition of bromine in methanol to the double bond, followed by cyclization and elimination of methanol gives the dihydro compound (**130**), a precursor of the β -carboline (**131**) (Scheme 38) <94JOC3219>.

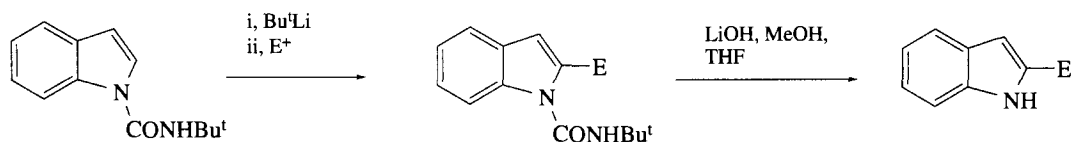


One of the most effective *N*-substituents for C-2 lithiation of indoles and pyrroles is the *t*-butylcarbonyl group, the lithiated derivatives of which react with a wide range of electrophiles such as trimethylsilyl chloride, *N,N*-dimethylbenzamide, and carbon dioxide, to give the substituted products in 45–95% yield <91S1079>. Removal of the *N*-group can be effected by treatment with lithium hydroxide in methanolic THF (Scheme 39). 1-Methoxyindole also can be lithiated selectively at C-2 and converted to substituted products: hydrogenolysis removes the methoxy group <91H(32)221>. Even *N*-phenylpyrrole can be selectively monolithiated at the pyrrole C-2 position,

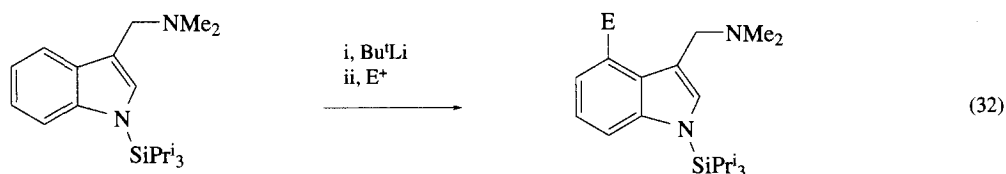


Scheme 38

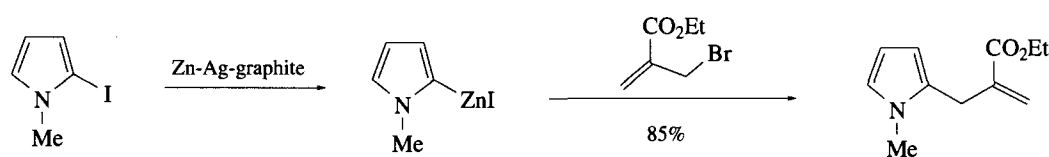
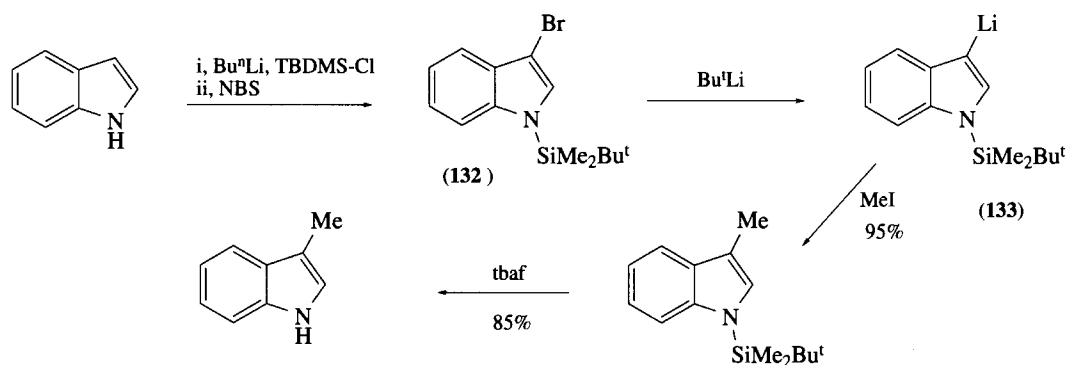
although it is easier to doubly lithiate at the *ortho* arylcarbon as well (93T10271). The most convenient reagent is a butyllithium and potassium *t*-butoxide mixture. Trapping with ketones, epoxides, alkyl halides, and disulfides gives product yields of 52–81%. On the other hand, use of two equivalents of butyllithium in the presence of TMEDA gives quantitative dilithiation. Lithiation can be diverted away from C-2 by the implementation of a bulky *N*-substituent. Although 1-methylgramine is cleanly lithiated at C-2, 1-(triisopropylsilyl)gramine is lithiated selectively at C-4 and can lead to useful 4-substituted indoles: electrophiles include 1,2-dibromoethane, DMF, and diphenyl sulfide (Equation (32)) (93H(36)29).



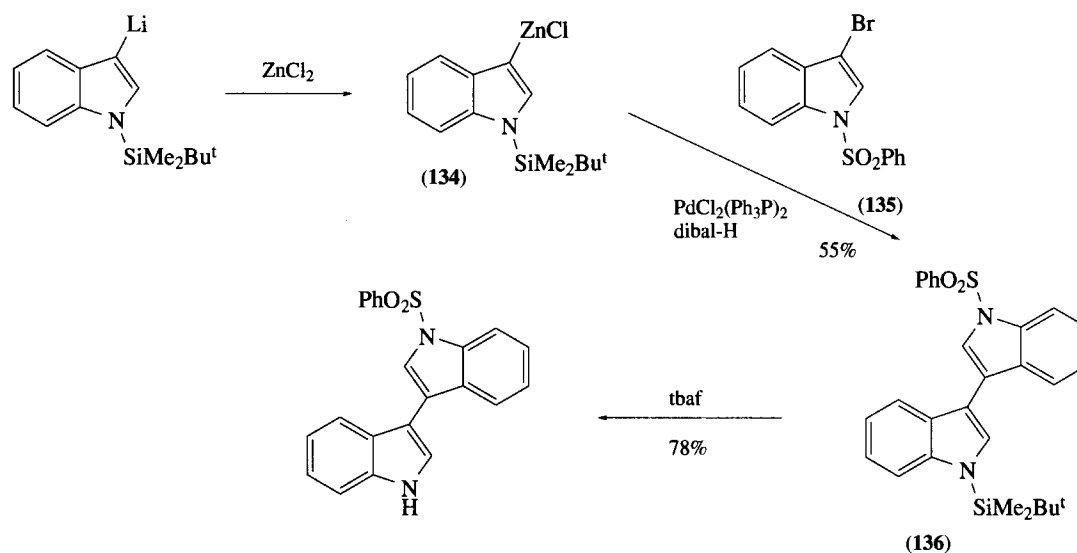
Scheme 39



Lithiation can also be introduced in the conventional way from halo compounds, for example, the sodium salt of 5-bromoindole can be converted to the 5-lithio derivative by treatment with *t*-butyllithium, and in turn leads to various 5-substituted indoles (92H(34)1169). Similarly, the potassium salts of the 4-, 6-, or 7-bromoindoles have been submitted to lithiation and subsequent substitution (86JOC5106). *N*-Protected 3-lithioindoles have received relatively little attention. A preparation of the 3-lithio compound (133) by halogen-metal exchange from the 3-bromo compound (132) has expanded the synthetic utility of this methodology (Scheme 40) (94JOC10). Pyrrolyl and indolyl derivatives of a range of metals other than lithium have received considerable attention, especially as precursors for coupling reactions. These derivatives can be prepared either directly from halo compounds or from the lithio compounds. Direct formation of the pyrrolylzinc compounds can be effected under very mild conditions by treatment of an iodide with a zinc-silver couple deposited on graphite. The zinc reagents are formed in excellent yields and can be converted into acylated or allylated products (Scheme 41) (94TL1047).

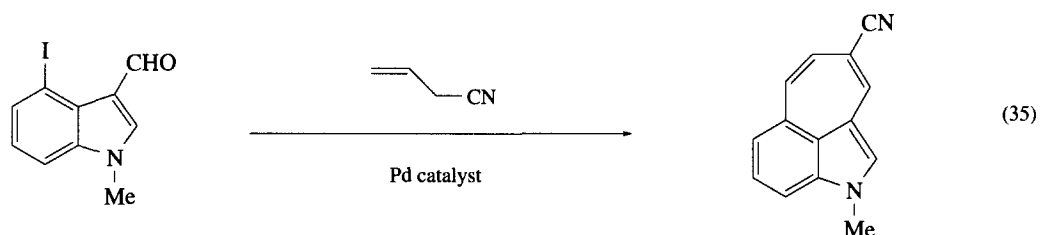
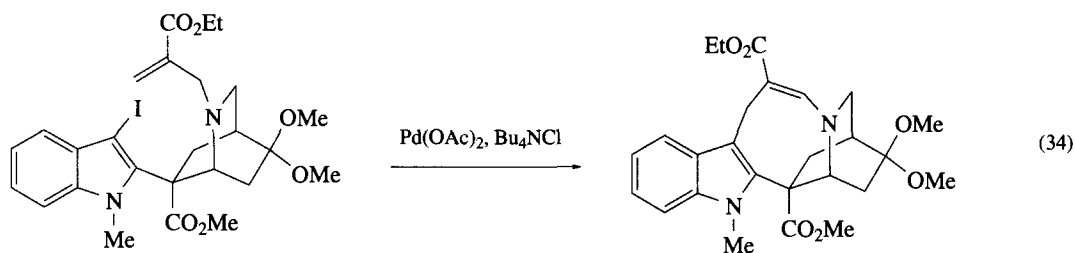
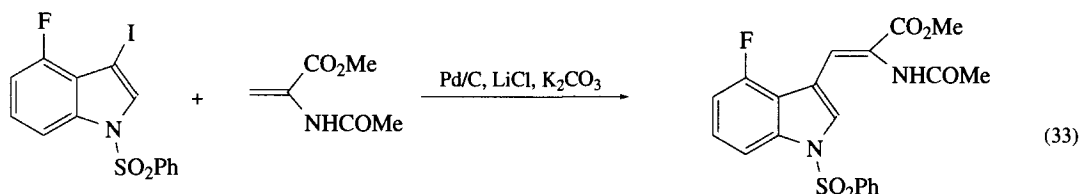


The 2-, 3- and 4-iodozinc derivatives of 1-benzenesulfonylindole have also been prepared from the corresponding iodides by reaction with activated zinc <93TL5955>. The indolylzinc compounds can also be formed by metal-metal exchange from the lithio compounds and have found wide utility in palladium-catalyzed arylation reactions <93H(36)941, 93TL5005>. Compatible N-protection can be provided by butoxycarbonyl(BOC), lithiocarboxy, benzenesulfonyl, and alkylsilyl groups. In particular, the 3-indolylzinc compound (134) can be coupled with the 3-bromoindole (135) to give the 3,3'-biindolyl (136) <94TL793> (Scheme 42). Indole-2-carbonitriles can be formed from the reaction of indolylzinc compounds with *p*-toluenesulfonyl cyanide <93TL4623>. 3-Methylthioindoles can similarly be obtained from organozinc intermediates by reaction with dimethyl disulfide <93TL6245>.

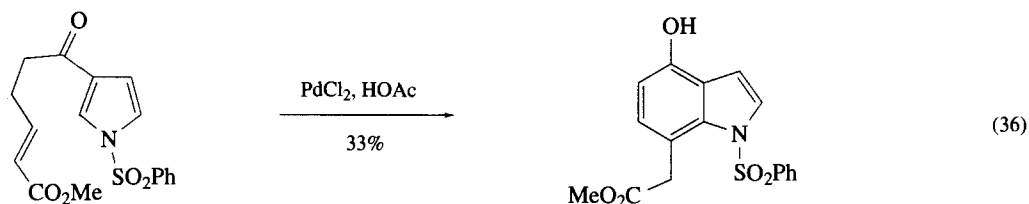


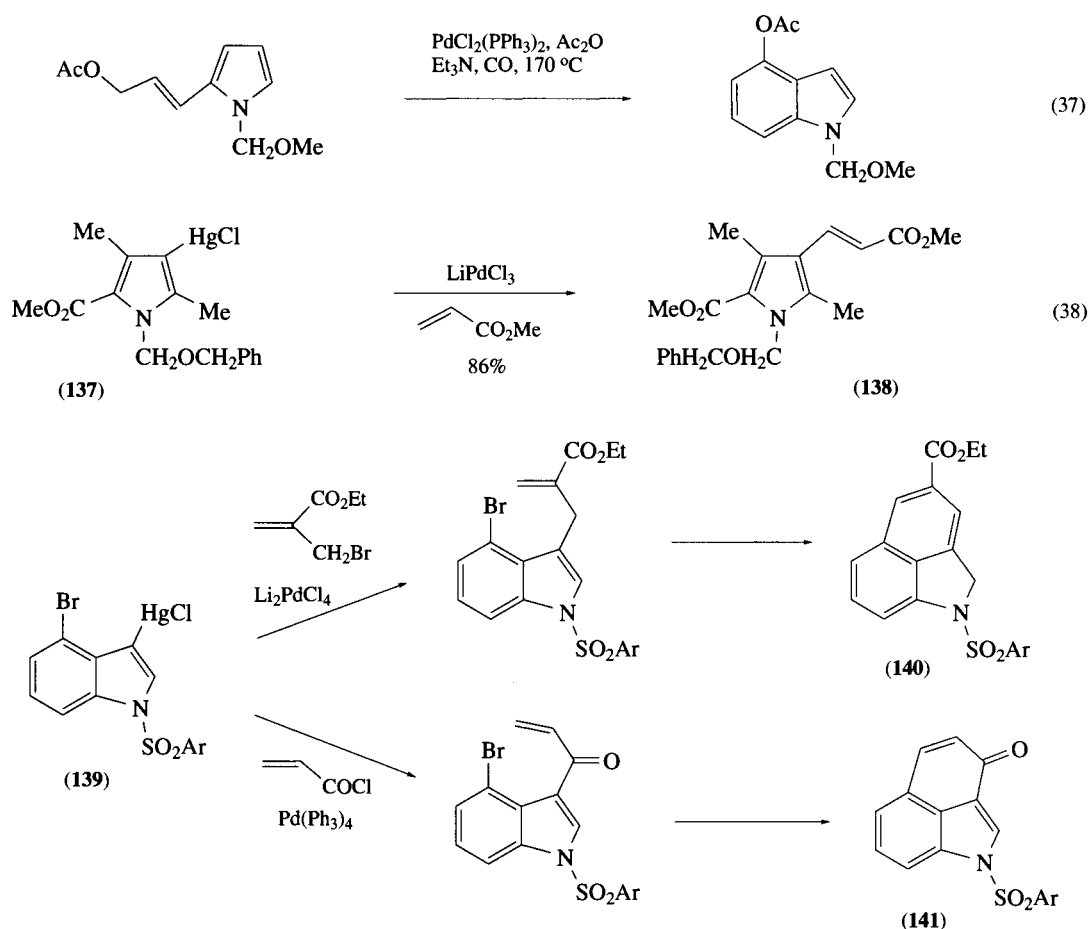
Palladium-catalyzed coupling reactions of the Heck type have in many instances involved indole and pyrrole derivatives. Although the mechanisms are complex, organopalladium species are impli-

cated <84H(22)1493>. Vinylation of *N*-substituted-3-iodoindoles with amidoacrylate groups provides a useful functionalization of indoles (Equation (33)) <90JOM(391)C23>. Yields are improved in intramolecular reactions (Equation (34)) <90JOC6028>. Palladium-catalysed coupling of 4-iodo-1-methylindole-3-carbaldehyde with 3-butenitrile can be used to build a seven-membered ring from C-3 to C-4 (Equation (35)) <92H(34)219>.

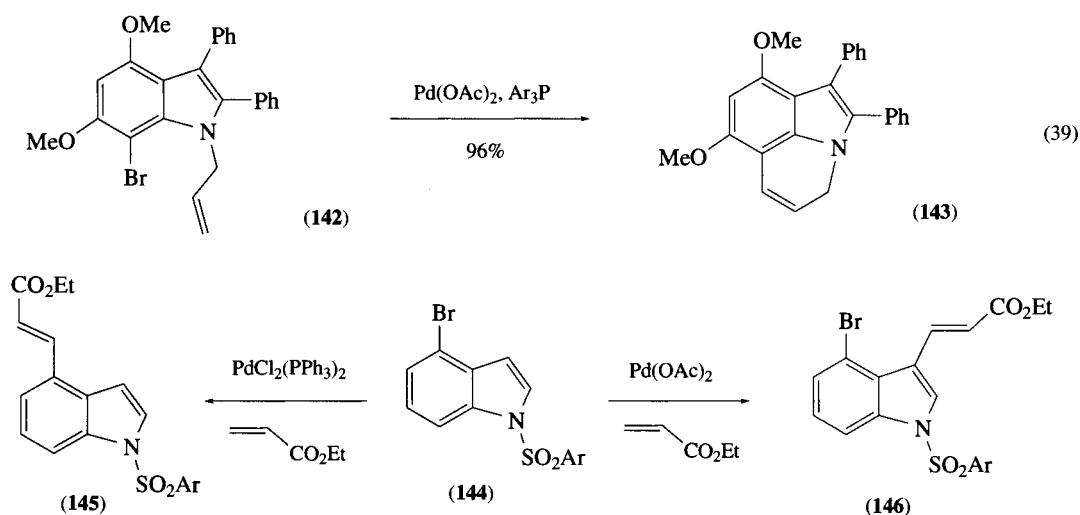


In the case of the intramolecular vinylation of pyrroles, a useful indole synthesis has been developed (Equation (36)) <91CPB2830>. Related processes have been reported to combine carbonylation with cyclization and these can be applied to the synthesis of indoles and carbazoles (Equation (37)) <89TL5883, 91JOC1922, 93TL7471>. Pyrrolylpalladium intermediates for vinylation can be formed from chloromercuripyrroles, which are readily available from the mercuriation of *N*-substituted pyrroles. Derivatives such as pyrrole (**137**) can be coupled with acrylic esters to give pyrrolylacrylates (**138**) (Equation (38)) <89JOC4801>. The related chloromercuriindole (**139**) undergoes palladium-catalyzed allylation and acylation, followed by vinylic coupling at C-4 to give the pyrroloquinoline derivatives (**140**) and (**141**) (Scheme 43) <89JOC4141>. A similar cyclization of a 4-bromo- α -allyltryptophan gives a mixture of annulated indoles containing 8- and 7-membered rings <94TL939>. Another pyrroloquinoline (**143**) is formed from a 1-allyl-7-bromoindole (**142**) on treatment with palladium acetate and tri-*o*-tolylphosphine (Equation (39)) <92T7601>. The palladium catalysed vinylation of bromoindoles can also be applied to 1-methoxyindole-3-carbaldehydes <86CPB4116>. Furthermore, a stoichiometric palladation extends the utility of this coupling process, for example the 4-bromoindole (**144**) can give either the 4-vinyl (**145**) or 3-vinyl (**146**) products, depending on the reaction conditions (Scheme 44) <93H(36)1739>. Palladium catalysed coupling has also been effected with a vinyl enolate, so that the product can be transformed into a ketone. This technique has been used in a synthesis of hapalindole Q <93JA3499>.





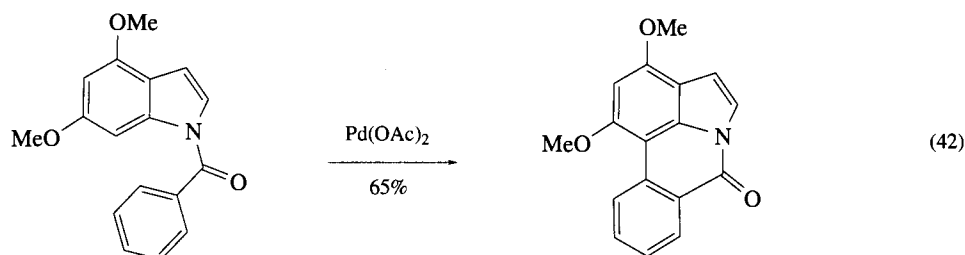
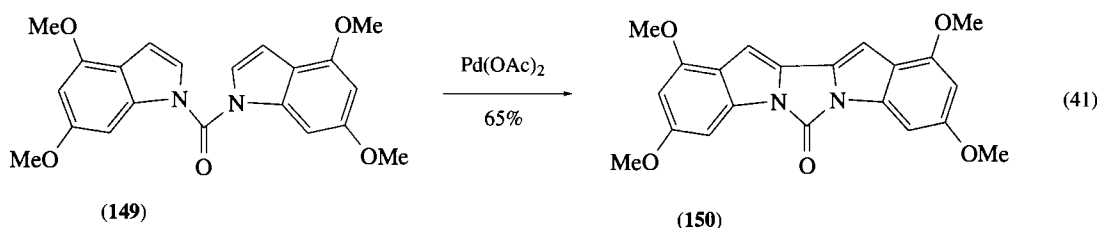
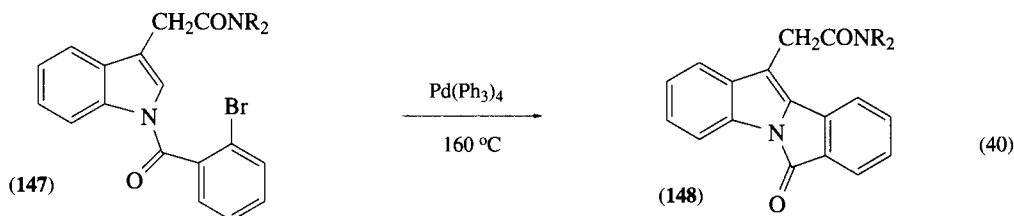
Scheme 43



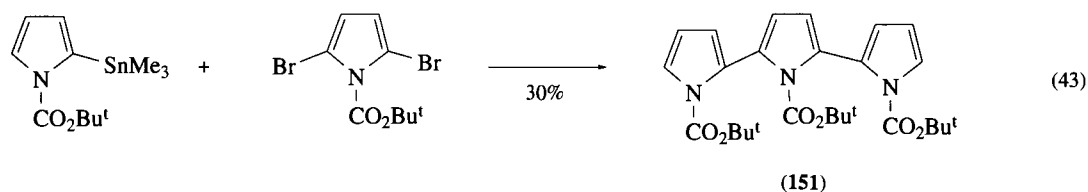
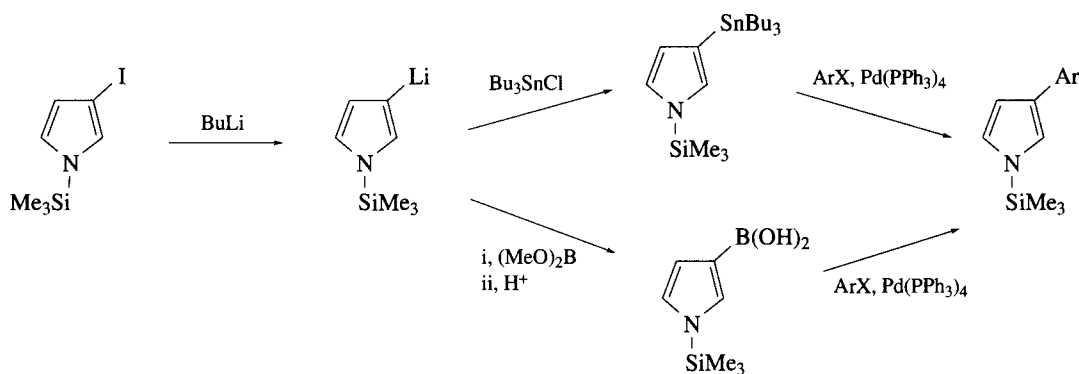
Scheme 44

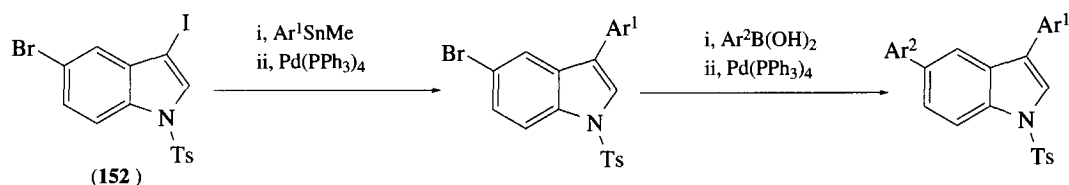
Aryl coupling can also be effected by palladium complexes and can be illustrated by the intramolecular coupling of the *N*-acylindole (147) to the tetracyclic compound (148) (Equation (40)) <91TL3317>. Unbrominated aryl rings can also be coupled using palladium acetate in a stoichiometric process and can also be considered within the current context. Relevant indole examples include the conversion of *N,N'*-carbonylbis-4,6-dimethoxyindole (149) into the biindolyl (150) <85CC1174>

(Equation (41)). Whereas the *N*-aryloindoles undergo similar cyclization exclusively at C-2 <89TL5807>, the 4,6-dimethoxy analogs are converted by cyclization at C-7 into pyrrolo-phenanthridones in moderate yields (Equation (42)) <93T151>.



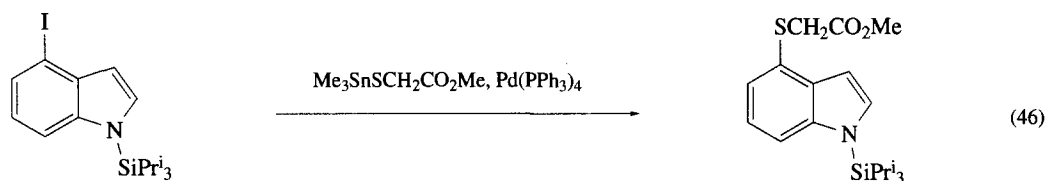
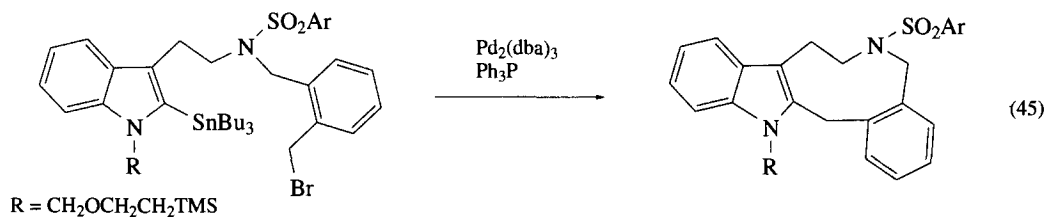
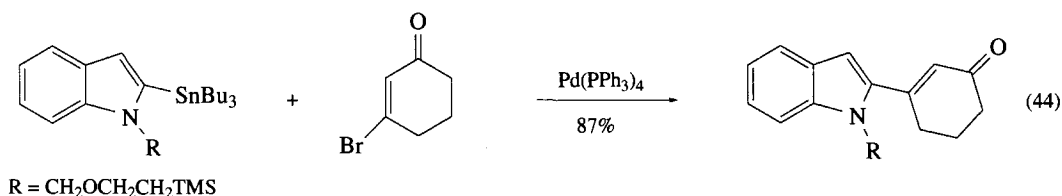
Pyrrolyl- and indolylstannanes and boronic acids, which can be prepared from the corresponding organolithium derivatives, have received increasing use in palladium-catalyzed coupling reactions with aryl halides (Scheme 45) <91S613, 92JOC1653>. This technique has allowed the preparation of a tripyrrole (151) (Equation (43)) <91SM403>. Different halogens on indoles can sometimes be differentiated. An excellent example is shown by the 5-bromo-3-iodoindole (152), which can be arylated selectively at C-3 by an arylstannane and subsequently at C-5 by an arylboronate. Both coupling reactions are catalyzed by palladium complexes (Scheme 46) <92SC1757>.





Scheme 46

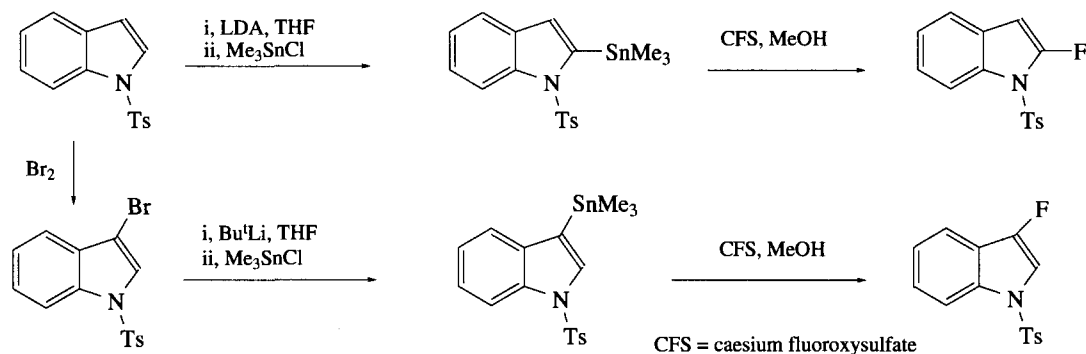
N-Substituted 2-indolylstannanes have been studied rather thoroughly with respect to both the N-substituents and electrophilic coupling agents. N-Methyl- and N-(trimethylsilyl)ethoxymethylindoles are very reactive and the silyl group can be readily removed from the 2-indolyl product by treatment with fluoride ion. Electrophiles include allyl, benzyl, vinyl, aryl, heteroaryl, acyl and vinylogous acyl halides, and triflates (Equation (44)) $\langle 93\text{HCA}2356, 94\text{JOC}4250 \rangle$. Intramolecular 2-vinylation and 2-benylation of a 2-indolylstannane has also been reported (Equation (45)) $\langle 93\text{SL}771 \rangle$. A 4-iodo group on an indole can be effectively replaced by a thioether group in a palladium-catalyzed reaction with a thiostannyl derivative (Equation (46)) $\langle 92\text{JCS}(\text{P}1)323 \rangle$.



An efficient method for the synthesis of 3-substituted indoles is afforded by the palladium-catalyzed reaction of 1-tosyl-3-tributylstannylindoles with a variety of vinyl, aryl, and heteroaryl halides and triflates $\langle 94\text{TL}2405 \rangle$. In a reverse process, 4-indolyltriflates and 5-pyrrolo[2,3-d]pyrimidinyltriflates undergo coupling with vinylstannanes and arylstannanes, respectively $\langle 88\text{JA}1557, 88\text{JOC}1170, 93\text{JOC}403 \rangle$. A 4-indolylthallate has been coupled with a 3-pyridylstannane, using palladium acetate in DMF $\langle 87\text{CPB}1322 \rangle$.

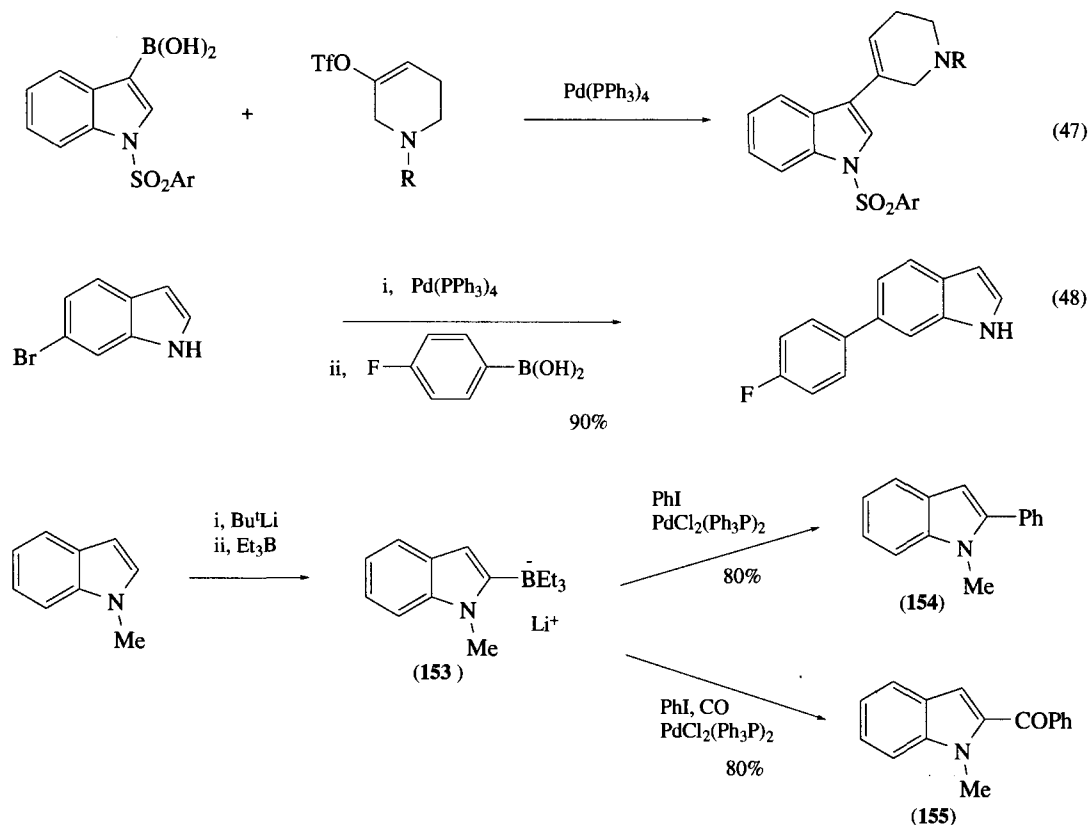
Fluorinated indoles can be obtained in good yield from the 2- and 3-stannyl derivatives by reaction with cesium fluoroxysulfate (CFS) and methanol $\langle 94\text{T}1899 \rangle$. The nitrogen must be protected and the stannyl compounds are prepared by routine exchange of the respective lithio derivatives (Scheme 47).

Boronic acids derived from 3- and 5-substituted indoles are useful intermediates for palladium-catalyzed vinylation, arylation, and heteroarylation (Equation (47)) $\langle 92\text{H}(34)1395, 93\text{TL}2235 \rangle$. As with the stannanes, a reverse process can be utilized. 6- and 7-Bromoindoles can be coupled directly with arylboronic acids to give the 6- and 7-arylindoles, respectively; a feature of the reaction is that protection of the indole nitrogen is unnecessary (Equation (48)) $\langle 94\text{SL}93 \rangle$. A new route to 2-substituted indoles has been developed from trialkyl-(1-methylindol-2-yl)borates. The borate (153),



Scheme 47

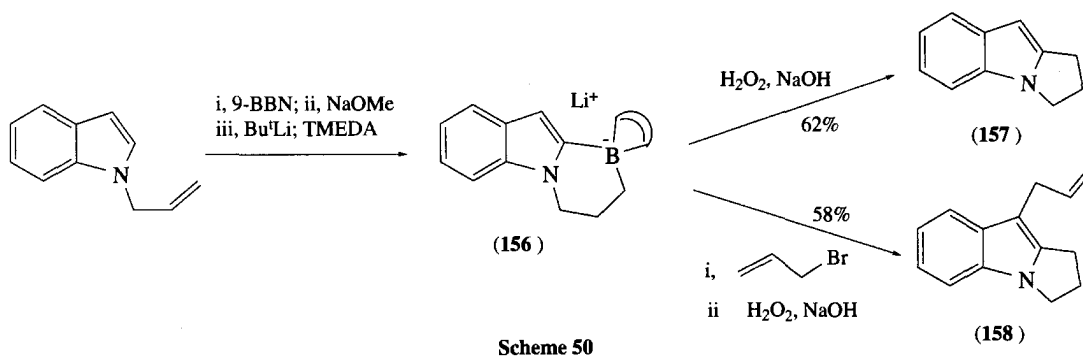
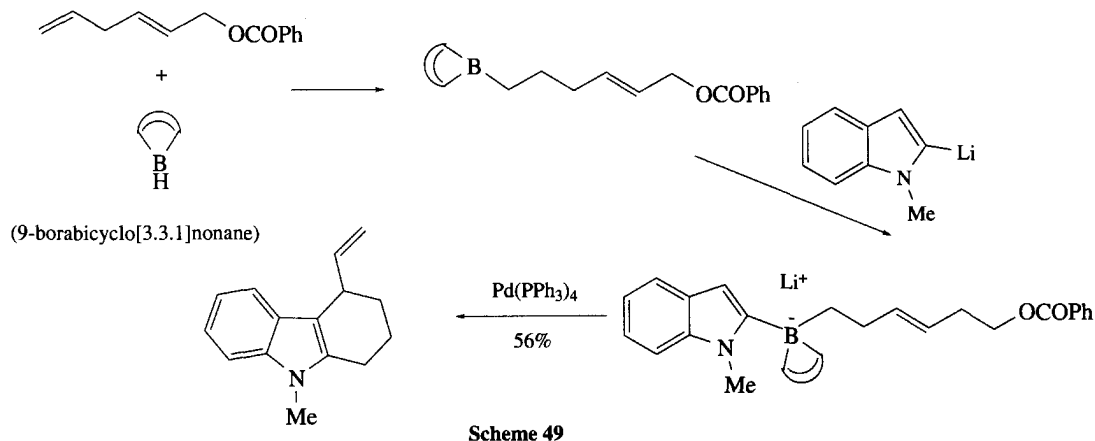
formed from 1-methylindole and *t*-butyllithium followed by treatment with triethylborane, can be converted into the 2-phenylindole (**154**) on reaction with iodobenzene in refluxing THF in the presence of a palladium catalyst <88H(27)203, 89CC135>. The same type of borate also undergoes a palladium-catalysed carbonylation and cross-coupling to yield indol-2-yl ketones (**155**); the coupling group can be an aryl iodide or a vinyl triflate (Scheme 48) <94JOC2634>. A similar organocopper species can be allylated and vinylated <89CC727>.



Scheme 48

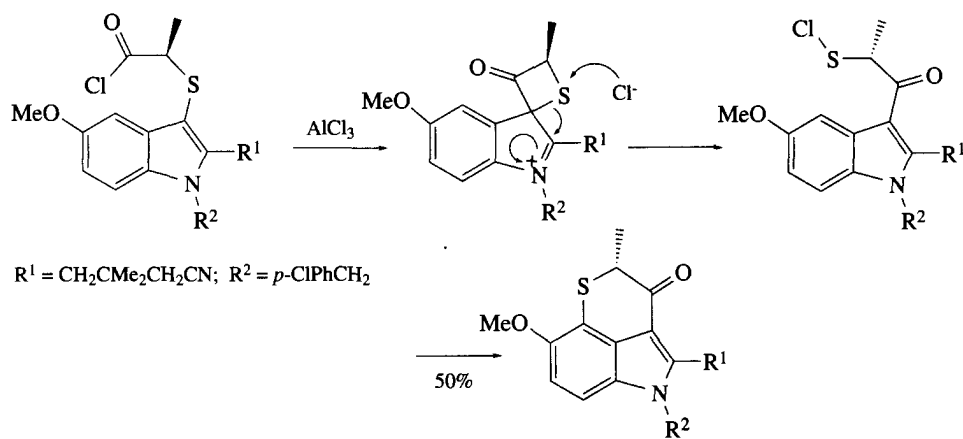
A tandem strategy for indole annulation has been developed from indolylborates. Initial *b*-annulation was effected by the following sequence; hydroboration of a terminally unsaturated allylic acetate with 9-BBN, reaction with 2-lithio-1-methylindole to generate the borate; palladium-induced cyclization at C-3, migration from boron to carbon; and aromatization (Scheme 49) <91CC1219>. A development of this uses the hydroboration derivative (**156**) of a 1-alkenylindole, followed by

lithiation and cyclization at C-2. Subsequent electrophilic substitution or palladium-catalyzed coupling at C-3, induces a boron to carbon migration and oxidative work-up yields the α -annulated indoles (157) and (158), respectively (Scheme 50) <92TL6849>.



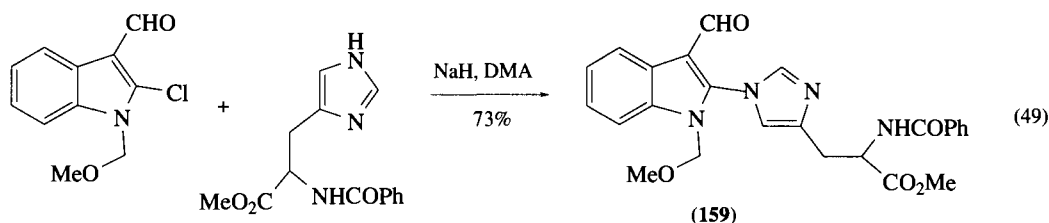
2.02.1.3.8 Miscellaneous electrophilic reactions

The formation of indole thioethers can be carried out at C-4 by a process involving migration from C-3. An intramolecular acylation of 2-(3-indolylthio)propionic acid undergoes reaction at C-3, with rearrangement of the sulfur moiety and substitution at C-4 via a sulfenyl chloride; there is also good optical retention in a chiral substrate (Scheme 51) <92TL4717>.

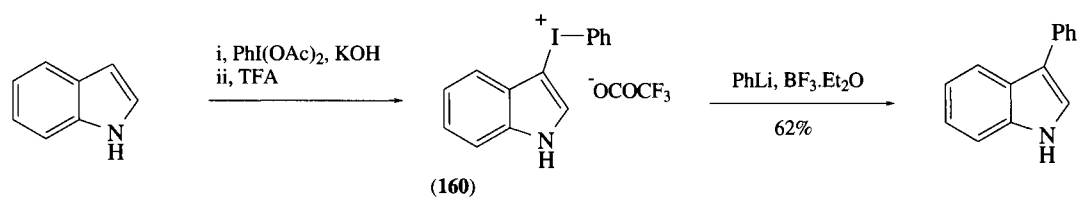


2.02.1.4 Reactions with Nucleophiles

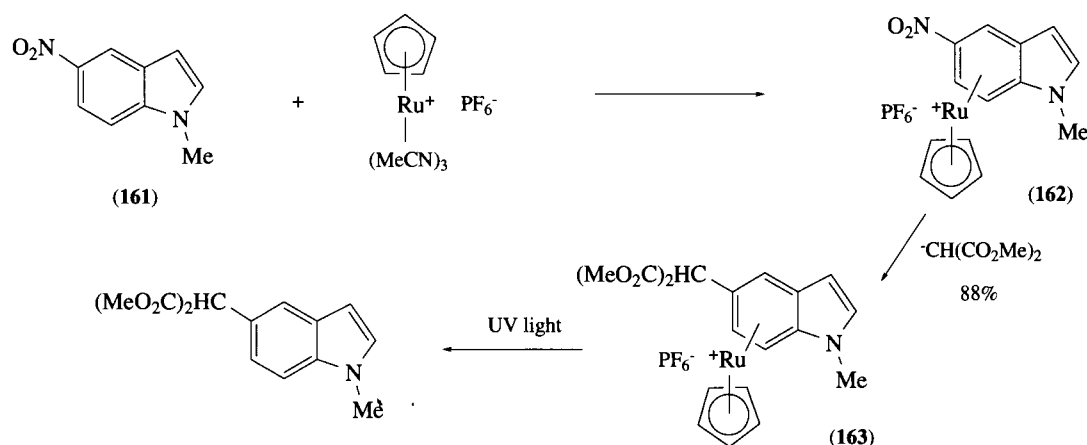
2-Chloroindole-3-carbaldehydes are excellent substrates for a range of nitrogen nucleophiles such as azide ion ^{<88CC1583>}, pyrrole, indole, and other nitrogen heterocycles ^{<92S731>}. Use of a histidine-based nucleophile gives an *N*-(2-indolyl)imidazole (**159**), which serves as a precursor for the tryptophan residue of moroidin, a bicyclic octapeptide (Equation (49)).



Nucleophilic substitution at C-3 of indoles is rare. However, phenyl(3-indolyl)iodonium trifluoroacetate (**160**) reacts with alkyl- and aryllithium compounds to yield the 3-alkyl- or 3-arylindoles, together with iodobenzene formed by reductive elimination (Scheme 52) ^{<87TL3071>}. Cyclopentadienylruthenium complexes of indoles have been found to undergo displacement of halogen atoms at C-4 and C-5, or nitro groups at C-4, C-5, and C-7, by a wide range of nucleophiles such as amines, alkoxides, thiolates, and malonate ions in excellent yields ^{<88CC1621, 88OM660>}, for example the ruthenium complex (**162**), which can be prepared directly from the nitroindole (**161**) by thermal ligand exchange, undergoes displacement of the nitro group by malonate to give the product (**163**) (Scheme 53).



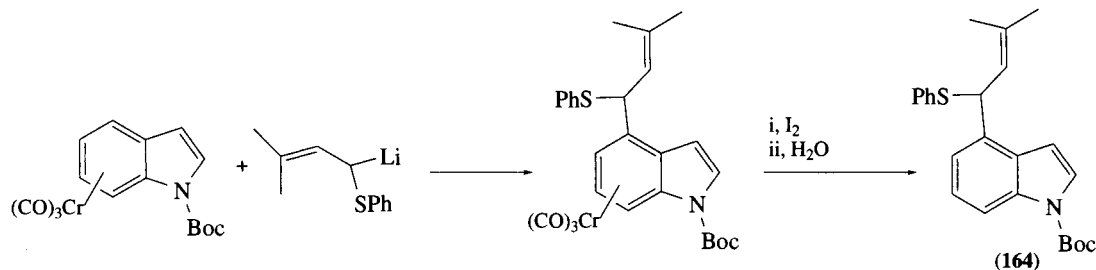
Scheme 52



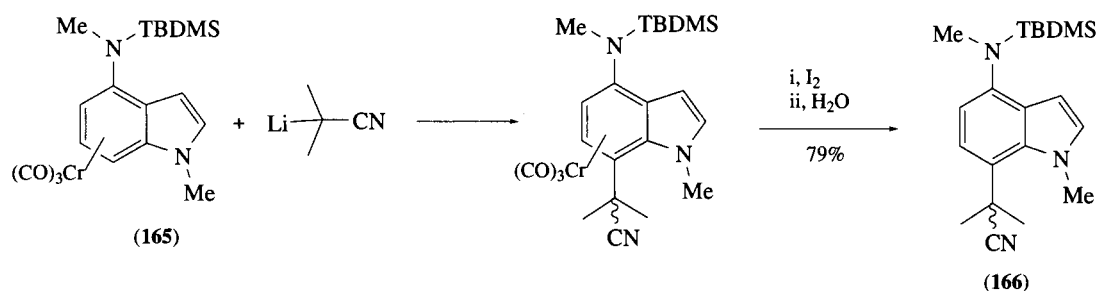
Scheme 53

Functionalization of indoles at C-4 can be achieved by the addition of nucleophiles to indole tricarbonylchromium complexes, followed by oxidative removal of the chromium. A recent example, from 1992, shows the formation of the 4-allylindole (**164**) following carbanion addition (Scheme 54) ^{<92AJC99>}. This technique has also been applied to formation of a 4-indolylbutenone, a key intermediate in the synthesis of clavicipitic acid ^{<93TL5051>}. 7-Substitution of the indole ring can

also be achieved by nucleophilic displacement reactions of the chromium carbonyl complexes of 4-methylaminoindoles <93TL1399>. The indole complex (**165**) reacts with the lithium carbanion of a tertiary nitrile to give the 7-substituted product (**166**) (Scheme 55).



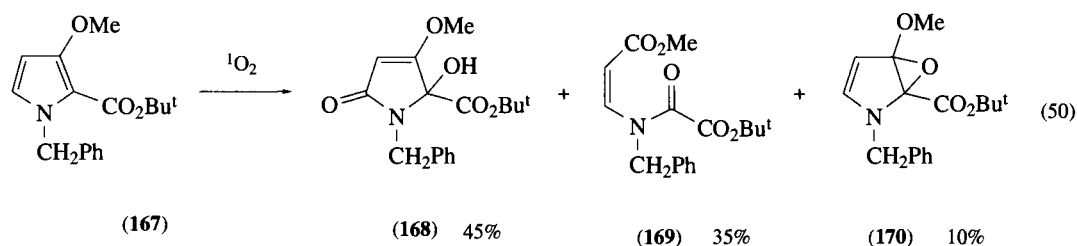
Scheme 54

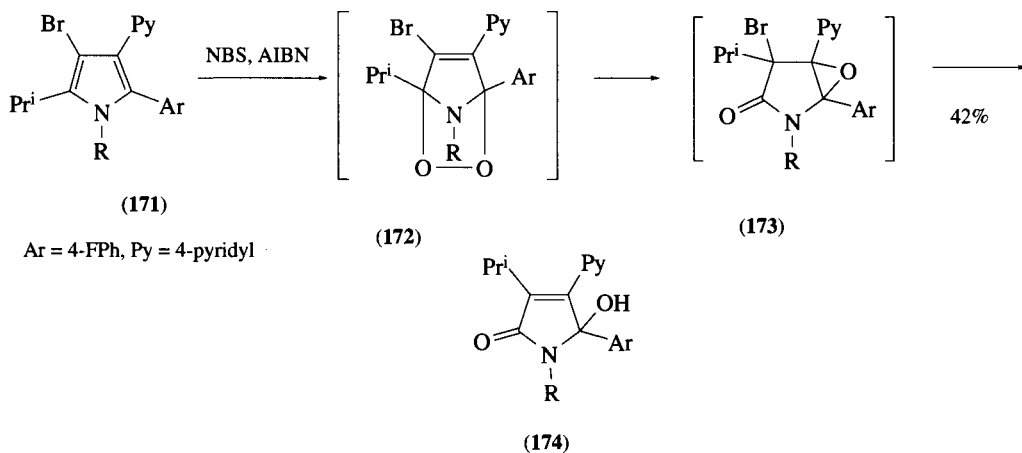


Scheme 55

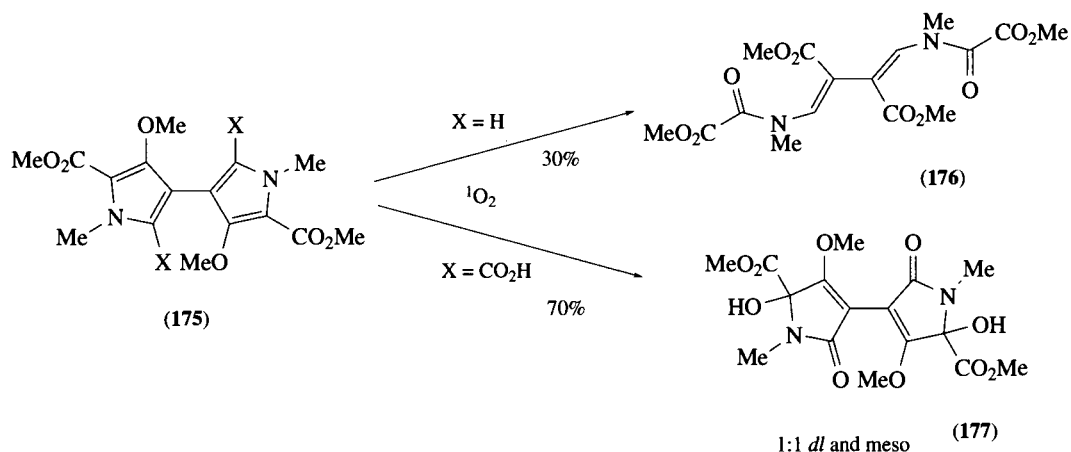
2.02.1.5 Oxidation of the Heterocyclic Ring

Both pyrroles and indoles are susceptible to a wide range of oxidation processes which are synthetically useful. These include oxygenation and cleavage reactions, with the latter usually arising from unstable cyclo-oxygenated intermediates. Photooxygenation of the trisubstituted pyrrole (**167**) has been shown to give the hydroxy lactam (**168**), together with lower yields of the cleavage product (**169**) and the epoxide (**170**) (Equation (50)) <91TL7571>. A similar transformation was observed when the bromopyrrole (**171**) was treated with NBS in the presence of AIBN, and gave rise to the hydroxylactam (**174**) (Scheme 56) <94JCS(P1)245>. Although this is presumably a radical process, it is included here because the proposed mechanism appears to involve typical oxygenation intermediates, such as the endoperoxide (**172**) and the epoxide (**173**). In comparison with the photooxygenation of the single pyrrole (**171**), the 3,3'-bipyrroles (**175**) are converted into the cleavage product (**176**) and the bipyrrolone (**177**) (Scheme 57) <93JA8457>. An example of the synthetic utility of the oxidative cleavage of the pyrrole C-2 to C-3 bond is the ring expansion of bicyclic pyrroles (**178**) to the lactams (**179**) (Equation (51)) <82SC390>.

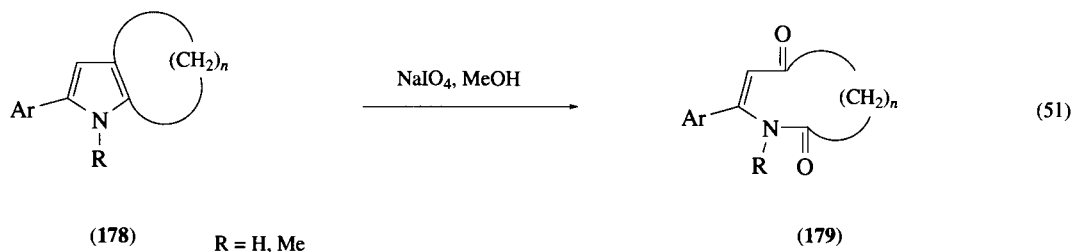




Scheme 56

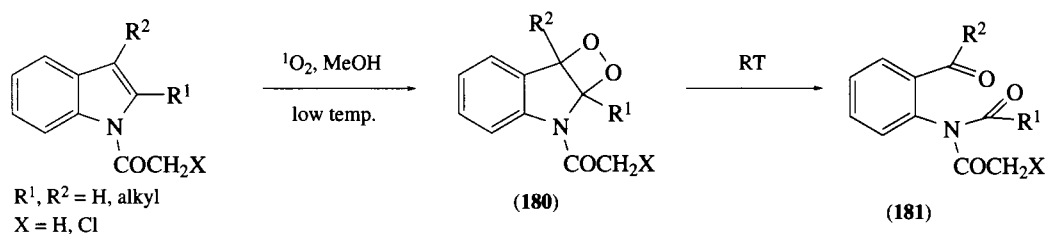


Scheme 57

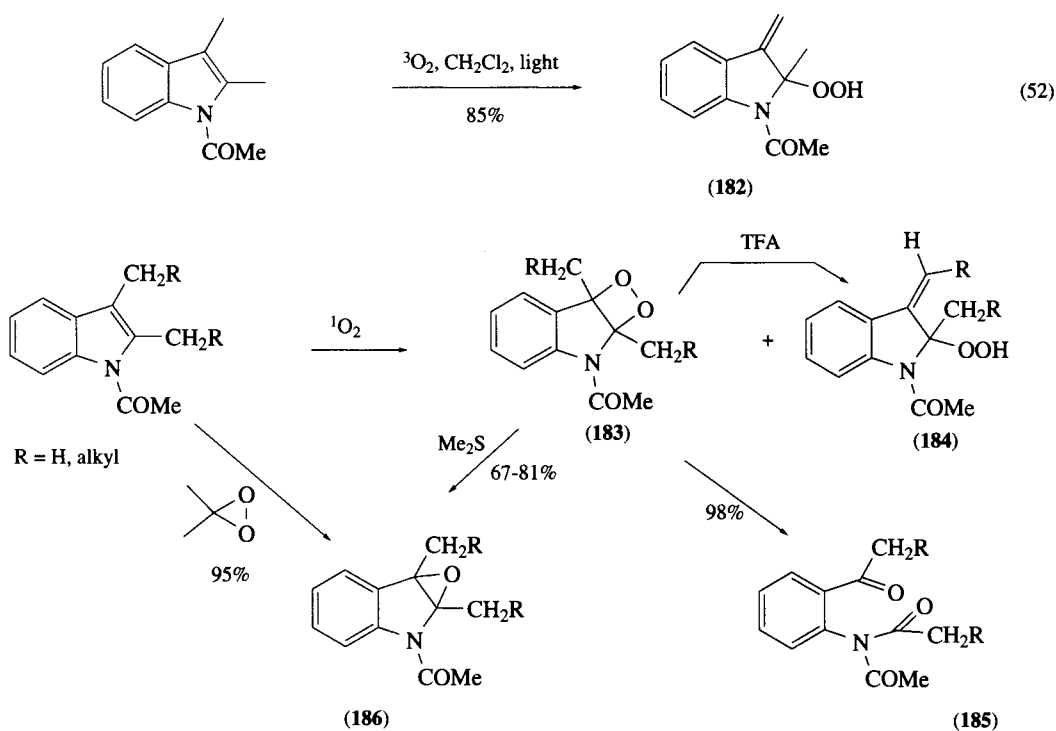


The oxidation of indoles continues to be studied in detail, and with the development of new reagents, more precise reaction conditions afford the isolation and characterization of significant oxygenated intermediates. Photosensitized oxidation of *N*-acylindoles using xenon lamp irradiation in methanol generates dioxetanes (**180**), whose structures could be characterized at low temperature by proton and carbon NMR spectroscopy, and which decompose smoothly on warming to give the oxo-imide cleavage products (**181**) (Scheme 58) <93JOC5524>. On the other hand, tpp-sensitized photooxygenation in dichloromethane gives exclusively the 2-hydroperoxy-3-methyleneindoline (**182**) (Equation (52)) <93JOC47>. A more recent study, in the early 1990s, has developed this chemistry to include transformations of some of the key intermediates <93TL5247, 94JOC2733>. The intermediate dioxetane (**183**) can be converted by acid catalysis into the hydroperoxide intermediate (**184**), to the oxo-imide (**185**) by thermolysis, and most significantly to the epoxide (**186**) by treatment with dimethylsulfide (Scheme 59). The epoxide can also be obtained by the reaction of the *N*-acyl indole with dimethyldioxirane. On warming to room temperature, the epoxide is transformed into the oxindole (**187**) resulting from migration of the group from C-2 to C-3 (Scheme 60) <92CB2719,

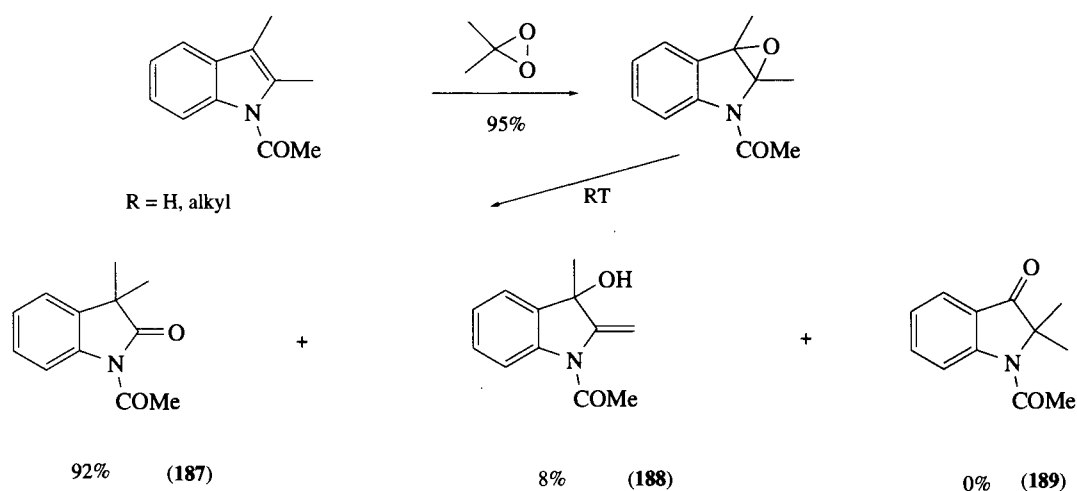
93JA8867, 94JOC2733). One of the epoxides has been characterized by x-ray crystallography. In the case of substrates bearing larger C-3 substituents, the yields of products corresponding to the methyleneindoline (188) and indolin-3-one (189) increase. For instance, with a 3-*t*-butyl group, the yields of oxindole, methyleneindoline, and indolin-3-one are 0%, 70%, and 30% respectively.



Scheme 58

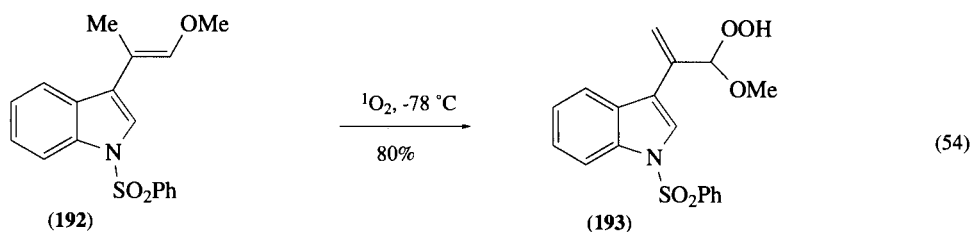
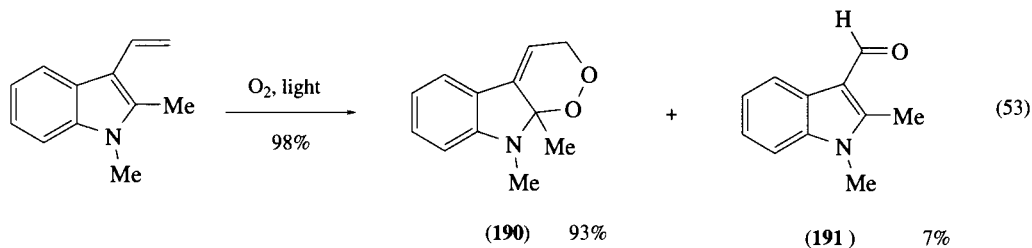


Scheme 59

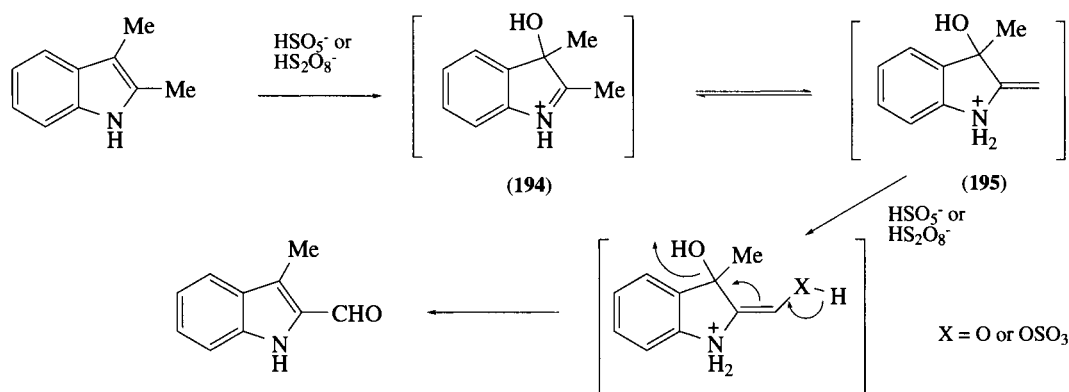


Scheme 60

A variety of 1-methyl- and 1-(benzenesulfonyl)-3-vinylindoles have been studied with respect to their oxidation with singlet oxygen. The predominant reaction is the [4 + 2] cycloaddition leading to an endoperoxide (**190**), but some vinyl cleavage also occurs to give the 3-carbaldehyde (**191**) (Equation (53)) <93JOC7839>. The methoxyvinyl group of the indole substrate (**192**) influences the predominance of an ene reaction over the [4 + 2] cycloaddition, resulting in the formation of the allylic hydroperoxide (**193**) (Equation (54)).

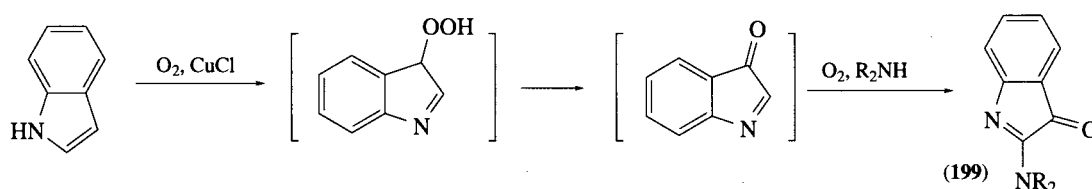
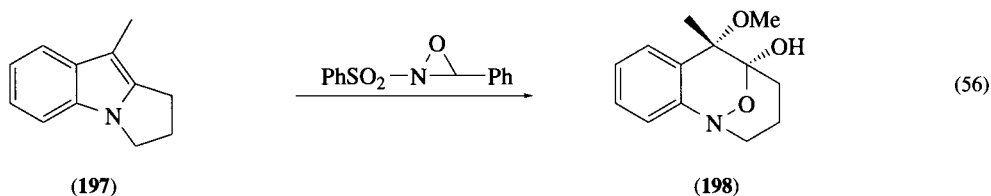
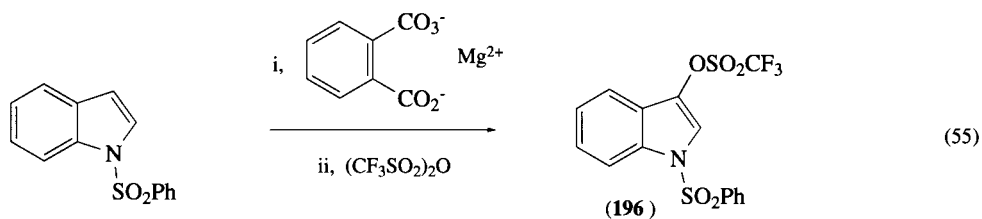


The oxidation of 2,3-dimethylindole with peroxodisulfate and peroxomonosulfate has been shown to yield 3-methylindole-2-carbaldehyde <88JCS(P2)1065>. Mechanistic studies have established that the reaction proceeds in three steps, namely electrophilic attack of the peroxide at C-3 to give an indolenine intermediate (**194**), peroxide attack on the enamine tautomer (**195**), and hydrolysis (Scheme 61) <93JOC7469>.



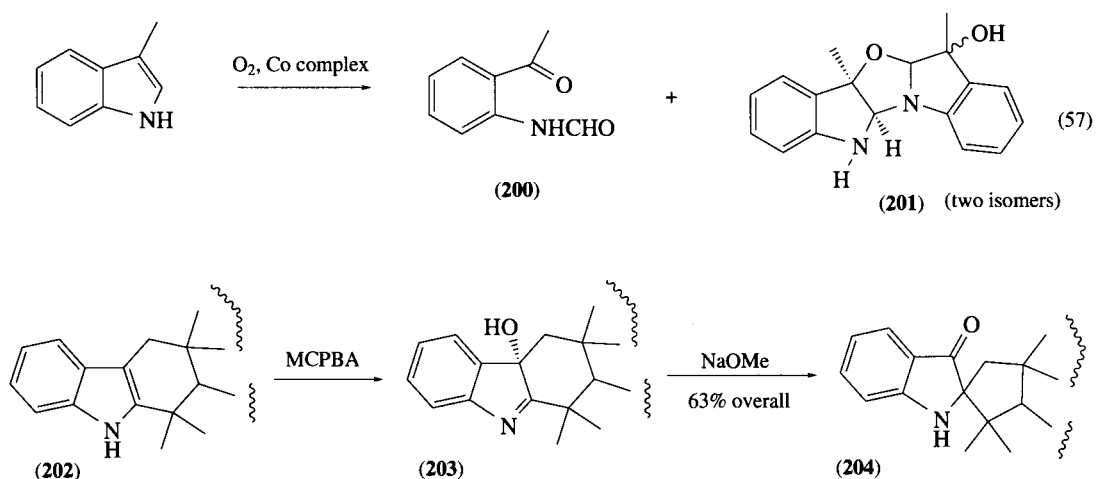
Scheme 61

1-Benzenesulfonylindole can be oxidized with magnesium monoperoxophthalate and the intermediate 3-hydroxy compound trapped with triflic anhydride to give the indole triflate (**196**), which is useful for coupling reactions (Equation (55)) <90H(30)627>. Peracid oxidation also converts the pyrrolo[1,2-*a*]indole (**197**) into the bridged structure (**198**), but the yield is higher with oxygen transfer from *N*-benzenesulfonyl-*C*-phenyloxaziridine (Equation (56)) <92TL5705>. Indole undergoes copper-catalyzed oxidation to a 3-hydroperoxy species which can be trapped by a secondary amine to yield a 2-dialkylamino-3-oxo-3*H*-indole (**199**) (Scheme 62) <93TL2953>.



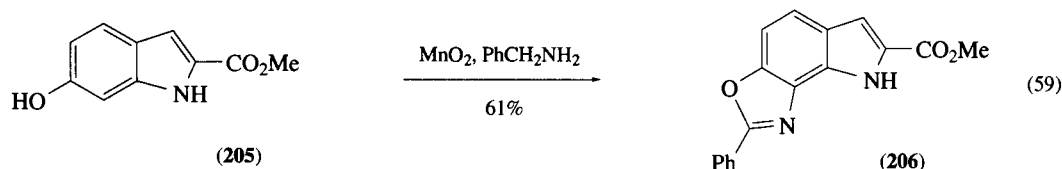
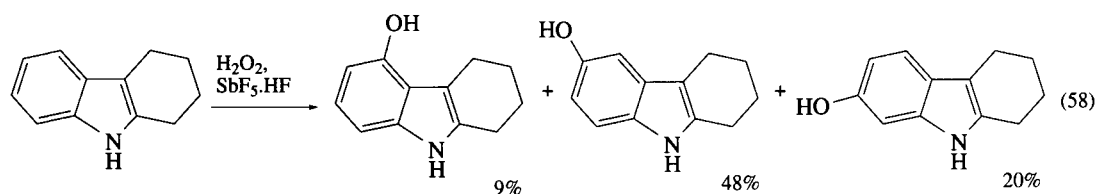
Scheme 62

The normal oxidation mode of indoles leads to cleavage of the C-2 to C-3 bond to produce an oxo-amide. Sometimes an intermediate along this pathway can be diverted to give a different outcome. Although the oxo-amide (**200**) is the major product of the cobalt-catalyzed oxidation of 3-methylindole, two minor products are the pentacyclic dimers (**201**) (Equation (57)) <85CPB1878, 85CPB4167, 89CPB2194>. The brevianamide precursor (**202**) can be converted by peracid oxidation into the 3-hydroxyindolenine (**203**), which is rearranged by treatment with sodium methoxide into the 3-oxo-3*H*-indole (**204**) (Scheme 63) <89TL5575, 90JA808>.

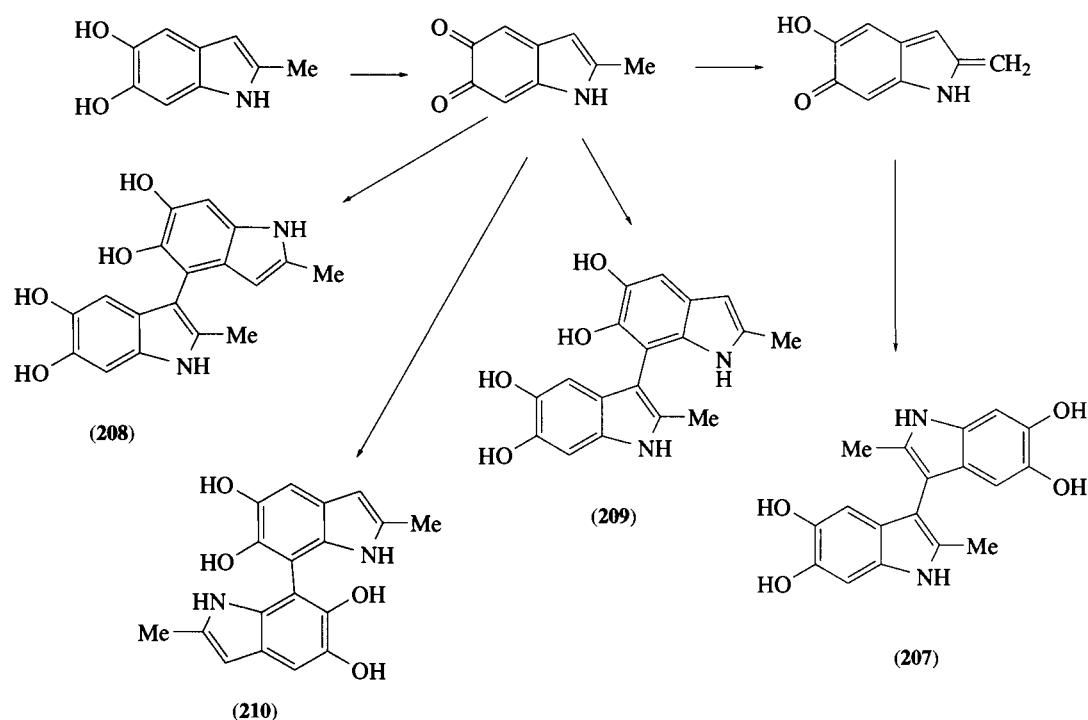


Scheme 63

Indoles undergo acid-catalyzed oxidation leading to mixtures of 4-, 5-, and 6-hydroxyindoles in rather poor yields (Equation (58)) <86TL4565>. Some hydroxylated indoles can be oxidized further to indoloquinones, which can be trapped with nucleophiles such as amines, or which can undergo dimerization. Manganese dioxide oxidation of the 6-hydroxyindole 2-carboxylic ester (**205**) in the presence of a primary methylamine leads to formation of an oxazoloindole (**206**) (Equation (59)) <86CC371, 88JOC5163>.

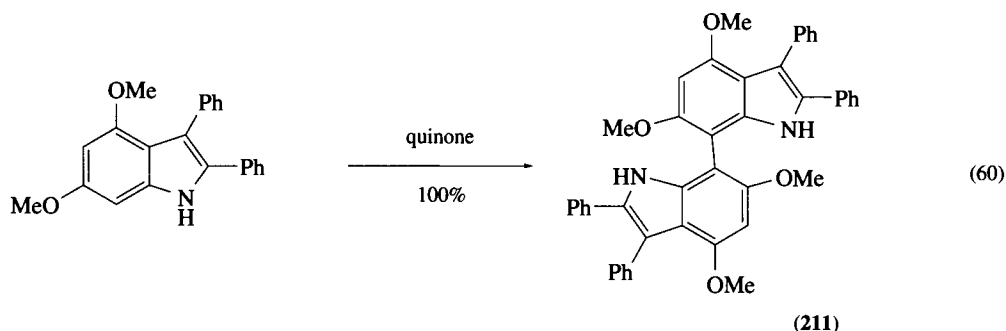


Oxidative dimerization of 5,6-dihydroxyindole-2-carboxylic acid and 5,7-dihydroxytryptamine gives 4,4'-biindolyl dimers in rather poor yields <87TL467, 89JA719>. In contrast, a 2,7'-biindolyl results from the oxidation of 5,6-dihydroxytryptamine <90JOC1484>. A 1993 study has shown that 5,6-dihydroxy-2-methylindole is rapidly oxidized by peroxidase and hydrogen peroxide at neutral pH to give a complex mixture of oligomers including the 3,3'-, the 3,4'-, the 3,7'-, and the 7,7'-biindolyls (**207**), (**208**), (**209**), and (**210**), respectively; these products could be characterised as their acetyl derivatives (Scheme 64) <93T9143>.

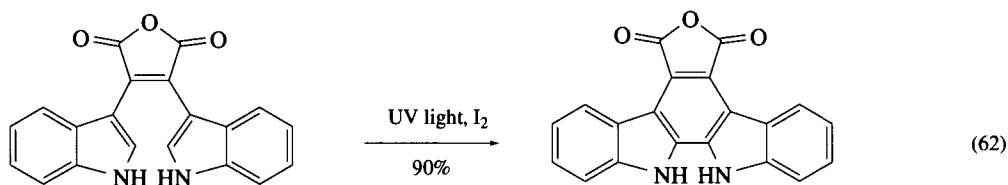
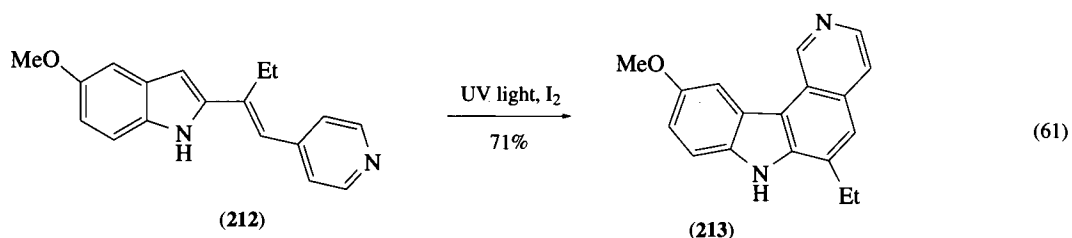


Scheme 64

The electrochemical oxidation of 1,2-dimethylindole leads to the 3,6'-biindolyl <91T737>. One of the highest-yielding examples of an indole oxidative dimerization is that of the electron-rich 4,6-dimethoxy-2,3-diphenylindole, which can be converted to the 7,7'-biindolyl (**211**) by a variety of oxidizing agents including quinones and iodine monochloride (Equation (60)) <89CC111, 94T10497>.

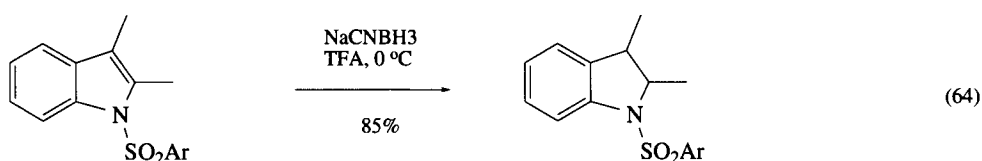
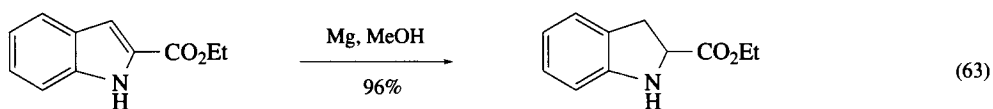


Photoinduced aryl-aryl couplings have found utility in the synthesis of some carbazoles and indolocarbazoles. Cyclization of the stilbene (212) gives the pyridocarbazole (213) (Equation (61)) <85TL4929>. Several groups have made use of this oxidative cyclization in the synthesis of staurosporines, at the stage where the crucial bond is formed between the two indole C-2 positions (Equation (62)) <94S25, 94TL5555>.



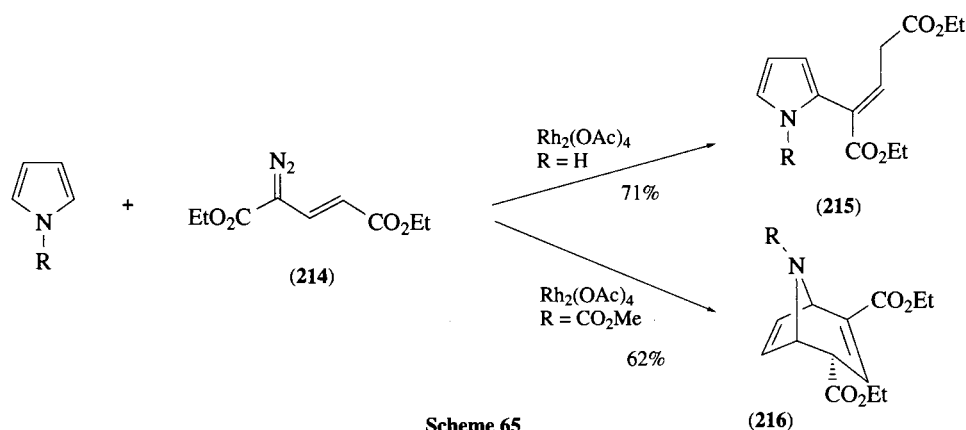
2.02.1.6 Reduction of the Heterocyclic Ring

In a general reduction of α,β -unsaturated esters, indole-2-carboxylic esters undergo reduction with magnesium in methanol to give the related indoline (Equation (63)) <86TL2409>. Indolines can also be produced in good yield from 1-benzenesulfonylindoles by reduction with sodium cyanoborohydride in TFA at 0°C (Equation (64)) <89TL6833>. If acyl groups are present at C-2 or C-3 in the substrate, they are reduced to alkyl groups.

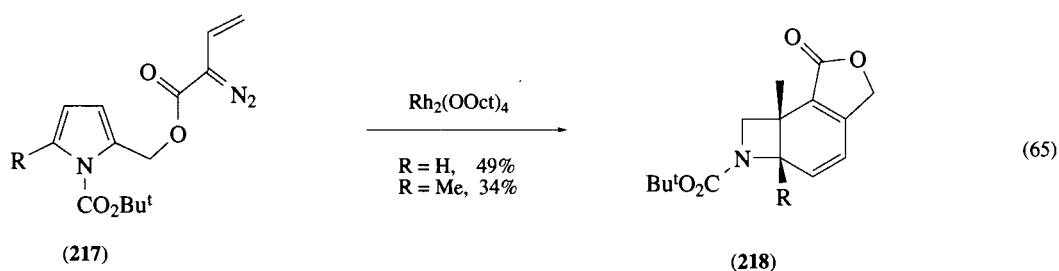


2.02.1.7 Reactions with Carbenes, Nitrenes, and Radicals

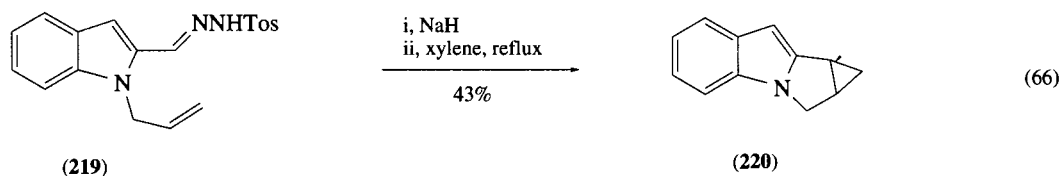
The metal-carbene complexes generated from the interaction of vinylidiazomethane with rhodium acetate undergo reactions with pyrroles in a variety of ways, depending on substitution patterns. The vinylidiazomethane (**214**) reacts with pyrrole itself to give an alkylation product (**215**), whereas it converts the 1-methoxycarbonylpyrrole into a product (**216**) with a tropane skeleton. Formation of the latter product probably results from a sigmatropic Cope rearrangement of a divinylcyclopropane intermediate (Scheme 65) <89TL4653, 93T5203>. The use of more bulky vinylidiazomethanes blocks the Cope rearrangement and a second vinylcarbenoid reaction can occur. The synthetic utility of this chemistry has been demonstrated by the synthesis of natural products such as the tropane alkaloid ferruginine <91JOC5696>.

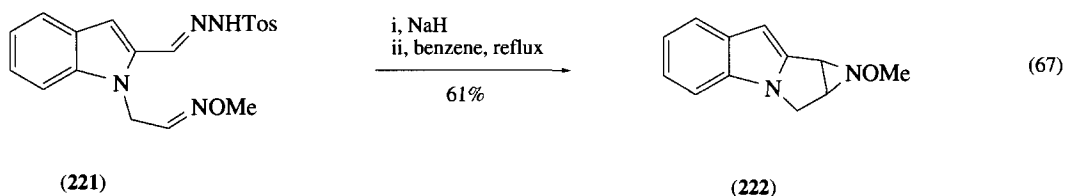


An intramolecular variant of this synthetic methodology has been developed. Pyrrole-linked vinylidiazomethanes (**217**) undergo rhodium-catalysed carbene formation and intramolecular insertion to give moderate yields of fused azetidines (**218**) of the 7-azabicyclo[4.2.0]octadiene type (Equation (65)) <94TL5209>.

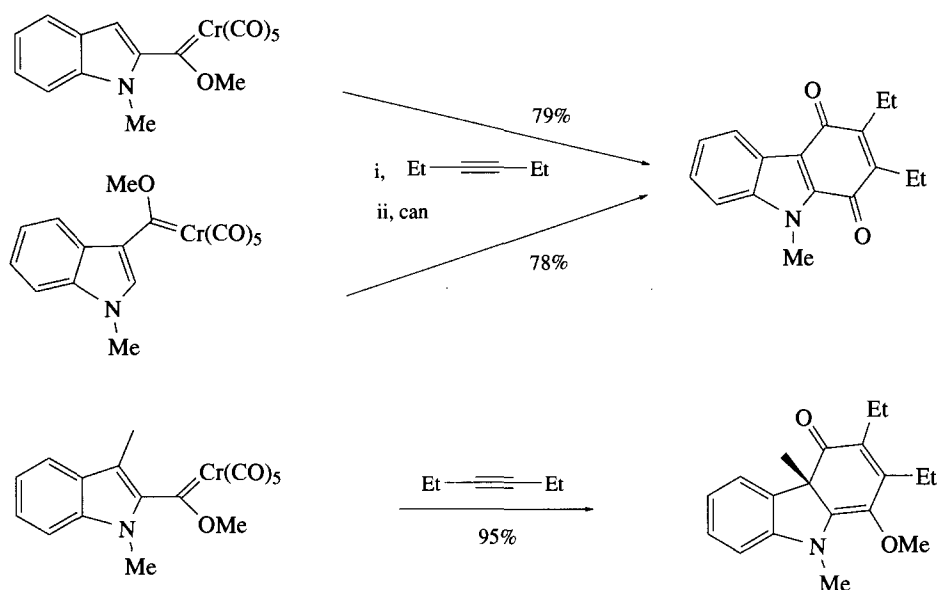


In connection with the synthesis of pyrrolo[1,2-*a*]indoles, intramolecular carbene insertion reactions have been developed. Although strictly involving interactions between two substituents, they will be considered here because of their chemical similarity. Thermal decomposition of the sodium salt of the tosylhydrazone (**219**) gives intramolecular cycloaddition followed by loss of nitrogen to yield the fused indole (**220**) (Equation (66)) <89JCS(P1)2449>. This technique can be extended to include oxime ethers (**221**) in place of alkenes and thus provide formation of an aziridine (**222**); insertion into alkynes could not be achieved (Equation (67)) <91JCS(P1)1721>.



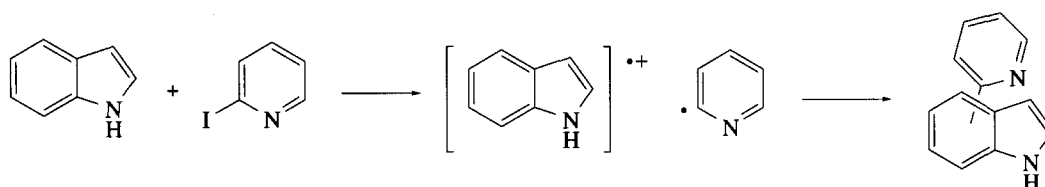


Chromium–carbene complexes can be prepared from 2- and 3-substituted indolyl lithium compounds and chromium hexacarbonyl, followed by methylation. These reactive species combine with alkynes to give cyclization products, which are oxygenated carbazoles. Oxidation to the quinone can be effected with ceric ammonium nitrate (can) where possible, otherwise the product is a cyclohexadiene (Scheme 66) <89JOC3249>.



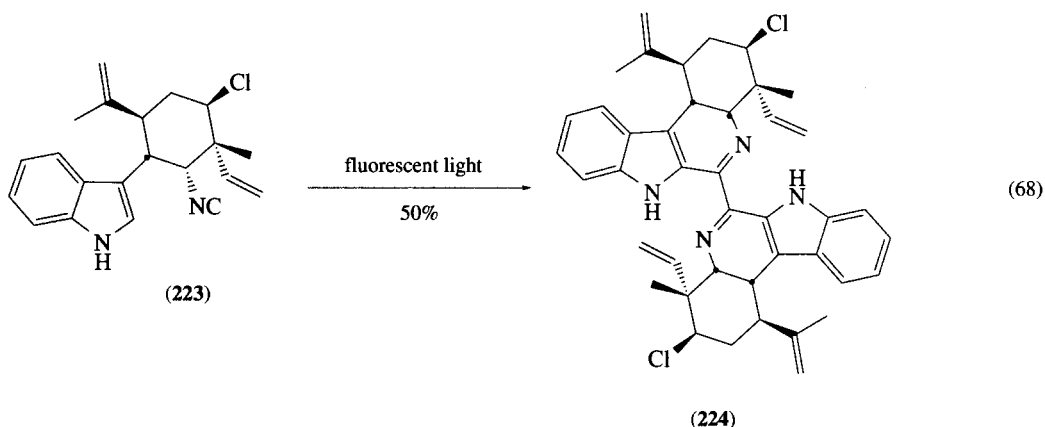
Scheme 66

Pyrroles and indoles are involved in numerous free-radical reactions and this area is receiving increased attention because of the synthetic potential available. Radical reactions are sometimes facilitated by the fact that pyrroles and indoles can form reasonably stable radical cations in some cases. For instance, photoarylation of indole by 2-iodopyridine is controlled by a photochemical electron-transfer reaction leading to the combination of the indole radical cation and the 2-pyridyl radical. Direction of attack is controlled by the relative spin density of the possible radical cations. In polar solvents, substitution is favored at positions 3, 6, and 4, whilst in nonpolar solvents, there is a preference for substitution at positions 2 and 7 (Scheme 67) <88CPB940>.



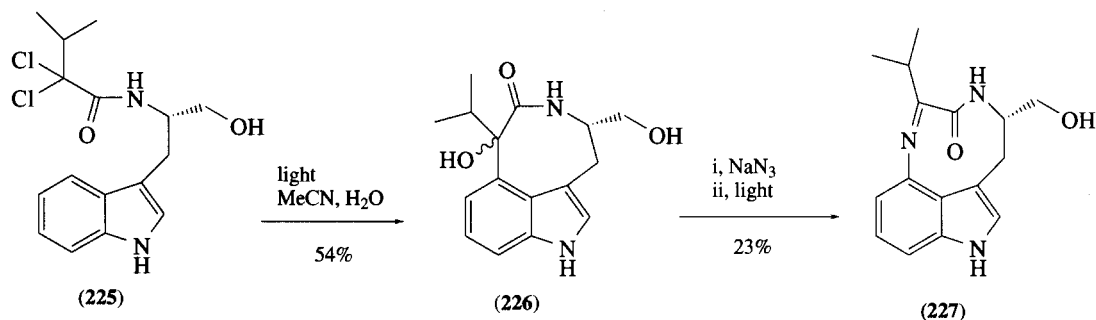
Scheme 67

There are examples when photochemically generated radicals undergo an oxidative dimerization similar to some examples shown in Section 2.02.1.5. In the case of hapalindole E (223), irradiation leads to the dimer (224), presumably by a mechanism which involves photoinduced interaction between the indole ring and the isonitrile group to give an indolyl radical (Equation (68)) <89JOC719>.

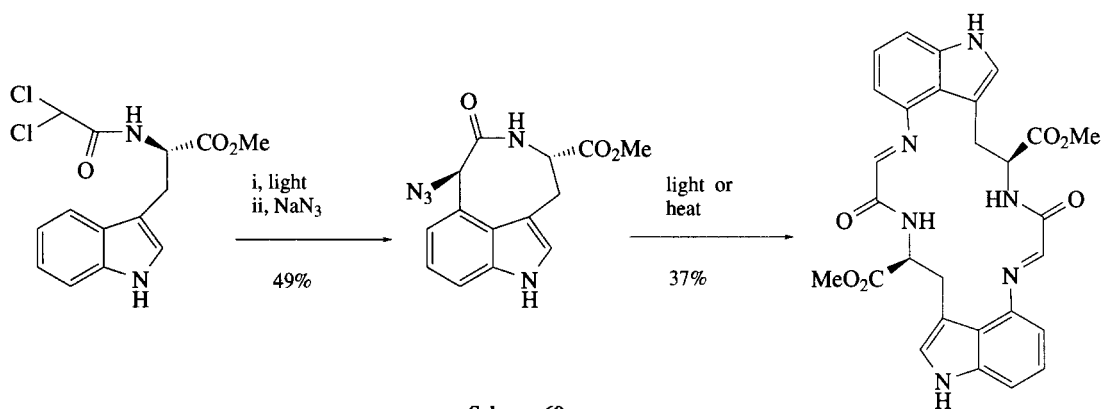


The formation of 3,4-linked indoles can be achieved by photochemical irradiation of *N*-dichloroacetyltryptophan derivatives. The reaction is general and gives moderate to good yields. The cyclization proceeds via a coupling of an alkyl radical with an indole radical cation, and has been applied to the synthesis of indolactam V. For example, the dichlorotryptophan (225) on irradiation in aqueous acetonitrile gives the cyclized intermediate (226), which is trapped by a nucleophilic azide ion, the resulting azide then undergoes a photochemical loss of nitrogen and rearrangement to the lactam product (227) (Scheme 68) <88CC587, 88CC589, 92JCS(P1)797, 92JCS(P1)813, 92JCS(P1)823>.

In a more recent study, in the early 1990s, an unstable 9-membered imine is generated and is immediately transformed into an 18-membered lactam dimer, with a left-handed double-helical structure controlled by hydrogen bonding (Scheme 69) <93JA813>.



Scheme 68

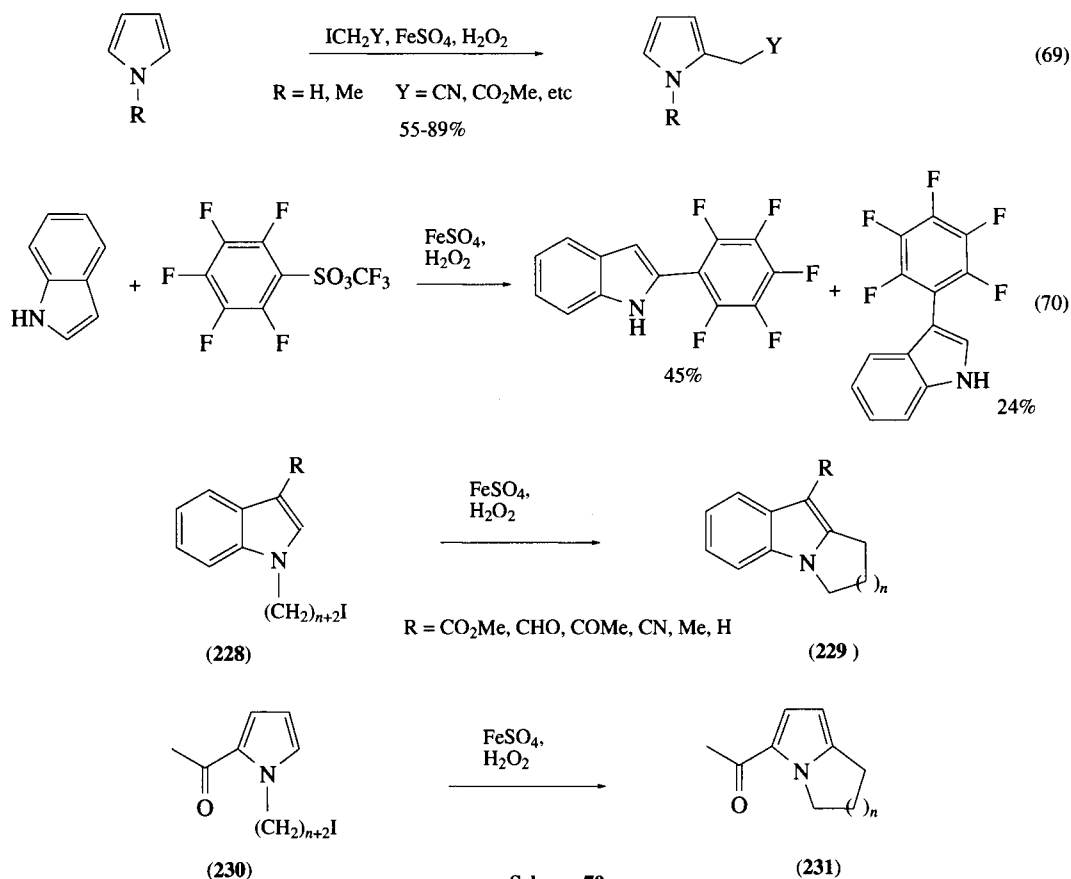


Scheme 69

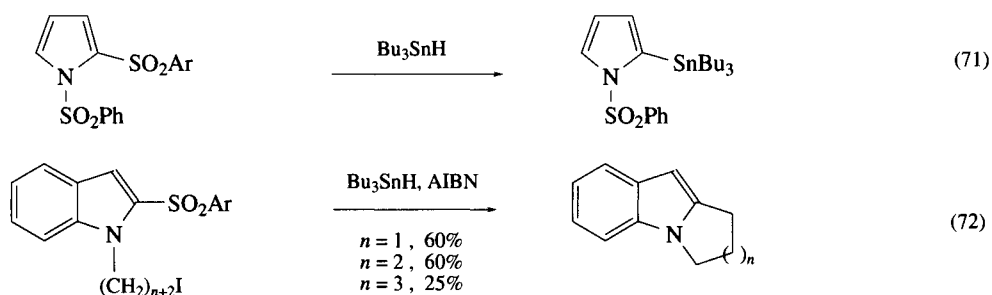
The pyrrole and indole rings are readily attacked by a range of electrophilic radicals, generated in various ways. The reaction of alkyl iodides with a combination of iron(II) sulfate and hydrogen peroxide generates alkyl radicals, which give 2-substitution products with pyrrole and 1-methyl-

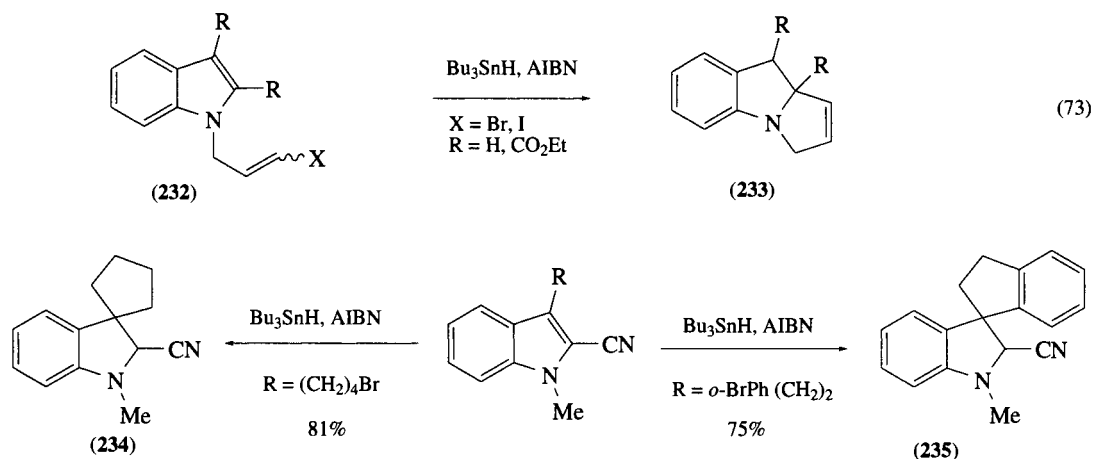
pyrrole (Equation (69)) <92JOC6817>. Indole is also substituted at C-2 with cyanomethyl and carbomethoxymethyl radicals. Perfluoroalkyl radicals have been generated in this way and give similar substitution results. 2-Acylated pyrroles are substituted at C-5, while 2,5-dimethylpyrrole gives mainly the 2-perfluoroalkyl-2*H*-pyrrole <93TL3799>. Pyrrole and indole undergo perfluoro-arylation on photoirradiation with perfluorophenyl triflate in reactions that involve electron-transfer processes; both 2- and 3-substituted products are obtained (Equation (70)) <93JOC2599>.

Treatment of *N*-(ω -iodoalkyl)pyrroles (**228**) and indoles (**230**) with excess hydrogen peroxide in a sonicated solution of iron(II) sulfate in DMSO gave the related cyclized products (**229**) and (**231**) respectively (Scheme 70) <94JOC2456>.



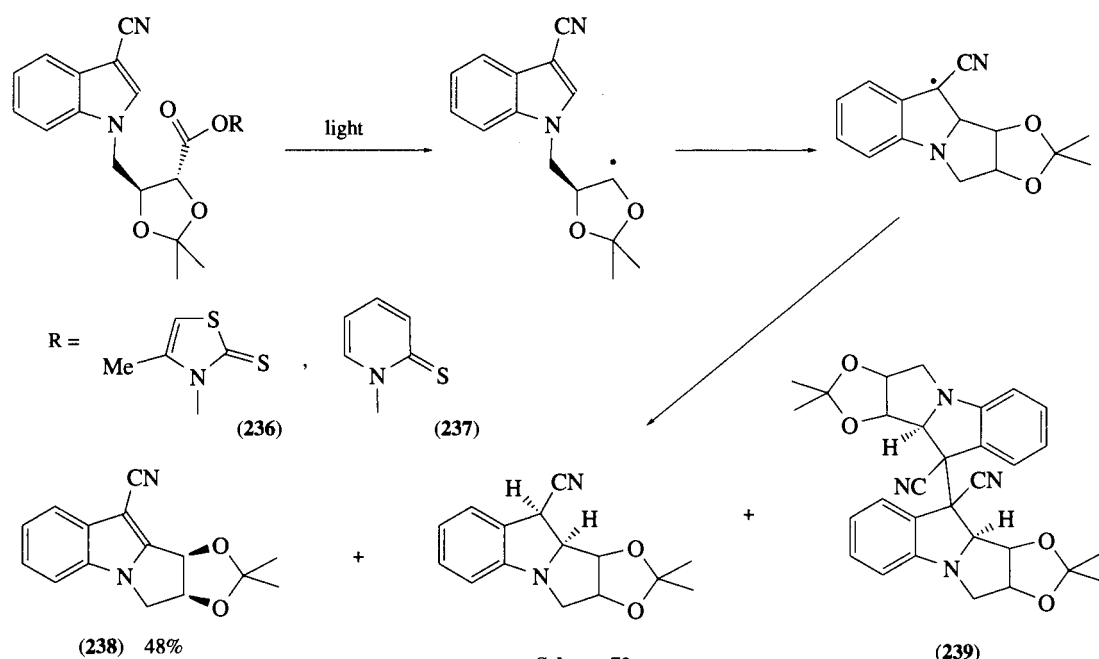
N-Protected 2-arenesulfonyl-pyrroles and indoles react with tri-*n*-butylstannane and AIBN as an initiator to give stannanes, presumably by an addition and elimination sequence (Equation (71)) <92SL805>. When the *N*-protecting group is ω -iodoalkyl, cyclization occurs (Equation (72)) <93SL231>. Similar conditions can be used for the cyclisation of *N*-protecting groups with terminal vinyl halides. Thus 1-haloallylindoles (**232**) can be converted into the pyrrolo[1,2-*a*]indoles (**233**) (Equation (73)) <91JOC3479>. Intramolecular addition of alkyl and aryl radicals appended to the C-3 of 2-cyanoindoles generates a range of spiro addition products such as (**234**) and (**235**) (Scheme 71) <93JOC3100>.



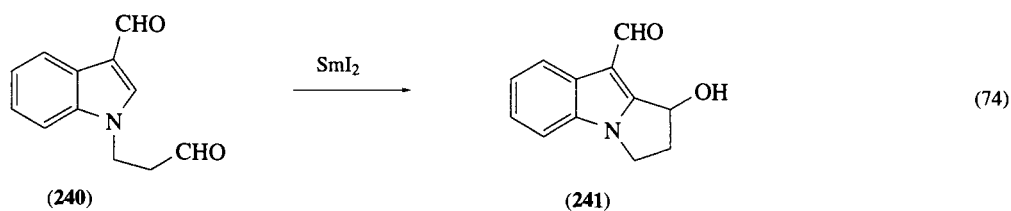


Scheme 71

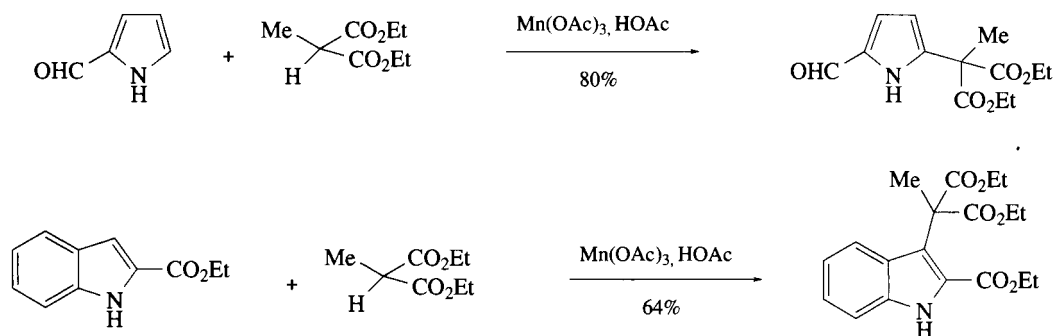
Free radicals can also be generated by the photolysis of thiohydroxamate systems derived from thiazolones and thiopyridones. Photolysis of the thiazolone derivative (236) yields the pyrrolo[1,2-*a*]indole (238), together with a trace of the related indoline and some dimeric product (239). Reaction of the pyridone thiohydroxamate (237) gives a dimer as the major product (Scheme 72) <93JOC2768>.



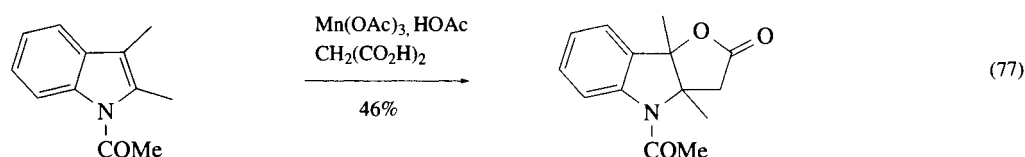
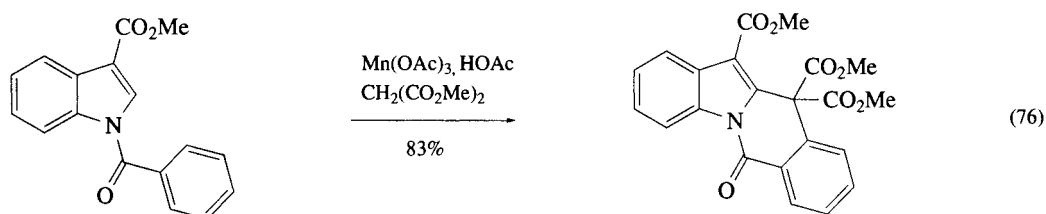
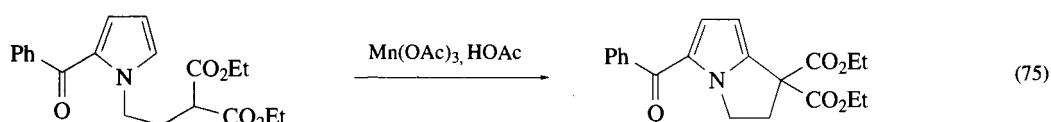
Samarium iodide interacts with indole-3-carbaldehydes to give a radical anion, which can be trapped by intramolecular attack on a carbonyl group. Thus, the 1-oxopropylindole (240) can be converted into the pyrrolo[1,2-*a*]indole (241) (Equation (74)) <93CC1277>.



Pyrroles and indoles undergo reactions with malonyl radicals generated by reaction of manganese(III) acetate with malonic acids or esters. A wide range of 2- or 3-acylpyrroles react at the free α -position with the diethyl methylmalonate radical; indole-2-carboxylic ester reacts at C-3 (Scheme 73) <93JOC7610>. An intramolecular analog of this reaction has also been developed in the case of pyrrole derivatives <92CJC1838> (Equation (75)). The manganese(III)-initiated oxidative free-radical reaction between *N*-aroylindoles and dimethyl malonate results in the linking of the two ring systems to give indolo[2,3-*a*]isoquinolines (Equation (76)) <94TL1283>. Oxidative addition of the malonic acid radical to 1-acetylindoles results in attack at C-2, with subsequent lactonization at C-3 and decarboxylation (Equation (77)) <93JHC1133>.



Scheme 73



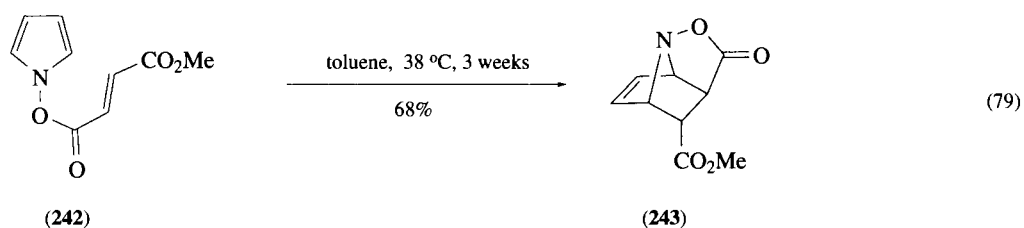
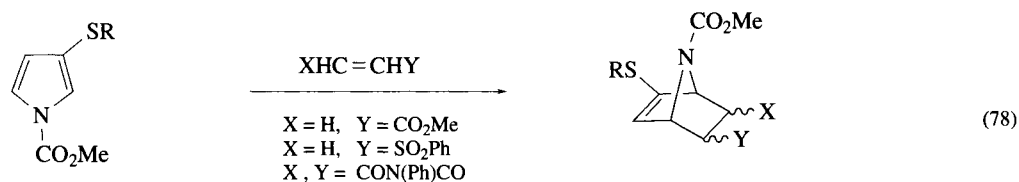
2.02.1.8 Cycloaddition Reactions

Pyrroles and indoles can give a wide variety of cycloaddition reactions and this area has seen vigorous activity since 1990 because of the potential of this methodology for the synthesis of complex indole alkaloids.

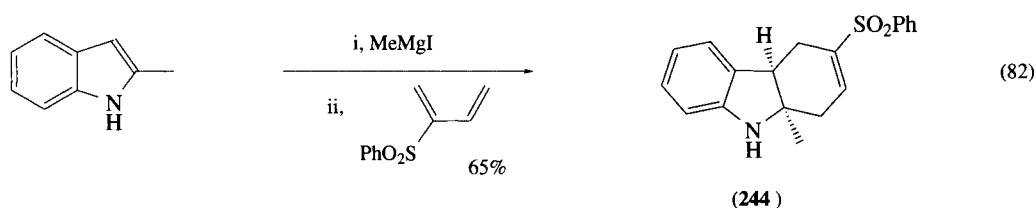
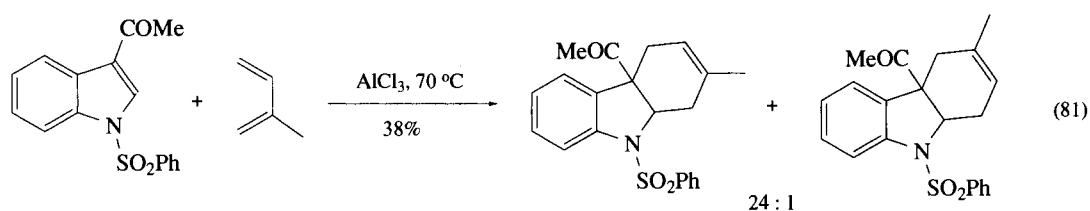
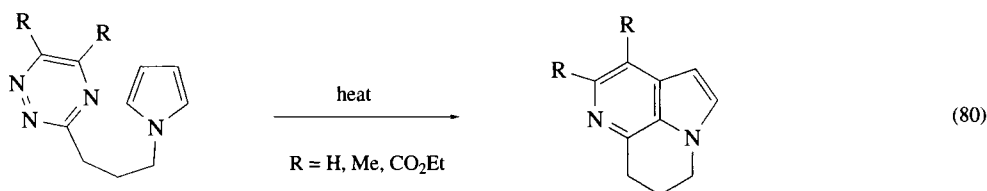
2.02.1.8.1 [4 + 2] Cycloadditions of pyrroles and indoles

N-Carbomethoxypyrroles bearing a 3-methylthio or 3-phenylthio group undergo high-pressure Diels–Alder additions to a range of dienes such as *N*-phenylmaleimide, methyl acrylate and phenylvinylsulfone (Equation (78)) <89H(29)79>. The thio substituents appear to activate the pyrrole ring to cycloaddition and some selectivity for the *endo* adducts is observed. An interesting but very slow

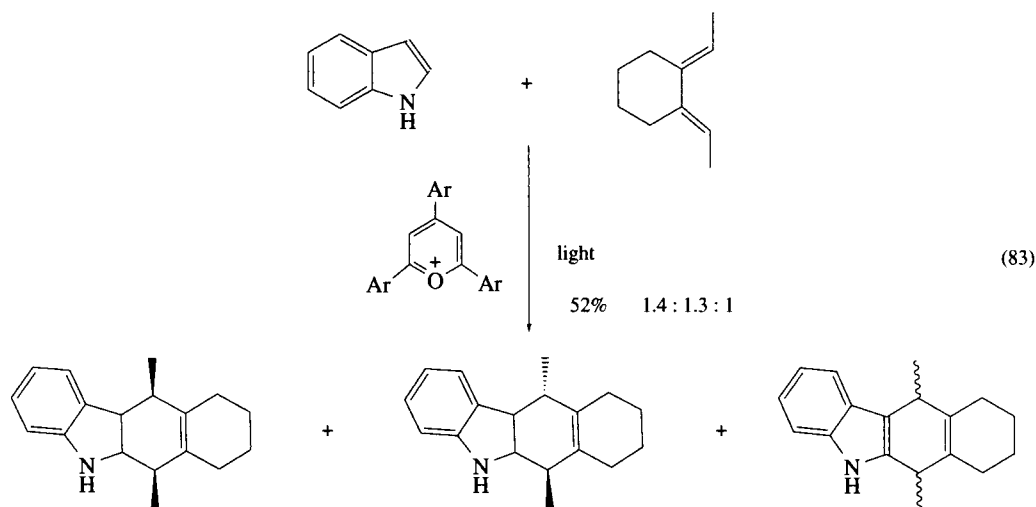
intramolecular addition of the 1-pyrrolyl fumarate (**242**) to the structurally useful product (**243**) has been achieved in reasonable yield (Equation 79) <84CC630>.



The pyrrole ring can also participate as a dienophile in an inverse electron demand Diels–Alder reaction with a tethered 1,2,4-triazine to give 1,7-bridged 6-azaindoles <93JOC516> (Equation (80)). Similar reactions have been reported for indoles <92JOC5285>. 1-Benzenesulfonyl-3-acyl-pyrroles and indoles also act as dienophiles and good regioselectivity is observed when the reactions are catalyzed by aluminum chloride (Equation (81)) <88JA7188>. Indolyl Grignard reagents, such as that derived from 2-methylindole, react with 2-benzenesulfonyl-1,3-butadiene to give cycloadducts such as the tetrahydrocarbazole (**244**); this methodology has been applied to the synthesis of ellipticine (Equation (82)) <89TL2589, 90JOC4528, 94H(37)175>.

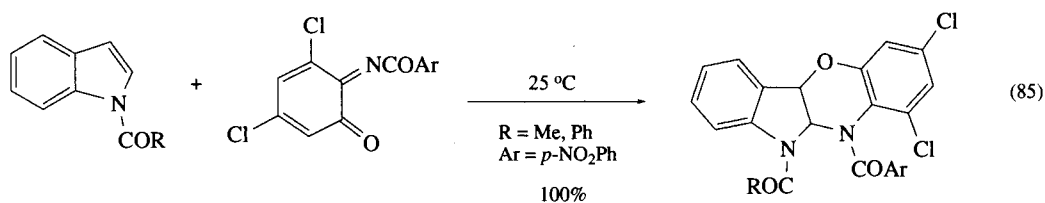
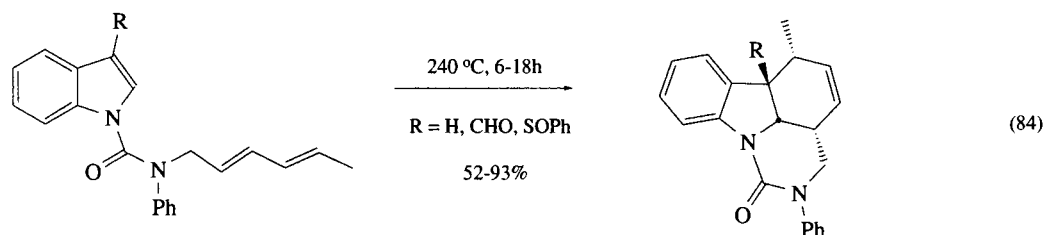


Indole has been shown to undergo a Diels–Alder reaction with both *endo*- and *exo*-cyclic 1,3-dienes, in which electron transfer catalysis is provided using a triarylpyrylium cation as a sensitizer (Equation (83)) <90SL275, 91JOC1405, 93TL6391>. The adducts are generally mixtures of isomers, with *endo* predominating in the case of cyclohexadienes. The indole radical cation is postulated as an intermediate.



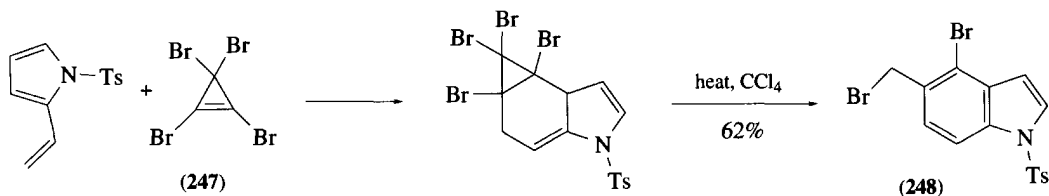
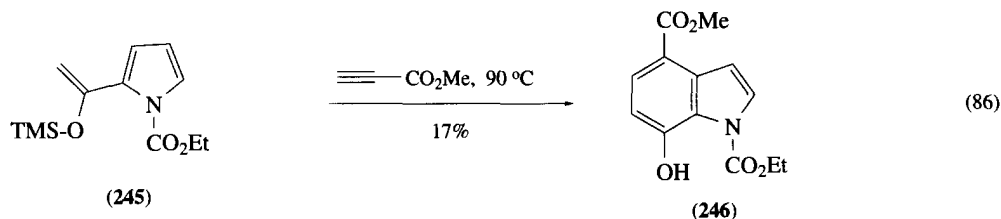
Intramolecular Diels–Alder reactions have been reported for a series of *N*-(hexa-2,4-dienyl) carbamoylindoles, which yield useful tetracyclic systems. Similar cyclizations occur for the related *N*-acylindoles (CH_2 replacing NPh), but not for the alkoxy carbonyl analogs (O replacing NPh) (Equation (84)) <88TL5605, 89JOC2425>.

1-Acylindoles act as dienophilic components of a $[4+2]$ cycloaddition when treated with an *o*-quinone monoimide (Equation (85)) <87TL6691>.

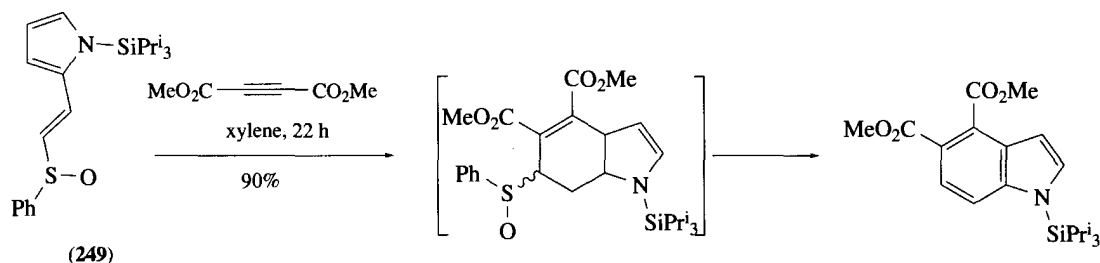


2.02.1.8.2 $[4+2]$ Cycloadditions of vinylpyrroles and vinylindoles

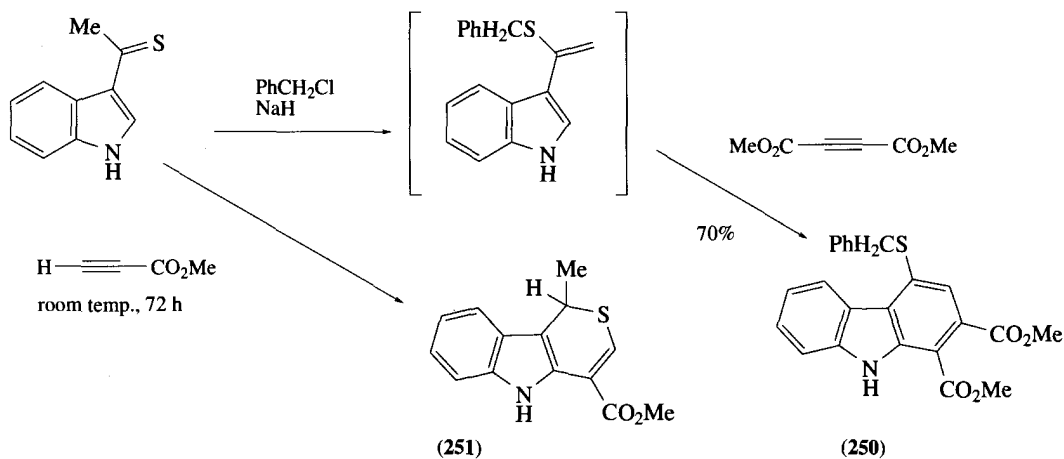
Vinylpyrroles undergo a variety of Diels–Alder cycloadditions which can lead ultimately to the synthesis of indoles, for example the 2-vinylpyrrole (**245**) gives only a rather low yield of the indole (**246**) in combination with methyl propiolate (Equation (86)) <90TL4613, 91H(32)1199>. Cycloaddition of the more exotic tetrabromocyclopropene (**247**) gives a better overall conversion to the indole (**248**) (Scheme 74) <90TL4581>. This synthetic methodology has been developed to provide a high-yielding indole synthesis from 2-phenylsulfinylvinylpyrroles (**249**) (Scheme 75) <87TL3453>. Furthermore, 3-thioacetylpyrroles can be converted to α -benzylthio-3-vinylpyrroles by *S*-benzylation. The resulting 3-vinylpyrroles undergo Diels–Alder reactions with electron-deficient dienophiles and oxidation affords the 4-benzylthioindoles <89CPB1999, 91CPB489>. This reaction sequence also occurs for 3-thioacetylindole to give the carbazole (**250**) with DMAD; the thioacetylindole can even participate as a diene analog in reaction with methyl propiolate to give the thiapyrano[4,3-*b*]indole (**251**) (Scheme 76) <94H(37)725>.



Scheme 74

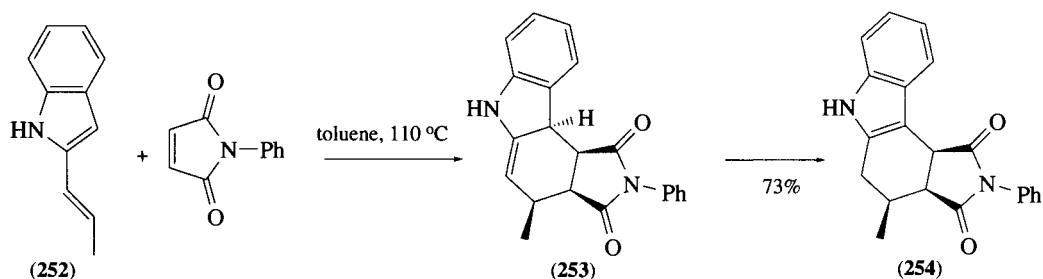


Scheme 75

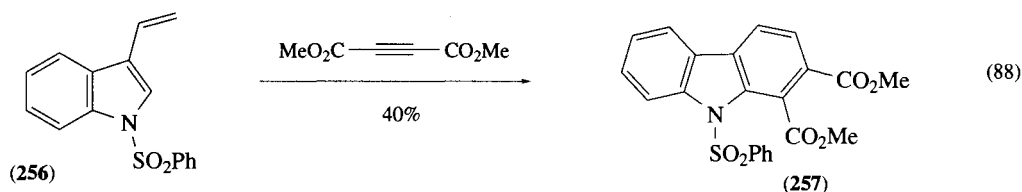
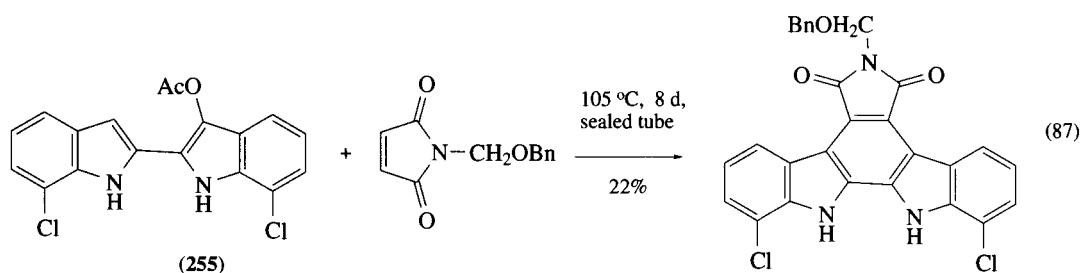


Scheme 76

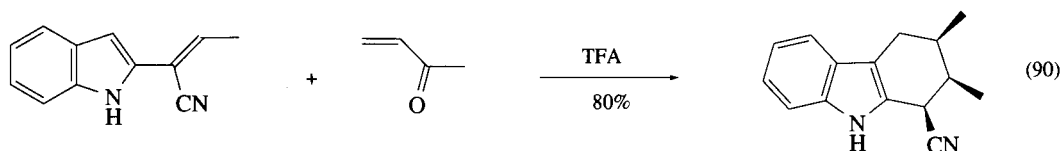
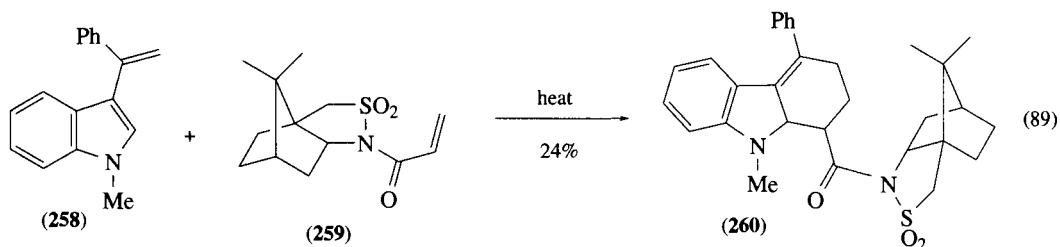
Vinyl indoles have been studied extensively with respect to their Diels–Alder reactions, and provide a broadly successful strategy for the construction of polycyclic systems. In general 2-vinylindole substrates are not *N*-protected, but the 3-isomers are usually *N*-methyl or *N*-benzenesulfonyl compounds. Many of the usual dienophiles are applicable to these reactions and include DMAD, *N*-phenylmaleimide, methyl acrylate, and benzoquinone. For example, the 2-vinylindole (**252**) reacts with *N*-phenylmaleimide in refluxing toluene to give the tetrahydrocarbazole (**254**), following re-aromatization of the intermediate adduct (**253**) (Scheme 77) <88HCA467, 88HCA1060, 90JOC5368, 93SL597, 93H(35)1259, 94CC1237>. A special case of this methodology is its application to the synthesis of the antitumor antibiotic rebeccamycin. A 2,2'-biindolyl (**255**) replaces the vinylindole in its reaction with a maleimide (Equation (87)) <85TL4015>. Also the 3-vinylindole (**256**) reacts with DMAD to give the carbazole (**257**), arising from ready oxidation of the dihydro compound formed initially (Equation (88)) <86S748>.

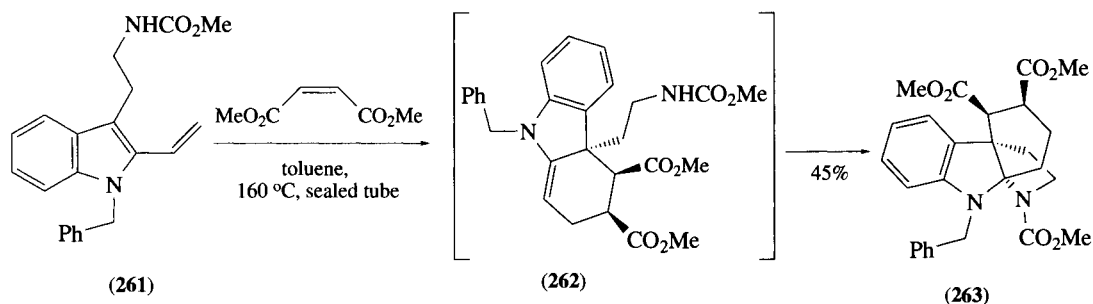


Scheme 77



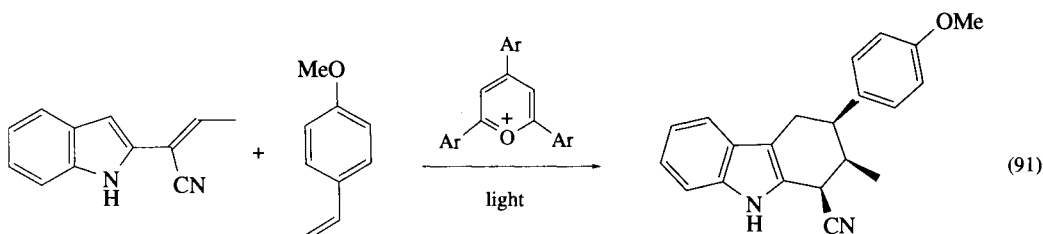
In the case of unsymmetrical dienophiles, the normal patterns of regioselectivity and stereoselectivity are observed (89H(29)11, 93T2863). This is exemplified by the reaction of the 3-vinylindole (258) with the (1*R*, 2*S*, 4*S*)-isomer of the *N*-propenoylbornanesultam (259) to give the adduct (260), whose absolute configuration was determined by x-ray crystallography. Good π -facial selectivity was found, but the chemical yield was poor (Equation (89)). 2-(1-Cyano-1-alkenyl)indoles undergo cycloaddition with α,β -unsaturated carbonyl compounds under conditions of acidic catalysis (Equation (90)) (92TL6621). The addition of a dienophile to a 2-vinyltryptamine (261) gives an adduct (262), which undergoes cyclization to the tetracyclic product (263) (Scheme 78) (92SL601). Further studies have been made, in the late 1980s and early 1990s, on 3-indolyl-enol ethers (89M157, 92JOC910), 2- or 3-indolyl-enamines (92T3515), and 3-(2-nitrovinyl)indoles (93JHC183). In the two former cases, the adducts can be aromatized with or without loss of the alkoxy or amino substituents. *In situ* cycloadditions have also been observed when indoles combine with aldehydes or ketones in the presence of maleimides and under conditions where the vinylindole can be produced by dehydration (93JHC81).



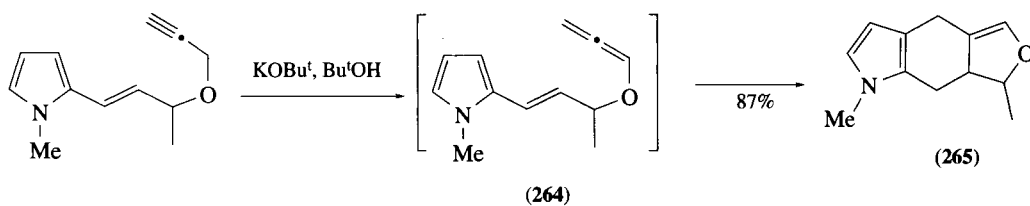


Scheme 78

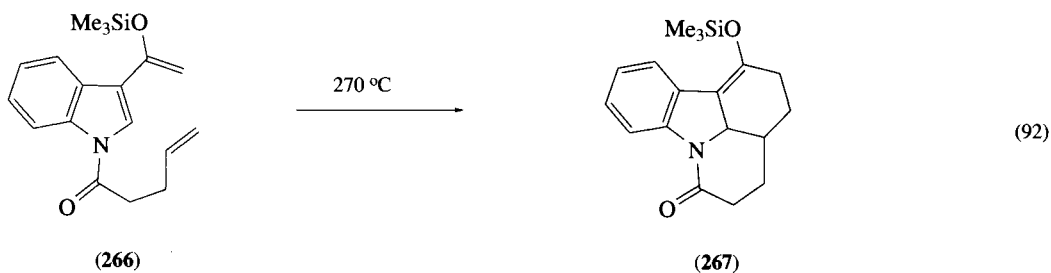
Electron-transfer-mediated cycloadditions of 2-(1-cyanovinyl)indoles can be carried out with cyclohexadienes and electron-rich styrenes; the reactions are catalysed by photoexcited pyrylium salts or triarylammonium radical cations (Equation (91)) <93AG(E)901>.

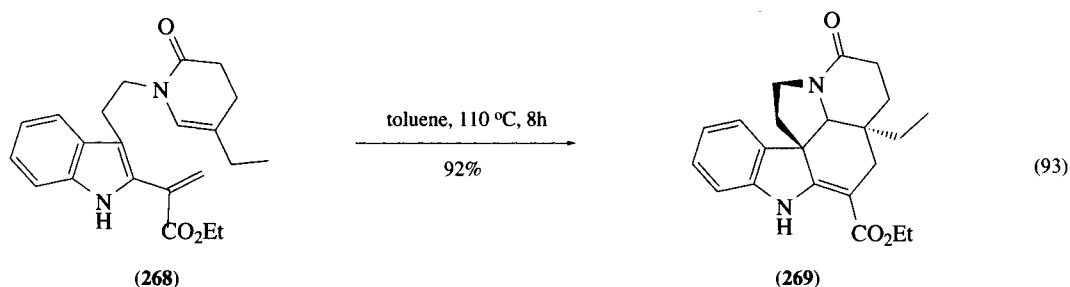


Intramolecular Diels–Alder additions have also been developed for vinylpyrroles and vinylindoles. The allenylvinylindole (**264**) can be converted to a tricyclic system (**265**) in high yield (Scheme 79) <84JA6735>. 3-Vinylindoles undergo intramolecular cycloaddition to a terminal alkene tethered to the nitrogen atom and give tetracyclic structures, for example, the silyl enol ether (**266**) generates compound (**267**) (Equation (92)) <90JOC1624>. 2-Vinylindoles tethered at C-3 to an enamine moiety have been used to great effect in the synthesis of indole alkaloids <84ACR35, 93JOC1434>. For example, the 3-oxovincadifformine ethyl ester (**269**) can be formed in excellent yield by the thermal cycloaddition of the 3-oxoscodine (**268**) (Equation (93)) <94T6941>.



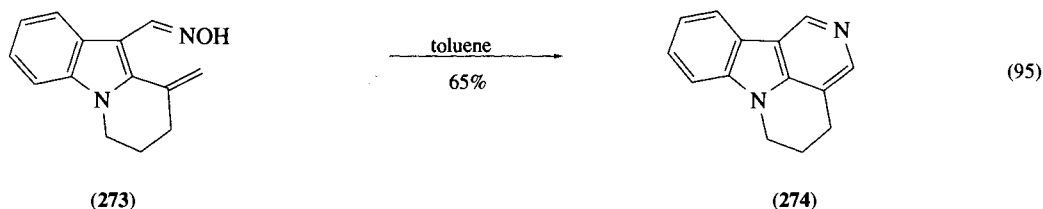
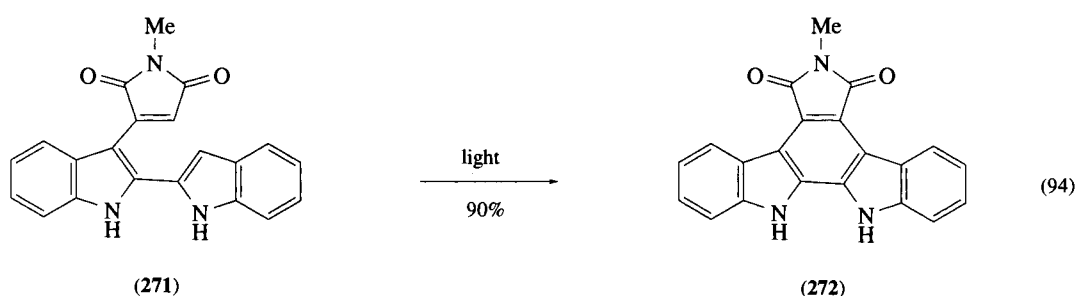
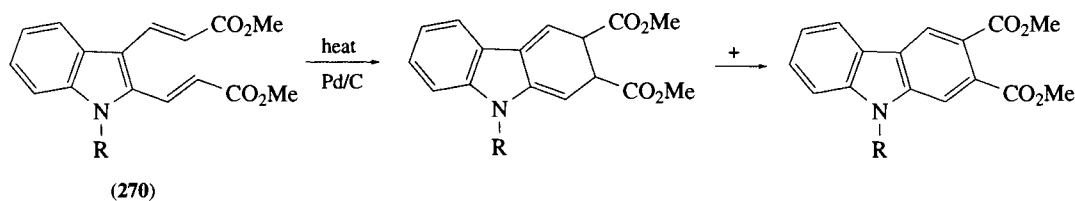
Scheme 79





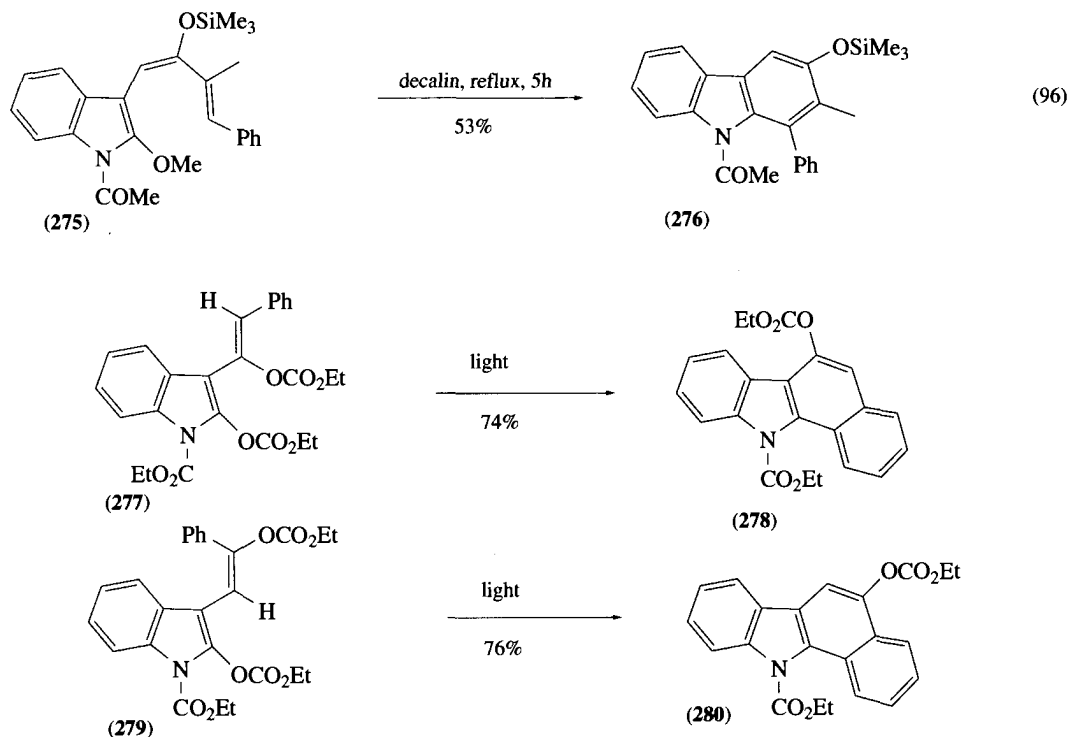
2.02.1.8.3 [2 + 2 + 2] Cyclizations of pyrroles and indoles

This category includes examples of electrocyclizations involving 2,3-divinylindoles and also systems in which the indole double bond is at the end of a suitable conjugated chromophore. Mixtures of dihydrocarbazoles and carbazoles can be obtained from the thermal treatment in the presence of Pd/C, of 2,3-divinylindoles (**270**), usually generated from either Wittig or Heck reactions (Scheme 80) <90H(31)587, 90HCA827>. In another special case adapted to the synthesis of indolocarbazoles, the 3-substituted-2,2'-biindolyl (**271**) can be photocyclized to the product (**272**) in high yield (Equation (94)) <93TL5329>. An aza-equivalent of this type of electrocyclic process is illustrated by the conversion of the indole alkenyl oxime (**273**) into the dihydroindolonaphthyridine (**274**) (Equation (95)) <92JOC5917, 94H(37)697>.



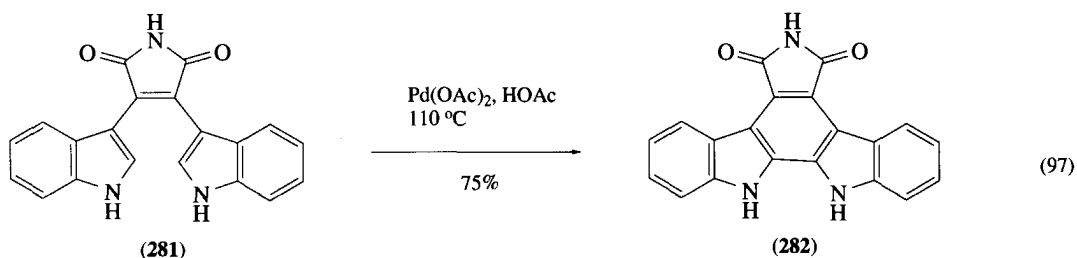
Indoles substituted at C-3 with butadienyl or styryl groups undergo electrocyclic reactions to give carbazole derivatives. The silyl enol ether (**275**) can be converted thermally directly into the carbazole (**276**), as the result of ready elimination of methanol; this sequence is crucial for a hyellazole synthesis (Equation (96)) <89CC43>. Several related 3-styrylindole sequences have been developed and differ with respect to the placement of ethyl carbonate groups on the alkene. Both the substrates (**277**) and (**279**) undergo photocyclization to the respective benzocarbazoles (**278**) and (**280**) as a result of

electrocyclization followed by aromatization with elimination of ethanol and carbon dioxide (Scheme 81) <92S891, 93T4741>. The phenyl group in the above sequences can be replaced by a range of heteroaromatic rings including indole.



Scheme 81

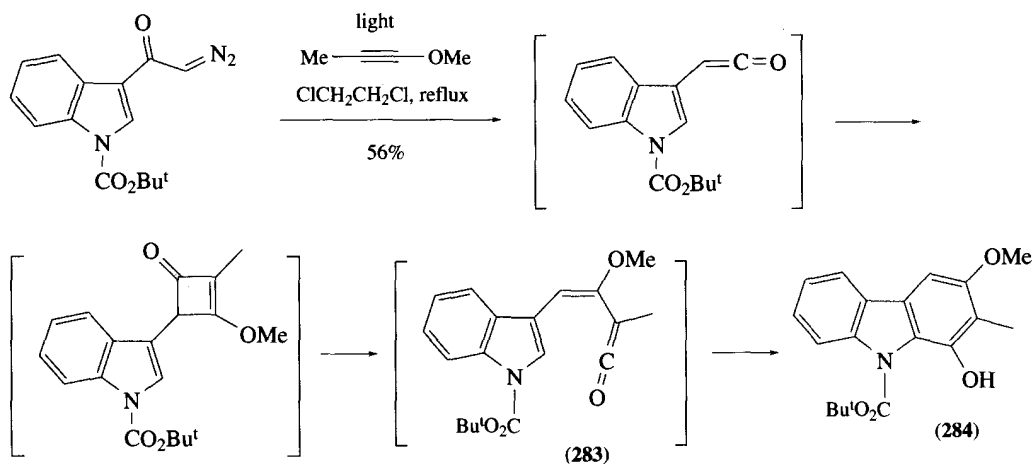
A related substrate is the diindolylmaleimide (**281**), which undergoes an oxidative electrocyclization on treatment with palladium acetate in acetic acid, to give the indolocarbazole (**282**) (Equation (97)) <85TL4015, 93TL8361>.



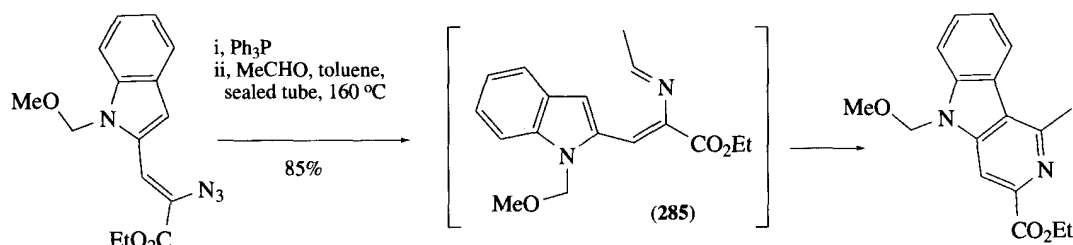
Another hyellazole synthesis has been based on an ingenious rearrangement leading to a 3-indolyl-vinyl ketene (**283**), which cyclizes to a hydroxycarbazole (**284**) (Scheme 82) <90JA3093>. A successful pyridocarbazole synthesis utilizes the cyclization of a 2-indolyl-vinyl ketene (**285**), generated in an aza-Wittig reaction (Scheme 83) <93TL4701>.

2.02.1.8.4 Cobalt-mediated [2 + 2 + 2] reactions of indoles with alkynes

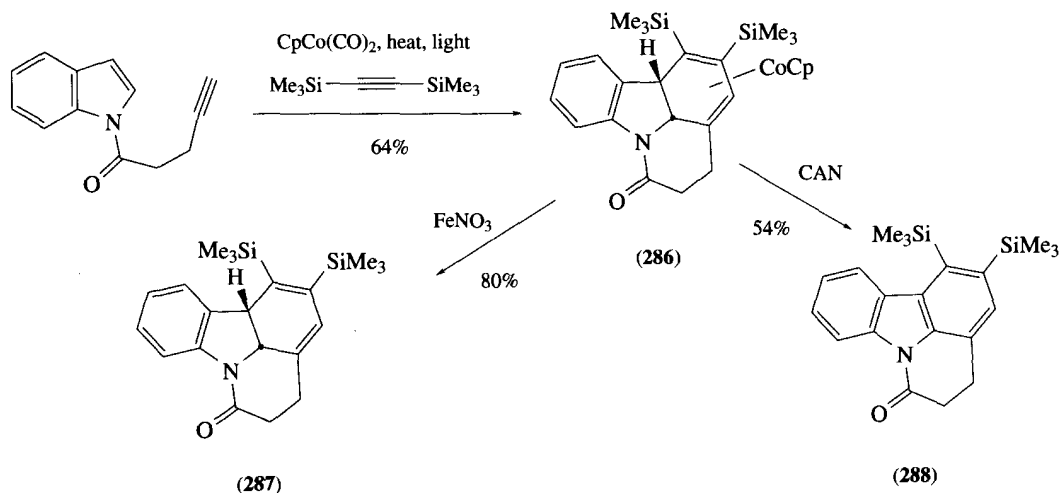
The indole 2–3 double bond can participate in a cobalt-mediated combination with two alkyne units. In particular, when one of the alkynes is tethered to the indole nitrogen atom by an acyl group, cyclization occurs to give an unusual dihydrocarbazole such as compound (**286**), which can be demetallated either to the dihydrocarbazole (**287**) or to the carbazole (**288**) (Scheme 84) <86JA2091>.



Scheme 82



Scheme 83



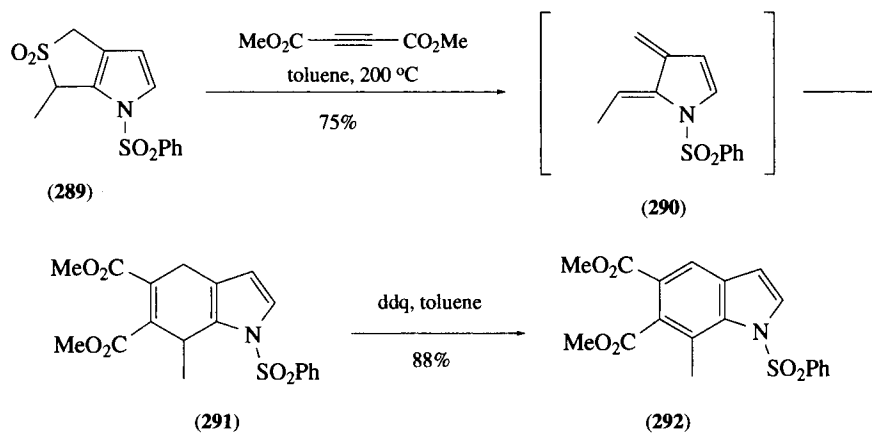
Scheme 84

2.02.1.8.5 [4 + 2] Cycloadditions of pyrrole- and indole-quinodimethanes

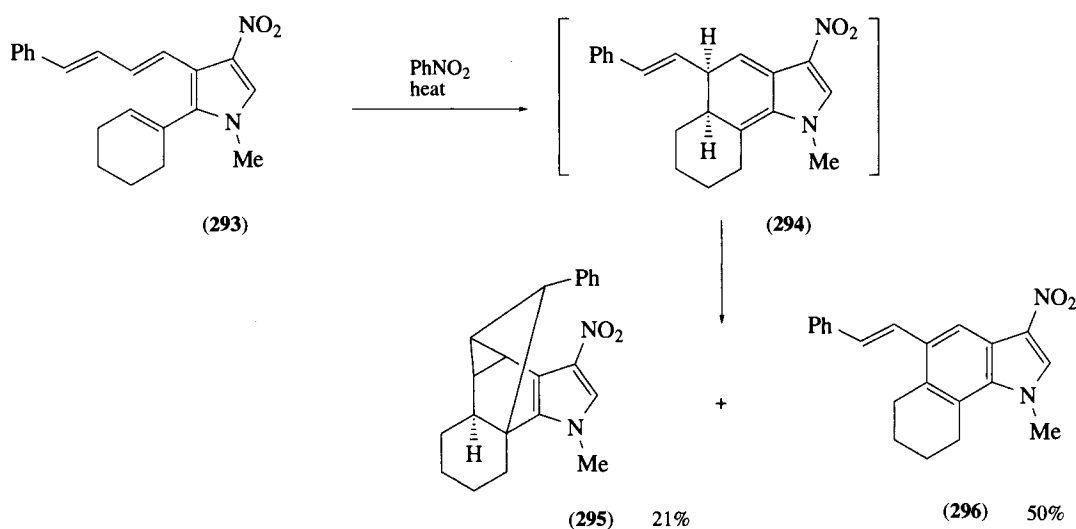
Many examples of Diels–Alder reactions have been carried out on quinodimethanes derived in a variety of ways from pyrrole and indole precursors. This area continues to be widely investigated because of its generality and its synthetic utility. In the case of pyrrole derivatives, this methodology leads to the synthesis of indoles, and particularly those substituted in the benzene ring.

The thieno[3,4-*b*]pyrrole-1,1-dioxide (289) reacts with DMAD at 200 °C to give the cycloadduct (291), presumably by trapping a pyrrole-2,3-quinodimethane (290). The cycloadduct can then be dehydrogenated to the indole (292) (Scheme 85) <92CC549>. Formation of a quinodimethane structure (294) is also postulated to result from the Cope rearrangement of the phenylbutadienylpyrrole

(293), and under the conditions of refluxing nitrobenzene gives the products (295) and (296) of cycloaddition and dehydrogenation respectively (Scheme 86) <92CC1401>.

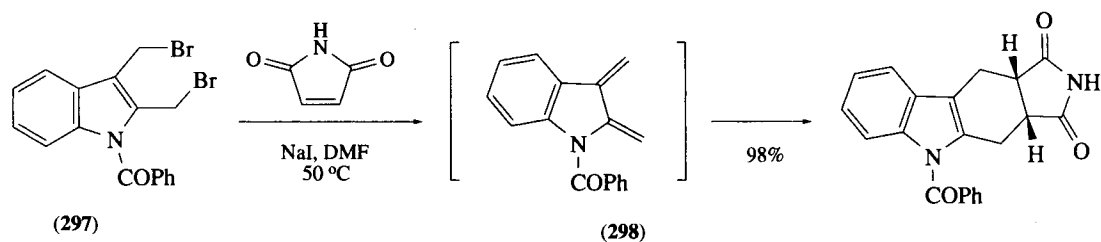


Scheme 85



Scheme 86

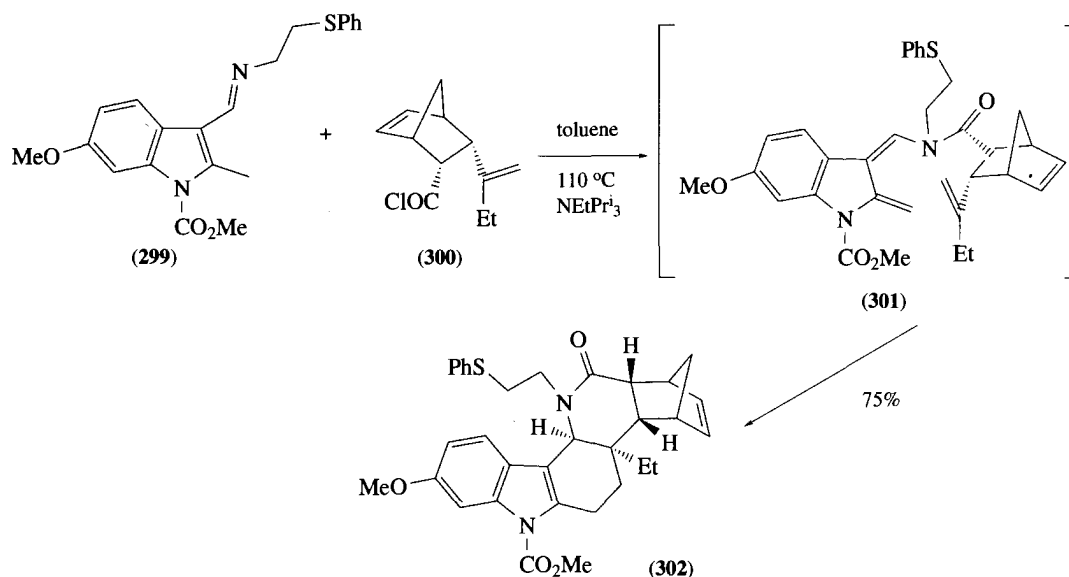
The 2,3-quinodimethane (298) of 1-benzoylindole has been generated from the 2,3-di(bromomethyl) indole (297) by treatment with sodium iodide and can be trapped by a range of electrophilic dienophiles, such as maleimides, DMAD, benzoquinone, and α,β -unsaturated carbonyl compounds and sulfones (Scheme 87) <89TL7289, 91T1925>.



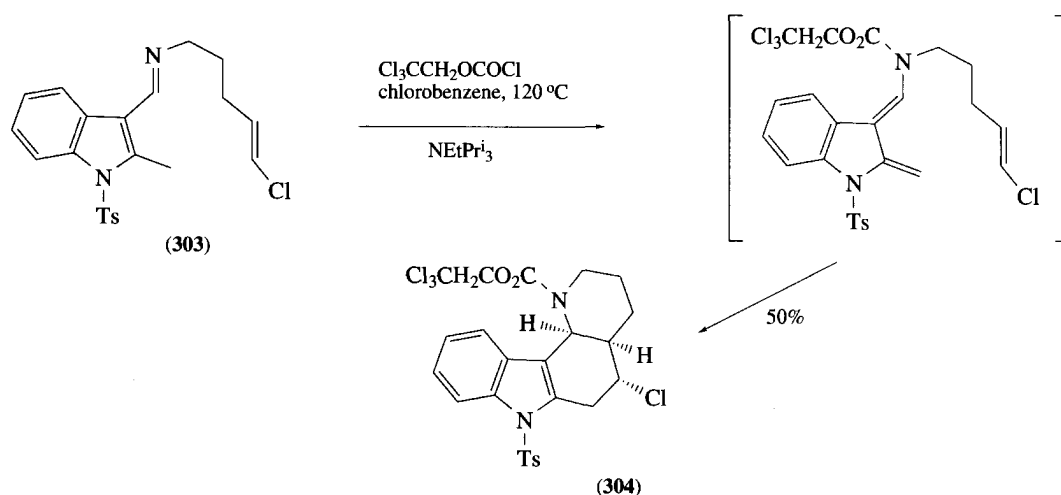
Scheme 87

The generation of indole quinodimethanes from 3-imino-2-methylindoles is one of the most versatile and effective routes, especially as it enables the attachment of substituents which feature

in complex alkaloid structures. This synthetic strategy has been reviewed and new applications have appeared <84ACR35, 84JA2105, 86JA212, 86JA217, 87TL3423, 88JA2242>. The methodology employs an intermolecular reaction leading to an intramolecular cycloaddition in the synthesis of the tabersonine framework, where the indole (299) reacts with the bicyclo[2.2.1]heptene acid chloride (300) to give a quinodimethane (301) which is trapped by the pendant alkene to give the adduct (302) (Scheme 88) <88JA2242>. An intramolecular variant is used in the cyclization of the indole (303) to the tetracyclic system (304) (Scheme 89) <84JA2105>. In the early 1990s, this strategy was applied to the formation of pyrrole quinodimethanes, and thus to the synthesis of indoles (Scheme 90) <92CC1401>.

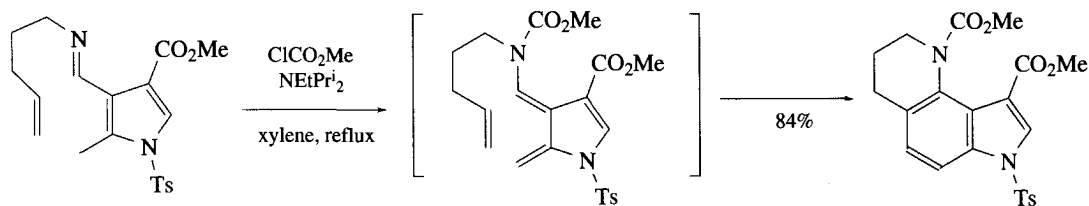


Scheme 88



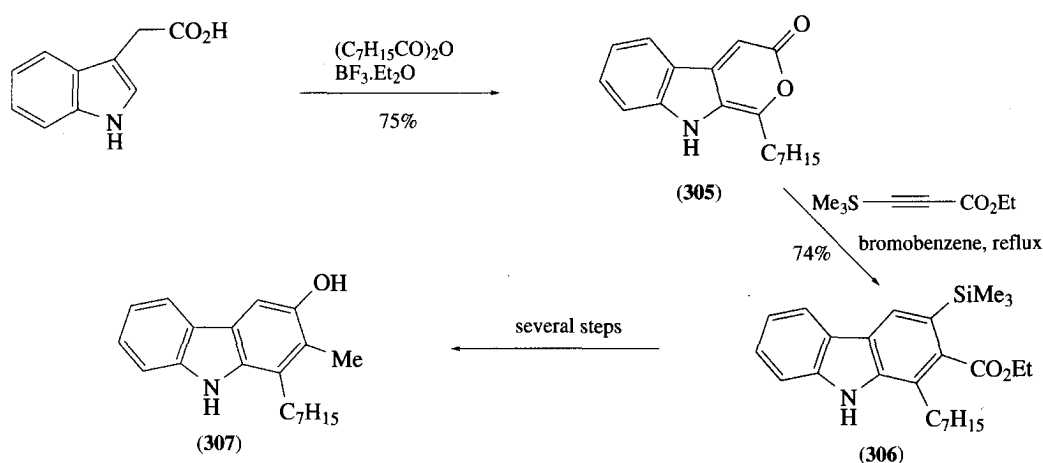
Scheme 89

Several types of stable indole quinodimethane systems have been developed as the basis for an effective carbazole synthesis, and there is vigorous activity in this area. In particular, the pyrano[3,4-b]indol-3-ones have been widely used and the area has been reviewed <88JCS(P1)1407, 88JCS(P1)3249, 89CR1681, 89H(29)11, 89JCS(P1)376, 89JCS(P1)2463, 91JCS(P1)2941, 92H(34)781>. This chemistry can be illustrated by the synthesis of carazostatin, in which the crucial pyranoindolone structure (305) is obtained by acylation at C-2 of an indole-3-acetic acid, with concomitant cyclization. Cycloaddition



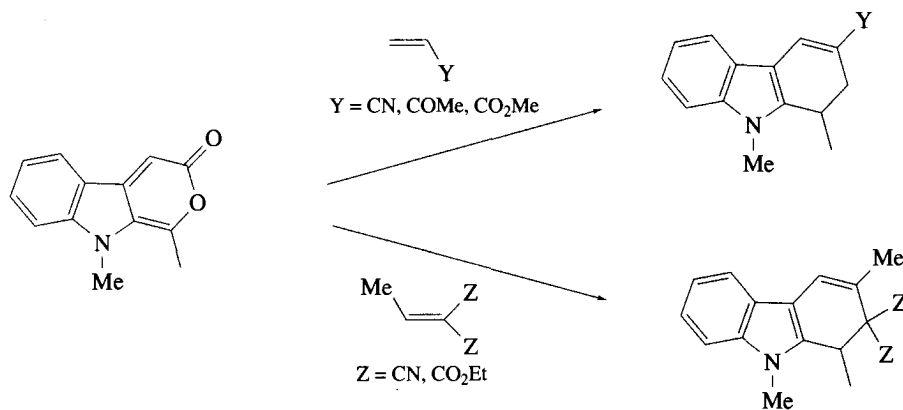
Scheme 90

then occurs with the appropriate alkyne to give the carbazole (306), which is a precursor of carazostatin (307) (Scheme 91) <91JCS(P1)2941>.

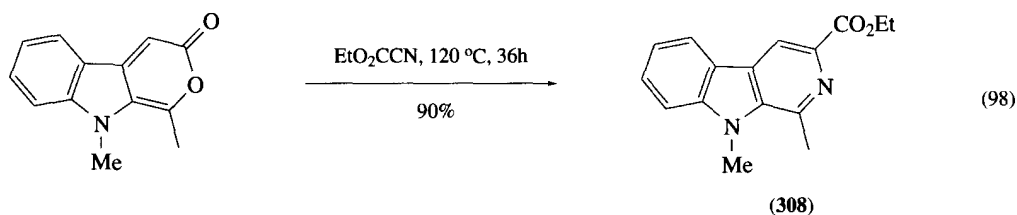


Scheme 91

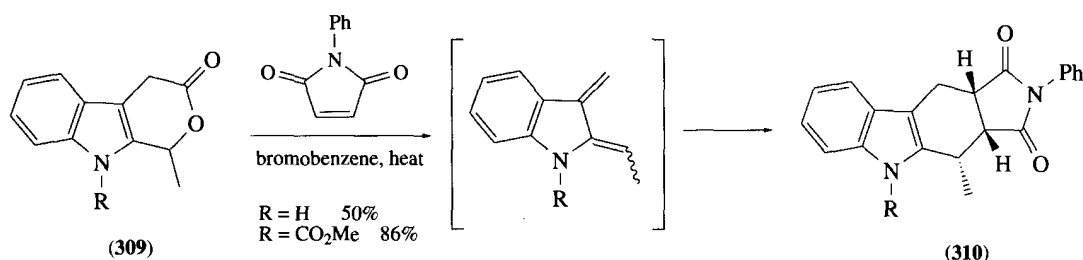
This general carbazole synthesis has also been studied with respect to unsymmetrical alkene addition. The regiochemical control is somewhat variable and is influenced not only by the alkene substituents but also by the indole *N*-substituent. For *N*-H or *N*-alkylindoles the addition of dienophiles such as enamides or α,β -unsaturated carbonyl compounds gave some regiochemical preference for the electron-attracting substituent appearing at C-3 in the dihydrocarbazole product. However, this preference seems to be switched to C-2 for *N*-acylindoles, and furthermore, doubly deactivated alkenes such as alkylidene malonates give products with the electron-attracting groups at C-2 (Scheme 92) <89T6761, 90T4023, 90T5523, 92JCS(P1)415>. The pyrano[3,4-*b*]indol-3-ones are also capable of undergoing cycloaddition to electron-deficient nitriles, such as ethyl cyanofornate and *p*-toluenesulfonyl cyanide, to yield 3-substituted pyrido[3,4-*b*]indoles (308) (Equation (98)) <92S473>. On the other hand, 3,4-pyridynes fail to cyclize under similar conditions <89JOC5189>.



Scheme 92

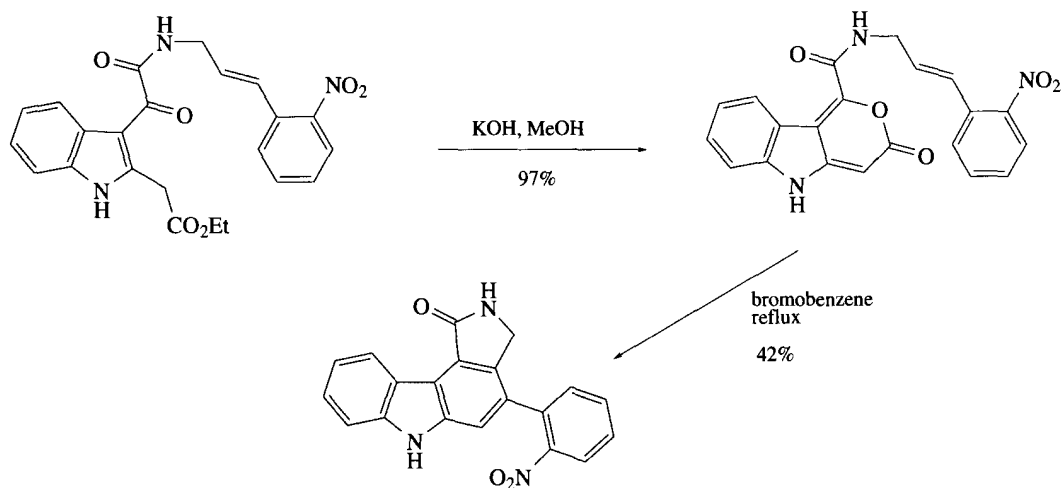


A dihydro version of the pyranoindol-3-one has also been used as a precursor of an indole quinodimethane, as the result of thermal loss of carbon dioxide <89TL7289, 93T439>. The dihydro compounds can be prepared effectively from the pyranoindolones by a sequence of lactone hydrolysis, carbonyl reduction and relactonization. They can then be decomposed thermally in the presence of suitable dienophiles such as maleimides, quinones, or unsaturated ketones or esters. For example, the dihydroindolone (309) in its thermal reaction with *N*-phenylmaleimide gives the tetracyclic adduct (310) (Scheme 93).



Scheme 93

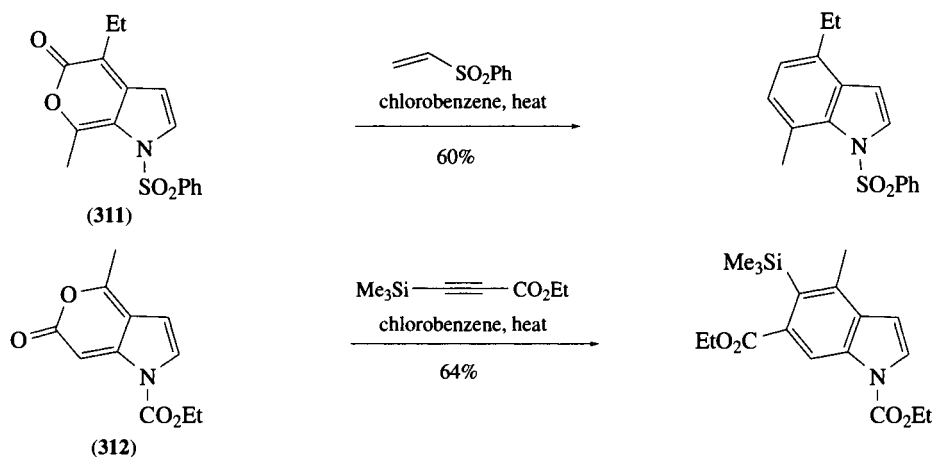
The isomeric pyranoindol-2-ones have also been used as relatively stable examples of indole quinodimethanes and offer a similar synthesis of carbazoles <90CC1667, 90JCS(P1)673, 92JOC2105>. An intramolecular version of this cycloaddition has been adapted to the synthesis of staurosporine derivatives and illustrates the synthetic process (Scheme 94).



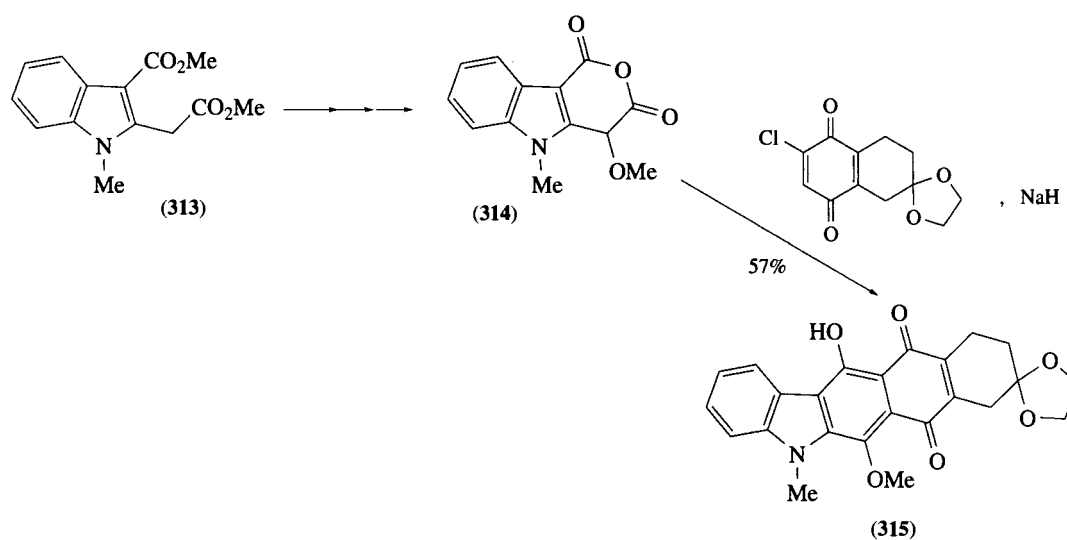
Scheme 94

The pyranoindolone strategy has also been applied to the analogous pyrrole systems. Similar quinodimethane structures (311) and (312) can be generated from pyrrole acetic acids and undergo cycloadditions to indoles (Scheme 95) <90JCS(P1)2156, 92T7447, 93T7353>.

The related pyranoindolo-2,4-dione (314), prepared from the indole diester (313), also undergoes reaction with a quinone to give a cycloadduct (315), which presumably arises from the intermediacy of a quinodiketene; the adduct is a precursor of the antitumor agent daunomycin (Scheme 96) <90CPB585>.

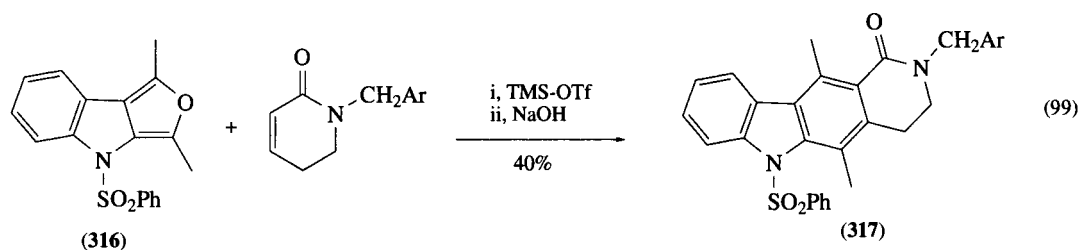


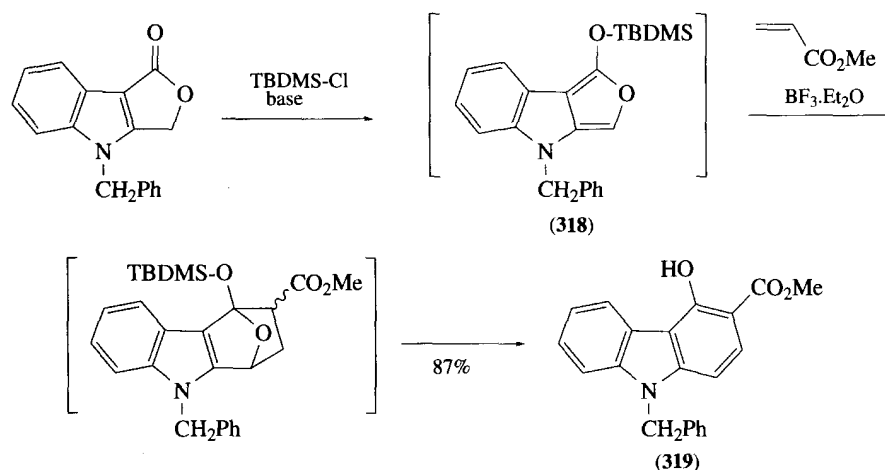
Scheme 95



Scheme 96

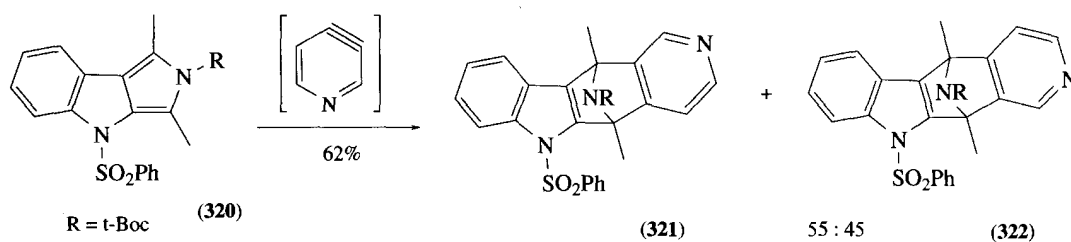
Another example of a quinodimethane structure is provided by the 4*H*-furo[3,4-*b*]indole system, which undergoes cycloaddition with dienophiles including benzyne, DMAD, maleimides, and α,β -unsaturated carbonyl systems (83TL5435, 90TL1081, 92JOC5878, 93SL333). The technique has been used in a trimethylsilyl triflate-accelerated cycloaddition to convert the furoindole (316) into the tetracyclic compound (317), which is a synthetic precursor of ellipticine (Equation (99)). A variant of this methodology also gives an effective synthesis of 4-hydroxycarbazoles (319), through the intermediacy of furoindole (318) (Scheme 97).





Scheme 97

A pyrrolo[3,4-*b*]indole (**320**), which can be prepared by an intramolecular aza-Wittig reaction and *N*-protection, acts as a quinodimethane and undergoes cycloaddition to 3,4-pyridyne to yield a mixture of isomeric adducts (**321**) and (**322**) which are precursors of ellipticine and isoellipticine (Scheme 98) <84CC1552, 92T10645>.



Scheme 98

2.02.1.8.6 [3 + 3] Cycloadditions

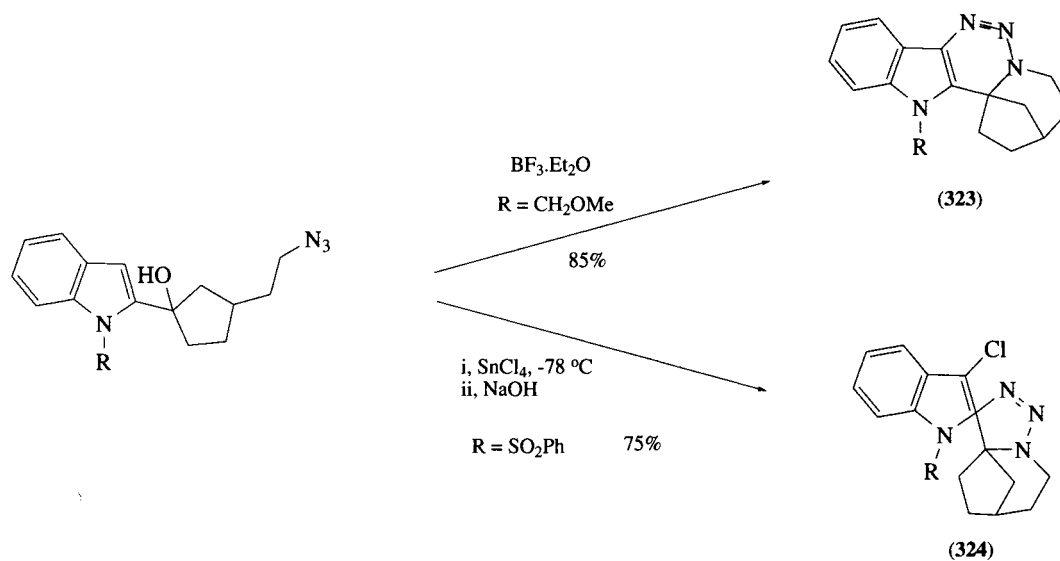
Azides undergo low-temperature [3 + 3] and [3 + 2] cycloaddition reactions to allylic carbocations to give dihydrotriazines and triazolines, respectively. Dihydrotriazines (**323**) are produced from *N*-alkylindole derivatives and triazolines (**324**) from *N*-benzenesulfonylindole derivatives (Scheme 99) <94JOC2682>.

2.02.1.8.7 [3 + 2] Cycloadditions

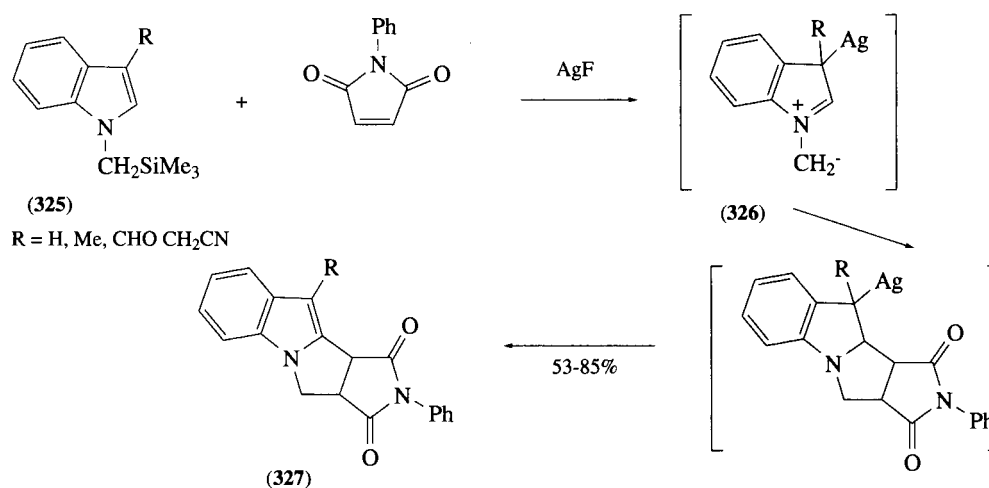
N-[(Trimethylsilyl)methyl]indoles (**325**), on treatment with silver fluoride, generate azomethine ylids (**326**), which undergo 1,3-dipolar cycloadditions with a range of electrophilic dipolarophiles including *N*-phenylmaleimide to give derivatives of pyrrolo[1,2-*a*]indoles such as (**327**), possibly via the proposed intermediate <89JOC644> (Scheme 100). Similar 1,3-dipoles can be produced from indolium salts and show similar behavior (Scheme 101) <88CB1651, 88TL5325>. An intramolecular example of this chemistry leads to the formation of a tetracyclic indoline ring system (**328**) (Scheme 102) <89TL4447>.

2.02.1.8.8 [2 + 2] Cycloadditions

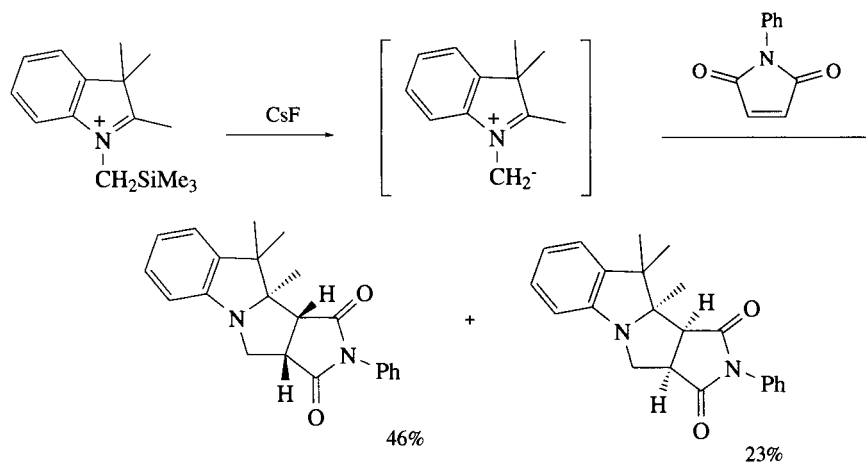
Ultraviolet light irradiation of *N*-acylindoles in the presence of alkenes gives cyclobutane ring formation. In intermolecular examples the major pathway involves bond formation of the indole



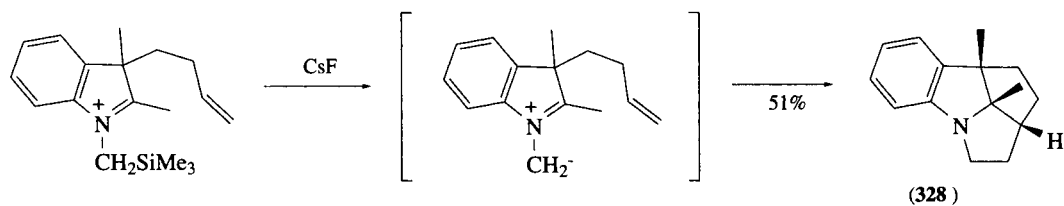
Scheme 99



Scheme 100



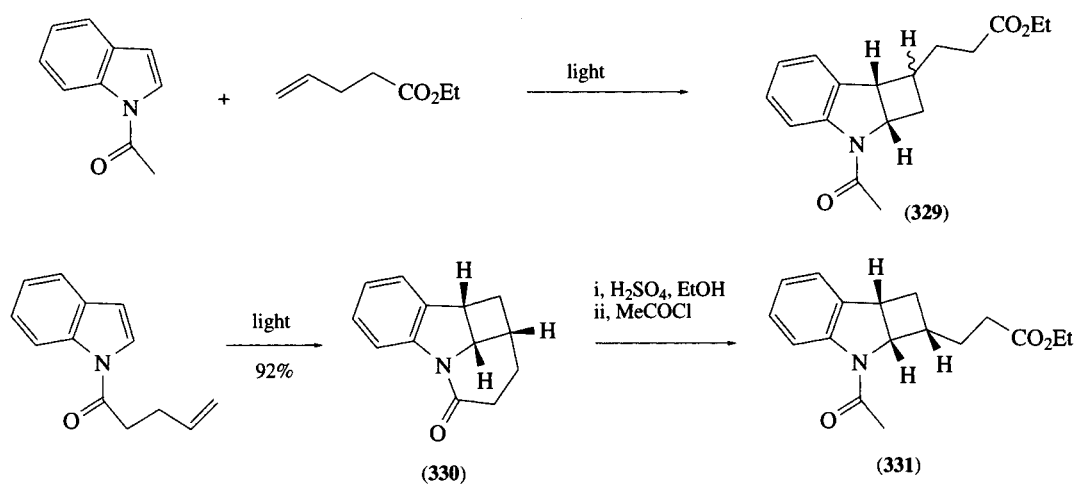
Scheme 101



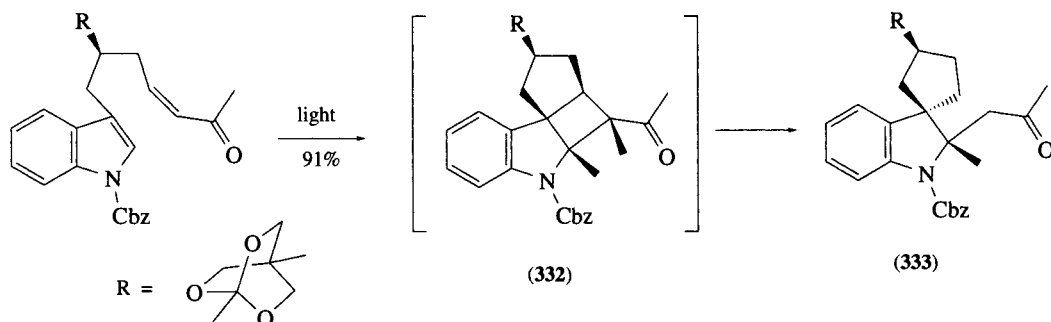
Scheme 102

C-2 with the less-substituted end of the alkene <84JCS(P1)405, 91CJC1171, 91JOC6326, 92PAC1327>. However, the use of intramolecular photocycloaddition offers a route to the alternative isomer. This is illustrated by the formation of compound (329) from the irradiation of *N*-acetylindole with ethyl pent-4-enoate, and formation of the isomer (331) by conversion of the photoadduct (330) from intramolecular reaction (Scheme 103) <92CC1491>.

An intramolecular photocycloaddition of a vinylogous amide tethered at C-3 of a 1-acylindole has also been achieved in a stereospecific reaction, in which the cyclobutane adduct (332) undergoes a retro-Mannich reaction to give the imino ketone (333), a synthetic precursor of vindorosine (Scheme 104) <90JA8971>.



Scheme 103

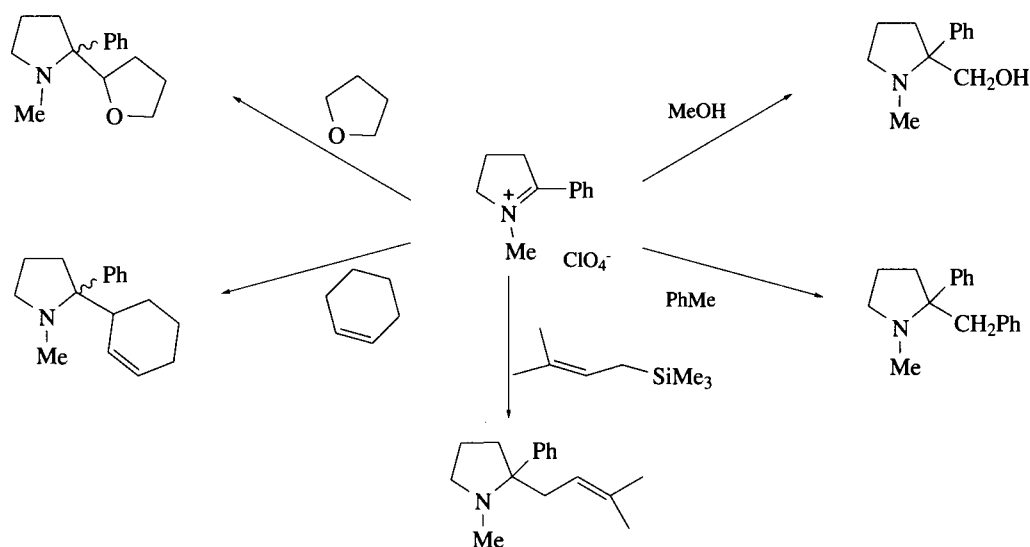


Scheme 104

2.02.2 REACTIVITY OF NONCONJUGATED RINGS

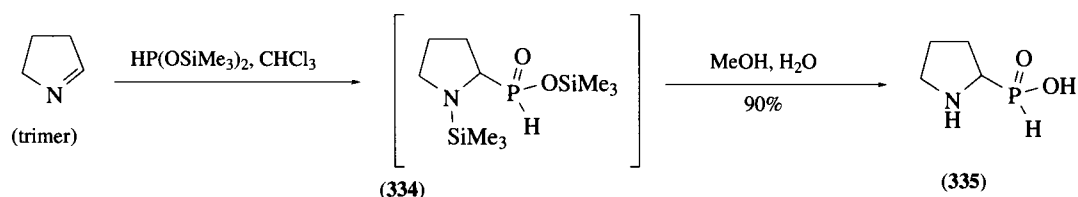
2.02.2.1 Pyrrolenines and Indolenines

Pyrroleninium salts undergo alkylation in photoinduced electron-transfer processes with ethers <94T575>, alcohols <81T3385>, alkenes <81JA3136>, alkylarenes <87JA2738>, silanes, and stannanes <84JOC213>. Imines can also be generated *in situ* by oxidation of related amines with 1,4-dicyano-naphthalene (Scheme 105) <92TL5107>.



Scheme 105

The trimer of 1-pyrroline reacts readily with bis(trimethylsilyl)phosphonite to yield 2-pyrrolidinylphosphinic acid (**335**), following the addition of aqueous methanol to the intermediate trimethylsilyl derivative (**334**) (Scheme 106) <94TL1103>.

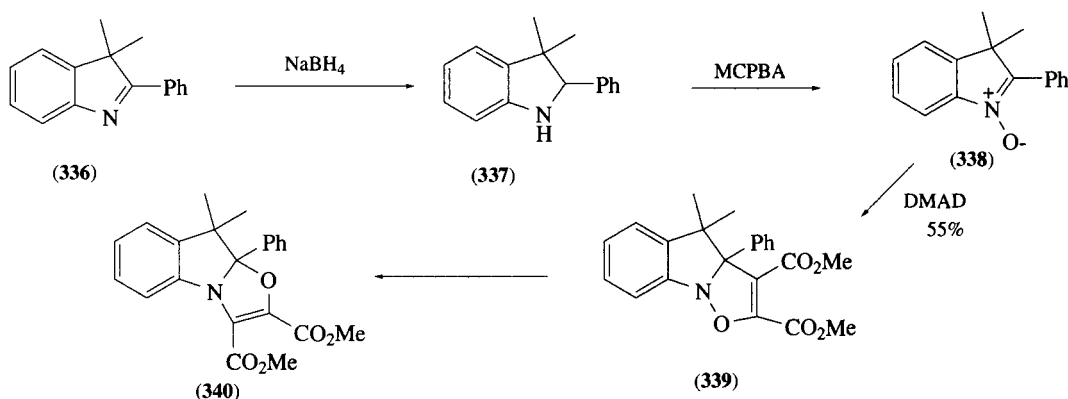


Scheme 106

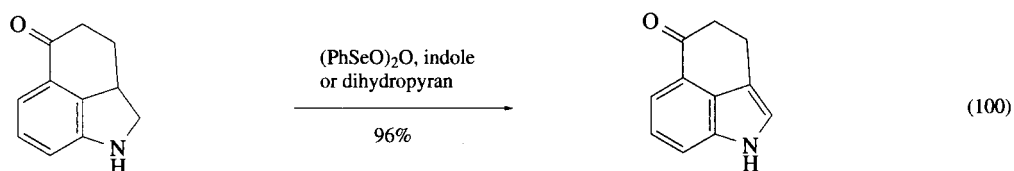
Indolenines exemplified by compound (**336**) can be converted into the related 3*H*-indole 1-oxide (**338**) by a sequence involving reduction to the dihydro compound (**337**) followed by oxidation with *m*-chloroperbenzoic acid. Compound (**338**) behaves as a typical cyclic nitrene and undergoes cycloaddition with DMAD and methyl propiolate. The initially formed cycloadduct (**339**) rearranges via an aziridine intermediate to an oxazolo[3,2-*a*]indole (**340**) (Scheme 107) <93JCS(P1)939>.

2.02.2.2 Dihydro Derivatives

From a synthetic point of view, indolines are used to provide different reactivity properties from the indoles. Once a required sequence of reactions is achieved, the indoline can then be oxidized readily to the indole. One of the most effective methods for the conversion of indolines to indoles involves treatment with benzeneseleninic anhydride, together with three equivalents of a sacrificial scavenger such as indole or dihydropyran to remove the phenylselenating by-product; the method has been used in the synthesis of ergot alkaloids (Equation (100)) <85TL4183, 90JCS(P1)707>.

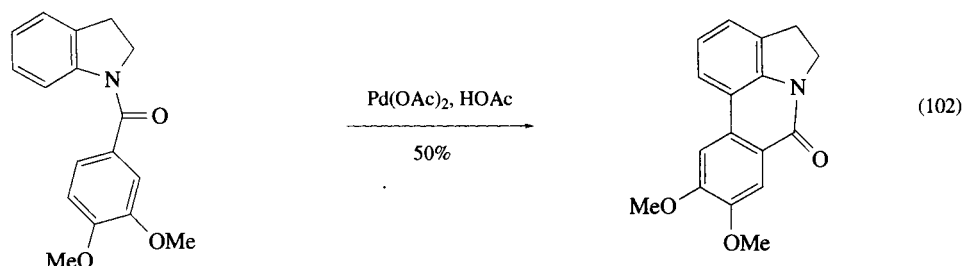
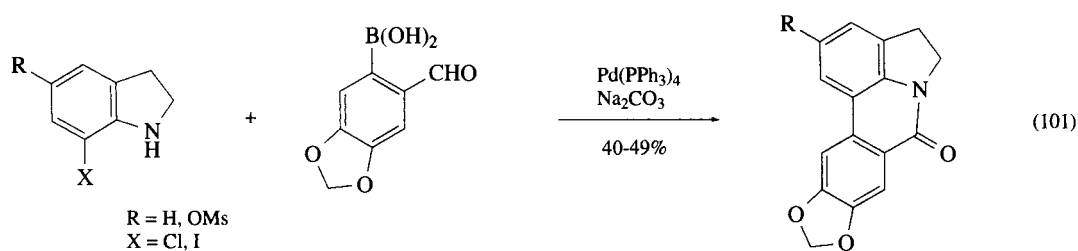


Scheme 107



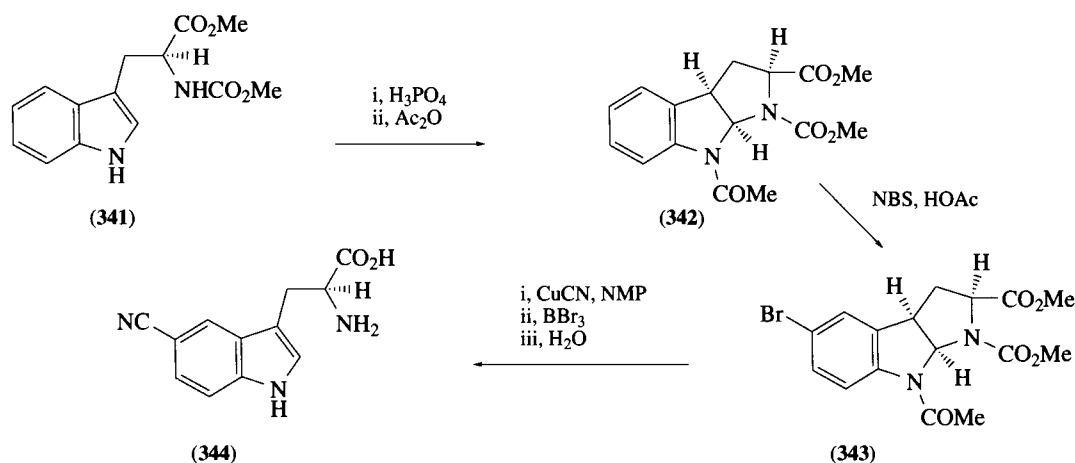
1-(*t*-Butoxycarbonyl)indolines undergo regioselective lithiation at C-7 using *s*-butyllithium and tetramethylethylenediamine at low temperature, and this reactivity allows a route to 7-substituted indolines <92H(34)1031>.

7-Haloindolines take part in cross-coupling reactions with *o*-formylboronic acid under modified Suzuki conditions to give pyrroloquinolines (Equation (101)) <90TL1523>. With the similar aim of achieving a short and effective route to the pyrrolophenanthridone alkaloids, palladium acetate-mediated coupling of a 1-aryloindoline has been shown to occur at C-7 and generate the biphenyl link; a similar reaction of the related indole results in coupling to C-2 (Equation (102)) <89TL5807>.

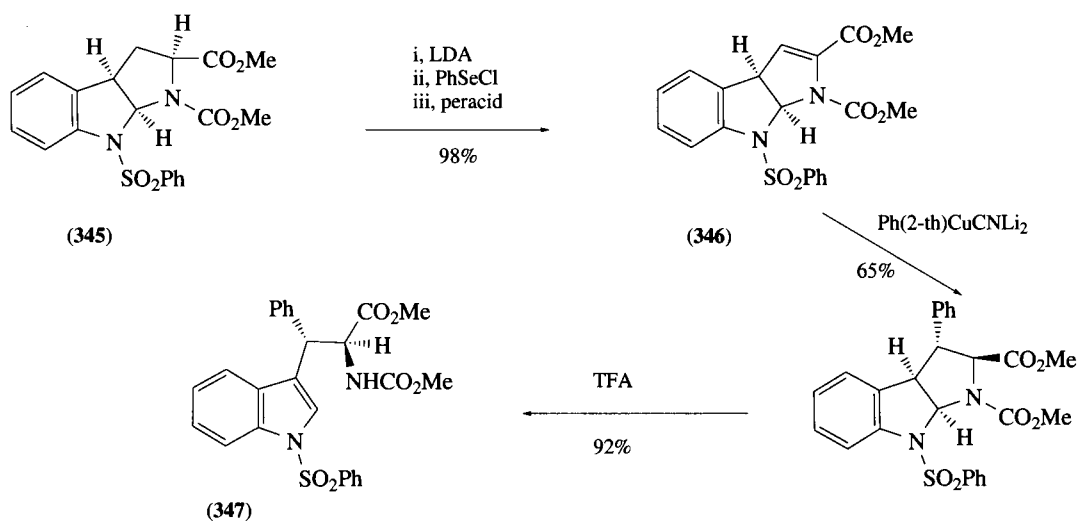


Dihydroindoles are readily produced from tryptamine and tryptophan derivatives through cyclization of amino groups at the indole C-2. This process can be reversed to regenerate the indole after advantage has been taken of the indoline reactivity. For instance, the tryptophan ester (341) can be cyclized to the indoline (342), which can then be brominated at C-5; the product (343) can also be converted to 5-cyanotryptophan (344) (Scheme 108) <92TL29>. The related indoline (345) has been

dehydrogenated to give an α,β -unsaturated ester (**346**), to which mixed 2-thienylcyanocuprates have been added in a highly stereoselective manner. After ring opening, the β -substituted tryptophan derivatives (**347**) can be obtained (Scheme 109) <92TL6251>.



Scheme 108

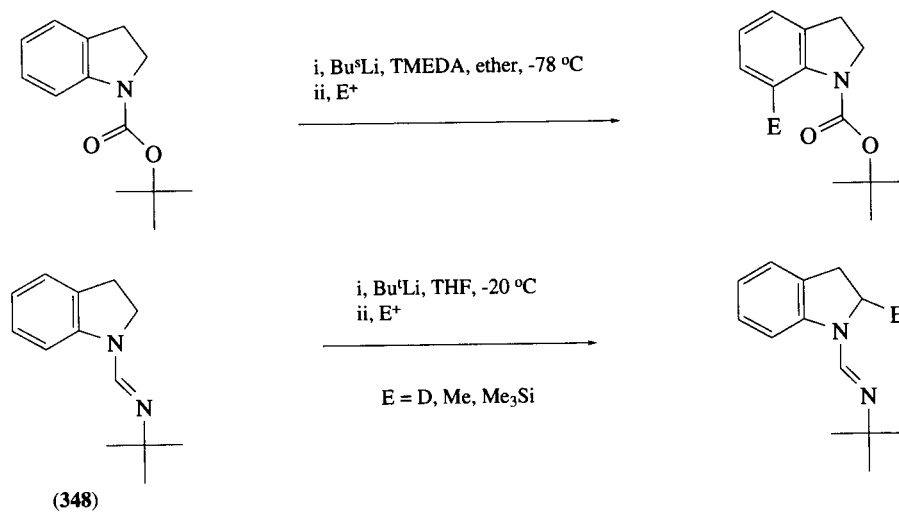


Scheme 109

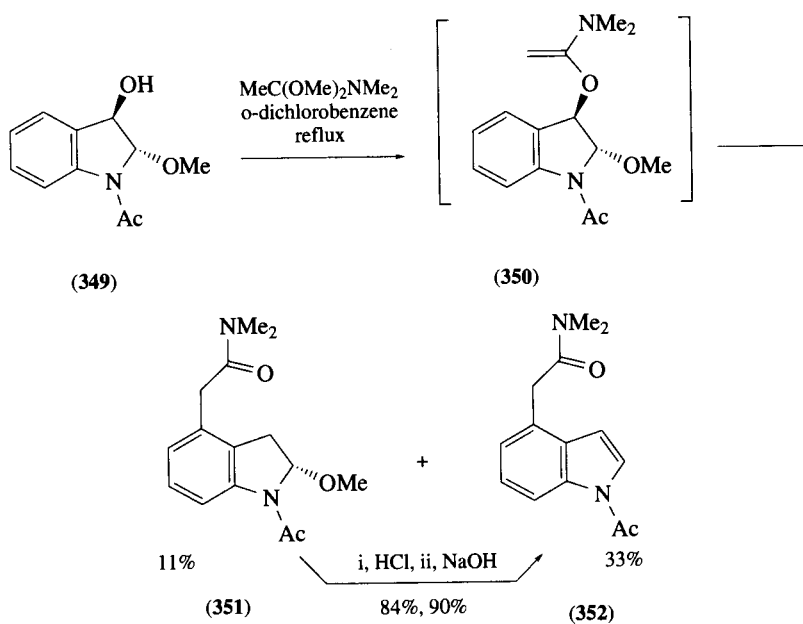
Indoline shows interesting metallation and alkylation properties, which are dependent on the nature of the nitrogen substituent. When the *N*-substituent is *t*-butoxycarbonyl, metallation and alkylation occur exclusively at the *ortho* (C-7) position, whereas reaction occurs exclusively at the alkyl C-2 position in the case of the *N*-formamidine (**348**) (Scheme 110) <89TL1197, 92H(34)1031, 93JOC6538>.

An interesting method of indole C-4 substitution has been developed from a Claisen rearrangement. Reaction of 3-hydroxy-2-methoxyindoline (**349**) with *N,N*-dimethylacetamide dimethylacetal gives the intermediate (**350**), which undergoes a Claisen rearrangement to yield a mixture of the 4-substituted indoline (**351**) and indole (**352**). The indoline can be converted into the indole by a sequence of methanol elimination and base hydrolysis (Scheme 111) <90CC781>.

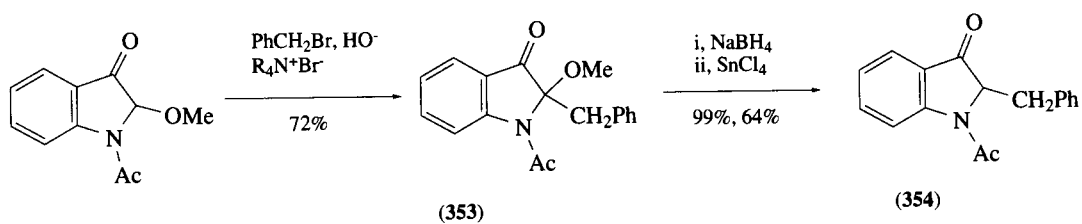
1-Acetyl-2-methoxy-1,2-dihydroindol-3-one can be alkylated under phase-transfer conditions to the indolone (**353**), which on reduction and acid-catalyzed rearrangement yields a 2-monoalkylated 1*H*-indol-3-one (**354**) <91JCS(P1)2445> (Scheme 112).



Scheme 110

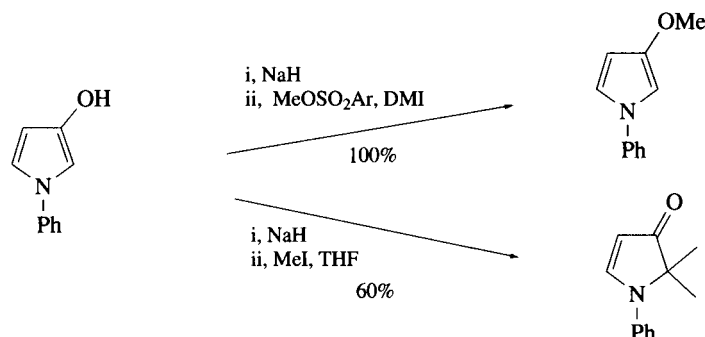


Scheme 111



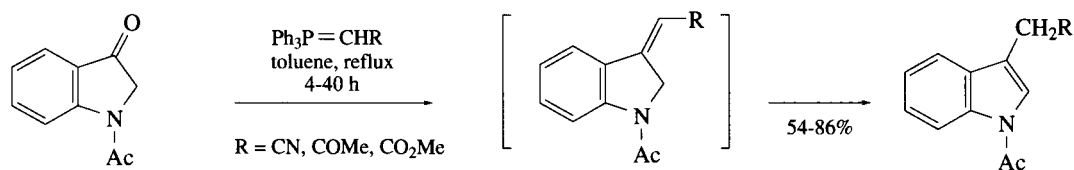
Scheme 112

The alkylation of 1*H*-pyrrol-3(2*H*)-ones gives a preference for *C*-alkylation with alkyl halides, but *O*-alkylation with methyl tosylate in the dipolar aprotic solvent *N,N*-dimethylimidazolidinone (DMI) (Scheme 113) <91JCS(P1)3245>.



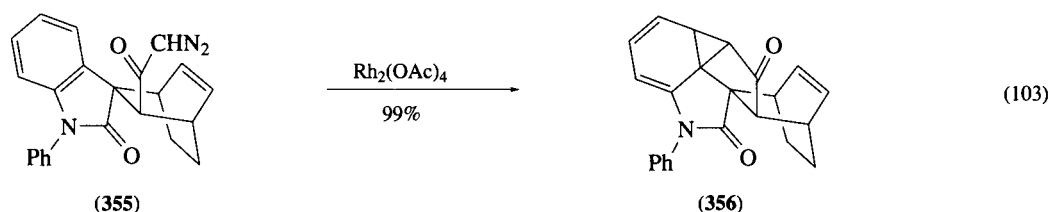
Scheme 113

The carbonyl group of 1-acetylindol-3-one is reactive towards phosphoranes and enables the resulting alkenes to generate indoleacetic acid esters and indolylmethyl ketones (Scheme 114) <91S701>.



Scheme 114

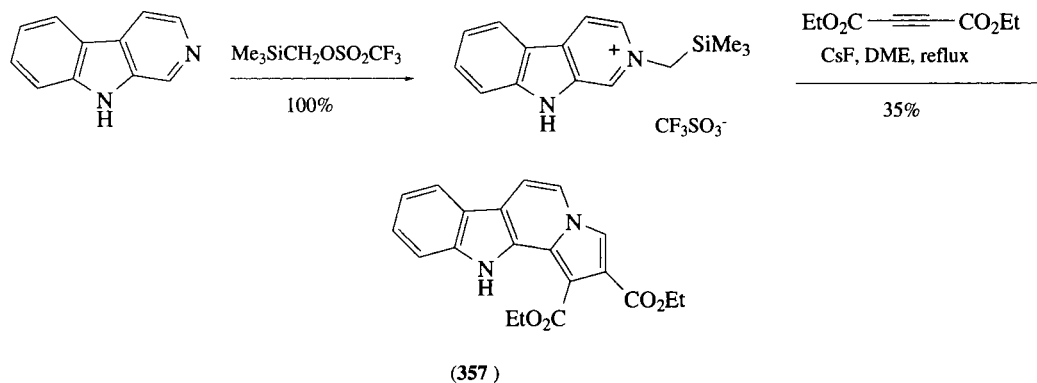
A range of diazoketones such as (355), generated from spiroindolones, undergo a surprising insertion of their carbenes into the C-3a to C-4 bond to yield the related norcaradiene (356) in preference to other alternatives (Equation (103)) <92S323>.



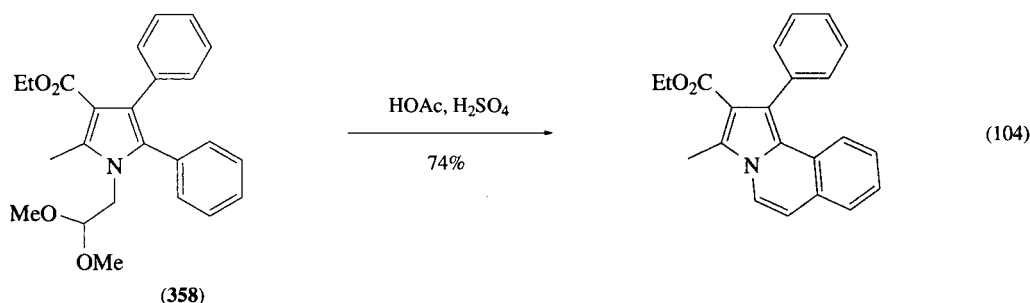
2.02.3 REACTIVITY OF SUBSTITUENTS ATTACHED TO RING CARBON ATOMS

2.02.3.1 Fused Benzene Rings and Aryl Groups

The quaternization of a β -carboline, followed by the generation of an azomethine ylid, enables cycloaddition of diethyl acetylenedicarboxylate to yield the fused pyrrole product (357) (Scheme 115) <89TL3423>. Similar systems, namely the pyrrolo[2,1-*a*]isoquinolines, have been formed by an intramolecular electrophilic substitution on the 2-aryl ring of a suitably substituted pyrrole (358) (Equation (104)) <93JCS(P1)2761>.

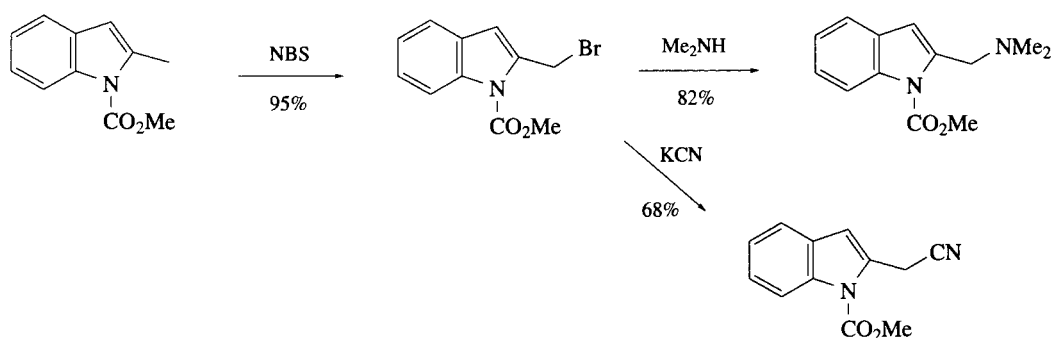


Scheme 115



2.02.3.2 Alkyl and Substituted Alkyl Groups

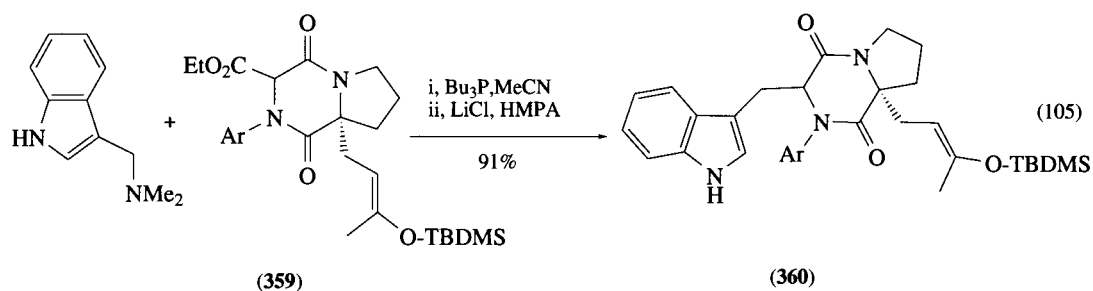
Radical bromination of pyrrole and indole methyl substituents continues to be investigated, as does the substitution of the resulting bromo compound. Selective bromination of the 2-methyl group in 1-acetyl-2,3-dimethylindole can be achieved and the bromide ion displaced by malonate $\langle 85\text{TL}5253 \rangle$. A recent example of an effective sequence of bromination and substitution is shown in Scheme 116 $\langle 92\text{S}743, 93\text{OPP}249, 93\text{TL}3215 \rangle$.



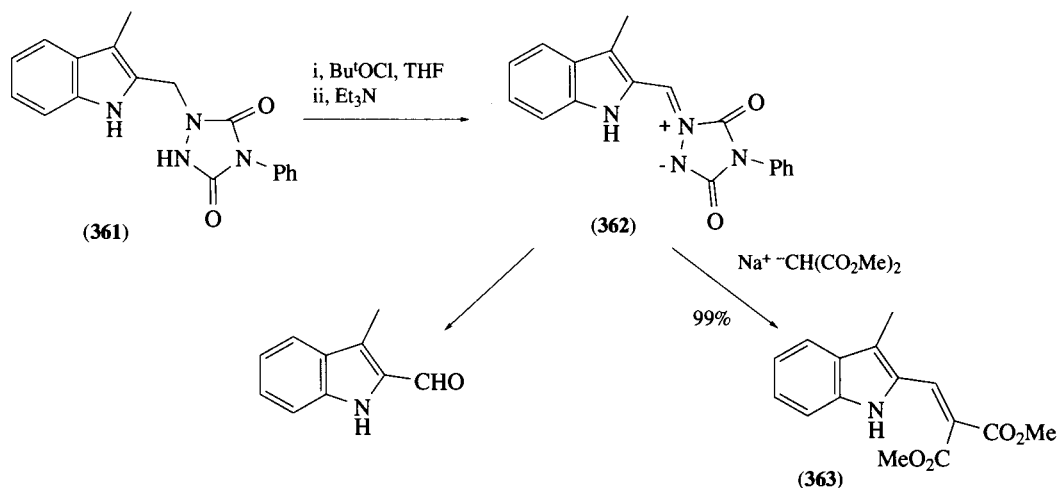
Scheme 116

Another useful intermediate is 3-(iodomethyl)-1-(benzenesulfonyl)indole which undergoes enolate alkylation. It has been used to introduce a 3-indolylmethyl group in the synthesis of the indole diterpene emeniveol $\langle 90\text{TL}4697, 93\text{TL}7383 \rangle$. 2- and 7-Hydroxymethylindoles undergo acid-catalyzed reactions with activated indoles to produce diindolylmethanes and higher oligomers including cyclic trimers $\langle 93\text{CC}819 \rangle$.

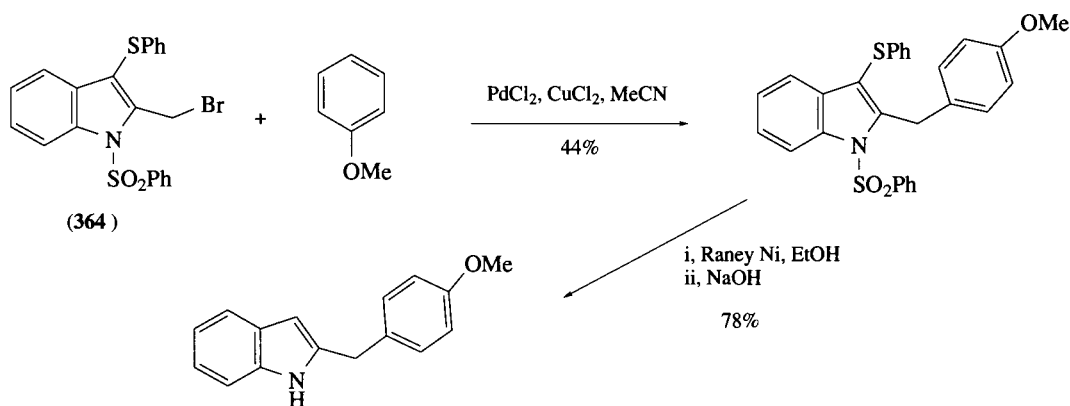
The phosphine-modified alkylation of gramine has also been used to alkylate the diketopiperazine (359) to give the brevianamide precursor (360) (Equation (105)) $\langle 93\text{T}8471 \rangle$.



Displacement of bromide ion from a 2-bromomethylindole has also been achieved with a triazolinedione to give compound (361), which on chlorination and dehydrochlorination forms the azomethine ylid (362); this in turn is attacked by nucleophiles such as water and malonate ion to give 3-methylindole-2-carbaldehyde and arylidenemalonate (363), respectively (Scheme 117) <85TL3673>. Palladium-mediated arylation of a 2-bromomethylindole (364) can generate a 2-benzylindole, with or without a 3-phenylthio substituent (Scheme 118) <92S835>. The tetrahydro- β -carboline (365) undergoes a useful ring expansion by destruction of the bond between two rings, as a result of acylation on nitrogen (Scheme 119) <89CC518>.

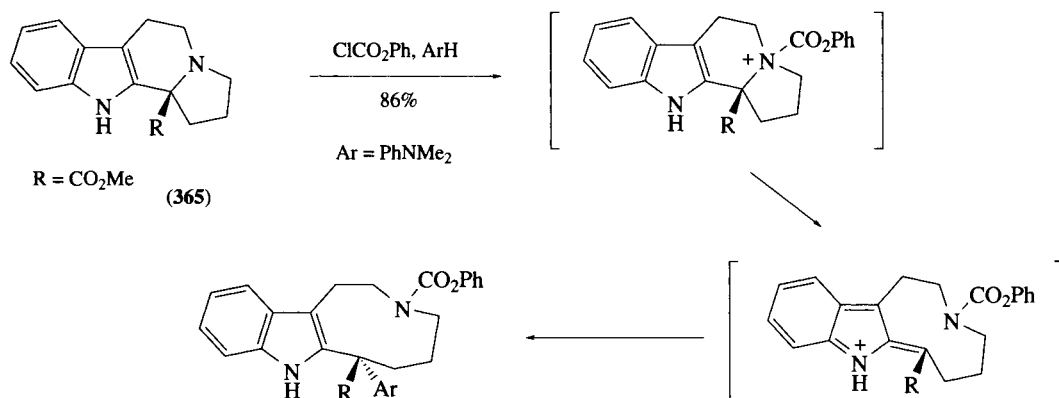


Scheme 117



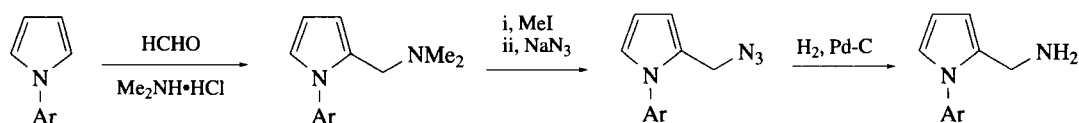
Scheme 118

The Mannich reaction provides a very convenient method for the introduction of a dialkylamino-methyl group into the 2-position of a pyrrole ring. The dialkylamino group has previously been displaced by various nucleophiles but not azides <73S703, B-77MI 202-01, 85CCA627, 90T1791>. This has



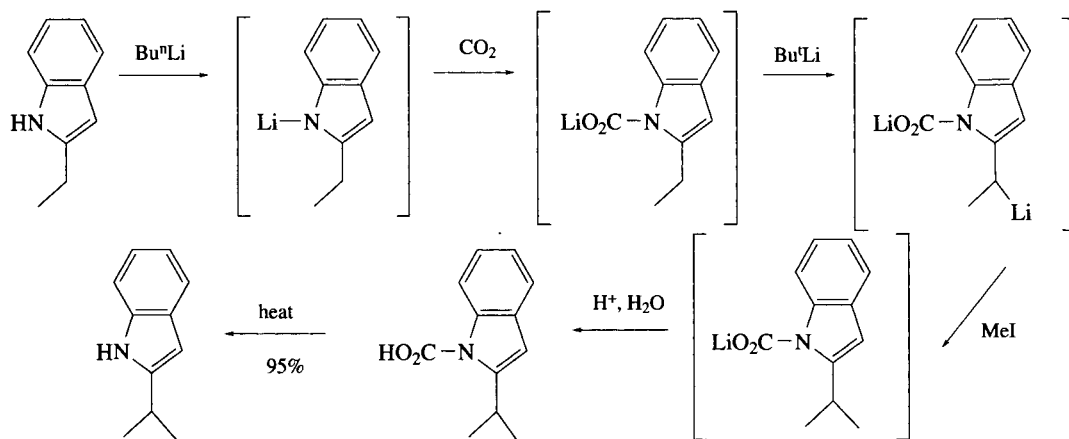
Scheme 119

now been accomplished by displacement of triethylamine from the quaternary salt derived from the Mannich base of a 1-arylpyrrole (94S164). The resulting azide can be reduced by catalytic hydrogenation to give the aminomethylpyrrole (Scheme 120). Another method for the introduction of nucleophilic groups on a 2-methylene of a pyrrole is by flash vacuum pyrolysis of 2-dimethylaminomethylpyrrole at 650°C . Dimethylamine is eliminated and the reactive intermediate is trapped by thiophenol. Similar behavior is observed for 2-pyrrolylmethyl phenyl sulfoxide, which loses phenylsulfinic acid at 110°C , and the intermediate can be trapped by amines, alcohols, thiols, and cyanide ion (93TL1187).



Scheme 120

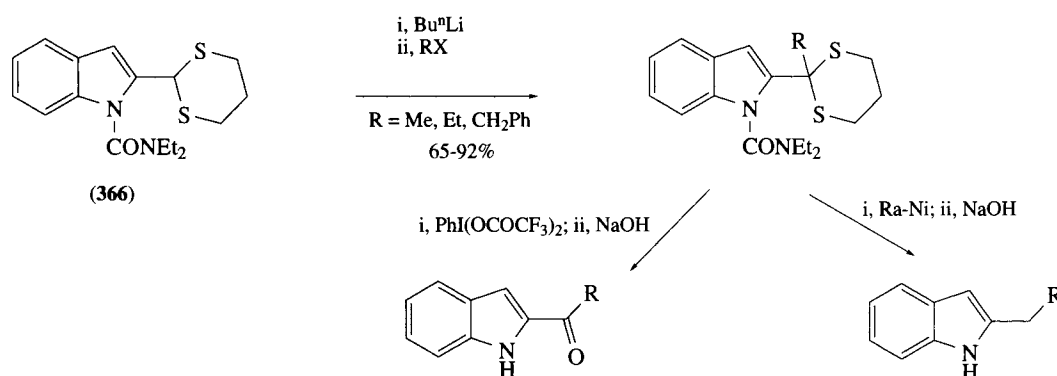
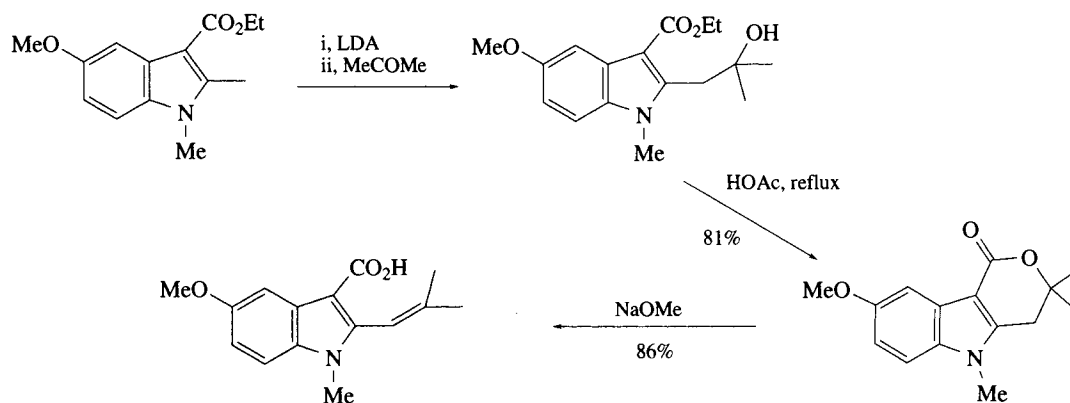
Side chain lithiation is a major source of reactivity in terms of the elaboration of pyrroles and indoles, and new examples and developments continue to be reported. One major development has been the lithiation of 2-alkyl groups of 2-alkylindole-1-carboxylate lithium salts. The carboxyl group protects the nitrogen atom, directs the lithiation, and can easily be removed after alkylation (Scheme 121) (86JA6808). Similarly, lithiation of 2,3-dialkylindoles takes place at the 2-methylene position via the C,N -dilithio derivative, using three equivalents of butyllithium (91JOC2256).



Scheme 121

Lithiation at the 2-methylene group of 1-substituted-2-alkylindole-3-carboxylate esters, followed by addition to ketones, gives aldols; these can be cyclized to form pyrano[4,3-*b*]indoles, which are precursors of 2-vinylindoles (Scheme 122) <89JOC4785>.

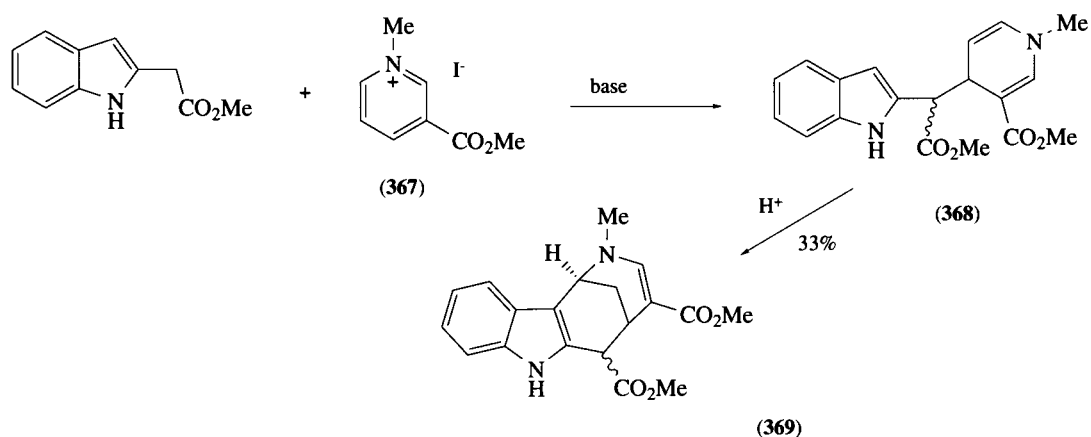
The *N,N*-diethylcarbamoyl group has been used to protect the indole nitrogen atom and to enable lithiation to occur at C-2. The lithio derivative can be converted into the dithiane (**366**), which can again be lithiated and alkylated to give a range of products derived from a variety of electrophiles. In this respect it is superior to the arenesulfonyl groups, which allow some 3-lithiation to occur <90H(31)173>. Subsequent removal of the dithiane and diethylcarbamoyl groups provides useful indole derivatives (Scheme 123) <91T7911>. Similar dithiane carbanions, but with unprotected nitrogen, have also been added to pyridinium salts to generate alkaloid precursors <90TL1893>.



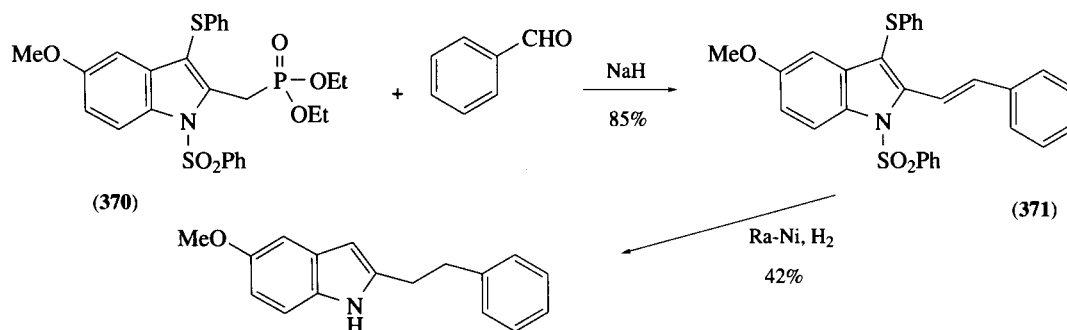
A procedure has been established for the synthesis of certain aspidospermine alkaloids, based on the addition of an enolate of methyl 2-indolylacetate to a pyridinium salt (**367**), followed by acid-catalyzed cyclization of the resulting 1,4-dihydropyridine (**368**) to the tetracyclic structure (**369**) (Scheme 124) <84H(21)325, 89JOC1166, 94T5233>.

2-Indolylmethylphosphonates (**370**) have been prepared from the corresponding bromides and undergo Wittig reactions with aryl aldehydes to yield styryl compounds (**371**); the 3-phenylthio group can be removed by reduction (Scheme 125) <85S188>.

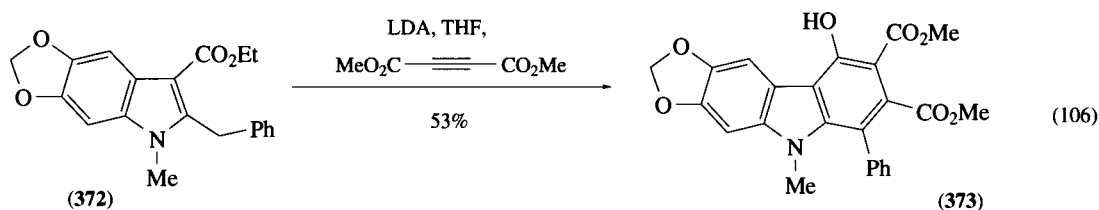
The 2-benzylindole-3-carboxylic ester (**372**) is postulated to form the carbanion on treatment with base, and the product of its addition to DMAD is the hydroxycarbazole (**373**) (Equation (106)) <92TL1655>. It is possible that the reaction is actually a cycloaddition involving a quinodimethane ketene structure (see Section 2.02.1.8).



Scheme 124

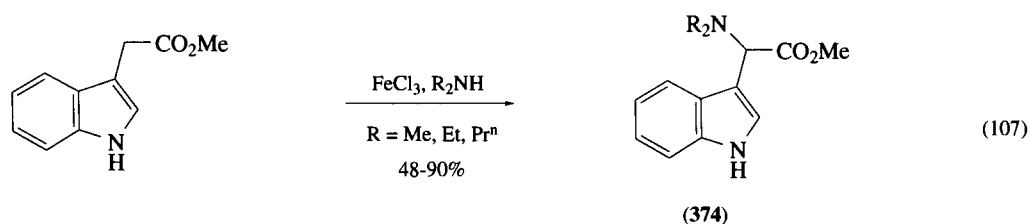


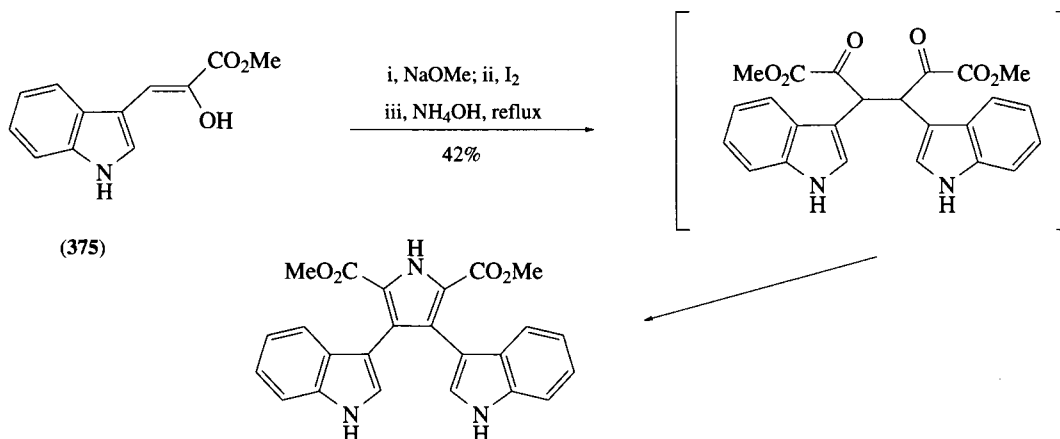
Scheme 125



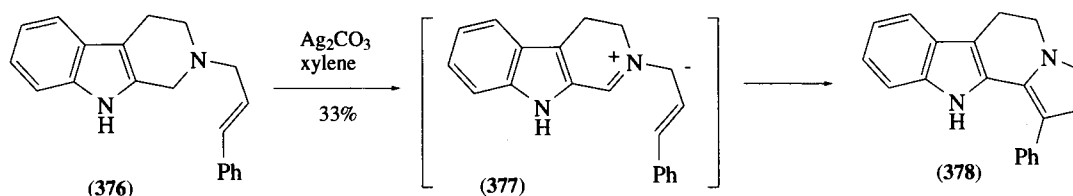
Oxidation of methyl 3-indolylacetate with iron(III) chloride in the presence of a secondary amine gave the amino esters (374) in good yield (Equation (107)) <89TL5337>. Oxidative dimerization of a 3-(indol-3-yl)pyruvate (375) has also been used as an entry to the diindolylpyrrole structures of the lycogalic acids (Scheme 126) <94TL1689>. The tetrahydro- β -carboline (376) also undergoes oxidation with silver carbonate to give an azomethine ylid (377) which cyclizes to the pyrrolocarbazole (378) (Scheme 127) <92T9735>. 2,3-Dialkylindoles can be oxidized to the corresponding 3-acylindole by treatment with nitroxide radicals <90H(30)1131>. A 4-(indolyl)ethanal has been used as a substrate for Fischer indole synthesis and gave rise to a 3,4'-biindolyl <85JA2943>.

The readily available 3-substituted indole (379) can be converted by triethyl orthoformate to the



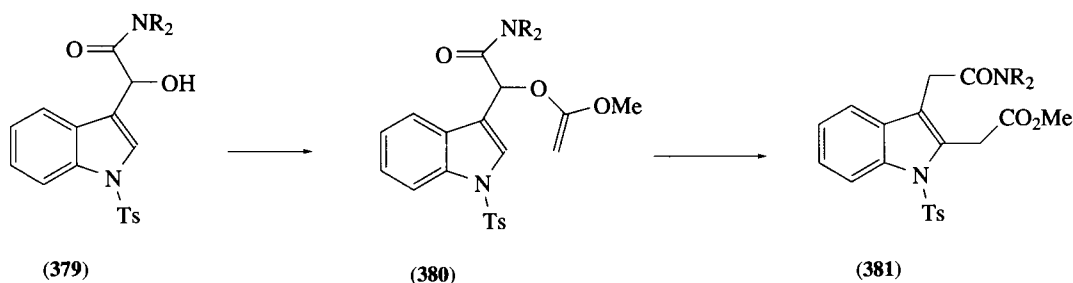


Scheme 126



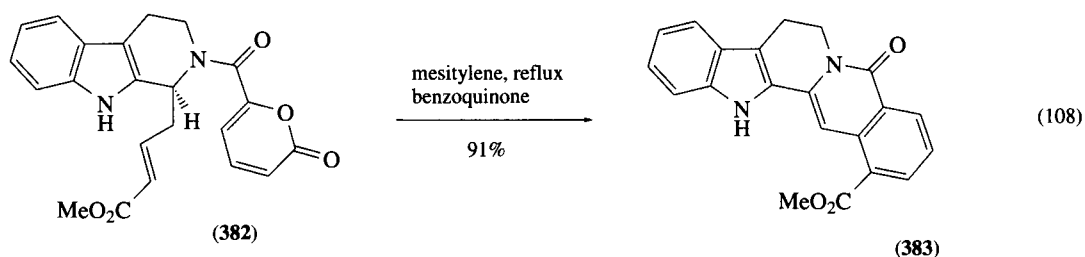
Scheme 127

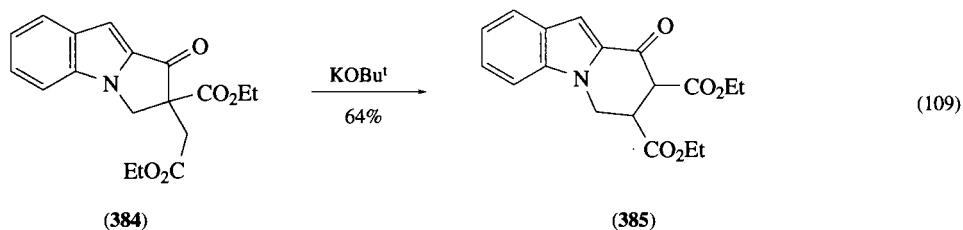
ketene acetal (380), which cannot be isolated because it undergoes Claisen rearrangement to the 2,3-disubstituted indole (381) (Scheme 128) <86JOC123>.



Scheme 128

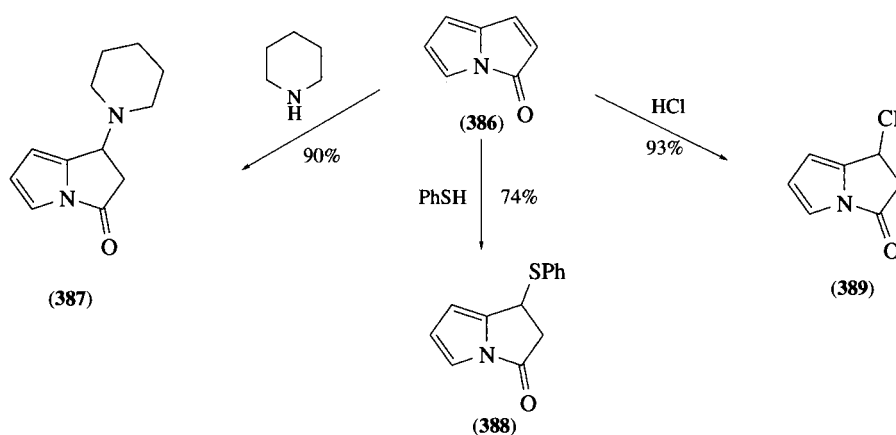
There are numerous examples of cyclizations between substituents located on pyrrole and particularly indole rings, and the linkages are substituted alkyl. However, in most cases the ring system has no real effect on the chemistry. An oxidative intramolecular Diels–Alder reaction converts the β -carboline (382) into the pentacyclic structure (383) (Equation (108)) <88TL6725>. The pyrroloindole (384) undergoes a ring expansion to the pyridoindole (385) (Equation (109)) <91T4645>. Remote cyclizations have been a feature of the syntheses of indolactam derivatives <86CC344, 94TL8549>, clavicipitic acid derivatives <88JOC5128>, and strychnos alkaloids <93JOC7482>.





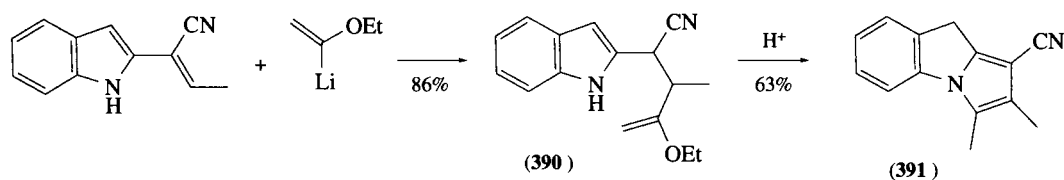
2.02.3.3 Alkenyl and Alkynyl Groups

Pyrrolizin-3-one (**386**) undergoes conjugate nucleophilic addition of piperidine and thiophenol and electrophilic addition of hydrogen chloride to give a range of 1-substituted 1,2-dihydropyrrolizin-3-ones (**387**), (**388**), and (**389**) respectively; the 1-chloro atom can readily be displaced by nucleophiles, including water (Scheme 129) <93CC1570>.



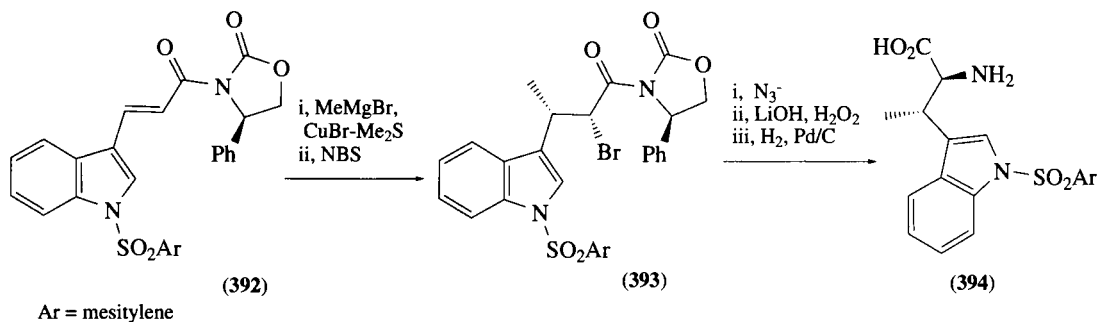
Scheme 129

Conjugate nucleophilic addition of methyl, vinyl, allyl, and malonate groups to 1-(2-indolyl)but-2-enecarbonitrile has been effected with a range of organometallic carbanions, for example, the lithium derivative of ethyl vinyl ether gives the product (**390**), which can be cyclized to a pyrrolo[1,2-*a*]indole (**391**) (Scheme 130) <91TL7237>.



Scheme 130

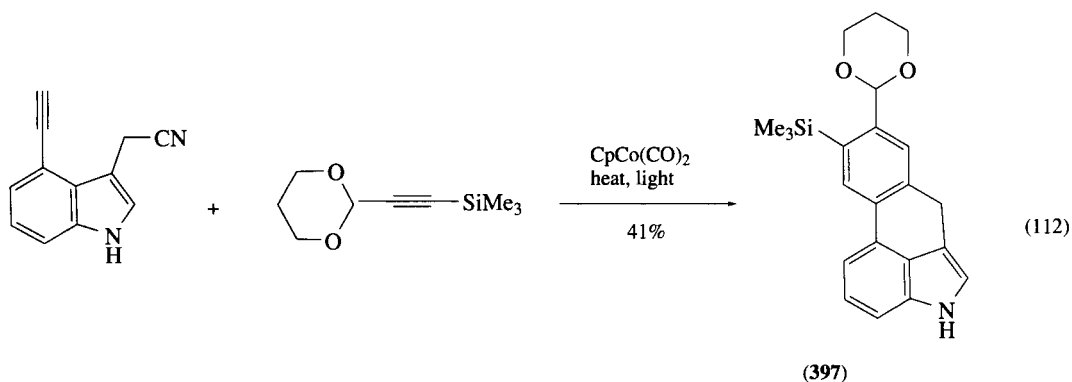
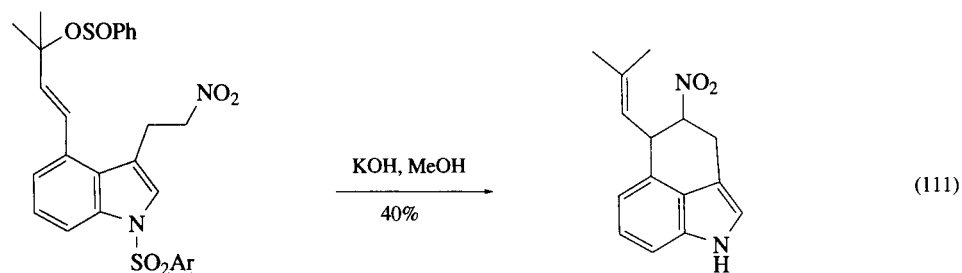
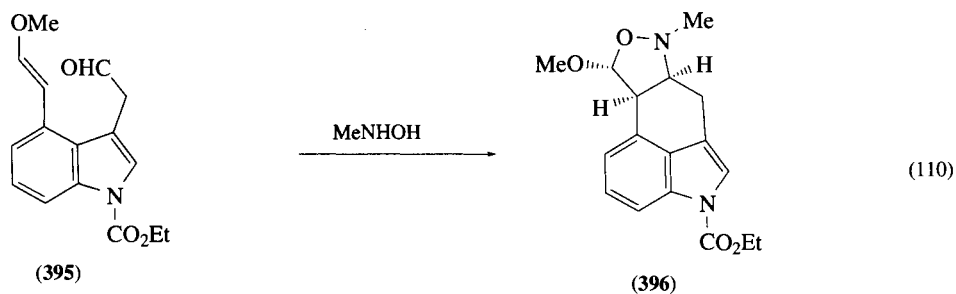
β -Methyltryptophan derivatives have been synthesized stereoselectively using chiral auxiliaries. Conjugate addition of magnesium methylcuprate to the indole acrylic acid (**392**) gives an enolate which can be trapped by bromination to give the product (**393**), which can then be transformed into the tryptophan (**394**) by azide displacement, reduction and hydrolysis (Scheme 131) <92TL7491>.



Scheme 131

Carbon-carbon double bonds directly attached to indole rings have been shown to participate in cyclization reactions. The vinyl ether (395), on treatment with *N*-methylhydroxylamine, undergoes a 1,3-dipolar cycloaddition to the adduct (396) (Equation (110)) <85JA2569>. The intramolecular addition of a nitroalkane anion to an allylsulfinate gives a similar ergot alkaloid precursor (Equation (111)) <86TL3169>.

The 3-cyanomethyl and 4-ethynyl substituents of an indole can be linked in a cobalt-catalyzed reaction with alkynes, to generate ergoline derivatives (397) in modest yield (Equation (112)) <94SL487>.

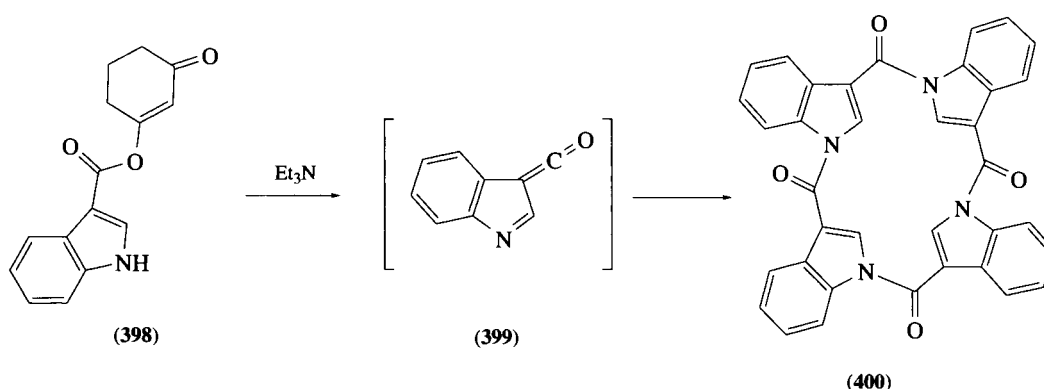


2.02.3.4 Carboxylic Acids and Their Derivatives

Improvement to the decarboxylation of indole-2-carboxylic acids has been made by the use of microwave radiation. Under these conditions the copper chromite can be eliminated from the standard reagent mixture of copper chromite in quinoline and reactions are faster and cleaner <93JOC5558>. The ketene (**399**) generated from either the acid chloride or enol ester (**398**) of indole-3-carboxylic acid undergoes cyclic tetramerization to give the macrocyclic product (**400**) (Scheme 132) <91JHC1569>.

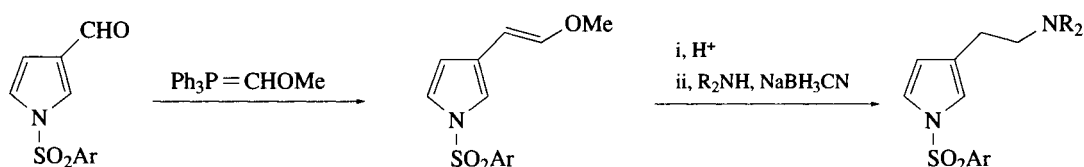
2.02.3.5 Aldehydes and Ketones

Pyrrolyl and indolyl aldehydes and ketones undergo the usual reactions of those functional groups. Activity in the indole area throughout the 1980s and 1990s, has made use of aldehyde and ketone reactions with resultant formation of imines <86CC634>, nitrones <92AJC611, 92AJC1051, 93AJC1725>, alcohols through Grignard and aryllithium addition <88AP(321)823, 89CC425>, vinyl indoles through Wittig reactions <89M157>, and nitrovinyl compounds through reaction with nitromethane <92AJC1879, 93T10817>. The Wittig reaction has also been used to introduce the dialkylaminoethyl group into pyrroles, through reaction of the 3-carbaldehyde with a methoxymethylenephosphorane and subsequent conversion of the methyl enol ether of pyrrole-3-carbaldehyde <88JHC635> (Scheme 133).

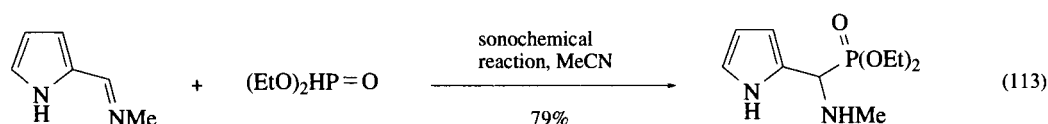


Scheme 132

Diethyl phosphonate undergoes sonochemically activated addition to the methylimine of pyrrole-2-carbaldehyde to yield an aminophosphonic acid (Equation (113)) <94S51>.



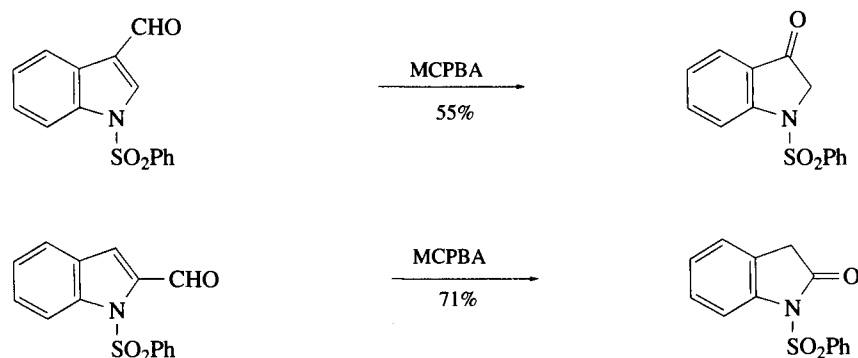
Scheme 133



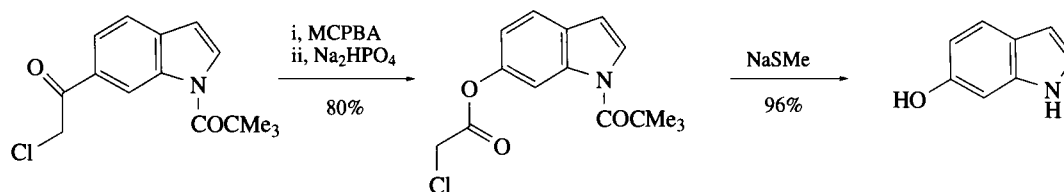
A sequence of bromination of a 3-indolyl acyl group followed by azide displacement and reduction, provides an effective route to 3-(α -aminoacyl)indoles <88JHC469>. Indoles react readily with oxalyl chloride at C-3 and the products have been used in combination with aryl acetic acid derivatives to generate indolyl-substituted maleic anhydrides and imides, as precursors for indolocarbazole antibiotics <90TL2353, 93TL5623>.

Indole-2-carbaldehyde can be effectively decarbonylated using a rhodium phosphine catalyst in refluxing xylene <84JOC3195>.

The Baeyer–Villiger oxidation of 2- or 3-formylindoles, suitably protected by *N*-acyl or sulfonyl groups, can give moderate yields of 1,2-dihydroindolones (Scheme 134) <94S411>. The similar oxidation of 6-chloroacetylindoles is more effective and allows the formation of 6-hydroxyindoles in high yield (Scheme 135) <94S1018>.

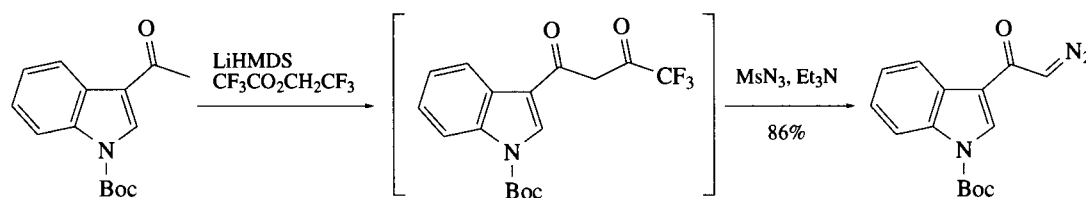


Scheme 134



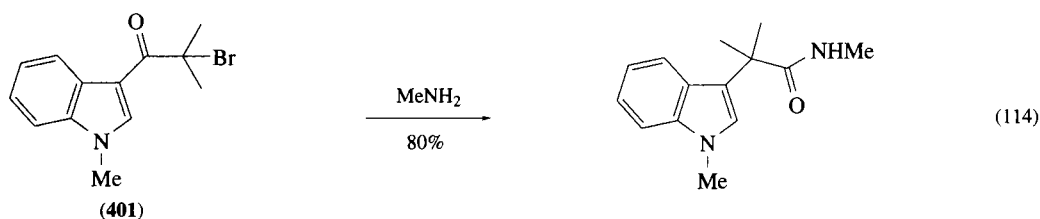
Scheme 135

The 3-acetyl group on an indole can be transformed into an α -diazoketone by a deformylative diazo transfer process, involving treatment with base and trifluoroacetylation followed by base-catalyzed reaction with mesylazide (Scheme 136) <90JOC1959>. This methodology has been used in a synthesis of the oxazolyl indole alkaloid pimprinine <94S1021>.



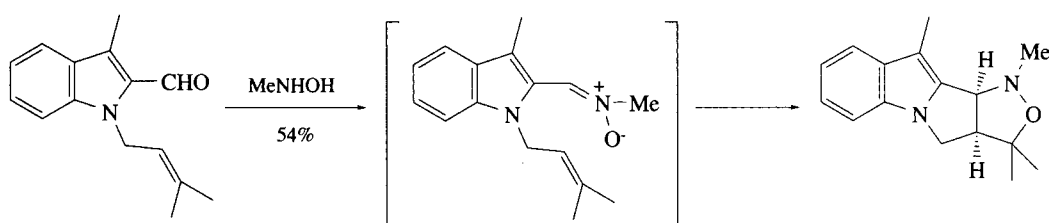
Scheme 136

Reaction of the α -bromoketone (401) with primary amines gives good yields of amides resulting from a Favorskii type mechanism; the amides in turn are a useful source of substituted tryptamines (Equation (114)) <90JHC1601>.

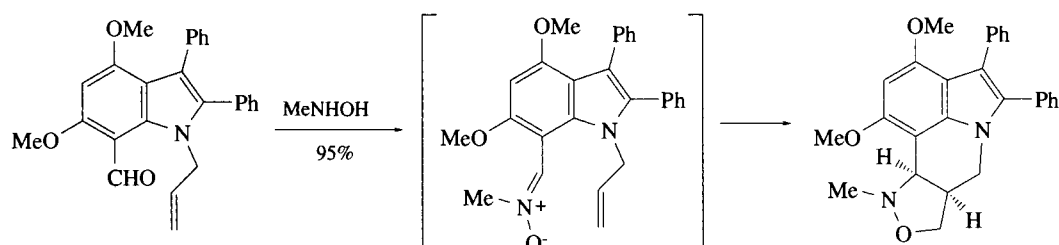


Aldehydes and ketones also provide a range of possibilities for cyclization on to adjacent functional groups on a ring. For example, the carbene generated from a tosylhydrazine sodium salt can be inserted into the double bond of an adjacent allyl group <89CC186, 89JCS(P1)2449, 89JCS(P1)2455> (see Section 2.02.1.7 and Equations (66) and (67)).

Nitrones generated from indole aldehydes readily undergo 1,3-dipolar cycloadditions to adjacent allyl double bonds. Pyrrolo[1,2-*a*]indoles are formed from 1-allylindole-2-carbaldehydes (Scheme 137) <89TL1421, 93AJC603>, whilst pyrrolo[3,2,1-*ij*]quinolines are obtained from 1-allylindole-7-carbaldehydes (Scheme 138) <93AJC843>.



Scheme 137



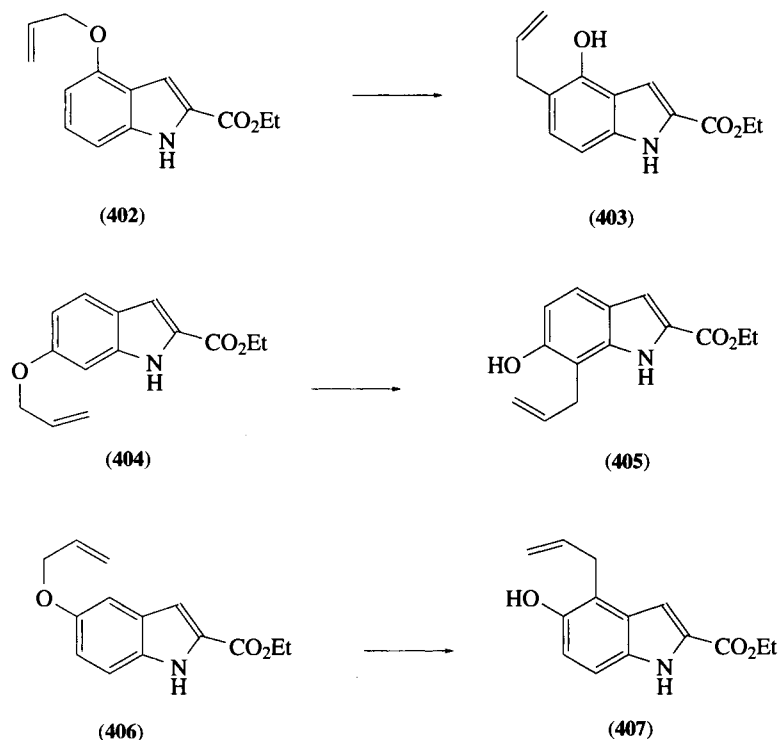
Scheme 138

2.02.3.6 N-Linked Substituents

Total syntheses of the makaluvamine group of tetrahydropyrroloquinoline alkaloids have made use of the cyclization of 4-aminoindole-3-acetic acid derivatives to lactams, which have been further transformed by a sequence of reduction and oxidation into iminoquinones <91CL1785, 94TL917>.

2.02.3.7 Hydroxy and Alkoxy Groups

Work in the 1980s, on the Claisen rearrangement of allyloxy indoles shows the regioselective rearrangement of the 4-allyloxy derivative (402) to the 5-allyl-4-hydroxyindole (403) and of the 6-allyloxy derivative (404) to the 7-allyl-6-hydroxyindole (405) (Scheme 139) <84JCS(P1)1333>. The latter transformation has been used in the synthesis of the alkaloid murrayaquino-B <85CC1391>. These results complement earlier observations of the rearrangement of 5-allyloxy indoles (406) to the 4-allyl-5-hydroxyindoles (407) (Scheme 139) <73BSF2046>.



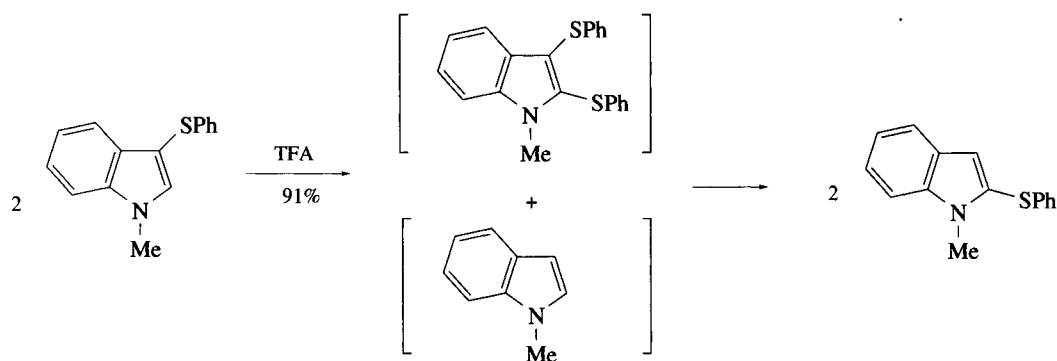
Scheme 139

The photo-Fries rearrangement of 5- and 6-acetoxyindoles has been studied. Although mixtures of products are obtained, the major products are the 4-acetyl-5-hydroxyindole and the 7-acetyl-6-hydroxyindole, respectively $\langle 89\text{TL}6483 \rangle$.

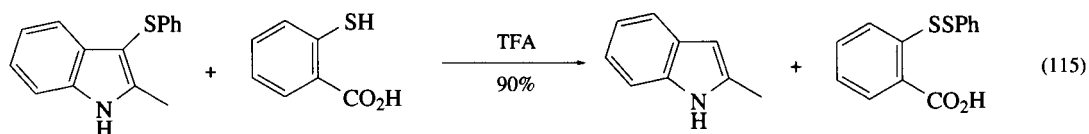
Oxidation of the 4-, 6-, and 7-hydroxyindoles has been carried out using sodium periodate and leads to complex mixtures of products including 3,4'- and 4,4'-biindolyls from 7-hydroxyindole $\langle 89\text{T}6749 \rangle$.

2.02.3.8 S-Linked Substituents

Indole 4-thioethers have been used as suitable substrates in intramolecular cyclizations leading to the chuangxinmycins $\langle 93\text{TL}489 \rangle$. The acid-catalyzed rearrangement of 1-methyl-3-thiophenylindole to 1-methyl-2-thiophenylindole has been shown to proceed by disproportionation to the 2,3-disulfide and 1-methylindole (Scheme 140) $\langle 89\text{CC}63, 92\text{JOC}2694 \rangle$. Similar treatment of 2-methyl-3-thiophenylindole with TFA, in the presence of thiosalicylic acid as a trapping agent for liberated thiophenol, gives a high yield of 2-methylindole in what is a useful nonreductive desulfenylation (Equation (115)) $\langle 94\text{JOC}6372 \rangle$.

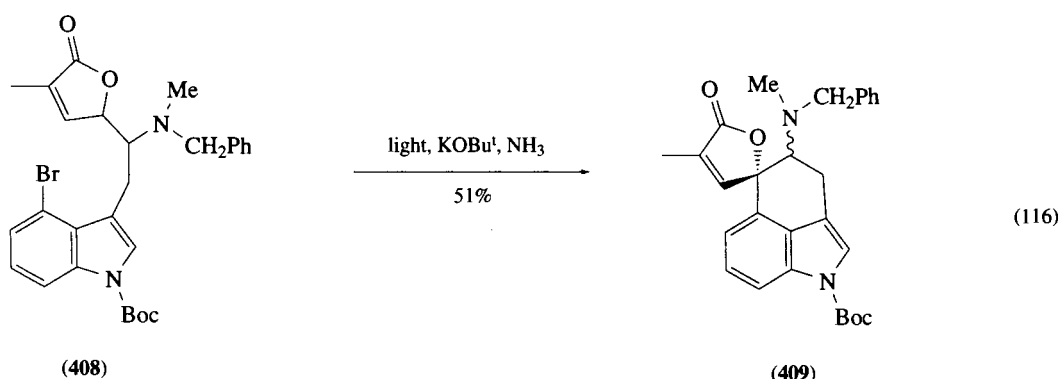


Scheme 140



2.02.3.9 Halogeno Groups

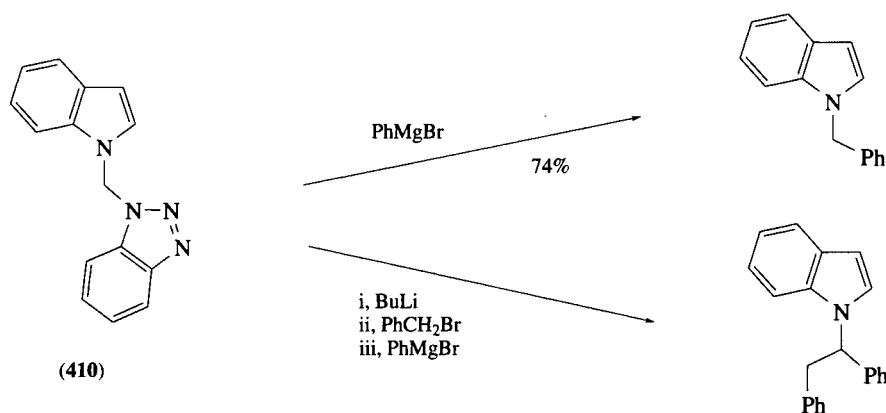
Most of the reactions of halogeno groups on pyrroles and indoles involve the formation of organometallic species. This chemistry has been dealt with in Section 2.02.1.3.7. Indolyl radicals are postulated in some photochemical reactions of chloro- and bromoindoles. Methyl 4-chloro-3-indolylacetate gives the dechlorinated product in good yield on irradiation in methanol <89JA8060>. Irradiation of the 4-bromo-3-substituted indole (**408**) under basic conditions gives the rugulovasine derivative (**409**), probably by an $S_{RN}1$ mechanism (Equation (116)) <93JA10450>.



2.02.4 REACTIVITY OF SUBSTITUENTS ATTACHED TO THE RING NITROGEN ATOM

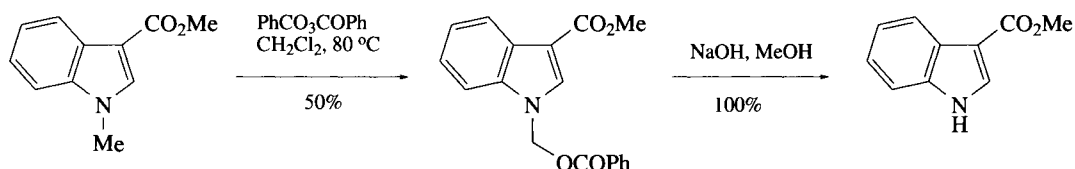
2.02.4.1 Alkyl and Substituted Alkyl Groups

Both pyrrole and indole can be substituted cleanly on nitrogen by the use of 1-(benzotriazol-1-ylmethyl) derivatives. Reaction of these compounds with Grignard reagents gives substitution products. For example, phenylmagnesium bromide reacts with the indolyl derivative (**410**) to give 1-benzylindole (Scheme 141) <93T2829>. The process can be extended by a sequence of lithiation and alkylation prior to Grignard attack to give branched alkyl products.

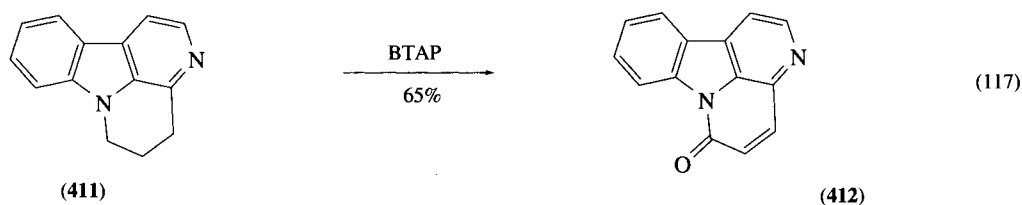


1-Methylindole-3-carboxylic esters can be deprotected at nitrogen by an oxidative process in which the methyl group is attacked by dibenzoyl peroxide. The resultant benzoyloxymethyl derivative can

be hydrolyzed by sodium hydroxide to deliver the deprotected indole (Scheme 142) <86H(24)2791>. Various canthines such as the simplest (**411**) can be oxidized regioselectively to the respective canthine-6-one (**412**) using the phase-transfer reagent benzyltriethylammonium permanganate (BTAP); the oxidation is sequential from the alcohol to the ketone and then to the final product (Equation (117)) <94TL1485>.

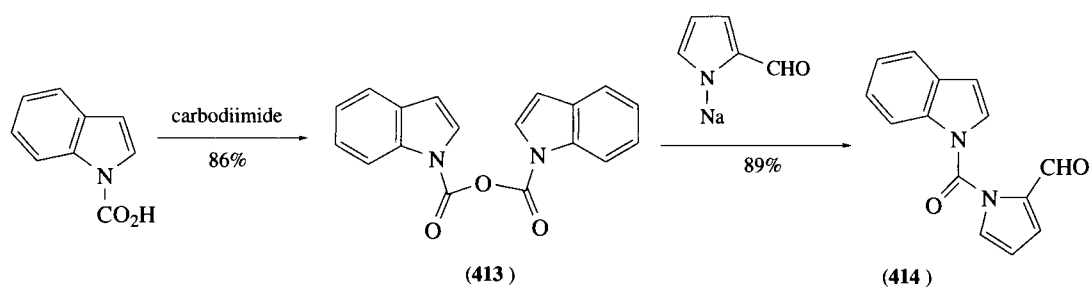


Scheme 142

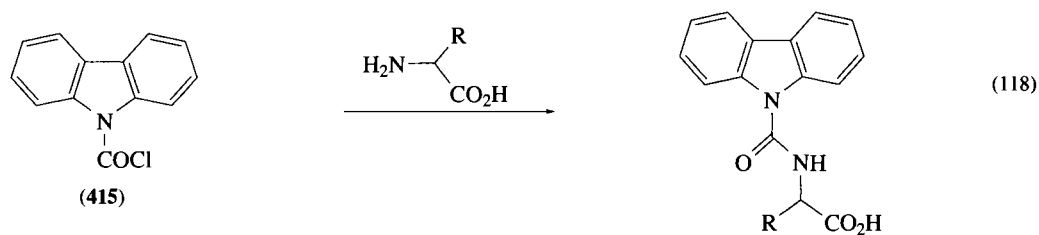


2.02.4.2 Carboxylic Acids and Their Derivatives

Indole-1-carboxylic acid can be converted by treatment with a carbodiimide to the anhydride (**413**), which can undergo reaction with amines or the sodium salts of phenols, thiophenols, pyrroles, and indoles. For example, it reacts with the sodium salt of pyrrole-2-carbaldehyde to give the product (**414**) (Scheme 143) <87JOC3934>. *N*-Chloroformylcarbazole (**415**), prepared by the reaction of carbazole with phosgene, has been used effectively for the fluorogenic labeling of amino acids (Equation (118)) <90TL1455>.

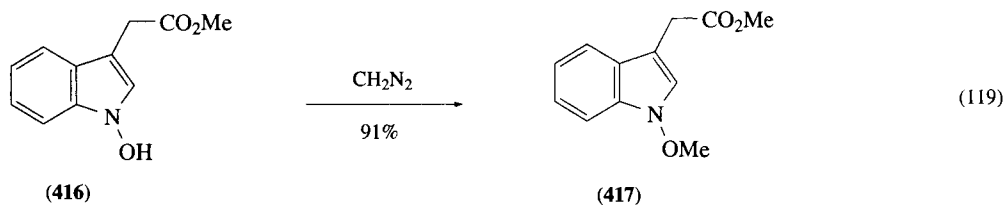


Scheme 143



2.02.4.3 Hydroxy and Alkoxy Groups

N-Hydroxyindoles are receiving increasing attention, but mainly for their reactions on the indole ring. However, the isolable but unstable 1-hydroxyindole-3-acetic ester (**416**) can be converted by treatment with diazomethane into the stable 1-methoxy analog (**417**) (Equation (119)) <91CPB1905>.



2.03

Pyrroles and their Benzo Derivatives: Synthesis

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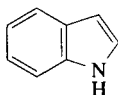
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2.03.1 INTRODUCTION AND OVERVIEW

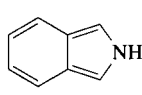
This chapter deals with the synthesis and synthetic elaboration of pyrrole (1) and its benzo derivatives, namely indole (2), isoindole (3) and carbazole (4).



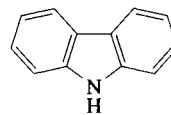
(1)



(2)

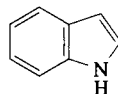
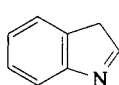


(3)

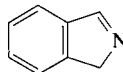
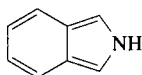


(4)

These compounds are classified as π -excessive heteroaromatic molecules. As a π -excessive molecule, pyrrole is reactive toward electrophiles and oxidants. The same is true of indole although it is less reactive than pyrrole towards electrophilic substitution. The partial rate factors for electrophilic substitution (relative to benzene) for pyrrole and indole have been estimated to be 10^{18} and 10^{13} , respectively $\langle 68\text{JCS(B)}392, 87\text{JCS(P2)}591 \rangle$. Recently developed photochemical methods for generation of the 3*H*-tautomer of indole have allowed its properties to be determined. The 3*H*-tautomer is persistent in diethyl ether at -100°C , but is converted to the 1*H*-tautomer at -20° (Equation (1)). The tautomerization is catalyzed by both protons and weak acids so the lifetime of the 3*H*-tautomer is extended in the presence of base. The estimated half-life in water at 25°C , pH 9, is 100 s $\langle 94\text{AG(E)}1153 \rangle$. Isoindole is generally considered to be somewhat less stable than indole because the carbocyclic ring is quinoid in character. It has been argued that isoindole should be considered to be a pyrrole ring substituted by a 2,4-butadienyl unit $\langle 77\text{JA}8248 \rangle$. The carbocyclic ring of 2-methylisoindole shows substantially more bond length alternation than does indole $\langle 84\text{UKZ}988 \rangle$. 2*H*-Isoindole is in equilibrium with the 1*H*-tautomer (Equation (2)). Although the parent compound exists as the 2*H*-tautomer, reactions can occur readily through the 1*H*-tautomer.



(1)



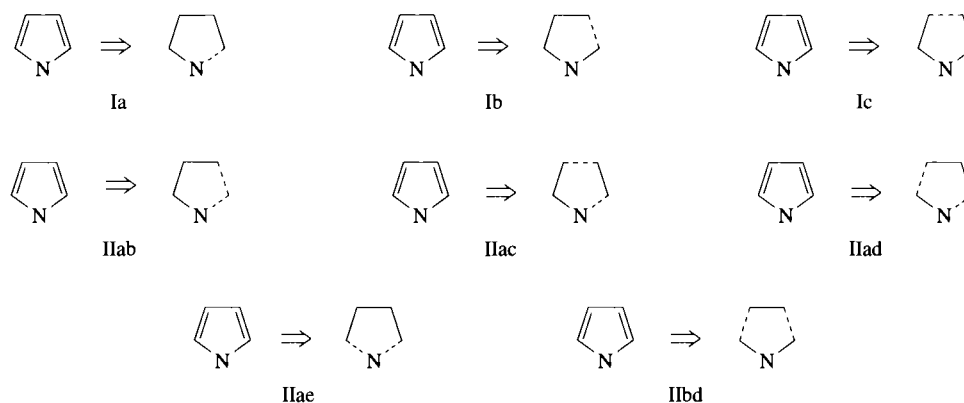
(2)

Isoindole is also more reactive toward oxygen than either pyrrole or indole. Indeed, its rate of deuterium exchange at C1 is 10^4 that of indole at C3 $\langle 87\text{JCS(P2)}591 \rangle$. The position of the 1*H*–2*H* equilibrium is substituent dependent. Donor substituents, for example 3-ethoxy, stabilize the 1*H*-tautomer and electron-attracting groups, for example 1-ethoxycarbonyl, favor the 2*H*-tautomer $\langle 75\text{S}252, 88\text{CB}243 \rangle$. Carbazole is the least reactive of the group, since it presents only carbocyclic sites. Chapter 2.10 of this volume discusses the reactivity of these molecules in more detail. Here the focus will be on the most useful methodology for formation and synthetic elaboration of these rings.

The chemistry of pyrroles and indoles has been actively and continuously investigated since the early days of organic chemistry and reviews and books have appeared regularly. Among the general references on pyrroles are a monograph published in 1977 $\langle \text{B-77MI } 203\text{-}01 \rangle$ and a two-part volume in *Chemistry of Heterocyclic Compounds* $\langle 90\text{HC(48-1)}1, 92\text{HC(48-2)}1 \rangle$. Indole chemistry was last comprehensively reviewed in a four-part volume in the *Chemistry of Heterocyclic Compounds* published between 1972 and 1983 $\langle 72\text{HC(25-1)}1, 72\text{HC(25-2)}1, 79\text{HC(25-3)}, 83\text{HC(25-4)}1 \rangle$. A monograph was published in 1970 $\langle \text{B-70MI } 203\text{-}01 \rangle$. The chemistry of isoindoles has been less extensively investigated since isoindole itself was not synthesized until 1972 $\langle 72\text{CC}393 \rangle$. Two reviews of isoindole appeared in 1981 $\langle 81\text{AHC(29)}341, 81\text{RCR}1087 \rangle$. A review of carbazole chemistry, including synthesis, was published in 1984 $\langle 84\text{AHC(35)}83 \rangle$. The first edition of *Comprehensive Heterocyclic Chemistry* summarized the area up to the early 1980s $\langle 84\text{CHEC(4)}1 \rangle$. Annual reviews of pyrrole and indole chemistry have appeared since 1989 $\langle 89\text{PHC(1)}111, 90\text{PHC(2)}70, 91\text{PHC(3)}90, 92\text{PHC(4)}81, 93\text{PHC(5)}110 \rangle$. More specialized reviews are noted later in the text.

Synthetic methods, particularly older ones, are frequently referred to as name reactions. While useful to the well-versed, this method provides no organizational framework and is not very helpful for new entrants in the field. In this chapter, I have adopted a systematic organization based on the structural relationship between the product and the starting material. Scheme 1 shows the des-

ignation of cyclization types which is based on the number and location of the bonds formed in the reaction. Solid lines represent the fragments from which the ring is formed and dotted lines represent the bonds formed during synthesis. This system is not without its ambiguities, since it depends on a specification of the 'starting material'. I have take the starting material to be any stable compound which can, if desired, be isolated and purified. Table 1 lists the classification for some of the well-established name reactions.



Scheme 1

Table 1 Correspondence with name reactions.

Name reaction	Category	Section
Leimgruber-Batcho indole synthesis	Ia	2.03.2.1
Reisser indole synthesis	Ia	2.03.2.1
Hemetsberger indole synthesis	Ia	2.03.2.1
Madelung indole synthesis	Ib	2.03.2.2
Bischler indole synthesis	Ic	2.03.2.3
Knorr pyrrole synthesis	IIac	2.03.2.5
Hantzsch pyrrole synthesis	IIac	2.03.2.5
Fischer indole synthesis	IIac	2.03.2.5
Gassman indole synthesis	IIac	2.03.2.5
Paal-Knorr pyrrole synthesis	IIae	2.03.2.7

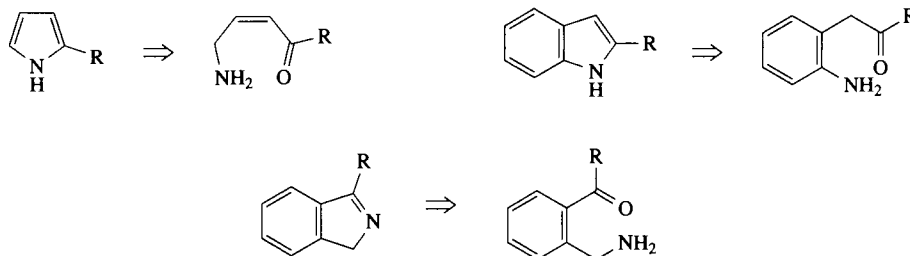
The classical pyrrole and indole syntheses usually involve polar cyclizative condensations in which nucleophilic and electrophilic centers react through one or more addition-elimination steps. The nucleophilic component is usually a nitrogen or the nucleophilic carbon of an enol, enolate or enamine. The electrophilic component is usually a carbonyl or imine group or an electron-deficient carbon-carbon double bond. These reactions often proceed by a series of equilibrium steps which are driven forward by the aromatization of the final product. Other reactions which are prominent pyrrole and indole syntheses are concerted cycloadditions and processes based on organometallic intermediates. We will discuss the mechanisms of the reactions only to the extent necessary to understand the synthetic aspects of the reaction, including such issues as range of applicable substituents and regioselectivity. Reactions have been chosen for inclusion on the basis of the author's impression of generality and utility, as judged primarily from frequency of use in the literature. Newer methods which appear to have promise of generality are also included. Availability of starting materials is a major practical aspect in the choice of a synthesis and some attention has been given to this consideration.

2.03.2 METHODS OF SYNTHESIS INVOLVING RING FORMATION

2.03.2.1 Category Ia Cyclizations

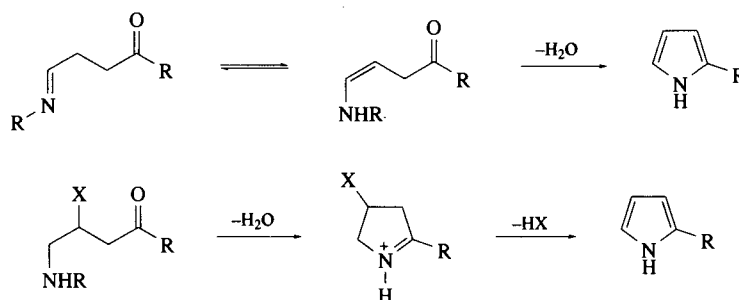
There are three prominent types of reactions which fall in the Ia classification. These are cyclizative condensations, metal-mediated cyclizations and nitrenoid insertion reactions. Scheme 2 shows

retrosynthetic transformations corresponding to pyrroles, indoles and isoindoles for the first type of reaction.



Scheme 2

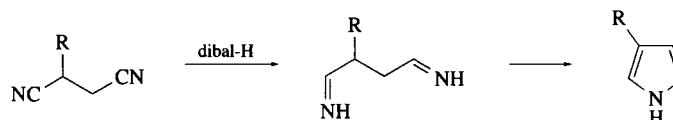
The mechanistic basis of the cyclizative condensations is the elimination of water from a carbinolamine resulting from an amine-carbonyl addition reaction. The amino group is often generated *in situ* from a nitro, cyano or other nitrogen-containing functional group. Because of the facility of amine-carbonyl addition reactions and the potential for aromatization by dehydration, any reaction sequence that generates a γ -amino-ketone is potentially capable of forming a pyrrole ring. Direct aromatization is possible if the intervening chain contains a double bond or a potential leaving group.



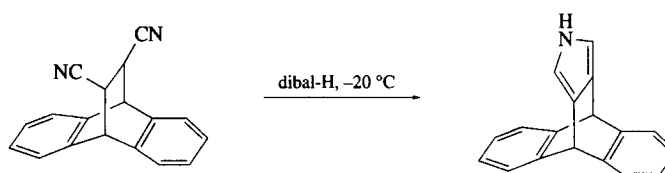
Scheme 3

Any number of synthetic schemes can be imagined which might provide such pyrrole precursors. The eventual effectiveness of any specific method, of course, depends on the yield that can be achieved and that is frequently a matter of adjusting reaction conditions to favor pyrrole formation relative to competing processes.

Substituted succinonitriles can be converted to 3-substituted pyrroles by reduction with dibal-H (84TL1659). dibal-H Reductions lead to diimine intermediates which undergo cyclization and aromatization (Scheme 4). This reaction was used successfully in the synthesis of unusual pyrroles which were used to prepare sterically shielded porphyrins (Equation (3)) (94AG(E)889).

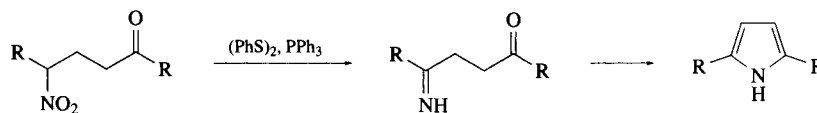


Scheme 4



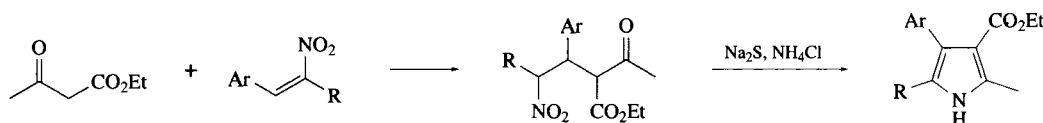
(3)

γ -Nitroketones are reduced to pyrroles by a mixture of diphenyl disulfide and triphenylphosphine (<86JCS(P1)2243>). Since γ -nitroketones are accessible by Michael additions of nitroalkanes to enones, this is a potentially versatile method (Scheme 5).



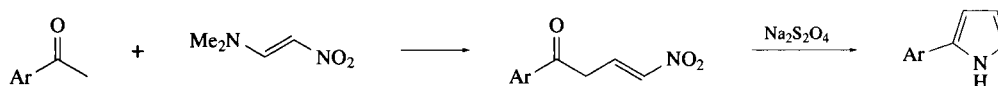
Scheme 5

The Michael adducts of β -ketoesters and β -nitrostyrenes can be reduced to 4-arylpyrrole-3-carboxylate esters (Scheme 6) (<85LA239>). It has been suggested that cyclization may occur at the oxime reduction level.



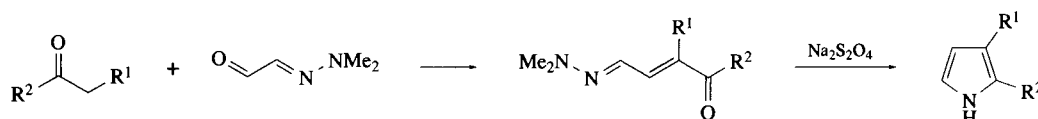
Scheme 6

Condensation of acetophenones and β -dimethylaminonitroethene gives γ -nitroenones which can be reductively cyclized to 2-arylpyrroles (Scheme 7) (<74CB1499>).



Scheme 7

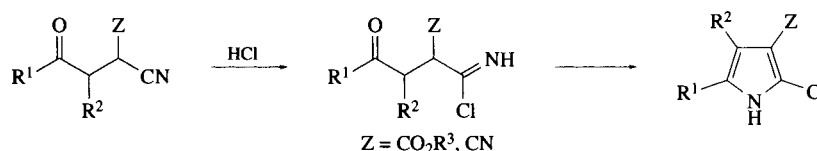
A related process involves the condensation of ketones with the mono-*N,N*-dimethylhydrazone of glyoxal. Base-catalyzed condensation gives the monohydrazone of a 2-ene-1,4-dione and reductive cleavage of the N—N bond affords 2,3-disubstituted pyrroles (Scheme 8) (<77CB491>).



Scheme 8

Each of these reactions is an example of reductive generation of an intermediate which can cyclize and aromatize by elimination under the reaction conditions.

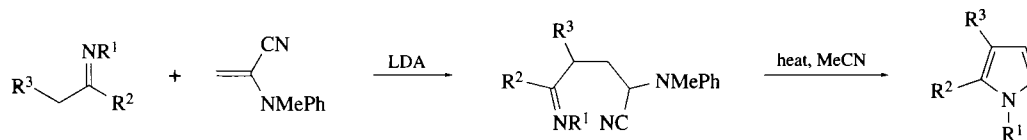
β -Ketonitriles can be cyclized to 2-chloropyrroles by HCl. This reaction presumably proceeds through an imino chloride and has been applied only to pyrroles with EW-groups in position 3 of the pyrrole ring (<89T6439, 94TL5989>). Starting with the alkylation of malononitrile by substituted phenacyl bromides, this procedure has been used to make a number of 2-chloro-3-cyano-5-arylpyrroles (Scheme 9) (<92MI 203-01>).



Scheme 9

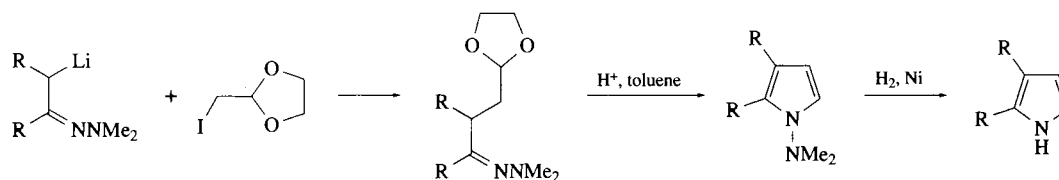
A variety of 1,2,3-trisubstituted pyrroles have been prepared using 2-(*N*-methylanilino) acrylonitrile as a starting material (<84S610>). The carbon chain is established by a conjugate addition of a lithioenamine. The cyclization step is conducted by heating in acetonitrile and presumably

proceeds by formation of an iminium group by elimination of cyanide from the α -aminonitrile. It is also possible to obtain 1,2,3,5-substituted pyrroles if the α -aminonitrile is alkylated before the cyclization (Scheme 10).



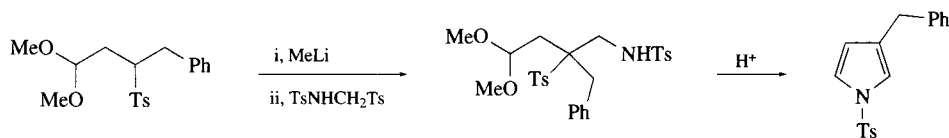
Scheme 10

Alkylation of α -lithio-*N,N*-dimethylhydrazones with 2-(iodomethyl)-1,3-dioxolane provides masked γ -hydrazonoaldehydes. Cyclization occurs after acid-catalyzed removal of the dioxolane protecting group and the *N*-unsubstituted pyrrole can be obtained by hydrogenolysis of the *N,N*-dimethylamino substituent (Scheme 11) <88JHC1135>.



Scheme 11

N-(*p*-Toluenesulfonylmethyl)-*p*-toluenesulfonamide functions as a Mannich-type electrophile which can aminomethylate enolates and enamines. When applied to β -sulfonylacetals a protected γ -aminoaldehyde is created. In this synthesis the sulfonyl substituent functions as a leaving group after cyclization (Scheme 12) <86CL1033>.

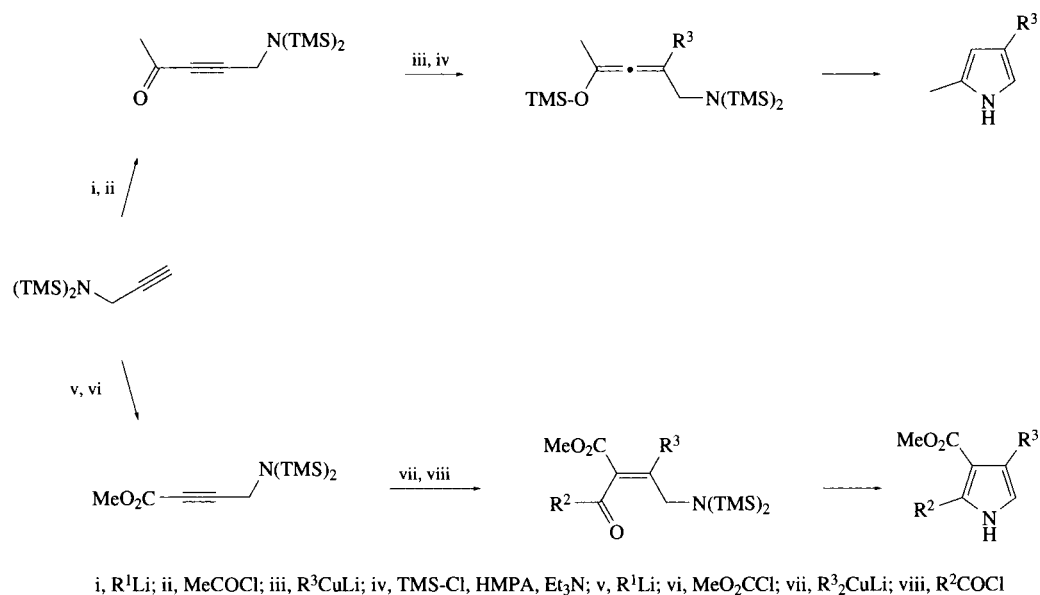


Scheme 12

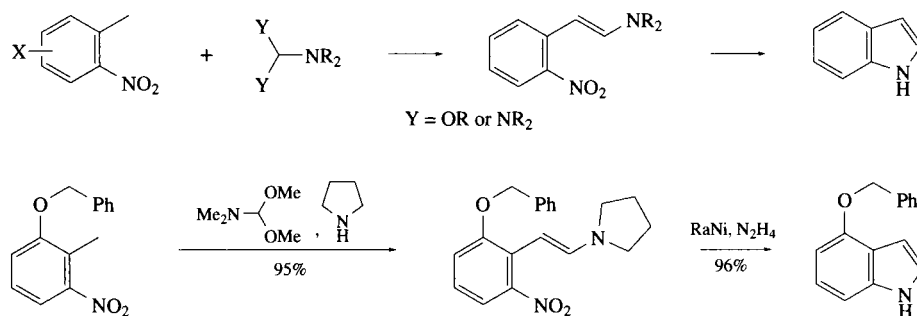
Scheme 13 shows additional examples of constructions of four-carbon chains functionalized appropriately for condensation to pyrroles. *N,N*-bis(trimethylsilyl)propargyl amines can be converted to ketones or esters by lithiation and acylation. These compounds then undergo cuprate addition. The resulting allenyl silyl ethers cyclize to pyrroles on treatment with acid <93T4603>.

Condensation reactions between amino groups and carbonyl or imine groups are important in indole synthesis. For indoles the required starting materials are *o*-aminobenzyl or *o*-nitrobenzyl ketones and aldehydes. One of the most useful syntheses of this type is the Leimgruber-Batcho synthesis which is a two-stage process involving condensation of an *o*-nitrotoluene with a formamide acetal, followed by an *in situ* reduction. The reaction is compatible with a wide variety of ring substituents <84H(22)195>. The basis of the Leimgruber-Batcho method is the weak acidity of the methyl group induced by the nitro substituent, the stabilization present in the enamine intermediate, and the availability of reducing conditions which are compatible with the cyclization. The original reaction conditions involved heating the *o*-nitrotoluene and *N,N*-dimethylformamide dimethyl acetal and distilling off methanol to drive the reaction to completion. An improved procedure includes a slight excess of pyrrolidine which exchanges with the dimethylamino substituent (Scheme 14) <82JHC845, 85OS214>.

An alternative procedure substitutes tripiperidinomethane for the DMF acetal. Condensation is done at 110°C with piperidine being removed by distillation at reduced pressure <83TL4561>. tris(Dimethylamino)methane can also be used <81H(16)1119>. The cyclization presumably occurs through an iminium ion derived from the *o*-aminophenyl enamine. The most commonly used reduction conditions are Raney nickel and hydrazine or hydrogenation over Pd/C. Other reductants which have been used include TiCl_3 <87JA3378> and iron powder with silica in acetic acid <87JHC1499>.



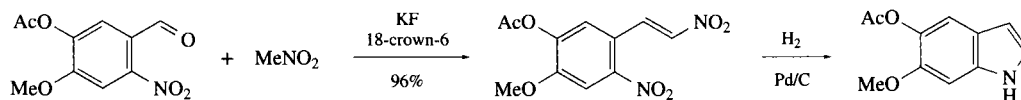
Scheme 13



Scheme 14

Use of Zn/NH_4Cl for reduction gives good yields of 1-hydroxyindoles, evidently by cyclization at the hydroxylamine stage. The *N*-hydroxyindoles can be alkylated or acylated (81H(16)1523, 84JCR(S)101, 86CPB677).

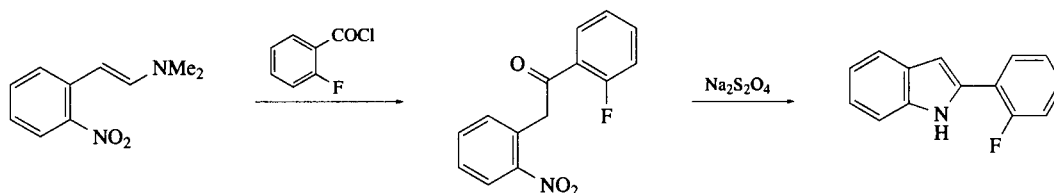
o-Nitrobenzaldehydes are the starting materials in a classical route to indoles which proceeds via *o*, β -dinitrostyrenes. Like the Leimgruber–Batcho method, this reaction is used primarily to make indoles having only carbocyclic substituents. The condensation of the aldehyde with nitromethane is usually done under alkaline conditions. Potassium fluoride/18-crown-6 in propanol was found useful for condensation of acetoxy-substituted aldehydes which would be susceptible to solvolysis under the normal conditions (Scheme 15) (87JHC941).



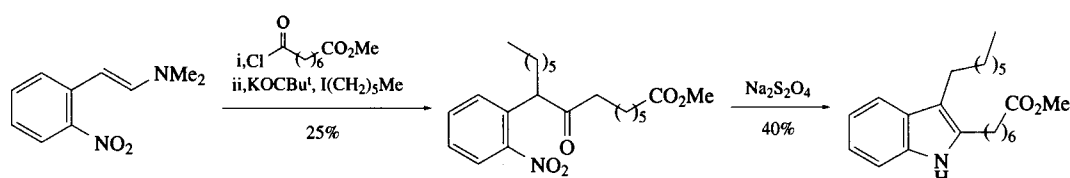
Scheme 15

The reduction can be done with iron powder or by catalytic hydrogenation or hydrogen transfer (89H(29)415, 93SL755). Inclusion of silica has been recommended for the iron reductions (83JOC3347, 85JMC1273). There is little evidence on the reduction stage at which the cyclization occurs. *o*-Nitrophenylacetoximes have occasionally been observed as by-products (60JOC1542, 90JCR(S)116).

There are several procedures by which *o*-nitrobenzyl ketones can be obtained. Enolates and enamines can be acylated with *o*-nitrophenylacetyl chloride. For example, the enamine intermediates from the Leimgruber-Batcho procedure can be acylated prior to reduction to give 2-substituted indoles (Scheme 16) <74JHC291>. Acylation followed by alkylation at the benzylic position leads to 2,3-disubstituted indoles (Scheme 17) <91G499>.

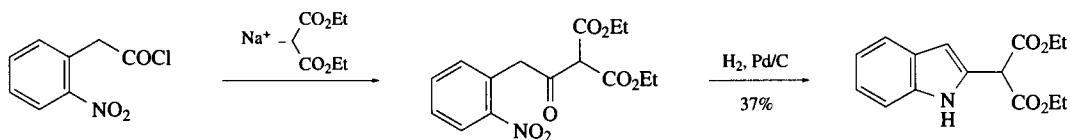


Scheme 16

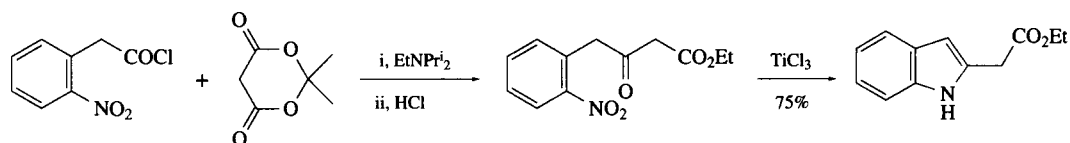


Scheme 17

The acylation of enolates of ethyl acetoacetate (Scheme 18) <76CB3282> and Meldrum's acid (Scheme 19) <90JCS(P1)673> by *o*-nitrophenylacetyl chloride leads to indole precursors.

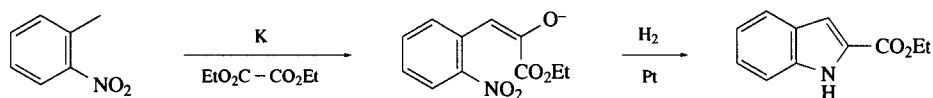


Scheme 18



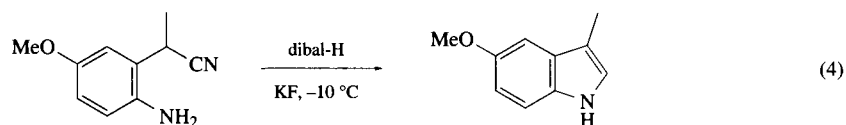
Scheme 19

The classical Reissert synthesis of ethyl indole-2-carboxylate involves acylation of the anion *o*-nitrotoluene by diethyl oxalate (Scheme 20) <73OSC(5)567>.

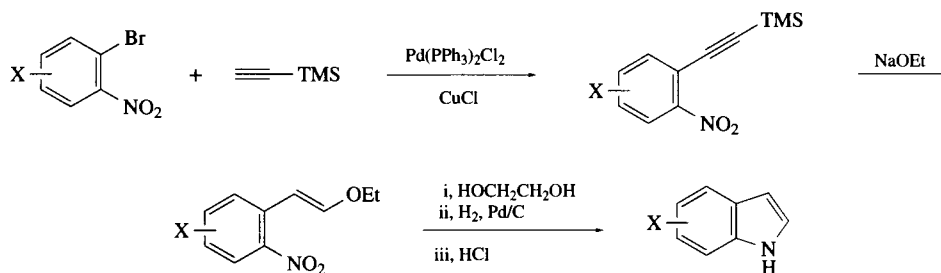


Scheme 20

Indoles have also been obtained from *o*-nitro and *o*-aminophenylacetonitriles by partial reduction of the cyano group (Equation (4)) <91T7195, 94SC839>.

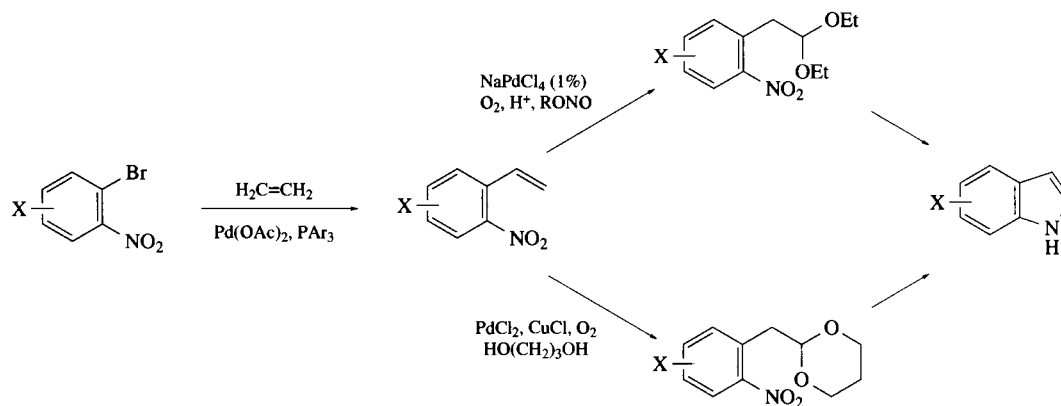


Several versions of palladium-catalyzed coupling reactions have been used to convert *o*-nitroaryl halides to *o*-nitrophenylacetaldehydes or *o*-nitrobenzyl ketones. Coupling with trimethylsilylethyne gives aryl alkynes which can be converted to *o*-nitrophenylacetaldehyde enol ethers by sodium ethoxide (Scheme 21) <86CPB2362>.



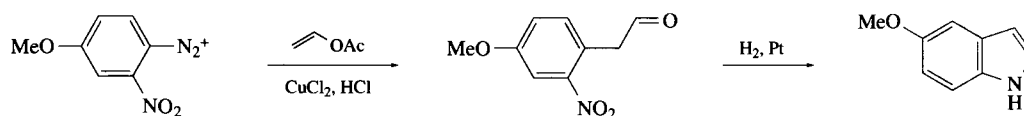
Scheme 21

o-Nitrophenylacetaldehyde acetals can also be obtained by Wacker oxidation of *o*-nitrostyrenes obtained by the Heck reaction <92JHC1625>. These reactions require conditions which favor oxygenation of the terminal carbon of the nitrostyrene. The Wacker oxidation can also be carried out in the presence of 1,3-propanediol, with the *o*-nitrophenylacetaldehyde being isolated as the dioxane derivative (Scheme 22) <89JHC1405>.



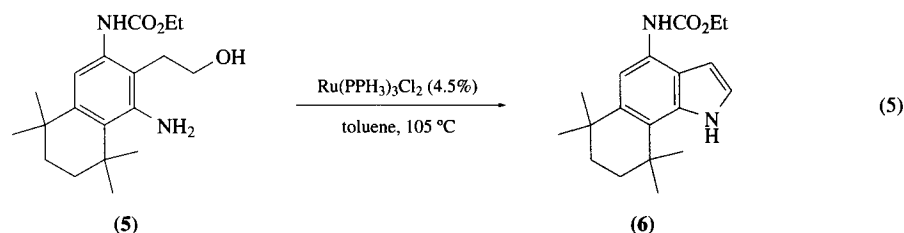
Scheme 22

Meerwein arylation of vinyl acetate provides an alternative route to *o*-nitrophenylacetaldehydes. Reduction with iron/acetic acid provides indoles in 50–90% yield (Scheme 23) <83JOC2066>.

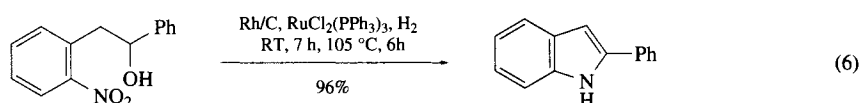


Scheme 23

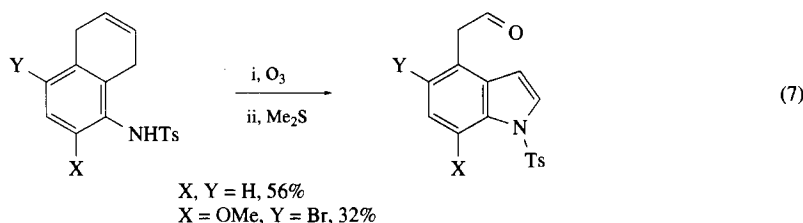
Another method for obtaining *o*-aminobenzyl ketones or aldehydes is to introduce the carbonyl group into an *o*-substituent by oxidation. 2-(*o*-Aminophenyl)ethanol derivatives yield indoles in the presence of Ru(PPh3)2Cl2 <90JOC580>. The reaction involves evolution of hydrogen. The method has been used successfully to synthesize substituted indoles and was applied to the substitution pattern found in the teleocidin analog (6) (Equation (5)) <91JOC4706>.



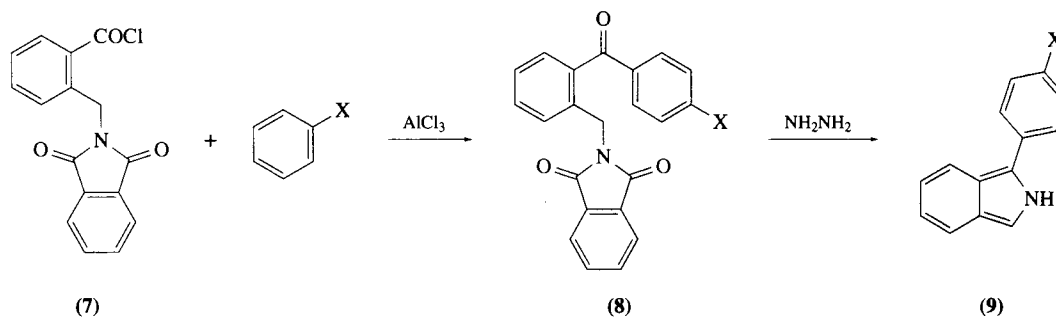
The reaction can also be carried out using 2-(*o*-nitrophenyl)ethanol derivatives as starting materials. A binary catalyst containing Rh/C and Ru (PPh₃)₂Cl₂ under a hydrogen atmosphere provides a means for both the reductive generation of the amino group and the oxidative cyclization (Equation (6)) <90JOC580>.



Carbonyl groups can also be introduced by ozonolysis. This approach has been used to obtain 4-substituted indoles starting with 1,4-dihydronaphthalenes (Equation (7)) <76LA1475, 85JA2943>.

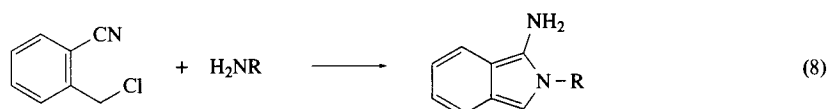


The methodology for synthesis of isoindoles is not as thoroughly explored as that of pyrroles and indoles and there is less information about the generality of specific methods. Nevertheless, reaction patterns for cyclizative condensation are similar to those for pyrroles and indoles. Generation of *o*-acylbenzylamines generates isoindoles. Several 1-arylisoindoles were prepared in two steps from (7) by a Friedel-Crafts reaction followed by release of the amino group from the phthalimide by hydrazinolysis (Scheme 24) <64JA4152>.

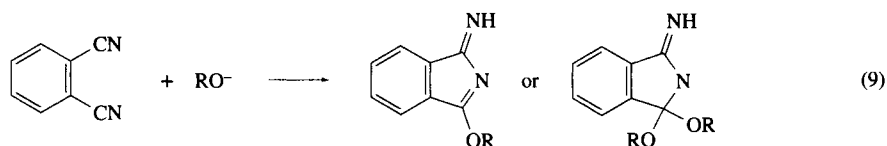


Scheme 24

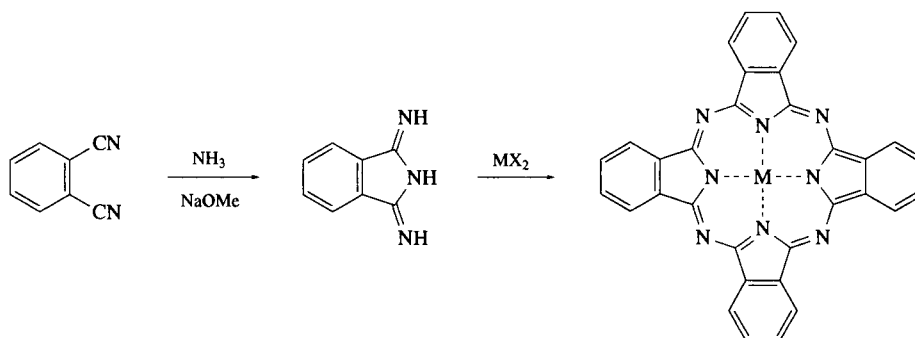
Reaction of *o*-cyanobenzyl chloride with amines gives 1-aminoisoindoles (Equation (8)) <71UKZ916>.



Phthalonitrile can also be cyclized by reaction with alkoxide ions with the product being either a 1,1-dialkoxy-3-imino-dihydroisoindole or 1-alkoxy-3-iminoisoindole (Equation (9)) <90JCR(S)322>.

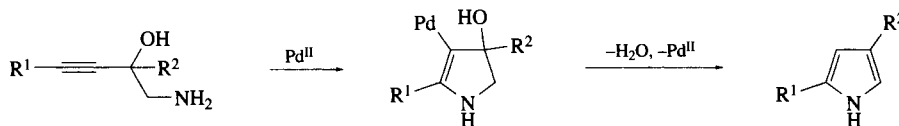


The isoindole ring is present in the phthalocyanine complexes and one method for their preparation is from 1,3-diminodihydroisoindole which is prepared by cyclization of *o*-phthalonitrile (Scheme 25) <83JA1539>.

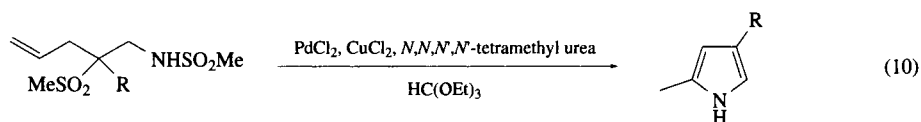


Scheme 25

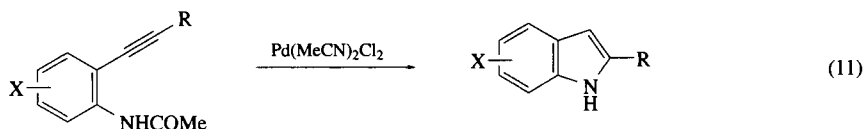
Pyrrole and indole rings can also be constructed by intramolecular addition of nitrogen to a multiple bond activated by metal ion complexation. 1-Aminomethyl-1-alkynyl carbinols, which can be obtained by reduction of cyanohydrins of acetylenic ketones, are cyclized to pyrroles by palladium(II) salts. In this reaction the palladium(II)-complexed alkyne functions as the electrophile with aromatization involving elimination of palladium(II) and water (Scheme 26) <81TL4277>. δ,ϵ -Unsaturated methanesulfonamides can also be cyclized to pyrroles by palladium(II), with the β -sulfonyl substituent being eliminated to complete aromatization (Equation (10)) <89CL737>.



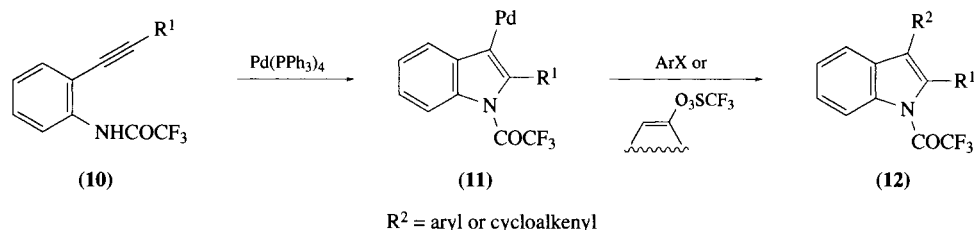
Scheme 26



The conversion of *o*-aminophenyl alkynes to indoles has been used occasionally in synthesis <66JOC4071, 84JA4218, 85TL5963>, but the practicalities of this method have been enhanced by improved methods for synthesis of the required starting materials. *o*-Acetamidophenylalkynes can be cyclized to indoles using PdCl_2 as a catalyst. The alkynes can be prepared by copper-catalyzed coupling of terminal alkynes with *o*-bromoacetanilide <89JOC5856>. The required arylalkynes can also be prepared by coupling of aryl triflates with tri-*n*-butylstannylalkynes <89TL2581>. Another route to the starting materials involves palladium-catalyzed coupling of 2-ethynylaniline with vinyl or aryl triflates, (Equation (11)) <89TL2581>.

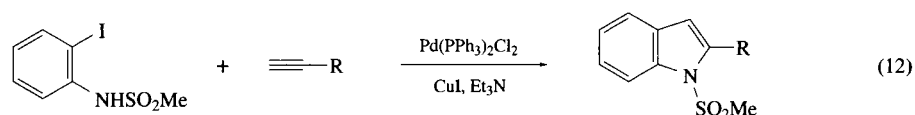


Indolylpalladium intermediates can be intercepted by aryl halides and vinyl triflates. Using 2-(trifluoroacetamido)phenylalkynes as reactants, palladium-catalyzed cyclization generates intermediate **(11)**. In the presence of an aryl halide or vinyl triflate coupling occurs to provide a 2,3-disubstituted indole (**(12)**) (Scheme 27) (92TL3915).

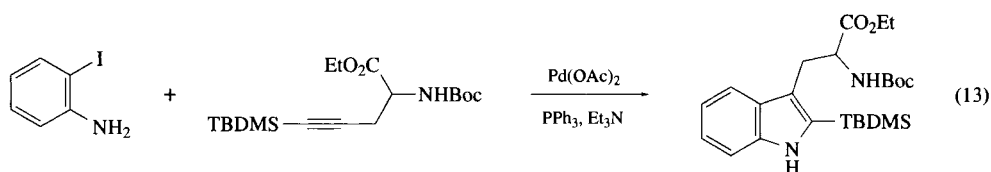


Scheme 27

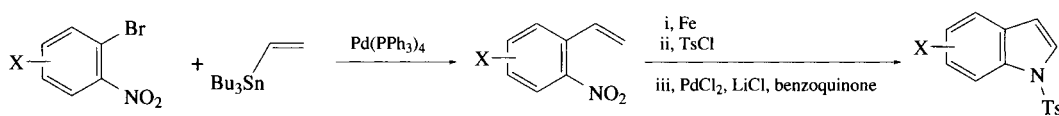
The use of terminal alkynes and *N*-methanesulfonyl-2-iodoaniline in the presence of both copper and palladium permits a one-pot synthesis of 2-substituted indoles (Equation (12)) (88CPB1305).



A procedure for palladium-catalyzed tandem coupling-cyclization has been developed using trialkylsilylalkynes and *o*-iodoaniline (93TL6471). The reaction is carried out as a single-step process using $\text{Pd}(\text{OAc})_2$ as a catalyst (Equation (13)).



Several indoles with carbocyclic ring substituents have been synthesized by palladium-catalyzed cyclization of *N*-(*o*-vinylphenyl)-*N*-arenesulfonamides. The starting materials are prepared by coupling 2-bromonitrobenzene with vinyl-tri-*n*-butylstannane, followed by reduction and sulfonylation (Scheme 28) (88JOC1170).

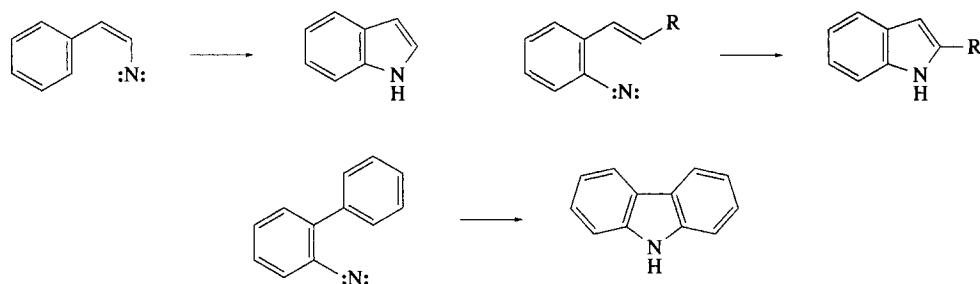


Scheme 28

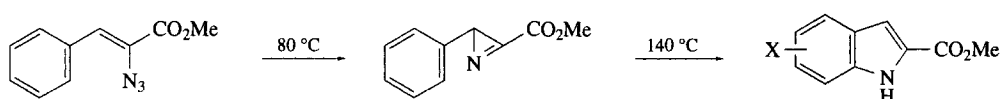
The Ia type classification also applies to a group of reactions attributable to nitrene or related (nitrenoid) intermediates. Although not as prominent as carbonyl condensation reactions, these reactions are especially useful for carbazole synthesis. These are formally insertion reactions but mechanistic scrutiny frequently reveals that the reactions are more complicated than a one step insertion process (Scheme 29).

Probably the most useful of these reactions for indole synthesis is the pyrolysis of β -azidocinnamate esters which was first developed by Hemetsberger (69M1599, 70M161). This reaction is a case where there is indeed a mechanistic complication since by conducting thermolysis of the starting azide at a minimal temperature it is possible to isolate an azirine, which, when heated to a higher temperature, gives the indole (Scheme 30) (85S186).

The β -azidocinnamates are normally obtained by condensation of an α -azidoacetate ester with an aromatic aldehyde. This base-promoted aldol condensation is in competition with decomposition of the enolate by elimination of nitrogen, so use of an excess of the α -azidoacetate and as mild as possible condensation conditions are needed to obtain good yields (84JCS(P)1333). The pyrolysis is normally done simply by heating the azidocinnamate in toluene or xylene and the yields are good.



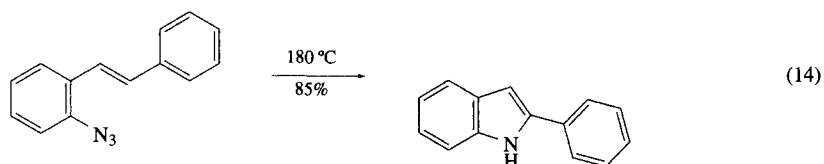
Scheme 29



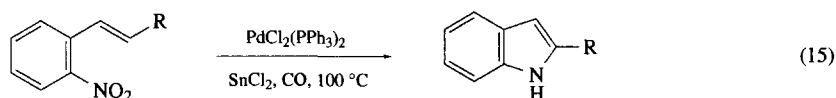
Scheme 30

A variety of carbocyclic substituents are compatible with the reaction <84JCS(P1)1333, 90AJC329, 90CPB2939>.

An alternative possibility places the 'nitrene' on the aromatic ring. *o*-Azidostyrenes can be obtained by diazotization of *o*-aminostyrenes, followed by reaction with azide ion. Pyrolysis leads to the corresponding indole (Equation (14)) <72JOC719>.



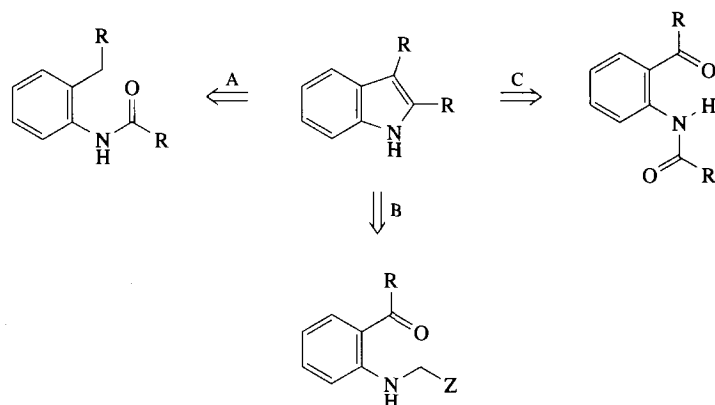
The main disadvantage of this route is the relatively cumbersome syntheses of the required *o*-azidostyrenes. The corresponding *o*-nitrostyrenes are more readily obtained, starting with *o*-nitrobenzaldehyde. *o*-Nitrostyrenes are converted to indoles by heating with triethyl phosphite <65JCS4831, 65JOC3604>. Again there are mechanistic complexities, since there is evidence that at least part of the reaction proceeds through an *N*-hydroxyindole intermediate, but there has been some synthetic application of the reaction <73JOC4074, 77JHC839, 94SC1701>. Other conditions for conversion of *o*-nitrostyrenes to indoles have been developed. Both $\text{Ru}_3(\text{CO})_{12}$ <86CC784, 94JMOC203> and $\text{PdCl}_2/\text{PPh}_3/\text{SnCl}_2$ <94JOC3375> can catalyze reactions in which carbon monoxide acts as the reducing agent. The latter system can provide indoles in 50–75% yield under modest pressure and temperature conditions (Equation (15)).



Both *o*-azidobiphenyls and *o*-nitrobiphenyls are useful precursors of carbazoles. The azidobiphenyls are converted to carbazoles by thermolysis or photolysis <51JA2435, 70JA3103>. 1-Nitrocarbazole, for example, can be prepared from 2-azido-2'-nitrobiphenyl by heating in *o*-dichlorobenzene at 170 °C <73OS829>. Deoxygenation of *o*-nitrobiphenyls by triethyl phosphite is also a convenient synthesis of carbazoles <65JCS4831>. Polyalkyl and polyalkoxycarbazoles have been successfully prepared by this method <68JOC4237, 79S48, 85MM1388, 94JMC2224>. Other examples can be found in a review of carbazole chemistry <84AHC(35)83>.

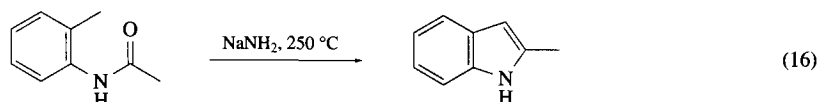
2.03.2.2 Category Ib Cyclizations

Category Ib cyclizations are relatively rare for pyrroles. Three retrosynthetic transformations correspond to indole cyclizations of this type (Scheme 31).

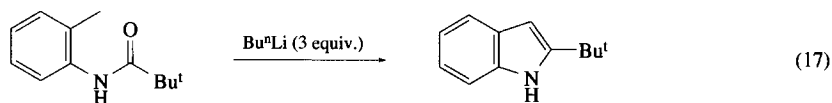


Scheme 31

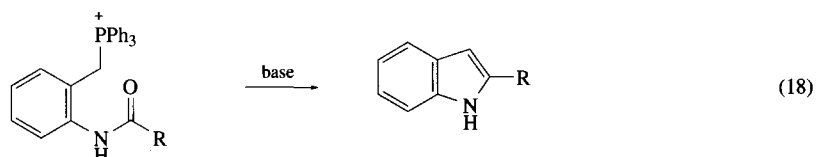
Two of these reactions are, in the most general terms, examples of intramolecular aldol condensations. Path A, which includes the Madelung method and its variants, uses *o*-substituted anilides as starting materials. Path B reverses the electrophilic and nucleophilic components for C2—C3 bond formation. Among the classic indole syntheses corresponding to the first pattern is the Madelung synthesis, exemplified by the preparation of 2-methylindole from *N*-acetyl-*o*-toluidine (Equation (16)) <55OSC597>.



The harsh reaction conditions that are required reflect the low acidity of the methyl substituent and the fact that under the reaction conditions the amide is presumably deprotonated and therefore hardly electrophilic. The Madelung cyclization can be done under much milder conditions by using two equivalents or more of an alkyllithium as the base (Equation (17)) <81JOC4511>.

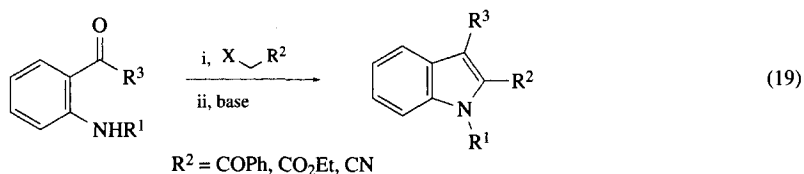


Placing an electron-withdrawing (EW) group at the benzylic position also facilitates cyclization. The triphenylphosphonio substituent converts the reaction to an intramolecular Wittig reaction and yields of 2-substituted indoles are quite good (Equation (18)) <81CC14, 86CB2069, 88S73, 89S364>.

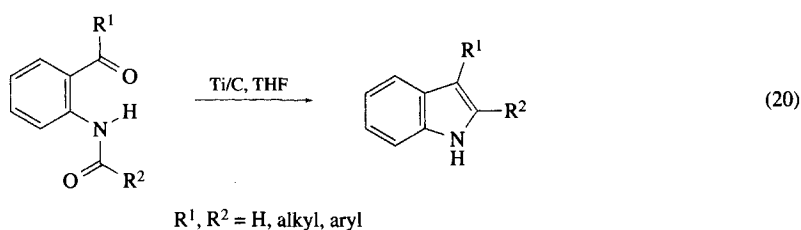


<i>R</i>	Base	Yield (%)	Ref.
CH_2Ph	$\text{KO}^\text{t}\text{Bu}^\text{t}$	64	88S73
$\text{HC}=\text{CMe}_2$	$\text{KO}^\text{t}\text{Bu}^\text{t}$	93	81JCS(CC)14
$\text{C}(\text{Me})=\text{CH}_2$	$\text{KO}^\text{t}\text{Bu}^\text{t}$	65	89S364
$\text{CH}_2\text{CO}_2\text{Et}$	$\text{KO}^\text{t}\text{Bu}^\text{t}$	72	86CB2069

An alternative retrosynthesis is represented by path B in Scheme 31. The most common route to the starting materials is by alkylation of *o*-acylanilines. Usually an EW substituent is required as the R substituent in the 2-position. For example, 2-benzoyl <92JOC3622>, 2-ethoxycarbonyl <92JMC4823>, and 2-cyanoindoles <72JHC161> (Equation (19)).

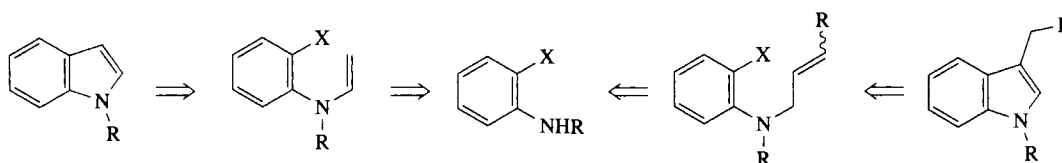


Reductive cyclization of *o*-acylanilides by low-valent titanium has recently been explored as a method of synthesis of indoles <92T5991, 93CC211, 94CB1125>. The reaction is most conveniently carried out using titanium-graphite. In addition to alkyl and aryl substituents, examples of cyclization with R² being styryl, acetyl and ethoxycarbonyl were successful (Equation (20)).



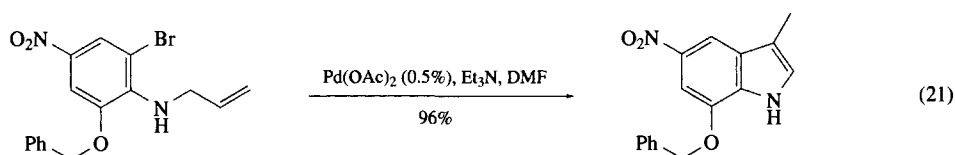
2.03.2.3 Category Ic Cyclizations

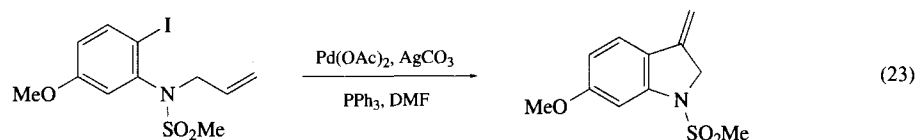
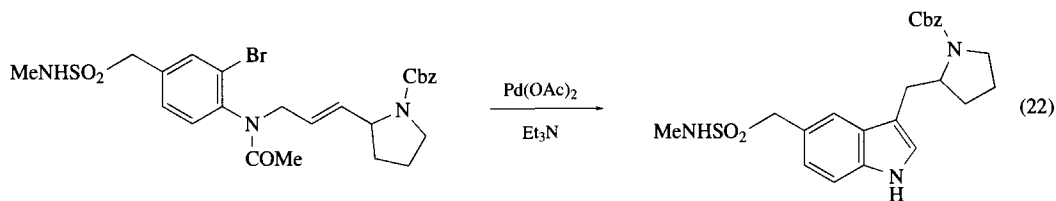
Category Ic cyclizations are rare for pyrroles. Palladium-catalyzed cyclizations of *N*-allyl and *N*-vinyl-*o*-haloanilines are useful indole syntheses which are in category Ic. The ultimate starting material is usually an *o*-haloaniline (Scheme 32).



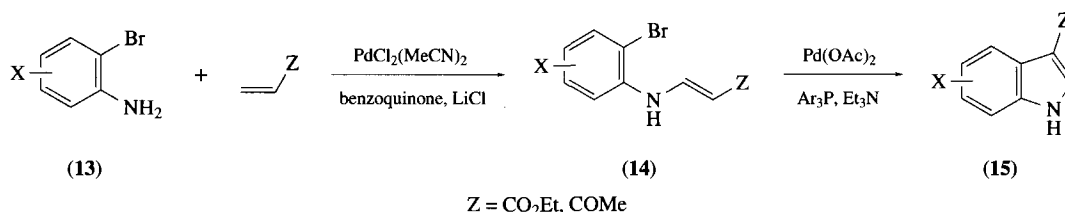
Scheme 32

The most useful of these routes is the intramolecular Heck reaction of *N*-allyl-*o*-haloanilines which was developed as an indole synthesis by Hegedus and co-workers <80JOC2709>. The reaction was initially carried out using Pd(OAc)₂, Ph₃P and triethylamine. An examination of alternative bases found NaO₂CCH₃ and Na₂CO₃ to be preferable in some cases <87TL5291>. Recently, a water-soluble phosphine ligand which permits mild cyclization conditions was introduced <92SL715>. The reaction presumably requires regeneration of palladium(0) *in situ* to maintain the catalytic cycle which involves oxidative addition at the aryl halogen bond. The reaction proceeds by 5-*exo*-trig cyclization via indolinylmethylpalladium and *exo*-methyleneindoline intermediates. The latter are sometimes isolated <93JCS(P1)1941>. The reaction is applicable to both ring- and *N*-substituted indoles (Equations (21)–(23)) <91JOC3048, 92TL8011>.



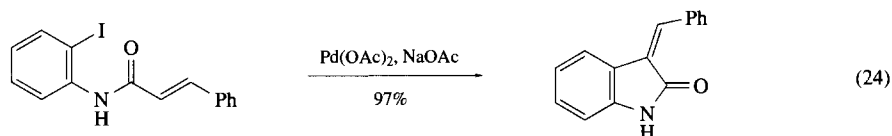


Indole-3-carboxylate esters and 3-acetylindoles can be prepared by palladium-catalyzed cyclization of enamines (14). These can be prepared from the corresponding anilines by condensation with β -dicarbonyl compounds or by palladium-catalyzed substitution reactions with ethyl acrylate or methyl vinyl ketone (<86BCJ927, 90S215>). Overall yields for the two-step process are 60–80% (Scheme 33).

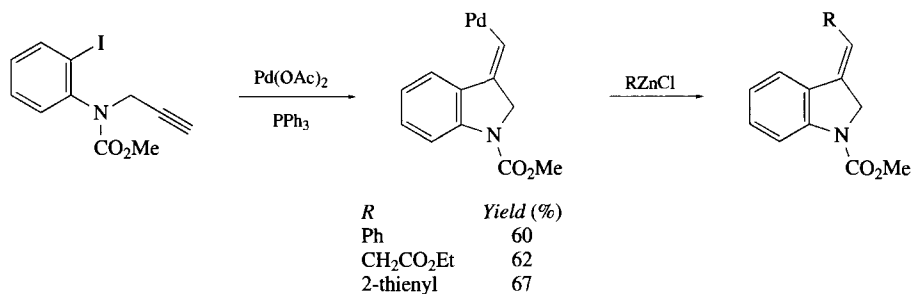


Scheme 33

A related reaction can be used to prepare oxindoles (Equation (24)) (<87TL5291, 92TL4859, 93JOC6949>).



The cyclization can also be done using *N*-propargylanilines in which case vinylpalladium intermediates are formed. These intermediates can undergo cross-coupling with organozinc reagents, permitting tandem extension with a 3-substituent (<89TL1135, 91H(32)2365>). The products isolated are 3-alkylideneindolines, but these can be isomerized to the corresponding indoles (Scheme 34).

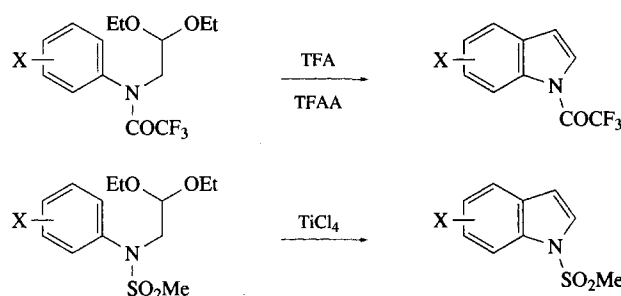


Scheme 34

Another indole synthesis which corresponds to the Ic pattern is the reaction of anilines with α -halomethyl carbonyl compounds. The reactions of *N*-alkylanilines with γ -bromo or γ -chloroacetoacetate esters gives indoles in fair to good yields. This method can be applied to indole-3-acetate esters and amides and can incorporate branching in the acetate side-chain (Equation (25)) (<62BSF1056, 64BSF1939>).

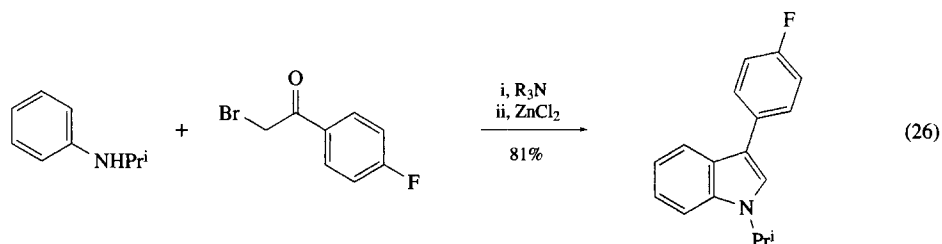


The use of simpler α -halocarbonyl compounds in indole synthesis is more limited. Several groups have explored the use of the diethyl acetal of bromoacetaldehyde as a potential indole precursor. Both *N*-trifluoroacetyl and *N*-methanesulfonyl derivatives of aniline can be alkylated by the acetal and cyclization occurs under acidic conditions (<81JOC778, 84JOC249>). These reactions require electron-donor substituents (Scheme 35).

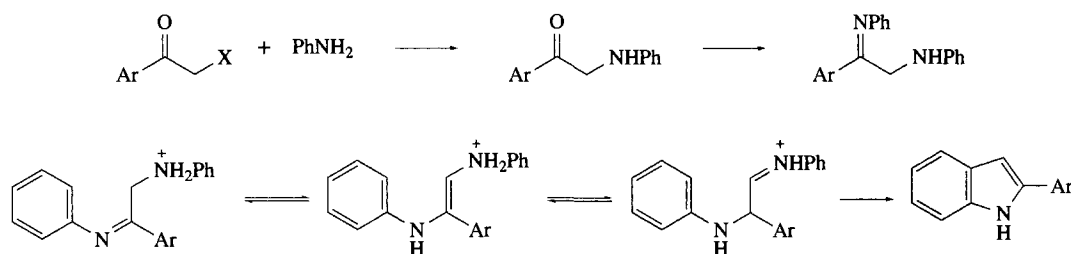


Scheme 35

3-Arylindoles can be prepared by acid-catalyzed cyclization of α -anilinoacetophenones (Equation (26)) (<85TL2155>).



The reaction of *N*-unsubstituted anilines with phenacyl halides is a useful way to make 2-arylindoles (<71JCS(C)2606>). This reaction is known as the Bischler indole synthesis. The reaction is typically carried out by heating a phenacyl halide with an excess of an anilinium salt. The reaction is believed to involve imine formation followed by protropic shifts and cyclization at the less hindered aldiminium carbon (Scheme 36) (<80AJC343>).

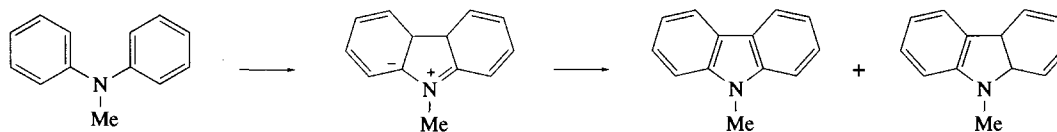


Scheme 36

An alternative procedure involves heating an *N*-phenacylpyridinium salt with an aniline. The mechanism is presumably similar to that of the Bischler procedure, involving initial formation of an imine (<78JOC(B)533>).

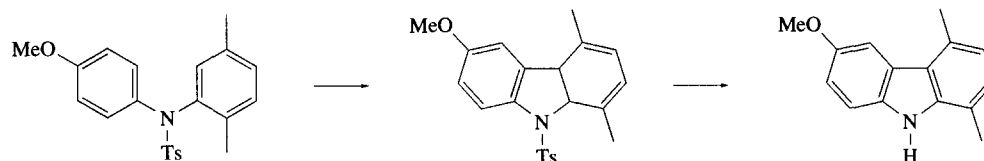
Type Ic cyclization can be applied to *N,N*-diarylamines to obtain carbazoles. Photolysis of diphenylamine leads to an electrocyclization which is followed by oxidation. Under anaerobic

conditions the cyclization intermediate disproportionates to a carbazole and a dihydrocarbazole (Scheme 37) <81JA6889>.



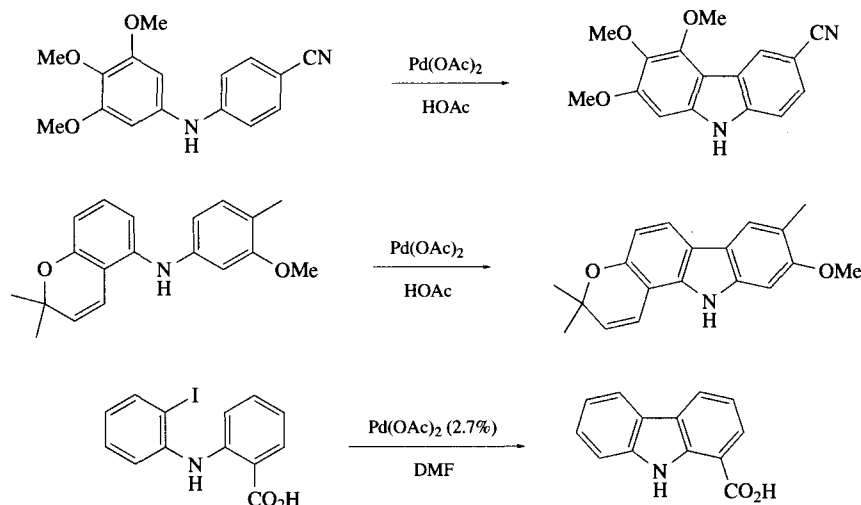
Scheme 37

Photocyclization can be advantageously carried out on the *N*-tosyl derivatives of diarylamines <92JCS(P1)3439>. Elimination of the sulfinate anion leads to aromatization (Scheme 38).



Scheme 38

Diarylamines can also be cyclized to carbazoles by heating with $\text{Pd}(\text{OAc})_2$ <75JOC1365>. A variety of substituted carbazoles have been prepared by this reaction <86CPB2672, 92JCS(P1)3439, 94TL1695>. *o*-Iododiarylamines are also potential precursors of carbazoles (Scheme 39) <84T1919>.



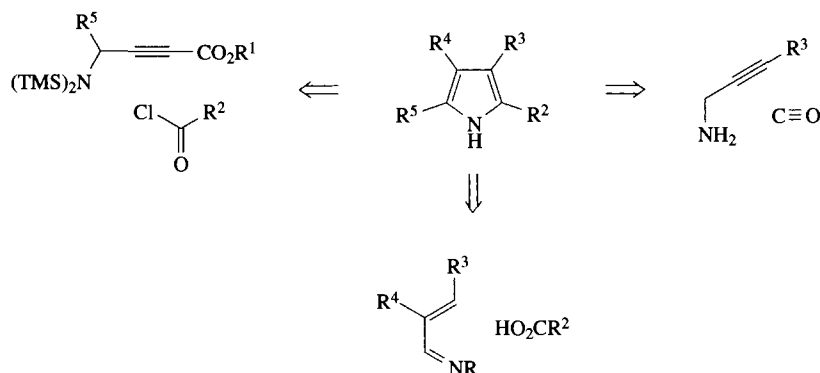
Scheme 39

2.03.2.4 Category IIab Cyclizations

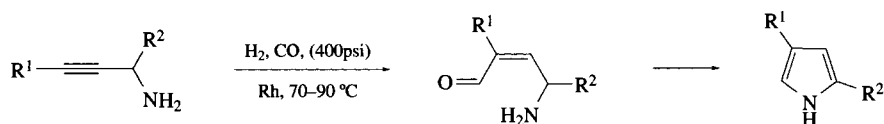
The IIab pattern corresponds to incorporation of a single carbon at C2 of a pyrrole or indole ring. Scheme 40 outlines some retrosynthetic relationships corresponding to pyrrole syntheses of this type.

One example of this pattern involves carbonylation of propargylamines under hydroformylation conditions. Excellent yields were obtained for 4-arylpyrroles, but 4-alkylpyrroles were accompanied by butanolides as by-products (Scheme 41) <92AJC1167>.

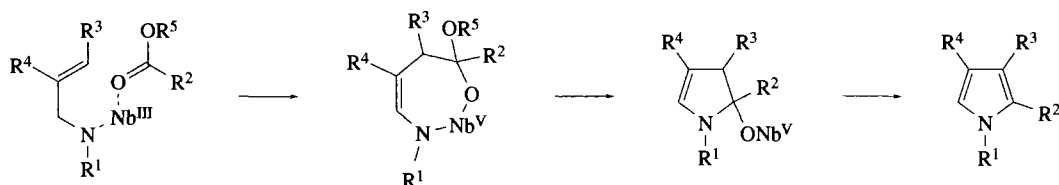
α,β -Unsaturated imines react with esters to give pyrrole derivatives in the presence of NbCl_3 . The niobium presumably functions both to form a ternary complex and to effect the reductive formation of the C—C bond. *N,N*-Dimethylformamide can also function as the carbonyl component, in which case a 3,4-disubstituted pyrrole is formed (Scheme 42) <89JOC4736>.



Scheme 40

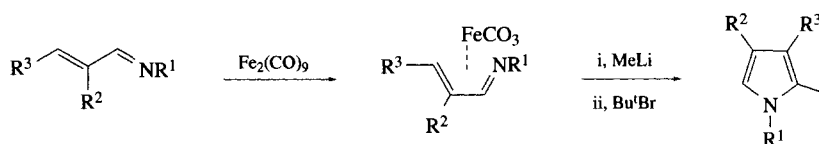


Scheme 41



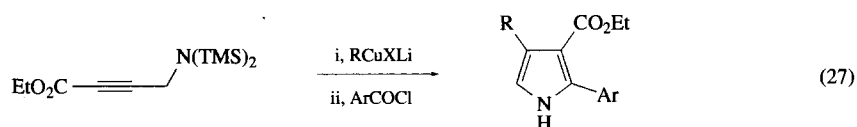
Scheme 42

α,β -Unsaturated imines also form complexes with $\text{Fe}(\text{CO})_3$. These react with methyllithium to give 2-methylpyrroles ^{<90JCS(P1)761>}. The reaction involves overall reductive acetylation of the β -carbon of the unsaturated imine, followed by cyclizative aromatization.



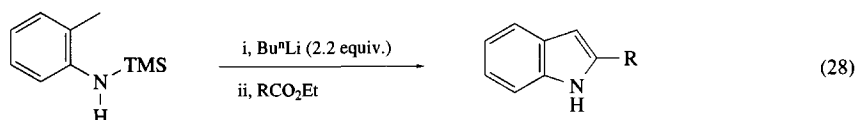
Scheme 43

Organocopper intermediates formed by carbocupration of ethyl 4-(bis-trimethylsilylamino)but-2-ynoate undergo cyclization with aryl chlorides to form 2-arylpyrrole-3-carboxylate esters (Equation (27)) ^{<93OM2478>}.

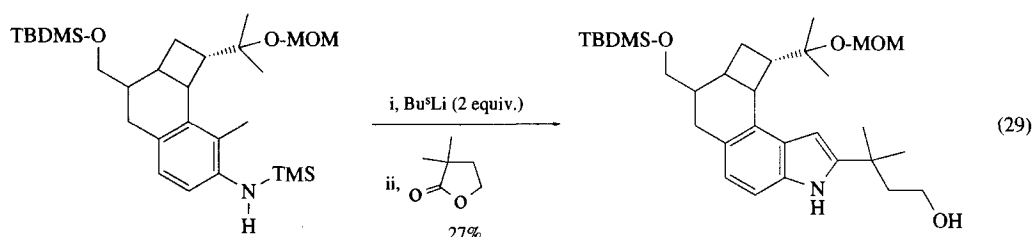


Category IIab indole syntheses involve acylation of *o*-aminobenzyl anion equivalents. These reactions are closely related to the Ia type syntheses since they typically involve an amine-carbonyl condensation in the final cyclization. The methods described here, however, are carried out without isolation of the intermediate. *N*-(Trimethylsilyl)-*o*-methyl-anilines condense with esters and lactones

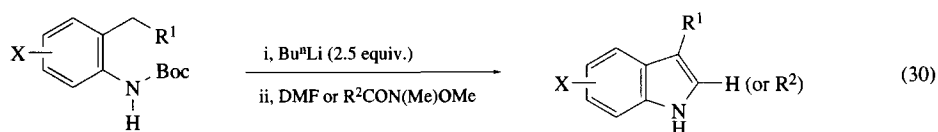
to give indoles. The reaction is effected using 2.2 equivalents of *n*-butyllithium and presumably involves *C*-acylation of a dilithiated intermediate <86T2957>. For simple alkyl and aryl esters yields of 2-substituted indoles are around 60% (Equation (28)).



The method was successfully applied to the synthesis of examples of the penitrem class of terpenoid indoles by introducing a 2-(1,1-dimethyl-3-hydroxypropyl) side-chain by using 3,3-dimethylbutyrolactone as the acylating agent (Equation (29)) <89T2431>.



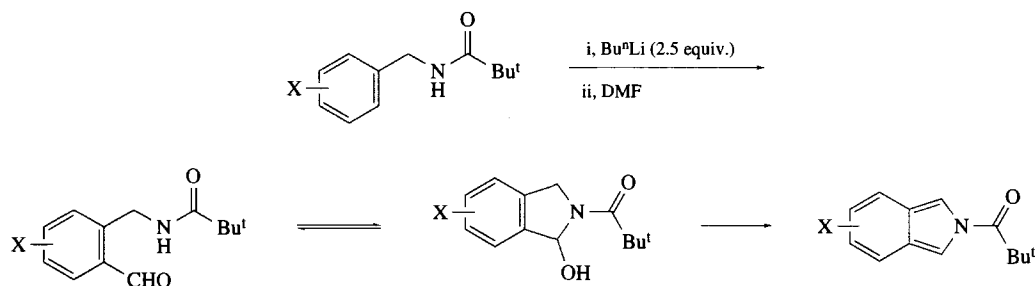
A similar synthesis of both 2- and 3-substituted indoles has been done using *N*-Boc-*o*-alkylanilines as the reactants <90SL207, 91S871>. Use of DMF gives indoles unsubstituted at C2 while *N*-methoxy-*N*-methyamides afford 2,3-disubstituted indoles.



Another type of *o*-aminobenzyl synthon is based on *o*-(bis-trimethylsilylamino)benzyl)copper-zinc reagents <89TL4795>. These are prepared from the corresponding benzylic bromides by reaction with zinc, followed by addition of cuprous cyanide. These reagents react with acid chlorides to give 2-substituted indoles. Since the reactions proceed by *C*-acylation rather than *N*-acylation, the conditions required for cyclization are mild and typically the *C*-acylated intermediates are not isolated.



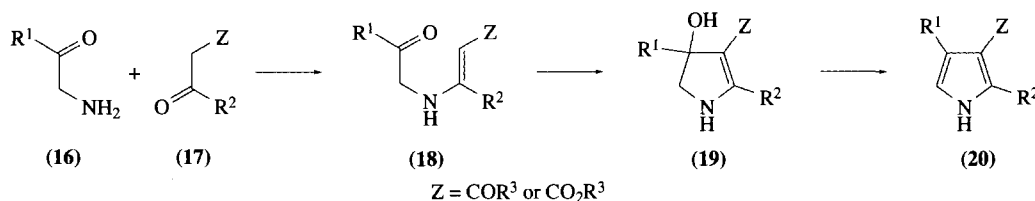
The IIab cyclization pattern has also been used to construct isoindoles. Lithiation of *N*-benzyl-pivalamides followed by formylation leads to 1-hydroxydihydroisoindoles. These can be converted by heating to isoindoles which can be trapped by dienophiles (Scheme 44) <90SL425>.



Scheme 44

2.03.2.5 Category IIac Cyclizations

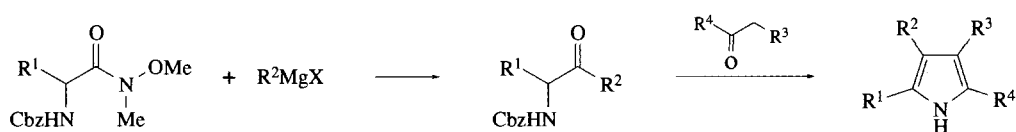
The condensation of carbonyl compounds with α -aminoketones gives pyrroles by a IIac cyclization known as the Knorr pyrrole synthesis. In most examples the carbonyl compounds are β -ketoesters or β -diketones (Scheme 45).



Scheme 45

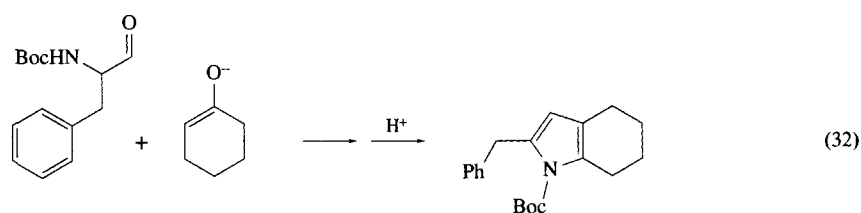
The cyclization normally proceeds by initial enamine formation. Enamines of this type can also be generated by addition of α -aminoketones to dimethyl ethynedicarboxylate or methyl propiolate and they can be converted to pyrroles (62JOC3346, 64JA107, 68T1567, 69T527, 91JCS(P1)3371). Intermediates of both structures (18) and (19) have been observed. The condensations are usually carried out in hot acetic acid.

The classical method of obtaining α -aminoketones for Knorr synthesis is by nitrosation of the carbonyl compound, followed by *in situ* reduction with zinc (82JOC3059, 82JOC5217). An alternative is to prepare *N*-methoxy-*N*-methylamides of *N*-protected (Boc, Cbz) amino acids. These react with Grignard reagents to give protected α -aminoketones which can be deprotected and used in the Knorr synthesis (Scheme 46) (93H(35)843).

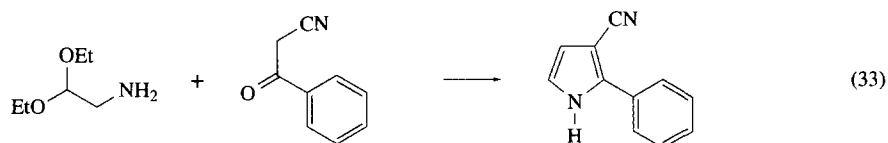


Scheme 46

A related pyrrole synthesis has been reported for cyclic ketones, but its scope is not fully established. Protected α -aminoaldehydes obtained from *N*-methoxy-*N*-methylamides by reduction with LiAlH_4 are condensed with enolates and the resulting aldols are cyclized to pyrroles by acid (Equation (32)) (92TL6939).



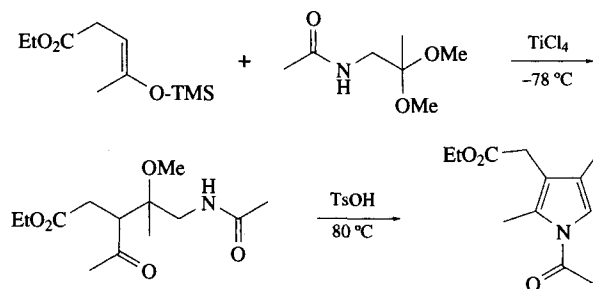
α -Cyanoacetophenones condense with the diethyl acetal of aminoacetaldehyde to give 3-cyano-2-arylprrroles in good yield (Equation (33)) (92MI 203-01).



α -Aminoacetophenones condense with β -dimethylaminoacrylonitrile to give 4-aryl-3-cyano-pyrroles (Equation (34)) (92MI 203-02).

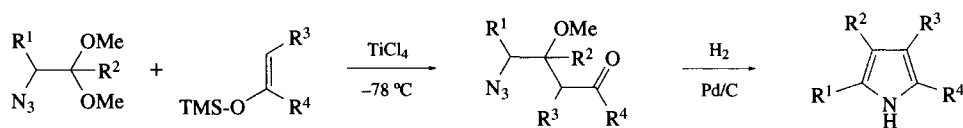


While the normal Knorr conditions involve initial N—C bond formation, the overall reaction can also be accomplished under conditions which begin with C—C bond formation. Mukaiyama conditions have been used to condense the dimethyl ketal of acetamidoacetone with silyl enol ethers. The resulting adducts can be cyclized to pyrroles (Scheme 47) <88S381>.



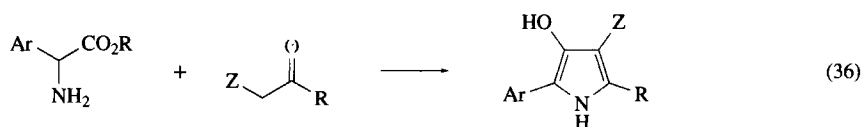
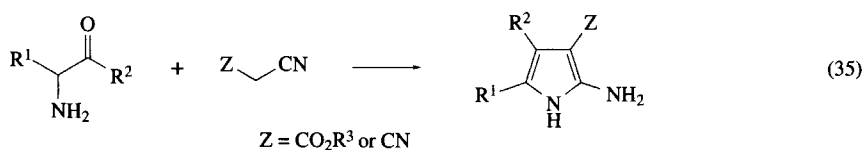
Scheme 47

The Mukaiyama reaction has also been used with the dimethoxy acetal of azidoacetaldehyde to give 2,3-disubstituted pyrroles (Scheme 48) <90AG(E)777>.

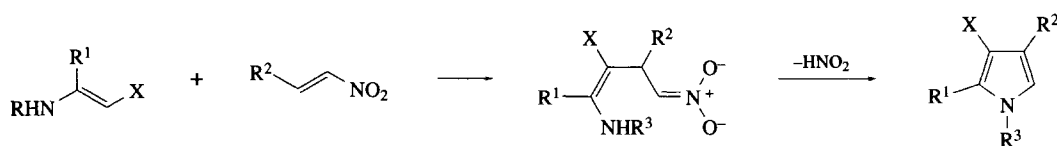


Scheme 48

The Knorr cyclization can also be used to construct amino- and hydroxypyrroles by reactions involving addition to a cyano or ester group. Use of an α -cyanoacetate ester instead of a ketoester under Knorr conditions results in the formation of 2-aminopyrroles <85S291>. Use of malonitrile gives 2-amino-3-cyanopyrroles (Equation (35)) <77JHC383>. α -Arylglycinate esters condense with β -ketoesters or β -diketones to give 2-aryl-3-hydroxypyrroles (Equation (36)) <93JHC1253>.

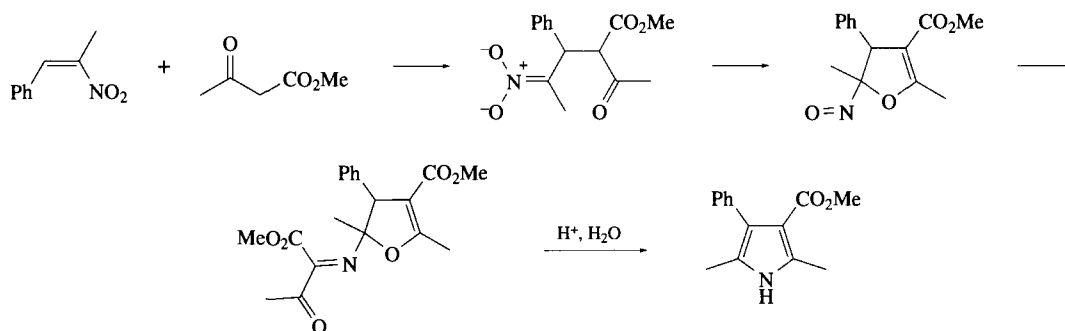


Pyrroles can also be obtained from enamines and nitroalkenes <53HCA49>. The reaction evidently proceeds by Michael addition followed by cyclization and elimination of nitrous acid (Scheme 49).



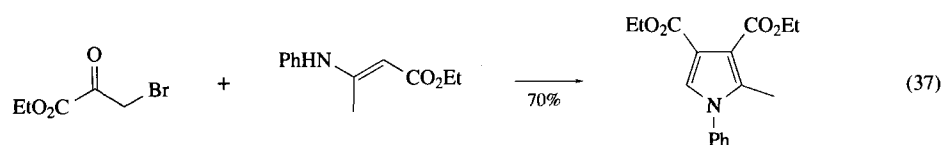
Scheme 49

This reaction appears to be quite general and the enamine nitrogen can be unsubstituted or substituted (81LA1534). Polycyclic structures incorporating pyrrole rings can be obtained from reactants in which either the enamine or nitroalkene is incorporated into a ring structure. Pyrroles can also be constructed from nitroalkenes and β -ketoesters. An initial 1:2 adduct is formed by Michael addition and cyclization of the nitronate intermediate. A nitroso intermediate is formed which condenses with a second molecule of β -ketoester. Mild acid hydrolysis results in pyrrole formation (Scheme 50) (84LA911, 85LA239). Despite this mechanistic complication, yields are in the 50–70% range for a number of substituted aryl derivatives.

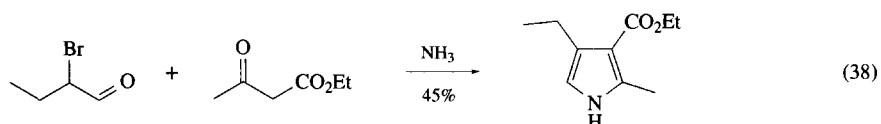


Scheme 50

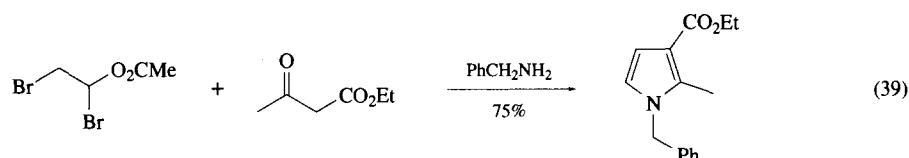
Another Hiac condensation pattern involves the reaction of enamines with α -halocarbonyl compounds (Equation (37)) (80T2125).



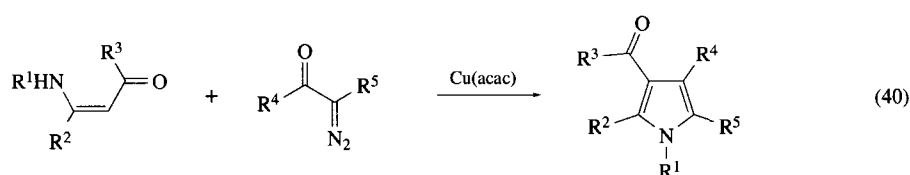
The Hantzsch pyrrole synthesis is a variation of this reaction which is conducted as a three component synthesis from a β -dicarbonyl compound, an α -haloketone or aldehyde and ammonia or an amine (Equation (38)). Yields for this procedure are typically 20–50% (70CJC1689). The regiochemistry is that resulting from N—C bond formation at the carbonyl carbon of the α -haloaldehyde.



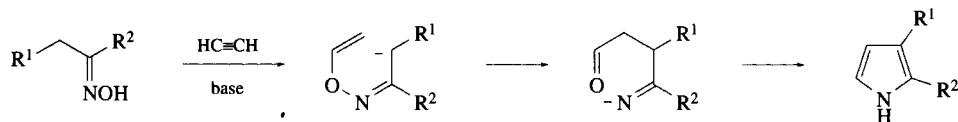
1,2-Dibromoethyl acetate is a useful α -bromoacetaldehyde surrogate in this reaction (Equation (39)) (90SC1923).



α -Diazoketones also react with stabilized enamines in the presence of a copper catalyst to give pyrroles (Equation (40)) (88JOC2084).

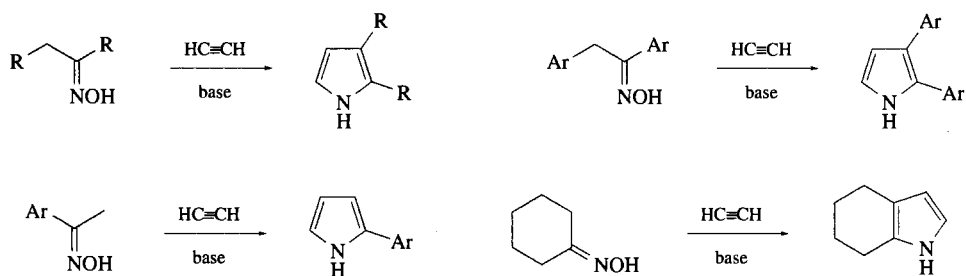


Trofimov and co-workers have developed a pyrrole synthesis which involves reactions of ketoximes with alkynes under strongly basic conditions (Scheme 51) <90AHC178, 94H(37)1193>. The key step in the reaction is a sigmatropic rearrangement of an *O*-vinylloxime which is followed by a typical imine-carbonyl condensation.



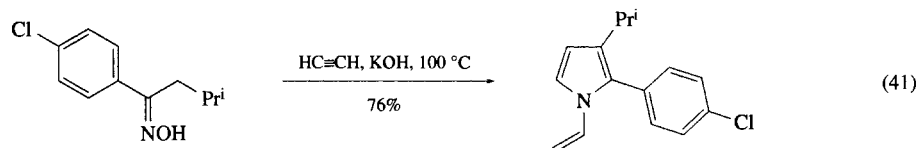
Scheme 51

Scheme 52 illustrates the application of this reaction to a variety of substitution patterns.

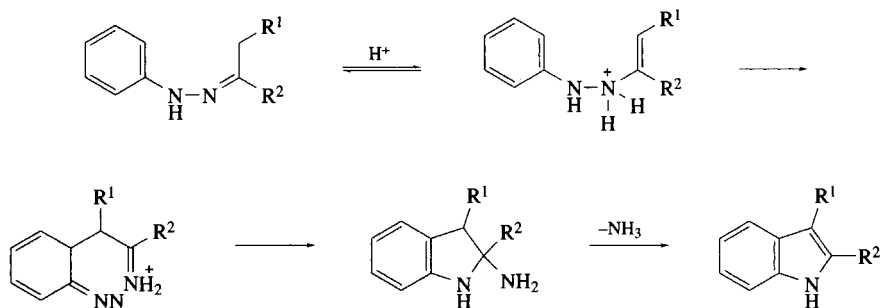


Scheme 52

Frequently an *N*-vinylpyrrole formed from the initial pyrrole is a major by-product (Equation (41)) <92CHE510>.



The Fischer indole synthesis is probably the most versatile method for making indoles; certainly it is the most widely applied <B-82MI 203-01, 88CHE709, 93OPP609>. The reaction involves acid-catalyzed conversion of an *N*-arylhydrazone to an indole. The mechanism involves a [3,3] sigmatropic rearrangement of an enehydrazine tautomer. For unsymmetrical ketones, two isomeric indoles are possible and the general result is that the indole derived from the more stable (usually the more highly substituted) enehydrazine is formed (Scheme 53).

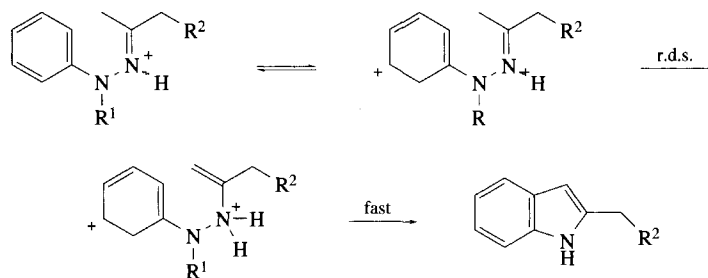


Scheme 53

The Fischer cyclization is very versatile in terms of the functional groups which can be tolerated at positions 2 and 3, and in the aromatic ring. The reaction is somewhat retarded by strong electron-withdrawing groups but even nitroaryl hydrazones can be cyclized under appropriate conditions <73CHE31, 84CPB2126, 86JMC2415, 88CHE154, 92JMC4086>. A variety of protonic and Lewis acids can be

used to effect the cyclization. Strong acids such as hydrochloric or sulfuric acid in water, methanol, ethanol or glacial acetic acid are frequently used <86JMC2415, 92JMC4823>. Polyphosphoric acid is also effective <89TL2099>. Zinc chloride is the most commonly used Lewis acid <93JMC2908>. A number of solid catalysts for high temperature reaction have been developed and these presumably function as Lewis acids. These include alumina, $\text{Al}_2\text{O}_3\text{-MgF}_2$, and AlPO_4 <83BSB715, 88CHE1191>. There have also been a number of cases reported where heating *N*-arylhydrazones in organic solvents without catalysts results in indole formation. Among the solvents used are pyridine (with the hydrazone hydrochloride) <77S645>, ethylene glycol or diethylene glycol <57JOC1703>, and sulfolane <87JOU1395>.

The regiochemistry of the Fischer cyclization can be controlled to some extent by the reaction conditions. Use of very strongly acidic conditions can shift the reaction to favor cyclization through the less stable enehydrazine. In general terms, this has been interpreted as resulting from competition between reaction through the thermodynamically favored enehydrazine or through a more rapid reaction of a less prevalent isomer. Recent detailed studies of the effect of acid strength and solvent isotope effects have provided evidence that the reaction in strongly acidic solution may proceed through an intermediate in which the aromatic ring is protonated <91JOC3001, 93JOC228>. This would be expected to substantially lower the activation energy of the sigmatropic rearrangement step. The interpretation of the effect of acid strength is that the less substituted (but kinetically preferred) enehydrazine is formed by *C*-deprotonation and rapidly rearranges (Scheme 54).



Scheme 54

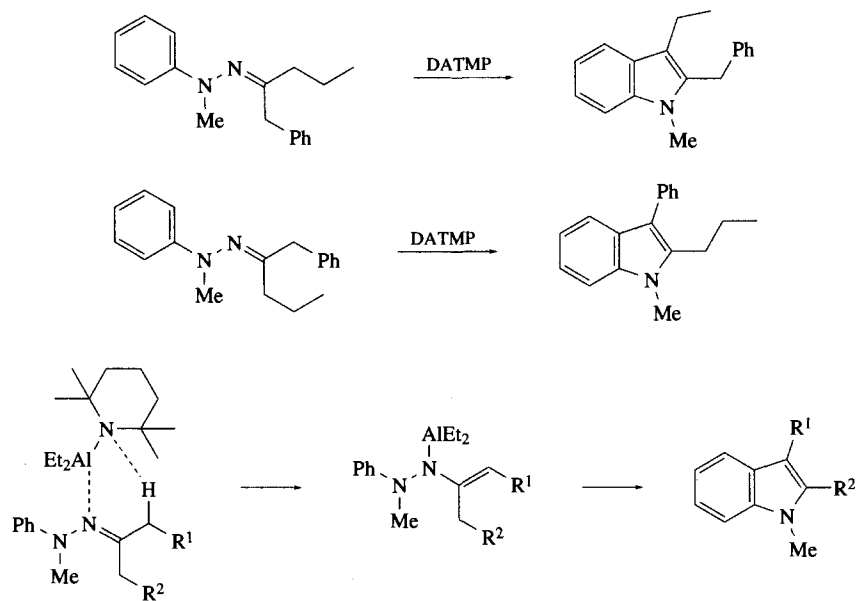
Another recent development opens a novel approach to control of the regiochemistry of Fischer cyclization. It has been found that aluminum amides, specifically diethylaluminum-2,2,6,6-tetramethylpiperidide (DATMP), promotes indolization of *N*-methyl-*N*-phenylhydrazones with the regioselectivity depending upon the stereochemistry of the hydrazone. The cyclization occurs into the substituent *syn* to the hydrazone lone-pair. This regioselectivity can be rationalized by a reaction mechanism in which the complexation of the aluminum at the hydrazone, followed by *syn* deprotonation, determines the regiochemistry (Scheme 55) <93JOC7638>. The generality of this approach, and in particular its extension to *N*-unsubstituted indoles, remains to be demonstrated, but if this approach proves to be widely applicable it will provide an important new means of regiochemical control.

ortho-Substituted aryl hydrazones sometimes give abnormal cyclization products <81ACR275>. For example, the *N*-(2-biphenyl)hydrazone of ethyl pyruvate gives both 4-phenyl and 7-phenylindole-2-carboxylate products. This can be attributed to sigmatropic rearrangement involving both the unsubstituted and substituted *ortho* positions, with the latter resulting in a subsequent 1,2-shift of the phenyl group (Scheme 56).

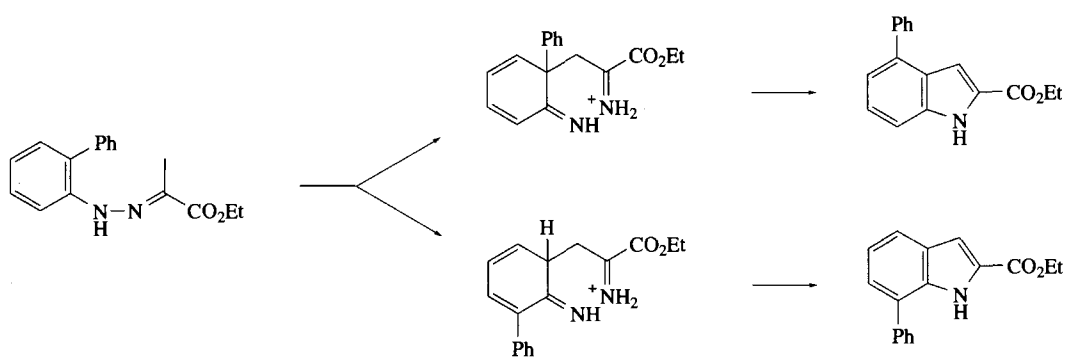
Products arising from 1,5-alkyl shifts have also been observed in the case of 2,4,6-trisubstituted *N*-arylhydrazones <80JA4772>. Products which arise from nucleophilic addition to the *ipso*-substituted rearrangement intermediate have also been observed. This is especially common with *N*-(*o*-alkoxyphenyl)hydrazones <81ACR275>. The nucleophile can be either solvent or chloride ion derived from the acidic catalyst (Scheme 57).

In addition to direct formation from an arylhydrazine and a carbonyl compound, *N*-arylhydrazones can be prepared from aryldiazonium ions by coupling with enolates or enamines (Japp-Klingemann reaction). This reaction has most frequently been applied to β -ketoesters. The coupling product undergoes deacylation so that the ultimate product of Fischer cyclization is an indole-2-carboxylate ester (Scheme 58) <92JMC4823>.

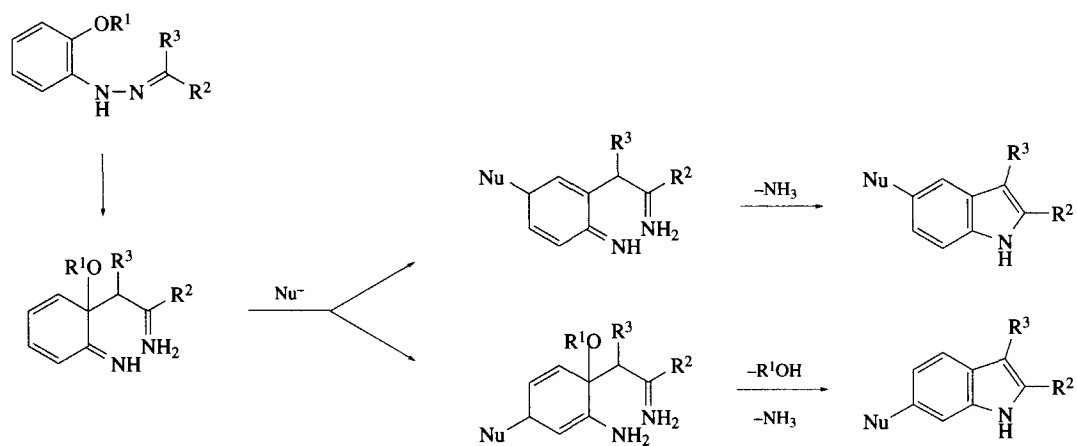
If the coupling is done on a β -ketoacid, decarboxylation of the coupling product occurs so that the final product is a 2-acylindole (Scheme 59) <92SC421>.



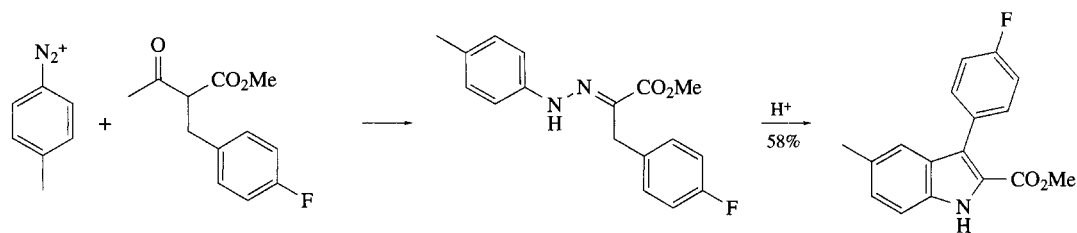
Scheme 55



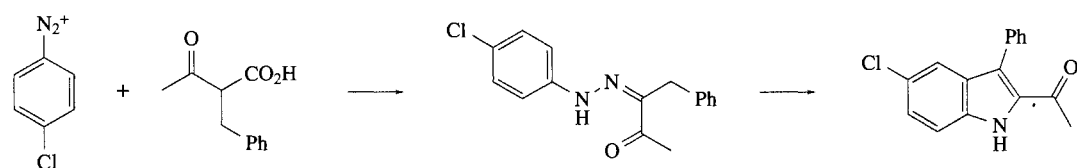
Scheme 56



Scheme 57

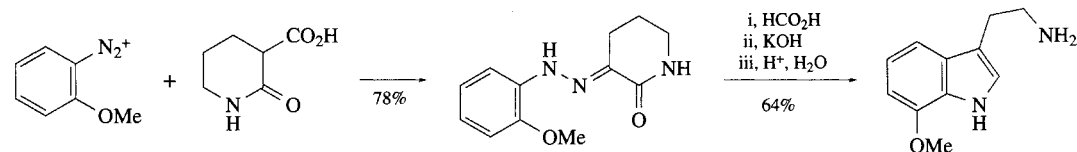


Scheme 58



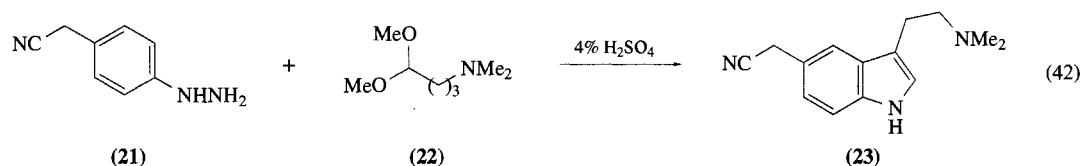
Scheme 59

A special application of the Japp–Klingemann coupling involves the use of 3-carboxy-2-piperidone (56JCS4593). After coupling, decarboxylation and cyclization, 1-oxo-1,2,3,4-tetrahydro- β -carboline are obtained. Under acidic conditions these can be hydrolyzed to tryptamines (Scheme 60) (89JOC2170, 93SC1689).

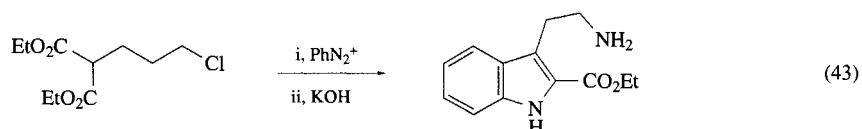


Scheme 60

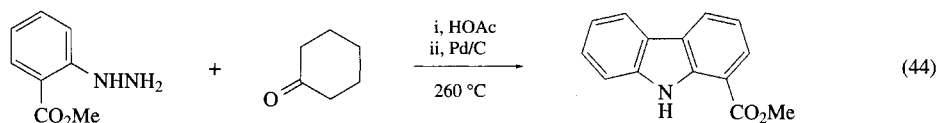
Because of the biological interest in tryptamines, other reactions which are applicable to their synthesis by Fischer cyclizations have been developed. The cyanomethyl compound (23) is an example. Its synthesis from *N,N*-dimethyl-4,4-dimethoxybutanamine (22) can be carried out on a kilogram scale with 4% sulfuric acid as the preferred reaction medium (Equation (42)) (94JOC3738).



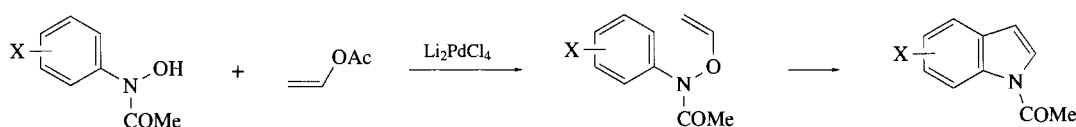
Chlorobutanal can serve as the precursor of *N*^b-unsubstituted tryptamines (68KGS1038). The halogen undergoes intramolecular nucleophilic displacement under the conditions of the Fischer cyclization. This approach can also be used in combination with Japp–Klingemann coupling (Equation (43)) (74S354).



Carbazoles can be synthesized by applying the Fischer cyclization to cyclohexanone hydrazones and then aromatizing the tetrahydrocarbazole product (88JHC671, 89JHC1213, 93T1223). An example is the synthesis of methyl carbazole-1-carboxylate (Equation (44)).

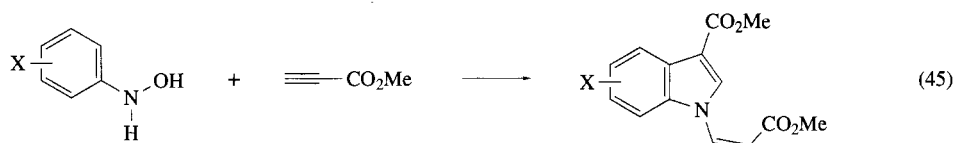


There are also several indole syntheses which are based on the use of arylhydroxyamines and which involve a [3,3]-sigmatropic rearrangement similar to that involved in Fischer cyclization. Substituted *N*-acetylarlyhydroxyamines can be vinylated by palladium-catalyzed addition–elimination with vinyl acetate. Under mild heating, rearrangement and cyclization occurs (Scheme 61) (84HCA1647, 88HCA344).



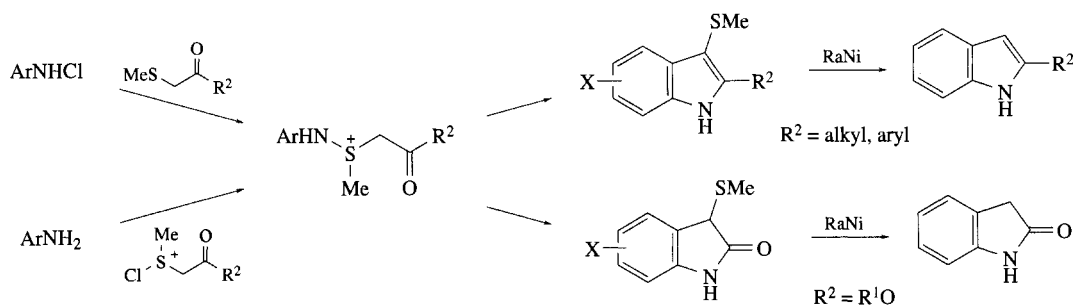
Scheme 61

In another example, arylhydroxyamines react with methyl propiolate to give indole-3-carboxylate esters (90H(31)1431). When the hydroxyamine has no other nitrogen substituent, the product is substituted on nitrogen by an acrylate group derived from addition to methyl propiolate (Equation (45)) (94JOC1577).



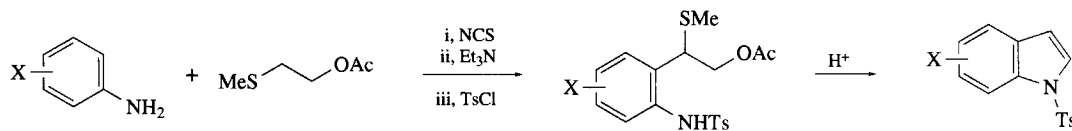
The rearrangement of hydroxyamine derivatives has also been applied to the synthesis of 2-substituted indoles (87T3237).

Gassman and co-workers developed a synthesis of indoles and oxindoles which involves [2,3]-sigmatropic rearrangement of *S*-anilinosulfonium salts. These can be prepared from *N*-chloroanilines or from the aniline and a *S*-chlorosulfonium salt. Rearrangement and cyclization occurs on reaction with triethylamine. If an α -methylthio ketone is used, the product is a 2-substituted 3-methylthioindole (74JA5495, 74JA5512). If an α -methylthio ester is used, the product is a 3-methylthio-oxindole (74JA5508). The cyclization products can be desulfurized with Raney nickel. The reaction is also adaptable to 2,3-disubstituted indoles by use of longer chain α -methylthio ketones. The cyclization products are 3-methylthio-3*H*-indoles which can be converted to indoles by reduction with LiAlH₄ (Scheme 62) (74CC201).



Scheme 62

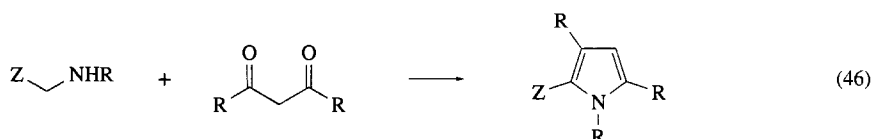
The Gassman method can also be applied to cyclic ketones and it has been applied to complex structures of the type encountered in terpenoid indole natural products (89JOC3449). A modification of the Gassman method uses 2-(2-propylthio)ethyl acetate as the sulfide and leads to the formation of indoles without the need for desulfurization. After the rearrangement an elimination ensues, leading to cyclization and aromatization (Scheme 63) (92H(34)1017).



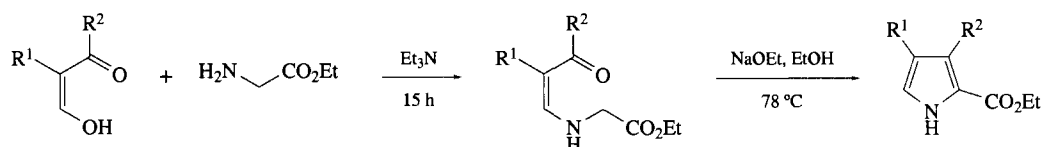
Scheme 63

2.03.2.6 Category IIad Cyclizations

1,3-Dicarbonyl compounds and their synthetic equivalents can give pyrroles by condensation with amines having an α -electron-withdrawing substituent such as an ester or a ketone (Equation (46)) (82S157).

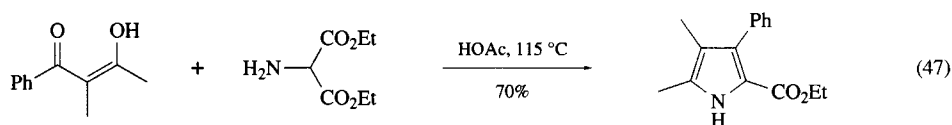


Such condensations have the potential for producing two isomeric products when the dicarbonyl component is unsymmetrical. The regioselectivity is usually governed by an initial condensation of the amino group at the more reactive carbonyl group. An example can be seen in the reaction of ethyl glycinate with hydroxymethylene ketones. Enaminone intermediates can be isolated and then cyclized by the use of a more strongly basic reagent (Scheme 64) (87JOC3986).



Scheme 64

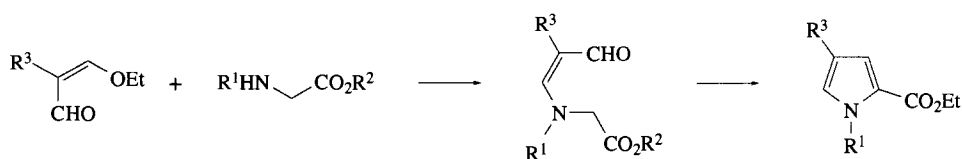
There are a number of variations of this pattern which have been developed. The α -aminocarbonyl compounds are usually generated *in situ* by nitrosation/reduction (93T2185). For some systems it is preferable to carry out the reduction by catalytic hydrogenation (85JOC5598). Aminomalonate esters can be used when the desired product is a pyrrole-2-carboxylate. Decarboxylation occurs during the course of the reaction so that the aminomalonate is a glycine equivalent (Equation (47)) (87JOC3986).



The reaction is most useful with symmetrical diketones but regioselectivity is observed in some circumstances, with the less hindered carbonyl group tending to become bound to nitrogen.

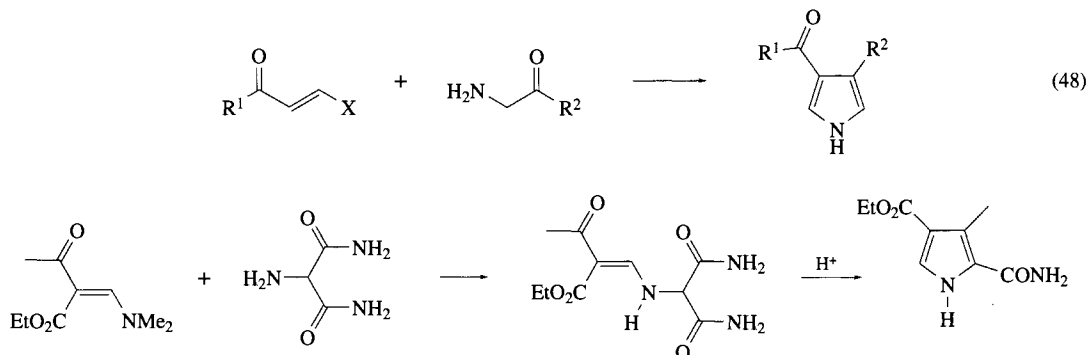
A number of β -substituted enones can function as 1,3-dicarbonyl equivalents by reactions which involve exchange of the amino group for a β -substituent. For example, α -alkyl- β -ethoxyacroleins can be converted to pyrrole-2-carboxylates by reaction with glycinate esters (Scheme 65) (89S337).

Ethyl glycinate, aminoacetonitrile and aminoacetamide each can condense with β -aminoenones.



Scheme 65

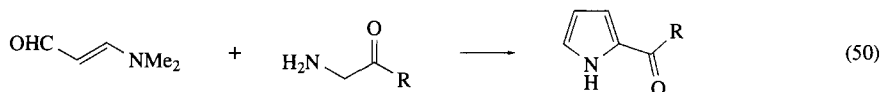
Such reactions have been used to obtain functionalized pyrroles (Equation (48) and Scheme 66) <87S566, 90H(31)1049>.



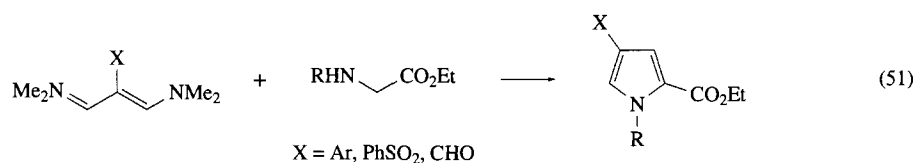
β -Chlorovinyl ketones are alternative β -dicarbonyl equivalents (Equation (49)) <72JPR353>.



β -*N,N*-Dimethylaminoacrolein condenses with some α -aminomethyl ketones with the alternative regiochemistry resulting from condensation at the enaminone carbonyl group (Equation (50)) <90JCS(P1)2681>.

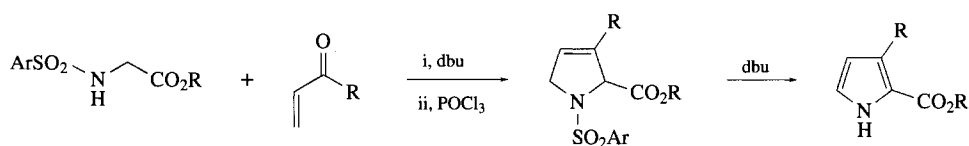


Arylvinidinium salts are also useful 1,3-dicarbonyl equivalents. Condensation with glycinate esters gives 4-arylpyrrole-2-carboxylate esters in generally excellent yields (Equation (51)) <90JOC4735>. There are a few examples where other substituted vinidium salts have been used.



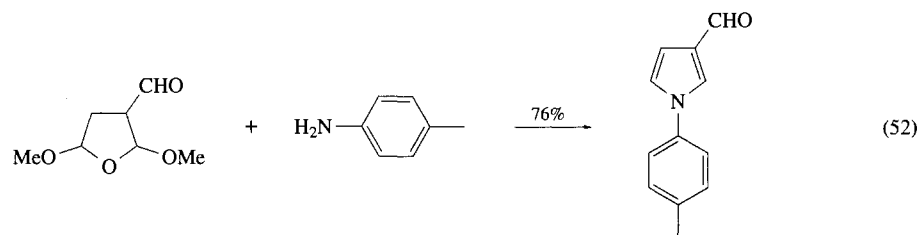
5-Arylpyrrole-2-carboxylate esters can be obtained from 3-aryl-3-chloropropeniminium salts and glycinate esters <92JOC5480>.

α,β -Unsaturated carbonyl compounds lacking β -leaving groups can be incorporated into pyrrole rings by reaction with *N*-tosylglycinate esters. Conjugate addition followed by aldol condensation generates dihydropyrroles. A subsequent elimination or oxidation provides the pyrrole. For example, with *N*-tosyl derivatives the aromatization is effected by elimination of sulfinate by *dbu* (Scheme 67) <65JCS4389, 91JHC1671>.



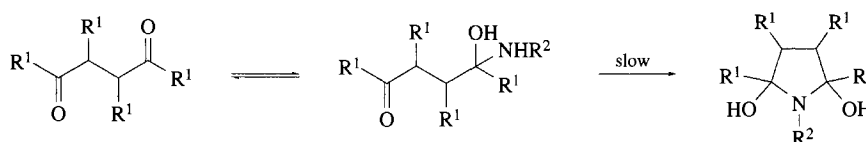
2.03.2.7 Category IIa: Cyclizations

The Paal–Knorr synthesis of pyrroles from 1,4-dicarbonyl compounds is one of the most general pyrrole syntheses. A variety of 1-substituted pyrroles can be prepared using 2,5-dimethoxytetrahydrofuran as a succinaldehyde equivalent $\langle 52\text{ACS}667, 78\text{JMC}962 \rangle$. Even nonnucleophilic amines such as 2,6-dinitroaniline and benzenesulfonamide can be converted to pyrroles in good yield $\langle 73\text{SC}303, 92\text{HCA}2608 \rangle$. 1,4-Dichloro-1,4-dimethoxybutane can also serve as a succinaldehyde surrogate $\langle 83\text{TL}1569 \rangle$. 2,5-Dimethoxytetrahydrofuran-3-carboxaldehyde, which is commercially available, serves as a precursor of pyrrole-3-carboxaldehydes (Equation (52)) $\langle 93\text{JMC}101 \rangle$.



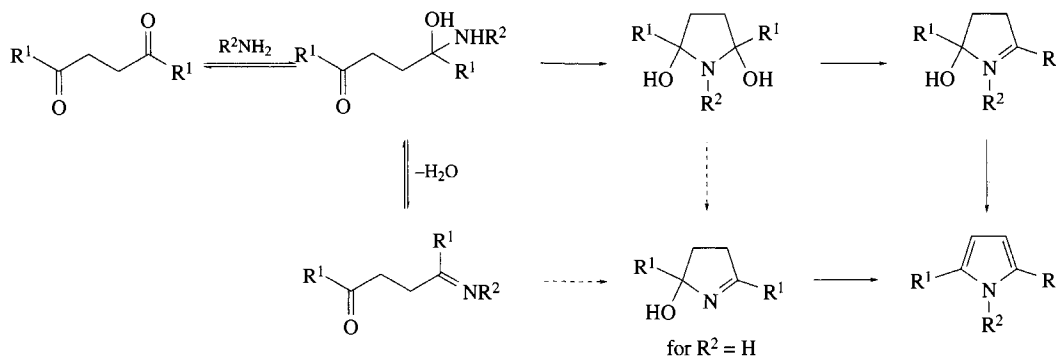
A variety of more complex 1,4-dicarbonyl compounds have been used in the synthesis of substituted pyrroles $\langle 83\text{JOC}2769, 84\text{JCS}(\text{P}1)2801, 91\text{TL}1765, 94\text{H}(37)1549 \rangle$.

There are several possible sequences for the addition, cyclization and dehydration steps involved in the overall transformation. A study of the effect of the size of 3,4-substituents in 2,5-hexanediones showed that rates decreased in the order Me > Et > Ph > Me₂CH. Both *dl* and *meso* isomers were compared and the *dl* isomers reacted more rapidly in each case $\langle 86\text{JOC}621 \rangle$. The fact that the stereoisomers do not interconvert under the cyclization conditions and the absence of a primary isotope effect for 3,4-hydrogen atoms have been taken to indicate that the cyclization of the hemiaminal is the rate-determining step $\langle 91\text{JOC}6924 \rangle$. This proposal is consistent with the observed steric effect, since the *meso* diketones result in *cis*-stereochemistry in the cyclization step (Scheme 68).



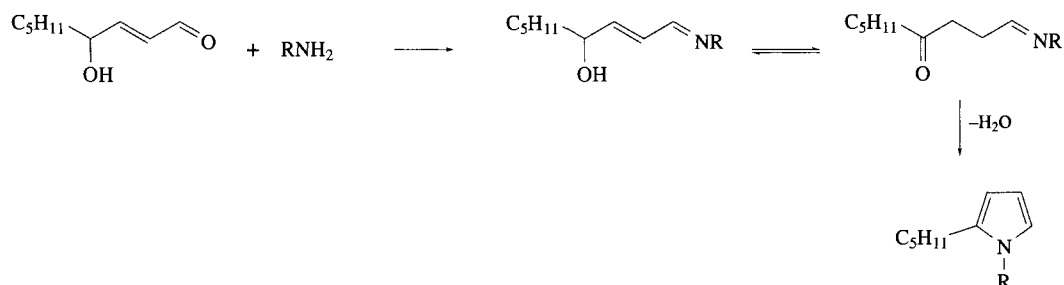
Scheme 68

Reaction of 1,4-diketones with liquid ammonia results in the formation of unstable 5-hydroxy-Δ^{1,2}-pyrrolenines which subsequently dehydrate to pyrroles $\langle 86\text{T}623, 88\text{T}3531 \rangle$. Monoimines of the diketones are also observable by NMR $\langle 86\text{T}623, 91\text{JOC}6924 \rangle$, but they may simply be in equilibrium with the ketone, amine and carbinolamine and may not be on the cyclization path. The currently available information suggests a minimal reaction system depicted in Scheme 69. None of the existing evidence appears to exclude cyclization at the imine stage.



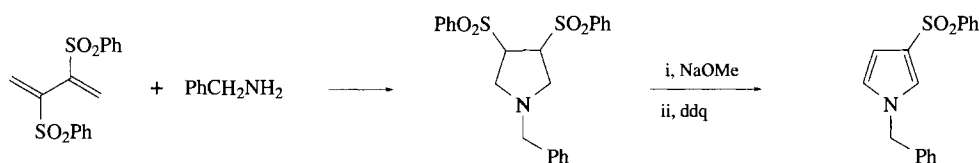
Scheme 69

Compounds that can isomerize to or equilibrate with 1,4-dicarbonyl systems are also potential pyrrole precursors. 4-Hydroxynon-2-enal, for example, condenses with primary amines to give 2-pentylpyrrole in a reaction which probably involves an isomerization of the imine intermediate (Scheme 70) <93MI 203-01>.

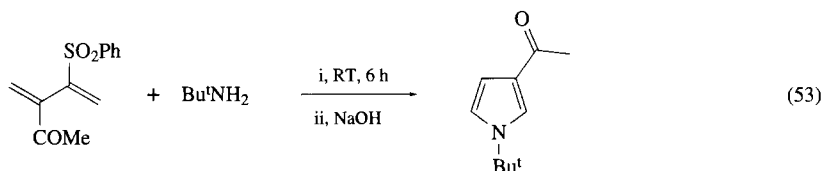


Scheme 70

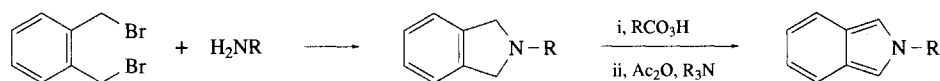
Another Ilae pyrrole cyclization involves nucleophilic addition of amines to electrophilic 1,3-dienes. The reaction pattern has been realized with 2,3-diphenylsulfonyl-1,3-butadiene and with 2-acetyl-3-phenylsulfonyl-1,3-butadiene <88TL3041, 91S171>. Elimination of phenylsulfinate, induced by base, introduces one double bond. The second can be introduced by ddq oxidation, as in Scheme 71. In Equation (53), oxidation is evidently the result of the exposure of the compound to the atmosphere.



Scheme 71

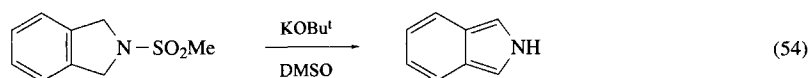


Category Ilae cyclizations play a prominent role in the synthesis of isoindoles. Simple 2-alkyl-2*H*-isoindoles can be prepared from 1,2-bis-(bromomethyl)benzene via dihydroisoindole-*N*-oxides. The oxides undergo Polonovsky elimination in acetic anhydride/triethylamine <88CZ85>. A similar reaction sequence can be applied to the synthesis of 2-aryl analogs (Scheme 72) <87CZ155>.

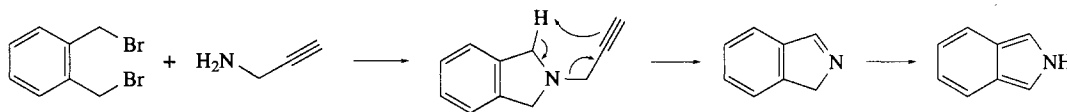


Scheme 72

The parent 2*H*-isoindole can be prepared as an 0.1 M solution in DMSO by reaction of 2-methanesulfonyldihydroisoindole with KOBu^t/DMSO (Equation (54)) <87CZ349>.

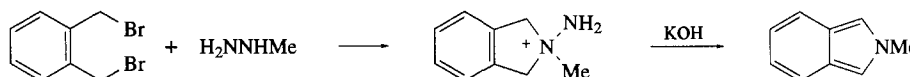


Thermolysis of *N*-(2-propynyl)dihydroisoindole at 500°C provides another synthesis of 2*H*-isoindole <84AG(E)517, 87CZ349>. The product can be obtained from the condensed (−196°C) pyrolyzate and used as a solution. The reaction presumably is a retroene process (Scheme 73).



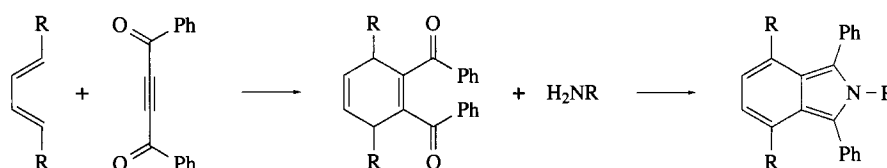
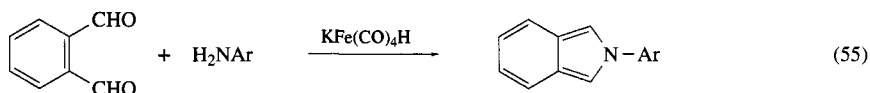
Scheme 73

2-Methyl-2*H*-isindole and 2-benzyl-2*H*-isindole have been prepared from bis-(bromomethyl)benzene by base-catalyzed elimination of hydrazinium salts (Scheme 74) <66T1011, 72S45, 85JOC1611>.



Scheme 74

Phthalaldehydes are alternative precursors of isindoles by *IIae* cyclization. Such reactions require a concomitant reduction. Heating 1,2-dibenzoylbenzene with ammonium formate generates 1,3-diphenylisindole <66T1011, 72S45, 85JOC1611>. Similarly, methylamine and NaBH₄ generate 2-methylisindole <73TL5185>. Anilines react with phthalaldehyde in the presence of K₂Fe(CO)₄ to give 2-aryl isindoles (Equation (55)) <79T1433>. The isindole reduction level can also be achieved by using the dihydro derivative of 1,2-dibenzoylbenzene in the reaction (Scheme 75) <71JOC1048>.



Scheme 75

1-Alkylthioisindoles are obtained from phthalaldehyde, primary amines and thiols (Equation (56)) <78JOC2886>.



2.03.2.8 Category IIbd Cyclizations

The most important pyrrole syntheses of this type are cycloadditions involving mesoionic oxazolium oxides, azomethine ylides or isonitriles. These syntheses are outlined in Figure 1.

The cycloaddition of oxazolium oxides (munchones) can be carried out by heating an α -amino acid in acetic anhydride. In the presence of a dipolarophile an unstable adduct is formed which then undergoes decarboxylation. Appropriate dipolarophiles include unsaturated ketones, esters, nitriles and nitro compounds, as well as electrophilic alkynes <86SC357, 87JHC1793>. By using dipolarophiles with a potential leaving group the cycloaddition can lead directly to pyrroles. Examples of such reactants include α -chloroacrylonitrile, which gives cyanopyrroles by HCl elimination <78JOC4273, 92MI 203-01>, as well as α -cyanoacrylates and alkylidene malononitriles which eliminate hydrogen cyanide <90T3515>. Adducts from nitroalkenes aromatize by loss of nitrous acid <89CAR(186)C7>. Munchones can also be generated directly from oxazolinones (azalactones) by alkylation with triethyloxonium salts (Equation (57)) <88S999>.

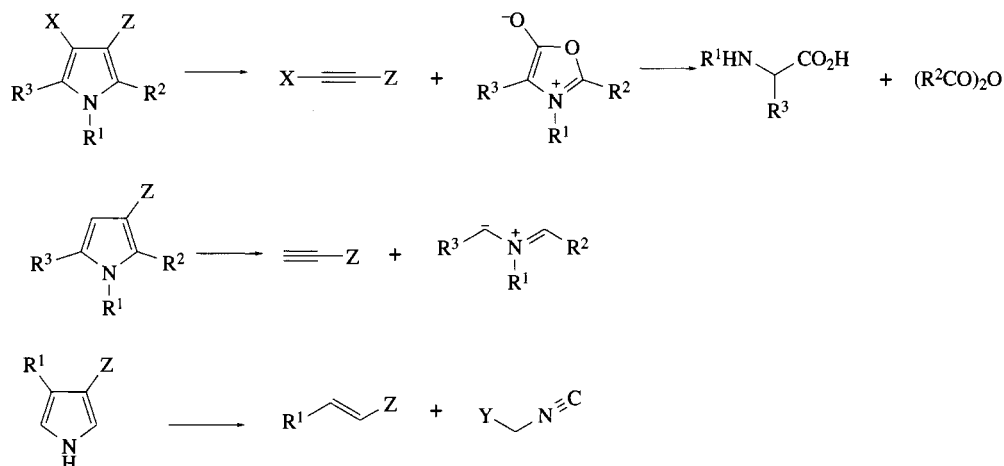
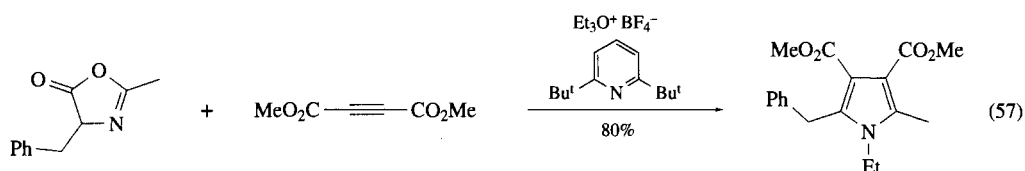
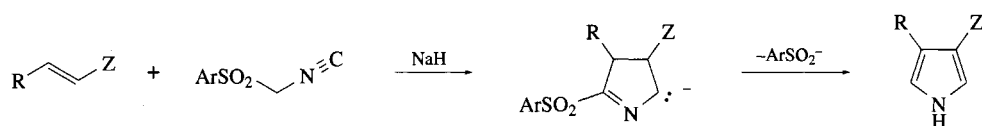


Figure 1 Examples of cycloaddition pyrrole syntheses.

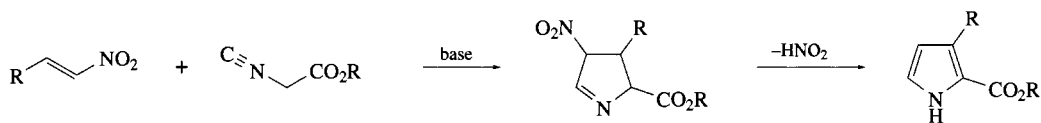


Anions of isocyanides, especially tosylmethyl isocyanide (TsMIC) and esters of isocyanoacetic acid, form pyrroles by addition to electrophilic alkenes and alkynes. For example, addition of TsMIC to acrylate esters in the presence of NaH is followed by cyclization at the isocyanide carbon leading to an intermediate which can aromatize by loss of sulfinate (Scheme 76) <80MI 203-01>.



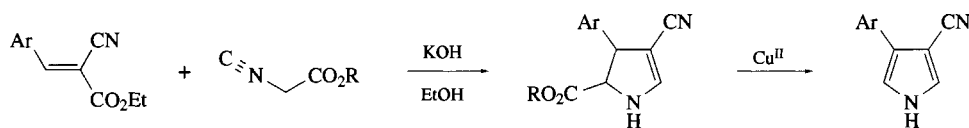
Scheme 76

Nitroalkenes are useful electrophiles for isocyanoacetate esters, with pyrroles being formed by elimination of nitrous acid (Scheme 77) <90T7587>.



Scheme 77

Addition to arylidene derivatives of cyanoacetate esters leads to adducts which can be aromatized and decarboxylated to 4-arylpyrrole-3-carbonitriles (Scheme 78) <92MI 203-03>.



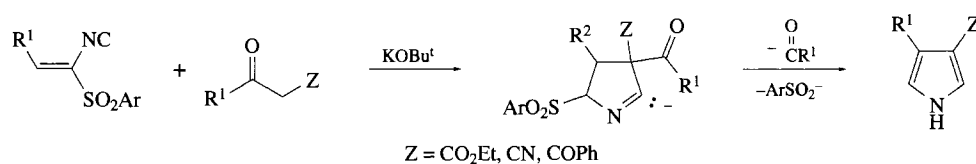
Scheme 78

Table 2 gives some additional examples of formation of pyrroles from isocyanides.

Table 2 Pyrroles by isocyanide cycloadditions.

Entry	Substituents	Reactant	Yield (%)	Reference
A. From α -isocyanoesters				
1	2-Ethoxycarbonyl-4-ethyl-3-methyl	3-Nitrohexene	80	92OS(70)68
2	2-Ethoxycarbonyl-3-(2-ethoxycarbonyl-ethyl)-3-methyl	Methyl 5-nitrohex-4-enoate	60	92TL6239
3	2-Ethoxycarbonyl-4-ethyl-3-trifluoromethyl	3-Nitro-1,1,1-trifluoropent-2-ene	65	89BCJ3386
4	2- <i>t</i> -Butoxycarbonyl-3-ethyl-4-methyl	3-Nitropent-2-ene	95	90T7587
5	2-Diethoxyphosphoryl-4-methyl-3-phenyl	2-Nitropropen-1-ylbenzene	58	93S473
B. From <i>p</i> -toluenesulfonylmethyl isocyanide (TsMIC)				
6	3,4-bis-(Ethoxycarbonyl)	Diethyl fumarate, KOBu ^t	44	91AJC323
7	3,4-Dicyano	Fumaronitrile	79	90JA2465
8	2-Cyano-3-ethoxycarbonyl	Ethyl β -cyanoacrylate NaH	54	94AJC975
9	4-Methyl-2-(4-methylphenylsulfonyl)-3-(4-methoxyphenyl)	1-(1-Nitropropenyl)-4-methoxybenzene	52	90T7587
10	2-Cyano-3-(2-furanyl)	Ethyl α -cyano-2-furanylideneacetate, KOH	92	87JAP61200984

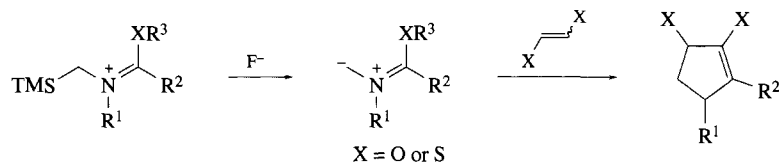
α -Tosylvinyl isocyanides also can serve as pyrrole precursors. They act as Michael acceptors towards enolates of β -ketoesters and similar acidic carbonyl compounds. The adducts are aromatized by deacylation and loss of sulfinate (Scheme 79).



Scheme 79

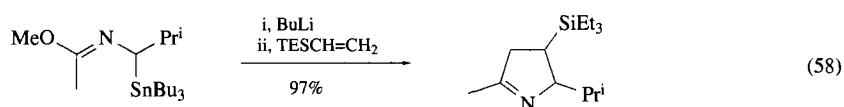
The reaction also proceeds for enolates of propiophenone and ethyl phenylacetate, in which case 3-methyl and 3-phenyl substituents are introduced (92JOC2245).

Several methods for making pyrroles involve *in situ* generation of azomethine ylides and anions. In the presence of electrophilic acetylenes these can be trapped and converted to pyrroles. One source of azomethine ylides are *N*-(α -trimethylsilylmethyl) derivatives of imidates and thioimidates. Azomethine ylides are generated by desilylation with fluoride ion. The salts are obtained by *N*-alkylation of imidate esters (83TL4303) or *S*-alkylation of thioacetamides (86JOC1997). The alkoxy or alkylthio groups are eliminated after cycloaddition. Electrophilic alkenes such as dimethyl maleate, dimethyl fumarate, maleonitrile and methyl acrylate give $\Delta^{2,3}$ -pyrrolenines (Scheme 80).



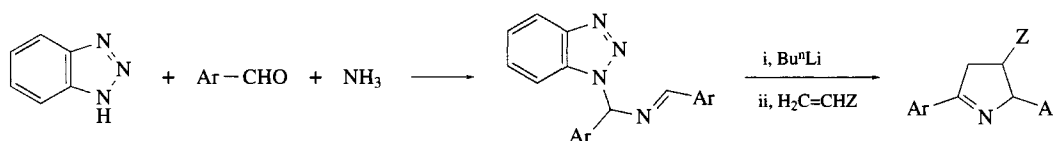
Scheme 80

Methoxy-substituted azomethine anions can also be generated by reaction of α -(tri-*n*-butylstannyl)imidate esters, which undergo lithium–tin exchange on reaction with alkyllithium reagents. The adducts eliminate methanol, generating $\Delta^{1,2}$ -pyrrolenines (Equation (58)) (94TL2641).



Benzotriazole also serves as a potential leaving group for generation of azomethine anions. Aromatic aldehydes and benzotriazole condense in the presence of ammonia to give α -benzo-

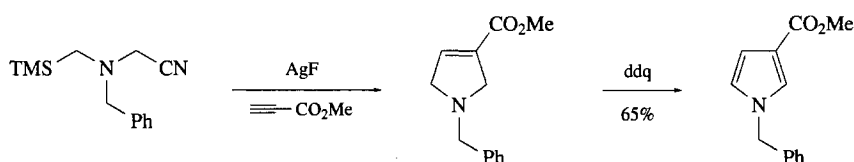
triazolylimines. These are deprotonated by *n*-butyllithium to generate anions which can then be trapped by electrophilic alkenes and alkynes (Scheme 81) <91S863>.



Scheme 81

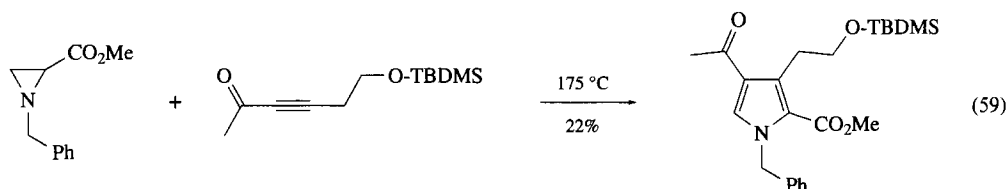
Dimethyl ethynedicarboxylate gives the expected pyrrole-3,4-dicarboxylate in essentially quantitative yield.

Azomethine ylides can also be generated from α -cyano- or α -phenylthiomethylamines <85JOC4006, 86CB813>. Silver fluoride promotes the elimination. The adducts from alkynes are 2,5-dihydropyrroles which can be oxidized to pyrroles by ddq (Scheme 82).

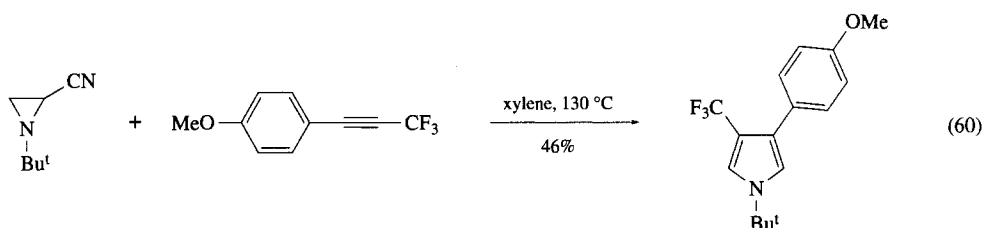


Scheme 82

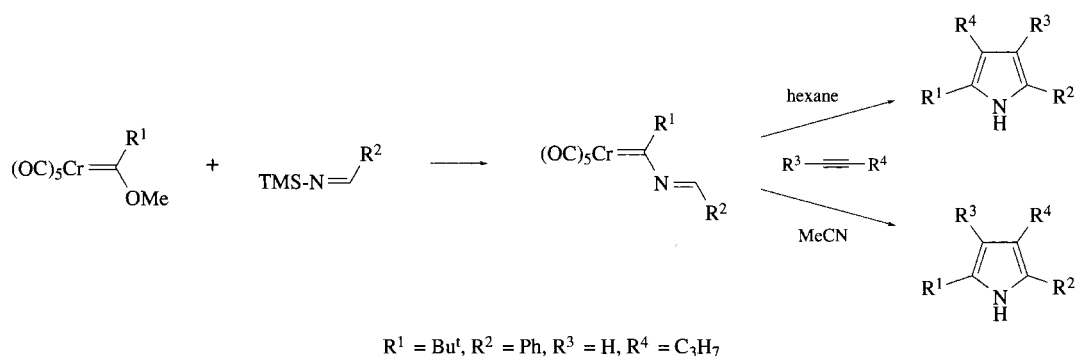
Azomethine ylides can also be generated by thermal ring-opening of aziridines. Pyrroles are obtained by addition to alkynic dipolarophiles <85JOC2309>. Better yields were obtained in intramolecular versions of this reaction (Equation (59)).



Azomethine ylides can also be trapped by aryl trifluoromethyl alkynes. When 1-*t*-butyl-2-cyanoaziridine was used, a pyrrole was formed by elimination of hydrogen cyanide <94S287>. Use of aziridine-2-carboxylate esters with this dipolarophile gave regioisomeric mixtures of dihydropyrroles which could be converted to pyrroles by oxidation with ddq (Equation (60)).



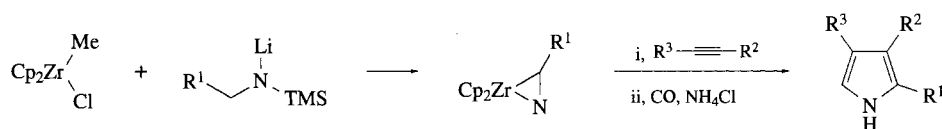
A [3 + 2] addition in which C3 and C4 are supplied by an alkyne uses iminocarbene complexes as the C2—N—C5 fragment. The carbene complexes are prepared from α -methoxycarbenechromium carbonyls (Fischer carbene complexes) by an exchange reaction with *N*-trimethylsilylimines <90JA1251>. Subsequent heating with the alkyne in hexane gives pyrroles in 80–97% yield. In nonpolar solvents the orientation of the addition is such that the smaller alkyne substituent becomes R⁴. Presumably this orientation is steric in origin. The regioselectivity is solvent dependent and in the more polar solvent acetonitrile, the opposite regioisomer is obtained, at least for terminal acetylenes (Scheme 83).



Scheme 83

2.03.2.9 Category IIIab Cyclizations

Zirconocene intermediates permit an unusual addition process which generates pyrroles from primary amines and alkynes with carbon monoxide serving as the C5 precursor (Scheme 84) <89JA776>.



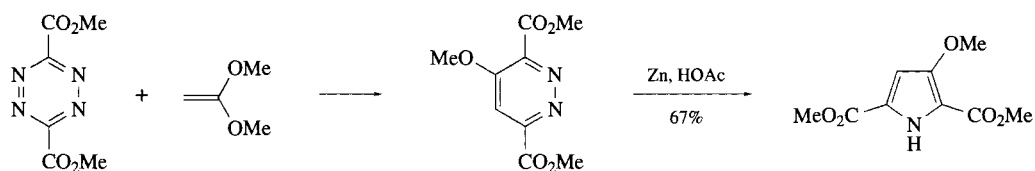
Scheme 84

The regioselectivity is governed by steric factors and the smaller alkyne substituent is found at the R^2 position. The yields are usually in the 40–60% range, but the method seems to be quite versatile with respect to the types of alkynes which can be used.

2.03.2.10 Syntheses Involving Contraction or Fragmentation of Existing Rings

There are a number of syntheses in which other heterocyclic rings serve as intermediates for the formation of pyrroles through reactions which involve ring contraction or fragmentation <89CHE237>. Most of these reactions are rather limited in scope but a few are versatile enough to have synthetic applicability.

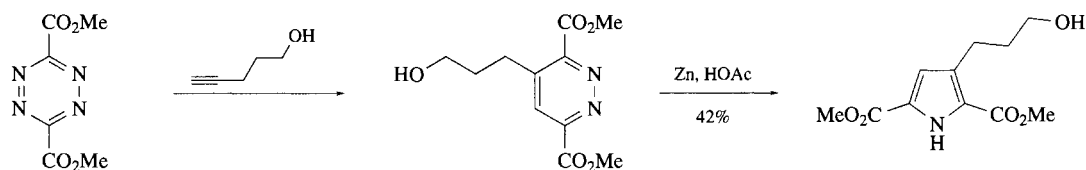
Pyrroles can be obtained by reduction of 1,2-diazines <80JMC481>. This reaction has been used in conjunction with inverse electron demand Diels-Alder reactions to prepare 3,4-disubstituted pyrrole-2,5-dicarboxylic acid derivatives. Silyl enol ethers or enamines serve as the electron-rich dienophiles. Silyl ethers of ester enolates give 3-methoxypyrroles (Scheme 85) <84JOC4405>.



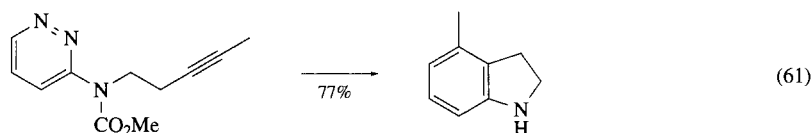
Scheme 85

The use of alkynes as the dienophile allows for the synthesis of 3-alkylpyrrole-2,5-dicarboxylate esters (Scheme 86) <88JOC1405>.

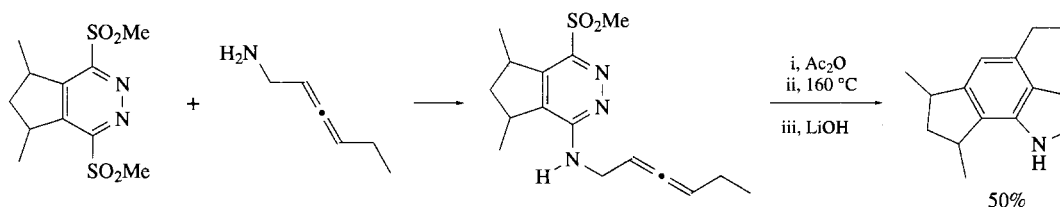
The carbocyclic ring of substituted indolines can be constructed by intramolecular Diels-Alder reactions of 2-(*N*-pent-3-ynylamino)pyridazines (Equation (61)) <84JOC2240>.



Scheme 86

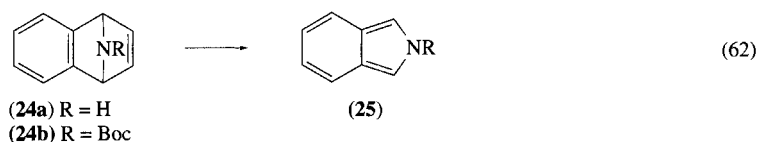


This synthetic strategy was successfully applied to the synthesis of structures of the type found in the antitumor compound CC1065 [88JA1321, 88JA4796]. Using 3,6-dimethanesulfonylpyridazine allows both for introduction of the nitrogen with its attached substituent by nucleophilic substitution and for ultimate aromatization by elimination of sulfinate. This strategy was employed in the synthesis of the substance trikentin, an antimicrobial indole isolated from a sponge (Scheme 87) [91JA4230].

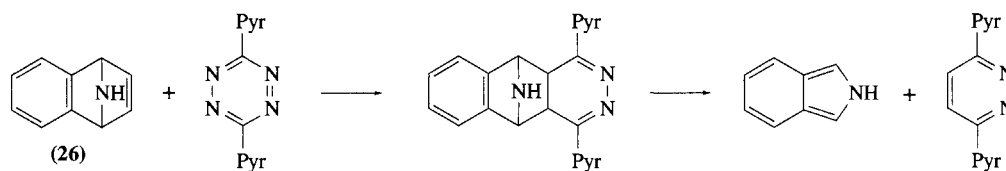


Scheme 87

Retrocycloadditions provide a route to some of the simpler isoindoles. 2*H*-Isoindole can be obtained by pyrolysis of (**24a**) at 600 °C [72CC1149]. The Boc derivative can also be used and gives isoindole by concomitant loss of the Boc group (Equation (62)) [79T1055].



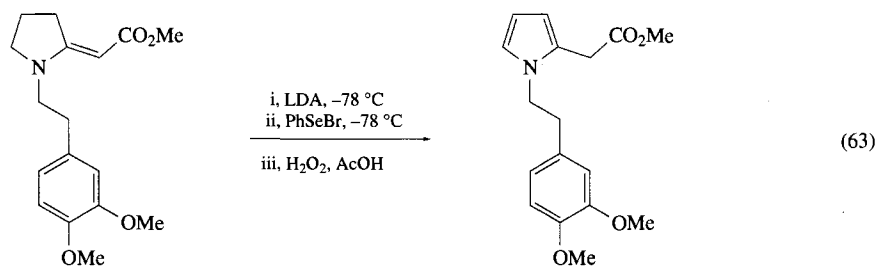
Cycloreversion occurs at a much lower temperature through pyridazines formed *in situ* from (**26**) and 3,4-dipyridyltetrazine (Scheme 88) [72TL4295].



Scheme 88

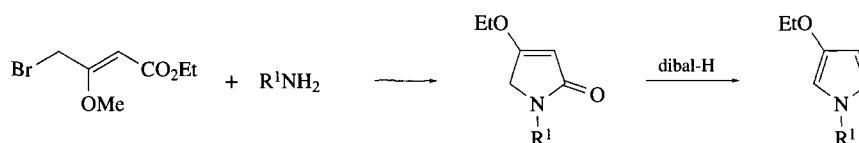
2.03.2.11 Syntheses by Oxidation/Reduction of Existing Rings

Although there are many syntheses of pyrrolidines, pyrrole synthesis is not usually approached through the saturated ring. There are some examples of syntheses in which dihydropyrroles are aromatized. α -Pyrrolidinylidene acetates, for example, can be aromatized by selenylation of the enolate followed by oxidative elimination of the selenoxide (Equation (63)) [75JOC800].



Aromatization by Pd/C has also been reported <76USP3957818>.

Reductive formation of pyrroles from pyrrolenones is also feasible. For example, dibal-H reduction of 4-alkoxy-3-pyrrolin-2-ones provides a route to 3-alkoxypyrroles (Scheme 89) <84JOC3222>.



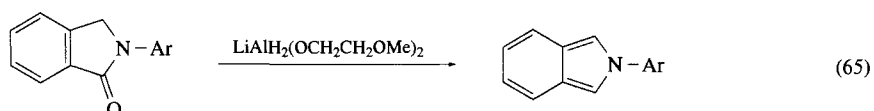
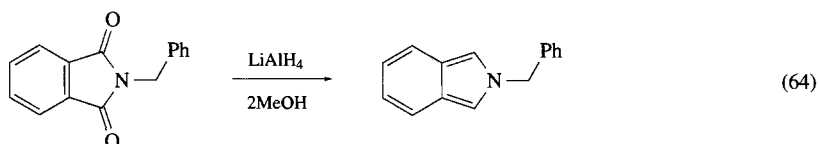
Scheme 89

Synthesis of indoles via 2,3-dihydroindoles (indolines) is sometimes done in order to achieve a specific substitution pattern in the carbocyclic ring <67RCR753>. Indolines, being aniline derivatives, readily undergo electrophilic substitution at C5. Indolines can also be used to achieve selective 7-substitution. The 1-Boc derivative of indoline can be lithiated at C7 <92H(34)1031> and 1-acetylindoline is thallated at C7 <89H(29)643>. These organometallic intermediates can be functionalized and then aromatized to indoles. There are a number of methods which have been developed for oxidative aromatization of dihydroindoles. Table 3 cites some examples.

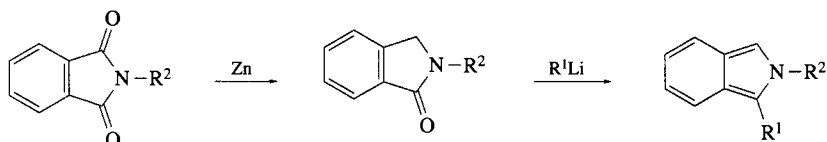
Table 3 Oxidative aromatization of indolines to indoles.

Entry	Substituents	Reagent	Yield (%)	Reference
1	None	Hg(OAc) ₂	72	88SC1331
2	None	R ₄ N ⁺ RuO ₄ ⁻ , <i>N</i> -Methylmorpholine- <i>N</i> -oxide	50–75	94TL6567
3	None	Iodosobenzene	38	88TL6917
4	2-Methyl	O ₂ , Co(salen)	79	80CL1287
5	5-Propyl	Mn(OAc) ₃	60	89JOC4350
6	1-Benzoyl-4-[2-(<i>N,N</i> -dipropylamino)ethyl]-5-methoxy	O ₂ , KOBu ^t	41	90JHC2093
7	3,4-Dihydrobenzo[<i>cd</i>]indol-5(1 <i>H</i>)one	(PhSeO) ₂ O	57	90JCS(P1)707
8	3-(1-Acetylpiperidin-4-yl)-5-nitro	MnO ₂	17	93CPB1589

Aromatization of existing rings is also an important means of synthesis of isoindoles. 2-Substituted 2*H*-isoindoles can be obtained by reduction of phthalimides or phthalimidines. 2-Benzyl-2*H*-isoindole can be prepared by reduction of *N*-benzylphthalimide using LiAlH₂(OMe)₂ (Equation (64)) <88JOC2565>. Reduction of *N*-arylphthalimidines with LiAlH₂(OCH₂CH₂OMe)₂ gives *N*-aryl-2*H*-isoindoles in 70–80% yields (Equation (65)) <88CHE1308>.

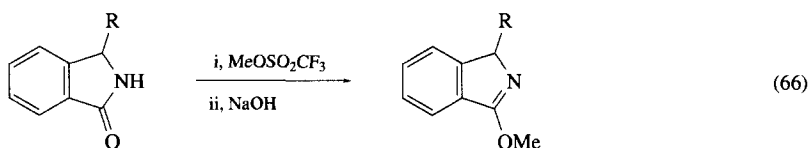


Addition of organolithium reagents to phthalamidines gives isoindoles with a 1-substituent derived from the lithium compound <79JOC1519>. The phthalamidines can be prepared in a number of ways, including partial reduction of phthalimides by zinc (Scheme 90).



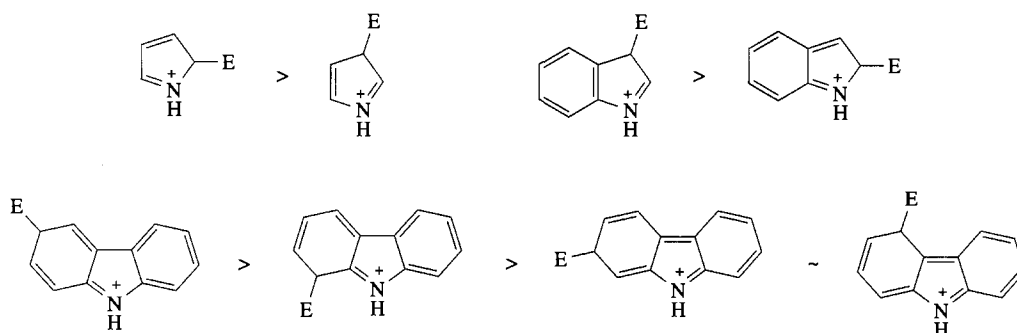
Scheme 90

3-Alkoxy-1*H*-isoindoles can be prepared by *O*-alkylation of phthalamidines using tri-alkyloxonium salts or alkyl triflates (Equation (66)) <82S842, 89ZN(B)1132>.



2.03.3 INTRODUCTION AND MODIFICATION OF SUBSTITUENT GROUPS

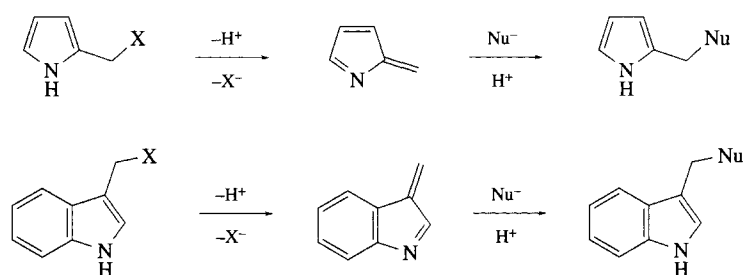
The preceding section focused on the formation of pyrrole, indole, isoindole and carbazole rings. This section will discuss the introduction of substituents on existing rings, with primary attention on addition or modification of carbon substituents. Many of the methods for introduction of substituents involve electrophilic substitution. Such reactions are generally favorable because of the relatively high reactivity of the heterocycles toward electrophiles. Electrophilic aromatic substitution is subject to directive effects. The inherently preferred positions are 2 > 3 for pyrrole, 3 > 2 > others for indole, and 3,6 > 1,8 > others for carbazole. These orientational effects can be rationalized in terms of qualitative resonance arguments about the stability of the addition intermediates and are supported by theoretical analysis (Scheme 91) <84JA421, 91JA4550, 93JST(288)199>.



Scheme 91

Electron-withdrawing (EW) groups superimpose their effect on the reactivity of the ring. In pyrrole, 3-EW substituents favor 5-substitution while 2-EW groups favor 4-substitution. In indole, 3-EW groups can make carbocyclic substitution (at C5 or C6) competitive with C2. Electron-withdrawing groups at C2 in indole attenuate the reactivity at C3, but it remains the dominant position for substitution.

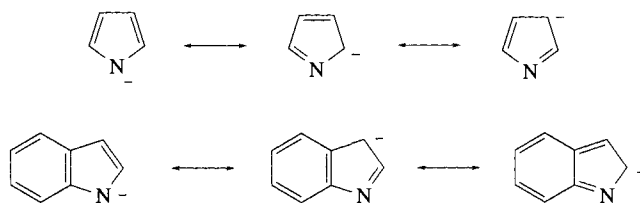
Another important method for modification of pyrroles and indoles involves substitution at the α -position on a side-chain. Displacement of leaving groups is exceptionally facile and these reactions frequently occur by an elimination-addition sequence in which an alkylidenepyrrolenine or alkylideneindolenine is formed (Scheme 92).



Scheme 92

2.03.3.1 N(1)-Substitution Including Introduction and Removal of Protecting Groups

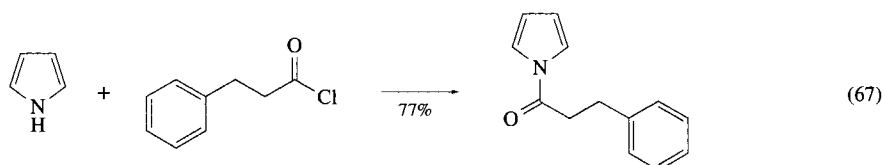
Introduction of 1-substituents is an important reaction, not only in its own right, but also as a means of nitrogen-protection during reactions at other positions on the heterocycle. Besides avoiding unwanted reactions at nitrogen, protecting groups can also serve to moderate the reactivity of the ring by electron-withdrawal or, in the case of metallation, by coordination effects (see Section 2.03.4.1). Bulky nitrogen substituents can influence reactivity by steric effects. The nitrogen atom in neutral pyrroles, indoles and carbazoles is very weakly nucleophilic since the lone-pair electrons are part of the delocalized aromatic π -sextet. However the corresponding anions are reactive nucleophiles. Pyrrole, indole and carbazole are all weakly acidic with pK_s (referenced to water) quoted as 17.2, 16.7 and 15.2, respectively <89T7501>. In DMSO the pK values are 23.0, 20.9 and 19.9 <91JOC3216>. The aromatic heterocycles are thus much more easily deprotonated than saturated amines whose pK_s are >30 . Synthetic procedures for introduction of nitrogen substituents on pyrrole, nitrogen and carbazole nearly always use a strong base to provide the reactive anion as the nucleophile. Reactions with acylating, sulfonylating and alkylating agents, including Michael acceptors, are facile under basic conditions. The anions are ambident nucleophiles with the regioselectivity dependent upon the solvent and the degree of cation coordination. The nitrogen atom is the site of harder nucleophilicity (Scheme 93).



Scheme 93

Alkyl groups are easily introduced on to pyrroles and indoles but most are not easy to remove. The benzyl, methoxymethyl and 3-trimethylsilylethoxymethyl are among those for which deprotection methods are available. Among the most useful protecting groups for pyrroles and indoles are acyl, alkoxycarbonyl, carbamoyl, sulfonyl and silyl substituents. All except the last are EW substituents which not only prevent reaction at nitrogen but also diminish the reactivity of the ring. This is frequently advantageous because of the high susceptibility of the unprotected rings to protons, electrophiles and oxidants.

N-Acyl groups can be introduced by reaction of pyrrole or indole anions with acyl halides in THF <87T3689, 88CPB3770, 88JOM209> or under phase transfer conditions (Equation (67)) <81S461>.



The EW-effect of the acyl groups makes the pyrrole and indole ring more comparable to benzene

in terms of stability and reactivity towards electrophiles. *N*-Acyl groups are relatively easily removed by alkaline hydrolysis. The reactivity is the consequence of weakened 'amide resonance' which is due to the involvement of the nitrogen lone-pair in the heterocyclic conjugation.

The *t*-butoxycarbonyl (Boc) group is the most frequently used of the alkoxycarbonyl groups. It was introduced as an indole protecting group in the context of lithiation <81JOC157> but is useful for other reactions, too, so long as acidic conditions are avoided. The Boc group can be introduced on pyrrole or indole using di-*t*-butyl dicarbonate and dmap, <84AG(E)296, 85TL6141> or with *t*-butoxy phenyl carbonate and NaH <84JOC4203, 86JCS(P1)2181>. The Boc group can also be introduced under phase transfer conditions <92S95>. Phase transfer conditions have also been used to introduce benzyloxycarbonyl (Cbz) and phenoxycarbonyl groups. The phenoxycarbonyl derivatives can be converted to other alkoxycarbonyl groups by alkoxide exchange (Equation (68)) <86JCS(P1)2181, 92S95>.



In addition to using the traditional acidic conditions, it is possible to remove Boc groups by pyrolysis. Heating the protected pyrroles or indoles to 180–185 °C frequently results in clean deprotection <85TL6141>. Other alkoxycarbonyl groups are usually removed by alkaline hydrolysis.

Both arenesulfonyl and methanesulfonyl groups have proven useful in the protection of pyrroles and indoles. Sulfonyl groups can be introduced by reaction of the heterocyclic anion with a sulfonyl halide in DMSO <73JOC3324> or under phase transfer conditions <79S136>. As long as the rest of the molecule is stable to aqueous base, sulfonyl groups can be removed by alkaline hydrolysis <73JOC3324>. Arenesulfonyl groups are also susceptible to reductive removal. Among the conditions which have been applied successfully are sodium amalgam in buffered methanol, <81JOC4836> sodium naphthalenide, <87JMC2051> and photoactivated reduction by NaBH₄ <87JA4335>.

Trialkylsilyl groups can be introduced on pyrrole by reaction of the appropriate silyl chloride with the anion of pyrrole <82JCS(P1)1833>. The triisopropylsilyl (tips) group has been introduced using pyrrole anion generated with BuⁿLi, NaH or potassium sand <89CB169, 90JOC6317>. The *t*-butyldimethylsilyl (TBDMS) group can be introduced using NaH as the base in THF <86JCS(P1)2181>. The trimethylsilyl (TMS) and TBDMS groups have also been introduced on indole by use of the corresponding trichloroacetates. Reaction occurs at 100 °C in the presence of K₂CO₃ and 18-crown-6 <86TL4995>.

The 1-tips derivative of pyrrole has proven to be a useful intermediate for synthesis of 3-substituted pyrroles. The steric bulk of the substituent blocks the 2-position so that electrophilic substitution, including acylation <90JOC6317>, bromination <84JOC3239, 89CB169>, and nitration <90JOC6317> take place at C3. Similar steric directing effects have also been reported for the TBDMS <85TL5035> and triphenylmethyl groups <83JCS(P1)93>.

There has been some use of alkoxymethyl protecting groups. Unlike acyl, alkoxycarbonyl and sulfonyl substituents, alkoxymethyl groups do not reduce the reactivity of the pyrrole and indole rings toward electrophiles. In contrast to saturated acetals and amins, the alkoxymethyl substituents on a pyrrole or indole ring require fairly vigorous conditions for hydrolytic removal <73JOC3324>. This may be a reflection of the effect of delocalization of the nitrogen lone-pair, which in saturated amins can participate in acid-catalyzed hydrolysis. Perhaps the most useful of the alkoxymethyl groups is the 2-trimethylsilylethoxymethyl group (SEM). It can be introduced on to pyrrole or indole using NaH followed by alkylation by the chloride <84JOC203, 84JOC3503>. The group can be removed with tetra-*n*-butylammonium fluoride in THF <84JOC3503>.

Aminoalkyl groups have some of the same characteristics as alkoxymethyl groups, for example, in being able to direct lithiation to C2 (see Section 2.03.4.1). 1-Dimethylaminomethylindole can be prepared directly from indole by reaction with formaldehyde and dimethylamine at 0–5 °C <66CB889>. This regioselectivity contrasts to the 3-substitution which occurs under acidic conditions. However, the dimethylamino group is rather resistant to acidic or basic hydrolysis <89H(29)849>. Interestingly, NaBH₄ in refluxing ethanol achieves reductive deprotection <90JOC3688>. The pyridylethyl group can be introduced on to pyrrole or indole by conjugate addition to 4-vinylpyridine using alkoxide ions as base. This group can be removed by quaternization with methyl iodide followed by basic elimination in acetone–water <87JHC641>.

N-Alkylation of pyrroles, indoles and carbazoles is an important synthetic reaction in its own

right. *N*-Alkylation is very general for reagents capable of reacting by either S_N2 substitution or conjugate addition. Besides the conditions mentioned for protecting group introduction, such as the use of NaH in THF, anions of pyrrole and indole can be generated using NaNH_2 in liquid ammonia. For example, 1-methylindole can be prepared easily by this method <73OSC(5)769>. Phase transfer conditions are also frequently excellent for alkylation of pyrroles and indoles by primary halides and sulfonates and are marginally effective for secondary groups <76S124, 76S414, 79S617, 80JOC3172, 83T2669, 84JCR(S)88>. Powdered KOH in acetone or DMSO can be used for alkylation of pyrrole or indole, as well as *C*-alkyl derivatives <73JCS(P1)499, 81S461>. Table 4 gives some examples of alkylation procedures.

Table 4 Nitrogen-alkylation of pyrroles, indoles, and carbazoles.

Entry	Substituents	Conditions	Yield (%)	Reference
A. Pyrroles				
1	1-(Carboxymethyl)-2-formyl	$\text{BrCH}_2\text{CO}_2\text{H}$, NaOH	63	78JMC962
2	1-(2-Aminoethyl)	$\text{ClCH}_2\text{CH}_2\text{NH}_2$, NaOH R_4N^+ HSO_4^-	93	92T8793
3	1-Butyl-2-methyl-3,4-diphenyl	$\text{Br}(\text{CH}_2)_3\text{CH}_3$, R_4N^+ Br^-	66	93AP157
B. Indoles				
4	1-Methyl	CH_3I , NaNH_2 , $\text{NH}_{3(l)}$	85–95	73OSC(V)769
5	1-Methyl	CH_3I , NaH, DMF	86	77JOC1784
6	1-Benzyl	PhCH_2Br , DMSO, KOH	95	73JCS(P1)499
7	1-Ethyl	$\text{C}_2\text{H}_5\text{I}$, acetone, KOH	88	81S461
C. Carbazoles				
8	9-Benzyl-3,6-dibromo	PhCH_2Cl , K_2CO_3	81	89JOC965
9	1-(Methoxycarbonylmethyl)	$\text{BrCH}_2\text{CO}_2\text{CH}_3$, K_2CO_3 , DMF	91	93JA3548

One of the general issues which must be considered in pyrrole and indole alkylation is that of carbon- versus nitrogen-alkylation. *N*-Alkylation is favored by strong nucleophilicity at nitrogen. *N*-Selectivity is favored by reaction conditions, such as highly polar solvents and weakly coordinating cations, which minimize the coordination of the cation at nitrogen. When the nitrogen atom is neutral or is tightly coordinated to a metal cation, reaction is more likely to occur at a carbon atom. The more reactive the alkylating agent, the more likely that *C*-alkylation will occur. For example, the magnesium salts of pyrrole and indole react with allylic halides to give more carbon- than nitrogen-alkylation. There have been a number of studies of the factors effecting nitrogen- versus carbon-alkylation of pyrroles and indoles. Table 5 shows some data from a recent study <90JCS(P1)111>. The trends observed are similar to those in other studies. These alkylations were performed in THF, a solvent which has only moderate capacity to dissociate ion pairs. The potassium and sodium salts are primarily *N*-alkylated under these conditions but lithium and magnesium salts show higher ratios of substitution at carbon (C2 for pyrrole and C3 for indole). Comparing *n*-butyl tosylate with the iodide shows that the tosylate has a slightly greater tendency for *N*-alkylation. Allyl bromide has a greater tendency for *C*-alkylation than the saturated alkylating agents.

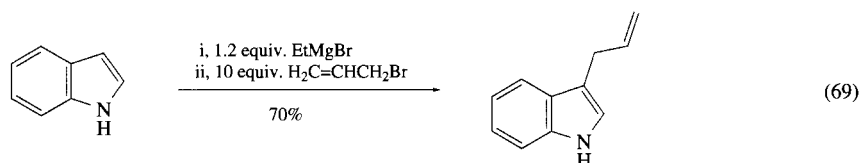
Table 5 Nitrogen-versus carbon-alkylation of pyrrole and indole.

Counterion	Solvent	Alkylating agent	N : C ratio
A. Pyrrole			
K^+	THF	<i>n</i> -Butyl iodide	100 : 0
Li^+	THF	<i>n</i> -Butyl iodide	4 : 96
K^+	THF	<i>n</i> -Butyl tosylate	100 : 0
Li^+	THF	<i>n</i> -Butyl tosylate	17 : 83
B. Indole			
K^+	THF	<i>n</i> -Butyl iodide	97 : 3
Li^+	THF	<i>n</i> -Butyl iodide	25 : 75
K^+	THF	<i>n</i> -Butyl tosylate	100 : 0
Li^+	THF	<i>n</i> -Butyl tosylate	74 : 26
Na^+	THF	Allyl bromide	65 : 35
Mg^{2+}	THF	Allyl bromide	2 : 98

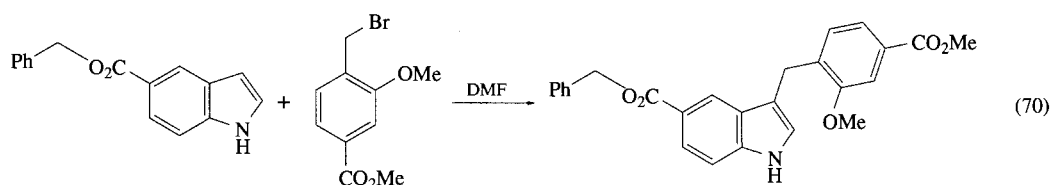
Similar trends were seen in a study of N1 versus C3 alkylation of indole under phase transfer conditions. These conditions involve a weakly coordinating counterion so that *N*-alkylation should be preferred. Benzyl chloride gives a higher N:C ratio than benzyl bromide, reflecting the generalization that more reactive alkylating agents will tend to favor *C*-alkylation <90H(31)447>.

2.03.3.2 Direct Carbon-alkylation

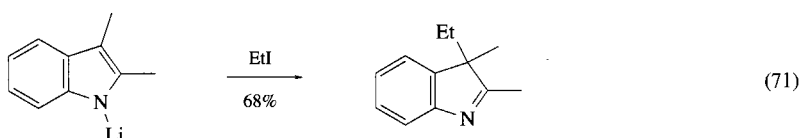
Direct carbon-alkylation of indole can be achieved by reaction of a magnesium halide salt of indole with a reactive halide such as allyl bromide. For example, indole can be converted to 3-allylindole in 70% yield (Equation (69)) <52JCS3172>.



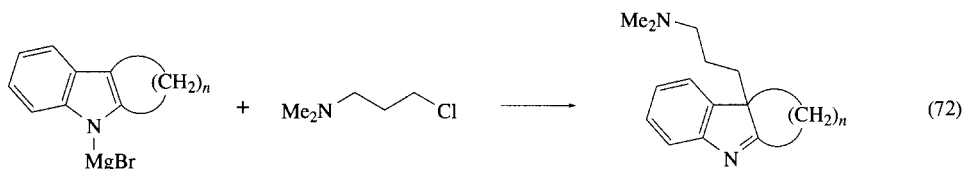
The *C*-alkylation of indoles by benzylic halides can be achieved by reaction with the neutral indole in DMF (Equation (70)) <93JMC394>.



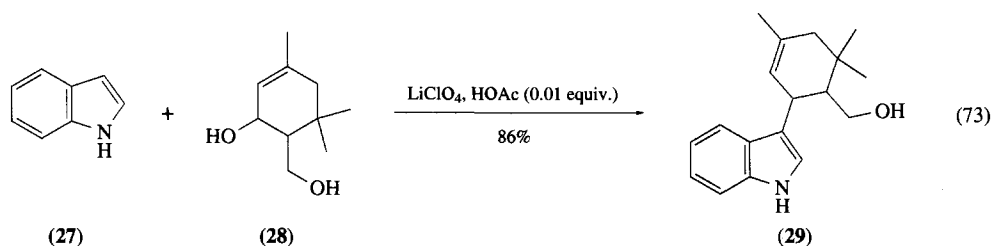
The lithium salt of 2,3-dimethylindole reacts in THF with primary iodides to give 3-substituted 3*H*-indoles. Interestingly, the reaction is dependent on the concentration of the salt, implying that an aggregated species may be involved (Equation (71)) <91H(32)685>.



Conditions for *C*-alkylation can also be applied to certain functionalized alkylating agents (Equation (72)) <91JHC1293>.

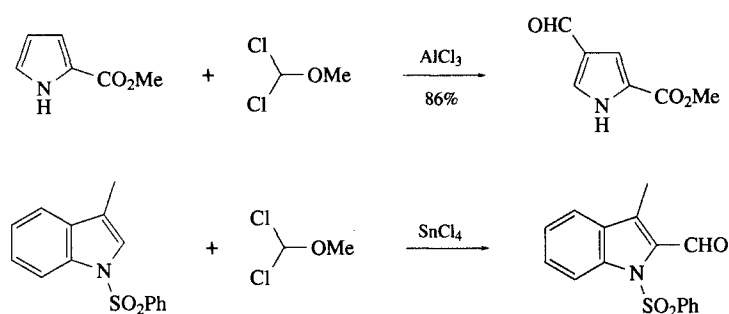


Carbon-alkylation is also observed under conditions where a carbocation is generated as the active electrophile. Reaction of pyrrole with *t*-butanol in the presence of a strongly acidic exchange resin gives a mixture of 2- and 3-(*t*-butyl)pyrrole in 1.6–1.7:1 ratio and 60–70% yield <88SC1261>. Reaction of ethyl bromide or isopropyl bromide with 1-phenylsulfonylpyrrole in the presence of AlCl₃ gives a mixture of 2- and 3-alkylation products. *t*-Butyl chloride/AlCl₃ gives an excellent yield of 3-(*t*-butyl)pyrrole <85CJC896>. Indole reacts with allylic alcohols in the presence of 3 M LiClO₄ and a small amount of acetic acid to give 3-alkylated products. This reaction presumably involves the generation of an allylic perchlorate ion pair and reaction occurs at the less substituted allylic terminus. For example, reaction of indole with (28) gives (29) which was used as an intermediate in the preparation of the diindolylterpene yuehchukene (Equation (73)) <93CC510>.



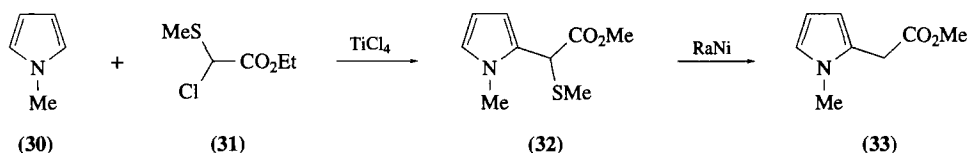
Carbazole is converted to 1,3,6,8-tetra-(*t*-butyl)carbazole in quantitative yield on refluxing in *t*-butanol/TFA. Under the same conditions *N*-methylcarbazole gives a high yield of the 3,6-di-(*t*-butyl) derivative <90CHE158>.

Relatively stabilized carbocations can be used to alkylate the pyrrole and indole rings. Introduction of formyl groups, for example, can be done with MeOCHCl_2 , via a reaction which likely proceeds through the 1-chloro-1-methoxymethyl cation, (Scheme 94) <72JMC97, 86SC1799>.



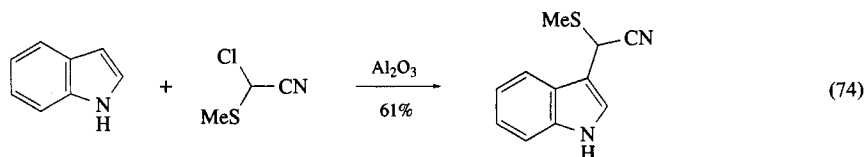
Scheme 94

Ethyl 2-chloro-2-methylthioacetate reacts with 1-methylpyrrole to give (32) which can be desulfurized to ethyl 1-methylpyrrole-2-carboxylate (Scheme 95) <82CPB915>.



Scheme 95

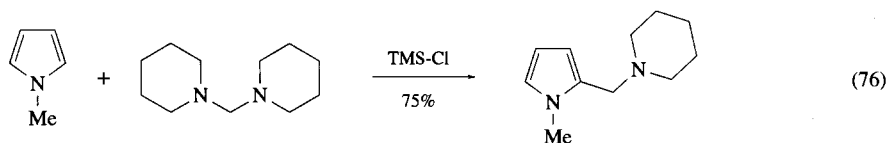
Similar reactions can be conducted with indole and 1-chloro-1-methylthioacetonitrile in the presence of alumina (Equation (74)) <92JCS(P1)2821>.



Mannich condensations of pyrroles and indoles are especially useful examples of alkylation by stabilized carbocations, in this case iminium ions. The traditional conditions involve reaction between the heterocycle, formaldehyde and a dialkylamine in acidic solution, as in the preparation of gramine (Equation (75)) <37CB567>.

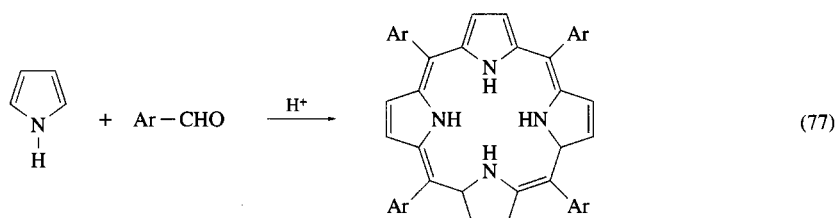


Iminium ions can also be generated from α -aminoethers or bis-(dialkylamino)methanes by reaction with TMS-Cl (Equation (76)) <88CC1161>.

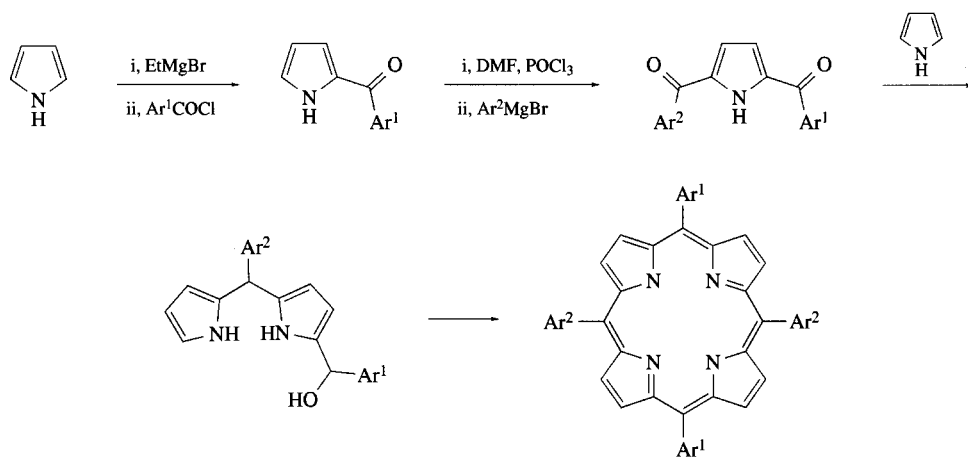


Similar reactions occur in acetonitrile using acetyl chloride or SO_2 to generate the active electrophile <88TL2997>.

The conversion of pyrrole to porphyrins involves a series of electrophilic substitutions and aromatizing oxidation. Condensation of pyrrole and aldehydes has been used extensively in the construction of porphyrin rings <87MI 203-01>. For aromatic aldehydes, heating with pyrrole in hot xylene with 3% $\text{ClCH}_2\text{CO}_2\text{H}$ provides tetraaryl porphyrins from a variety of substituted benzaldehydes (Equation (77)) <67JOC476, 86CHE629>.

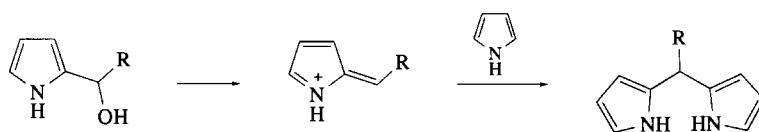


5,10,15,20-Tetrasubstituted porphyrins have also been obtained by starting with 2-acylpyrroles. The procedure involves reduction to the carbinol using a limited amount of LiAlH_4 , followed by condensation in propanoic acid with $\text{Zn}(\text{OAc})_2$ <89TL2411>. Differentially substituted porphyrins can be built up by a series of acylations and organometallic additions which generate the desired pyrrolylcarbinols (Scheme 96) <90TL7265>.



Scheme 96

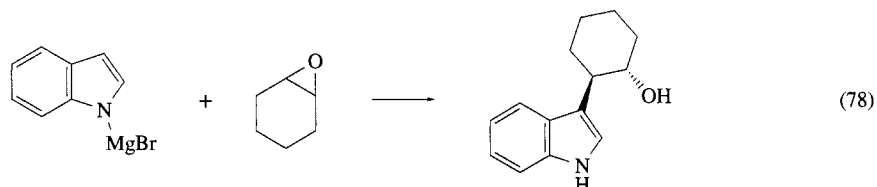
All of these reactions involve a 2-pyrrolylcarbinol acting as the precursor of an electrophilic alkylidenepyrrolenine which attacks another pyrrole molecule. The porphyrin skeleton is eventually reached when four pyrroles have condensed (Scheme 97).



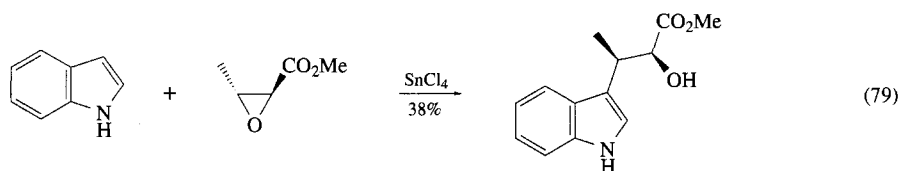
Scheme 97

Under similar conditions indoles give diindolylmethanes, since the reaction can not proceed beyond dimerization <78JHC433>.

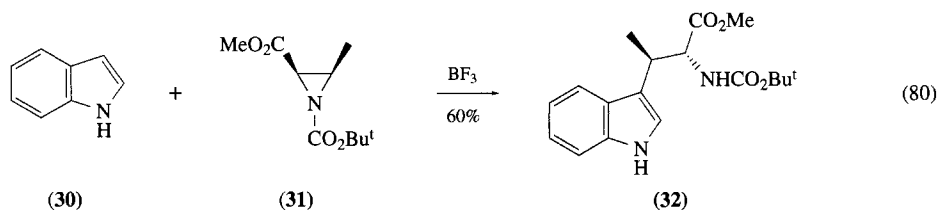
Epoxides and aziridines can also act as alkylating agents toward indoles. For example, indole is alkylated by cyclohexene oxide (Equation (78)) <91TL3779>.



Methyl 2(*S*),3(*R*)-epoxybutyrate reacts with indole under the influence of SnCl_4 to give a 3-substitution product. The reaction proceeds with inversion of configuration at C3 of the butyrate (Equation (79)) <90CPB323>.



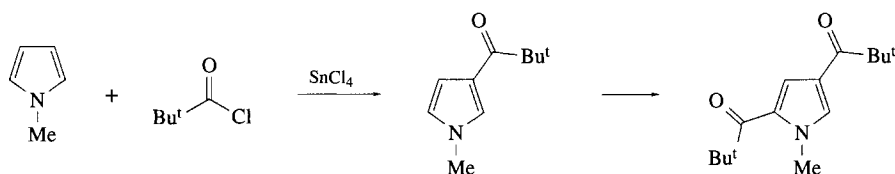
Alkylation by aziridines is of interest because it can introduce the 2-aminoethyl group which is found in many biologically active indoles. Zinc triflate was found to be the best catalyst for effecting enantiospecific alkylation of indoles by 1-(Cbz)aziridine-2-carboxylate esters <89TL4073>. Aziridine (**31**) was used to stereospecifically introduce an α -methyltryptophan side-chain using BF_3 as the catalyst (Equation (80)) <90CPB564>.



2.03.3.3 Carbon-acylation

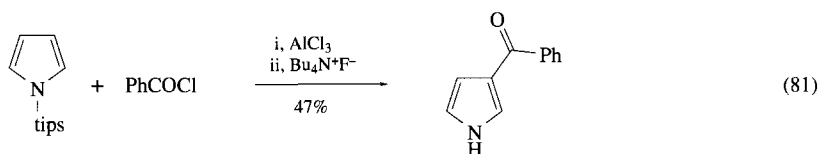
Direct carbon-acylation is a very useful synthetic method for the introduction of carbon substituents on both pyrroles and indoles. The rings are reactive under relatively mild acylation conditions and the acyl groups stabilize and deactivate the rings so that yields of mono-substitution products are often excellent. Pyrroles and indoles already containing EW substituents can be used because the rings remain reactive toward substitution. Among conditions which have proven most successful from a synthetic point of view are Lewis acid-catalyzed reactions, reactions of acyl halides with magnesium salts of the heterocycles and Vilsmeier-Haack conditions involving amides and POCl_3 .

1-Methylpyrrole reacts with pivaloyl chloride and SnCl_4 to give mainly the 3-acyl and 2,4-diacyl products. The diacyl product arises by subsequent acylation of the initial product. The preference for initial 3-acylation is the result of a steric effect and illustrates that it is relatively easy to overcome the normal 2 > 3 preference for acylation (Scheme 98) <87JOC2209>.

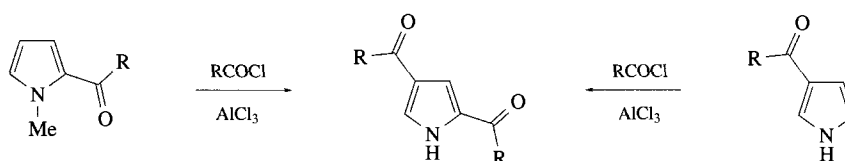


Scheme 98

Pyrroles with bulky *N*-substituents, for example 1-tips-pyrrole, undergo acylation at the 3-position. Highly reactive acylating agents such as ethyl oxalyl chloride and trichloroacetyl chloride require no catalyst. For other acid chlorides AlCl_3 is required (Equation (81)) <90JOC6317>.

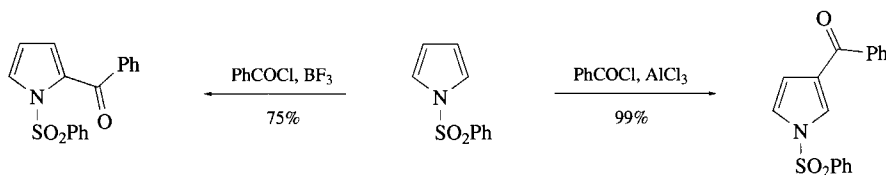


Friedel–Crafts acylations are subject to some regiochemical control based on the presence of EW-substituents which both attenuate reactivity and influence position selectivity. 2-Acylpyrroles are acylated mainly at C4 while 3-acylpyrroles undergo acylation at C5, both leading to 2,4-diacylation (Scheme 99) <93JCS(P1)273>.



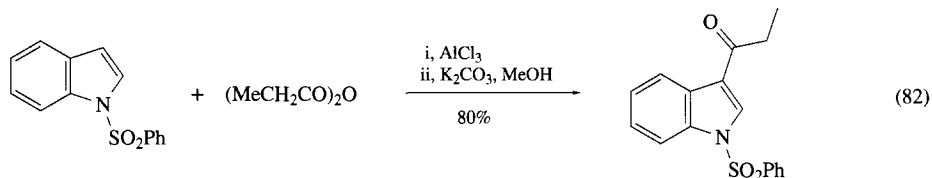
Scheme 99

1-Phenylsulfonylpyrroles can provide either 2- or 3-acylation products. Acyl chlorides and AlCl_3 give mainly the 3-substitution products while with BF_3 , acyl chlorides or anhydrides gives 2-substituted derivatives <83JOC3214, 93JOC7899>. The mechanistic basis of this selectivity may lie in a difference in the electrophilicity of the intermediates involved in the two procedures, with AlCl_3 generating a more reactive species. Alternatively, the selectivity may reflect a steric and/or chelation effect originating in the phenylsulfonyl substituent (Scheme 100).

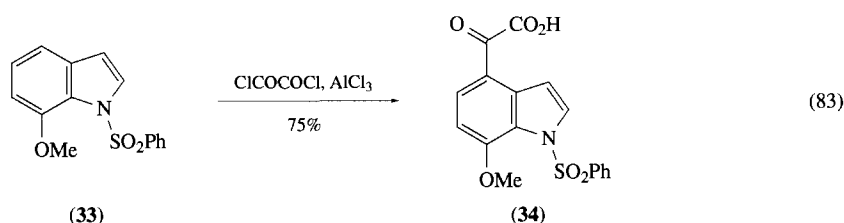


Scheme 100

1-Phenylsulfonylindole is a useful reactant for preparation of 3-acylindoles. High yields can be obtained with acid anhydrides or chlorides using AlCl_3 in CH_2Cl_2 <85JOC5451>. The phenylsulfonyl group can be readily removed by alkaline methanolysis (Equation (82)).

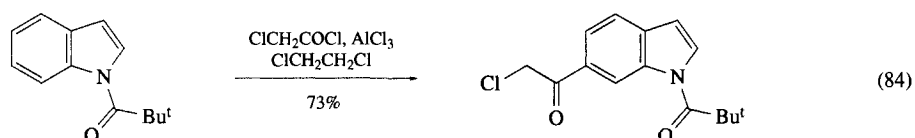


The combination of a 1-phenylsulfonyl and a 7-methoxy substituent makes C4 the preferred position for acylation of (33) by oxalyl chloride (Equation (83)) <93SC2717>.



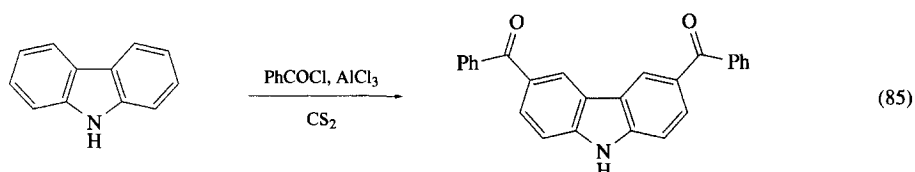
The Friedel–Crafts acylation of ethyl indole-2-carboxylate has been compared under a number of conditions with a variety of acylating agents. While several acid chlorides, catalyzed by AlCl_3 , give 3-acylation, others give substantial amounts of 5- and 7-substituted products (88CPB2023, 90CPB3261). Both the solvent and the Lewis acid catalyst can effect the product composition, but the overall patterns are not clear. In general terms, milder Lewis acids tend to favor 3-substitution while use of excess AlCl_3 in CH_2Cl_2 promotes 5-substitution.

3-Acetylindole and ethyl indole-3-carboxylate are acylated primarily at C5 under Friedel–Crafts conditions, but there is also some 6- and 7-substitution (82CPB2349). 1-Acetyl-, 1-pivaloyl- and 1-methoxycarbonylindole can all be acylated in quite good yield at the 6-position by chloroacetyl chloride in the presence of AlCl_3 (Equation (84)). In contrast, acetyl chloride under the same conditions gives the 3-substitution product. Longer chain α -halocarbonyl chlorides also show C6 selectivity (94TL2699). The cause of this regioselectivity is uncertain.

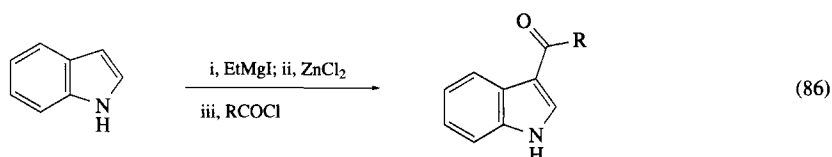


Selective acylation of the indole ring at C2 can be achieved by using a trimethylsilyl group as an *ipso*-director (89JOC4350). Similarly, 1-acetyl-4-TMS-indole undergoes acylation at C4 (84JOC4409). These results indicate some of the ways substituents can be used to control the position of acylation of indole. But, as with pyrroles, it appears that selectivity of the acylation is also affected by the precise structure of the acylating reagent.

The benzoylation of carbazole under Friedel–Crafts conditions gives mainly the 3,6-dibenzoyl derivative, although some of the 1- and 3-monosubstituted products are also found (Equation (85)) (91JHC1035).



An alternative approach to acylation of pyrroles and indoles is by reaction of a magnesium salt with an acid chloride or acid anhydride (Equation (86)) (86CZ307). This procedure involves conversion of the heterocycle to the anion by reaction with a Grignard reagent (87JOC2209, 88JHC469). Recent work suggests that the zinc salts, which can be prepared from the magnesium salts, may be advantageous (90T6061). The zinc salts evidently have reduced reactivity and there is less competing *N*-acylation.



The Vilsmeier–Haack reaction is a reliable and relatively mild method for introduction of formyl and acyl groups at C2 of pyrrole and C3 of indole. The reaction can be used to introduce a formyl group at C2 of pyrroles using DMF and POCl_3 (63OSC831, 93JMC101) or an acyl group (89S783, 93JOC7245). Excellent yields of 3-formylindoles are obtained under these conditions (54JCS3842, 92SC2077). Vilsmeier reagent prepared from DMF and oxalyl chloride has also been used (94SC1389). *N*-Methylformanilide and POCl_3 were the preferred reagents for formylation of 9-benzylcarbazole at C3 (92CPB230). Table 6 provides some examples of acylation procedures.

2.03.3.4 Conversion of Acyl Substituents to Alkyl Groups

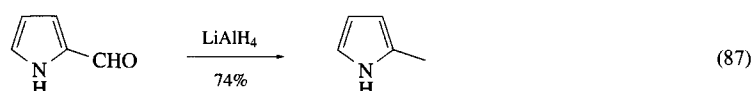
The reductive deoxygenation of acyl substituents is one of the classic methods for introduction of primary alkyl groups on aromatic rings. For benzene derivatives, the Wolff–Kishner and Clem-

Table 6 Acylation of pyrroles and indoles.

Entry	Substituents	Conditions	Yield (%)	Reference
A. Under Friedel-Crafts conditions				
Pyrroles				
1	3-Propanoyl-1-phenylsulfonyl	Propanoyl chloride, AlCl ₃	84	93JOC7899
2	4-Acetyl-2-benzoyl	Benzoyl chloride, AlCl ₃	90	93JCS(P1)273
3	2,4-Dihexanoyl	Hexanoyl chloride, AlCl ₃	94	93JCS(P1)273
Indoles				
4	2-Ethoxycarbonyl-3-(3-carbo-ethoxypropanoyl)	Methyl 3-(chloroformyl)-propanoate, AlCl ₃	95	90CPB3261
5	2-Ethoxycarbonyl-3-(4-methoxybenzoyl)	4-Methoxybenzoic acid, TFAA, H ₃ PO ₄	—	85CPB4707
6	3-(4-Methoxycarbonylpyridinoyl)-1-phenylsulfonyl	3-Chloroformyl-4-methoxycarbonylpyridine, AlCl ₃	50	85JOC5451
B. Using magnesium derivatives				
Pyrroles				
7	2-Pivaloyl	Pivaloyl chloride	71	87JOC2209
8	2-(2-Methoxybenzoyl)	2-Methoxybenzoyl chloride	58	94JHC941
9	2-(3-Carboxy-2-methylbutenoyl)	2,3-Dimethylmaleic anhydride	—	90JCS(P1)1463
Indoles				
10	3-Cyclopentylcarbonyl	Cyclopentanoyl chloride	49	88JHC469
11	3-Acetyl-5-benzyloxy	Acetyl chloride	65	60JA1180
12	3-(3-Methylbut-2-enoyl)	3-Methylbutenoyl chloride ^a	70	90T6061
C. Under Vilsmeier-Haack conditions				
Pyrroles				
13	2-Formyl	DMF, POCl ₃	85	63OSC831
14	2-Formyl-1-(4-methylphenyl)	DMF, POCl ₃	100	93JMC101
15	2-Benzoyl	Benzoylmorpholine, POCl ₃	74	93JOC7245
16	2-Chloroacetyl	<i>N,N</i> -Dimethylchloroacetamide, POCl ₃	75	89S783
17	2-Formyl-4- <i>t</i> -butyl	DMF, ClCOCOCI	83	94SC1389
Indoles				
18	3-Formyl	DMF, POCl ₃	95	54JCS3842
19	3-Formyl-6-methoxy	DMF, POCl ₃	95	92SC2077
Carbazoles				
20	9-Benzyl-3-formyl	<i>N</i> -Methylformanilide	74–86	92CPB230, 88H(27)2341

^aReaction with indolyl zinc chloride.

mensen reactions can be used. In the case of *C*-acylpyrroles and *C*-acylindoles, the transformation can also be effected by typical hydride donors because of the lability of the carbinol intermediates. For example, pyrrole-2-carboxaldehyde can be reduced to 2-methylpyrrole by LiAlH₄ (Equation (87)) (93T1343).



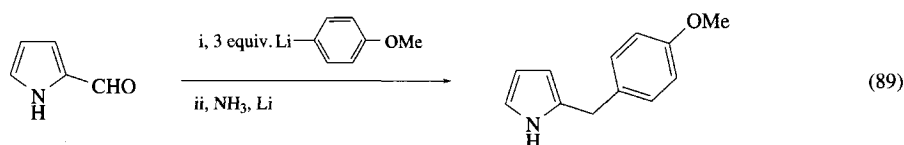
The reduction proceeds by elimination of the (complexed) α -oxy substituent, which is assisted by the ring nitrogen atom. This reactivity is especially favorable for C2 in pyrrole and C3 in indole but can also occur at the other ring positions.

Conditions involving various hydride donors have been developed. NaBH₄ in hot isopropanol reduces 2- and 3-acylpyrroles to the corresponding alkyl derivatives (85JOC2961). Both 2-acyl and 3-acyl-1-phenylsulfonylpyrroles can be reduced to the corresponding alkylpyrroles by borane-*t*-butylamine complex in the presence of AlCl₃ (89JOC4350, 90SC1647). 3-Acyl-1-phenylsulfonylindoles can be reduced to the corresponding 3-alkyl derivatives by NaBH₄-CF₃CO₂H (85JOC5451, 89JOC4350).

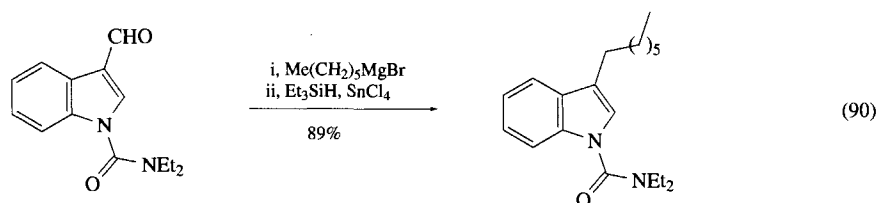
3-Benzylindoles can be obtained by *in situ* reduction of carbinols formed by addition of 2-substituted indoles to benzaldehydes in the presence of Et_3SiH and TFA (Equation (88)) <93TL1529>.



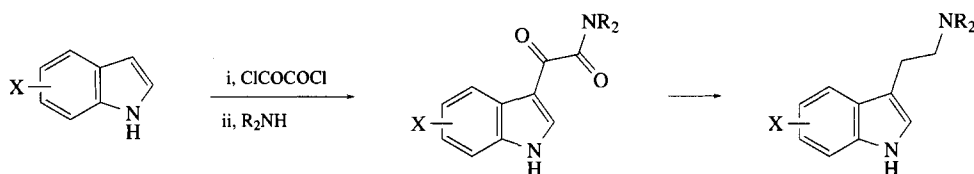
Alkyl substituents can also be built up by organometallic additions to acylindoles and pyrroles, followed by reduction of the resulting carbinols. Pyrrole-2-carboxaldehyde, for example, reacts with aryllithium reagents to give carbinols which can be reduced to 2-benzylpyrroles *in situ*. In this case, reduction was done by distilling liquid ammonia into the reaction mixture, followed by addition of lithium metal (Equation (89)) <81JOC5060>.



Similar tactics can be applied to synthesis of 3-substituted indoles (Equation (90)).

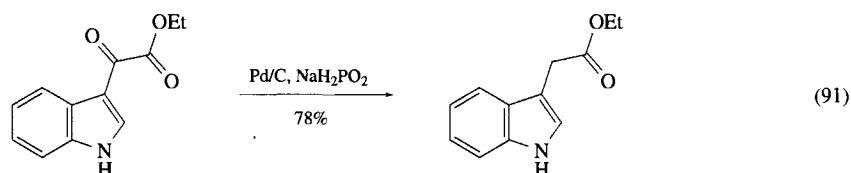


One of the general methods for introduction of a tryptamine side-chain on to indole involves acylation with oxalyl chloride, followed by reaction with an amine and then reduction <54JA6208>. In addition to LiAlH_4 , which was used in the original work, diborane <83S935, 92T1039, 93SC65> and alane <92JOC3636, 94TL5413> can be used for the reduction (Scheme 101).



Scheme 101

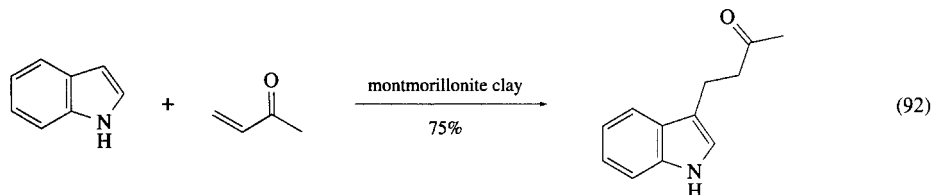
Partial reduction of glyoxalate esters to an acetate side-chain has been achieved under transfer hydrogenolysis conditions using $\text{Pd/C-NaH}_2\text{PO}_2$ (Equation (91)) <89SC2585>.



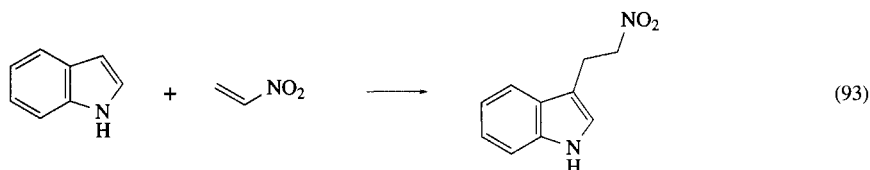
2.03.3.5 Alkylation by Conjugate Addition

Both pyrrole and indole anions are sufficiently nucleophilic to react with Michael acceptors such as unsaturated esters, ketones and nitriles. When the reaction is done under alkaline conditions the alkylation usually occurs at nitrogen. Pyrrole and indole are also reactive Michael acceptors under

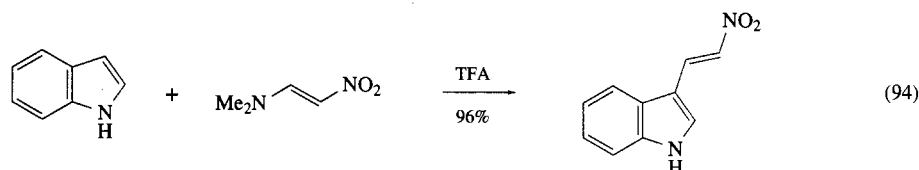
acidic conditions, in which case C-alkylation usually occurs. Reaction of indoles with unsaturated ketones in acetic acid/acetic anhydride results in addition at C3 <57JA2819>. Good results have been reported for the catalysis of the reaction of indole by acidic clay (Equation (92)) <88TL2577>.



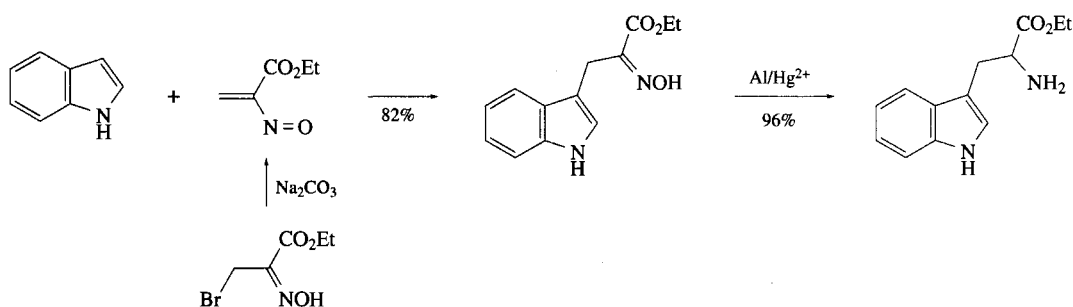
Nitroethylene is reactive toward indole in the absence of a catalyst. Up to 80% yields of the 3-alkylation product have been obtained (Equation (93)) <80JOC1185>.



1-Dimethylamino-2-nitroethene is also a useful reagent for 3-substitution of indoles (Equation (94)) <77JOC1784, 93JMC4069>. The resulting nitrovinyl indoles can be reduced to tryptamines with LiAlH₄.



The oxime of ethyl 3-bromopyruvate can be used to introduce a substituent which can be conveniently converted to a tryptophan side-chain (Scheme 102) <83JCS(P1)1283>. The active electrophile is an α -nitrosoacrylate ester. Pyrrole and *N*-methylpyrrole give the corresponding 2-substituted derivatives, accompanied by some of the 3-substitution product. This procedure has been used to prepare a number of substituted tryptophans <88S73, 90T5113, 92JHC335>.

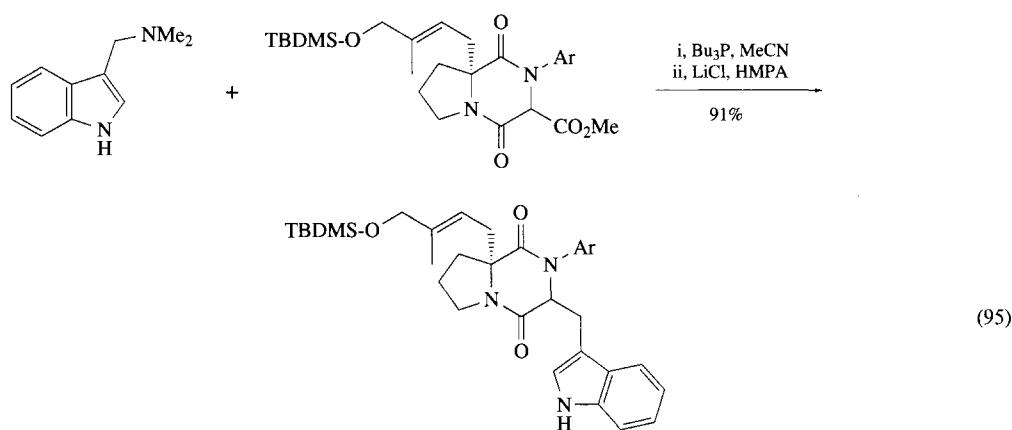


Scheme 102

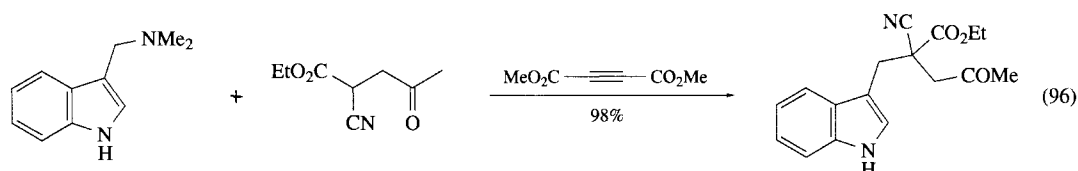
2.03.3.6 Modification of Alkyl Substituents by α -Substitution

Both pyrroles and indoles can be synthetically modified by reactions at the α -position of alkyl substituents. The special reactivity of benzylic positions in carbocyclic aromatics is well-appreciated and in pyrroles and indoles this reactivity is further enhanced by the electron-releasing capacity of the heterocyclic nitrogen. In pyrroles and indoles, these substitution reactions frequently take place under base-catalyzed conditions by an elimination-addition mechanism. Even groups such as alkoxy,

dialkylamino and cyano, which are usually poor leaving groups, can be subject to nucleophilic displacement. The widest application of this method has been in the reactions of 3-dialkylaminomethylindoles. 3-(Dimethylaminomethyl)indole (gramine) can be prepared from indole, formaldehyde and dimethylamine in acetic acid <37CB567>. Either as the neutral amine or the quaternary salt, it is a useful reagent for introduction of indolyl-3-methyl groups at a variety of nucleophilic sites. Nucleophiles such as nitroalkanes <90T833>, α -nitroesters <60JOC1548, 86T5065, 88JOC2323>, and formamidomalonates <45JA36, 89JOC2170> have been used. There have been some recent modifications of procedures for gramine alkylation which have proven useful in the synthesis of relatively complex structures. Inclusion of 30–40 mol% of Bu_3P was found to improve the reactivity and selectivity for monoalkylation in reactions of gramine with dimethyl malonate and nitromethane <81H(16)941>. This effect is attributed to reaction taking place through a phosphonium ion intermediate. This procedure gave excellent results in the alkylation of α -carboethoxydiketopiperazines (Equation (95)) <93T8471, 93JA9323>.



Gramine substitutions have also been carried out in the presence of dimethyl ethynedicarboxylate (Equation (96)) <71LA(743)95, 90T2943>. Presumably this reagent serves both to activate the amino group by *N*-alkylation and to generate a base which deprotonates the potential nucleophile.



Similar displacement reactions can be applied to branched aminoalkyl heterocycles <86T5065>. Another route to branched aminoalkylpyrroles involves addition of organolithium reagents to the dimethylaminomethylene derivatives that are formed by Vilsmeier–Haack reactions (Scheme 103). These can be isolated by treatment of the reaction mixture with NaH <87SC863>.

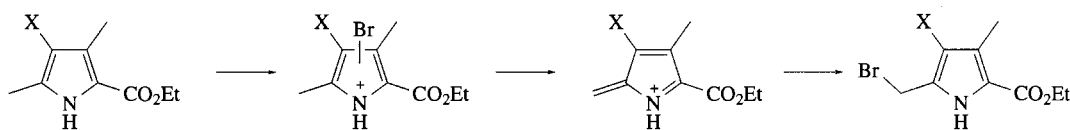


Scheme 103

While benzylic halides are very useful synthetic reagents, α -haloalkyl pyrroles and indoles are rather unstable compounds and special precautions are needed both in their preparation and application in synthesis. Synthetic methods for side-chain halogenation are also complicated by the high reactivity of the rings towards the usual halogenating agents. Both the stability and reactivity problems can be overcome to some extent by the use of a nitrogen EW-protecting group.

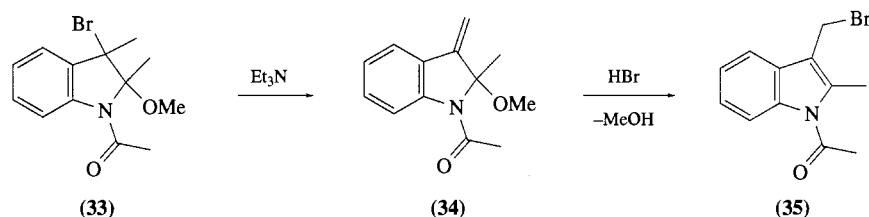
3,5-Dimethylpyrrole-2-carboxylate esters having 4-substituents are brominated in excellent yield at the 5-methyl group (Scheme 104) <80JOC1786>. Mechanistic studies indicate that this reaction

occurs by an initial nonselective ring bromination which is followed by elimination of HBr and then nucleophilic addition of Br^- <80JA1377>.



Scheme 104

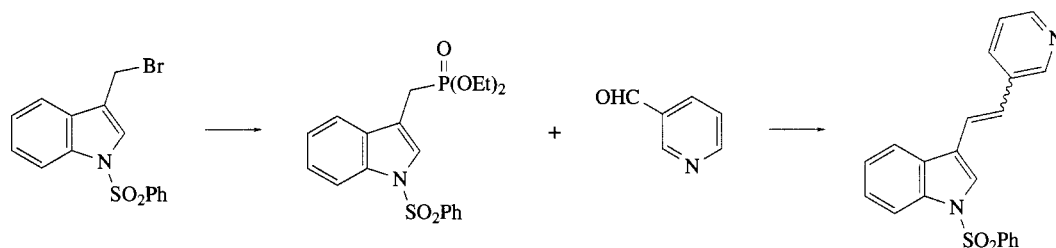
Related reactions have been observed in the indole series. Bromination of 1-acetyl-2,3-dimethylindole in methanol first generates the adduct (33) which is dehydrohalogenated to (34) by triethylamine. Exposure to HBr then generates (35) (Scheme 105) <82CJC1233, 85TL5253>.



Scheme 105

Since side-chain halogenation of indoles faces the problem of competing ring bromination, the usual approach is to install a EW group on nitrogen. Side-chain halogenation can be further favored by using radical chain halogenation conditions. These tactics have been used successfully to prepare 2-, 3-, 4- and 5-bromomethylindoles <75CPB2990, 82S926, 89H(29)783, 92JHC953, 92S743, 93JMC4230>.

Halomethylpyrroles and indoles with adequate stability can be used effectively in enolate alkylations <85TL5253, 90TL4697, 92SC2077, 93TL7383>. Scheme 106 illustrates the preparation and subsequent synthetic application of an indol-3-ylmethyl-phosphonate <92JHC953>.

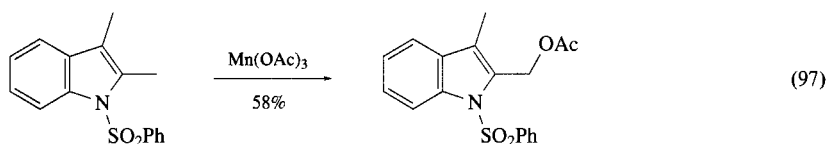


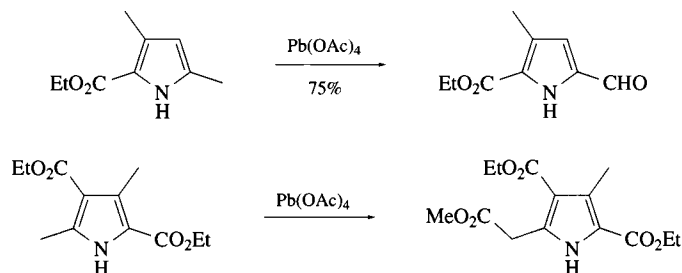
Scheme 106

2.03.3.7 Modification of Alkyl Substituents by Oxidation

Since the pyrrole and indole rings are quite susceptible to oxidation, the potent oxidants which are used to oxidize substituents on benzenoid rings are usually not applicable. Several oxidizing agents have been developed which are capable of selective oxidation of substituents at both the 2- and 3-positions. Lead tetraacetate has proven to be useful for oxidation of methyl groups on pyrrole carboxylate esters to either formyl or acetoxymethyl groups (Scheme 107) <85LA2301, 93BSF625>.

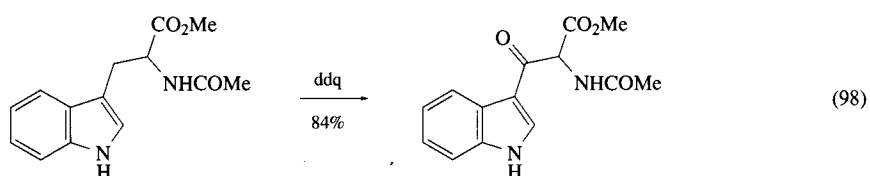
The 2-methyl group of 1-phenylsulfonylindoles can be acetoxylated by $\text{Mn}(\text{OAc})_3$ (Equation (97)) <94SC565>.



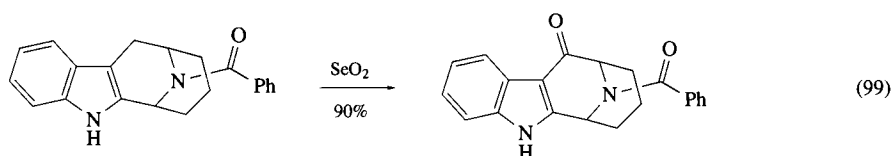


Scheme 107

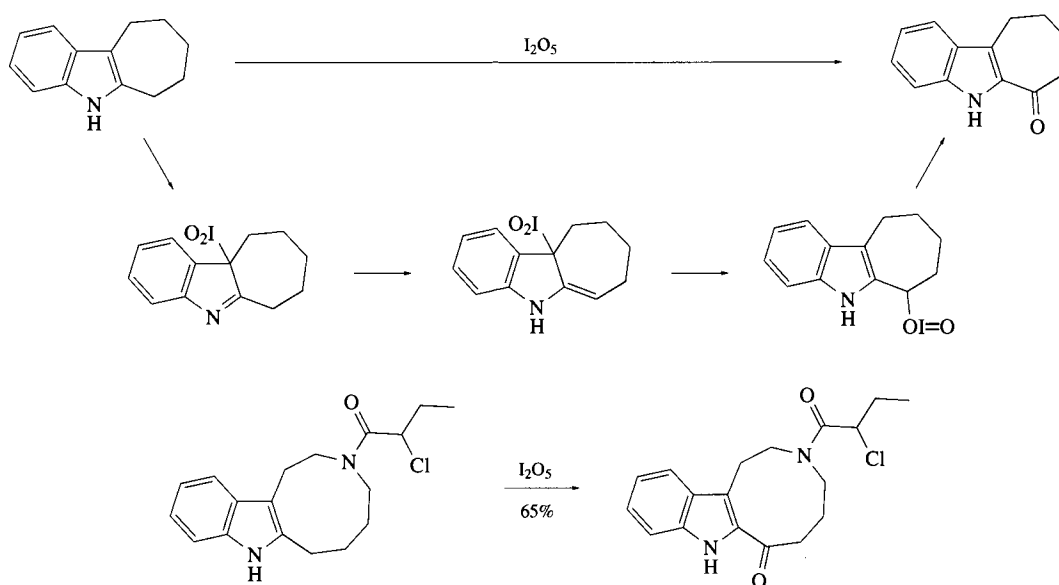
Oxidation of 2,3-dialkyl and 3-alkylindoles by ddq gives the corresponding 3-acylindoles. Yields are good to excellent for cycloalkan[b]indoles and typically in the 40–50% range for noncyclic analogs (<86TL5735, 87JHC1321, 92JOC70>. The protected side-chain of tryptophan can be oxidized in 84% yield (Equation (98)) (<77JOC1213>).



Selenium dioxide has also been used for oxidation of indoles to 3-acylindoles (Equation (99)) (<83JA907>).



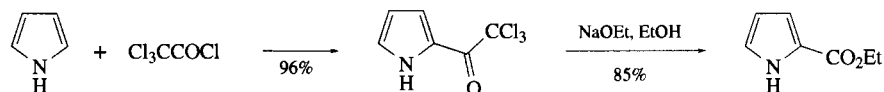
Cycloalka[b]indoles are converted to 2-acyl derivatives by I_2O_5 (<81JA6990, 87CPB4700, 93JOC7469>). The mechanism invoked to explain this reaction involves initial electrophilic attack at C3, deprotonation of the α -carbon at C2 and nucleophilic addition at the α -position (Scheme 108).



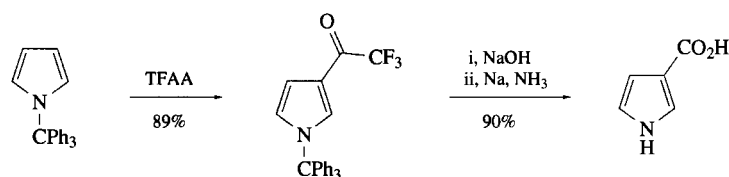
Scheme 108

2.03.3.8 Introduction of Carboxylic Acids, Esters, Amides, and Nitriles

One of the most convenient means of introducing carboalkoxy groups on to the pyrrole ring involves trichloroacetylation followed by alkaline alcoholysis (Scheme 109) <88OSC618>. Similar reactions occur with trifluoroacetylpyrroles (Scheme 110) <83JCS(P1)93>.



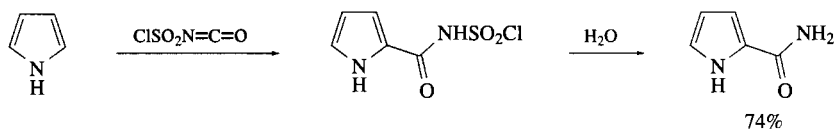
Scheme 109



Scheme 110

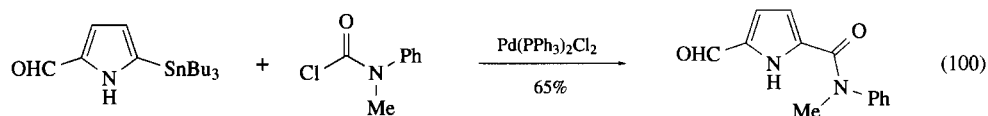
The traditional hypohalite oxidation can be applied to acetylpyrroles, so long as the ring is deactivated by appropriate substituents. For example, 3-acetyl-1-phenylsulfonylpyrrole can be oxidized to 1-phenylsulfonylpyrrole-3-carboxylic acid in 86% yield using NaOBr <93ACS57>.

There are examples of direct introduction of carboxamide substituents by electrophilic substitution. For example, pyrrole-2-carboxamide can be prepared in good yield using chlorosulfonylisocyanate as an electrophile (Scheme 111) <80CJC409>.



Scheme 111

ipso-Substitution on a pyrrolylstannane by a carbamoyl chloride has been reported (Equation (100)) <93SL117>.



Pyrrole and indole carbonitriles are readily prepared by dehydration of oximes. Because the aldehydes are easily available by Vilsmeier–Haack formylation, this represents an efficient entry into the carboxylic acid derivatives <80CJC409, 88SC671, 93JMC101>.

2.03.3.9 Halogenation

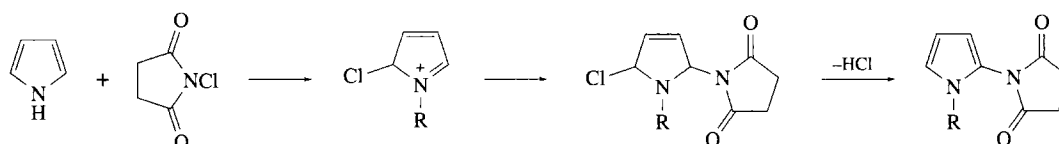
Because of the inherently high reactivity of pyrrole and indole to electrophilic substitution, halogenation can be carried out under very mild conditions. The ring halogenation products of pyrrole and indole are very sensitive to acid unless the ring is substituted by EW groups. The halogenated heterocycles are quite stable in basic solution. They can be stabilized to storage by addition of small amounts of a tertiary amine. A wide variety of halogenating agents have been examined, including *N*-chlorosuccinimide (NCS) <86H(24)2879, 86TL1051>, pyridinium bromide perbromide <63CJC2399, 86H(24)2879>, and 1,3-dibromo-5,5-dimethylhydantoin <92OS(70)151>.

Bromination of 1-substituted pyrroles with one equivalent of Br_2 in CCl_4 leads to a mixture of 2- and 3-monobromination products, as well as di-, tri- and tetrabromo derivatives. Use of NBS improves selectivity so that 75–100% yields of 2-bromination products can be obtained from 1-substituted pyrroles. With two equivalents, high yields of 2,5-dibromo products are obtained <81JOC2221>. 2-Bromopyrrole can be obtained by bromination of pyrrole with 1,3-dibromo-5,5-dimethylhydantoin in the presence of a catalytic amount of AIBN at -50°C and is isolated as the Boc derivative <92OS(70)151>.

Use of the halogens in DMF were found to give satisfactory results for the bromination and iodination of indole, 1-methylindole and 2-methylindole <82S1096>. Indole can be converted to 3-bromoindole in 64% yield using pyridinium bromide perbromide <63CJC2399>. This procedure works for indoles with both EW and ER substituents. 3-Chloroindole can be obtained by use of NCS <86H(24)2879>. Use of two equivalents of sulfonyl chloride has been reported to give 2,3-dichloroindole. Sulfonyl chloride has been used successfully to chlorinate the 3- and 2-positions of indoles having carbocyclic bromine substituents. For example, 4,7-dibromoindole can be converted to the 3-chloro (63% yield) or 2,3-dichloro (88% yield) derivatives by use of 1.0 or 2.2 equivalents of sulfonyl chloride <89H(29)1663>. A procedure in which *N*-bromosuccinimide (NBS) is used as the halogenating agent in a silica/ CH_2Cl_2 slurry has also been recommended for indole <86TL1051>. With one equivalent, the reaction occurs at C3 but a second equivalent leads to bromination at C6.

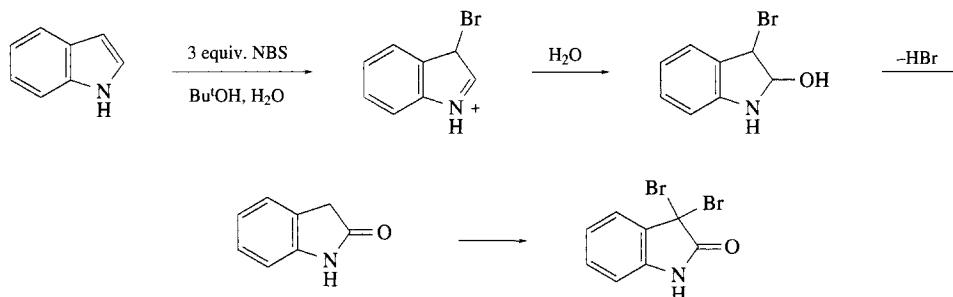
Carbazole and its *N*-substituted derivatives undergo 3,6-dibromination under conventional halogenation conditions <86MM516>. *N*-Methylcarbazole can be converted to the 3,6-dibromo derivative by heating with HBr in DMSO <92CHE1260>. The use of NBS with a silica slurry has been reported to give some control of bromination on the basis of the amount of reagent used. With two equivalents the 3,6-dibromo product is obtained quantitatively. With one equivalent a 1:3:1 mixture of carbazole, 3-bromocarbazole and 3,6-dibromocarbazole is obtained <92T7479>.

Occasionally the halogenation of pyrroles results in an addition product. For example, while pyrrole itself undergoes chlorination, reaction of 1-substituted pyrroles with NCS gives 2-succinamidopyrroles <89JOC5347>. This reaction probably occurs as a result of trapping of the initial chlorination intermediate, followed by elimination of HCl (Scheme 112).



Scheme 112

This reaction pattern involving nucleophilic capture of an initial halogenation intermediate is also observed in indoles and forms the basis for a convenient method of converting indoles to bromooxindoles. Reaction of indole with NBS in aqueous *t*-butanol gives 3,3-dibromooxindole (Scheme 113) <64JOC1206, 89JCS(P1)2009>.



Scheme 113

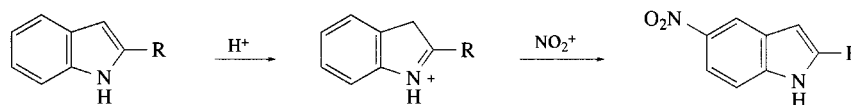
This reaction proceeds by capture of the 3-bromo-3*H*-indolenine intermediate by water, leading to formation of an oxindole which is further brominated. By the use of a limited amount of NBS, the subsequent bromination can be avoided.

Regioselective thallation (see Section 2.03.4.2) can be used to introduce halogen at C4 or C7 of the indole ring. For example, 5,7 and 6,7-dibromoindole-3-carboxaldehyde can be converted to the corresponding 4-bromo derivative by reaction with $\text{Ti}(\text{O}_2\text{CCF}_3)_3$ and CuBr_2 (89H(29)1663).

2.03.3.10 Introduction of Nitrogen Substituents

The nitration of pyrrole is rather difficult to carry out in good yield. The ring is susceptible to both oxidation and acid-promoted polymerization in the usual nitrating media (90HC(48-1)330). The best yields for nitration of simple pyrroles are usually obtained using nitric acid and acetic anhydride, in which the active nitrating species is acetyl nitrate. For pyrrole, a yield of 60–70% of a 4 : 1 mixture of 2-nitro and 3-nitropyrrole can be obtained (70T5101). 1-Substituted pyrroles react similarly, although the ratio of 3-nitro product increases with the steric bulk of the nitrogen substituent (79JOC2321). 2-Methylpyrrole gives a 4.3 : 1 ratio of 5-nitro and 3-nitro product in 12–15% yield (70JHC399). A quite good yield (73%) of the 3-nitration product was obtained from 1,2,5-trimethylpyrrole using $\text{KNO}_3\text{--H}_2\text{SO}_4$ (87OPP48). Use of a 1-EW group would be expected to improve the selectivity of pyrrole nitration. Among the groups explored have been acetyl, methoxycarbonyl and phenylsulfonyl (71T245, 85CJC896). 3-Nitropyrrole can be obtained in 77% yield by nitrating 1-tips-pyrrole with $\text{Cu}(\text{NO}_3)_2$ in acetic anhydride, followed by desilylation with $\text{Bu}_4\text{N}^+ \text{F}^-$ (90JOC6317). With EW substituents at C2, mixtures of 4- and 5-nitration products are obtained. With 3-EW groups, 5-nitration is observed (59CJC2053, 68JMC282, 71T245).

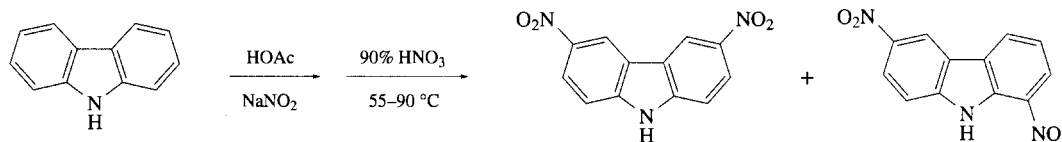
The mechanistic foundations of indole nitration have been well established (72HC(25-2)539). While the 3-position is the preferred site for electrophilic attack, nitration of indole itself is complicated by oxidation and dimerization. In strongly acidic solution, the 3-position is protonated and reaction takes place at the 5-position under the directing influence of the iminium moiety (Scheme 114).



Scheme 114

When the 3-position is occupied by an EW substituent, nitration occurs at C6 and C4. For example, the nitration of gramine, which proceeds through the N^{b} -protonated species, gives a 6 : 1 mixture of the 6-nitro and 4-nitro products in 49% total yield (60G525). Methyl indole-3-carboxylate is nitrated by HNO_3 at 60°C to give a 60% yield of a 1 : 1 mixture of 4- and 6-nitration products (86TL4327). Selectivity for the 4-position can be enhanced by blocking the 6-position or by the steric effect of a 7-substituent (87H(26)1475).

Nitration of carbazole gives an approximately 2 : 1 mixture of 3,6- and 1,6-dinitrocarbazole. It is synthetically advantageous to carry out the reaction on 9-nitrosocarbazole, with the nitroso group being lost during nitration (Scheme 115) (64JOC2474).



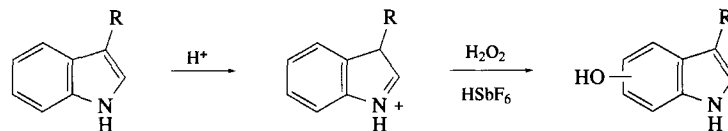
Scheme 115

Substituted carbazoles can be nitrated and reduced to obtain aminocarbazoles (91JHC1165). The Curtius reaction is an alternative means for synthesis of aminocarbazoles from carbazole carboxylic acids (90H(31)241).

2.03.3.11 Introduction of Oxygen Substituents

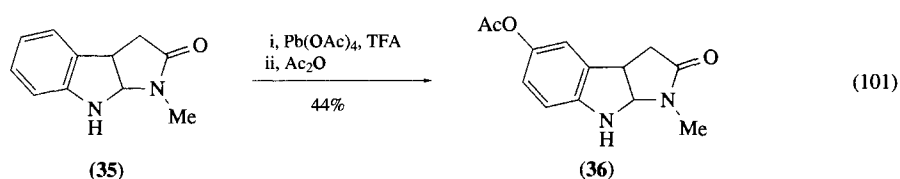
Direct oxidative hydroxylation of pyrrole and indole derivatives is rare. The susceptibility of the heterocyclic rings to oxidation facilitates ring cleavage. Indole, 3-methylindole and tetrahydrocarbazole have been hydroxylated using H_2O_2 in $\text{SbF}_5\text{--HF}$. A mixture of 4-, 5-, and 6-

hydroxylation products is obtained, with the 5-hydroxy product being the major one. Both the position of substitution and the preference for carbocyclic hydroxylation would suggest that the reaction proceeds by hydroxylation of a 3-protonated indolenine intermediates (Scheme 116) <86TL4565, 87NJC611>.

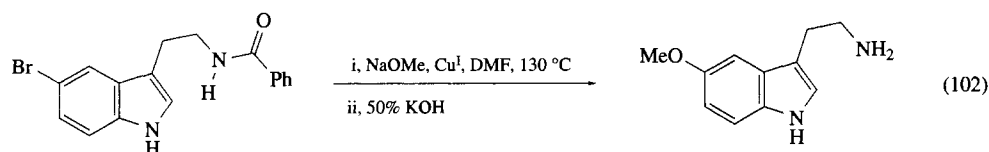


Scheme 116

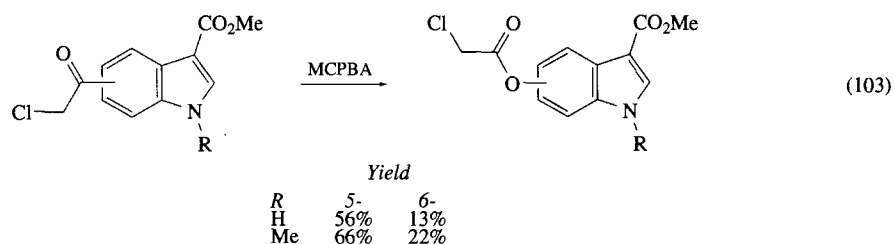
The dihydroindole (35) has been directly acetoxylation by $\text{Pb}(\text{OAc})_4$ (Equation (101)) <90CPB2632>. This reaction, too, depends on both the activation of the carbocyclic ring by the nitrogen substituent and the masking of the reactivity of the heterocyclic ring.



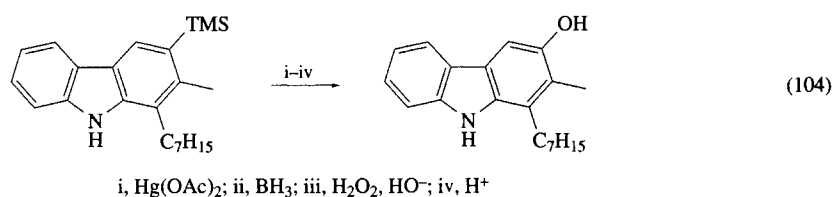
Indole 3-boronic acid has been oxidized to indolin-3-one by H_2O_2 , but the yield is low <90H(30)627>. This reaction might be applicable to hydroxylation at carbocyclic positions, since it is a fairly general method for aromatic hydroxylation <57JOC1001>. The boronic acids can be obtained from aryllithium compounds. There are a few examples of direct displacement of halogen by oxygen substituents, but as with other aromatics, vigorous conditions are required (Equation (102)) <79JHC1325>.



Successful Baeyer–Villiger oxidations have been reported for 5-chloroacetyl and 6-chloroacetyl derivatives of methyl indole-3-carboxylate and the corresponding 1-methyl derivatives <87H(26)1471>. The yields were much better for the chloroacetyl compounds than for the acetyl derivatives (Equation (103)).



Vilsmeier–Haack formylation, followed by oxidation with acidic H_2O_2 was used to introduce a hydroxy group at C3 in *N*-methylcarbazole <89T1759>. A TMS group was used for the introduction of a C3 hydroxy group in a carbazole in the course of synthesis of carazostatin by a sequence of mercuri-desilation, hydroboration and oxidation (Equation (104)) <90SL521>.



2.03.3.12 Introduction of Sulfur Substituents

The high reactivity of pyrrole and indole toward electrophilic substitution permits direct sulfenylation, sulfinylation and sulfonylation under relatively mild conditions. Direct sulfenylation of pyrroles and indoles can be carried out using *N*-(methylthio)morpholine. With 1-methylpyrrole at a 1:1 molar ratio a mixture of 2-methylthio and 2,5-bis-methylthio pyrrole is obtained. The latter becomes the major product when the ratio of sulfenylating agent is increased to 4:1 <91JHC1025>. A similar product composition is observed using methanesulfonyl chloride. The 3-substitution product is obtained from 1-methylindole and *N*-(methylthiomorpholine) in 76% yield. Both pyrrole <71AJC1229> and indole <93JMC1425> derivatives have been sulfenylated by reaction with thiophenols and I_2 . Reaction of indole with dimethyl disulfide and CuI gives 3-(methylthio)indole in 41% yield <89JOC2985>. Reaction of the zinc salt of indole with dimethyl disulfide also gives 3-(methylthio)indole <93TL6245>. The *S*-trifluoroacetoxysulfonium salt prepared by reaction of methyl phenyl sulfoxide with TFAA reacts with indole in the presence of Et_3N to give 3-phenylthioindole in 71% yield <93TL6245>.

Pyrrole and 1-methylpyrrole can be sulfinylated by sulfinyl chlorides or *N*-(phenylsulfinyl)succinimide. The products are prone to rearrange to the 3-isomers on contact with acid <80JOC5336>. A phenylsulfinyl group has been introduced at C3 of the indole ring by 3-sulfenylation of *N*-lithioindole with diphenyl disulfide, followed by *N*-sulfonylation and oxidation to the sulfoxide <89JOC1782>. Sulfinylindoles can be oxidized to sulfones by MCPBA <93JMC1425>.

2.03.4 SYNTHETIC MODIFICATION VIA ORGANOMETALLIC INTERMEDIATES

The synthetic methodology based on organometallic chemistry which has been developed for aliphatic and benzenoid compounds also has many applications to heteroaromatic chemistry. A number of valuable methods for the introduction and modification of substituent groups on pyrrole, indole and carbazole rings are based on reactions involving organometallic reagents and intermediates. In this section we will discuss reactions which proceed through four types of organometallics: organolithium compounds, mercury and thallium derivatives prepared by electrophilic metallation, transition metal intermediates, and π -complex intermediates.

2.03.4.1 Reactions Involving Organolithium Intermediates

In aliphatic and benzenoid chemistry Grignard and organolithium reagents are frequently prepared from halogenated precursors. This is less common in pyrrole and indole chemistry. Instead, direct lithiation is the usual route to organolithium compounds. The metallation is usually done with an alkylolithium reagent and is selective for the 2-position in both pyrrole and indole because of the directive effect of the heteroatom. Other strategies, including halogen-metal exchange, or substituent-directed lithiation, must be used to introduce lithium at other positions. Because of the acidity of the N—H bond, it is usually substituted during lithiation, frequently by one of the protecting groups discussed in Section 2.03.3.1. If the protecting group also acts as a directing group, reactivity toward lithiation is further enhanced. This is the case for phenylsulfonyl, *t*-butoxycarbonyl and alkoxymethyl groups.

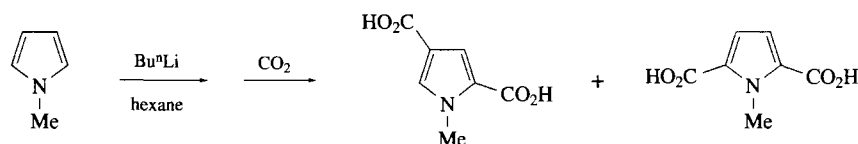
Both 1-methylpyrrole and 1-methylindole can be easily lithiated and the resulting intermediates have been used extensively in synthesis. The range of electrophiles which can be used with lithiated pyrroles and indoles is quite broad. Aldehydes and ketones give carbinols. *N,N*-Disubstituted formamides give aldehydes. Carbon dioxide and alkyl chloroformates have been used to make carboxylic acid derivatives. Alkyl halides, sulfates and sulfonates can be used to introduce alkyl groups. Interestingly, even *t*-butyl bromide has been used successfully for alkylation (see entry 3, Table 7). Some examples are given in Table 7.

In the presence of excess *n*-butyllithium, 1-methylpyrrole is dilithiated to give a mixture of both 2,4- and 2,5-substitution products (Scheme 117) <77JCS(P1)887, 91TL4313>. Inclusion of TMEDA in the lithiation reaction favors the 2,5-dilithiation, but not much synthetic application has been made of the dilithio intermediate.

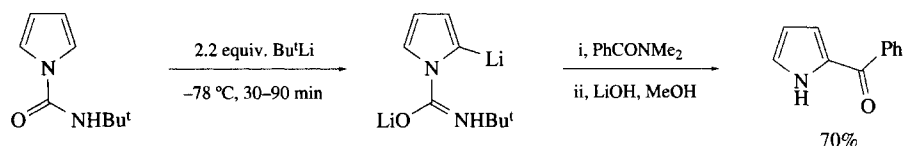
Several of the *N*-protected pyrrole and indole derivatives can be lithiated by amide bases as well as by organolithium reagents. 1-(*t*-Boc)pyrrole can be lithiated by lithium 2,2,6,6-tetramethylpiperidide <81JOC157, 91S613>. 2-Lithio-Boc-pyrrole can also be prepared from the 2-bromo precursor by halogen-lithium exchange <92OS(70)151>. 1-Phenylsulfonylindole has been used extensively as a

Table 7 Examples of synthetic procedures involving lithiation.

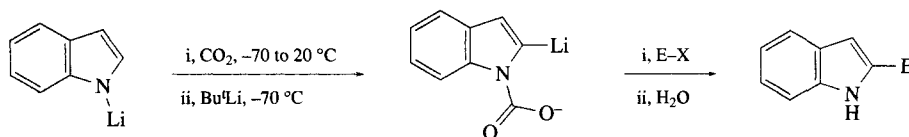
Entry	Lithiation substrate	Conditions	Electrophile	Yield (%)	Reference
A. Pyrroles					
1	1-Methyl	Bu ⁿ Li, 25, 12h	Benzonitrile	70	82SC231
2	1-Methyl	Bu ⁿ Li, TMEDA, 15 min	Cyclopropanoyl/benzene	100	93JMC1716
3	1-Methyl	Bu ⁿ Li, 25, 12h	<i>t</i> -Butyl bromide	90	82SC231
4	1- <i>t</i> -Butoxycarbonyl	Li TMP	Triethyl borate	83	93T1009
B. Indoles					
5	1-Phenylsulfonyl	LDA	Pyridine-3,4-dicarboxylic anhydride	78	82JOC2810
6	5-Methoxy-1-phenylsulfonyl	Bu ⁿ Li	Dimethyl oxalate	54	89JOC3084
7	5-Methoxy-1-phenylsulfonyl	LDA	3-Dimethylaminocarbonyl-4-formyl-2-methoxypyridine	71	93T2885
8	1-Carboxy-3-methyl	Bu ⁿ Li	Methyl chloroformate	70	88SC1151

**Scheme 117**

synthetic intermediate and both *t*-butyllithium <73JOC3324> and LDA <81JOC2979, 82JOC757> have been used for lithiation. 1-Boc-indole can be lithiated at C2 by *t*-butyllithium <81JOC157>. The *t*-butylcarbamoyl group is also useful for the directed lithiation of pyrroles and indoles <91SI079>. It may be preferable to both the Boc and phenylsulfonyl groups for pyrrole lithiation. Lithiation proceeds using *t*-butyllithium at -78°C and good yields of substitution products are obtained with typical electrophiles. The carbamoyl protecting group can be removed by alkaline hydrolysis (Scheme 118).

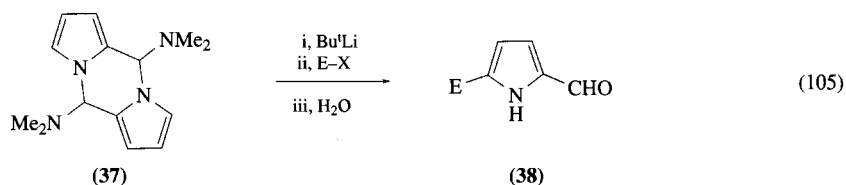
**Scheme 118**

An efficient methodology for C2 lithiation involves treatment of the lithium salt of indole with CO₂ at -70°C to -20°C , followed by another equivalent of *t*-butyllithium. The *N*-carboxy group introduced by the carbonation serves as a directing group for the C2 lithiation <85TL5935>. Since the *N*-carboxy group is unstable to hydrolysis, *N*-deprotection occurs during work-up (Scheme 119). Similar methodology has been applied to pyrrole <88OPP585> and to 3-methylindole <88SC1151>.

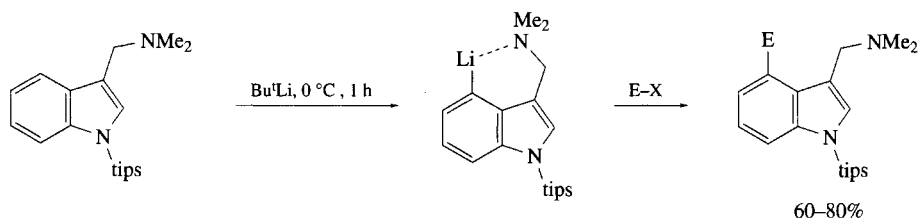
**Scheme 119**

Early studies included the methoxymethyl group as one of the *N*-protecting groups examined for indole lithiation <73JOC3324>. While lithiation occurred readily, the subsequent removal of the protecting group was troublesome. The 1-(2-trimethylsilylethoxymethyl) (SEM) group has been used in lithiations of pyrroles and indoles <84JOC203, 86T3723>. Using *n*-butyllithium in DME, lithiation occurs in less than an hour at -5°C to 0°C . *N*-Deprotection can be accomplished with

tetra-*n*-butylammonium fluoride. There are also examples of use of *N*-(dialkylaminomethyl) as protecting groups in pyrrole and indole lithiation. 1-Methyl-2-(dimethylaminomethyl)pyrrole undergoes lithiation primarily at C5 indicating that the heteroatom is the controlling factor <82JOM(234)123>. The dimer (37) serves as a versatile synthon for 5-substituted pyrrole-2-carboxaldehydes <88TL777>. Lithiation occurs adjacent to the heteroatom and after hydrolysis the 5-substituted aldehydes can be obtained (Equation (105)).



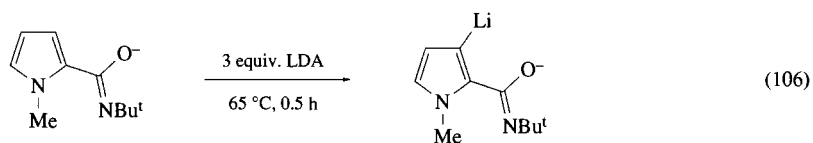
1-(Dimethylaminomethyl)indole is lithiated by *n*-butyllithium in ether at room temperature and good yields of 2-substituted products are obtained from carbonyl compounds <89H(29)849, 90JOC3688>. When the indole nitrogen is substituted with a tips group, gramine undergoes lithiation at C4 <93H(36)29>. The steric blocking effect of the tips group is presumably important in this case since the 1-methyl derivative of gramine is lithiated at C2, as would have been expected (Scheme 120).



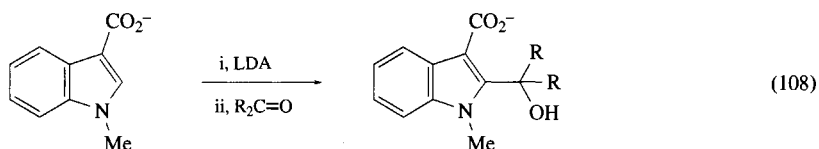
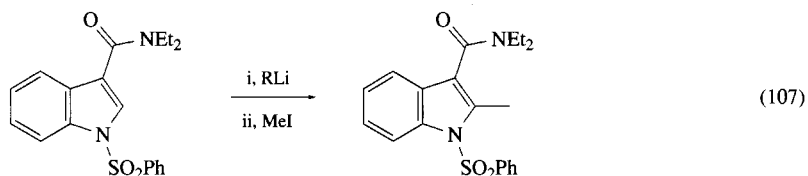
Scheme 120

The *N*-(pyrrolidinomethyl) derivative of carbazole is lithiated at C1 using either *t*-butyllithium or *n*-butyllithium/TMEDA. Good yields of 1-substituted carbazole can be obtained with the usual electrophiles <88JOC794>.

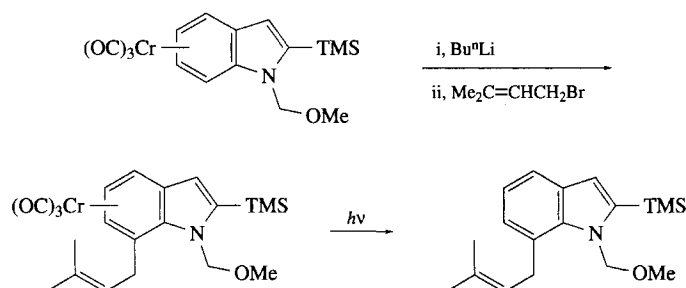
Several of the directing groups developed for benzenoid lithiation have also been employed with pyrroles and indoles. 1-Methylpyrrole-2-(*N*-*t*-butyl)carboxamide is lithiated at C3, although in modest yield (Equation (106)) <85JOC4362>.



The *N,N*-diethylamide of 1-phenylsulfonylindole-3-carboxylic acid is lithiated at C2 and gives fair to good yields of addition products with typical carbonyl compounds (Equation (107)) <93JCS(P1)1425>. The acid can also be lithiated at C2 using LDA (Equation (108)).

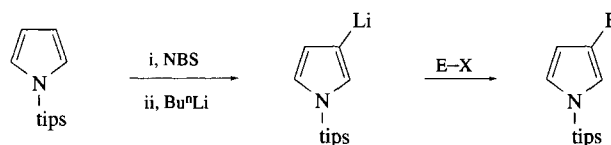


Among the less conventional substrates for lithiation are the η^6 -chromium complexes of indole. Lithiation occurs preferentially at C2 but can be directed to the carbocyclic ring if the 2-position is blocked. The complex of 1-(methoxymethyl)indole is lithiated at position 7, if the 2-position is blocked by a TMS group. 7-Substituted indoles can be obtained in 70–95% yield <89T5955>. The chromium can be removed photochemically (Scheme 120).



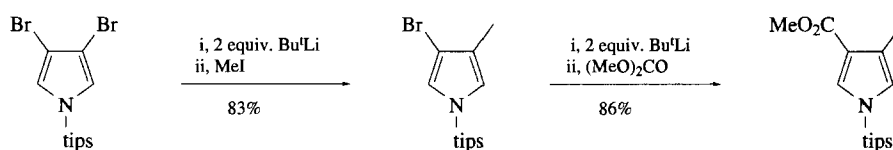
Scheme 121

When organolithium reagents are required that are not accessible by directed metallation, the usual tactic is to prepare an appropriate halogenated precursor and introduce the lithium by halogen-metal exchange. With pyrroles and indoles, this approach is somewhat complicated by the poor stability of the halogenated heterocycles but with use of an appropriate *N*-protecting group the goal can usually be achieved. 1-tips-pyrrole is a useful compound for the preparation of 3-substituted pyrroles via 3-lithio intermediates (Scheme 122). It can be brominated selectively at C3 and converted to the organolithium intermediate by halogen-metal exchange <84JOC3239>. Among the electrophiles used successfully with 3-lithio-1-tips-pyrrole are alkyl halides, CO_2 , DMF, *N,N*-dimethylacetamide, formaldehyde and various aldehydes and ketones <89CB169, 90JOC6317>.



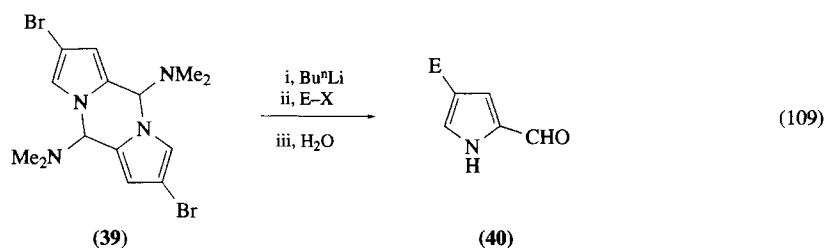
Scheme 122

Sequential halogen-lithium exchange starting with 3,4-dibromo-1-tips-pyrrole has been used to prepare 3,4-disubstituted pyrroles (Scheme 123) <90TL6785>.

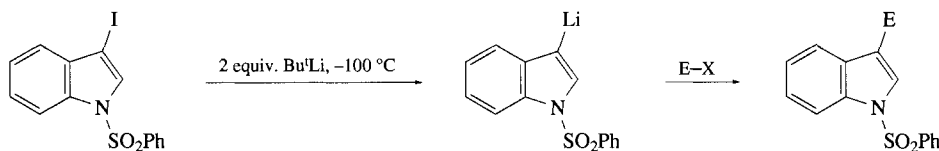


Scheme 123

The dibromo derivative of the dimeric pyrrole (**39**) is a useful precursor of 4-substituted pyrrole-2-carboxaldehydes (Equation (109)) <88TL3215>.



3-Lithio-1-phenylsulfonylindole can be prepared from the 3-iodo precursor at -100°C . At room temperature it rearranges to the more stable 2-lithio isomer. Good yields of 3-substituted indoles can be obtained as long as the rearrangement is avoided (Scheme 124) <82JOC757>.



Scheme 124

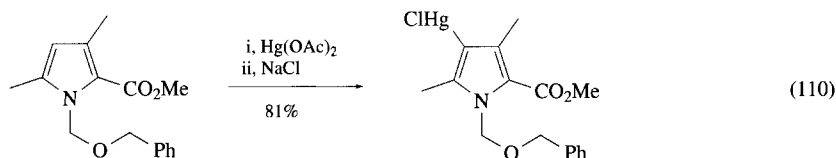
3-Lithio-1-arenesulfonylindole intermediates are also prone to ring fragmentation. Lithiated 1-phenylsulfonylindole-2-carboxamides fragment at -78°C <86H(24)2127>. 3-Lithiated indoles with *N*-alkyl substituents are much less susceptible to fragmentation. This relative stability relationship presumably reflects the anion stabilization provided by the sulfonyl group.

A related observation was made during the study of lithiation of 1-arenesulfonylindol-2-ylidithianes. The indole ring fragmented as a result of 3-lithiation when 1-phenylsulfonyl was used as the protecting group but this problem was avoided by use of *p*-methoxyphenylsulfonyl as the protecting group <88T443, 90H(31)173>. 3-Bromo-1-(TBDMS)-indole is a useful precursor of 3-substituted indoles. Lithiation can be done using *t*-butyllithium and good yields of substituted products were obtained with a range of typical electrophiles <94JOC10>. All of the isomeric indoles lithiated in the carbocyclic ring have been prepared by halogen-metal exchange using *t*-butyllithium and the potassium salt of the various bromoindoles <86JOC5106>. The lithiated intermediates have been formylated (with DMF) and acylated (with *N*-methoxy-*N*-methylcarboxamides).

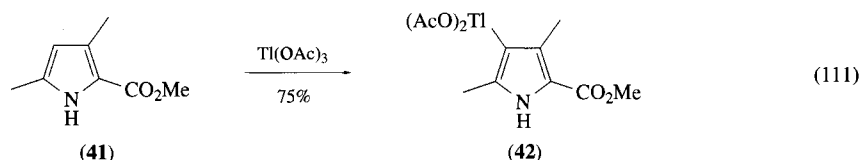
2.03.4.2 Electrophilic Metallation by Mercury and Thallium Salts

The high reactivity of the pyrrole and indole rings makes them reactive toward electrophilic cations such as Hg^{2+} and Tl^{3+} . As a result, certain organomercury and organothallium intermediates are available by direct substitution. The mercury derivatives are usually prepared by reaction of the heterocycle with $\text{Hg}(\text{OAc})_2$ and reaction occurs at the position favored for electrophilic attack, primarily C3 in indoles. Thallation, on the other hand, is directed by chelating substituents so the position of substitution depends on the placement of directing groups. The most useful procedures involve 4- and 7-substitution on indoles directed by 3- or 1-substituents, respectively. The thallated derivatives of pyrroles and indoles can be converted to halides, cyanides and nitro compounds by procedures that are general for arylthallium compounds.

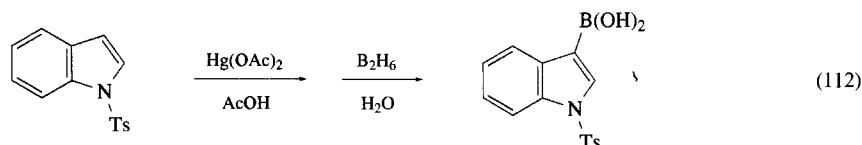
Mercuration products have been characterized for several pyrroles (Equation (110)) <89JOC4801>.



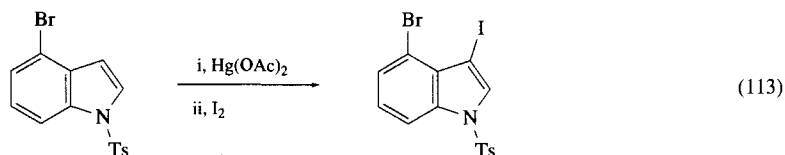
In a study of thallation of pyrroles it was found that only reactants such as (41), which were trisubstituted, gave good results (Equation (111)) <90G587>. Thallation of 1-methylpyrrole-2-carboxaldehyde appears to be anomalous, giving a 3,4-dithallation product in preference to mono-thallation <79JHC993>.



There is some uncertainty about the detailed structure of the mercuration product of indole itself. It is likely that substitution occurs at both N1 and C3. Both 1-methyl and 3-methylindole give pure mercuration products which can be isolated as acetates or chlorides <82MI 203-02>. *N*^b-Acetyltryptophan gives a mixture resulting from C2 and C7 mercuration, including some of the 2,7-dimercuration product <94TL1239>. 1-*p*-Toluenesulfonylindole is reactive toward $\text{Hg}(\text{OAc})_2$ and gives the 3-mercuration product which can be used to introduce a boronic acid substituent at C3 (Equation (112)) <93TL2235>. Indoleboronic acids are useful intermediates for palladium-catalyzed coupling reactions (see Section 2.03.4.5).



Mercuration has also been used to introduce iodine at C3 in indoles (Equation (113)) <84JOC2657>.



Perhaps the most important current synthetic application of thallated intermediates is in the preparation of 4- and 7-substituted indoles. Indole-3-carboxaldehyde, indole-3-carboxylate esters and 3-acetylindole all undergo thallation at C4. Thallation can be directed to the 7-position by 1-acyl substituents and 1-acetylindole is the preferred reactant <89H(29)643>. The thallated intermediates have been used to introduce halogen, nitro, and azido substituents <79JHC993, 85CPB3696, 87H(26)2817, 89H(29)643>. Thallated intermediates can also be subjected to palladium-catalyzed coupling (see Section 2.03.4.5).

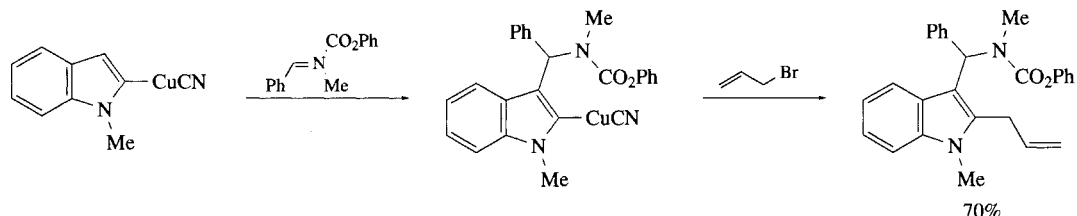
2.03.4.3 Substitution Reactions Involving Transition Metals

Transition metals have a prominent role in methods for effecting carbon-carbon bond formation, with organocopper and organopalladium being the most important in organic chemistry as a whole. Organocopper intermediates are usually prepared stoichiometrically and are used as nucleophiles. Organopalladium species are usually generated *in situ* as transient intermediates and many reactions involve only a catalytic amount of palladium. Synthetic procedures involving organopalladium intermediates developed in aliphatic and aromatic chemistry have proven to be useful in the synthesis of both pyrroles and indoles <88AG(E)1113, 88H(27)2225>.

2.03.4.4 Pyrrolyl and Indolyl Organocopper Derivatives

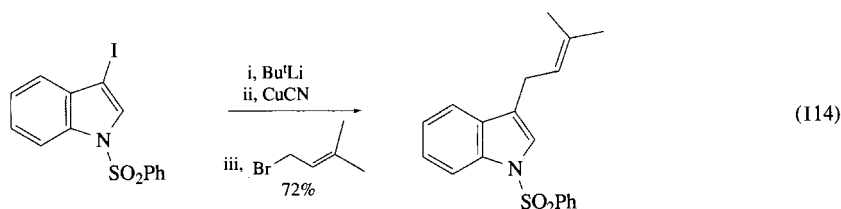
Organocopper intermediates are relatively unexploited in the synthesis of pyrroles and indoles. Early work demonstrated that 1-methylpyrrol-2-ylcopper could be coupled with aryl iodides, but the symmetric coupling product 1,1'-dimethyl-2,2'-dipyrrole was also formed <71ACS2596>. Treatment of 1-lithio-2-methylindole with CuBr gives 1-methylindol-2-ylcopper which is a solid that is sufficiently stable to be stored for several weeks <80T1439>. It gives coupling products with aryl iodides. Recent synthetic work on pyrrolyl- and indolylcopper reagents has focussed on cyanocuprates. A cyanocuprate can be prepared from 2-lithio-1-methylindole and it gives a good yield of coupling product with allyl bromide <89CC727>. In other cases, the cyanocuprate has been found to react at C3. This was observed using 1-acylpyridinium ions and acyliminium ions as electrophiles. In the

case of *N*-phenoxycarbonyl-*N*-methylbenzaldiminium ions, a sequential C3, C2 substitution was achieved (Scheme 125).



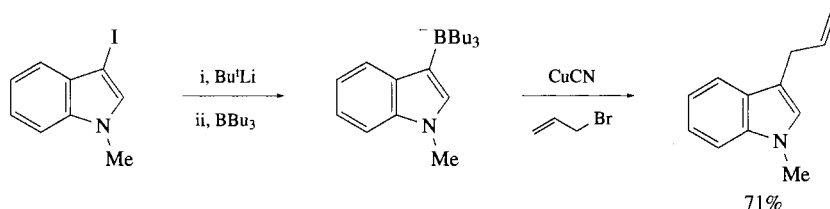
Scheme 125

3-Lithio-1-benzenesulfonylindoles can be converted to the cyanocuprate which undergoes C3 coupling with substituted allyl bromides (Equation (114)) <86JOC2343>.



(114)

Pyrrolyl-2-borates and indolyl-3-borates, which can be prepared from the corresponding lithium compounds, can be coupled with allylic halides in the presence of CuCN (Scheme 126) <87JHC377>.



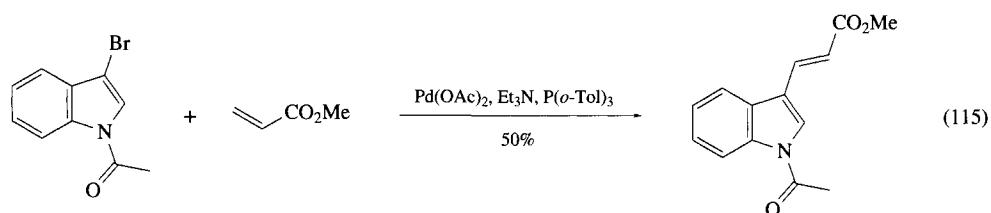
Scheme 126

While studies of pyrrolyl and indolyl copper reagents have not yet been comprehensive, it appears that these compounds are quite stable and less reactive than phenylcopper and cuprate reagents. The stability of thienylcopper reagents is well known and has been put to use in development of organocopper chemistry, and perhaps the stability of the pyrrolyl and indolyl reagents is related. The fact that certain electrophiles attack reactive C—H positions suggests rather low reactivity of the C—Cu bond.

2.03.4.5 Reactions Involving Pyrrolyl and Indolyl Palladium Intermediates

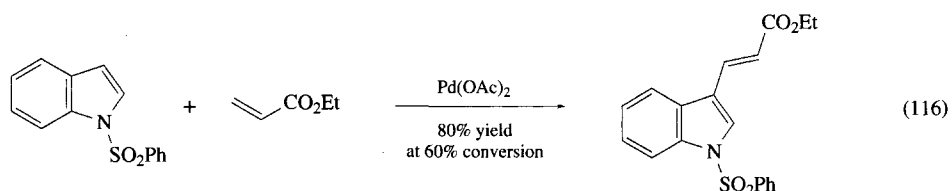
The most important palladium-catalyzed processes include Heck vinylation of halides and sulfonates and various cross-coupling reactions in which a nucleophilic intermediate (stannane, organozinc halide or boronic acid) is coupled with an electrophile (halide or sulfonate). These coupling reactions are usually restricted to arylation and vinylation because of the tendency of alkylpalladium species to undergo elimination. The pyrrole and indole rings can participate in cross-coupling as either the nucleophilic or electrophilic component.

1-Acetyl-3-bromoindole was among the compounds studied in the early stages of the development of the Heck reaction. It reacts in a typical manner with methyl acrylate (Equation (115)) <78JOC2947>.

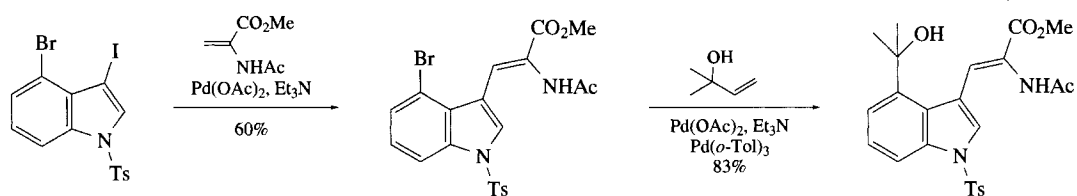


1-Phenylsulfonylindol-3-yl triflate is also a satisfactory reactant and it can be coupled with acrylate esters, acrolein, enones and styrene <92SC2129>. Heck reactions can also be used to introduce vinyl substituents on the carbocyclic ring in indoles and carbazoles using appropriate halogenated precursors <85CPB2162, 87CPB3146, 88JOU583>.

Vinylation can be carried out directly at the 3-position on indoles without a halogen or sulfonate substituent <84H(22)1493>. This reaction presumably involves an electrophilic palladation at C3 followed by a Heck reaction (Equation (116)). These reactions can also be carried out in the presence of a cooxidant such as $\text{Cu}(\text{OAc})_2$ or AgOAc which serves to reoxidize palladium. Similar reactions have been carried out on 1-benzenesulfonylindole <84S236> and 1-acylindoles <83JCS(P1)1361>.

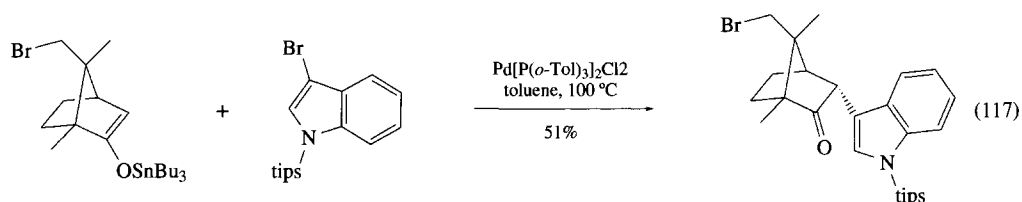


Examples of specialized uses of palladium-catalyzed Heck reaction include the synthesis of the ergot metabolite clavicipic acid, where two sequential vinylations were carried out by taking advantage of the greater reactivity of a 3-iodo over a 4-bromo substituent (Scheme 127) <87JA4335>.

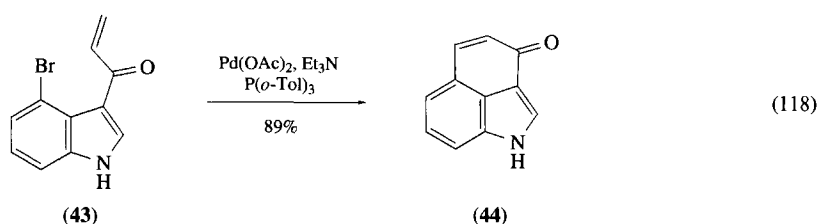


Scheme 127

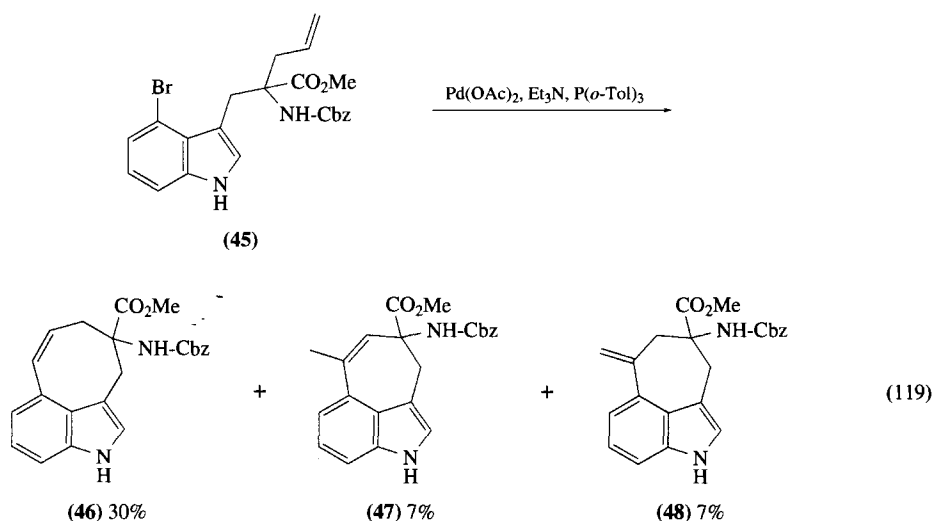
In the synthesis of hapalindole Q, an indolyterpene isolated from algae, coupling was achieved under Heck conditions with a tin enolate (Equation (117)) <93JA3499>.



Several intramolecular Heck reactions have been reported. An 89% yield was obtained in the cyclization of (43) (Equation (118)) <89JOC4141>.



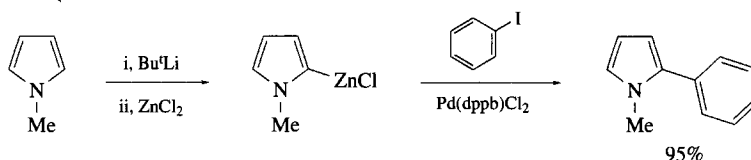
With (45) both 7-membered and 8-membered ring formation was observed (Equation (119)) <94JOC4418>.



Similar results were obtained in a cyclization involving the 3-position of the indole ring <90JOC6028>.

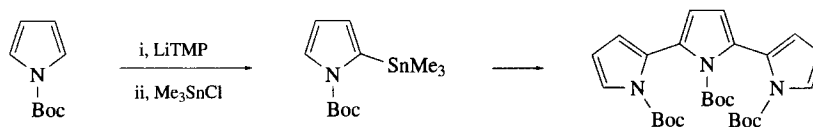
The other important synthetic application of palladium intermediates is in cross-coupling. This involves reaction of a nucleophilic reagent, typically an organozinc, stannane or boronic acid with an aryl or vinyl halide or sulfonate. The fundamental mechanism involves an oxidative addition of the halide (or sulfonate) to a palladium intermediate formed by metal-metal exchange with the nucleophile. Carbon-carbon bond formation occurs by reductive elimination. The heteroaromatic ring can be introduced either as the nucleophilic or electrophilic component. Detailed study has been directed to such issues as the oxidation state of palladium at the various stages and the role of the added ligands and anions which are important components of the reaction process. Synthetic optimization can frequently be achieved by adjustments in the ligands and added salts.

Both 1-methylpyrrol-2-ylmagnesium bromide and 1-methylpyrrol-2-ylzinc chloride give good yields in cross coupling with aryl iodides (Scheme 128) <81TL5319>.



Scheme 128

1-Boc-pyrrole has been coupled both through the 2-boronic acid derivative and the 2-trimethylstannyl derivative, both of which can be prepared from the lithiopyrrole <91S613>. Cross-coupling of 1-Boc-2,5-dibromopyrrole with 1-Boc-2-trimethylstannylpyrrole gave a tripyrrole (Scheme 129) <91SM403>.

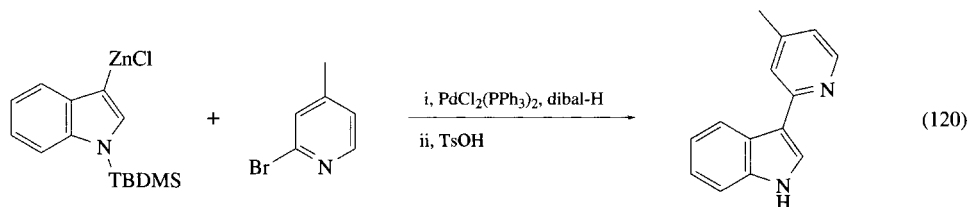


Scheme 129

1-tips-Pyrrole-3-boronic acid can also be coupled with aryl halides <92JOC1653>.

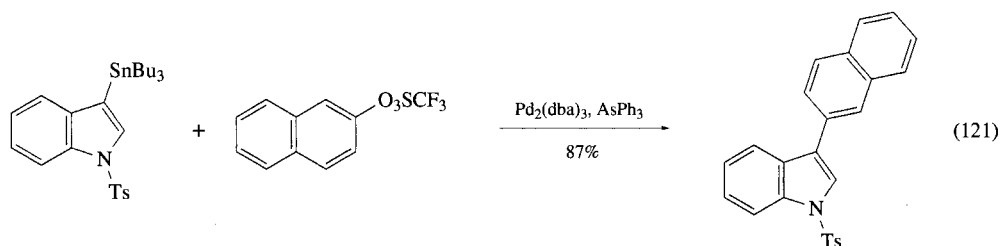
There are also a number of procedures for cross coupling of indoles. 3-Bromoindole protected with a 1-TBDMS group can be converted to 3-zinc chloride reagents by lithiation followed by

metal-metal exchange with ZnCl_2 . The zinc reagents couple with pyridyl and other heteroaryl halides (Equation (120)) <94TL793>.



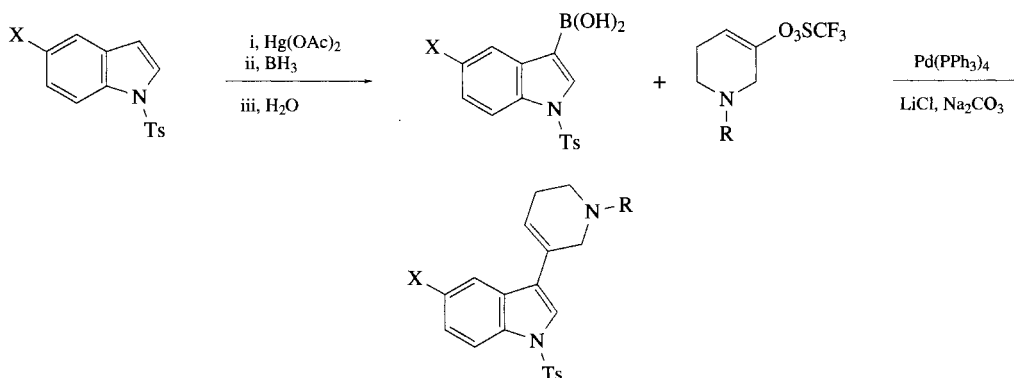
Several *N*-protecting groups were explored with indol-2-ylzinc reagents and the best results in cross-coupling with iodobenzene were obtained with the 1-Boc-derivative <93H(36)941>. Both 2-iodo and 3-iodo-1-phenylsulfonylindole have been converted directly to zinc reagents with active zinc and then coupled with aryl halides <93TL5955>.

Palladium can also catalyze cross-coupling between aryl halides and aryl or vinylstannanes and there are examples of application of this reaction in the synthesis of pyrroles and indoles. 1-Tosyl-3-(tri-*n*-butylstannyl)indole gives good yields of coupling products with vinyl and aryl iodides and triflates. The recommended catalyst is tris-(dibenzylideneacetone)-dipalladium (5 mol%) used with triphenylarsine (10 mol%) (Equation (121)) <94TL2405>.



A number of 2-aryl and 2-vinylindoles have been prepared by cross-coupling with 1-(trimethylsilylethoxymethyl)-2-(tri-*n*-butylstannyl)indole <94JOC4250>.

The Suzuki method for cross-coupling involves use of an arylboronic acid as the nucleophilic component. These conditions have been successfully applied to indoles. Indole-3-boronic acids, which can be prepared via electrophilic mercuration, can be coupled with vinyl triflates (Scheme 130) <93TL2235>.



Scheme 130

Indole-5-boronic acid has also been used successfully in Suzuki coupling <92H(34)1395>.

Organothallium derivatives have also been used in Suzuki coupling reactions. The 4-substituted thallation intermediate from indole-3-carboxaldehyde can be coupled with both aryl and vinyl-boronic acids (Equation (122)) <86CPB3971>.

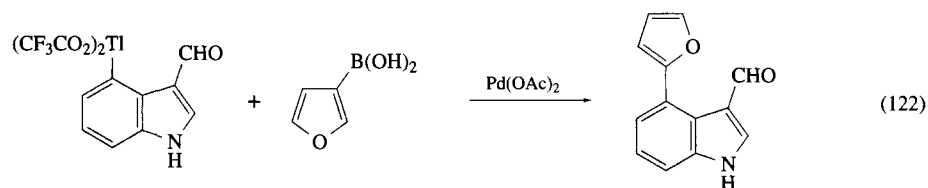


Table 8 gives some examples of palladium-catalyzed cross-coupling reactions.

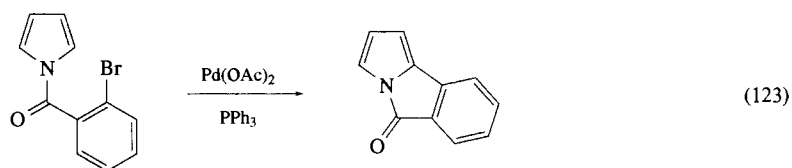
Table 8 Palladium-mediated cross-coupling reactions.

Entry	Reactant	Reagent	Catalyst	Yield (%)	Reference
A. Organomagnesium and organozinc reagents					
1	1-Methylpyrrol-2-yl-magnesium bromide	Iodobenzene	$\text{PdCl}_2(\text{dppb})$	93	81TL5319
2	1-Methylindol-2-yl-magnesium bromide	Iodobenzene	$\text{PdCl}_2(\text{dppb})$	79	81TL5319
3	1-Boc-indol-2-yl-zinc chloride	Iodobenzene	$\text{Pd}(\text{PPh}_3)_4$	68	93H(36)941
4	1-Phenylsulfonylindol-3-yl-zinc iodide	2-Bromopyridine	$\text{Pd}(\text{PPh}_3)_4$	83	93TL5955
5	1-Phenylsulfonylindol-2-yl-zinc chloride	2-Bromo-4-methylpyridine	$\text{PdCl}_2(\text{PPh}_3)_2$ dibal-H	92	93TL5005
6	1-TBDMS-indol-3-yl-zinc chloride	2-Bromopyridine	$\text{PdCl}_2(\text{PPh}_3)_2$ dibal-H	95	94TL793
B. Stannane intermediates					
7	1-SEM-2-tri- <i>n</i> -butylstannylindole	4-Cyanophenyl bromide	$\text{PdCl}_2(\text{PPh}_3)_2$	94	94JOC4250
8	1-(<i>p</i> -Toluenesulfonyl)-3-(tri- <i>n</i> -butylstannyl)indole	2-Naphthyl triflate	$\text{Pd}_2(\text{dba})_3 \text{AsPh}_3$	87	94TL2405
9	3-Bromo-1-methanesulfonyl-indole	2-Ethoxyvinyltri- <i>n</i> -butylstannane	$\text{Pd}(\text{PPh}_3)_2\text{Cl}_2$	83	91T1877
10	2-Bromo-3-formylindole	2-Methoxycarbonylvinyl-tri- <i>n</i> -butylstannane	$\text{Pd}(\text{OAc})_2, \text{R}_4\text{N}^+ \text{Cl}^-$	67	88H(27)1585
11	3-Formylindole ^a	3-Trimethylstannylpyridine	$\text{Pd}(\text{OAc})_2$	52	87CPB1322
C. Boronic acid intermediates					
12	1-tips-Pyrrole-3-boronic acid	4-Bromobenzaldehyde	$\text{Pd}(\text{PPh}_3)_4$	90	92JOC1653
13	1-(<i>p</i> -Toluenesulfonyl)indole-3-boronic acid	1-Methyl-3-trifluoromethanesulfonyloxypyridine	$\text{Pd}(\text{PPh}_3)_4$	90	93TL2235
14	6-Bromoindole	4-Fluorophenylboronic acid	$\text{Pd}(\text{PPh}_3)_4$	90	94SL93
15	Indole-5-boronic acid	Bromobenzene	$\text{Pd}(\text{PPh}_3)_4$	87	92H(34)1395

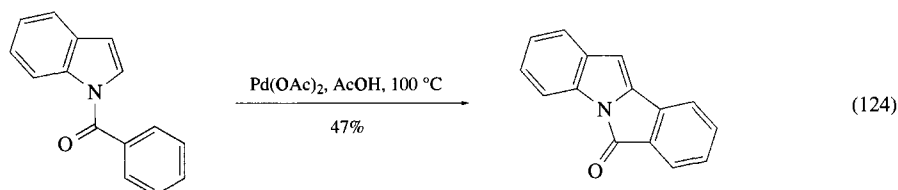
^aThallation at C-4 by $\text{Ti}(\text{O}_2\text{CCF}_3)_3$ prior to coupling.

Alkynes can be coupled with halopyrroles and haloindoles in the presence of a palladium catalyst and Cu^I . This reaction has been applied to both 3-iodo and 4-iodo indoles (<88CPB2248, 93H(36)2241>) and to 1-tips-3-iodopyrrole (<92JOC1653>).

Palladium can effect cyclization of pyrroles and indoles with appropriately placed haloaryl substituents. The reaction presumably involves generation of a palladium(0) species which undergoes oxidative addition at the carbon—halogen bond followed by electrophilic metalation to form a diarylpalladium intermediate (Equation (123)) (<90T4003, 91TL3317, 93TL5449>).

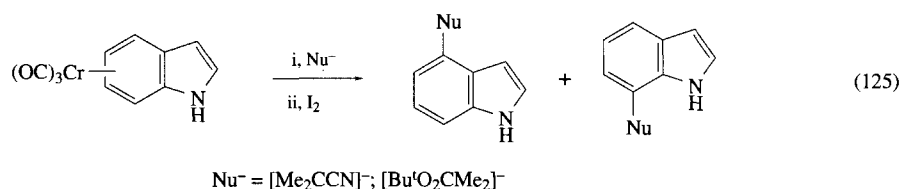


Similar cyclizations can be carried out by a stoichiometric amount of $\text{Pd}(\text{OAc})_2$ on compounds without a halogen substituent (Equation (124)) (<79S151>).

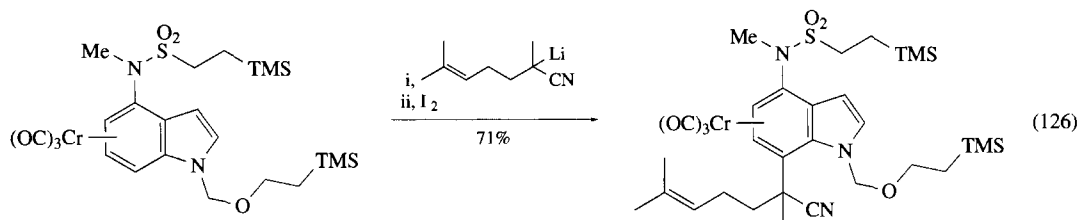


2.03.4.6 Synthesis Via π -Complexes

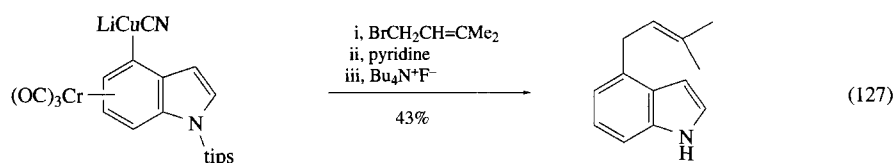
Pyrrole and indole form π -complexes with several transition metal species. The metal-containing groups are strongly electron-attracting so that the complexed ring becomes more susceptible to nucleophilic attack. The $\text{Cr}(\text{CO})_3$ complexes have been the most thoroughly explored. The complexes of various pyrroles $\langle 71\text{JOM}(30)211, 90\text{H}(31)383 \rangle$, indoles $\langle 68\text{JOM}(14)359 \rangle$ and of carbazole $\langle 82\text{JOM}(231)5 \rangle$ have been characterized. Complexation to the benzenoid ring is stronger than to the pyrrole ring and $\text{Cr}(\text{CO})_3$ is readily transferred from pyrroles to benzenoid compounds $\langle 93\text{JOM}(470)\text{C4} \rangle$. In indoles, complexation of the carbocyclic ring permits substitution by nucleophilic addition. The complex of indole itself gives 4- and/or 7-substitution products with nucleophiles such as the anion of isobutyronitrile (97 : 3) and *t*-butyl isobutyrate (99 : 1) (Equation (125)) $\langle 81\text{T3957} \rangle$.



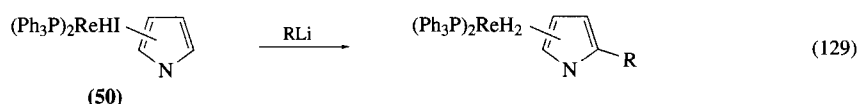
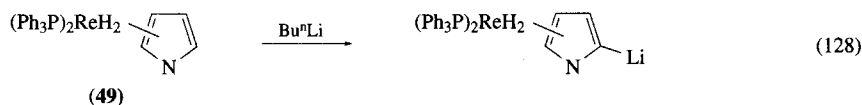
2-Lithio-1,3-dithian is also an effective nucleophile but it shows selectivity for C7 substitution (14 : 86) $\langle 81\text{T3957} \rangle$. The substituted indoles are obtained from adducts by oxidation with iodine. The $\text{Cr}(\text{CO})_3$ complexes of $\text{N}^a\text{-N}^b$ -diprotected 4-(methylamino)indoles undergo nucleophilic addition at C7 by anions of tertiary nitriles (Equation (126)) $\langle 93\text{TL1399} \rangle$.



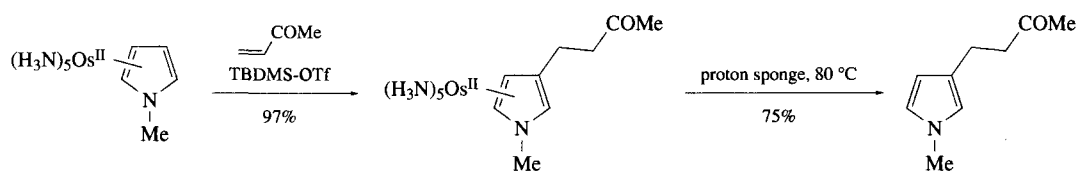
The $\text{Cr}(\text{CO})_3$ complexes of indole are also activated toward lithiation. The complex of 1-methylindole is lithiated at C2 by *n*-butyllithium at -78°C . If C2 is blocked by prior silylation, lithiation occurs primarily at C7 although there is some C4 lithiation $\langle 81\text{CC1260} \rangle$. Use of the 1-(SEM) protecting group achieves clean C7 lithiation $\langle 89\text{T5955} \rangle$. 1-(tips) Indole- $\text{Cr}(\text{CO})_3$ is selectively lithiated at C4 and 4-substituents can be introduced by electrophiles such as ethyl chloroformate and isopentenyl bromide $\langle 82\text{CC467} \rangle$. The 4-indolylcopper derivative can be prepared using CuBr-SMe_2 and it undergoes coupling with allylic and vinylic bromides (Equation (127)) $\langle 88\text{T7325} \rangle$.



Although they have not yet been developed to the point of synthetic application, pyrrole-rhenium complexes display the reactivity expected for a very strongly electron-attracting substituent. Complex (49) undergoes ring lithiation readily (Equation (128)). Use of the iodo rhenium complex (50) leads to addition at C2 (Equation (129)) $\langle 87\text{JOM}(333)71, 89\text{JOM}(362)\text{C31} \rangle$.

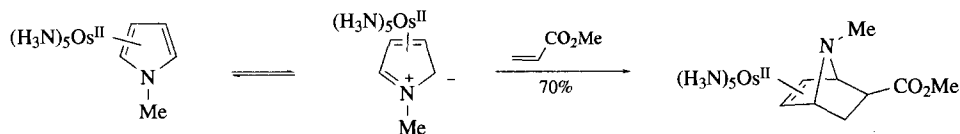


Harman and coworkers have explored the reactivity of η^2 -osmium complexes of pyrroles. These complexes can react as nonaromatic species in which the Os has localized two of the π -electrons. For example, the complex of 1-methylpyrrole gives a conjugate addition product with methyl vinyl ketone in the presence of TBDMS triflate <93JOC4788>. Decomplexation by reaction with a base gives a 3-substituted pyrrole (Scheme 131).



Scheme 131

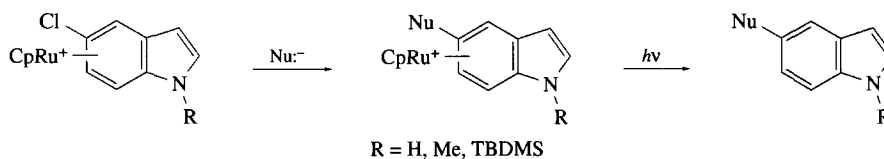
In the absence of TBDMS-triflate, a [3 + 2] cycloaddition occurs. This is attributed to reaction through a minor isomer which is in dynamic equilibrium with the major complex (Scheme 132).



Scheme 132

Another type of structure which has been examined are $[\text{RuCp}]^+$ complexes of 5-chloroindoles <88OM660>. These complexes undergo nucleophilic substitution by amine, alkoxide, thiolate and malonate nucleophiles (Scheme 133).

At the present time these π -complexes have only specialized synthetic applications but as their chemistry is further developed they may provide entirely new synthetic approaches.



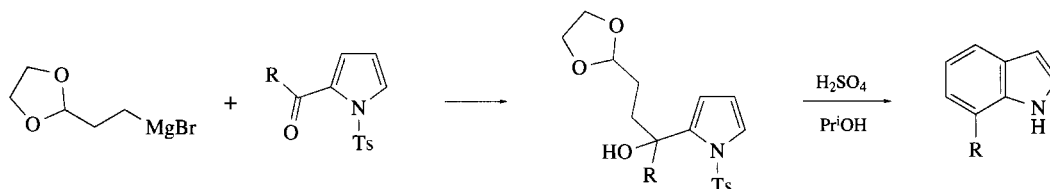
Scheme 133

2.03.5 SYNTHESIS INVOLVING ANNELEMENT OF PYRROLES AND INDOLES

2.03.5.1 Annellation by Electrophilic Condensation

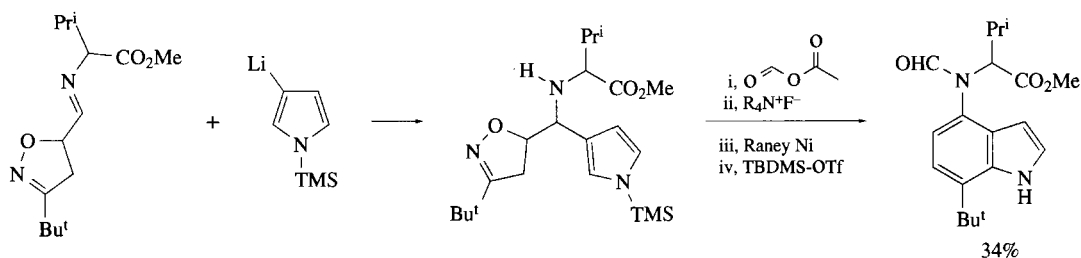
Although most indole and carbazole ring syntheses involve the construction of the nitrogen-containing ring, it is sometimes advantageous to assemble a carbocyclic ring on an existing pyrrole or indole structure. One approach to annellation is to build a substituted side-chain which can be cyclized by an electrophilic attack on the nitrogen-containing ring. A fairly general protocol for construction of 4-alkyl or 7-alkylindoles involves addition of 2-(dioxolanyl)ethylmagnesium

bromide to an *N*-protected 2-acyl or 3-acyl pyrrole. The combination of the masked aldehyde and carbinol set the stage for cyclization and aromatization. Yields of 7-substituted indoles of 65–80% were obtained with sulfuric acid-isopropanol as the cyclization medium (Scheme 134) <9H(29)783, 90H(31)683>.

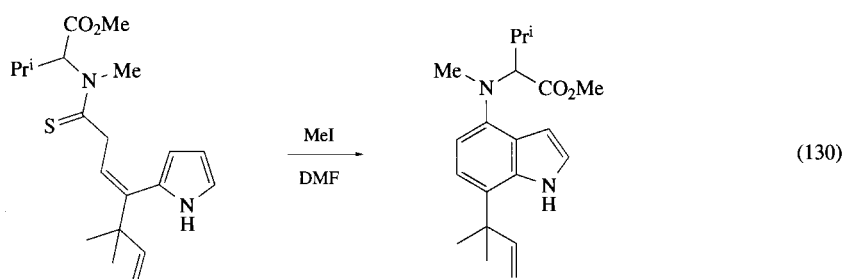


Scheme 134

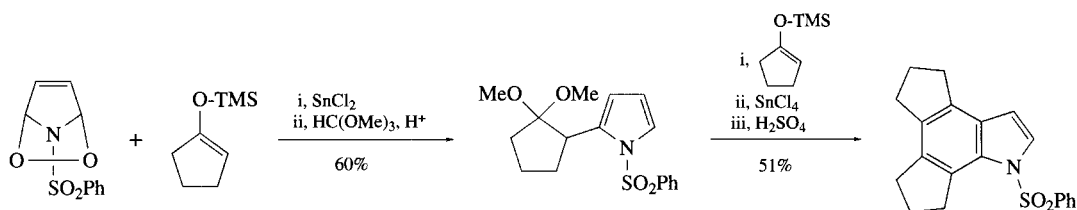
Annulation methodology has been used to prepare 4-amino-7-alkylindoles of the type found in the lyngbyatoxin group of natural products <89JA6228>. An interesting example of this type of synthesis is the use of the oxazoline ring as a source of both the electrophilic carbon and a hydroxyl group which can be eliminated to effect aromatization (see Scheme 135). The lyngbyatoxin structure has also been achieved using a thioamide as the electrophile <90T5113> (Equation (130)).



Scheme 135

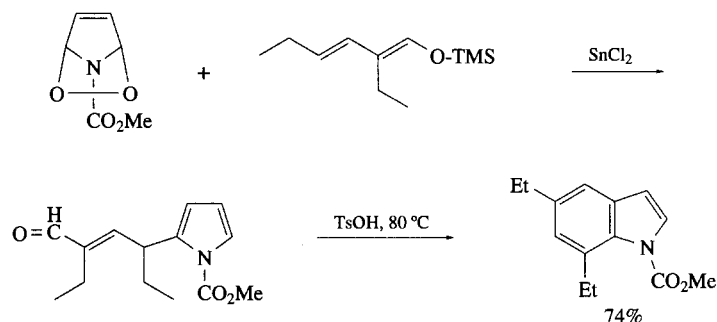


Another means of putting a functionalized side chain in place is by nucleophilic addition to the endoperoxides obtained by singlet oxygen oxidation of *N*-protected pyrroles. The initial adduct is converted to an acetal which can be elaborated by Mukaiyama condensation conditions (Scheme 136) <90H(31)691>.



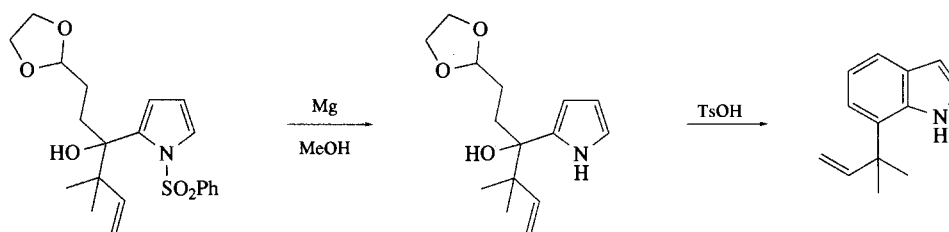
Scheme 136

Alternatively, the side-chain can be constructed by nucleophilic addition of a silyloxydiene promoted by stannous chloride (Scheme 137) <89H(29)771>.



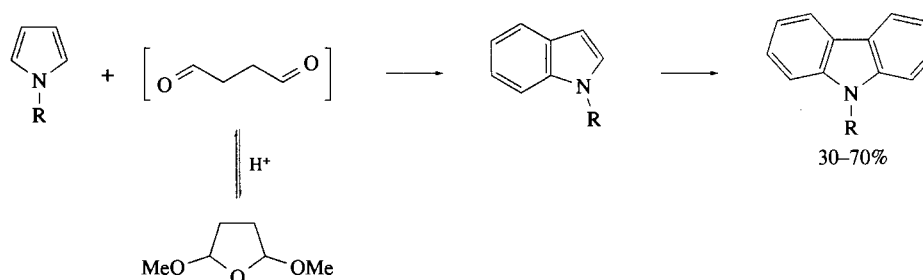
Scheme 137

Cyclization can be carried out on either protected or unprotected pyrroles, with the latter being more reactive. There are examples where the protected reactant can not be cyclized but cyclization occurs successfully with the deprotected compound (Scheme 138) <89H(29)783>.



Scheme 138

Electrophilic annelation has also been used to convert indoles to carbazoles. The condensation of amines with 2,5-dimethoxytetrahydrofuran, which is a very useful method of synthesis of pyrroles (see Section 2.03.2.7), if carried out for an extended period, gives *N*-substituted carbazoles. This reaction occurs by two successive annelations (Scheme 139) <86T2131>.

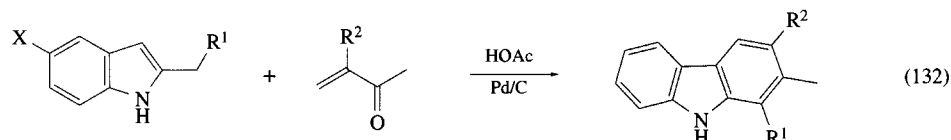


Scheme 139

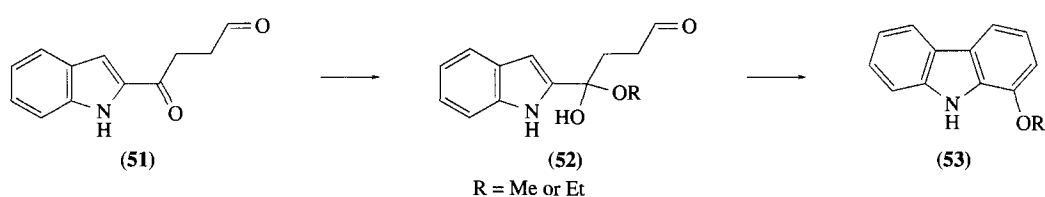
Substituted carbazoles have been made by acid-catalyzed condensation of indole with 2,5-hexanedione (Equation (131)) <67AJC2715, 77JCS(P1)1698, 87CPB425, 90H(31)401>.



Michael adducts of enones and 2-substituted indoles such as 2-methyl or 2-benzylindoles can be cyclized to carbazoles in acetic acid in the presence of Pd/C. This reaction involves an electrophilic addition at C3 and aromatization (Equation (132)) <88T5215>.



1-Alkoxy carbazoles are formed when the aldehyde (**51**) is heated in acidic methanol or ethanol. The introduction of the alkoxy group evidently occurs because cyclization is faster through an acetal or hemiacetal intermediate than through the less reactive 2-acylindole (Scheme 140) <88JCS(P1)235>.



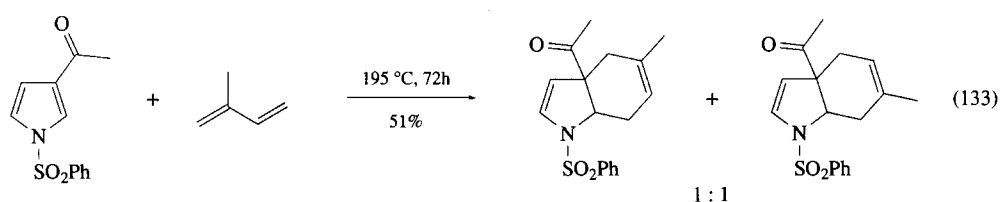
Scheme 140

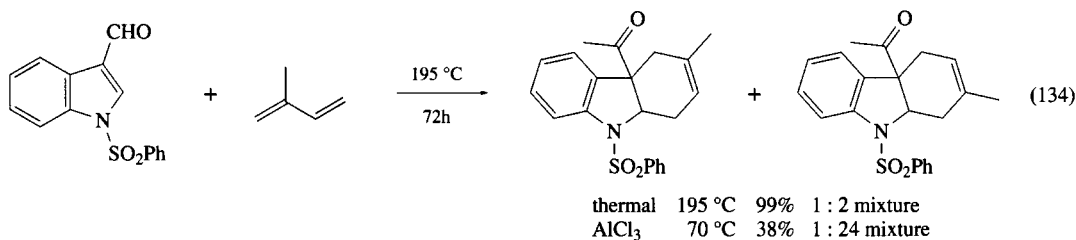
2.03.5.2 Cycloadditions as Annellation Reactions

Another strategy for annellation of pyrroles and indoles involves cycloaddition reactions. Because the heteroaromatic rings have substantial aromatic stabilization, the C2—C3 bond is not very reactive toward cycloaddition and there are only a few examples of the heterocycles acting as dienophiles. The carbocyclic ring of indole, like benzene, is unreactive toward cycloaddition. However, vinylpyrroles and indoles are quite reactive and react as electron-rich dienes because of the electron-donating nature of the ring. Other cycloaddition reactions involve the 2,3-dimethylene derivatives of pyrrole and indole, the so-called quinodimethanes, which are very reactive dienes. These intermediates, and more stable synthetic equivalents, are useful in cycloadditions with a variety of dienophiles. 2*H*-Isoindole, which itself can be thought of as a quinodimethane, is a very reactive diene.

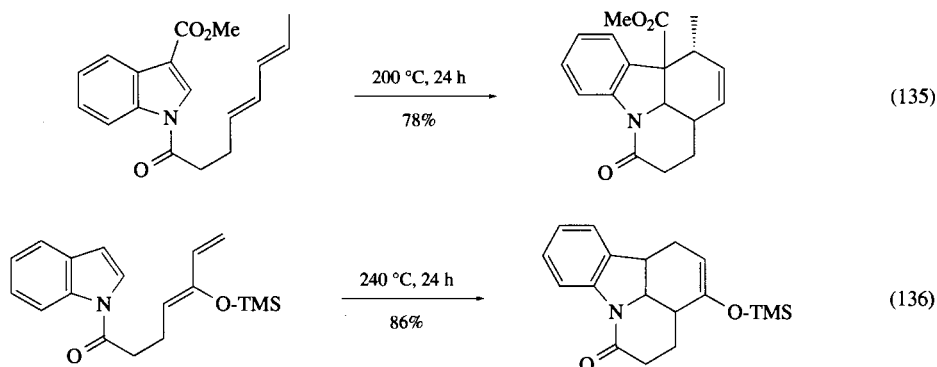
2.03.5.3 Pyrroles and Indoles as Dienophiles

There are a few reactions in which pyrroles and indoles act as dienophiles at the 2,3-double bond. These reactions typically occur with strong N-EW substituents which tend to diminish the aromatic character of the heterocycle. An additional EW-substituent activates the 2,3-bond toward addition to conventional dienes. This reaction is accelerated by AlCl₃ and the regioselectivity is also improved under catalyzed conditions (Equations (133) and (134)) <88JA7188>.

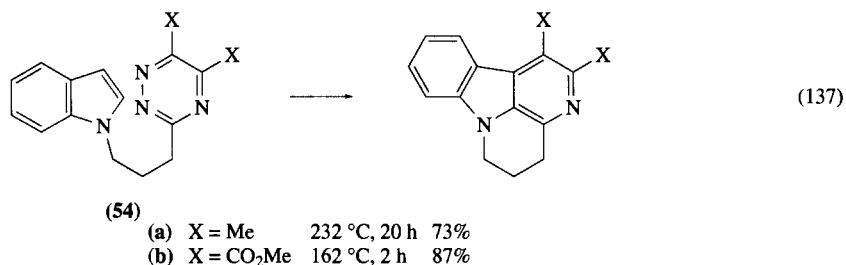




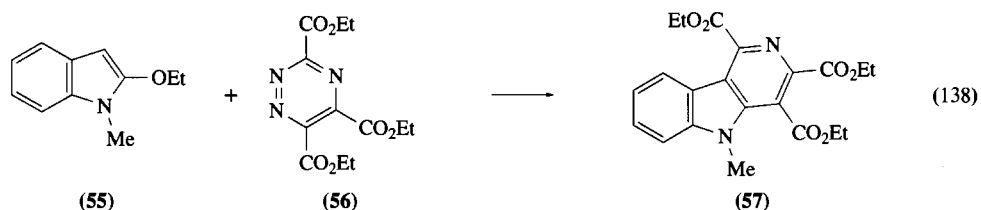
Examples of indole 2,3-bonds acting as dienophiles have been observed in situations where intramolecular addition is possible (Equations (135) and (136)) <88TL5605>.



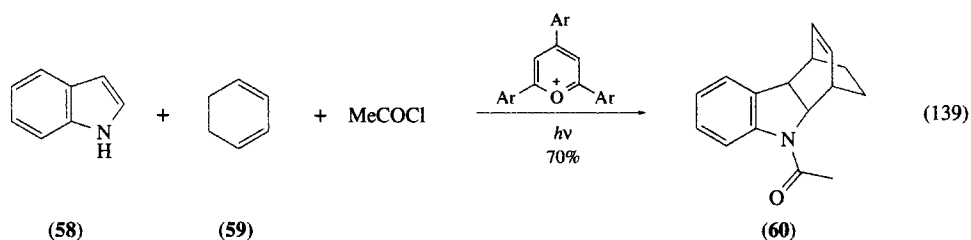
Examples of reactions in which the 2,3-bond acts as a dienophile in an intramolecular inverse electron demand Diels–Alder cycloaddition have also been recorded. The diester (**54b**) was more reactive than (**54a**), as would be expected (Equation (137)) <92JOC5285>.



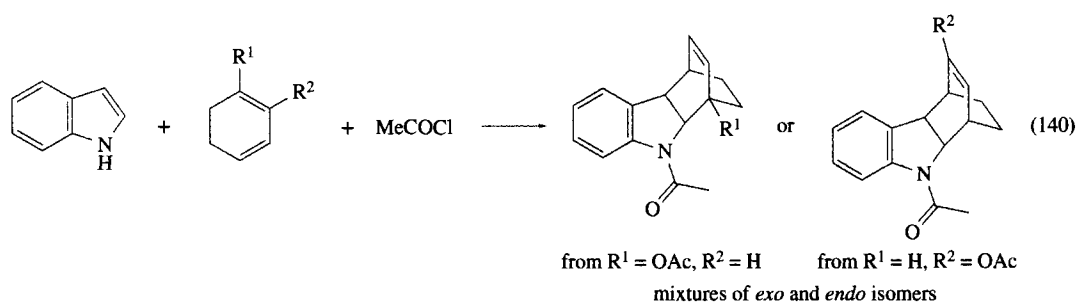
The electronic character of the indole ring can be adjusted by introduction of substituents so as to become more reactive as a dienophile in inverse-electron-demand Diels–Alder reactions. For example, 2-ethoxy-1-methylindole reacts with the electrophilic heterocycle (**56**) (Equation (138)) <87ZN(B)1032>.



The indole ring can act as a dienophile under the conditions of electron-transfer catalysis. In this mechanism, the indole radical cation is generated as a transient intermediate by single electron oxidation. Indole (**58**) and cyclohexadiene (**59**) react to give (**60**) in the presence of triarylpyrylium salts which act as photosensitizers. The reaction is carried out in the presence of acetyl chloride which traps and stabilizes the adduct (Equation (139)) <90SL275, 91JOC1405>.



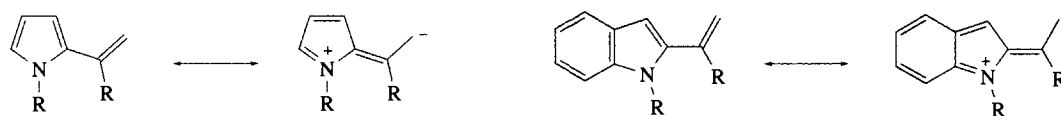
Substituted cyclohexadienes show good regioselectivity which corresponds to that expected if C3 is the dominant electrophilic center of the radical cation LUMO (Equation (140)).



2.03.5.4 Cycloaddition Reactions of Vinylpyrroles and Vinylindoles

The most fully explored synthetic applications of Diels–Alder reaction in annelation of pyrroles and indoles involves use of vinyl derivatives. Because of the donor character of the heteroaromatic ring, vinylpyrroles and vinylindoles react as electron-rich dienes. Various aspects of both the synthesis and cycloaddition reactions of vinylindoles have been reviewed. <85JHC585, 86CZ95, 88H(27)1253>.

A qualitative comparison of several α -substituted 1-methyl-2-vinylindoles found them to be 10–100 times less reactive than the corresponding pyrroles toward dimethyl ethynedicarboxylate. This appears to be the result of a combination of two factors. First, because of the quinoid character of the structure in the indole case, an indole ring is expected to be a poorer donor than a pyrrole ring to a 2-vinyl substituent. This is probably reinforced by a steric buttressing effect of the 7H on the indole ring which would tend to disfavor the *cisoid* conformation when $R > \text{H}$ (Scheme 141) <84T4837>.

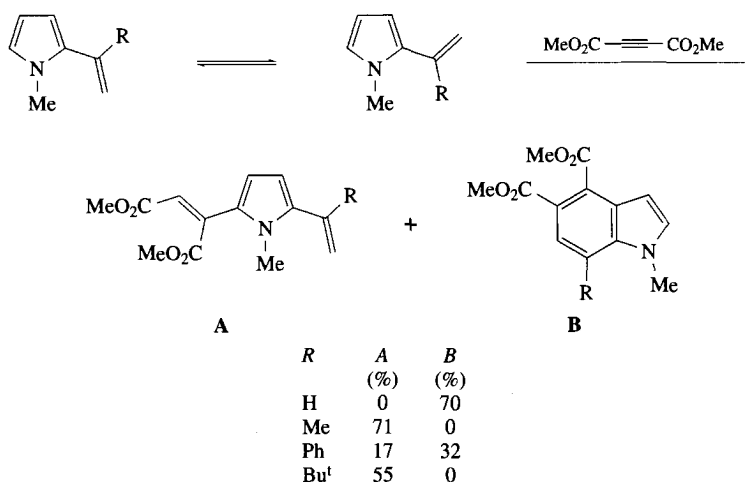


Scheme 141

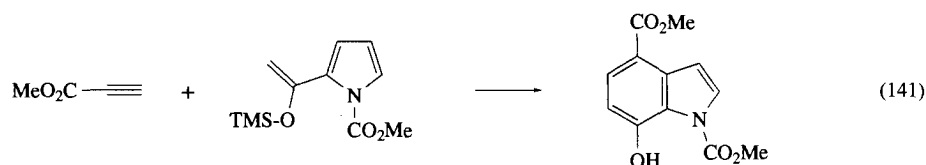
The use of unsubstituted 2-vinylindoles also tends to be complicated by their limited stability, although this problem can be diminished by use of EW groups on nitrogen <71JOC1759>. There are cases, such as with diethyl diazodicarboxylate, where electrophilic substitution at C3 competes with cycloaddition <88H(27)1253>.

A study of a series of 2-vinylpyrroles with α -substituents of increasing size revealed that alkyl groups prevented [4 + 2] cycloaddition with dimethyl ethynedicarboxylate. This is attributed to steric restriction on access to the necessary *cisoid* conformation. The phenyl analogs gave 6,7-dihydroindole-4,5-dicarboxylate esters along with the substitution product (Scheme 142) <84JCS(P1)2541>.

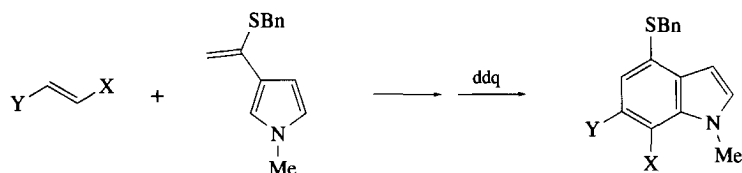
Electron-donor substituents on the vinyl group further enhance reactivity. Methyl 2-(1-trimethylsilyloxyethyl)-pyrrole-1-carboxylate reacts with methyl propynoate to give a 4,7-disubstituted indole (Equation (141)) <91H(32)1199>.



Scheme 142

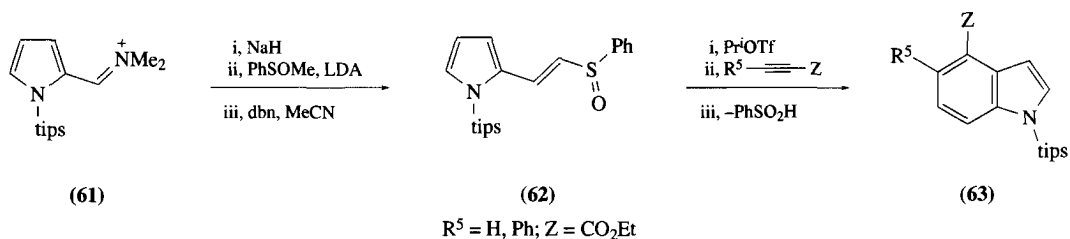


A similar reaction has been observed with 1-methyl-3-(1-benzylthioethenyl)pyrrole. Adducts can also be obtained from dienophiles such as acrylonitrile, acrolein, methyl acrylate, and diethyl fumarate (91CPB489). In this case the initial products are tetrahydroindoles which can be aromatized with ddq (Scheme 143).



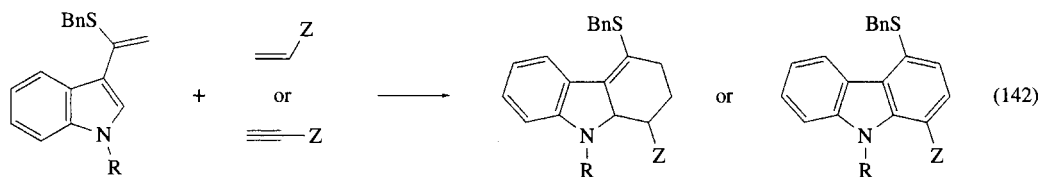
Scheme 143

1-tips-2-(2-Phenylsulfinylvinyl)pyrrole, which can be made from the Vilsmeier intermediate (61), reacts with alkynes to give 4,5-disubstituted indoles (87TL3453). In this case, elimination of sulfinate accomplishes the aromatization (Scheme 144).



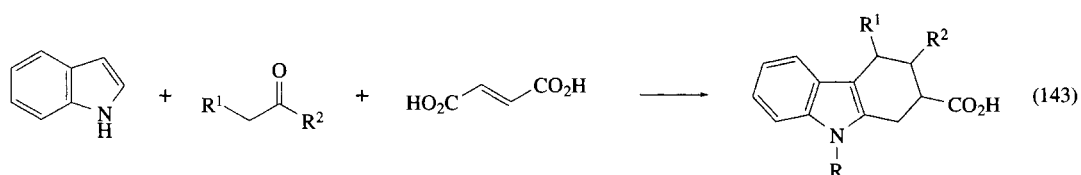
Scheme 144

The reactivity of 3-vinylindoles is also enhanced by an ER group at the vinyl substituent. 3-Thioacetylindole can be converted to 3-(1-benzylthioethenyl) indole by treatment of the conjugate base with benzyl chloride. This derivative reacts with a range of activated alkenes and alkynes to give 1,2-dihydrocarbazoles or carbazoles, respectively (89CPB1999). It is interesting that in the case of the alkynic dienophiles aromatization occurs by loss of hydrogen rather than by elimination of the thiol (Equation (142)).

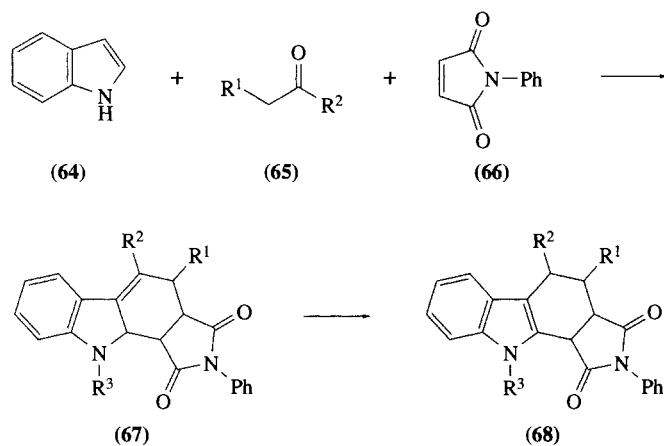


A number of successful cases of Diels–Alder reactions involving conversion of vinyl pyrroles to indoles and vinylindoles to carbazoles are illustrated in Table 9.

In some cases it is possible to obtain Diels–Alder adducts from vinylindole intermediates which are generated *in situ*. The yields in such reactions are usually not very high but the simplicity of operation is a compensating factor. For example, heating indole, a ketone and maleic acid together gives 1,2,3,4-tetrahydrocarbazole-2-carboxylic acids in around 20% yield (Equation (143)) <79JOC4402>.



This reaction presumably proceeds by electrophilic substitution at C3 and dehydration of the resulting carbinol, followed by cycloaddition. The reaction also involves the decarboxylation of an indole-2-acetic acid, which is known to be facile. Similarly, reaction of indole, a carbonyl compound and *N*-phenylmaleimide generates tetrahydrocarbazoles of structure (68). The yields vary from 15–20% for simple aldehydes to 70% and above for cyclic ketones <93JHC81>. The stereochemistry, which is predominantly all-*cis*, arises as the result of an *endo* addition followed by protonation from the less hindered side of the molecule in the step resulting in aromatization (Scheme 145).



Scheme 145

Intramolecular versions of vinylindole cycloaddition can occur with suitable reactants (Equation (144) and Scheme 146) <87JOC4661, 90JOC1624>.

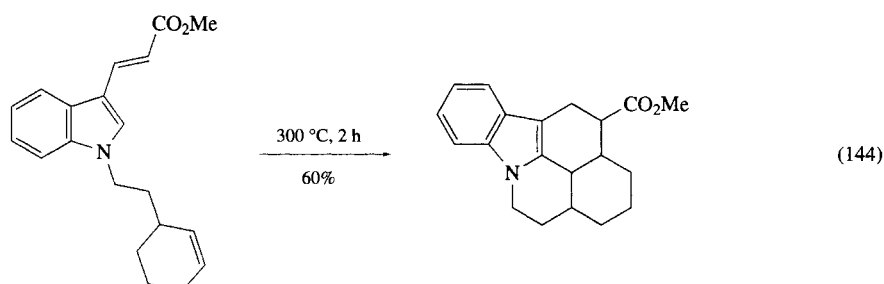


Table 9 Diels–Alder reactions of vinylpyrroles and vinylindoles.

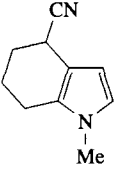
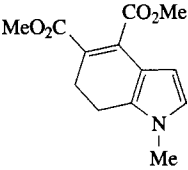
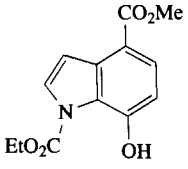
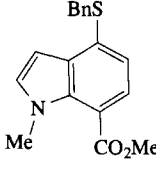
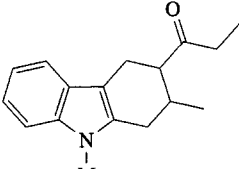
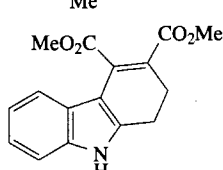
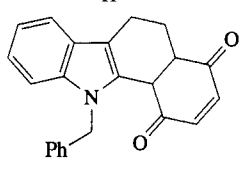
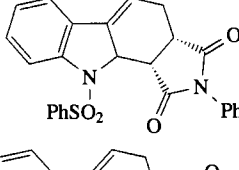
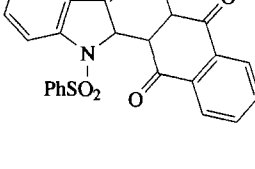
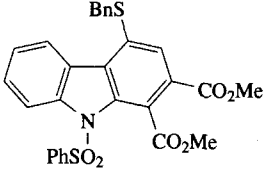
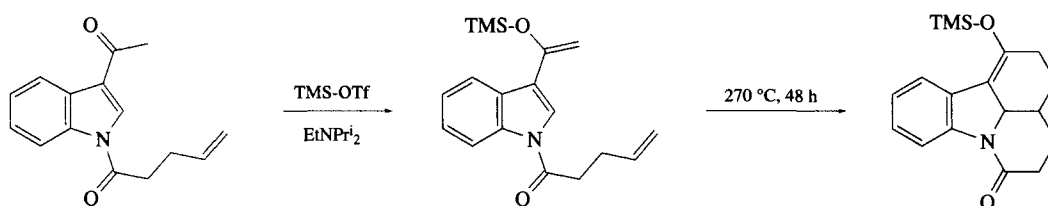
Entry	Diene	Dienophile	Product	Yield (%)	Reference
A. Vinylpyrroles					
1	1-Methyl-2-vinyl	Acrylonitrile		61	80JOC4515
2	1-Methyl-2-vinyl	Dimethyl ethyne dicarboxylate		70	81T1597
3	1-Ethoxycarbonyl-2-(1-trimethylsilyloxyvinyl)	Methyl propynoate		17	91H(32)1199
4	1-Methyl-3-(1-benzylthiovinyl)	Methyl acrylate		31	91CPB489
B. Indoles					
5	2-(1-Propenyl)	Pent-1-en-3-one		46	88H(27)2353
6	2-Vinyl	Dimethyl ethynedicarboxylate		46	88HCA1060
7	1-Benzyl-3-vinyl	Benzoquinone		86	81AJC1483
8	1-Phenylsulfonyl-3-vinyl	N-Phenylmaleimide		60	88HCA467
9	3-(2-Methoxyvinyl)-1-phenylsulfonyl	Naphthoquinone		86	92JOC910

Table 9 Continued.

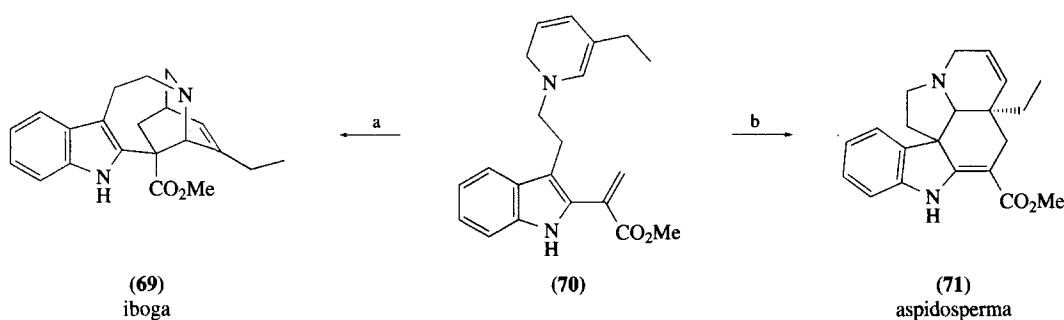
Entry	Diene	Dienophile	Product	Yield (%)	Reference
10	3-(1-Benzylthiovinyl)	Dimethyl ethyne-dicarboxylate		70	89CPB1999

^aInitial adduct was aromatized with ddq.



Scheme 146

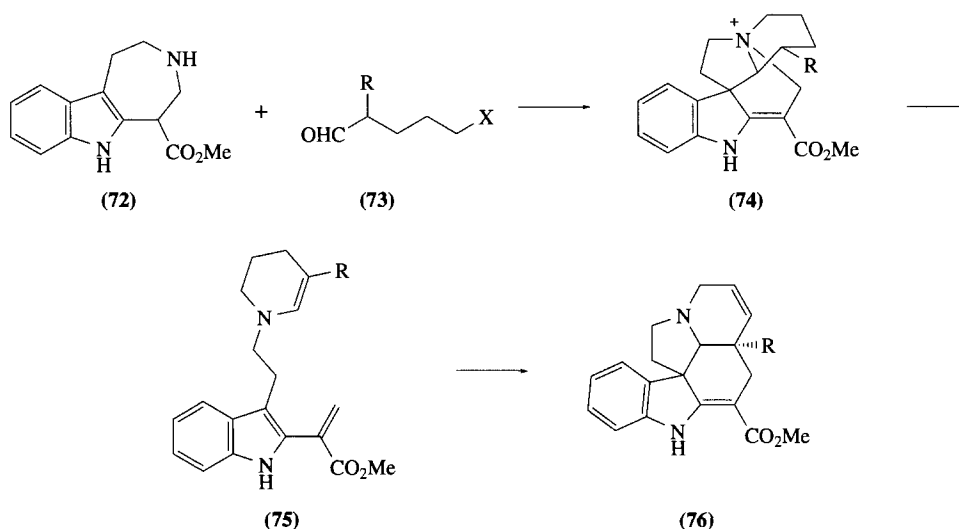
Two alternative intramolecular Diels-Alder reactions of the putative alkaloid precursor dehydrosecondine (**70**) have been postulated as a branch point in the biosynthesis of *aspidosperma* and *iboga* alkaloids <62JA98>. Reaction by path a, in which the acrylate moiety acts as the dienophile generates the *iboga* skeleton, (**69**) while path b, with the vinylindole acting as a diene, gives the *aspidosperma* structure (**71**) (Scheme 147).



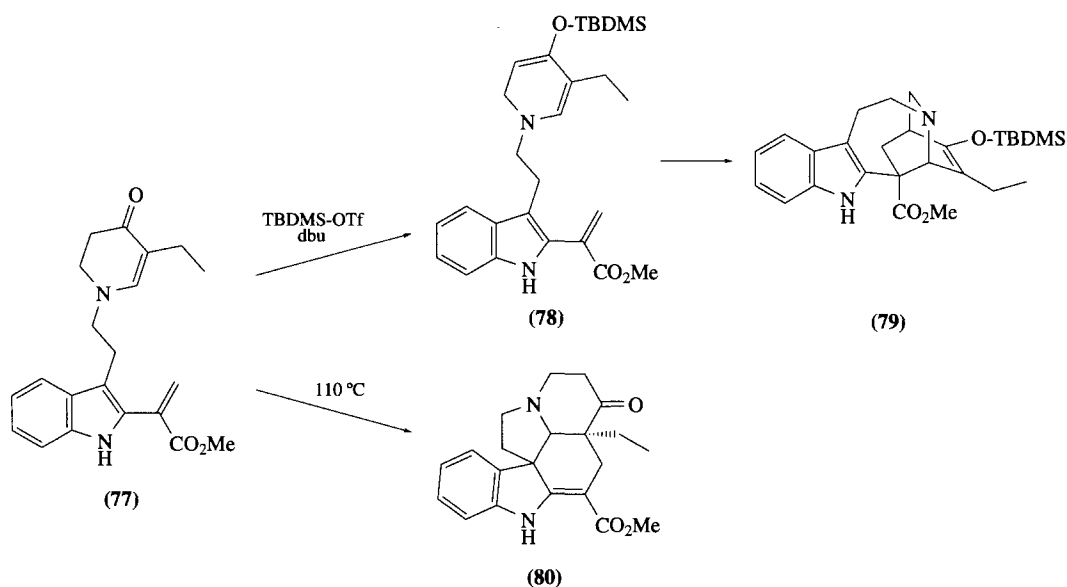
Scheme 147

This biogenetic proposal has spurred interest in the synthesis of structures such as (**70**). An especially efficient example of the use of intramolecular Diels-Alder reactions to synthesize *aspidosperma* alkaloids has been developed by Kuehne and co-workers <83T3715, 85JOC924, 85JOC4790, 87JOC347>. The azepino[4,5-*b*]indole structure (**72**) is condensed with a δ -haloaldehyde generating (**74**), which undergoes fragmentation to (**75**) which contains the secodine synthon. With the secodine type structure only cycloaddition path b is available and *aspidosperma* structures are formed (**76**) (Scheme 148).

Incorporation of an α,β -*cis* double bond generates dehydrosecodeine, the putative biosynthetic intermediate. By using the piperidone (**77**) as a starting material the reaction can be directed to either the *aspidosperma* or *iboga* skeleton. The ketone (**77**) cyclizes to (**80**) but conversion to the TMS enol ether (**78**) results in the *iboga* structure (**79**) (Scheme 149) <86JOC2913>.



Scheme 148

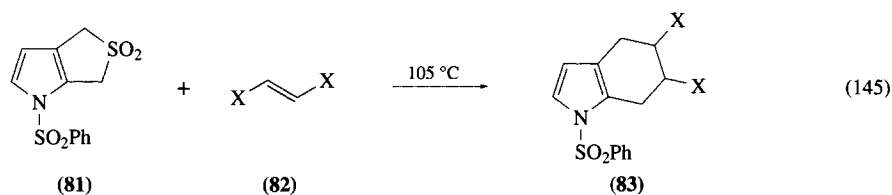


Scheme 149

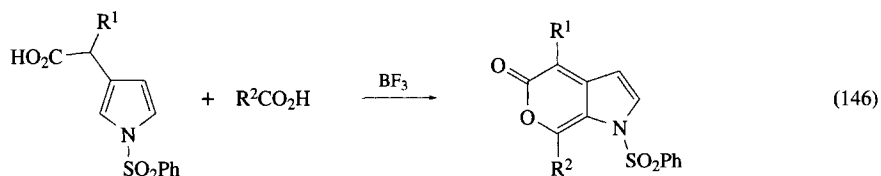
Use of related intermediates has led to the synthesis of several alkaloids, including the anti-neoplastic alkaloids of the vinblastine type <90MI 203-01>.

2.03.5.5 Annulation Through Quinodimethane Intermediates

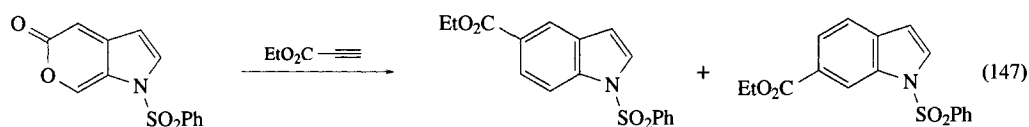
The quinodimethane derivative of benzene is known to be an extremely reactive diene with the aromaticity of the adduct contributing strongly to the driving force for the reaction. Substantial interest has developed in the synthetic potential of the corresponding pyrrole and indole quinodimethanes and several routes to such intermediates have been developed <89CR1681>. The sulfone (81) has been synthesized. By thermal elimination of SO₂ 2,3-dimethylenepyrrole is generated and it gives the expected adducts when trapped by dimethyl fumarate or *N*-phenylmaleimide (Equation (145)) <92CC549>.



Pyrano[3,4-*b*]pyrrol-5-ones function as pyrrole-2,3-quinodimethane equivalents. These compounds can be obtained by BF_3 -mediated cyclization of 1-(phenylsulfonyl)pyrrole-3-acetic acid with carboxylic acids. Use of α -substituted pyrrole-3-acetic acids can introduce a 4-substituent (Equation (146)) <90JCS(P1)2156>.

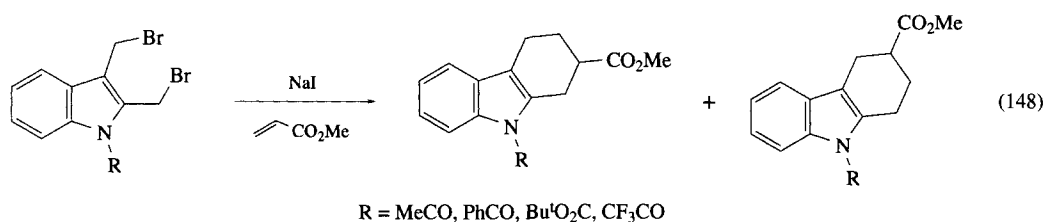


Reaction of the parent compound with ethyl propynoate gives a 1:1 mixture of 5- and 6-indolecarboxylate esters (Equation (147)) <90JCS(P1)2156>.

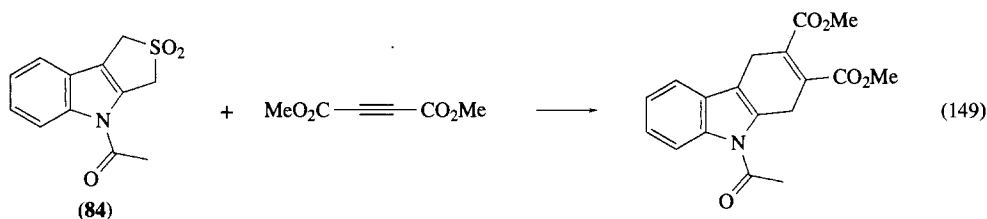


Symmetrical dienophiles such as dimethyl ethynedicarboxylate and benzyne avoid this regiochemical problem. Vinylsulfones can serve as ethyne equivalents in this reaction <90JCS(P1)2156>.

Indole-2,3-quinodimethanes stabilized by 1-EW groups can be generated by treatment of the corresponding 2,3-bis-(bromomethyl)indoles with sodium iodide. This reaction proceeds by halide exchange and reductive elimination of iodine. Methyl acrylate can trap the intermediate <84TL5429, 89TL7289>. There is a slight preference for the 2-substituted tetrahydrocarbazole (Equation (148)).

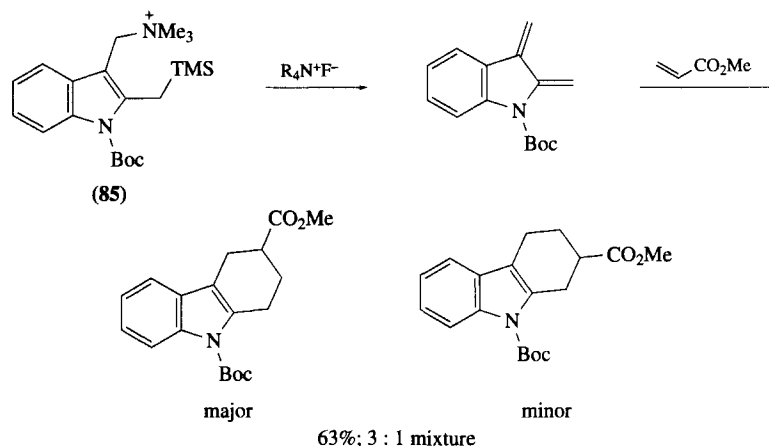


The quinodimethane can also be trapped by SO_2 which gives the sulfone (**84**), which can itself serve as a quinodimethane precursor (Equation (149)).



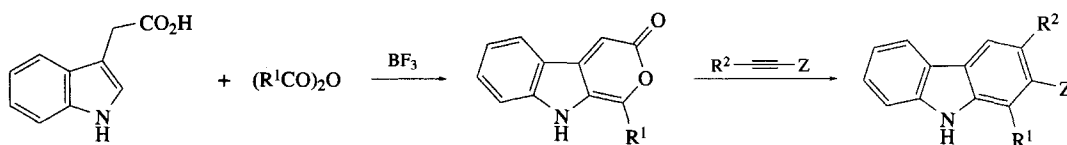
Another indole-2,3-quinodimethane precursor is the Boc-protected 2-(TMSmethyl)gramine

methiodide (**85**) which is converted to the quinodimethane on treatment with fluoride ion (Scheme 150) <82TL2745>.



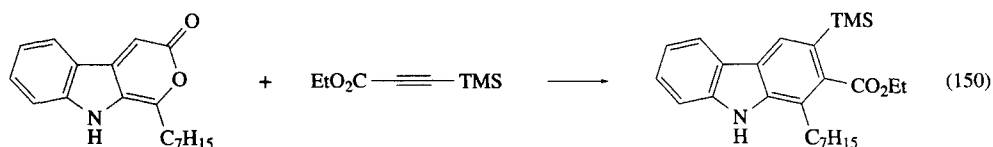
Scheme 150

Pyrano[3,4-*b*]indol-3-ones are useful indole-2,3-quinodimethane surrogates. Various 1-substituted derivatives can be prepared by BF_3 -catalyzed condensation of indole-3-acetic acid with carboxylic anhydrides <88JCS(P1)1407>. These compounds act as dienes toward electrophilic alkynes to give carbazoles. The reaction is not subject to strong regiochemical control and mixtures of both possible adducts are usually observed, although internal alkynes give mainly products with the EW group at C2 of the carbazole ring (Scheme 151).

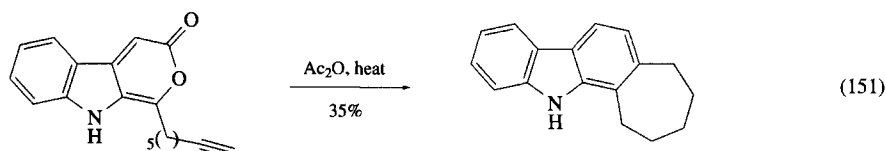


Scheme 151

Ethyl 3-(TMS)propynoate has been found to be a highly regioselective dienophile and the silyl substituent can be readily removed by protonolysis or used as a site for further reaction (Equation (150)) <91JCS(P1)2941>.

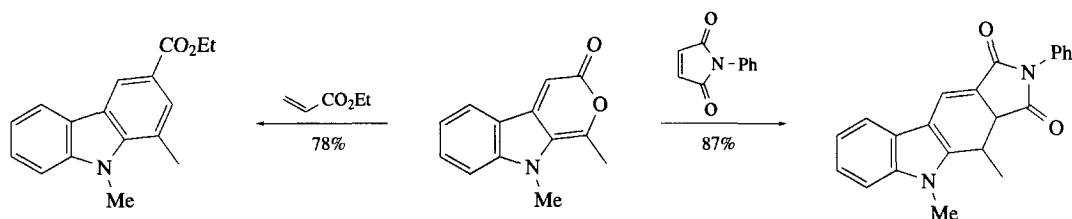


Terminal alkynes lacking an EW group react only slowly and give 1,3-disubstituted carbazoles. The reaction can also occur intramolecularly (Equation (151)) <88JCS(P1)3249>.

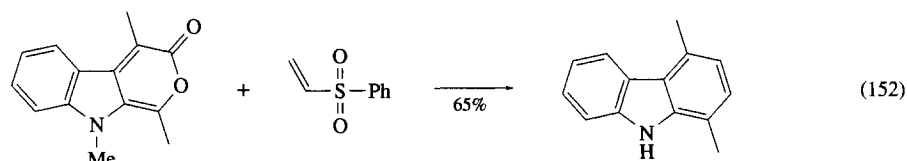


Dienophiles such as methyl vinyl ketone, acrylonitrile, methyl acrylate, and *N*-phenylmaleimide give products which are either 1,2-dihydrocarbazoles or fully aromatized carbazoles, depending upon the substituent (Scheme 152) <89T6761>.

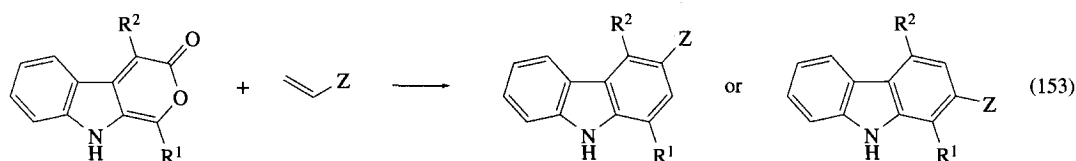
The use of phenyl vinyl sulfone or phenyl vinyl sulfoxide as the dienophile allows for aromatization by elimination of the sulfur substituent (Equation (152)) <89LA1275>.



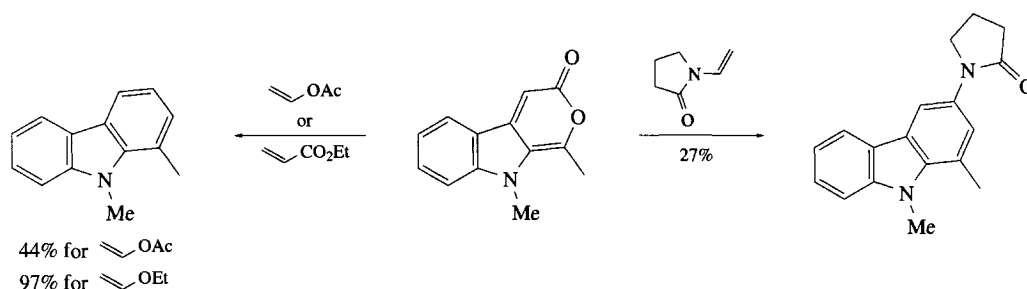
Scheme 152



The regioselectivity of additions with typical dienophiles is low for the 1-methylpyrano[3,4-*b*]indol-3-ones but the 1,4-dimethyl derivative shows a fairly strong preference for the activating EW group at the 3 position of the carbazole ring (Equation (153)) <89H(29)11>.



Pyrano[3,4-*b*]indol-3-ones also react with alkenes having electron-donor substituents such as ethyl vinyl ether, vinyl acetate, dihydropyran and *N*-vinylpyrrolidone. The oxygen substituents are eliminated to provide fully aromatized carbazoles. The amido substituent, on the other hand, is retained (Scheme 153) <92JCS(P1)415>.

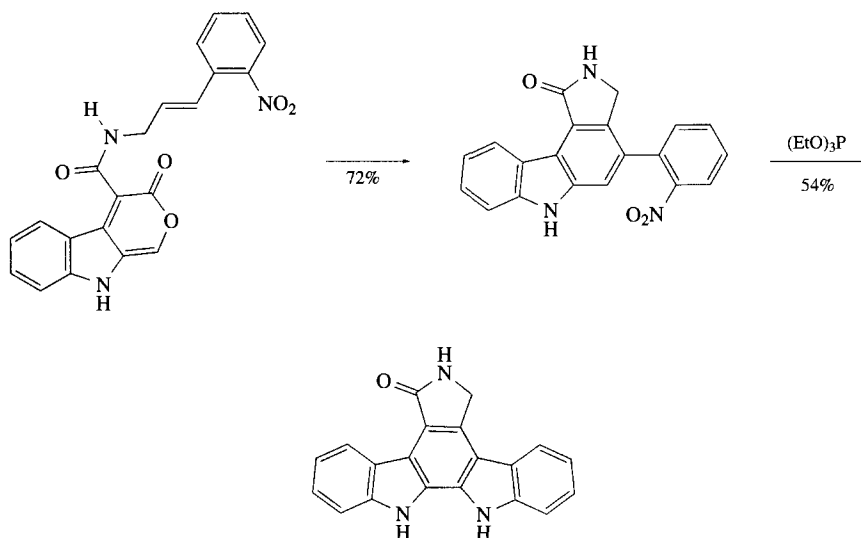


Scheme 153

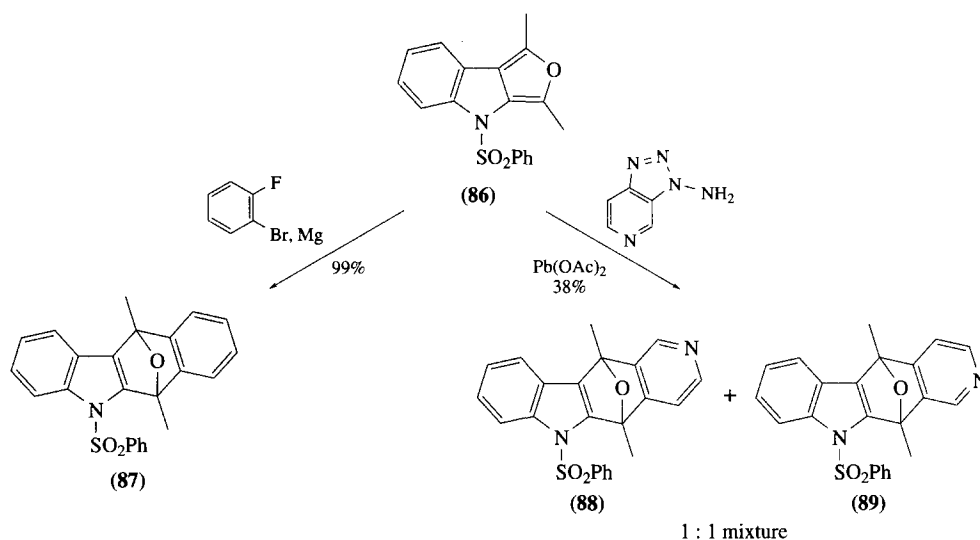
An intramolecular version of the reaction was used to construct the aglycone of the naturally-occurring indole staurosporine (Scheme 154) <92JOC2105>.

A synthesis of the furano[3,4-*b*]indole (**86**) has been developed and it has been shown to give cycloaddition products with benzyne and 3,4-pyridyne (Scheme 155) <84JOC4518>.

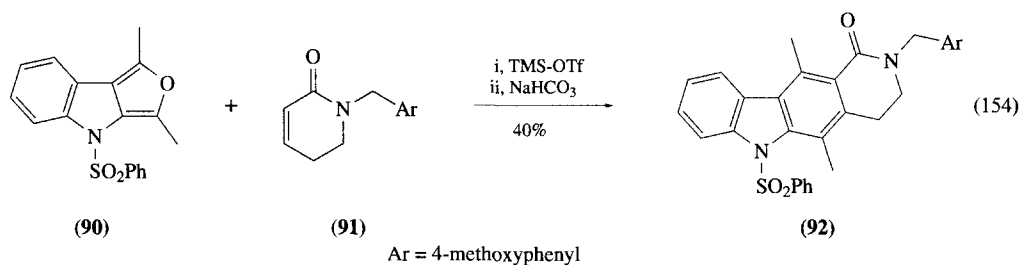
Product (**88**) is an ellipticine precursor, although the lack of regioselectivity detracts from the efficacy of the process. The low regioselectivity is evidently due to the high reactivity of the pyridyne, because reaction of (**86**) with ethyl acrylate is regioselective. The regioselectivity problem has been overcome by use of the piperidone (**91**) as the dienophile (Equation (154)) <92JOC5878>. This reaction presumably proceeds through an *O*-silylated intermediate.



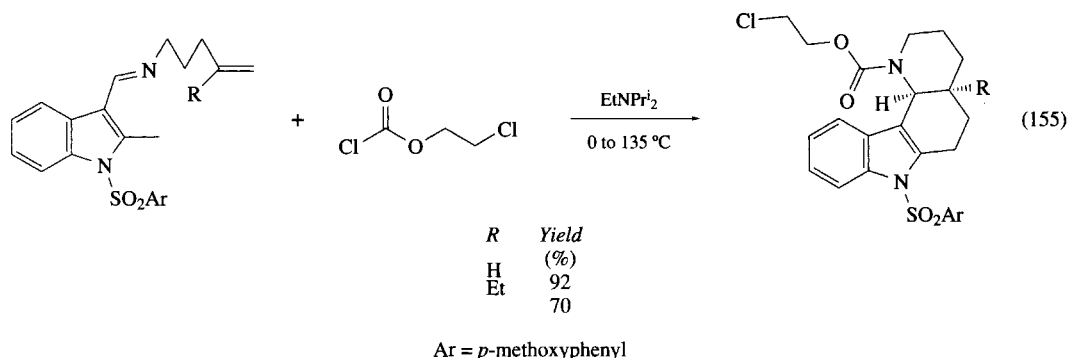
Scheme 154



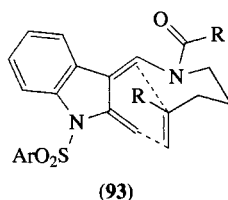
Scheme 155



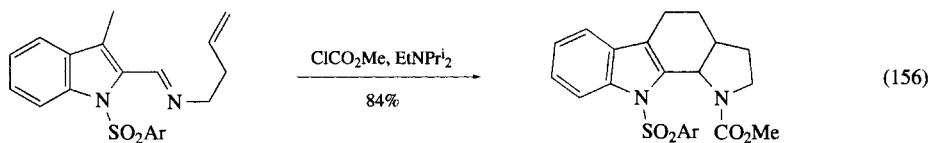
Much of the impetus for synthetic application of indole-2,3-quinodimethane intermediates came from the work by Magnus and co-workers (84ACR35). After developing conditions for generating tetracyclic intermediates as illustrated in Equation (155), the strategy was successfully applied to the synthesis of indole alkaloids including aspidospermine (83JA4739), kopsanone (84JA2105), kopsine (89JA6707), 16-methoxytabersonine (88JA2242), kopsijasmine (88JOC5772), and eburnamonine (86T3215).



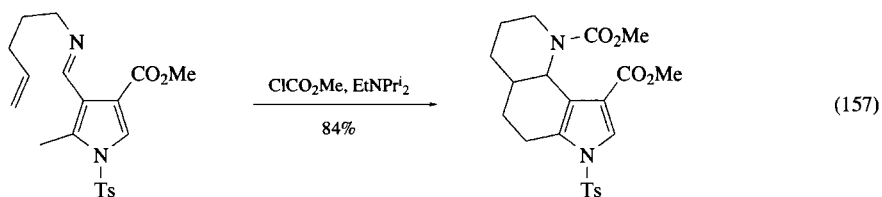
The generation of the quinodimethane under these conditions occurs by *N*-acylation of the imine, followed by deprotonation of the C2 methyl group. The stereochemistry of the cycloaddition is governed by a transition state conformation which favors a *cis cd* ring junction (**93**).



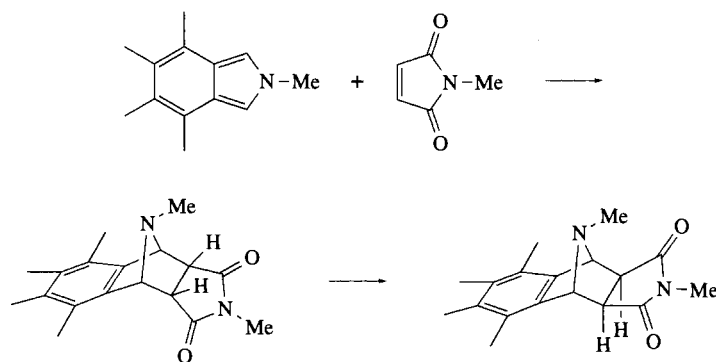
The positions of the formimine and methyl substituents on the ring can be interchanged (Equation (156)) <83T3725>.



The same strategy for generating quinodimethane intermediates has been applied to annelation of 2-methylpyrrole-3-carboxaldehyde derivatives (Equation (157)) <92CC1401>.

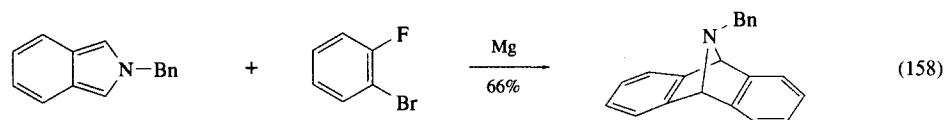


2*H*-Isoindoles are benzenequinodimethanes and react as dienes towards a variety of dienophiles. Maleimide, for instance, gives an *endo* adduct which rearranges easily to the *exo* isomer (Scheme 156) <92CB183>.



Scheme 156

2-Benzyl-2*H*-isoindole reacts with benzyne generated in refluxing THF <88JOC2565>. 2-Methyl, 1,2-dimethyl and 1,2,3-trimethyl-2*H*-isoindole react similarly <79JOC1519>. Adducts have also been obtained from 1,2-naphthyne and 2,3-naphthyne (Equation (158)) <83JOC2364>.



2.04

Pyrroles and their Benzo Derivatives: Applications

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2.04.1 INTRODUCTION

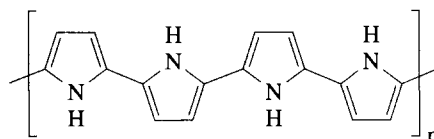
Pyrroles, indoles, and, to a lesser extent, carbazoles comprise an immense group of natural products, medicinal agents, polymers, and other materials. According to *Chemical Abstracts*, during

the period from 1983 to early 1995, there were more than 74000 citations involving applications of these ring systems. Therefore, present coverage is severely limited, and, in some cases, is restricted to literature from the mid-1990s.

2.04.2 POLYMERS

2.04.2.1 Pyrroles

Following the initial synthesis of polypyrrole (**1**) (PPy) <68CR(C)433>, and the discovery of its conducting properties <80SM329>, an enormous amount of work has been devoted to the study of this polymer and its possible applications. A number of reviews are available <86CJC76, B-86MI 204-01, 88CRV183, 89ACR249, 90MI 204-01>.



(1) (PPy)

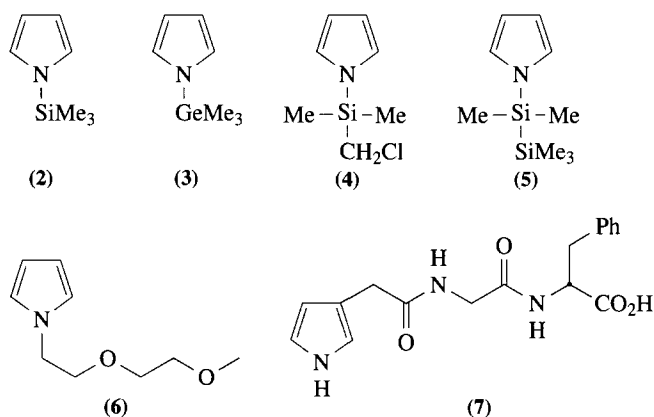
Polypyrrole has been discussed as a possible electrochromic material <83MI 204-01>, and this concept has been realized in the form of a PPy–Prussian blue composite as the oxidatively colored material <92CM1415, 94SM17>. PPy has also been used as a high-density information storage device <85SM255>, a microwave absorber to weld thermoplastic materials <93MI 204-01>, a precoat for the metallization of insulators, such as those on printed circuit boards <93SM3760>, and a material for shielding electromagnetic radiation and for reducing or eliminating electromagnetic interference <93AM(5)281, 94MI 204-01>. In these applications, compared to standard metals, PPy films do not corrode, are flexible, and are lightweight.

The most intensively studied application of PPy has been in the manufacture of capacitor devices <93MCLC61, 94MI 204-02>. For example, a PPy–aluminum solid electrolytic capacitor exhibits good frequency and temperature characteristics and good thermal and moisture stabilities <94SM157>. This capacitor can function continuously for more than 3600 h at 150°C without deterioration. A PPy–Ta₂O₅ capacitor has also been developed <92MI 204-01, 93BCJ2449>. Films of PPy function as cathodes in rechargeable lithium batteries, and are superior to conventional MnO₂ cathodes <91CC986, 93SM1495>. PPy films have been considered as possible electrode material in supercapacitor devices and should have better performance with respect to sustained power <94MI 204-03>. Polypyrrole has been incorporated in a lithographic structured conducting polymer <90MI 204-02>.

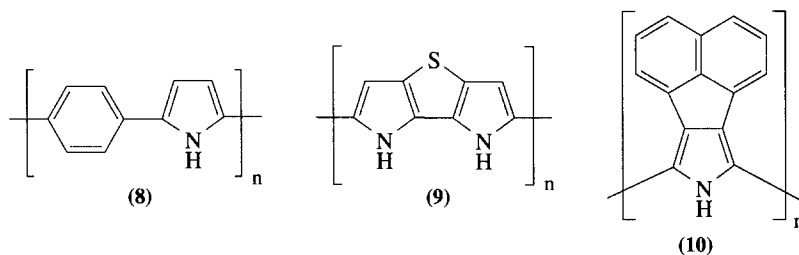
The use of PPy electrodes in chemical sensor and enzyme-based biosensor devices has received extensive evaluation <93SM15>. PPy film composites have been used to detect and measure ammonia <84MI 204-01>, yeast metabolism <94MI 204-04>, glucose levels <86JCS(F1)1259, 86ANC2979>, amino acids <93MI 204-02>, ascorbic acid <91JCS(F1)115>, flavin <92MI 204-02>, proteins <93MI 204-03>, and beer aroma components <94MI 204-05>. Polypyrrole has been used as a switchable membrane to measure the permeation rates of gases from the liquid phase <91SM425, 92AM(4)428>, and as mechanically stable and pinhole-free membranes for the electrochemical control of Na⁺/K⁺ transport across these membranes <94MI 204-06>. Related H⁺-selective electrodes have also been developed based on PPy <94MI 204-07>, and PPy has served as the reducing agent in the reduction of carcinogenic chromium(VI) <93MI 204-04>. Strips of PPy have also been used as electrical muscles in various devices <92AM(4)277, 93MI 204-05>. Composite PPy materials can act as binders and releasers of cationic neuroleptic drugs <92PP106>, and have also been used to bind cationic polypeptides <91SM979>. Polypyrrole latex particles have been used as solid phase supports for immunoassays for the hepatitis B surface antigen, the AIDS antibody, and the pregnancy marker human chorionic gonadotropin <92MI 204-03>. A nonconducting surface coated with polypyrrole serves as a sensor for ammonia and hydrazine <90MI 204-03>, and a PPy-coated platinum electrode acts as a nitrate ion sensor <90MI 204-04>. Silver ion can also be detected using a PPy-coated electrode <90JEC87>. PPy doped with dodecylsulfate ion on a gold surface is an electrode that acts as a humidity sensor <93SM3671>. A promising new composite for both sensor and electrochemical applications is PPy in montmorillonite clays <94MI 204-08>.

Several simple substituted polypyrroles and polypyrrole copolymers have been prepared and studied <83MI 204-02, 94H(37)2069>. For example, poly(3-alkylpyrroles) have been prepared by electrochemical polymerization and found to have conductivities comparable to that of PPy itself <89CC475,

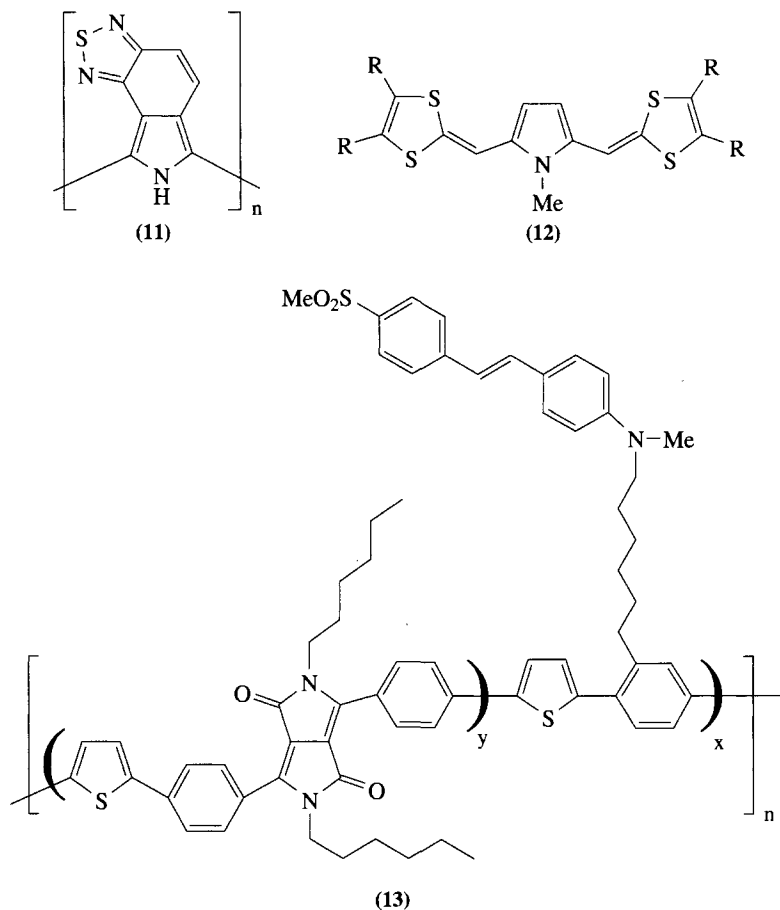
89CC725). Likewise, the properties of a series of electrochemically polymerized 3-acylpyrroles have been reported (93SM1489). The polymerization of 3-ethylpyrrole on gold electrodes modified with pyrrole-containing alkanethiols affords a composite film that is better conducting than those from PPy (95L296, 95L302). Poly(3-methylpyrrole-4-carboxylic acid) silica composites have been used as HPLC stationary phases for protein separations (93JLC1023). Electrodes prepared from poly(*N*-oxaalkylpyrrole) and poly(*N*-butylpyrrole) have been used in solid state rechargeable lithium batteries (93MI 204-06). Novel conducting polymers consisting of bilayers with PPy and poly(*N*-methylpyrrole chloride) and poly(styrenesulfonate) exhibit dual ion transport depending on the potential (93CC258), and can serve as the cathode for a lithium battery (93MI 204-07). Poly(*N*-methylpyrrole) has been used to bind and release dopamine as a function of electrical potential (87MM1594). Novel conducting polymers made by the electrochemical polymerization of pyrroles (2)–(5) have been described (90BCJ1716). These interesting films have conductivity comparable to PPy and have distinguishable colors at different potentials and may find use in multicolor electrochromic devices. As a new, potential molecular recognition polymer, poly(3-crown ether pyrrole) has been prepared (94PP205), and a new lithium-rechargeable battery has been synthesized with the polymer from *N*-(3,6-dioxaheptyl)pyrrole (6) as the electrolyte (91SM1147). Mixed polymers of PPy with 1,3-hexachlorobutadiene and poly(*p*-phenylenevinylene) have been prepared as novel electrochemical diodes and secondary batteries (91SM1495, 92CL2061). A PPy–lead dioxide composite electrode has been prepared for electroanalytical applications (92TAL481). Several polypyrroles, which are functionalized with amino acids and peptides, have been prepared and examined as enzyme recognition materials (94JA8813). An example of such a monomer is (7), the polymer from which interacts with carboxypeptidase. Oxidized PPy also forms complexes with DNA and this binding appears to be linked with the positive charge sites responsible for the observed conduction in the PPy films (92MI 204-04).



A copolymer of pyrrole and *N*-methylpyrrole operates as a redox switching device (91SM241), and novel conducting copolymers have been prepared from pyrrole and *N*-methylpyrrole with *N*-vinylcarbazole (93SM1483). A copolymer of pyrrole and pyrrole-3-carboxylic acid has found application as a glucose biosensor by incorporating glucose oxidase in the film (94MI 204-09). The resulting biosensor expresses higher immobilized enzyme activity than earlier PPy composites based on this enzyme. A “self-doping” polymer of alternating pyrrole and benzoquinone monomers has been fashioned (91SM435), as has been the alternating PPy–benzene polymer (8), which is conducting when doped with iodine or AsF₅ (88CC1432). The electropolymerization of the corresponding monomer has afforded (9) (89SM199), and a series of novel pyrrole-fused polycyclic aromatic hydrocarbons have been synthesized for low band gap polymers, two of which are illustrated ((10)



and (11)) <94CC1019>. Several pyrrole-based extended tetrathiafulvalene systems such as (12) have been designed as potential conducting charge-transfer complexes <92JA5035, 92TL6457>. Novel multifunctional "photorefractive polymers" such as (13) have been devised <93JA11735>.



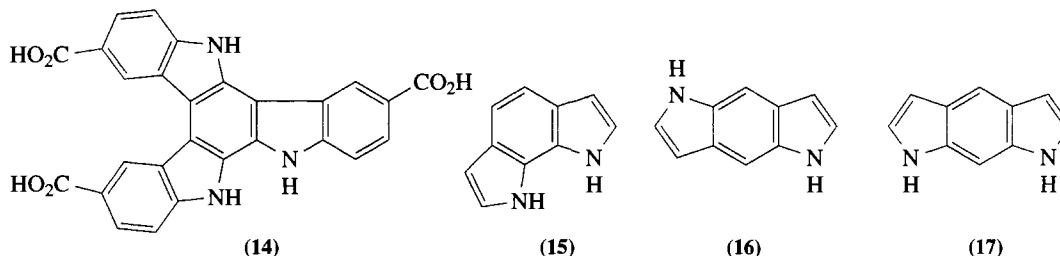
Since the late 1980s several innovative syntheses of polypyrroles have been discovered. The photosensitized polymerization of pyrrole in aqueous solution and in polymer matrices using tris(2,2'-bipyridine)ruthenium(II) as a photosensitizer has been reported <89CC132>, and PPy can be photochemically deposited on to any type of surface under visible light irradiation conditions <89CC657, 90CC387>. The preparation and potential applications of surface-functionalized polypyrrole-silica nanocomposite particles have been discussed <94PP217>.

The unique electronic properties of polypyrroles and the mechanism of their conductivity have been the object of many theoretical studies <94MI 204-10>. The future technical applications for conducting PPy polymers seems bright indeed <94MI 204-11>.

2.04.2.2 Indoles

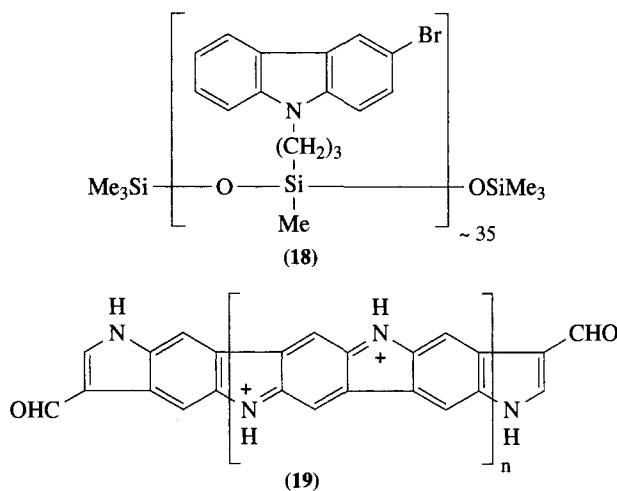
Compared with the extensive investigations of polypyrrole, studies of indole polymers are in their infancy. A series of substituted indoles was electrooxidized and the conductivities of the resulting polymers were measured and compared <84JPC4343>. Electropolymerized films of indole-5-carboxylic acid are stable and show rapid response to changes in solution pH. Therefore, these polymeric films are well suited for the fabrication of micro pH sensors <92MI 204-05>, and have been utilized in a modified glassy carbon electrode to measure ascorbate and NADH levels <93CC1629>. The electropolymerization of indole-5-carboxylic acid leads to the initial formation of trimer (14) as a major product, the structure of which has been established by NMR <94JCS(F1)1121, 94MRC559>. The novel

pyrroloindoles (15)–(17) have been prepared and electrochemically polymerized <91TL5035>. The polymer from (16) produces the most ordered polymer but it has the lowest conductivity <87CC1176, 89MI 204-01>.



2.04.2.3 Carbazoles

The carbazole polymer poly(*N*-vinylcarbazole) functions as a positive electrode material for a secondary lithium battery <85CC553>, and as a memory photoreceptor <91MI 204-01>. The related poly[3-(3-bromocarbazol-9-yl)propyl]methylsiloxane (18) forms novel electrochromic films <89CC196>. Carbazole anions have been used to initiate the polymerization of acrylates and methacrylates <95CC275>. The novel polymeric pyrrolocarbazole (19) displays physical properties similar to those of polyanilines.

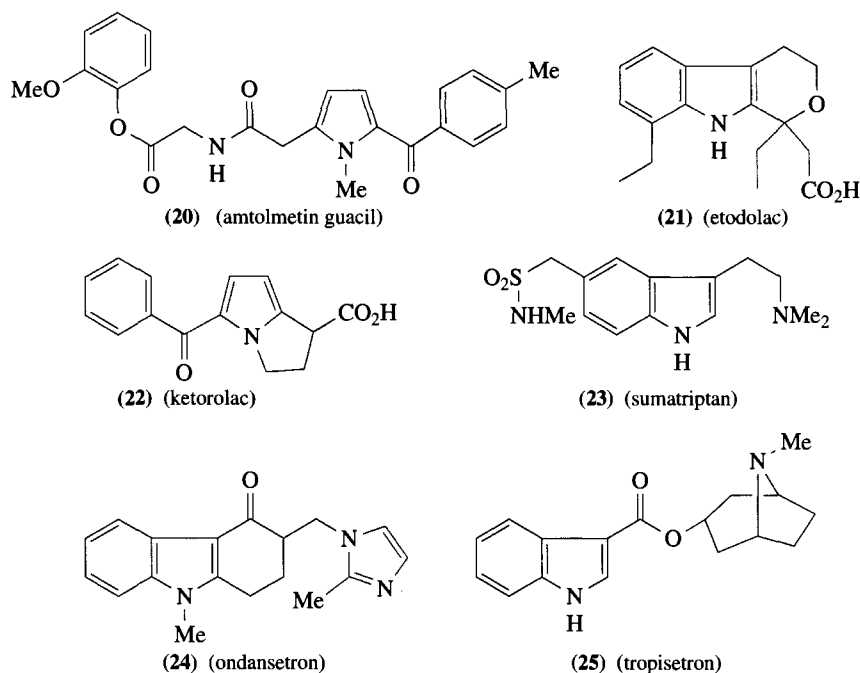


2.04.3 DRUGS

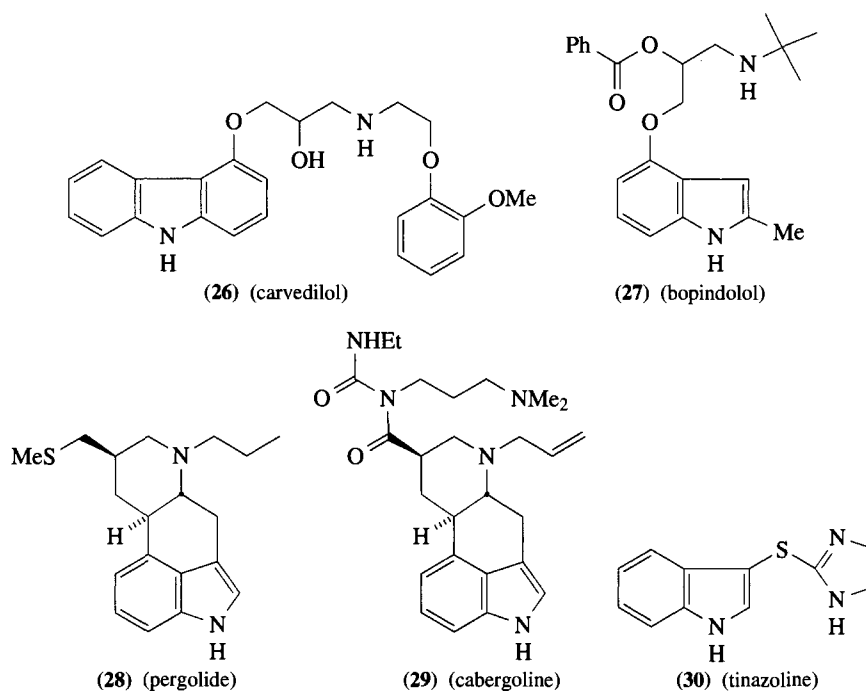
A number of new drugs have been approved for human therapeutic use since the early 1980s that contain pyrrole, indole, carbazole, and related ring systems.

The orally active antiinflammatory drug amtolmetin guacil (20) is used for the treatment of osteoarthritis, rheumatoid arthritis, and postoperative pain <B-94MI 204-12>. Etodolac (21) is also available for these ailments <B-86MI 204-02>. Ketorolac (22) is an antiinflammatory drug with both analgesic and antipyretic activity, with efficacy comparable to morphine for the management of moderate to severe postoperative pain <B-91MI 204-02>. The tryptamine derivative sumatriptan (23) is a highly selective 5-hydroxytryptamine receptor-1 (5-HT_{1D}) agonist and a new drug for the treatment of migraine <B-92MI 204-06>. The first 5-HT₃-receptor selective antagonist for the treatment of nausea and vomiting induced by cancer chemotherapy and radiotherapy to reach the market was

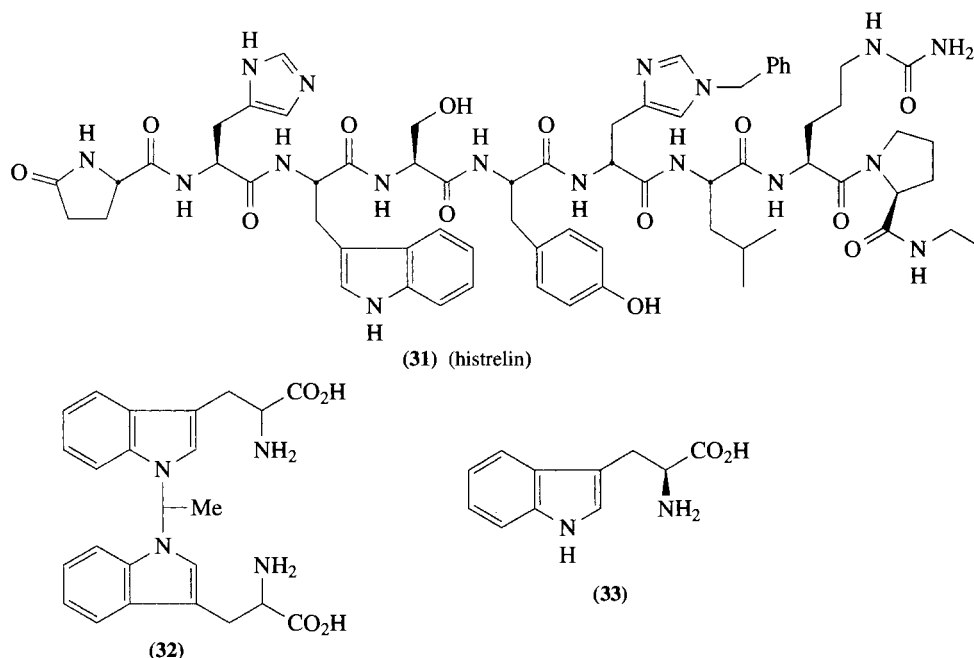
ondansetron (**24**) <B-91MI 204-02>, and a further such drug, also a selective 5-HT₃ antagonist, is tropisetron (**25**) <B-93MI 204-08>.



One of the few carbazole-containing drugs is carvedilol (**26**), a vasodilating beta-blocker useful in the treatment of hypertension and angina pectoris <B-92MI 204-06>. Bopindolol (**27**) is also a potent, long-acting β -adrenergic blocker used to treat hypertension, and related to the well known pindolol <B-86MI 204-02>. Pergolide (**28**) is a potent, long-acting dopamine agonist for Parkinson's disease <B-89MI 204-07>. The dopamine D₂ receptor agonist cabergoline (**29**), which is potent, selective, and long-lasting, is used as a prolactin inhibitor and has undergone clinical trials for Parkinson's disease and breast cancer <B-94MI 204-12>. The vasoconstrictor tinazoline (**30**) is useful as a nasal decongestant <B-89MI 204-07>.



The novel peptide histrelin (**31**) is a gonadotrophin-releasing hormone agonist for the treatment of central precocious puberty and is superior to progestational agents <B-94MI 204-12>. A mysterious and rare disease, eosinophilia myalgia syndrome, which has afflicted more than 1500 people with 27 deaths <90MI 204-05, 90MI 204-06>, has been traced to the contaminant (**32**) in some batches of dietary L-tryptophan (**33**) <91TL991>.



2.04.4 MEDICINAL COMPOUNDS

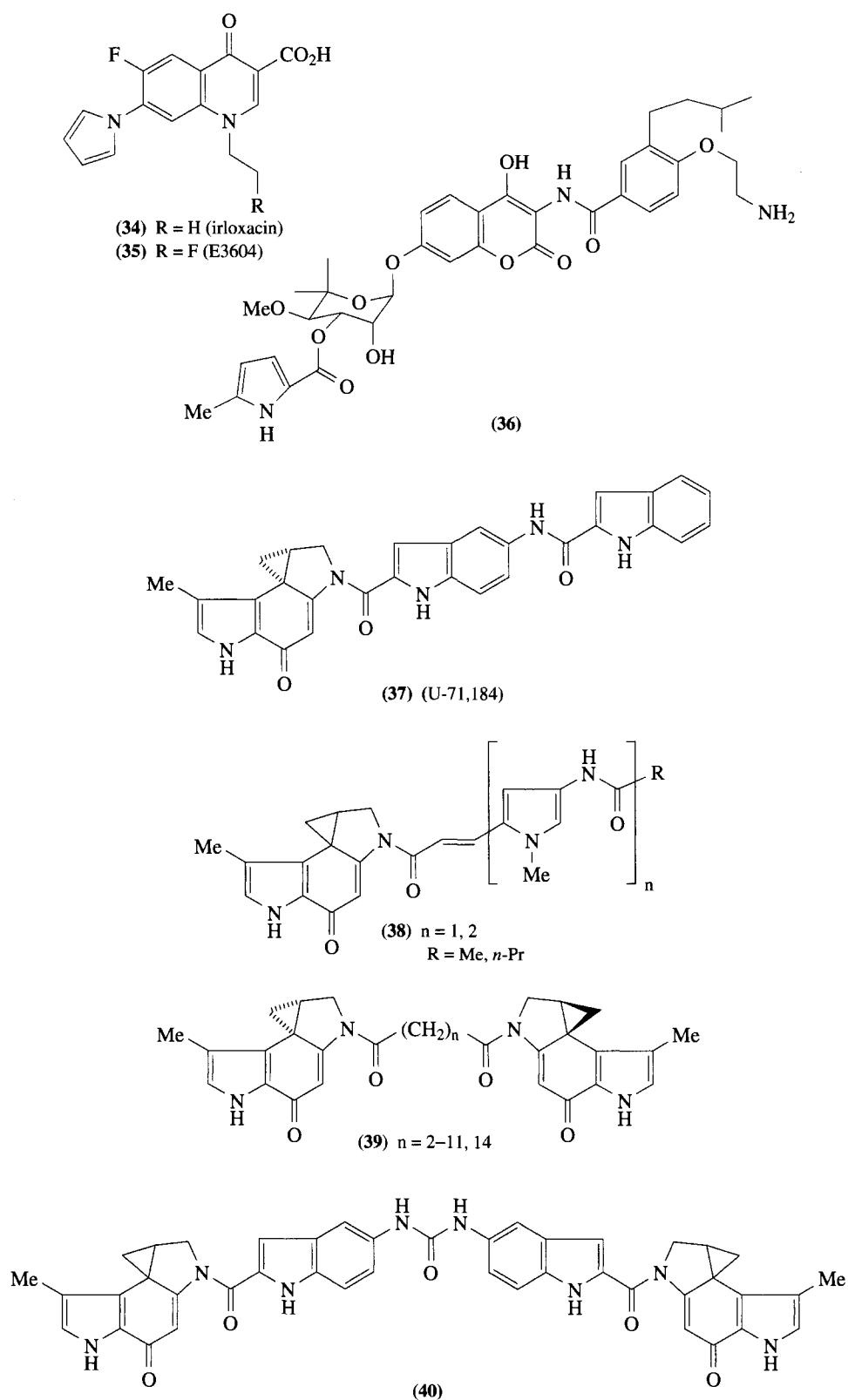
For every newly approved drug, there are dozens more promising candidate drugs undergoing detailed examination and preclinical or clinical evaluation. Even if these candidate compounds never progress to the stage of human treatment, they often provide essential insight into biochemical and pharmacological mechanisms.

2.04.4.1 Antibacterials

Few new promising synthetic antibacterial compounds have been discovered. The fluoro derivative of the pyrrole-containing irloxacin (**34**), is known as E3604 (**35**) <B-88MI 204-01>, and pyrrole (**36**) is an inhibitor of DNA gyrase and is active against methicillin-resistant *Staphylococcus aureus* <B-90MI 204-07>.

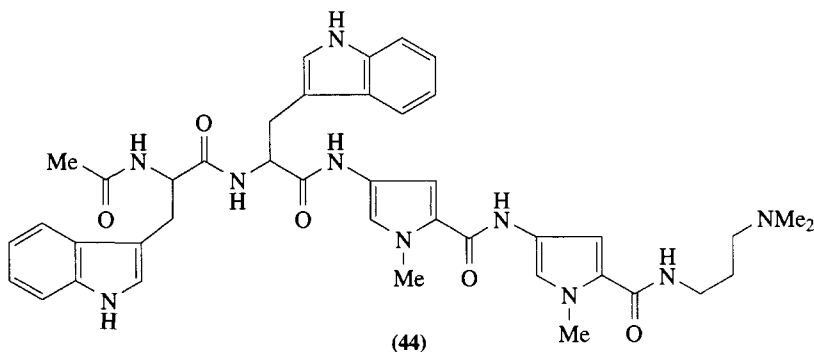
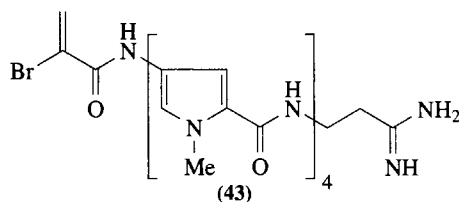
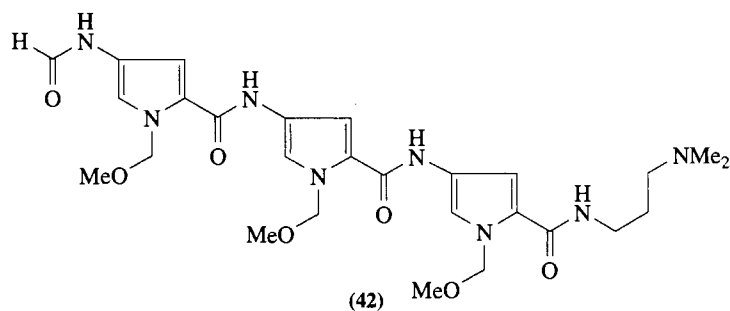
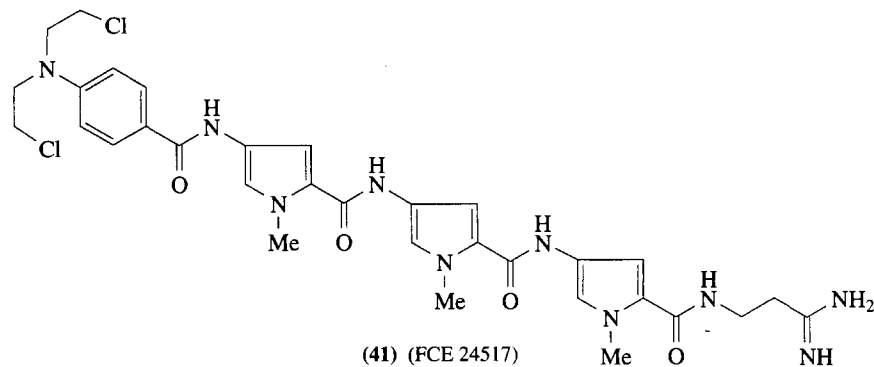
2.04.4.2 Antitumor Agents

The less toxic synthetic CC-1065 analogue, U-71,184 (**37**), is in preclinical development <86TL4103, 86MI 204-03>, and the analogues (**38**) are 1000 times more active against KB cells *in vitro* <95MI 204-01>. For a review of CC-1065, see <86ACR230>. The interesting dimeric compounds (**39**) and (**40**) show strong DNA affinity and cytotoxicity <89JA6428, 91JA8994>. The former compounds display a good correlation of binding with cytotoxicity, with the best compounds having $n = 3$ and 5.



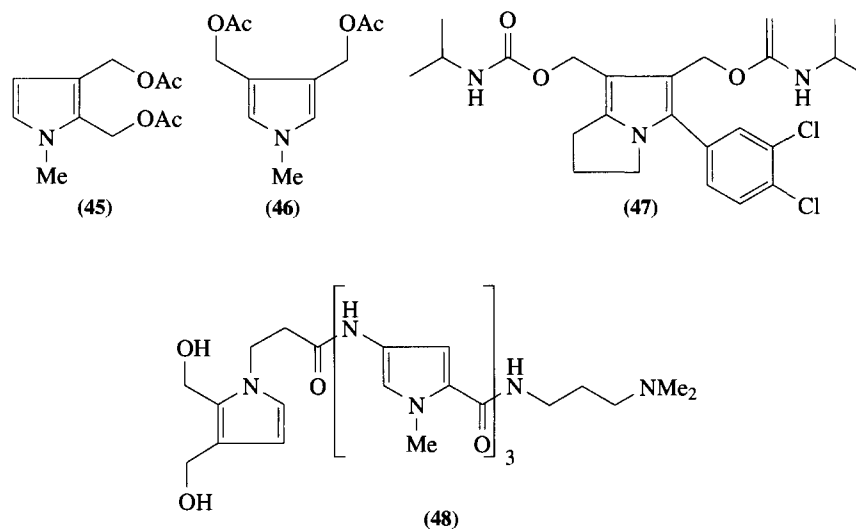
A number of synthetic analogues of distamycin and netropsin have been found to have excellent antitumor activity. For example, FCE 24517 (41), which is a nitrogen mustard–distamycin hybrid, is in clinical trials (B-93MI 204-09). The distamycin derivative (42) has increased water solubility and enhanced cellular uptake (95H(41)337), and (43) is one of several related antitumor compounds with

potent *in vitro* and *in vivo* activity <94BMC1467>. The netropsin tryptophan composite (**44**) shows strong DNA binding <93BMC2623>. Several simple pyrroles (**45**)–(**47**) have been designed and synthesized as novel DNA interstrand cross-linking agents, similar in mechanism to mitomycin <93JA3407>. Pyrrole (**45**) has also been attached to distamycin to produce the DNA cross-linking agent (**48**) <93JA12633>.

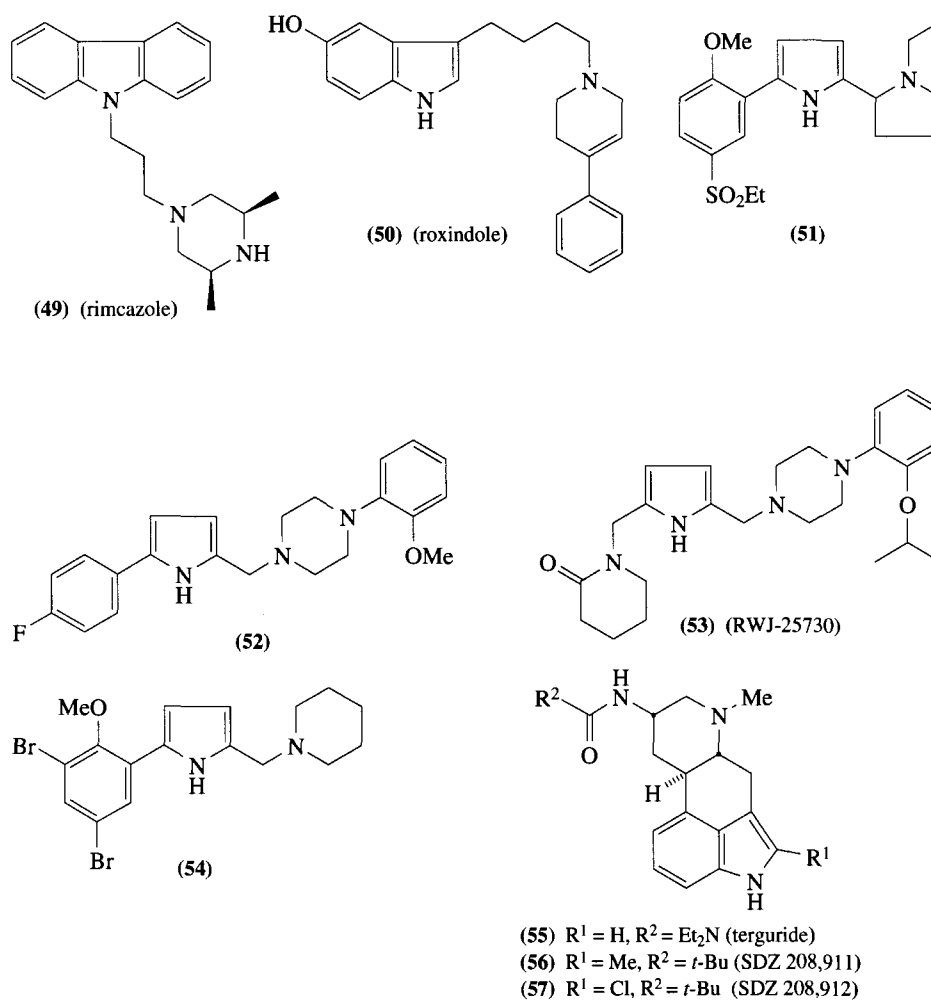


2.04.4.3 Antipsychotic Agents

Rimcazole (**49**) binds to sigma opiate sites in the brain <B-87M1 204-01>, and roxindole (**50**), which is in clinical trials, is a dopamine receptor agonist <92JMC4020>. Pyrrole (**51**) is three times more

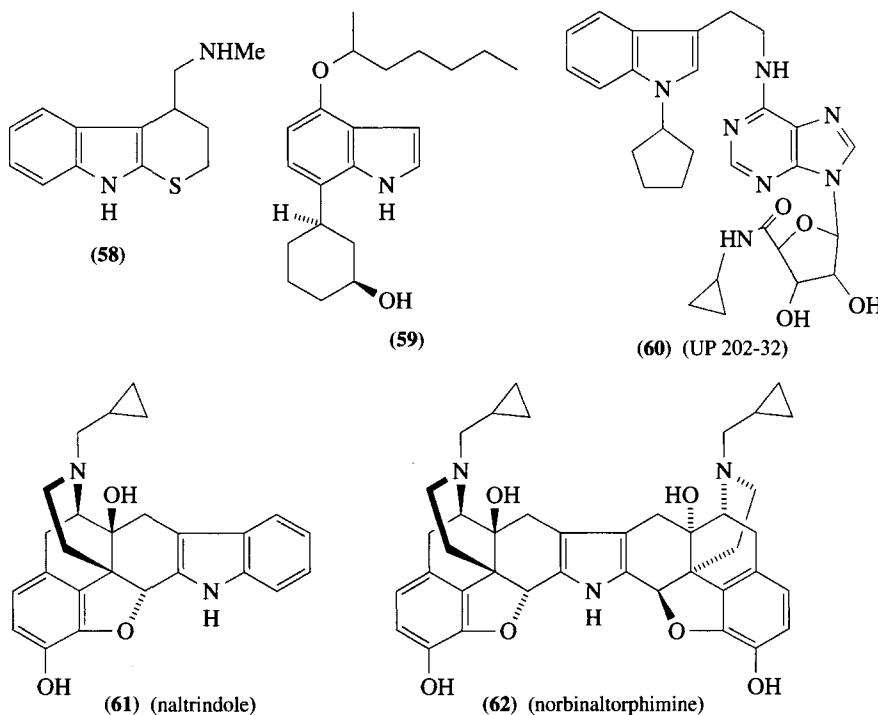


effective than sultopride as an antipsychotic agent, and pyrroles (52)–(54) represent new classes of sodium-independent dopamine receptor antagonists <B-88MI 204-02, B-93MI 204-10, B-94MI 204-13>. Indole derivatives (55)–(57), especially terguride (55) have some activity against schizophrenia <B-92MI 204-07>.



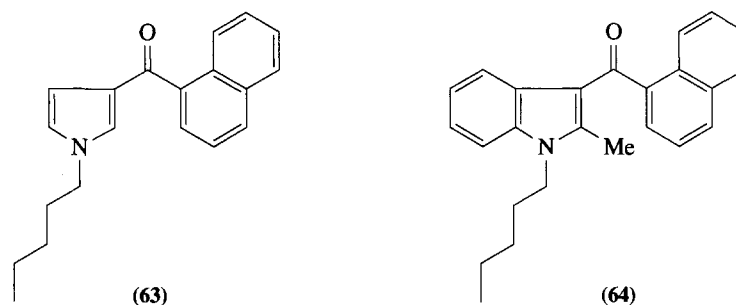
2.04.4.4 Analgesics

The simple indole derivative (**58**) is equipotent with morphine in analgesic tests but is devoid of opioid activity <B-85MI 204-01>. Likewise, (**59**) was prepared as an indole analogue of a known analgesic, although it lacked the desired activity <86JMC1457>. The adenosine receptor agonist UP 202-32 (**60**) is a potent analgesic <94JMC4307>, and several fused indole and pyrrole opioid antagonists such as naltrindole (**61**) and norbinaltorphimine (**62**) have been synthesized <94JMC1495, 94JMC1882>.



2.04.4.5 Cannabinoids

Several pyrrole- and indole-based cannabinoids such as (**63**) and (**64**) are found to exhibit biological activity comparable to Δ^9 -THC <94BMC563, 95TL1401>.

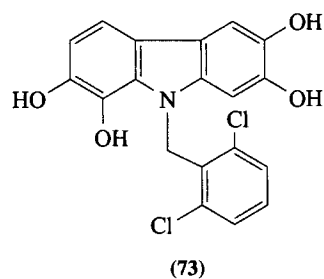
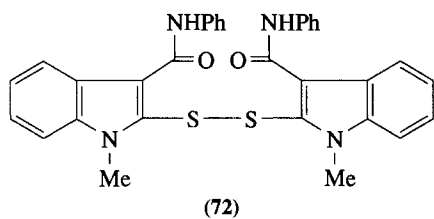
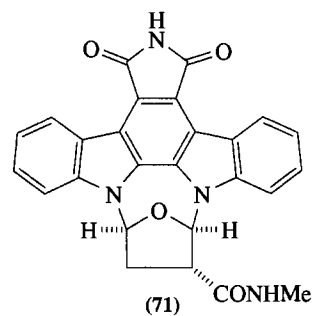
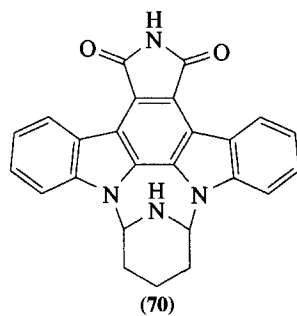
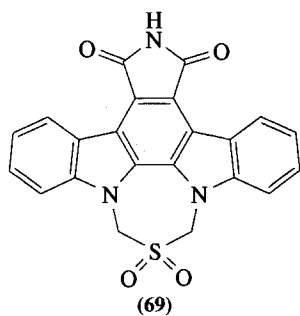
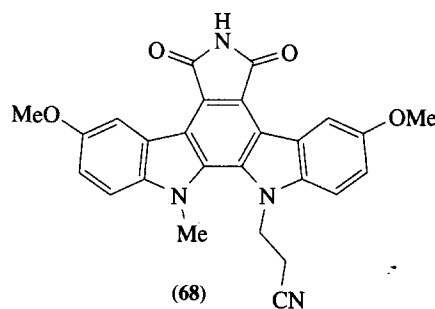
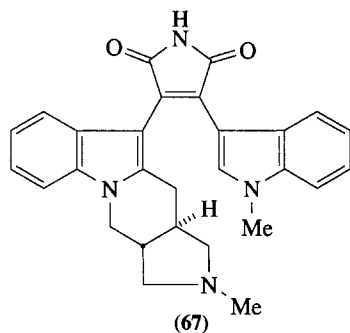
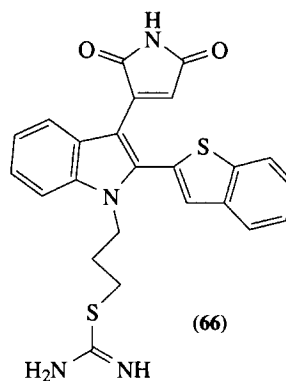
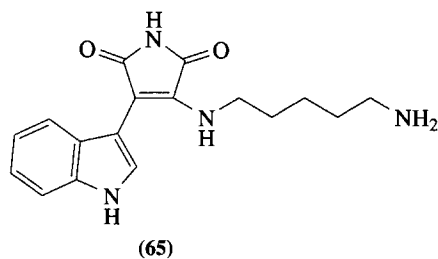


2.04.4.6 Protein Kinase Inhibitors

Several natural and synthetic inhibitors of the family of enzymes known as protein kinases have been discovered, and many of these compounds have antitumor activity. The natural indolo-carbazole alkaloids are discussed separately.

Several indolylmaleimides (**65**)–(**67**) are strong inhibitors of protein kinase C (PKC), but not

of protein kinase A (PKA) <94BMC1303, 94BMC2485, 95BMC67>. Likewise, the synthetic indolo[2,3-*a*]carbazole derivatives (68)–(71) are four of the dozens of potent and selective inhibitors of PKC that have been discovered <93BMC1537, 94BMC1333, 94BMC495, 95BMC55>. The indole disulfide (72) is a potent inhibitor of two types of tyrosine kinases <94JMC2033>, and carbazole (73) is a potent, selective inhibitor of tyrosine kinase <94JMC2224>.

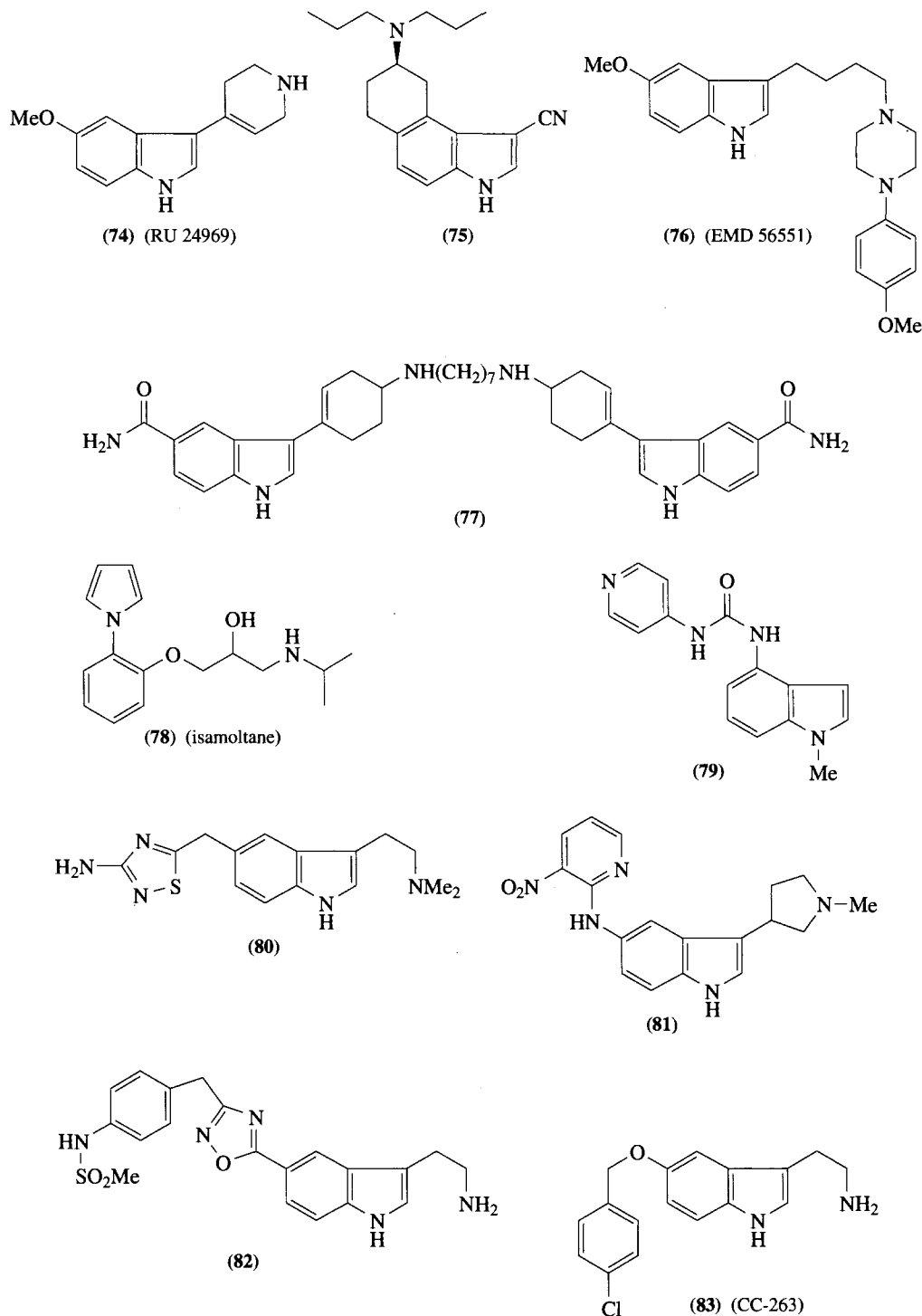


2.04.4.7 Serotonin Uptake Inhibitors

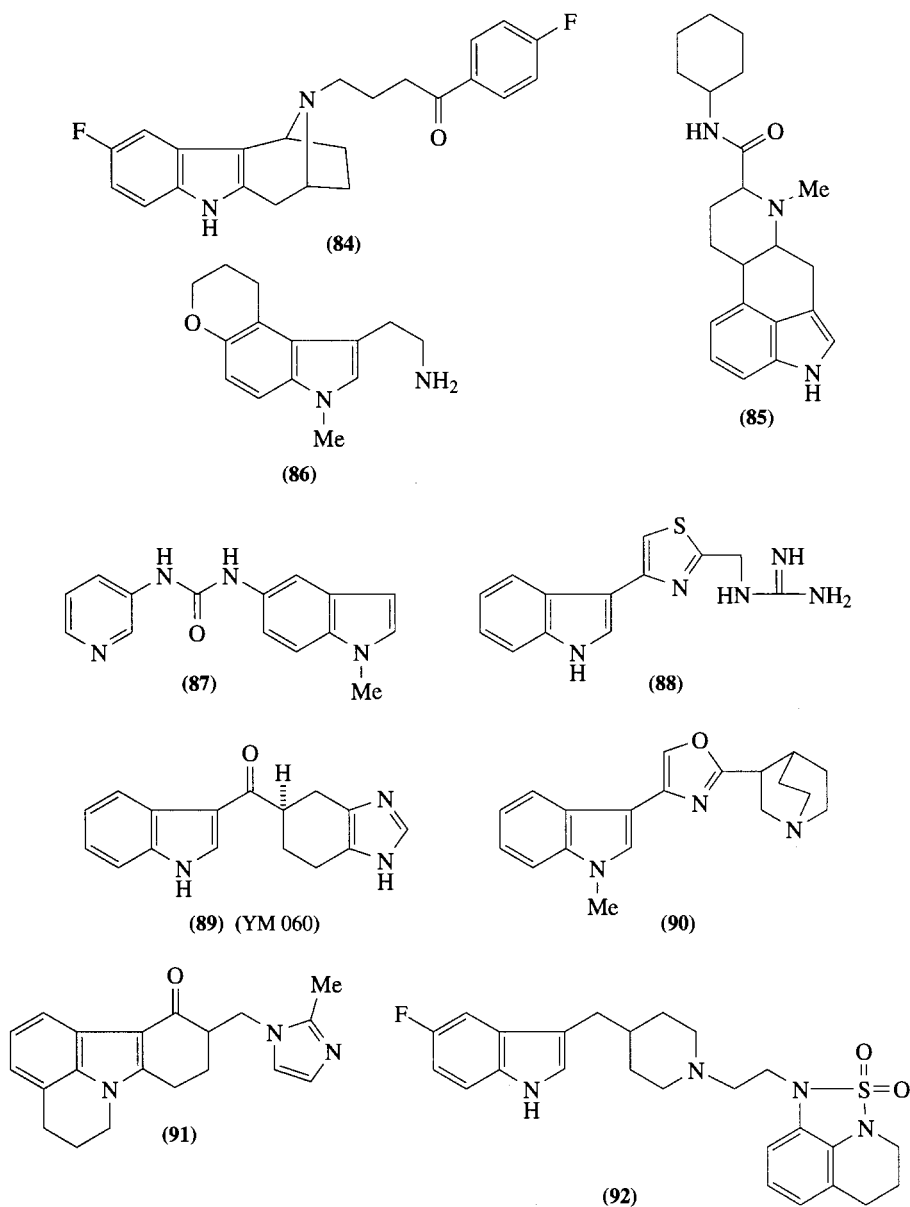
In view of the importance of the neurotransmitter serotonin (5-hydroxytryptamine, 5-HT) in the etiology or treatment of numerous medical problems (anxiety, depression, stroke, migraine,

hypertension, nausea, schizophrenia, and obsessive-compulsive disorders), an enormous effort has been made to develop selective inhibitors of the various 5-HT receptors.

Indoles RU 24969 (**74**), (**75**), EMD 56551 (**76**), and dimer (**77**) are potent agonists of the 5-HT_{1A} receptor <B-87MI 204-02, B-92MI 204-08, 94JMC3263, 95BMC123>. Isamoltane (**78**) is a potent 5-HT_{1B} receptor agonist <B-89MI 204-02>. One of several selective 5-HT_{1C} receptor antagonists to be discovered is (**79**) <93JMC1104>. In addition to (**77**), compounds (**80**)–(**82**) have 5-HT_{1D} receptor affinity <93BMC993, 93JMC1529, 94JMC2509>. Sumatriptane (**23**), which is also a 5-HT_{1D} receptor binder, is a drug for the treatment of migraine <B-92MI 204-08>. Indole (**83**) (CC-263) is 15 times better than sumatriptane (**23**) at binding to the 5-HT_{1D} receptor <B-92MI 204-08>.



Compounds **(84)** and **(85)** bind selectively to the 5-HT₂ receptor <B-92MI 204-08, 93JMC3073>. Indole **(86)** is a 5-HT_{2A} agonist and **(87)** is a 5-HT_{2C} antagonist <B-94MI 204-14>. Several indoles **(88)**–**(90)** have high affinity for the 5-HT₃ receptor <B-92MI 204-03>, and **(91)**, which is more potent than ondansetron **(24)**, is in clinical trials <93JMC3693>. Compound **(92)** is one of several related compounds that is a clinical candidate for depression because of its potent and selective inhibition of serotonin uptake <93JMC1194>.

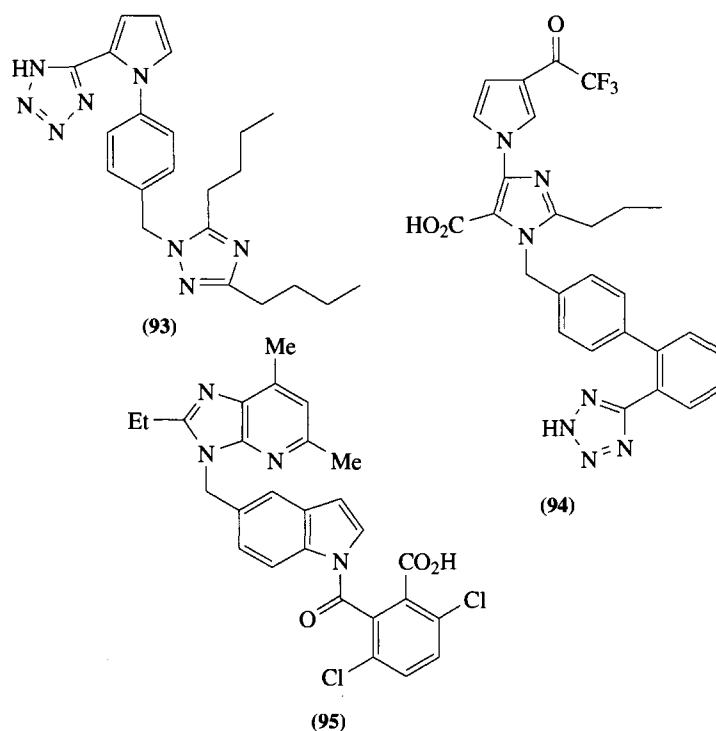


2.04.4.8 Angiotensin Receptor Antagonists

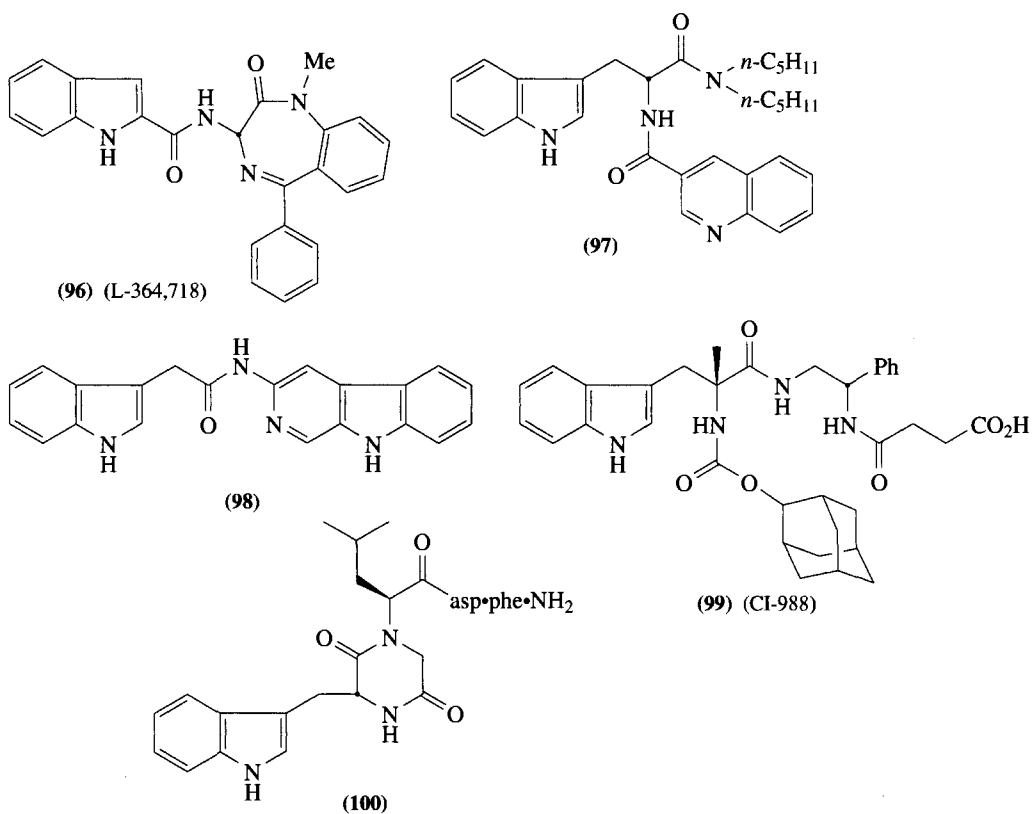
A few new nonpeptide angiotensin II receptor antagonists have been synthesized **(93)**–**(95)** <93JMC101, 93JMC2253, 93JMC4230>.

2.04.4.9 Cholecystokinin Receptor Antagonists

Cholecystokinin (CCK) is an important peptide involved in anxiety. A number of highly selective antagonists for CCK receptors have been discovered. For example, devazepide (L-364,718) **(96)** is

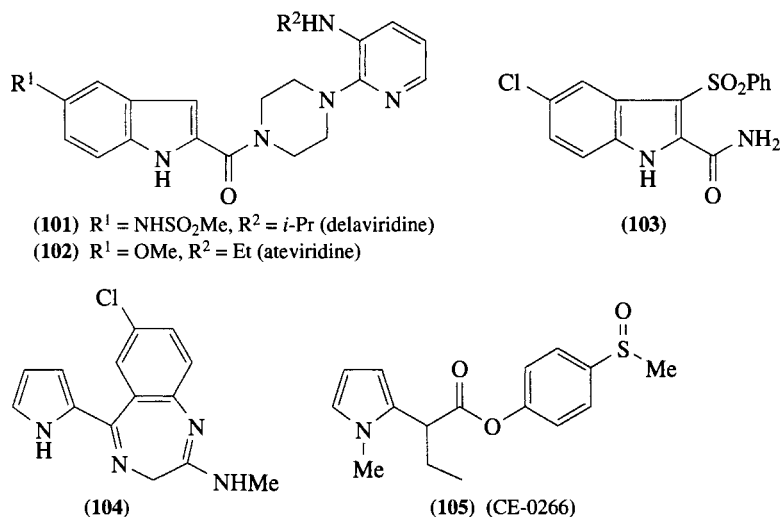


active at the CCK-A receptor, and is comparable in affinity to CCK itself $\langle$ B-88MI 204-03 $\rangle$. Indoles **(97)** and **(98)** are also selective for the CCK-A receptor $\langle$ B-91MI 204-03, 93BMC867 $\rangle$, and antagonists **(99)** (CI-988), **(100)**, and others are selective for the CCK-B receptor $\langle$ B-91MI 204-03, 93BMC2805 $\rangle$.



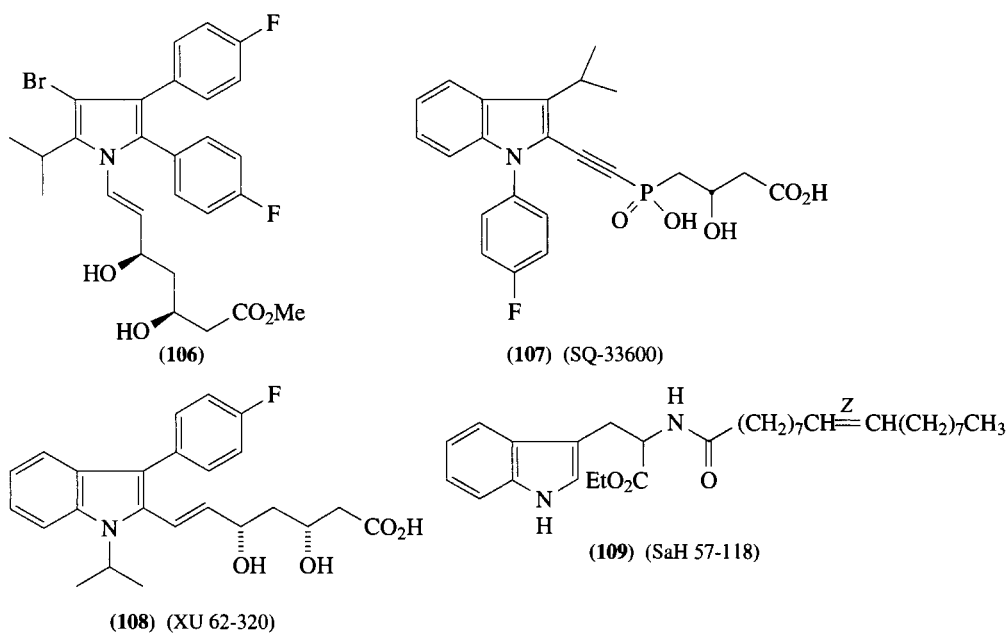
2.04.4.10 Anti-HIV Agents

Delaviridine (**101**) and ateviridine (**102**), which are in clinical trials, are reverse transcriptase inhibitors and prevent the spread of HIV-1 in human lymphocytes <94JMC999, B-94MI 204-15>. The simple indole (**103**) is also an inhibitor of this enzyme <93JMC1291>, while pyrrole (**104**), which is in clinical trials, inhibits the Tat regulator <B-93MI 204-11>, which is a regulatory gene product required for HIV-1 replication and which is encoded by the HIV-1 virus. Pyrrole (**105**) (CE-0266) is a serine proteinase inhibitor <B-93MI 204-12>.



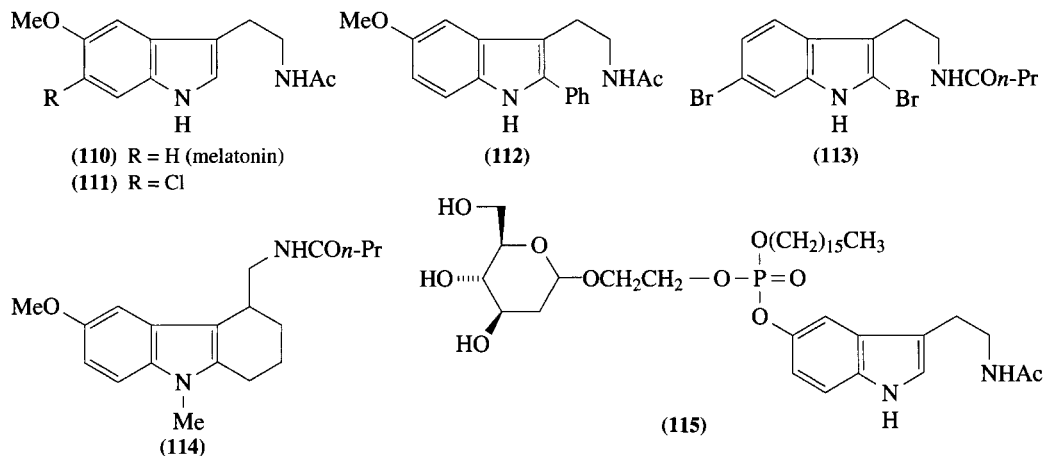
2.04.4.11 Cholesterol Lowering Agents

Several target enzymes in the cholesterol biosynthesis pathway have been targeted in attempts to lower blood cholesterol. The HMG-CoA reductase inhibitor (**106**) is 10 times more potent than lovastatin <93JMC3658>, and both SQ-33600 (**107**) and XU 62-320 (**108**) also inhibit this enzyme <B-89MI 204-03, B-93MI 204-13>. The indole compound SaH 57-118 (**109**) inhibits acyl CoA cholesterol acyltransferase and lowers cholesterol absorption in rabbits by 65% <B-89MI 204-03>.



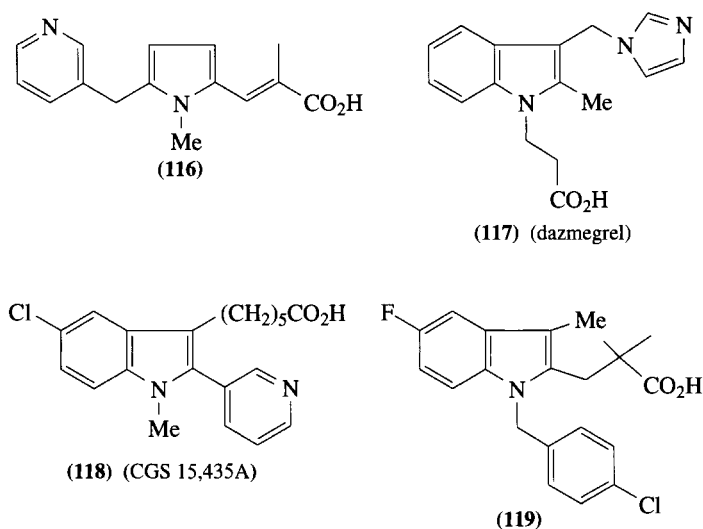
2.04.4.12 Melatonin Receptor Binders

Melatonin (**110**) is a neural hormone that is implicated in many disorders. Several melatonin receptor antagonists and agonists have been synthesized in order to study the pharmacology of melatonin and to map its receptor, including 6-chloromelatonin (**111**) and (**112**)–(**114**) <B-86MI 204-04, 93JMC4069, 94BMC1555, 94BMC1559>. Glycosyl derivatives such as (**115**) have been prepared to effect the brain transport of melatonin <94BMC1485>.



2.04.4.13 Thromboxane Synthase Inhibitors

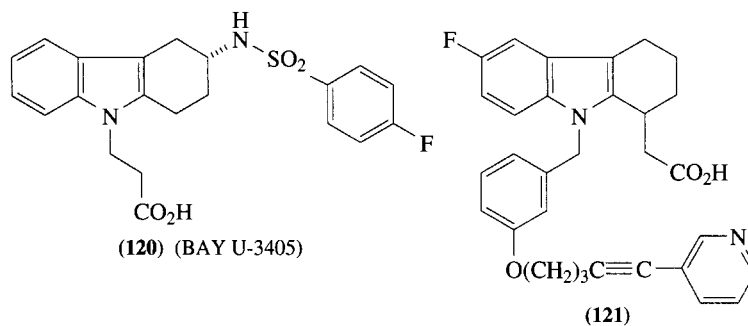
A number of compounds have been found to inhibit thromboxane synthase, an enzyme involved in the synthesis of thromboxane A₂, which is a potent pro-aggregatory and vasoconstrictor substance thought to play a role in vascular homeostasis. For example, (**116**)–(**118**) are inhibitors of this enzyme <B-87MI 204-03, B-90MI 204-08> while (**119**) and (**120**) are thromboxane receptor antagonists <B-90MI 204-08>. The structurally interesting (**121**) is dual acting <92BMC979>.



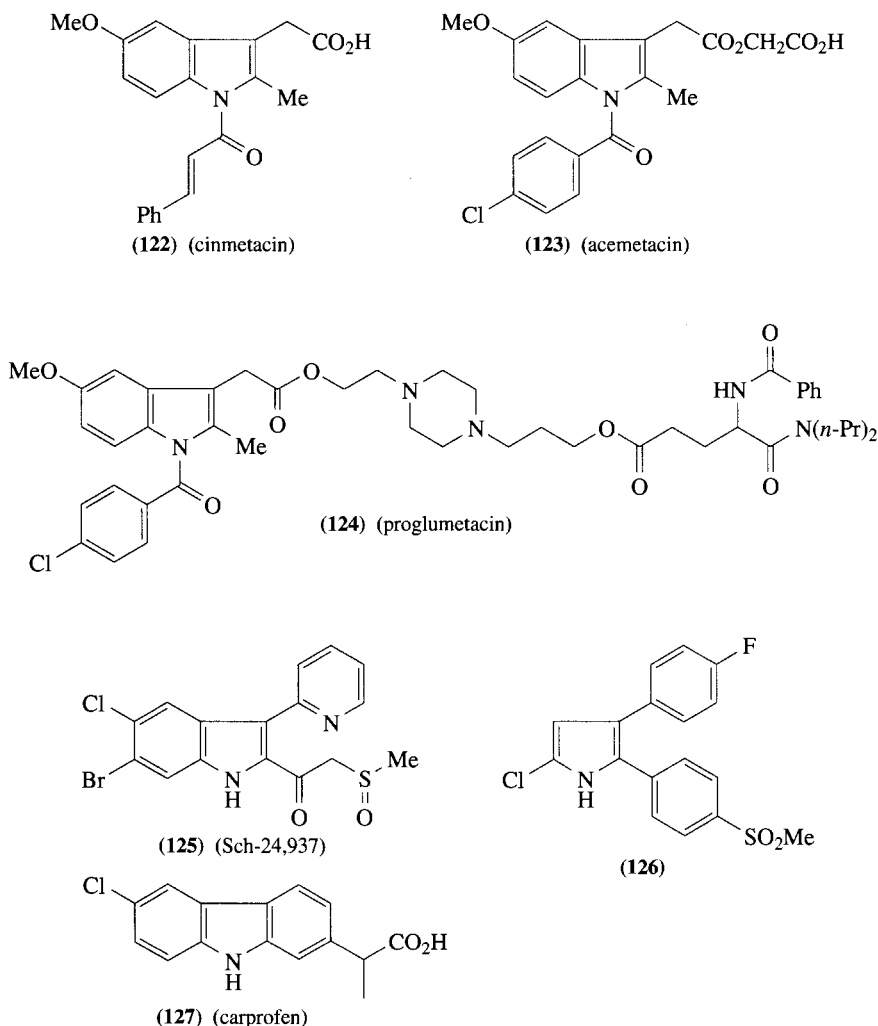
2.04.4.14 Antiinflammatory Agents

Several disease states result from tissue inflammation and the development of new anti-inflammatory agents continues to be an important focus of pharmaceutical companies.

The indomethacin derivative cinmetacin (**122**) has been studied, and the indomethacin prodrugs

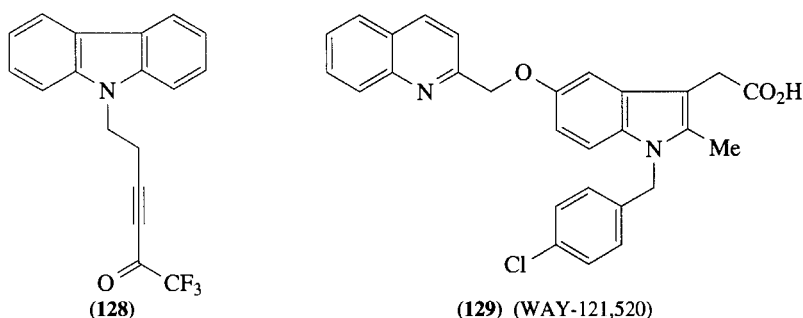


acemetacin (**123**) and proglumetacin (**124**) exhibit fewer gastroduodenal lesions than indomethacin in humans <B-88MI 204-04>. The antiarthritis indole (**125**) suppresses the secondary response in rat adjuvant arthritis <B-86MI 204-05>, and pyrrole (**126**) and carbazole (**127**) also display anti-inflammatory properties <B-88MI 204-04, 94JMC988>.



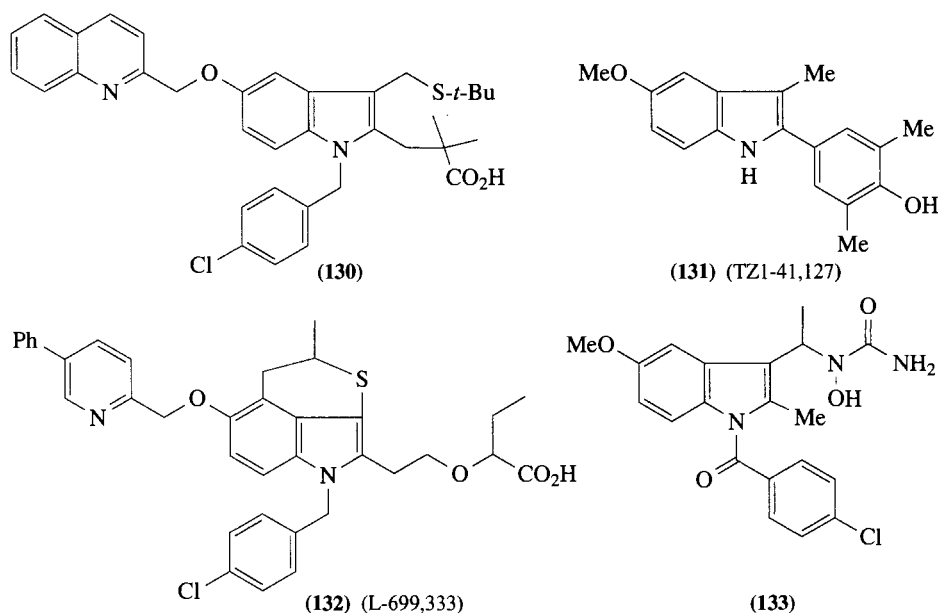
2.04.4.15 Phospholipase Inhibitors

Compounds (**128**) and (**129**) inhibit human phospholipase A and the latter compound displays antiinflammatory activity <B-93MI 204-14>.



2.04.4.16 Lipoygenase Inhibitors

Several compounds, such as (130)–(133), have been discovered to be potent inhibitors of 5-lipoxygenase, an enzyme involved in the synthesis of leukotrienes from arachidonic acid. Of these agents, (133) also inhibits cyclooxygenase <91MI 204-04, 92BMC1655, B-93MI 204-14, 94JMC1153>. Compound (130) (MK 591), which is also a potent inhibitor of leukotriene B₄ (LTB₄) biosynthesis, is currently in clinical evaluation <92BMC1395>.

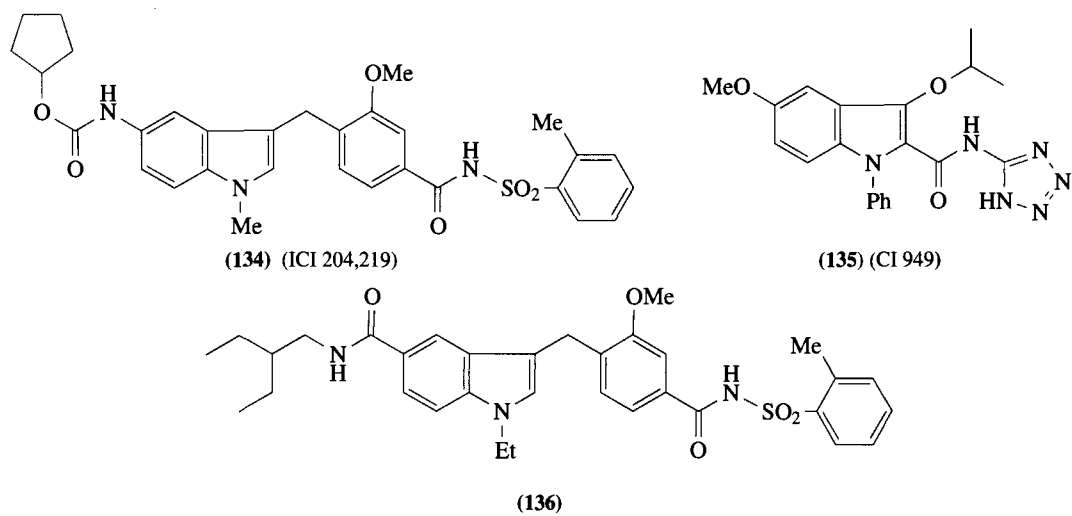


2.04.4.17 Leukotriene Synthesis Inhibitors

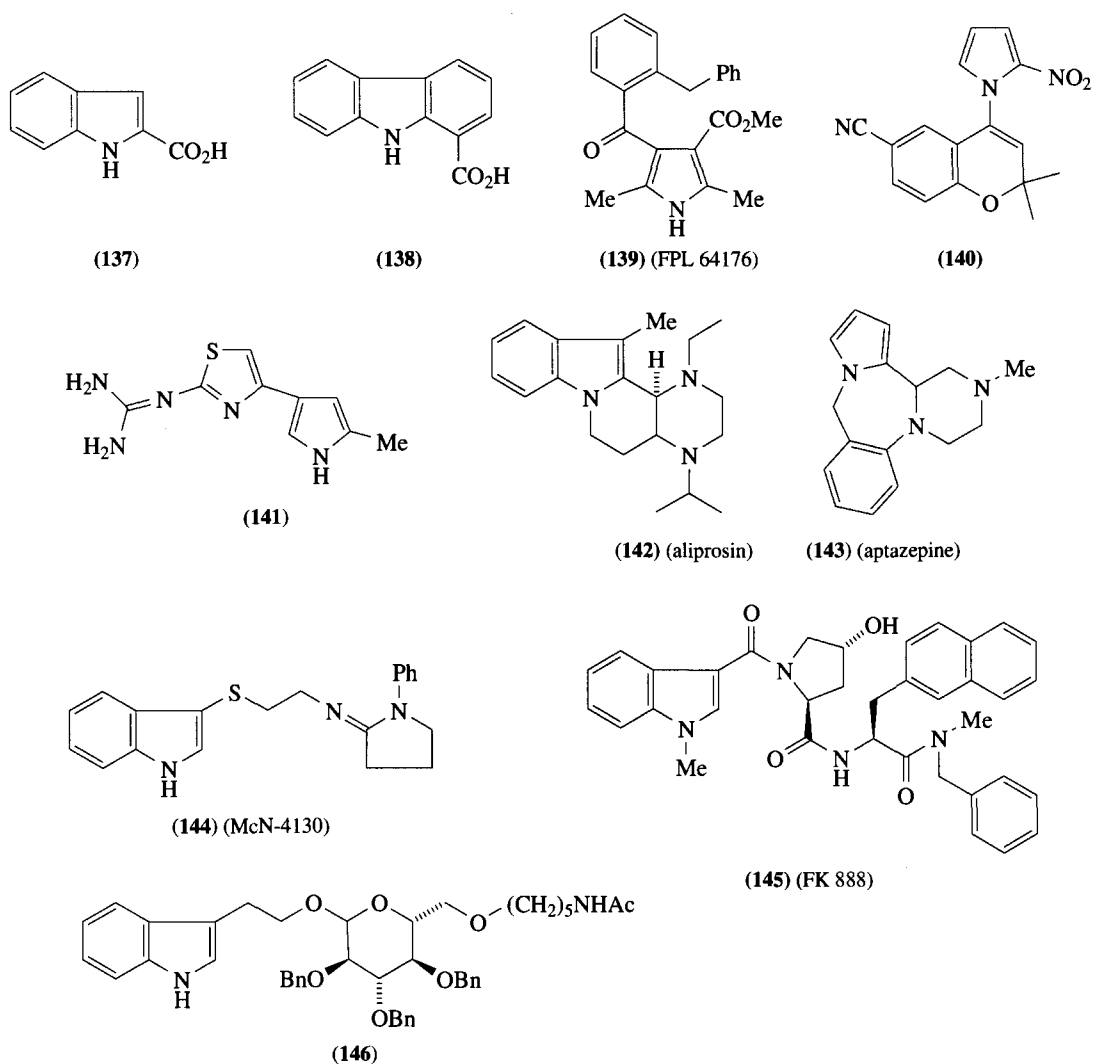
Compound ICI 204,219 (134) is a potent selective antagonist of leukotriene D₄ (LTD₄) and has clinical activity against antigen-induced bronchoconstriction in asthmatic patients, and CI 949 (135) is a mediator release inhibitor which protects against allergen-induced anaphylaxis <B-89MI 204-04>. Compound (136), which is very similar to (134), is a potent and selective antagonist of the peptidoleukotrienes <93JMC394>.

2.04.4.18 Miscellaneous Medicinal Compounds

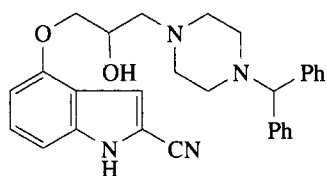
The simple 2-indolecarboxylic acid (137) and 1-carbazolecarboxylic acid (138) inhibit parathyroid hormone stimulated bone resorption <B-87MI 204-04>. A new class of calcium ion channel activators is FPL 64176 (139) <93JMC2739>, and nitropyrrole (140) is related to the potassium ion channel activator cromakalin and has smooth muscle relaxant properties <92BMC1595>. Pyrrole (141) is a reversible potassium ion competitive inhibitor of the gastric H⁺/K⁺-ATPase <B-90MI 204-09>. Ali-



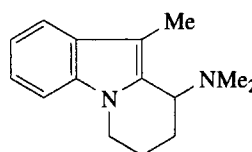
prosin (142) and aptazepine (143) are two antagonists of the α -2-adrenoreceptor <B-88MI 204-05, B-89MI 204-05>. Class I antiarrhythmic properties have been discovered in McN-4130 (144) <B-86MI 204-06>, and two tachykinin antagonists are (145) (FK 888) and (146) <B-93MI 204-15>.



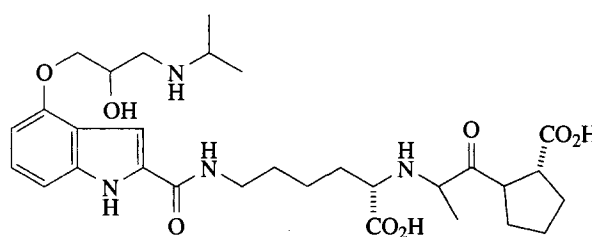
Indole DPI 201,106 (**147**) has positive inotropic and vasodilator activity, is bradycardic and prolongs the action potential <B-87MI 204-05>, and (**148**) is a cerebrovasodilator <B-88MI 204-06>. BW A575C (**149**) is an angiotensin converting enzyme inhibitor- β -blocker <B-89MI 204-06>. Several tryptophan derivatives like (**150**) are inhibitors of metalloproteinases <B-90MI 204-10> and (**151**) is a nonpeptide mimic of somatostatin <92BMC135>. Indole amide derivatives of type (**152**) are inverse agonists at the benzodiazepine receptor <92JMC2214>.



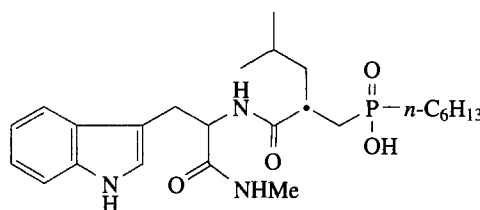
(147) (DPI 201, 106)



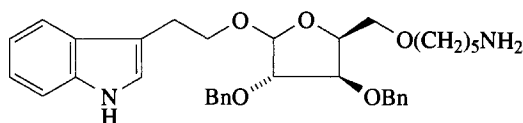
(148)



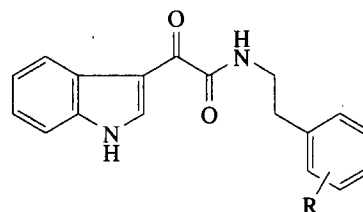
(149) (BW A575C)



(150)



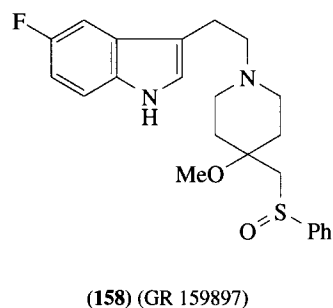
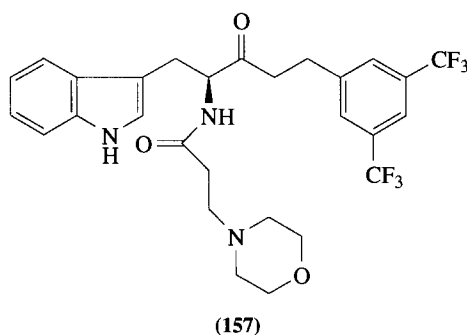
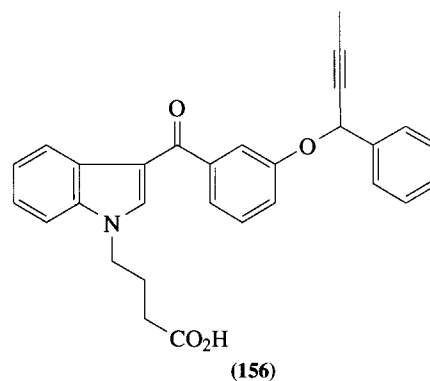
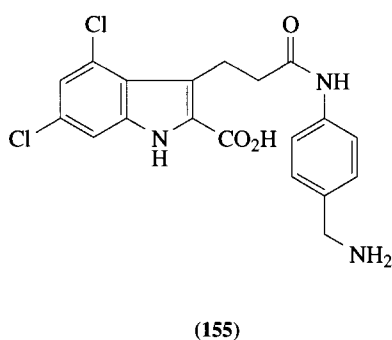
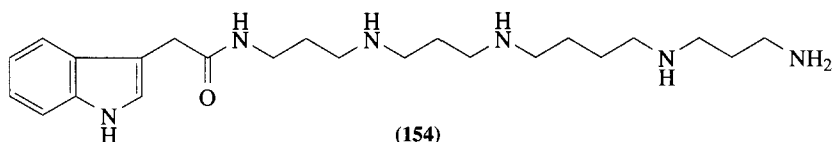
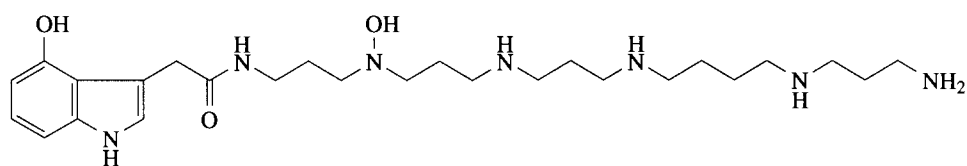
(151)



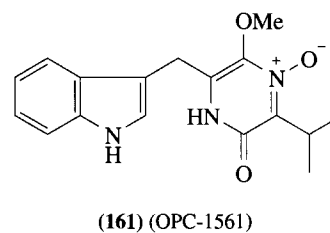
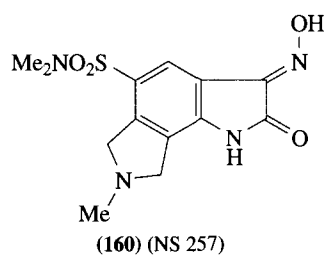
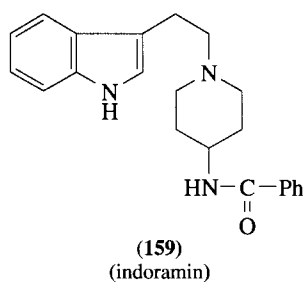
(152)

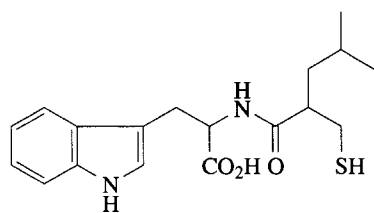
Indole funnel web spider metabolites, such as (**153**), are potent NMDA antagonists <B-91MI 204-05>, and the related synthetic (**154**) is a polyamine antagonist <B-94MI 204-16>. Several antagonists at the glycine site of the NMDA receptor have been discovered, such as (**155**) and related indole-2-carboxylic acids <92BMC1627, B-94MI 204-16>. Indole derivative (**156**) is an inhibitor of 5α -reductase <B-94MI 204-17>, and the newly discovered (**157**) is a substance P receptor antagonist <95MI 204-02>. Substance P is a peptide neurotransmitter implicated in the transmission of pain and in the initiation of inflammatory responses. The drug candidate GR 159897 (**158**) is a highly potent, orally active nonpeptide neurokinin NK_2 receptor antagonist <94BMC1951>.

Several novel compounds have been discovered to have potential utility in the treatment of the effects of ischemia following stroke. For example, indoramin (**159**) increases the rate of ATP synthesis in the brain and may be useful in treating ischemia <B-86MI 204-07>. Isatin oxime NS257 (**160**), which is an AMPA receptor antagonist, is a candidate for the treatment of stroke <94BMC371>. The generation of superoxide anion, which is associated with injury to the central nervous system,

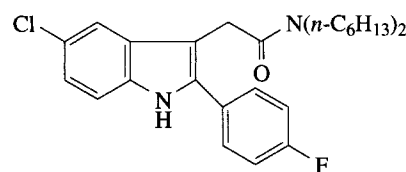


is inhibited by OPC-1561 (**161**) <91JAN53>. A new inhibitor of endothelin converting enzyme (ECE) has been uncovered in indole (**162**) <93BMC1953>. The production of neurosteroids is linked to the mitochondrial DBI receptor complex, and several new ligands have been discovered for this complex, including indole (**163**) <93JMC2908>. Biindolyl (**164**), which is prepared from indigo, is a potent inhibitor of ATP binding to DNA A protein <94BMC1771>. This protein is involved in DNA replication. The non-peptide neurotensin (NT) mimetic (**165**) binds strongly to NT receptors <94BMC1241>.

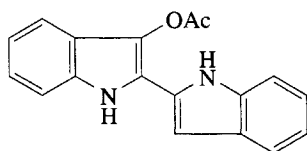




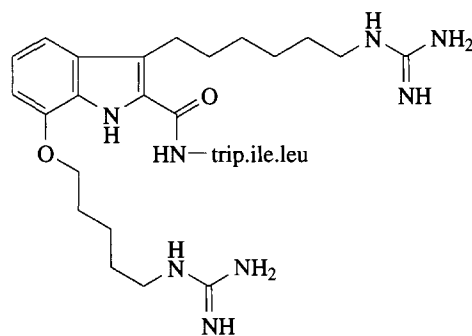
(162)



(163)



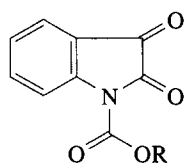
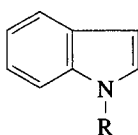
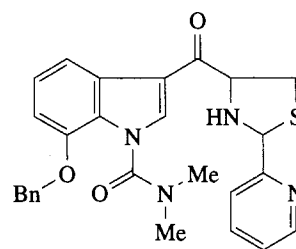
(164)



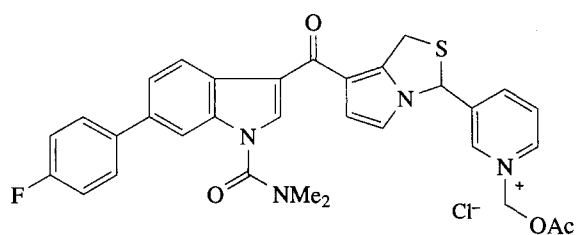
(165)

The simple *N*-carboalkoxyisatins (**166**) are inhibitors of chymotrypsin $\langle 95\text{BMC}89 \rangle$, and *N*-alkylindoles (**167**), which form stable vesicles, serve as models for studying transmembrane proteins and, in particular, the role of tryptophan in the insertion and anchoring of membrane-spanning proteins $\langle 95\text{JA}1265 \rangle$. Several compounds, (**168**)–(**170**), are potent platelet activating factor antagonists $\langle \text{B-93MI 204-16, 94JMC2011, 94JMC4423} \rangle$, and ABT-299 (**169**) is actually the prodrug ($-\text{AcOCH}_2\text{Cl}$) of an agent that is in clinical trials for the treatment of sepsis. This compound is one of the most potent antagonists of platelet activating factor yet described. The nitropyrrole sugar (**171**) has been designed and synthesized to function as a universal replacement for any of the natural nucleosides in DNA $\langle 95\text{JA}1201 \rangle$, and the novel tryptophan phosphoric acid analogue (**172**) has been described $\langle 92\text{SL}708 \rangle$.

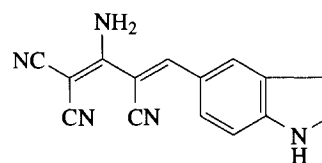
The novel biindolyl (**173**) and others have been constructed as possible urea binders $\langle 93\text{JA}12601 \rangle$,

(166)
(R = CH_2Ph , *t*-Bu)(167) (R = Me, *n*- C_6H_{13} ,
n- $\text{C}_{10}\text{H}_{21}$, *n*- $\text{C}_{18}\text{H}_{37}$)

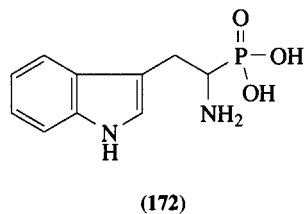
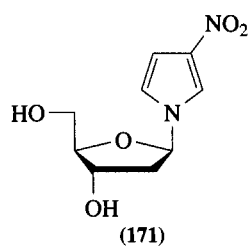
(168)



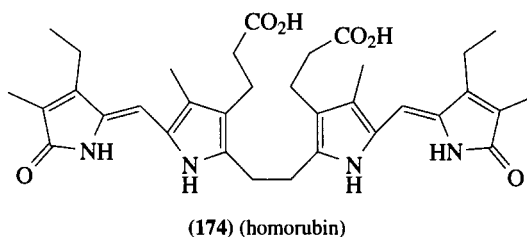
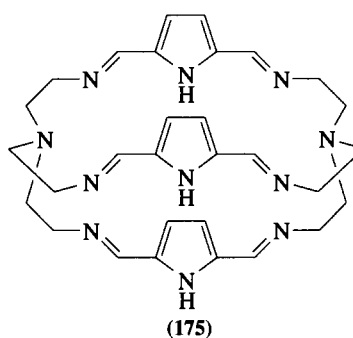
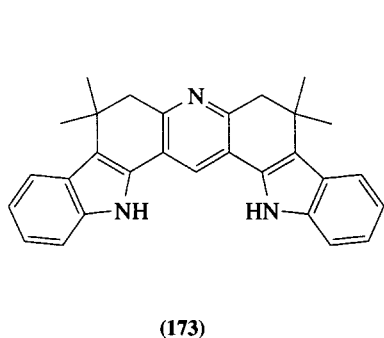
(169) (ABT-299)



(170) (AG-370)

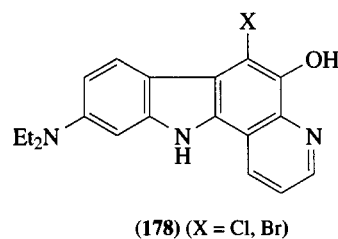
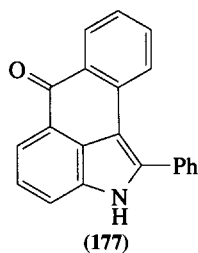
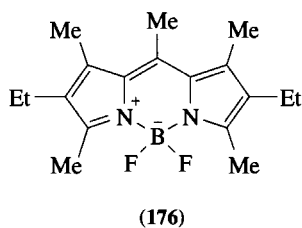


and “homorubin” (174) has been synthesized for study and comparison with natural bilirubin and related tetrapyrroles <94TL9673>. Likewise, the pyrrole-based cryptand L (175) has been designed to act as a dicopper ligand <94CC649>.



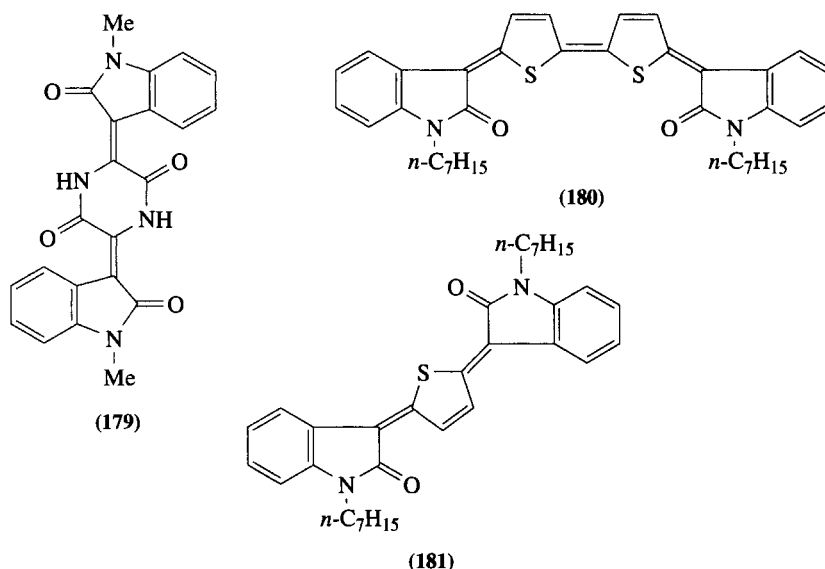
2.04.5 DYES

Several pyrroles, indoles, and carbazoles have found use both as laser dyes and textile dyes. For example, a number of pyrromethene-BF₂ complexes such as (176) show broadband laser activity in the region 530–580 nm under flashlamp excitation <90HAC389>. Indoloanthraquinone (177) is an efficient laser dye in the 500–550 nm region <91BCJ3417>. Pyridocarbazole (178) is a new visible or near-IR color-former, depending on oxidation state and metal chelate complexation <92CC1114>.

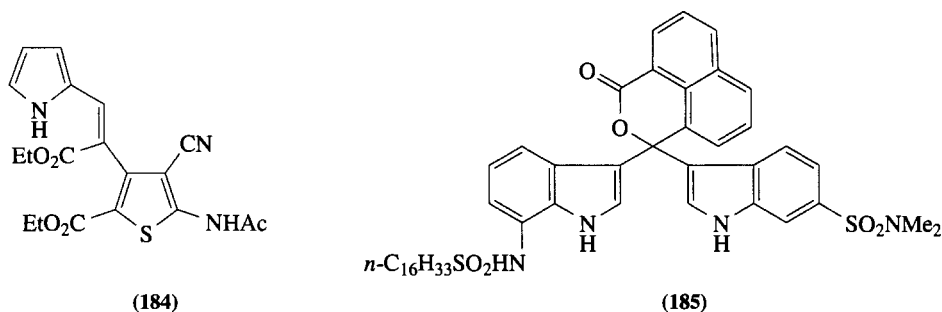
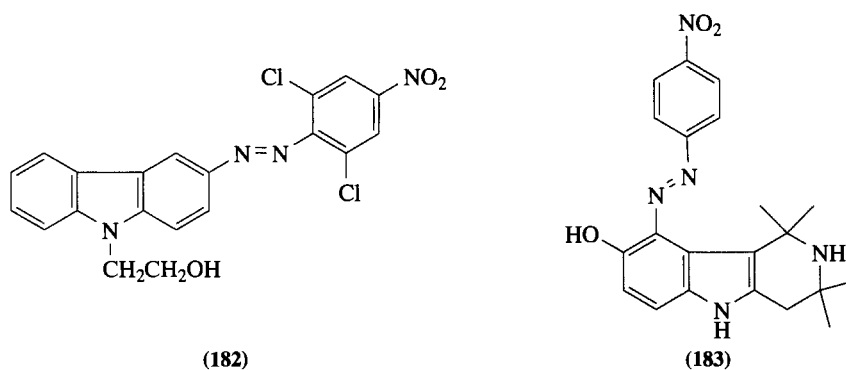


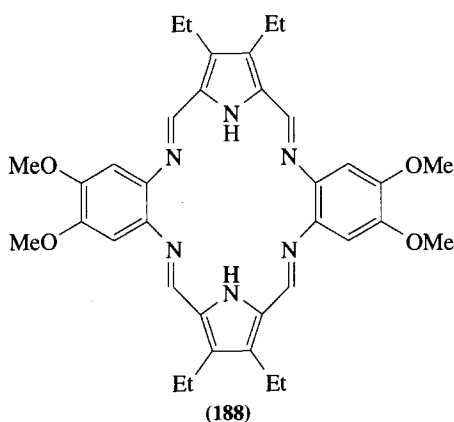
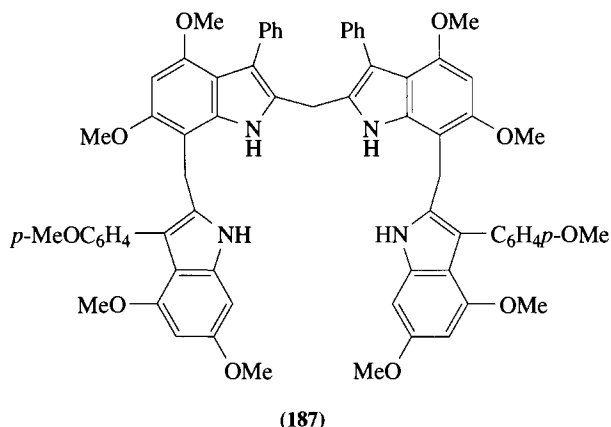
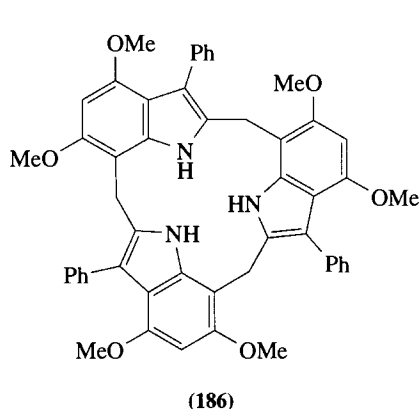
Interest in indigo chemistry remains strong. Thus, the novel indigoid *para*-quinodimethanes, such as (179), are known <92AG(E)1202>. The historically famous indophenine dye of von Baeyer has been reinvestigated and the product from *N*-heptylisatin is found to be a mixture of several geometric

isomers, one of which is (180). The simpler compound (181), formed from only one unit of thiophene, was also reported for the first time <93JA11512>.



The carbazole azo dye (182) is used for dyeing polyester fabrics <94CA(120)32867>, and the related γ -carboline (183) represents a new class of dyes that is very light stable in a nylon-6 matrix <91DP83>. The thiophene pyrrole derivative (184) is a novel polyester dye <93MI 204-17>. Diindole (185) is a new dye for instant photography <91JOC4576>. In connection with possible use as light-fast dyes for wool, a series of calix[3]indoles and indole oligomers have been synthesized as novel metal ligands <93CC819, 93SL246>. Examples include (186) and (187). In this regard, the novel pyrrole complex (188) has been designed and synthesized to complex uranyl ion <92IC529>.





2.04.6 AGROCHEMICALS

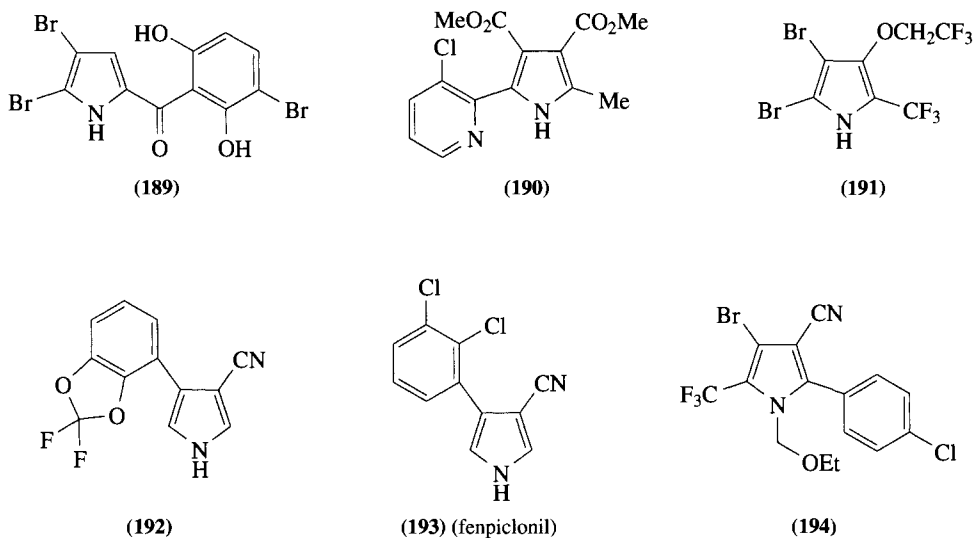
Surprisingly, there are relatively few examples of agrochemicals or other pesticides that incorporate pyrrole, indole, or carbazole units. However, a few examples of insecticidal and herbicidal pyrrole compounds are known. Indeed, discovery of the potent antibacterial activity of the naturally produced pyoluteorin has led to several synthetic analogues of potential commercial importance. Bromopyrrole (**189**) proved to have the greatest herbicidal activity of 20 related pyrroles <90MI 204-11>, and pyrrole (**190**) has both pre- and postemergence herbicidal activity <90MI 204-12>. Dibromopyrrole (**191**) shows strong herbicidal efficacy <94MI 204-18>, and cyanopyrroles (**192**) and (**193**) (fenciclonil) are important new fungicides for cereal seed treatment <B-91MI 204-06>. Pyrrole (**194**) is in commercial development as a broad spectrum insecticide and miticide <B-91MI 204-07, 94MI 204-19>.

2.04.7 NATURAL PRODUCTS

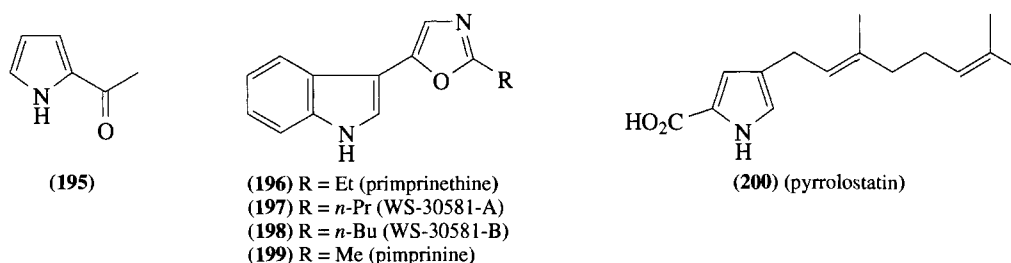
It is impossible to cover in this short chapter the myriad natural products discovered between 1983 and 1995 that contain a pyrrole, indole, or carbazole ring. Therefore, coverage is limited to the literature since 1990 and highlights from 1983 to 1990.

2.04.7.1 Bacterial Products

A number of extraordinarily complex and biologically potent metabolites have been discovered in bacteria. Despite the existence of an estimated three million species of bacteria, only a small percentage have thus far been examined for their chemical content. A variety of structurally fascinating and biologically important compounds have been isolated from *Streptomyces* sp. For



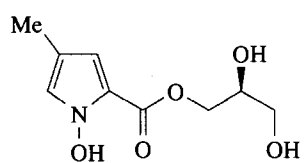
example, a *Streptomyces* sp. produces the simple 2-acetylpyrrole (**195**), a compound with hepatoprotective properties <91ABC2117>. Pimprinethine (**196**) is produced by several microorganisms, such as *S. cinnamomeus* <82JAN549>, and the related WS-30581-A (**197**) and -B (**198**) are metabolites of *Streptoverticillium waksmanii* <84JAN1153>. All three are similar to pimprinine (**199**). Pyrrolostatin (**200**), which inhibits lipid peroxidation in the brain, is produced by *Streptomyces chrestomyceticus* <93JAN892>, and glycerinopyrin (**201**) has been identified from cultures of *S. violaceus* <91LA77>. Another *Streptomyces* sp. has yielded the antitumor BE-18591 (**202**) <93JAN1799>. The interesting diolmycins A1 (**203**) and A2 (**204**) are products of *Streptomyces* sp. <93JAN762>, and hydroxytryptophan is present in aborycin, a tricyclic 21-peptide from *S. griseoflavus* <94LA741>. Chromoxy-mycin (**205**) is a novel pyrrole metabolite found in cultures of *S. rubropurpureus* <85TL3273>.



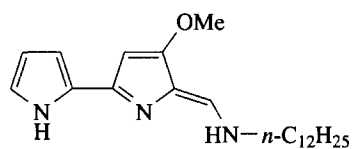
Not unlike the potent antitumor compound CC-1065, several structurally and mechanistically related compounds, the duocarmycins, such as (**206**), are produced by *Streptomyces* sp. These metabolites display exceptionally potent cytotoxicity activity <91JAN1045>. Since the late 1980s, a series of carbazole derivatives have been found that are potent free radical scavengers. Thus, *S. chromofuscus* produces carazostatin (**207**) <89JAN1879>, and *Streptomyces* sp. has afforded neo-carazostatins A–C (**208**)–(**210**) <91JAN903>, all of which are antioxidants and inhibit lipid peroxidation. Epocarbozolin A (**211**) and B (**212**) are produced by *S. anulatus* <93JAN25>, and a series of aminocarbazoles, such as antiostatin A₁ (**213**), is found in cultures of *S. cyaneus* <90JAN1337>. Carquinostatin A (**214**) is a potent neuronal cell protecting substance and radical scavenger from *S. exfoliatus* <93TL4943>.

A number of related carbazoles are secreted by *Streptoverticillium ehimense*. For example, at least eight carbazomycins (e.g., (**215**) and (**216**)) have been isolated from cultures of this microbe <87JAN157, 88JAN602>. Cytoblastin (**217**), which is related to indolactam V and the teleocidins, is produced by *S. eurocidicum* <91MI 204-08>, and *S. blastmyceticum* has provided blastmycetin F (**218**) and the strong tumor promoter (–)-7-geranylindolactam V (**219**) <94MI 204-20>. The similar pendolmycins (**220**) and (**221**) are produced by *Nocardioopsis* sp., and (**221**) inhibits phorbol ester binding to protein kinase C <88MI 204-07, 91MI 204-09>.

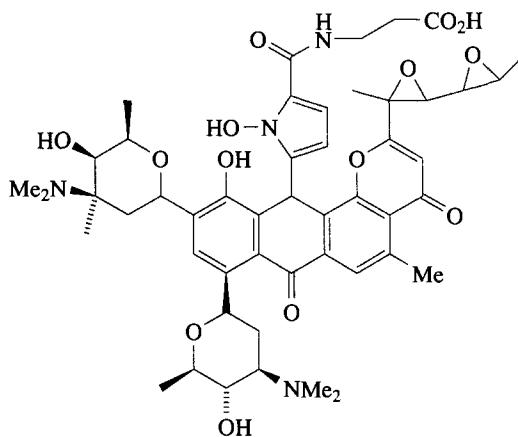
The interesting sulfur-containing indoles (**222**) and (**223**) have been isolated from the hyper-



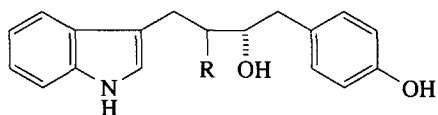
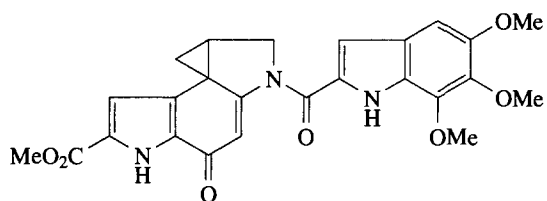
(201) glycerinopyrin



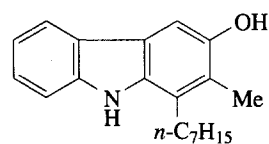
(202) (BE-18591)



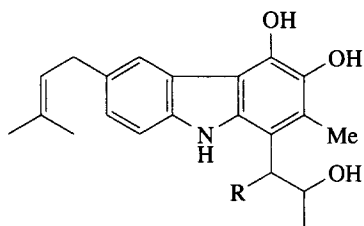
(205) (chromoxymycin)

(203) R = α OH (diolmycin A1)(204) R = β OH (diolmycin A2)

(206) (duocarmycin SA)



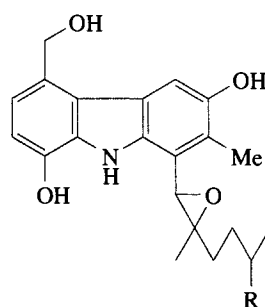
(207) (carazostatin)



(208) R = OH (neocarazostatin A)

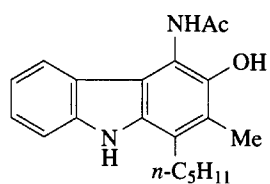
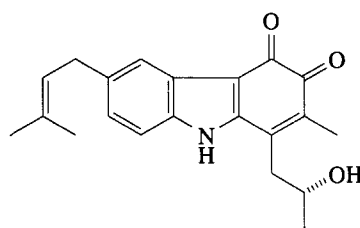
(209) R = H (neocarazostatin B)

(210) R = OMe (neocarazostatin C)

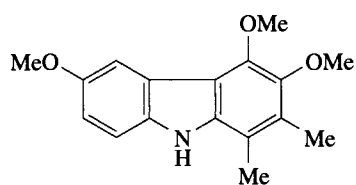


(211) R = Me (epicarbozolin A)

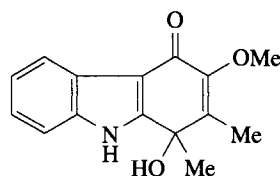
(212) R = Et (epicarbozolin B)

(213) (antiostatins A₁)

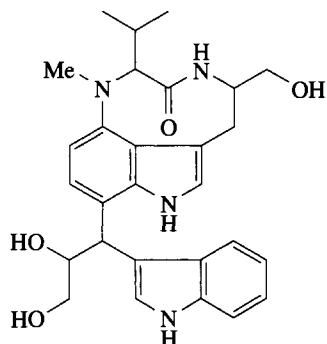
(214) (carquinostatins A)



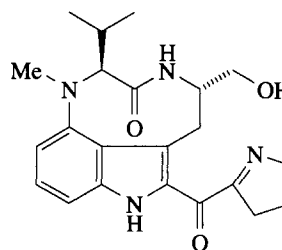
(215) (carbazomycin D)



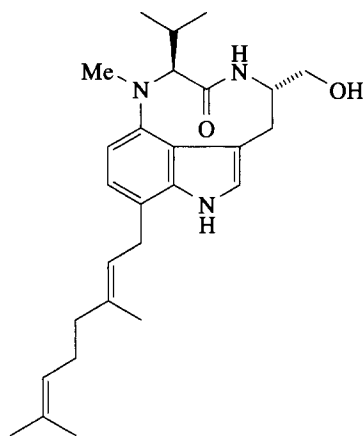
(216) (carbazomycin G)



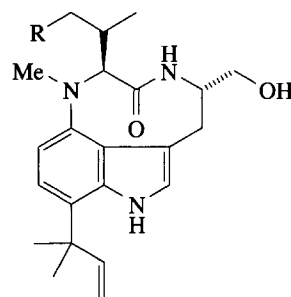
(217) (cytoblastin)



(218) (blastmycetin F)

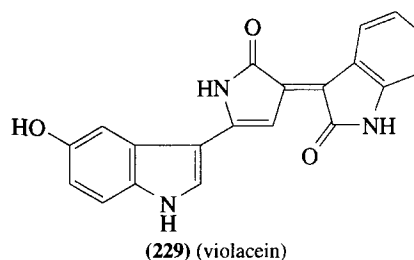
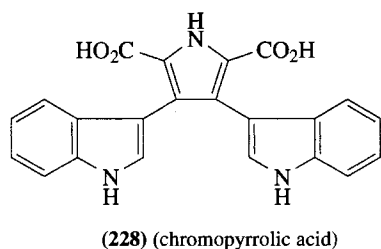
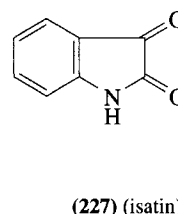
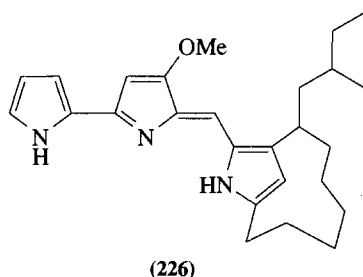
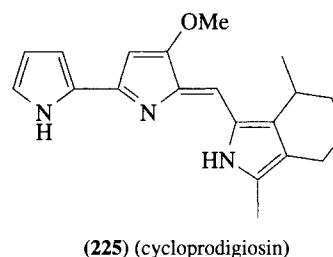
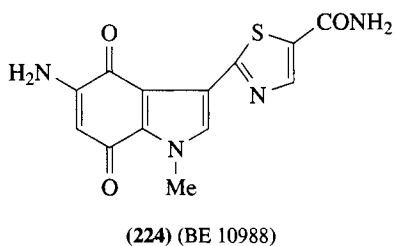
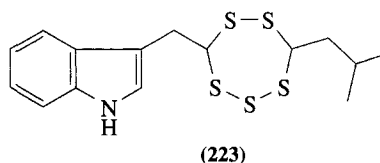
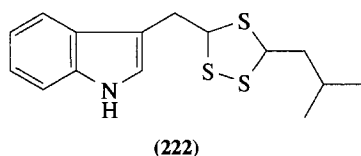


(219)

(220) R = H (pendolmycin)
(221) R = Me

thermophila archaea *Thermococcus tadjuricus* and *T. acidaminovorans* <93LA871>. The topoisomerase II inhibitor BE 10988 (**224**), which contains a thiazole ring, is produced by an unidentified organism <91TL2791>. The novel pyrroles cycloprodigosin (**225**) and (**226**) are produced by the marine bacteria *Beneckea gazogenes* and *Alteromonas rubra* <83TL2701, 83TL2797, 91JAN187>. Isatin (**227**), which is also produced by *Alteromonas* sp., protects embryos of the shrimp *Palaemon macrodactylus* from the pathogenic fungus *Lagenidium callinectes* <89SCI116>. Several related metabolites, such as chromopyrrolic acid (**228**) and violacein (**229**), have been isolated from *Chromobacterium violaceum* <93MI 204-18>. The cofactor tryptophan tryptophylquinone (TTQ), which is present in bacterial methylamine dehydrogenases (*Paracoccus denitrificans*), has structure (**230**) <95JA1485>.

Species of *Streptomyces* also produce an array of halogenated metabolites. Thus, cultures of *Streptomyces* sp. have yielded pyrroxamycin (**231**) <87JAN961> and the optically active neopyrrolomycin (**232**) <90JAN1192>. The novel metabolite roseophilin (**233**) from *S. griseoviridis* has significant cytotoxicity activity <92TL2701>. Complestatin (**234**), from *S. lavendulae* <89TL4987>, is closely related to the kistamicins found in cultures of *Microtetraspora parvosata* <93JAN1812>. Complestatin is a potent inhibitor of the complement system and the kistamicins are active against

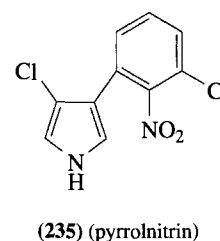
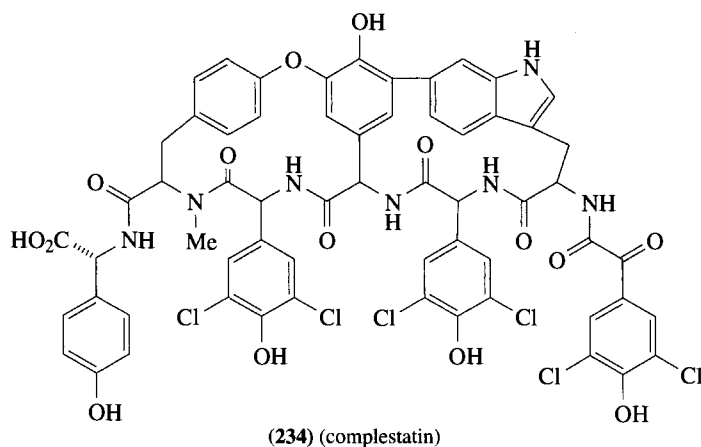
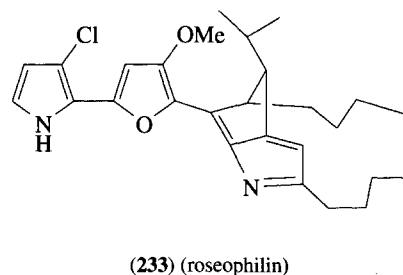
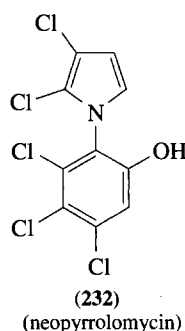
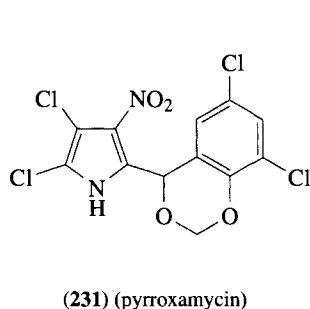
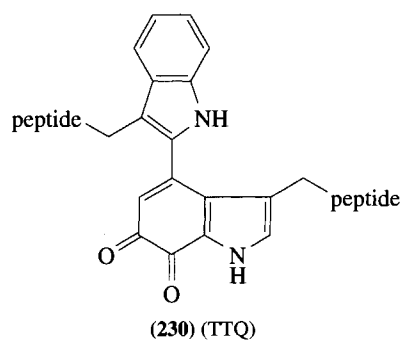


the influenza Type A virus. Another strain of *Streptomyces* produces chloropeptin I, which is identical to complestatin except for the attachment of the indole ring [94JAN1173]. Halogenated pyrroles are also produced by *Pseudomonas* bacteria. Pyrrolnitrin (235), known since 1965, has been found in *P. cepacia* along with related pyrroles [90MI 204-13]. Pyrrolomycins C-E (236)–(238) are produced by *Actinosporangium vitaminophilum* [83JAN1263, 83JAN1483]. Thiazohalostatin (239), which was isolated from cultures of *Actinomadura* sp., has a cytoprotective effect and prevents cell death caused by calcium overload [93JAN1638]. The microbe *A. melliaura* produces indolocarbazoles AT2433-A1 (240) and -A2 (241), which have strong antitumor and antibiotic activity [89JAN1784]. The Panamanian soil microorganism *Nocardia aerocolonigenes* (renamed *Saccharothrix aerocolonigenes*) has afforded the related rebeccamycin (242) and (243) [85TL4011, 87JAN668]. A *Microspora* sp. produces the novel cochinnimicins I–III (e.g., (244)) [92JAN1709].

2.04.7.2 Fungal Products

Like bacteria, the 1.5 million estimated species of fungi are abundant on Earth and produce a multitude of secondary metabolites, many of which contain heterocyclic ring systems.

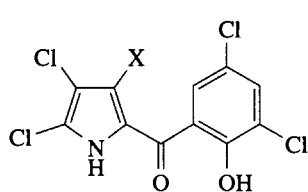
The fungus *Penicillium crustosum* produces an array of stunningly complex penitremes A–F, for example (245) [83JCS(P1)1857, 90CPB3473], and *P. nigricans* has yielded pennigritrem (246) [92JCS(P1)23]. The okaramines A–F, such as (247), are produced by *P. simplicissimum* [91ABC3143].



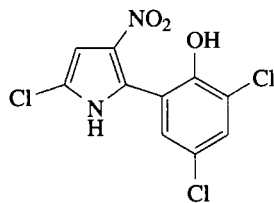
The fungus *Emericella desertorum* contains several related indoles such as emindole DA (**248**) <88JCS(P1)1689>, and *E. nivea* has yielded the isomeric pollen growth inhibitor emeniveol <92TL6987>. Emindole PA (**249**) was found in *E. purpurea* from an Egyptian desert soil sample <94JCS(P1)1673>.

Species of *Aspergillus* fungi produce many different structural types such as the ochrindoles A–D, for example (**250**) from *A. ochraceus* <94MI 204-21>, and (**251**) from *A. flavipes* <94MI 204-22>. Several related metabolites, such as (**252**), which have insect antifeedant activity, were isolated from *A. flavus* <88JOC5457>. The carbazole, aflavazole, which is related to (**252**), is also produced by this fungus <90JOC5299>, as are the aflatrems, such as (**253**) <92MI 204-09>. Tubingensin B (**254**) is produced by *A. tubingensis* <89TL5965>. A wide variety of ergot alkaloids are produced by *Claviceps* fungi, including two new fructosides of chanoclavine (**255**) from *C. fusiformis* <90MI 204-14>.

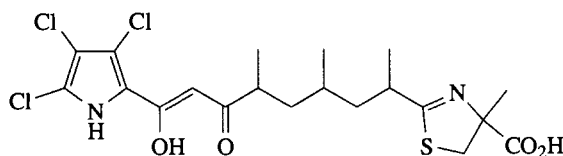
The fungus *Thielavia minor* contains OPC-15161 (**256**), which is an inhibitor of superoxide anion generation by guinea pig macrophages <91JAN52>. Fiscalin B (**257**) is found in *Neosartorya fischeri* <93JAN545>, and terezine D (**258**) is a metabolite of *Sporormiella teretispora* <95MI 204-03>. The potent inhibitor of squalene synthase, viridifungin C (**259**), is found in cultures of *Trichoderma viride* <93TL5235>. The fungus *Auxarthron umbrinum* produces rumbrin (**260**), which prevents membrane



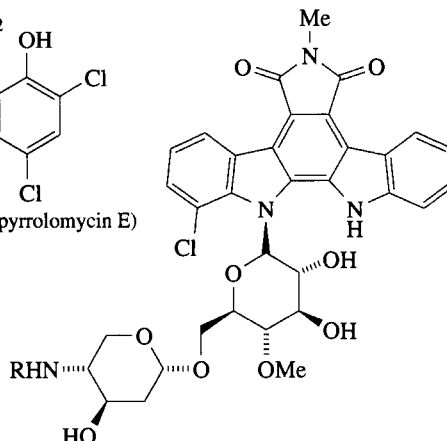
(236) X = H (pyrrolomycin C)
(237) X = Cl (pyrrolomycin D)



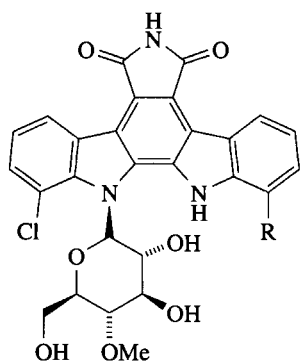
(238) (pyrrolomycin E)



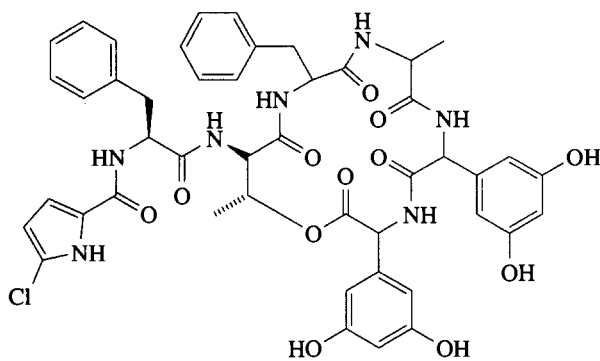
(239) (thiazohalostatin)



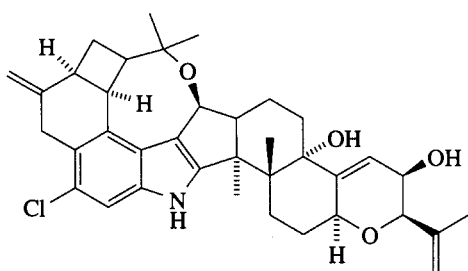
(240) R = Me (AT2433-A1)
(241) R = H (AT2433-A2)



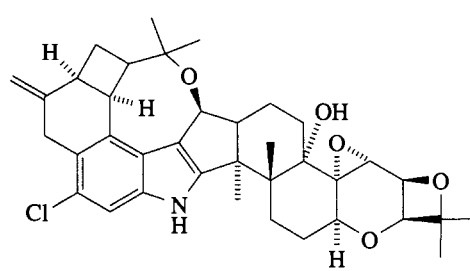
(242) R = Cl (rebeccamycin)
(243) R = H



(244) (cochinmicin III)



(245) (penitrem C)

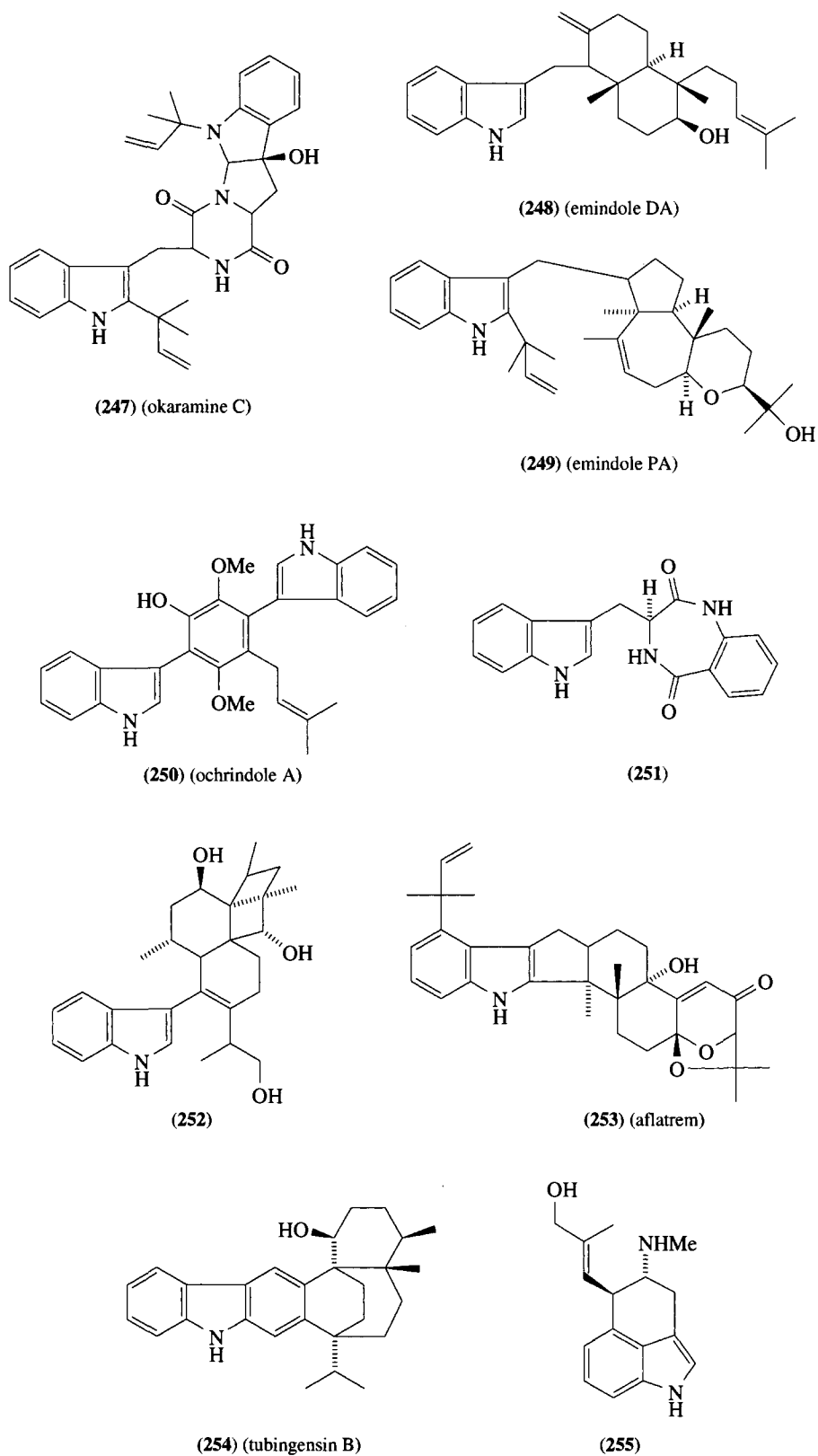


(246) (pennigritrem)

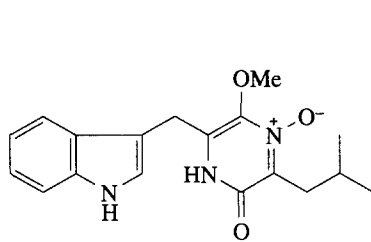
lipid peroxidation and calcium overload, and, thus, may be useful in the treatment of myocardial and cerebral ischemia <93JAN888>. The slime mold *Lycogala epidendrum* secretes lycogarubins A–C, for example, (261), which display some anti-HSV-1 virus activity <94TL2559>.

2.04.7.3 Marine Natural Products

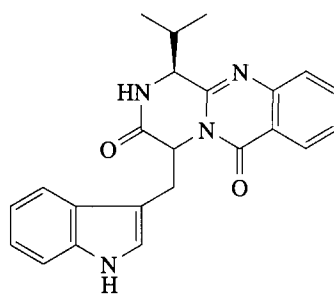
Since the mid-1980s there has been an explosion of activity in the area of marine natural products isolation and characterization.



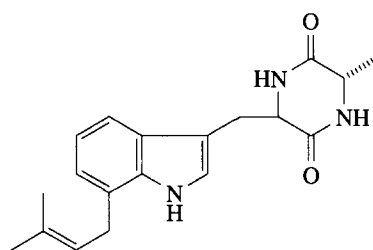
Marine sponges have furnished a rich collection of pyrroles, indoles, and a few carbazoles—many of which are halogenated. The simple pyrroles (262) and (263) have been isolated from *Agelas oroides* [94MI 204-23], from which oridin had earlier been isolated. Another *Agelas* sp. afforded



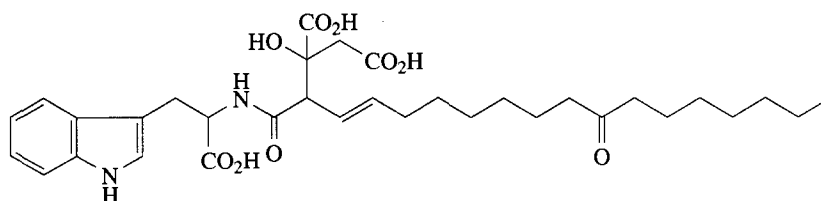
(256) (OPC-15161)



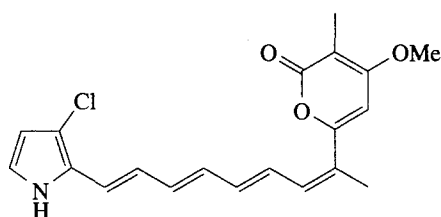
(257) (fiscalin B)



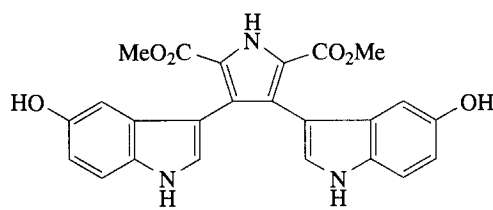
(258) (terezine D)



(259) (viridifungin C)

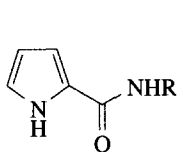
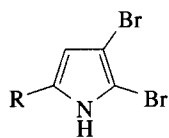
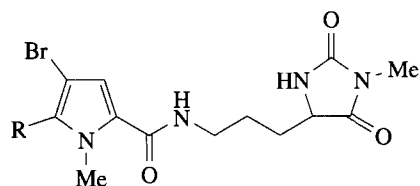


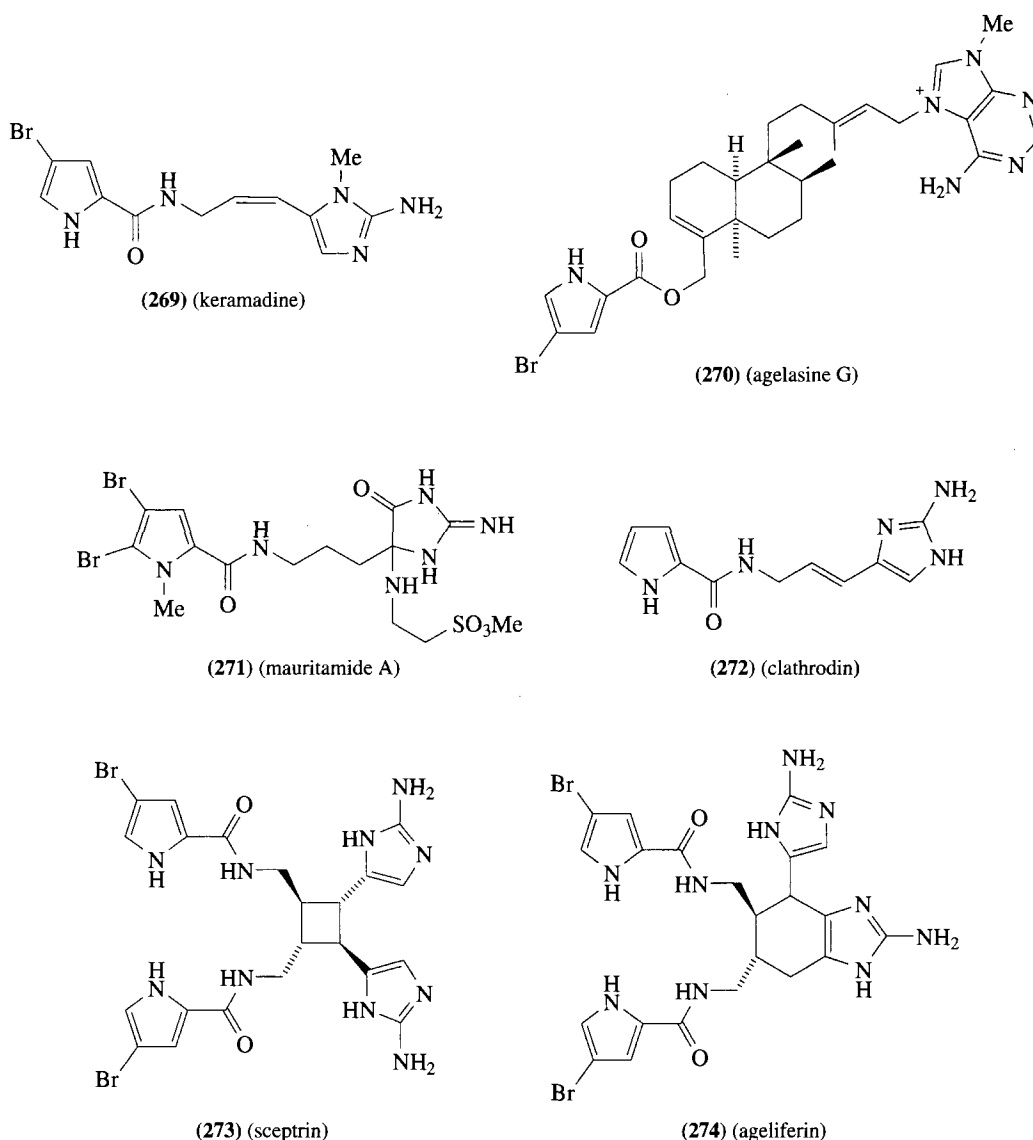
(260) (rumbrin)



(261) (lycogarubin A)

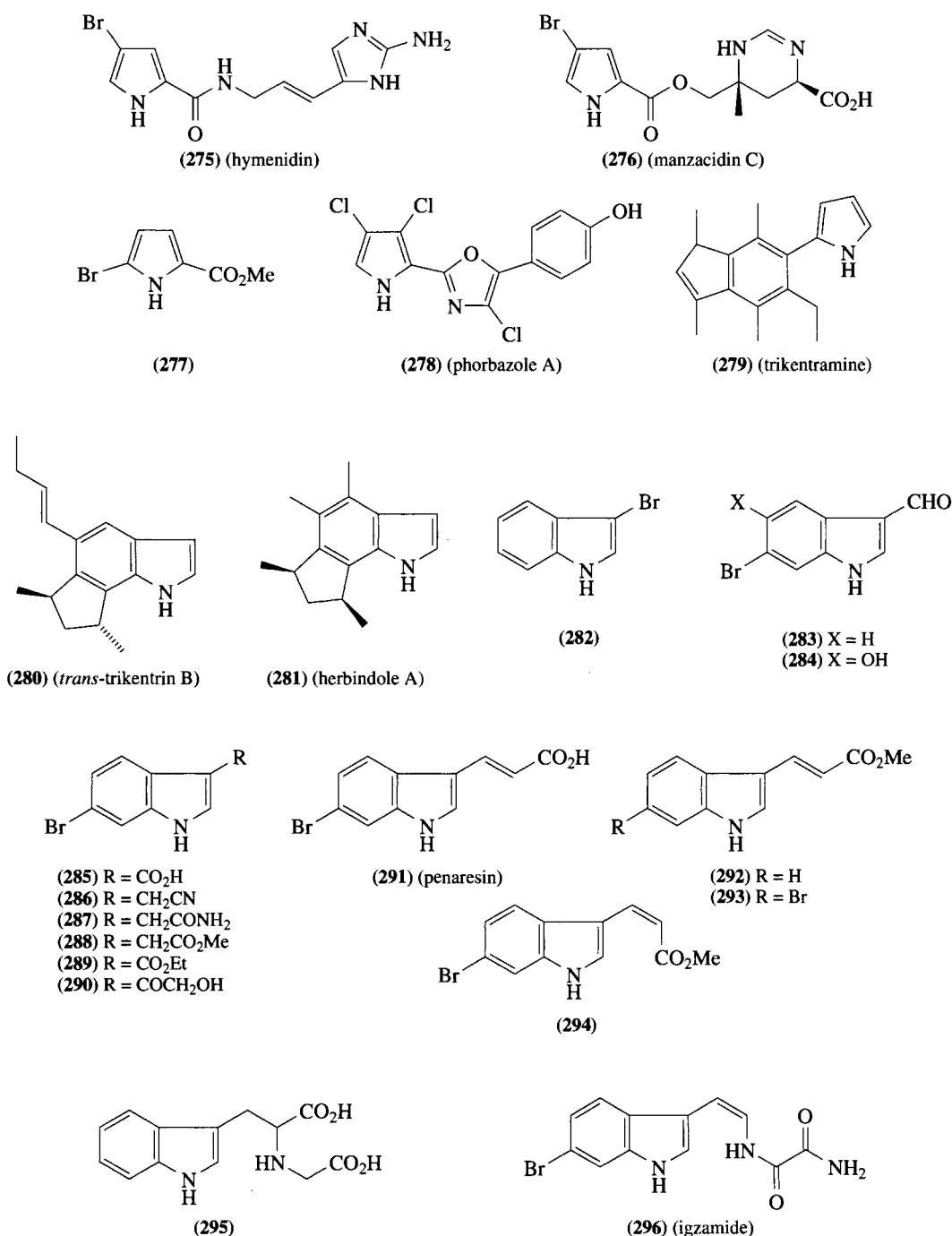
pyrroles including (264)–(266) <88CL803>. Midpacamide (267) and (268) are found in *A. mauritiana* <88CJC45>. Kermadine (269) is produced by an Okinawan *Agelas* sponge and is a novel antagonist of serotonergic receptors <84TL2475>. Another study of an Okinawan *Agelas* sp. revealed the anti-leukemic agelasine G (270) <92CPB766>. The Fijian *A. mauritiana* contains (267) as well as mauritamide A (271) <94TL1375>, and the Caribbean *A. clathrodes* produces clathrocin (272) (3-debromohymenidin) <91MI 204-10>. A set of dimeric sceptrins, such as (273) and ageliferrins, such as (274), have been isolated from *A. sceptrum* and *A. conifera* <89PAC525, 91JOC2965>. Some of these metabolites are potent actomyosin ATPase activators <90T5579>.

(262) R = H
(263) R = CHO(264) R = H
(265) R = CH₂OMe
(266) R = CO₂Me(267) R = Br (midpacamide)
(268) R = H



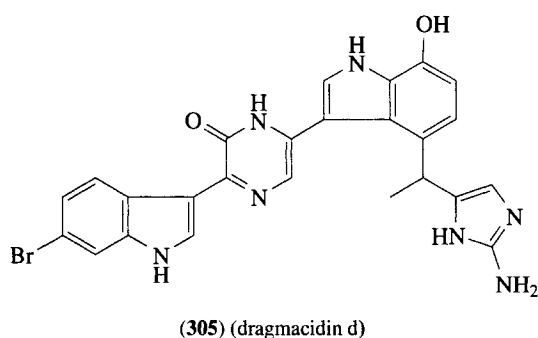
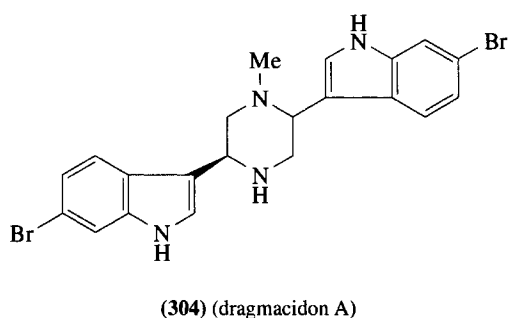
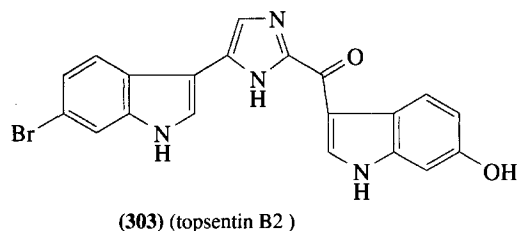
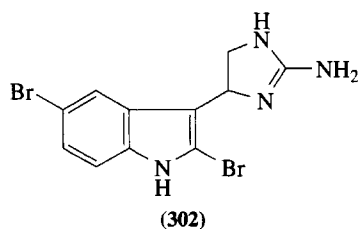
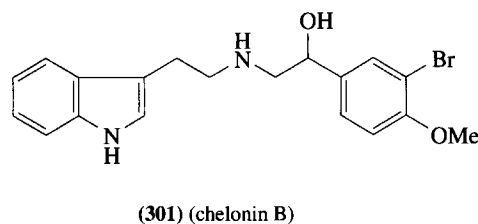
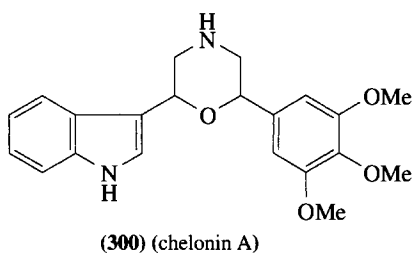
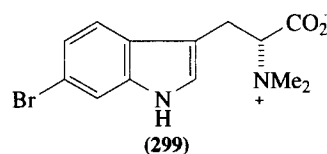
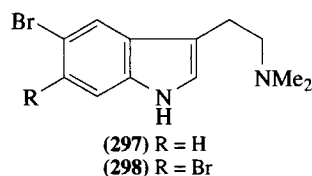
Hymenidin (**275**), which is an antagonist of serotonergic receptors, is found in an Okinawan *Hymeniacidon* sp. <86E1176>. This sponge also produces manzacidins A–C (e.g., **276**) <91JOC4574>, as well as several new cyclic heptapeptides that contain tryptophan <93T2391>. The Sri Lankan sponge *Lissodendoryx* sp. contains the simple pyrrole (**277**) <85MI 204-02>, and the novel chlorinated phorbazoles A–D (e.g., **278**) are produced by *Phorbos aff clathrata* sponge <94TL2589>. Species of the sponge *Trikentrion* have yielded several novel pyrrole and indole metabolites, including trikentramine (**279**) from *T. loeve* <90TL2979>, and a series of trikentrins (e.g., **280**) from *T. flabelliforme* <86T6545>. The Western Australian sponge *Axinella* sp. produces the related herbindoles A–C, such as (**281**), which have cytotoxic and antifeedant activity <90T3089>. The Western Indian ocean *Axinella* cf. *carteri* sponge produces the cytotoxic tryptophan-containing cyclic peptide axinastatin 4 <93H(35)711>. The three brominated indoles (**282**)–(**284**) are released by the sponge *Oceanapia bartschi* <93ZN(B)1408>, and (**283**) is also found in the sponges *Pleroma menoui* <89ZN914>, *Plocamissma igzo* <93MI 204-19>, and *Pseudosuberites hyalinus* <93MI 204-20>, as well as in the coral *Dendrophyllia* sp. <89HCA1444>. The deep water sponge *Pseudosuberites hyalinus* also contains 6-bromoindoles (**285**)–(**288**) <93MI 204-20>, and *Pleroma menoui* from the Coral Sea produces (**289**) and (**290**) <93MI 204-20>. Penaresin (**291**), which is 10 times more potent than caffeine as a calcium inducer, is found in the Okinawan sponge *Penares* sp. <90H(31)2205>. The New Caledonian sponge *Corallistes undulatus* produces serotonin and acrylate derivatives (**292**)–(**294**) <93MI 204-21>. An *Ircinia* sp. from Okinawa contains indole-3-acrylic acid (debromopenaresin) and the novel cytotoxic tryptophan derivative

konbamidin (**295**) <94MI 204-24>. The sponge *Plocamissma igzo* has also yielded igzamide (**296**) <93MI 204-19>.



Several tryptamines are also found in marine sponges. Thus, (**297**) and (**298**) have been identified in *Smenospongia aurea* <85T1039>, and D-6-bromohypaphorine (**299**) is found in *Aplysina* sp. <94MI 204-25>. The Palauan sponge *Chelonaplysilla* sp. is the source of chelonin A (**300**) and B (**301**) <91JOC4403>. The sponge *Discodermia polydiscus*, collected in deep Bahamian waters, contains the cytotoxic (**302**) <91JOC4307>. A number of related sponge metabolites having two indole rings have been discovered since the late 1980s, only a few of which are shown here. For example, topsentin B2 (**303**) has been found in the Mediterranean sponge *Topsentia genitrix* <87CJC2118>, the deep water Caribbean sponge *Spongisorites* sp. <88JOC5446, 91JOC4304>, and the British Columbian sponge *Hexadella* sp. <90T715>. The latter study also identified dragmacidon A (**304**) and related metabolites

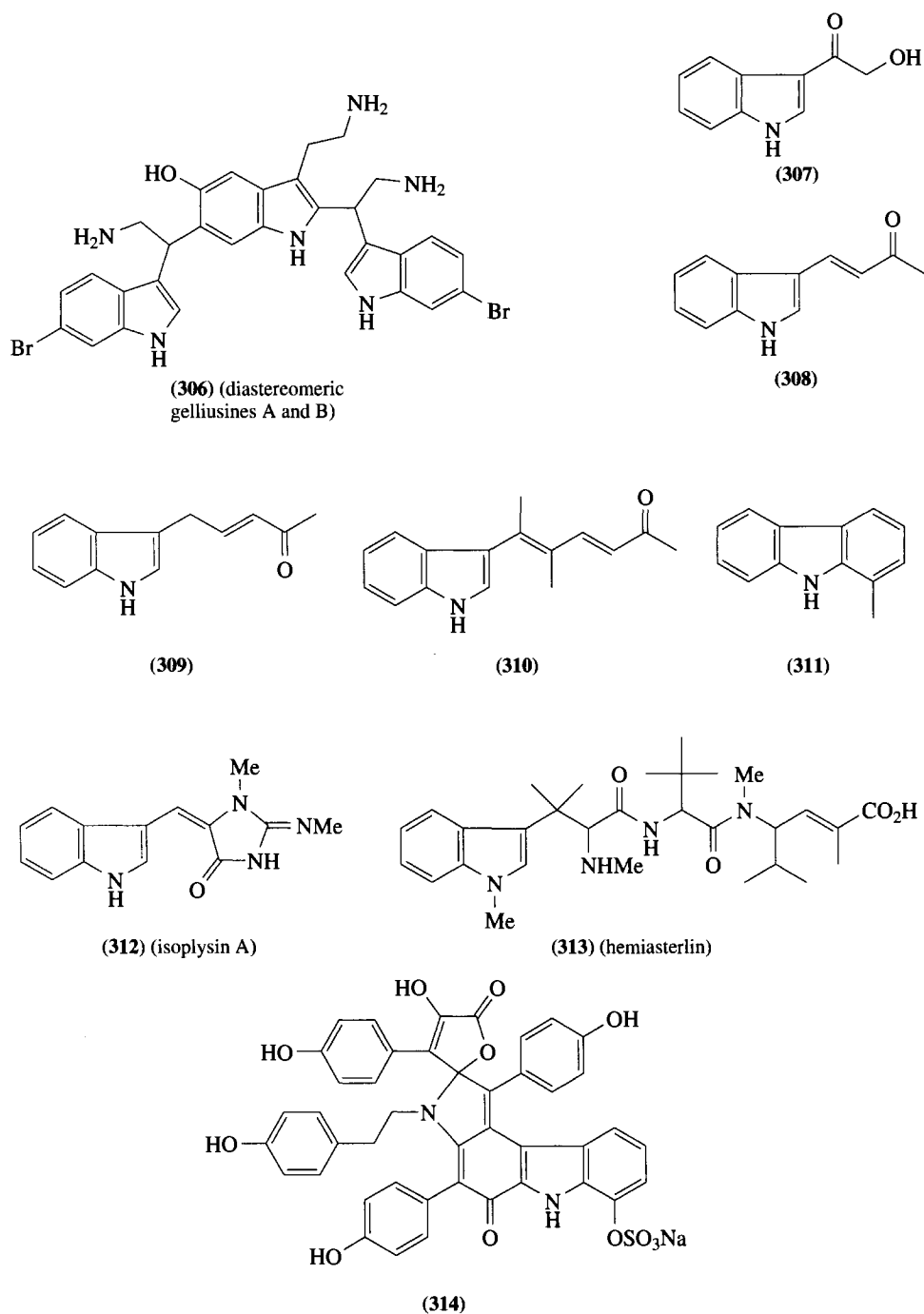
<90T715>. Another study of *Spongosorites* sp. revealed the presence of dragmacidin d (**305**), which is active against feline leukemia <92JOC4772>.



A deep-water Caledonian sponge (*Gellius* or *Orina* sp.) produces the novel tris-indoles gellusines A and B (**306**) as a pair of diastereomers <94MI 204-26>. The sponge *Tedania ignis* contains a series of simple indoles (**307**)–(**310**) and carbazole (**311**), although some of the former may be isolation artifacts <91MI 204-11>. A large number of imidazolyl-indoles have been discovered in sponges and soft corals—including isoplysin A (**312**) from *Aplysina* sp. <94MI 204-25>; for a leading reference, see <94T2241>. The photochemical isomerization of these metabolites has been investigated <89HCA1444>. The unique tripeptide hemiasterlin (**313**) has been isolated from *Hemiasterella minor* <94TL4453>. The sponge *Dictyodendrilla* sp. produces three reductose inhibitors such as (**314**), which contains the unique pyrrolo[2,3-*c*]carbazole spirolactone ring system <93JOC7632>.

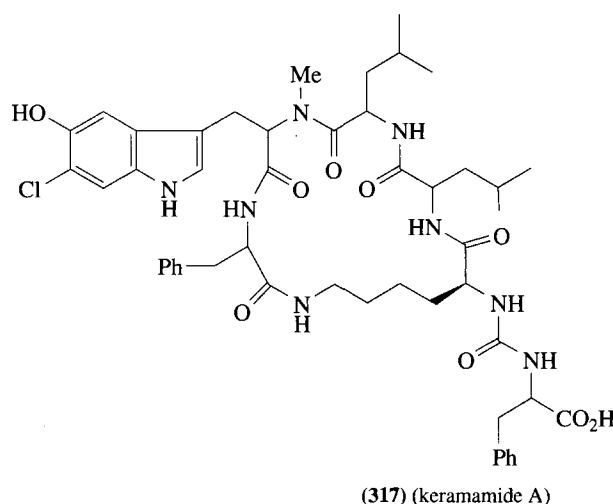
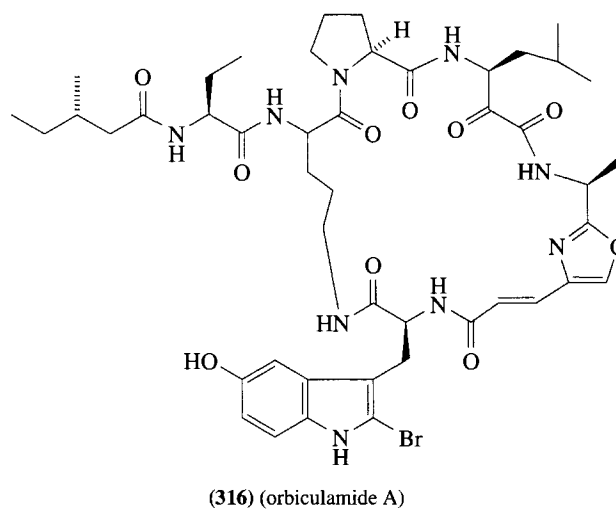
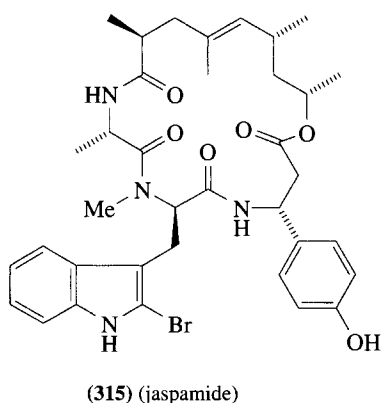
Several indole-containing cyclic peptides are found in sponges. For example, *Jaspis johnstoni* produces jaspamide (**315**), which contains the rare 2-bromotryptophan amino acid and which is a potent antitumor, antifungal, and insecticidal compound <86JA3123, 86TL2797, 87MI 204-06>. The Okinawan sponge *Theonella* sp. has yielded several structurally similar peptides, such as orbiculamide A (**316**) <91JA7811>, and keramamides A–H, J (e.g., **317**) <91JA7812, 95T2525>. The deep-sea sponge *Discodermia* sp. contains several novel peptides, such as polydiscamide A (**318**) <92JOC1767> and discobahamins A and B <94MI 204-27>.

Marine tunicates have also afforded several indole- and pyrrole-containing natural products of structural and biological interest. For example, the Brittany tunicate *Dendrodoa grossularia* has furnished several novel indoles, such as grossularine-1 (**319**) and (**320**) <86TL2621, 89T3445>. The Gulf



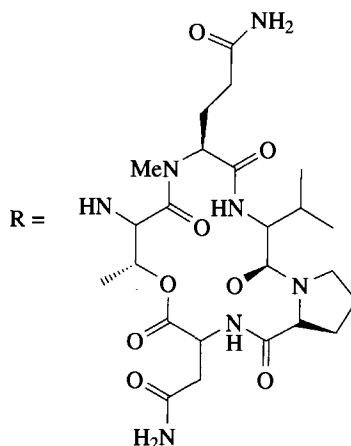
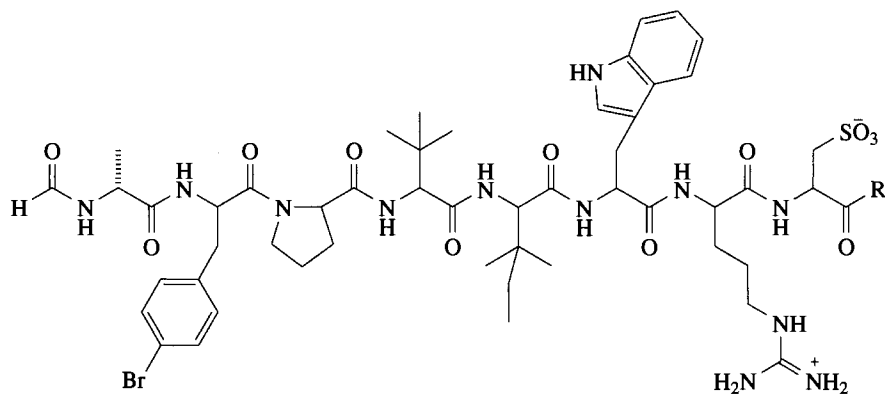
of California tunicate *Didemnum candidum* produces 6-bromotryptamine (321) and the bis-indole (322) <91MI 204-12>, while a tunicate from Fiji (*Polycitrella mariae*) contains citorellamine (323) <87TL749>. The solitary ascidian *Halocynthia roretzi* produces halocyamines A (324) and B, which inhibit the growth of fish viruses and marine bacteria <90B159>. The Philippine ascidian *Polyandrocampa* sp. produces polyandrocaramides A–D, (such as (325) and (326)) <90TL2521>, and *Eusynstyela misakiensis* has yielded the novel eusynstyelamide (327) <94MI 204-28>. An ascidian *Polycitor* sp. produces the novel polycitrins A (328) and B (329), and polycitone A (330) <94JOC999>. When disturbed, the marine ciliate *Pseudokeronopsis rubra* secretes the four chemical defense agents keronopsins (331)–(334) <94AG(E)1495>.

The marine ascidian *Atapozoa* sp. produces the new tambjamine E (335) and F <91E504>, and wakayin (336) is the first pyrroloiminoquinone from an ascidian (*Clavelina* sp.) <91JOC4596>. This

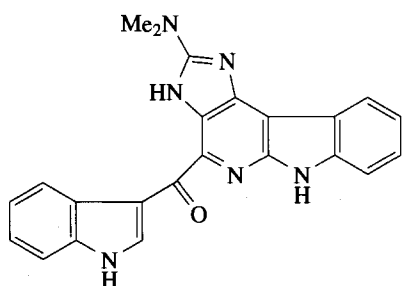


compound is cytotoxic and exhibits topoisomerase II inhibitory activity. The Caribbean ascidian *Lissoclinum fragile* has furnished the novel eudistomin U (**337**) and isoeudistomin U (**338**) <94MI 204-28>. The ascidian *Diazona chinensis* contains the potent cytotoxic diazonamides A (**339**) and B (**340**) <91JA2303>. Bryozoans, or “moss animal,” have yielded a rich array of novel metabolites, ranging from the simple indoles (**341**)–(**343**) from *Zoobotryon verticillatum* <93MI 204-22> to the extraordinarily intricate chartellines A–C (e.g., **344**) <85JA4542> and chartellamides A (**345**) and B <87JOC5638>. The bryozoan *Sessibugula translucens* and their predatory nudibranchs *Tambje eliora* and *T. abdere* contain tambjamine B (**346**) and others <83JOC2314>. The bryozoan *Bugula dentata* also contains tambjamines G–J <94AJC1625>, similar in structure to (**346**), and the blue pigment (**347**) <86E84>.

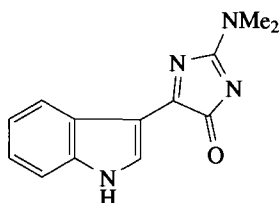
Marine acorn worms have proven to be a rich source of natural indoles, most of which were isolated prior to 1983 <B-83MI 204-03>. More recent examples include (**348**)–(**350**) from *Ptychodera* sp. <87T1063> and *Glossobalanus* sp. <85E1487>. The marine worm *Polyphysia crassa* produces the antibacterial and labile 2,3,4-tribromopyrrole (**351**) <90MI 204-15>. An *Aplysia* sp. sea hare contains bromoindoles (**352**) and (**353**) <89IJC(B)322>. The long and fascinating history of the ancient royal indigoid dyes from *Murex* molluscs <74MI 204-01, B-83MI 204-03> continues in 1992 with the isolation for the first time of 6-bromoindigotin (**354**) from *M. trunculus* <92MI 204-10>. The Japanese Ivory shell, *Babylonia japonica*, produces several highly toxic compounds, including prosurugatoxin (**355**) <85CPB2890>, although it now appears that bacteria associated with this animal actually produces these toxins <85CPB3059>. Likewise, bacteria (*Vibrio parahaemolyticus*) from the toxic mucus of the boxfish *Ostracion cubicus* produce vibrindole A (**356**) and (**357**) <94MI 204-29>. The mollusc *Monodonta labio* from Japan produces monodontamides D (**358**) and E (**359**) <94T6805>.



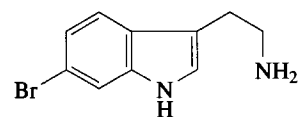
(318) (polydiscamide A)



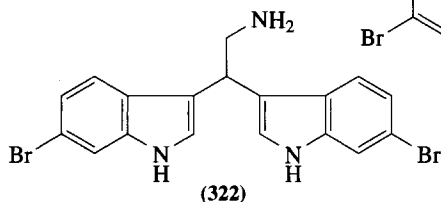
(319) (grossularine-1)



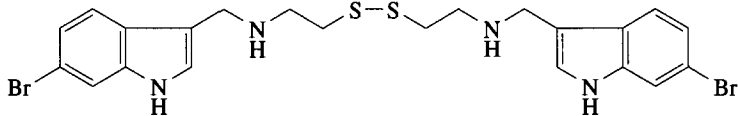
(320)



(321)

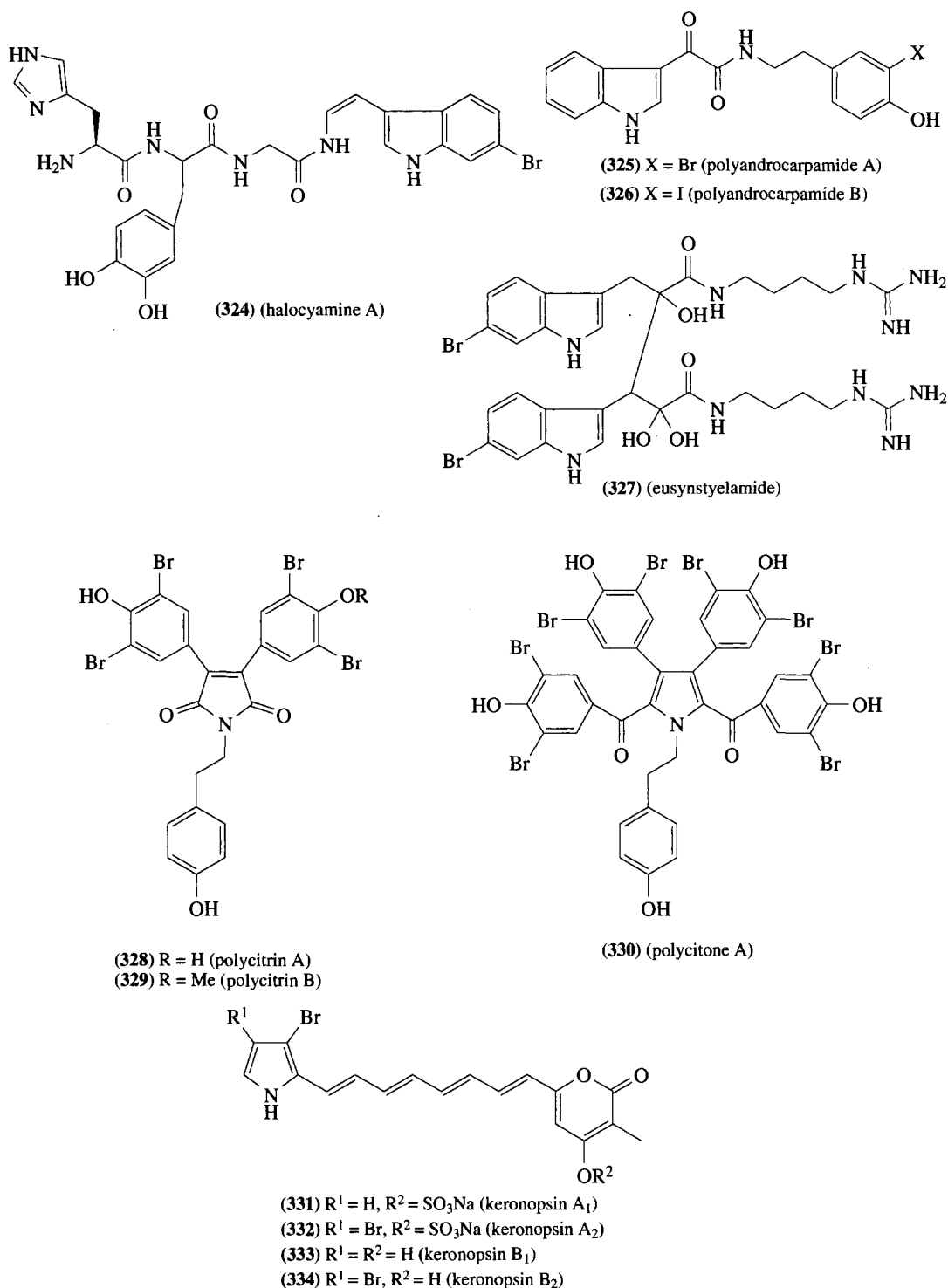


(322)



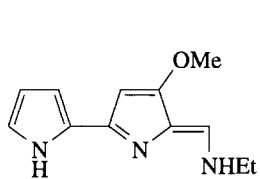
(323) (citorellamine)

Seaweeds produce an array of novel compounds including those that contain the indole or pyrrole ring. For example, the red alga *Gracilariopsis lemaneiformis* from Oregon contains the simple pyrroles (360) and (361) (91MI 204-13), and nostodione A (362) is a mitotic arrestor from a blue-green alga (94MI 204-30), which is related to the dimeric scytonemin (363), the structure of which has been determined (93E825). This blue-green algal metabolite is a photoprotective pigment present in many fresh water algae. The seaweed *Martensia denticulata* produces denticins A–C (e.g., 364),

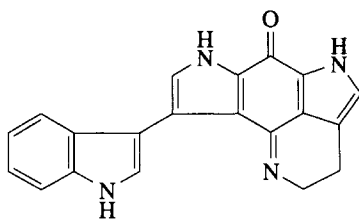


which have antiphotooxidative activity [94MI 204-31]. The novel sulfur-containing itomanindoles A and B (365), the bis-indole (366), and several other brominated sulfur-containing indoles are found in the red alga *Laurencia brongniartii* [88TL6091, 89T7301]. The bis-indole (367) is the latest of several such metabolites to be found in the blue-green alga *Rivularia firma* [91MI 204-14]. The blue-green alga *Tolypothrix tjipanasensis* is the source of 15 new indolo[2,3-*a*]carbazoles, the tjipanazoles, which include (368)–(370) [91T7739].

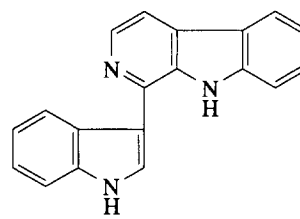
A large number of chlorinated indoloisonitriles and related indoles have been isolated from the



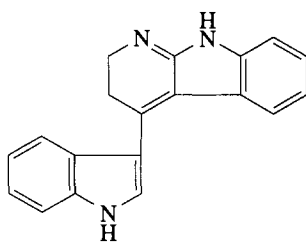
(335) (tambjamine E)



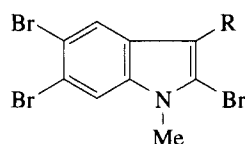
(336) (wakayin)



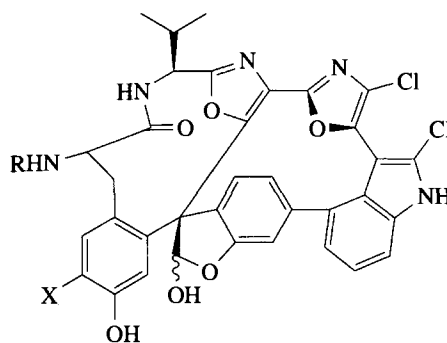
(337) (eudistomin U)



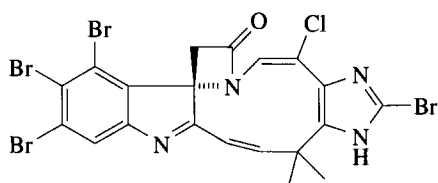
(338) (isoeudistomin U)



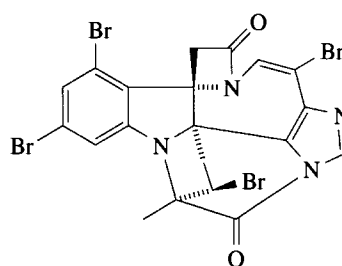
- (341) $R = \text{CH}_2\text{NMe}_2$
 (342) $R = \text{CH}_2\text{N}(\text{O})\text{Me}_2$
 (343) $R = \text{CHO}$



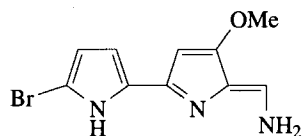
- (339) $X = \text{H}$, $R = \text{COCH}(\text{NH}_2)\text{CHMe}_2$
 (diazonamide A)
 (340) $X = \text{Br}$, $R = \text{H}$ (diazonamide B)



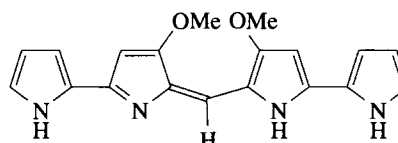
(344) (chartelline A)



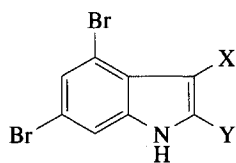
(345) (chartellamide A)



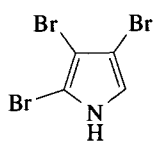
(346) (tambjamine B)



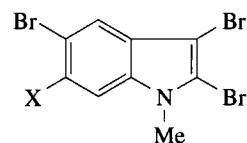
(347)



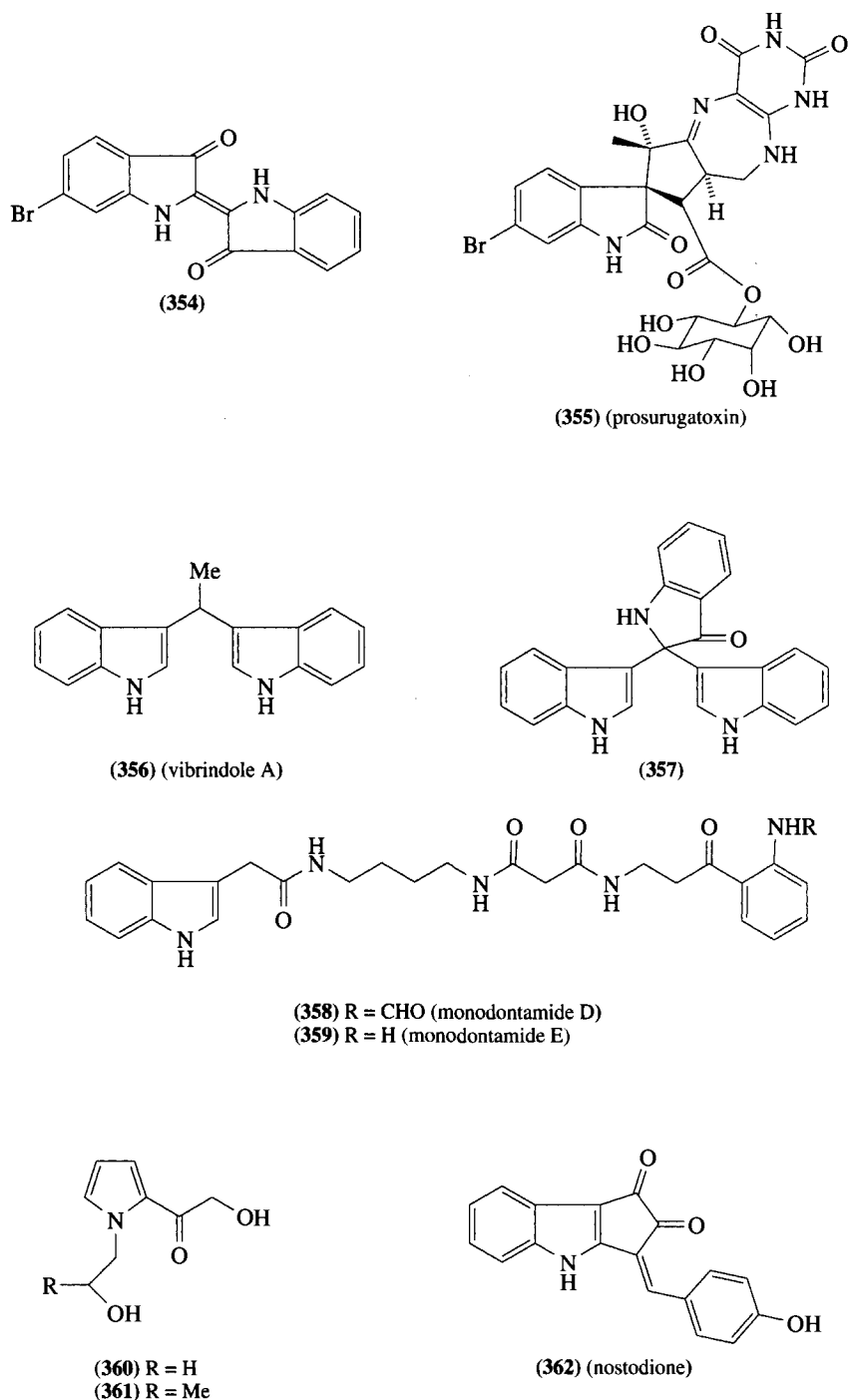
- (348) $X = \text{Br}$, $Y = \text{H}$
 (349) $X = Y = \text{H}$
 (350) $X = \text{H}$, $Y = \text{Me}$



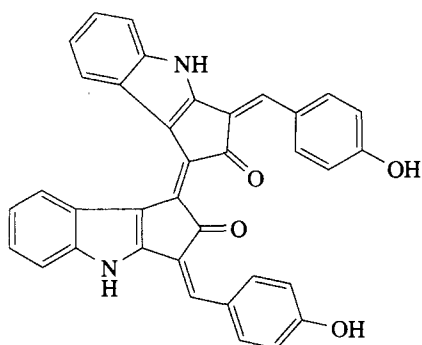
(351)



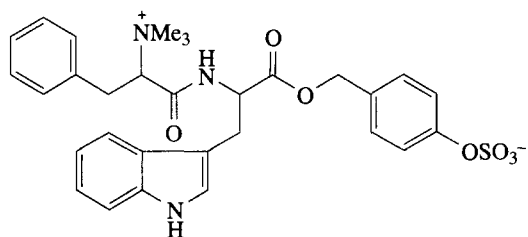
- (352) $X = \text{H}$
 (353) $X = \text{Br}$



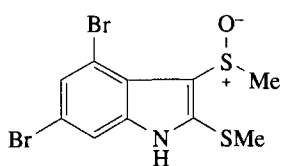
blue-green alga *Hapalosiphon fontinalis* <87JOC1036>. Two of these hapalindoles are shown as (371) and (372). The terrestrial blue-green alga *Fischerella muscicola* has yielded fischerindole L (373) <92TL3257>, while *F. ambigua* produces ambigaine isonitrile F (374) along with other similar metabolites <92JOC857>. The oxindole (375) has also been cultured from *Hapalosiphon fontinalis* <89P1565>, and *H. welwitschii* and *Westiella intricata* produce several complex indoles, the major metabolite of which is (376) <94JA9935>. The blue-green alga *Lyngbya majuscula* contains lyngbyatoxins B (377) and C in addition to teleocidins <90MI 204-16>. The compound responsible for the bioluminescence of krill (*Euphausia pacifica*), which was isolated in 1968, has been shown to be (378) <93TL2779>, and is similar to the dinoflagellate (*Pyrocystis lunula*) luciferin <89JA7607>.



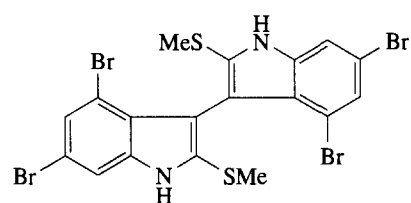
(363) (sytonemin)



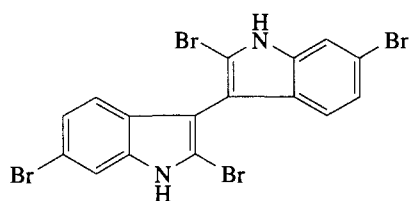
(364) (denticin A)



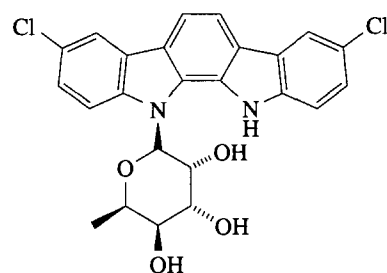
(365) (itomanindole B)



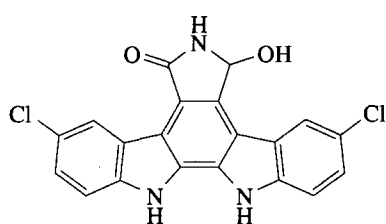
(366)



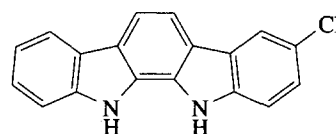
(367)



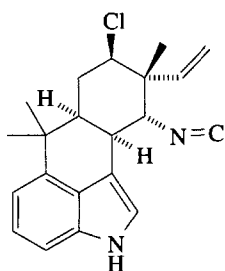
(368) (tjiapanazole A1)



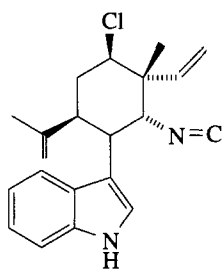
(370) (tjiapanazole J)



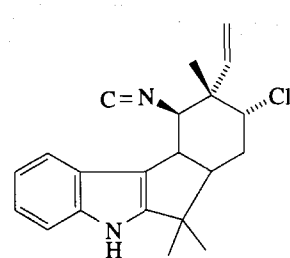
(369) (tjiapanazole I)



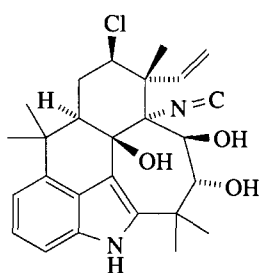
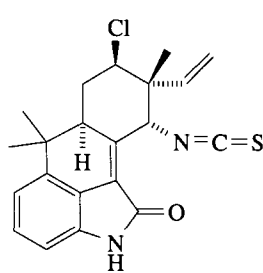
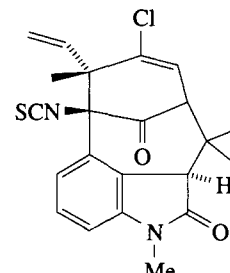
(371) (hapalindole A)



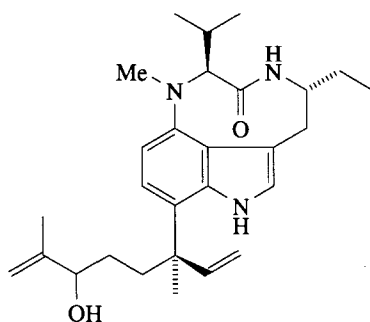
(372) (hapalindole E)



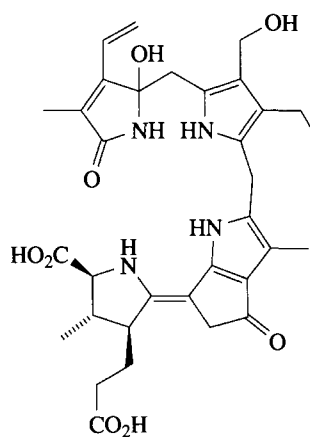
(373) (fischerindole L)

(374)
(ambiguine isonitrile F)(375)
(anhydrohapaloxindole B)

(376)



(377)

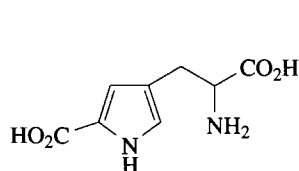


(378)

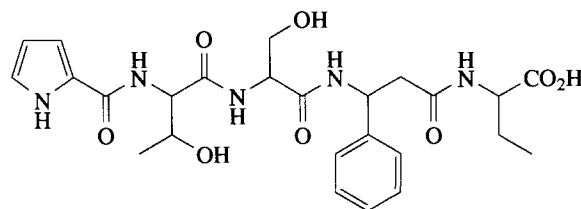
2.04.7.4 Plant Products

The myriad indole-containing plant alkaloids of the indoloquinolizidine type and other types are not covered. The list of simple indole, pyrrole, and carbazole plant alkaloids, which is discussed here, is far shorter.

Several simple pyrroles have been discovered in plants. For example, the poisonous mushroom *Clytocybe acromelalga* contains pyrrole amino acid (379) <91CL1541>, and *Aster tataricus* produces the pyrrole-containing peptides asterinins A–C (e.g., 380) <94P945>. The Chinese herbal medicine *Pseudostellaria heterophylla* has yielded (381) <88MI 204-08>. The interesting metabolite (382) of zeatin riboside from tobacco crown gall cells has been identified <90TL1419>. The Mexican folk plant *Quararibea funebris* has yielded several novel pyrroles, including funebrine (383), funebral, and funebradiol (384) <90MI 204-17>. The epilupinine derivative (385), and a related compound, are found in the plants *Virgilia divaricata* and *V. oroboides* <91PI891>. The complex 9,21-didehydroryanodine (386) is produced by *Ryania speciosa* <84CC1265>.

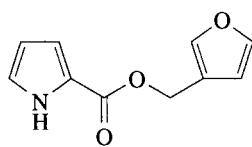


(379)

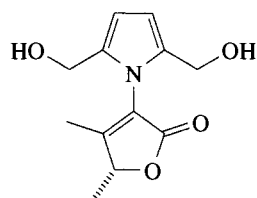


(380) (asterinin A)

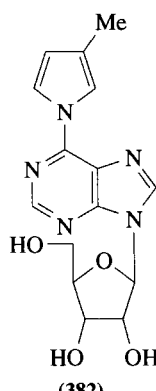
Several terrestrial plants utilize methyl 4-chloroindole-3-acetate (387) and acid (388) as growth hormones, including peas and beans. Studies in the late 1980s have involved *Vicia faba* (fava bean) <87ABC3081>, *V. amurensis* <87MI 204-07>, and *Pinus sylvestris* <86MI 204-08>. Green peas (*Pisum sativum*) also contain (*S*)-4-chlorotryptophan (389) and malonyl derivatives (390) and (391) <93TL1057>, and *V. faba* contains (389) and 4-chloro-6-methoxyindole (392) <92P2327>. Monatin



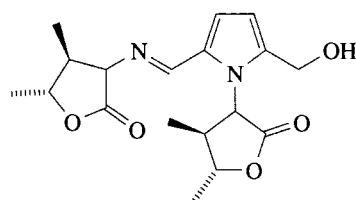
(381)



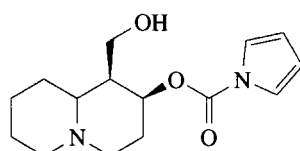
(384) (funebradiol)



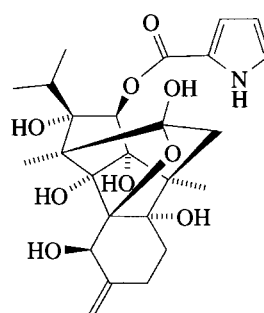
(382)



(383) (funebrine)



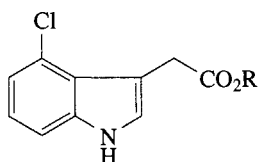
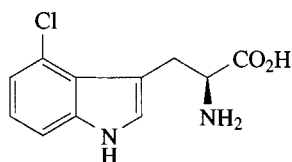
(385)



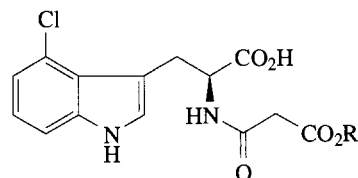
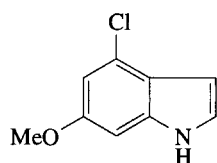
(386)

(393), which is 1400 times sweeter than sucrose, is produced by *Schlerochiton ilicifolius* <92JCS(P1)3095>. The simple enone (394) is found in *Pamburus missionis* <94P879>, and indolone (395) has been isolated from the edible mushroom *Pleurotus salmoneostramineus* <94JA8849>. This labile compound may play a very important part in the photochemical generation of oxygen from water. Paniculidine A (396) and B (397) and paniculol (398) have been characterized from *Murraya paniculata* <85CPB1770, 90JCS(P1)2047>, and *M. gleniei* has yielded the novel bis-indole (399) <90TL5217>. Another bis-indole, sciadole (400), is found in *Tricholoma sciodes* <94MI 204-32>, and the fruit bodies of this plant as well as those of *T. virgatum* produce simple indoles (401)–(403) <94T3571>.

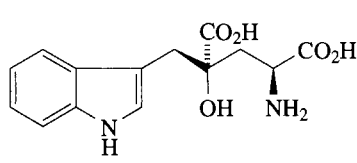
The novel indole dimers peronatin A (404) and B and two related oxygenated compounds are

(387) R = Me
(388) R = H

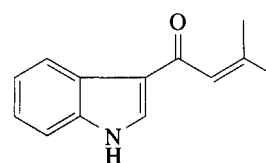
(389)

(390) R = Et
(391) R = Me

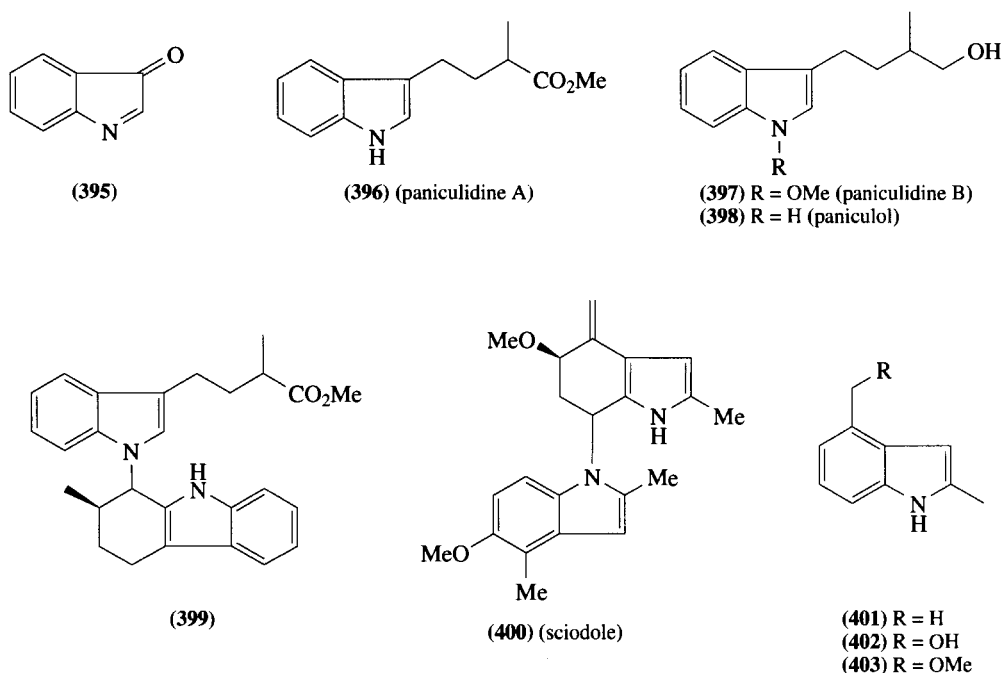
(392)



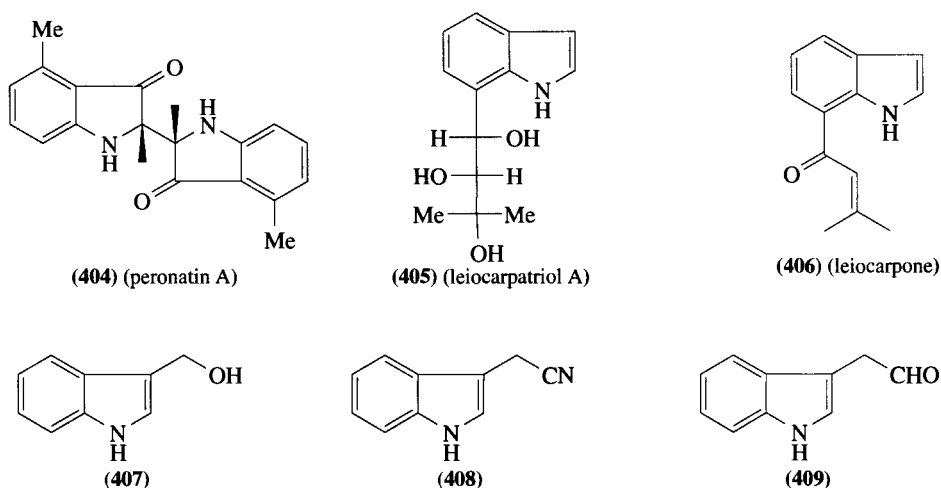
(393) (monatin)



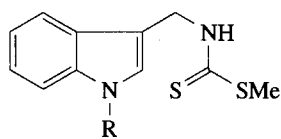
(394)



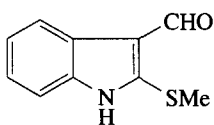
found in *Collybia peronata* and *Tricholoma sculpturatum*, respectively <94MI 204-33>. Several indole derivatives have been isolated from the roots of *Esenbeckia leiocarpa*, including leiocarpatriol A (405) and leiocarpone (406) <90G387>. In view of their suspected anticancer properties, cruciferous vegetables have been extensively examined for their chemical content. For example, kale, broccoli, collards, and brussels sprouts contain varying amounts of indoles (407)–(409) <88MI 204-09>, and Chinese cabbage (*Brassica campestris*) when inoculated with *Pseudomonas cichorii* produces the phytoalexins brassinin (410) and (411) <86CC1077, 90P1499>, and several brassicanals (412)–(415) <91P2915>. White cabbage (*B. oleracea*) produces similar compounds when provoked by *Pseudomonas cichorii* <90P1499>. Dithyreanitrile (416) is an insect antifeedant produced by *Dithyrea wislizenii* <91E304>, and camalexin (417) forms in the leaves of *Camelina sativa* (false flax) following elicitation by the fungus *Alternaria brassicae* <91T3909>.



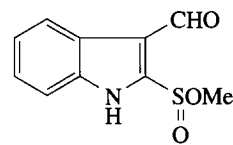
Yuehchukene (418) is a biogenetically novel alkaloid produced by *Murraya paniculata* <85CC47>, as is spermacoceine (419) (*Borreria verticillata*) <91P997>. Caulerpinic acid (420) is the latest of several similar indole alkaloids to be found in *Caulerpa racemosa* <91P3041>. The acetylcholinesterase inhibitor, crooksine (421), was isolated from *Haplophyton crooksii* <93P217>, and *Evodia rutaecarpa* contains (422) <88MI 204-10>. In addition to containing aspidochibine, *Aspidosperma quebracho*



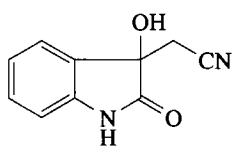
(410) R = H (brassinin)
(411) R = OMe



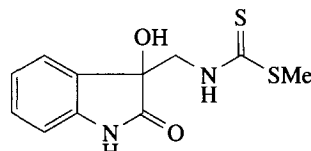
(412) (brassicinal A)



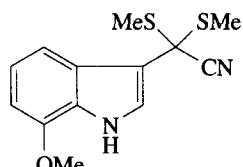
(413) (brassicinal C)



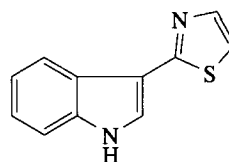
(414)



(415) (dioxibrassinin)

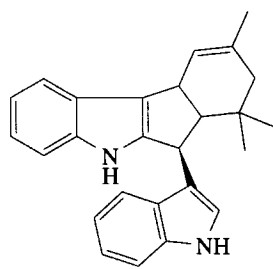


(416) (dithyreanitrile)

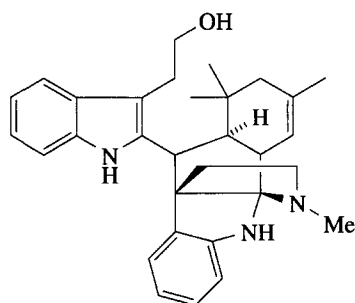


(417) (camalexin)

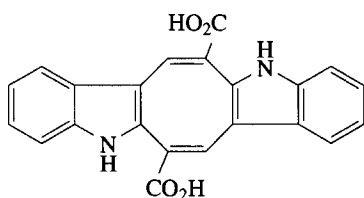
blanco has yielded (423), a compound that has apparently been formed by indole ring cleavage [91TL4949]. Monomargine (424), which also contains a novel structure, is found in *Monocarpia marginalis* and has cytotoxicity activity [93TL1795]. Also of biogenetic interest is the growing group of alkaloids from *Aristotelia* and several examples are known. Thus, *A. chilensis* contains (425) [90P1354], *A. fruticosa* has yielded aristofruticosine (426) [88TL3355], and *A. australasica* has provided the new 17-hydroxyhobartine (427) [90MI 204-18]. Several novel tryptophan-containing cyclic peptides have been identified in orange (*Citrus sinensis*) [91ABC2923].



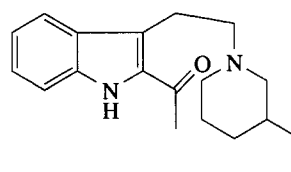
(418) (yuehchukene)



(419) (spermacoceine)

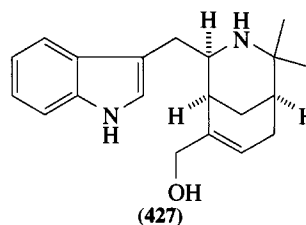
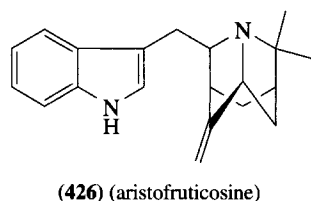
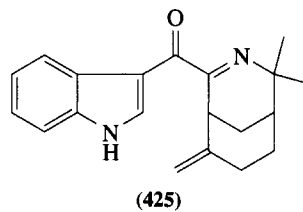
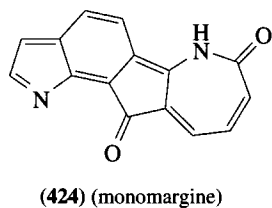
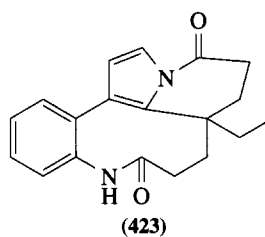
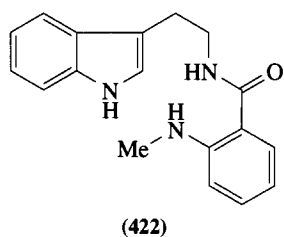


(420) (caulerpinic acid)

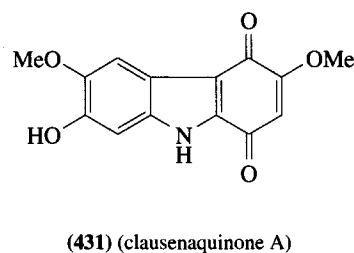
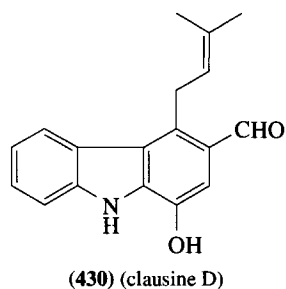
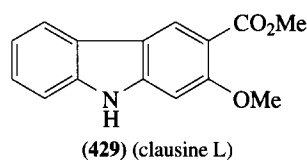
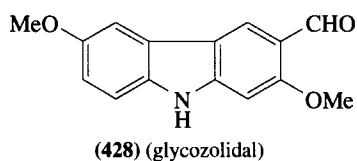


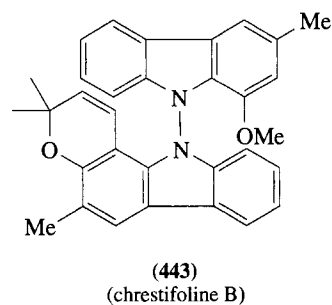
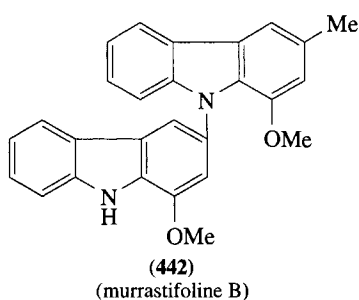
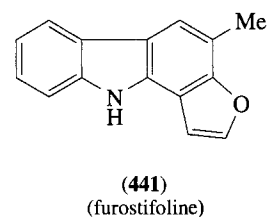
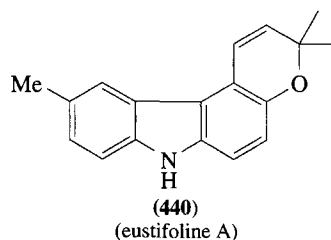
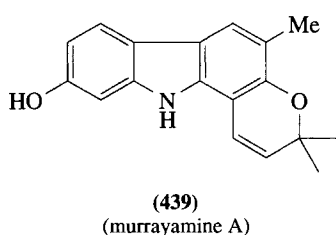
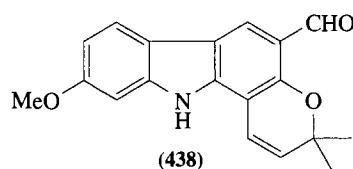
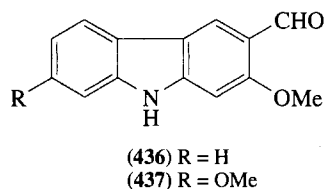
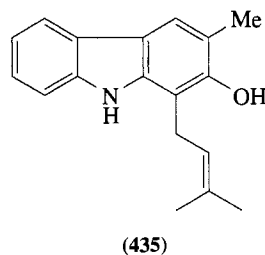
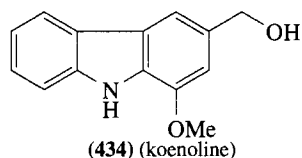
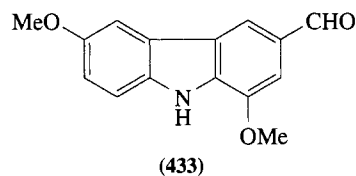
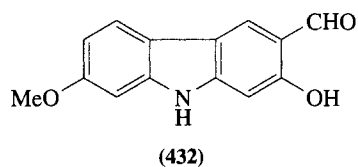
(421) (crooksidine)

Over the years, numerous carbazole-containing alkaloids have been discovered in plants, and several examples have been reported. Glycosolidal (428) is found in *Glycosmis pentaphylla* [85MI 204-03], and several carbazoles, such as (429)–(431), are produced by *Clausena excavata* [92CPB1069, 93P449, 94BMC2395], by *C. harmandiana* (e.g., 432) [88MI 204-11], and by *C. lansium* (e.g., 433) [91P343].



Species of *Murraya* are also rich sources of carbazole alkaloids, such as the cytotoxic koenoline (434) and (435) from *M. koenigii* <85P3041, 94P1073>, and the novel (436)–(438) from *M. siamensis* <90MI 204-19>. Several carbazoles related to (438) have been isolated from *M. euchrestifolia*, such as murrayamine A (439) <91P1048>, eustifoline A (440), furostifoline (441), and others <90CPB1548>. In addition, this Taiwanese plant contains several novel binary carbazole alkaloids, such as murrayamine B (442), chrestifoline B (443), and oxydimurrayafoline (444) <87CPB450, 90CPB1143>. Dimer (445) was identified in *M. exotica* <92MI 204-11>. In 1995, clausenal (446) was isolated from *Clausena heptaphylla* <95P787>.

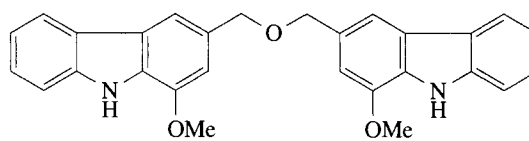




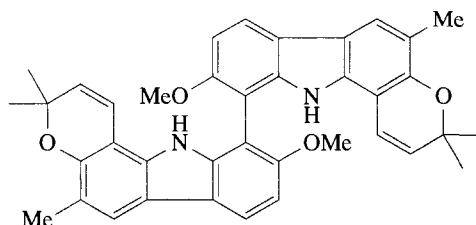
2.04.7.5 Miscellaneous Natural Products

A few instances of pyrrole-, indole-, carbazole-containing natural products are known from insects and higher animals, in addition to tryptophan-containing peptides.

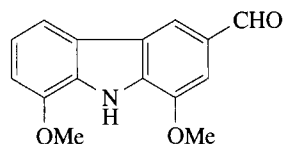
The funnel-web spider *Argiope* sp. produces argiotoxins 659 (447) and the related 673 <B-89MI 204-08>. These compounds exhibit excitatory amino acid antagonism. The ladybird beetle *Exochomus quadripustulatus* contains exochomine (448) <92TL1281>, and a very similar compound chilocorine is secreted by the ladybird beetle *Chilocorus cacti* <94T2365>. The Amazonian frog *Phyllomedusa bicolor* produces a tryptophan-containing peptide that interacts with an adenosine receptor and which is used by the natives as a stimulant for “hunting magic” <92PNA(89)10960>. Another indole



(444) (oxydimurrayafoline)

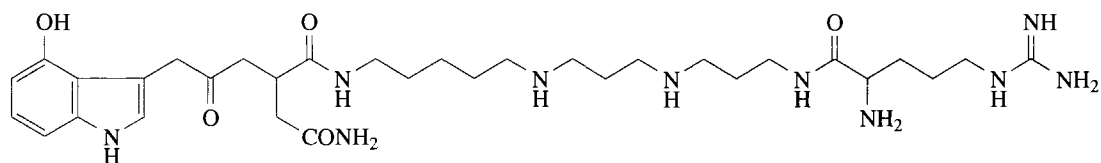


(445)

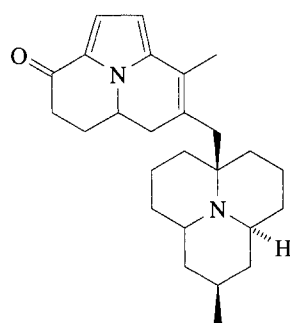


(446) (clausenal)

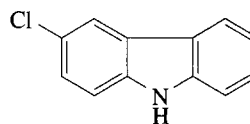
peptide, spinorphin, first isolated from the bovine spinal cord, is a natural inhibitor of enkephalin-degrading enzymes <93BBR(194)713>. The first carbazole of any type to be isolated from mammals was 3-chlorocarbazole (449), a compound that exhibits potent monoamine oxidase inhibition and is present in bovine urine <86MI 204-09>. Isatin (227) has been shown to be identical with “tribulin” and is present in human urine and rat brain and heart <88MI 204-12>. This compound is a monoamine oxidase inhibitor and is a normal constituent of tissues and body fluids of both humans and rats.



(447) (argiotoxin 659)



(448) (exochomine)



(449)

2.05

Furans and their Benzo Derivatives: Structure

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Università di Modena, Italy

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2.05.1 INTRODUCTION

This chapter surveys the structure of furans and their benzofuran derivatives in order to complement the first edition of *Comprehensive Heterocyclic Chemistry* (CHEC-I) with materials published between 1983 and 1993. The determination of the structural features of the furan ring has received remarkable attention, since this five-membered heterocyclic ring is encountered as a building unit in a variety of natural and synthetic products. Besides terpenes, it suffices to mention ribose and deoxyribose, which play an important part in the vital function of nucleic acids.

The knowledge of the structure of a molecule is of primary importance for ascertaining its physical and chemical properties. At the same time, these properties may provide information about the molecular structure. Chemical reactivity and magnetic properties, for instance, are closely related to molecular structure, and even biological and pharmaceutical activities seem to depend on molecular structure and dimension. The basis for the fundamental concept of aromaticity rests on geometric requirements. Many efforts have been made to determine structure and related properties. The development of technology has yielded new and ever more powerful and accurate instrumentation, enabling the employment of more and more precise and specifically designed experimental techniques. All this has enabled structural analysis to be extended to very large systems and more precise information about molecular structure to be obtained in different physical states (gaseous, liquid, and solid). Data collected in different phases may be compared either to determine the possible structural differences or to explain the observation of similar or different behaviors of some properties. Simultaneously, there has been a rapid evolution in theoretical calculations, with a rapid increase in their reliability. This has led to a process recently characterized as "mutation of chemistry" (91JST(230)5). Experimental results are often analyzed in parallel with data from computational studies, which, as regards their accuracy, are considered not only as useful for explaining experimental observations but also as very reliable for making predictions. Moreover, theoretical calculations enable the dynamic structural aspect to be analyzed in greater detail and information to be obtained about processes or chemical species not detectable experimentally.

In reviewing the material, experimental data are reported together with theoretical results when these are available, in line with the approach outlined above. The outline of the chapter has been kept similar to that of the previous edition. Proton and ^{13}C NMR spectra have been treated jointly, since in the majority of studies the NMR spectra for both these nuclei are reported and analyzed. Two short outlines have been introduced concerning boiling points and gas chromatography.

2.05.2 THEORETICAL METHODS

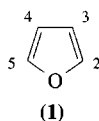
2.05.2.1 *Ab Initio* Methods

The continuing rapid increase in computer power has made it possible to undertake *ab initio* calculations even for quite large systems. The use of *ab initio* programs is no longer limited only to mainframe computers: packages for use on workstations or PCs are also available now. The development of calculation methodologies (geometry optimization with gradient method; localization of transition state as saddle point in the potential energy surface; inclusion of electron correlation; configuration interaction (CI); multiconfiguration self-consistent field (MCSCF); and so on) have made possible very useful results for analyzing or forecasting experimental behavior.

The *ab initio* method, which does not involve the assumptions and approximations of the semi-empirical methods, is very sensitive to the basis set and the theory level employed, as can be seen for the calculated equilibrium geometry of furan at different levels reported in Table 1. The increase of basis set size, and especially the inclusion in the calculation of electron correlation, improves the agreement between experimental and calculated geometrical parameters. Similar trends are found in the calculation of harmonic frequencies (88JPC1739, 91JST(235)185). The minimal basis set STO-3G is even better in the computation of dipole moments: 0.43 D at STO-3G level and 2.37 D at 6-31G** level for furan in comparison with an experimental value of 0.7 D ($1\text{ D} = 3.336 \times 10^{-30}\text{ C m}$) (87JA7347).

The *ab initio* techniques have also been employed to estimate other molecular properties such as electronic spectra and ionization potentials. These studies have been performed on furan (84CPH(90)231, 85JCP(83)723, 89JCS(P2)263, 93JA6184) in comparison with other 5-membered rings and only in one paper (83JST(105)375) was the analysis extended to benzoheterocyclic derivatives. Although all of these *ab initio* calculations provide values for molecular properties in accordance with experimental trends, only when the effects of electron correlation are introduced do the calculated values better reproduce the experimental ones quantitatively.

Furan obeys the $4n + 2\pi$ electron rule and is therefore considered to be an aromatic compound. Different procedures were applied to results of *ab initio* molecular orbital theory for determining the degree of aromatic character of furan with respect to other cyclic compounds (81JST(85)163, 88JA4204, 88JST(163)173, 90IJQ843). The commonly accepted order of aromaticity (85CHE717) thiophene > pyrrole > furan is confirmed in all the approaches.

Table 1 Experimental and computed geometry of furan (1).

	Experi- mental	STO-3G	4-21G	4-21G**	DZP MP2	MNDO	AMI	MM2
Bond length (pm)								
O—C(2)	136.2	137.6	138.6	135.2	136.4	136.7	139.7	136.0
C(1)—C(3)	136.1	134.0	133.8	133.5	137.4	139.0	139.7	136.4
C(3)—C(4)	143.1	144.5	145.1	144.3	143.3	144.4	144.7	142.8
C(2)—H	107.5	108.2	106.3	107.0	107.8	108.4	108.5	110.2
C(3)—H	107.7	107.7	106.4	107.2	107.9	107.9	108.6	110.2
Bond angles (deg)								
C(2)—O—C(5)	106.5	105.4	106.8	106.4	106.9	102.0		106.3
O—C(2)—C(3)	110.7	111.2	109.7	111.1	110.5	110.2		111.0
C(2)—C(3)—C(4)	106.0	106.1	106.8	105.7	106.0	106.0		105.9
O—C(2)—H	115.9	116.5	116.1	116.1	115.7	117.1	114.3	117.5
C(2)—C(3)—H	127.9	127.2	126.9	126.8	127.9	126.6	125.5	127.1
Reference	79MI 205-01	78JA3981	81JST(85)163	81JST(85)163	88JPC1739	77JA4907	85JA3902	89JCC634

The chemical reactivity of the ring is linked with the aromaticity. The preferred protonation site in furan was determined at 6-31G**//3-21G level <86JCS(P2)147>. The protonation of the oxygen atom is less favored and occurs at the C₂, in agreement with experimental evidence. This result was also examined in terms of simple resonance structure arguments supported by MCSCF calculations.

A chemical reaction pathway can be studied with the *ab initio* method, not only determining the geometrical features and electronic structure of reactants and products but also exhaustively characterizing the transition state. This was achieved for the cyclization reaction of ethylene with furan <86IJQ511>, for conversion of 1-ethynyl-2-hydroxyethene to furan <89JCS(P2)683>, and for decomposition of tetrahydrofuran to ethylene and CO <90JA3374>. The reactivity of other derivatives containing the furan nucleus has received scant attention using *ab initio* methods. Examples are the study of differing regioselectivity in 1,3-dipolar cycloaddition in furan and benzofuran employing the PMO model <91JCS(P2)1243> and selectivity in cycloadditions to 2,3-dihydrofuran <84T441>.

A larger variety of derivatives containing the furan nucleus has been examined with this methodology along with studies on the conformational properties of the substituents on the ring. In the study of rotational isomerism, the determination of the more stable conformations, the energy differences between conformers, and the energy barriers for internal rotation has been achieved using minimal or large basis sets and at different levels of theory. The use of a minimal basis set generally enables the conformational behavior and the more stable conformers to be predicted correctly, but sometimes does not reproduce well the relative stabilities of conformers. An augmentation of the basis set employed can invert the relative stability of conformers and also increase the energy differences and the value of the energy barrier for interconversion. An example is furan-3-carbaldehyde <88JCS(P2)1501, 92ACS923>, for which the effect of an increase of the basis set and of the introduction of electron correlation effects, whether on the energy content or on the structure, was analyzed. The results of *ab initio* calculations for conformational properties are frequently combined with experimental evidence, with good agreement between theoretical and experimental data.

Another interesting conformational problem, in the case of the reduced furans, is related to the possibility of the ring assuming different conformations. The ring is puckered and undergoes free pseudorotation. Tetrahydrofuran is by far the most important of the reduced furans; its nucleus occurs in the ribose and deoxyribose sugar units (as well as in sucrose) and is therefore an integral part of the nucleic acids, RNA and DNA. Information about the equilibrium conformation, structural parameters, and energy minimum of the free molecule has also come from *ab initio* calculations at various levels for tetrahydrofuran <75JA1354, 83IJ72, 90JA3374, 93JPC7844> and

2,5-dihydrofuran <93JSP(160)158>. The influence on the ring conformation ascribable to substituents has also been investigated (see Table 2) <85IJQ517, 87JA5297, 93JA9734>.

2.05.2.2 Semiempirical Methods

Semiempirical MO methods are used extensively to study the properties and behavior of furan and related heterocycles. The reliability of the different approaches varies, so that it is difficult to recommend one in preference to the others, and it seems impossible to generalize the field of applicability of individual methods. The CNDO method, for example, gives reasonable results for dipole moments and charge densities but, in conformational analysis problems, does not always give the correct order of stability of conformers (e.g., in acyl derivatives of heterocyclic compounds <87AHC(41)75>). The more sophisticated MNDO method, as regards the formalism, is the nearest to *ab initio* calculations with respect to CNDO and INDO. The possibility of minimizing the total molecular energy as a function of all internal coordinates enables one to obtain fairly accurate molecular geometry (as reported in Table 1 for furan) and heat of formation (furan -8.7 kcal mol $^{-1}$, experimental -8.3 kcal mol $^{-1}$ (-36.4 and 34.7 kJ mol $^{-1}$, respectively)). Although MNDO has demonstrated its ability to reproduce many ground-state properties of molecules of several kinds, a number of weaknesses have been reported such as failure to reproduce hydrogen bonds, and activation energies that tend to be too large <77JA4899, 77JA4907>. Dewar and co-workers <85JA3902> have proposed a new version of MNDO (Austin Model AM1) in which the errors associated with MNDO seem to have been largely corrected. Nevertheless, for the furan molecule, as for pyrrole and thiophene, AM1 gives erratic results for the heats of formation (furan 3 kcal mol $^{-1}$; 12.5 kJ mol $^{-1}$) <85JA3902, 89H(28)1135>, whereas the molecular geometry is satisfactory (Table 1).

Application of semiempirical methods to conformational analysis, especially when rotation about an essential single bond in conjugated molecules is studied, may give unrealistic results. CNDO and MNDO, for example, often give a perpendicular or nearly perpendicular arrangement of the two parts of the molecule as the minimum in the potential energy function. The AM1 method seems better-behaved: the conformational preferences are quite satisfactorily reproduced, while the energy differences between the conformers are usually much too low <88JCC369>.

In spite of these problems, the semiempirical techniques at various levels of theory (especially PPP, EXT, CNDO, INDO, MINDO, MNDO, AM1) have been employed widely to estimate several properties of heterocycles containing the furan ring. Several studies have been published on the calculation of charge densities and dipole moments <87CCC409, 88AQ(C)156, 88EJM341, 89BSB327, 89H(29)657, 89JST(196)227, 90CJC1477, 92JST(253)179>, ionization potentials <82JST(86)231, 83JST(105)375, 91JST(249)305>, ESCA chemical shifts <89JST(198)427>, electronic spectra <82TCA559, 83JST(94)115, 84BCJ844, 86ZN(A)1425, 87SA(A)1431, 92BCJ2067>, photoelectronic spectra <84BCJ856, 84JST(108)25, 90CJC747, 92JES143>, force constants and vibrational frequencies <86JST(142)351, 88JCP7295, 88JSP(127)200>, studies on the site of protonation <81NJC505, 85HCA2037>, acidities <88JOC4427, 90CCC72>, chemical reactivity and reaction paths <83JCS(P)353, 84T441, 85NJC25, 86OMS(21)459, 87AG(E)471, 88CL1285, 88JA4533, 91JCS(P)1243, 91JOC4671, 92JST(253)243, 93JST(280)211, 93JST(284)37>.

Frequently, different semiempirical techniques are employed simultaneously and the results are compared; sometimes they are used by turns to reproduce a particular property. A study of the molecular geometry and electronic structure of condensed pentatomic heterocycles was carried out in this way, optimizing the molecular geometry at MNDO level while the electronic spectra were calculated at different CNDO levels <87JCPB1147>.

The MNDO method has been applied to characterization of the ground-state properties of a few oligomers of furan. The results for the relevant electronic properties (ionization potential, vibrational frequencies, and so on) derived as functions of the number of monomer units were then extrapolated to polymers <88SM109, 90JST(219)397, 91JPC9746, 91SM789, 91MI 205-01, 93JCP(98)769, 93SM(57)4467>. Minor applications are available for other semiempirical methods in the studies of polymers. Molecular properties of conjugated polymers containing the furan nucleus have been evaluated using the CNDO method, e.g. hyperpolarizabilities <91JCS(F2)3009>, valence bands XPS spectrum <93CPH(175)283>, and conformation defect <91MI 205-01>. A modified extended Hückel method (for calculating electronic properties) together with a partial retention of diatomic differential overlap (PRDDO) method (to predict the conformational potential energy surfaces) was employed to study phenylene-furan copolymers <92MM3591, 93MM2095>.

A new method for obtaining optimized parameters for semiempirical methods has recently been

Table 2 Theoretical methods employed in investigation of the properties (such as geometry) of furans.

<i>Basis and method^a</i>	<i>System</i>	<i>Property</i>	<i>Reference</i>
GDZ, HF	Furan	Geometry, crystal, gas phase	88JST(164)175
GDZ, HF		Geometry, IR, aromaticity	91JST(235)185
GDZP, MP2		Geometry, IR	88JPC1739
GDZP, HF		Geometry, delocalization	88JA4204
GDZ, GDZP, HF		Geometry, aromaticity	81JST(85)163
GMIN, HF		Geometry, aromaticity	88JST(163)173
GDZP, HF		Aromaticity	90IJQ843
GEXT, HF		Electronic spectra	85JCP(83)723
GDZP, CI		Electronic spectra	80CPH(47)313
ANO, CASPT2		Electronic spectra	93JA6184
GEXT, CI		Electronic spectra, IP	84CPH(90)231
DZ, spin coupled		Resonance energy, IP, electronic spectra	89JCS(P2)263
GDZ, GDZP, GEXT MCSCF		Geometry, protonation	86JCS(P2)147
GDZ, GDZP, MP4SDQ		Geometry, formation of furan	89JCS(P2)683
GMIN, GDZ, HF		Geometry, cyclization	86IJQ511
GMIN, GDZ, GDZP, HF		Protonated system, charge transfer	84JPC3539
GDZP, HF		Electronic distribution	92JST(255)221
GMIN, HF		Geometry, conformation	78JA3981
GMIN, GDZ, GDZP HF	Furan and substituted furans	Geometry, vibrational overtone	91JPC7659
GMIN, GDZ, HF, AM1		Geometry, cycloaddition	91JCS(P2)1243
GMIN, GDZ, HF, CNDO/S, INDO/S, HAM/3	Furan, benzofuran, dibenzofuran	Electronic spectra, IP	83JST(105)375
GMIN, GDZ, GDZP, HF	Furan, tetrahydrofuran	Hydration	87JA7346
GMIN, HF	Substituted furans	Geometry, conformation	88G753
GDZP, MP2		Geometry, conformation, microwave spectra	93ACS849
GTO, HF		Geometry, conformation	81SA(A)643
GDZP, MP2		Geometry, conformation, microwave spectra	92ACS923
GMIN, GDZ, HF		Geometry, conformation	88JCS(P2)1501
GMIN, GDZ, HF		Geometry, conformation	87JCS(P2)961
GMIN, GDZP, HF		Geometry, conformation	91JST(228)71
GMIN, GDZ, GDZP, CI		Geometry, diradicals, singlet–triplet energy	86JA8086
GDZ, HF, PM3		Conformation	91CB2481
GMIN, HF		Geometry, conformation	87JCS(P2)1443
GMIN, GDZ, HF	Bifurans	Geometry, conformation	84JST(108)199
GMIN, GEXT, HF	Hydrofurans	Geometry, conformation	87JPC545
GMIN, GDZ, GDZP HF		Geometry, ring puckering, microwave spectra	93JSP(160)158
GMIN, HF, MINDO/3	Tetrahydrofuran	Cycloadditions	84T441
GDZ, GDZP, CI		Geometry, protonation	86OMS(21)209
GDZ, GDZP, MP2		Geometry, deoxygenation	90JA3374
GDZP, HF		Heat of formation	92JA2880
GDZ, CI		Geometry, hyperfine coupling constant in radical cation	92TCA291
GDZP, HF		Pseudorotational pathway	83IJ72
GMIN, GDZ		Pseudorotational pathway	75JA1354
GMIN, GDZ, GDZP, MP2		Geometry, ring puckering, IR	93JPC7844
GDZ, GDZP, HF		Electronic spectra, IP	90JA538
GMIN, GDZ, HF		Geometry, conformation, pseudorotational pathway	87JA5297
GDZ, HF	Substituted tetrahydrofurans	Geometry, conformation, pseudorotational pathway	85IJQ517
GDZ, HF		Geometry, conformation, pseudorotational pathway	93JA9734
GDZ, GDZP, HF, AM1	Polyfuran	Geometry, ring puckering	93JST(280)211
GDZ, HF		Geometry, electronic	92JST(254)243
GMIN, HF		Electronic	88JCP(88)386
GDZ, HF		Electronic	92JCP(96)2339
GMIN, GDZ, HF	Dibenzofuran	Electronic	90JST(208)163
GMIN, HF		Electrostatic potential	86JA915
ANO, CASSCF	2,3-Dihydrobenzofuran	Electronic state, reactivity	93ACS176
GDZ, HF		Geometry	90JPC7418
GTO, GDZ, GDZP, HF	Fused 5-membered	Relative stability	90BCJ3117

^a Codes for basis sets: GMIN (GTO minimal including STO-NG); GDZ (GTO double zeta); GDZP (GTO double zeta + polarization); GEXT (GTO extended); ANO (atomic natural orbital). Codes for methods: HF (Hartree–Fock); CI (Configuration interaction); MCSCF (multiconfiguration self-consistent field); MPn (*n*-order Møller–Plesset correlation energy correction).

developed and applied to MNDO <88JCC209, 88JCC221>. This methodology, named PM3, has been utilized to determine and analyze the structure of some furan derivatives <91CB2481, 92JOC5271, 93BCJ2707>.

2.05.2.3 Molecular Mechanics

The molecular mechanics (MM) or force field method is an empirical method based on classical mechanics and adjustable parameters. It has the disadvantage of being limited in its application to certain kinds of compounds for which the required parameters have been determined (experimentally or by theoretical calculations). Its advantage is a considerably shorter computation time in comparison with other procedures having the same purpose. This method has been shown to be very reliable and efficient in determining molecular structures, energies, and other properties for a wide variety of compounds.

In the analysis of the structural properties of heterocyclic compounds containing an oxygen atom, the most frequently used force field among several available seems to be Allinger's MM force field. The earlier version was developed for application to conjugated systems by including a π -system molecular orbital treatment in calculations <87JCC581, 88JA2050> and with the parameters extended to include furan and related compounds <85TL2403, 88JOC5471, 89JCC635>. The molecular structure obtained for the furan ring is reported in Table 1 and compares favorably with experiment. Very few studies are yet available on furan derivatives <90T4473, 91T6427, 92JA3926>, whereas the method has been applied extensively to conformational analysis of hydrofurans. For 2,3-dihydrofuran and tetrahydrofuran, the MM2 method correctly identifies the preferred equilibrium conformation with good accuracy and does reasonably well in predicting the barriers to planarity and pseudorotation <87JSP(124)369, 90JPC1830>. The application of this methodology to substituted tetrahydrofurans has been shown to be very efficient for the analysis of conformational preferences, whether for derivatives containing several types of substituents on tetrahydrofuran ring <86JMC1917, 86JST(142)387, 88JCC188, 88T429, 91T297, 91T1291, 92JST(265)225> or for derivatives in which the tetrahydrofuran ring is condensed with another cyclic system <86JST(142)383, 91CJC521, 91JOC6180, 92CAR(229)245, 92JOC5271, 93CAR(244)49>. MM2 has also been employed in reactivity studies, as in the nitrile oxide cycloaddition to unsaturated sugar <89JOC793>, and the intramolecular Diels–Alder furan reaction <92JA10738>. The results seem encouraging for further applications.

A new molecular mechanics force field termed MM3 has been developed that is better parametrized for carbohydrates <89JA8551, 90JA8293, 93CAR(244)49>. Among other force fields available, the consistent force field method (CFF) <79JST(57)215> has been adopted for energy calculation in the furanose derivatives <85JCS(P2)789, 89JCS(P2)209>.

The results from these theoretical methods are presented in the context of whatever physical property is under discussion in the following sections and compared with experimental evidence.

2.05.3 MOLECULAR STRUCTURE

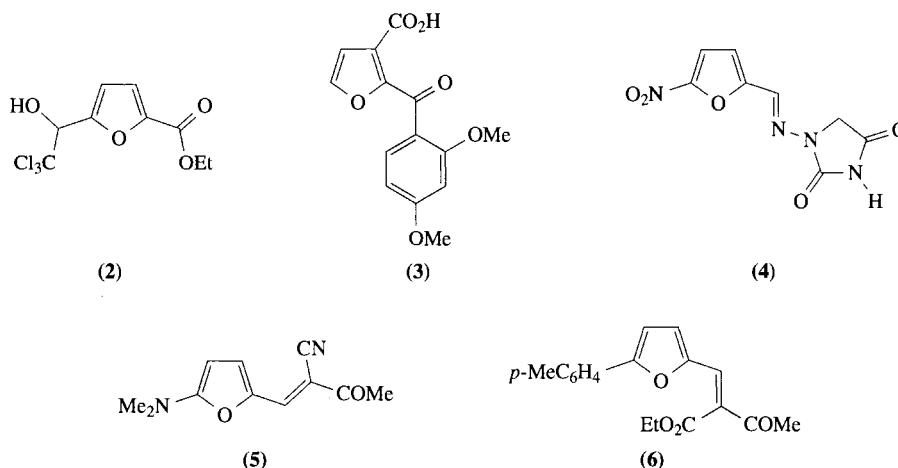
2.05.3.1 The Furan Nucleus

X-ray diffraction, electron diffraction methods, and microwave spectroscopy have provided abundant information about the structure of many furan derivatives. The bond lengths and angles determined for the same compounds by different methods are sometimes substantially different, although those obtained by microwave methods are accepted as the best now available. Furan presents this problem. The structural parameters of furan obtained by various techniques are assembled in Table 3. The major discrepancy is between the C—O and C=C distances, which are apparently almost equal in the gas phase, but differ by more than 4 ppm in the crystal. Attempts have been made to clarify this discrepancy: either the short C=C bond length is an artifact of the x-ray diffraction experiment <88JST(164)175> or the crystal structure of furan is affected by errors in measurements <89JST(187)161>. A structure consistent with data obtained by several techniques (gas-phase electron diffraction, rotational spectroscopy, and liquid-crystal NMR spectroscopy) has also been proposed <89JST(196)1>. Table 3 also collects bond lengths and angles of some furan derivatives. In all the derivatives reported, the furan ring remains strictly planar, while its dimensions can be affected by substituents, though usually only moderately. The dimensions of furfurylamine <90JST(223)33>, and furan-2-carbaldehyde <78JST(50)325> from microwave spectroscopy, and 2-furoyl

chloride <85JST(130)255> from electron spectroscopy very closely resemble those of unsubstituted furan determined using the same techniques. The furan ring seems lightly strained in the pseudo structure proposed for reproducing the ground rotational constants in 2-nitrofuran <92JST(269)97>. Microwave spectra of furan-3-carbaldehyde <92ACS923> and 3-furanmethanol <93ACS849> have also been investigated with assistance from *ab initio* computations at 6-31G* and MP2/6-31G* levels of theory. For these compounds, too, the dimensions of the furan part are estimated to be very close to those previously reported.

The data obtained in the condensed phase seem to exhibit the same trend. The short C=C bond distance in x-ray determinations for furan lengthens in substituted furans, while the other geometrical parameters range between typical values. In every case the C(3)—C(4) bond is always longer than the other C—C distances in the ring. Moreover, the bond angles may deviate from usual values while the substituents may be located out of the plane of the furan ring, as in furan-3,4-dicarboxylic acid <86ACS(A)559>. All of this can be attributed to strain. The strain is accommodated in several ways, while the furan ring itself remains strictly planar. In general, the furan nucleus is very resistant to bending.

Although it does not appear easy to rationalize systematic differences, the dimensions of the furan ring in a series of derivatives containing the same or a comparable substituent remain about the same. In the furancarboxylic acids (2) <93AX(C)558> and (3) <90AX(C)2487> (Table 3) the ring is always strictly planar with slight modifications in its dimensions. The greater variations apply to the C(3)—C(4) bond, which becomes longer compared with that in 2-furoic acid (141.1 pm) on introduction of a second substituent on the ring (142.8 pm in (2), 143.4 pm in (3), and 145.2 pm in 3,4-furandicarboxylic acid). A short C(3)—C(4) bond length of 139.7 pm has also been found by an x-ray diffraction study of nitrofurantoin (4) <93AX(C)741> (complete structural data for the furan ring are reported in Table 3). This bond returns to the usual length of 143.0 pm, the mean value of a set of several 2-nitronaphthofurans analyzed by x-ray diffraction (Table 3). Apart from this bond, the whole ring structure appears twisted: the bond angles close at O and C-3 atoms but open at C-2, while the other bond angles present the usual values. 2-Furylethylene derivatives (5) <89AX(C)533> and (6) <90AX(C)1558> present similar features: the C(3)—C(4) bond is very short (133.7 pm in (5) and 139.9 pm in (6)), but bond angle deformations are less than in nitro derivatives. In all these systems the furan ring always appears perfectly planar and the whole molecule is nearly planar. Thus there is some evidence here for extensive π -delocalization involving the furyl ring and substituents, as shown by the pattern of bond lengths and the planarity of the system.

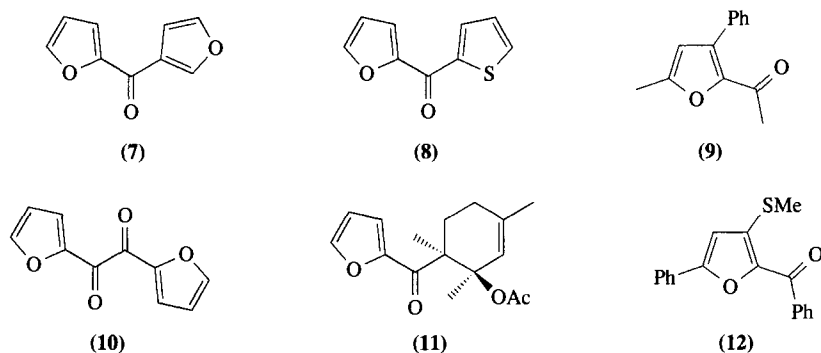


From x-ray diffraction studies of acyl derivatives ((7)–(12)), the dimensions of the furan ring appear very similar to one another. In order to have the highest degree of π -conjugation, the carbonyl group tends to reach the coplanar orientation with the furan ring while the other substituents (at the furan ring or on the carbonyl group) may deviate from the furan plane in order to accommodate steric effects.

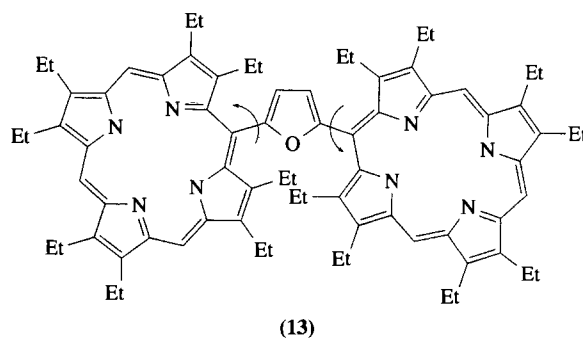
Evidence that the furan nucleus is not easily deformed or bent can be derived from the crystal structure of octaethylporphyrin (13) dimer bridged by a 2,5-disubstituted furan <93AJC1313>. Steric interaction of the furan ring with the flanking porphyrin substituents forces the ring planes to be almost orthogonal (porphyrin mean plane/furan plane dihedral angle 89.2° and 87.1°). Bond distances and angles in the furan ring and adjacent parts of the porphyrin rings are distorted owing to

Table 3 Experimental bond lengths and angles in substituted furans.

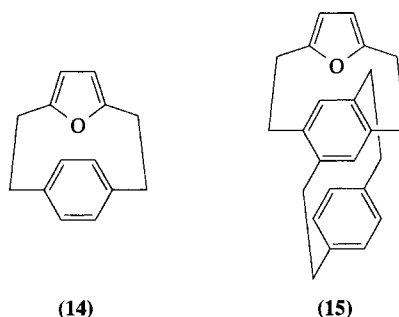
Substituent	Bond length (pm)					Bond angle (deg.)					Method	Reference
	O—C(2)	O—C(5)	C(2)—C(3)	C(3)—C(4)	C(4)—C(5)	1	2	3	4	5		
None	136.2		136.1	143.1		106.5	110.7	106.0				79MI 205-01
(See structure (1))	136.8		132.2	142.8		106.17	110.14	106.76			X-ray	72AX(B)2984
	136.2		136.1	143.0		106.56	110.65	106.07			Microwave	78JST(48)157
	136.6		132.0	141.4		106.0	110.1	106.9			X-ray	89JST(187)161
	136.4		136.4	143.0		106.74	110.49	106.14			Several	89JST(196)1
2-COOH	136.6	135.9	134.5	141.1	133.9	105.7	110.5	106.3	106.6	110.8	X-ray	83AX(C)1111
2-NO ₂	131.8	135.8	135.0	143.0	136.3	105.5	114.0	103.9		110.6	Microwave	92JST(269)97
2-CH ₂ NH ₂	136.2	136.2	136.1	143.0	136.1	106.55	110.65	106.07	106.07	110.66	Microwave	90JST(223)33
2-COCl	137.8		135.7	142.6		105.6	110.9	106.3			Electron diffraction	85JST(130)255
2-CHO	136.2		136.1	143.1		106.5	110.7	106.1			Electron diffraction	78JST(50)325
Structures (7) and (8)	137.2	136.3	134.4	141.6	132.8	105.8	109.4	107.2	106.0	111.3	X-ray	89JCS(P2)1741
3,4-COOH	135.8	135.9	136.4	145.2	136.4	107.6	110.6	105.6	105.8	110.5	X-ray	86ACS(A)559
3-SOCH ₃ , 4-I	136.0	135.0	135.0	142.0	134.0	107.4	109.6	106.1	106.7	110.2	X-ray	91JST(246)99
2I, 3-SOCH ₃	137.2	134.9	135.4	142.4	136.4	107.6	109.6	106.5	106.5	109.8	X-ray	91JST(246)99
Structure (9)	137.9	135.5	137.2	141.8	135.0	107.5	109.2	105.7	107.9	109.6	X-ray	92MI 205-02
Structure (4)	134.8	136.9	133.9	139.7	135.3	104.8	113.0	104.8	107.8	109.6	X-ray	93AX(C)741
2-NO ₂ averaged	136.0	138.0	134.0	143.0	139.0	104.0	115.0	105.0	106.0	111.0	X-ray	88EJH341
Structure (5)	141.2	134.4	138.1	137.7	138.2	105.4	107.8	108.3	105.7	110.9	X-ray	89AX(C)533
Structure (6)	137.4	137.1	135.7	139.9	139.5	107.3	108.9	107.6	107.2	109.0	X-ray	90AX(C)1558
Structure (10)	134.4	137.4	135.5	140.1	131.4	106.03	110.39	105.55	107.99	109.7	X-ray	87CPL(134)541
Structure (11)	137.1	135.9	134.6	141.2	132.3						X-ray	86AX(C)1797
Structure (12)	138.5	137.1	135.8	144.4	136.7	107.3	109.8	106.5	106.5	109.7	X-ray	89JHC439



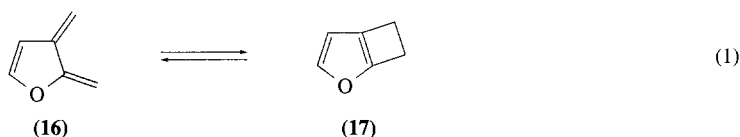
steric congestion involving the ethyl substituents, but the furan ring remains planar. The compression of the porphyrin centroids towards the furan oxygen, at the expense of the porphyrin distortion and angle strain at the C-2 and C-5 of the furan, is the noteworthy feature of this structure.



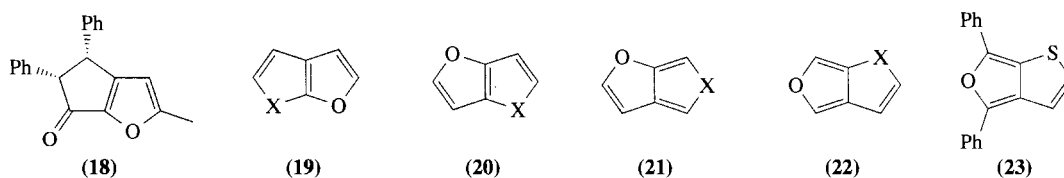
In contrast, in (2,5) furanophane (**14**) <84BCJ3552> and (**15**) <80AX(B)2276> the furan ring is slightly puckered. The furan ring takes an envelope form with the oxygen atom away from the benzene ring: the dihedral angle formed between the planes by the O, C-2, C-5 atoms and C-2, C-3, C-4, and C-5 is 3.3° in (**14**) and 3.8° (**15**). Other consequences of the strain can be seen in the opened angles of the methylene bridges, but the most noticeable effect is on the benzene ring. This ring adopts a symmetrical boat form in (**14**) with the carbon atoms linked to the bridge deviating by 13.8° and 14.8° from the plane defined by the other four carbon atoms of the ring. In (**15**) the benzene ring has a twisted boat form.



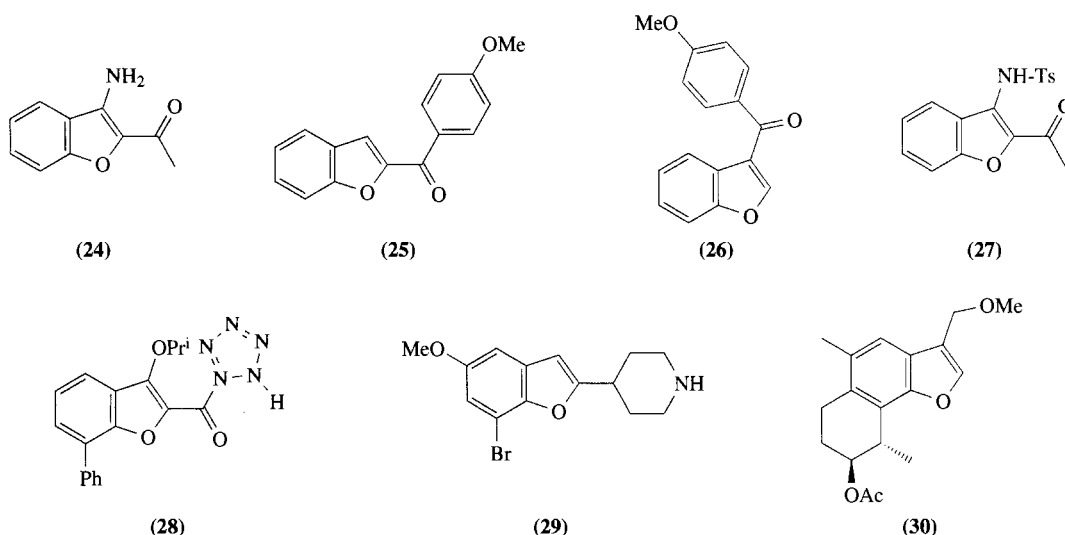
The dimensions of furan are such that its radial bonds are set at angles larger than those of benzene. These large angles clearly have a strong influence on the construction of annelated systems, which must be very strained. Studies of the ring closure reaction of (**16**) (Equation (1)), utilizing theoretical calculations (MNDO) <87AG(E)471>, show that compound (**17**) is thermodynamically unfavorable because of restricted mobility, high ring strain, and lack of resonance stability. The thermally inaccessible compound (**17**) has instead been generated photochemically from its thermodynamically more favorable isomeric form and identified by spectral methods (IR, PE, and UV/vis).



Very few structural data are available for systems containing the furan ring fused with a 5-membered ring. The x-ray structure of (18) is known <90AX(C)1129>. The two fused rings are coplanar and the dimensions of the furan ring are similar to those found for other nonannelated systems. The possible compounds containing two condensed pentatomic rings containing O, S, and N ((19)–(22)) have been investigated theoretically at MNDO level <87JCPB1147>. The [2,3-*b*] and [3,2-*b*]-isomers ((19)–(20)) are characterized by an “essentially double central bond,” and the [3,4-*b*]- and [3,4-*c*]-isomers ((21)–(22)) by an “essentially single central bond.” The [3,2-*b*]-isomer was found to be the most stable among furofurans and the [3,4-*b*]-isomer among furothiophenes. Furo[3,2-*b*]-pyrrole and pyrrolo[3,4-*b*]furan are practically isoenergetic compounds. An experimental x-ray structure for (23) is available <89CB1119>. The whole molecule is planar and the dimensions of the two 5-membered rings do not show any particular distortion with respect to the two unsubstituted moieties. The theoretical calculations at various levels of theory <87JCP1147, 89CB1119, 91CB2481> on the unsubstituted system correctly reproduce experimental features, except for the length of the central bond (experimental 139.7 pm; theoretical 144.0–147.0 pm according to the level used).



The dimensions of the furan ring in a set of benzo[*b*]furan derivatives ((24)–(30)) are reported in Table 4. In all of the compounds the benzo[*b*]furan nucleus always appears planar, with the dimensions of the furan moiety being rather similar to one another.

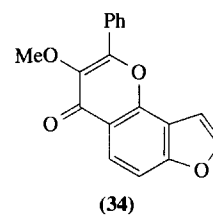
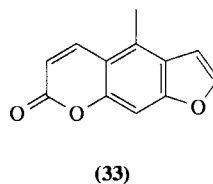
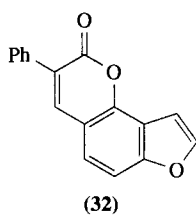
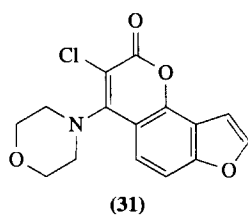


The location of substituents seems to influence the geometry of the furan ring only slightly, while the C(3a)—C(7a) bond is significantly longer in benzo-condensed derivatives as compared to furan (C(2)—C(3) and C(4)—C(5) in Table 3). The structure of 2-nitronaphthofurans determined by x-ray techniques <88EJM341> shows similar features (Table 3, 2-NO₂ averaged).

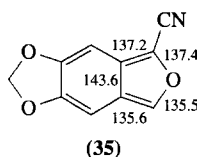
Annellation of benzo[*b*]furan with a pyran ring may lead to tricyclic systems ((31)–(34)) whose x-ray structures are available ((31) <91FES759>, (32) <88JHC407>, (33) <91AX(C)2144>, (34) <90AX(C)437>). In all these systems the benzo[*b*]furan moiety is planar with normal values for bond lengths and angles. The pyran ring in furocoumarins (31), (32), and bergaptol (33) is fairly planar, whereas in karanjin (34) it is distorted.

Table 4 Experimental bond lengths and angles in substituted benzo[*b*]furans.

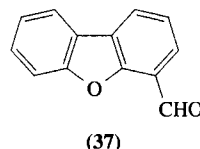
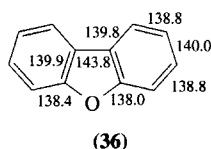
Compound	Bond length (pm)					Bond angle (deg.)					Method	References
	O—C(2)	O—C(7a)	C(2)—C(3)	C(3)—C(3a)	C(3a)—C(7a)	1	2	3	3a	7a		
(24)	140.3	136.0	137.8	144.7	139.9	105.3	111.6	105.6	105.7	111.8	X-ray	88AX(C)1449
(25)	138.0	137.4	134.5	142.3	138.8	105.9	110.8	107.6	105.3	110.3	X-ray	87JCS(P2)1443
(26)	136.2	136.9	135.7	144.8	138.4	105.7	112.5	105.5	105.3	111.0	X-ray	87JCS(P2)1443
(27)	138.1	136.9	137.4	142.9	140.0	106.0	110.7	107.1	105.0	111.2	X-ray	88AX(C)300
(28)	138.4	136.7	136.1	144.2	139.4	105.9	110.9	107.1	104.7	111.4	X-ray	91AX(C)611
(29)	139.3	136.5	133.3	144.1	138.1	104.7	112.3	106.2	105.6	111.2	X-ray	86BSB139
(30)	140.1	138.8	132.8	144.3	137.8	104.6	111.7	107.3	105.4	110.9	X-ray	86T4493



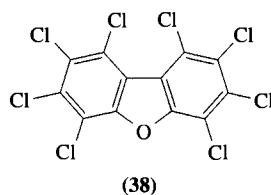
Benzo[*c*]furan is too unstable to be subjected to the usual techniques for measuring bond lengths and angles. Two stable benzo[*c*]furan derivatives have been synthesized and the crystal structure of one of these (35) is now available <86JOC3973>. The results indicate that (35) is an essentially planar molecule; only the carbon atom of the methylenedioxy group is slightly out of plane. Remarkably good agreement is found between the bond lengths of the furan moiety of (35) and the experimental values for furan itself (Table 3).



There is deviation of planarity between annulated rings in dibenzofuran. Crystalline dibenzofuran <84AX(C)1572> and dibenzofuran-*d*₈ <93AX(C)398> show disordered structures. The disordered molecules are related to the ordered molecules by 180° rotation about an axis passing through the molecular center of mass and perpendicular to the furan ring. The molecules are bisected by a mirror plane normal to the furan ring and containing the O atoms. Ordered molecules have the dimensions shown in Structure (36). The furan ring and the benzenoid ring are separately planar, but form a dihedral angle of 1.5° with one another. Similar features are present in the structure of 4-dibenzofurancarbaldehyde (37) <91AX(C)2134>, with a dihedral angle of 1.4°.

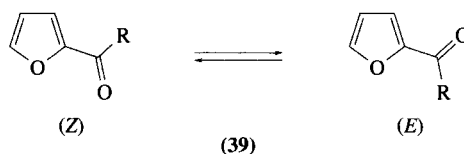


A number of physical properties of the polychlorinated dibenzofurans have been studied in order to better understand their formation and subsequent environmental fate. Several properties indicated unusual behavior when the 1- and 9-positions were halogenated. This behavior was thought to be due to steric crowding, causing the molecule to twist out of planarity to relieve the crowding. An x-ray diffraction study of octachlorodibenzofuran (38) is available <89MI 205-01>. The molecule does not maintain perfect planarity, having a dihedral angle between the aromatic rings of 0.2°, and serious crowding of the chlorines occurs. The benzenoid C—C bonds vary from 134.8 to 143.9 pm, while in nonsubstituted derivatives (36) they show a mean value of 139.2 pm and the C(9a)—C(9b) bond (diphenyl bond) is significantly longer (149.0 pm). Lengthening eased the crowding at 1- and 9-positions, with the essential planarity of the molecule being maintained by what is believed to be extensive π -electron delocalization.



2.05.3.2 Rotational Isomerism

The possibility for a substituent on the furan ring to assume preferential orientations with respect to the ring plane or to heterocyclic oxygen leads to rotational isomerism. A typical case is provided by the 2-acylfuran derivatives. The planar (*Z*)- and (*E*)-conformers (39) should represent the ground rotational state of these molecules with a rotational energy barrier between them. The transition state should correspond to the situation where the planes of the ring and the C=O bond are perpendicular.



A review has dealt with the conformations of acyl groups in heterocyclic compounds and one of the most studied compounds is furan-2-carbaldehyde <87AHC(41)75>. Studies of the conformational equilibrium in furan-2-carbaldehyde have employed different experimental and theoretical methods to determine conformational preferences together with the energy differences between the two planar conformers and the height of the rotational barrier. Table 5 summarizes the experimental data obtained by different techniques and in different phases. The molecule can exist in the two conformations (*E*) and (*Z*) and, while the (*E*)-conformer is the most stable in the gas phase, the (*Z*)-conformer is the one preferred in the liquid phase and is the only rotamer present in the solid state. This behavior has been attributed to the difference in polarity ((*E*) is the less polar conformer) and to the small difference in stability between the conformers (in the vapor phase $\Delta H^\circ = 0.818$ kcal mol⁻¹ (3.42 kJ mol⁻¹)). Hence the equilibrium is dependent on solvent polarity. A conformational analysis studying the variation in solvents with different polarity of the long-range coupling constants ($^nJ_{C,H}$ involving the formyl proton or carbon and the carbons or protons of the furan ring) confirms this behavior <87OMR(25)804>. The solvent effect on conformational equilibrium has also been studied theoretically. The experimental trend of solvent effects on the equilibrium and on activation energies is predicted correctly either using classical solvent effect theory together with *ab initio* MO calculations <87JCP(P2)961> or by introducing the solvent effect into MO calculations in the MNDO-MO method <88JST(168)265>.

Table 5 Relative free energy, conformer population, and free energy of activation for 2-carbonyl furan derivatives.

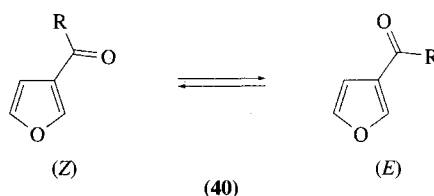
<i>R</i>	ΔG° (kcal mol ⁻¹) [kJ mol ⁻¹]	Population (% (<i>Z</i>))	ΔG^* (kcal mol ⁻¹) [kJ mol ⁻¹]	Method	Phase or solvent	Reference
H	$\Delta H^\circ = 0.818$ [3.42] $\Delta H^\circ = 1.07$ [4.48]	(<i>E</i>) > (<i>Z</i>) 100 (<i>Z</i>) > (<i>E</i>)	9.31 ((<i>E</i>) → (<i>Z</i>)) [38.95]	IR-Raman	Vapor	89SA(A)789
	0.692 (195 K) [2.89]	19.4 (245 K)	10.9 ((<i>E</i>) → (<i>Z</i>)) [45.6] (245 K)	¹³ C-T ₁ ⁻¹ H NMR	Et ₂ O/CD ₃ COCD ₃	87CCC541
		42 (295 K) 69 73 80		$^nJ_{C,H}$ NMR	Cyclohexane CDCl ₃ CD ₃ COCD ₃ DMSO- <i>d</i> ₆	87OMR(25)804
		90 (158 K)	10.3 ((<i>E</i>) → (<i>Z</i>)) [43.1] (158 K)	¹³ C NMR	Me ₂ O	85JSC(P2)1839
Me		79 (216 K)				
Et		47 (173 K)	9.3 (173 K) [38.9]			
Pr ⁱ		40 (173 K)	9.1 (173 K) [38.1]			
Pentan-3-yl		56 (173 K)	9.0 (173 K) [37.7]			
Bu ^t		77 (173 K)	8.6 (173 K) [36.0]			
Bu ⁱ		15 (134 K)	7.5 (134 K) [31.4]	DM, KC	CCl ₄	84JST(116)377
Cl		23 69.8 (359 K)		Electron diffraction	Gas phase	85JST(130)255

The effect of substituents on the conformational properties in 5-*R*-2-formylfurans (*R* = Me, Br, CN, NO₂, 4-NO₂C₆H₄) has also been studied by difference spectroscopy <89JST(197)193>. The ratios

of the conformers were determined from the solvent dependence of the IR spectra, and the energy differences of the conformers from the temperature dependence of liquid-state Raman spectra and ^1H NMR spectra, while the magnitudes of rotational barriers were calculated using the semiempirical CNDO/2 method. The result is that for all the compounds the (*E*)-form is more stable in nonpolar solvents and the (*Z*)-form is more stable in polar solvents and in the liquid phase. The (*Z*)-form is more stabilized by electron-donor groups than by electron-acceptor groups.

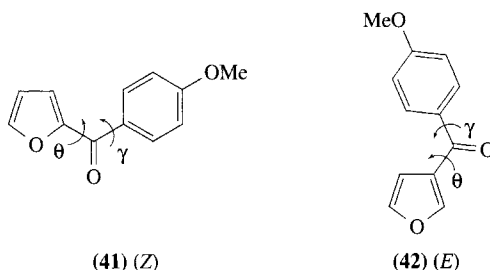
The situation in 2-acetylfuran ($\text{R} = \text{Me}$) qualitatively resembles that of the 2-formyl analogue with an equilibrium between the conformers. Accurate NMR measurements $\langle 85\text{JCS(P2)1839} \rangle$ at low temperature and in dimethyl ether solution performed on 2-acyl derivatives of furan have enabled direct detection of the amount of the two conformers. At 173 K the (*E*)-conformer population for 2-acetylfuran amounts to 53%. The percentage of (*E*)-conformer as a function of the increase of the alkyl group has also been determined and is reported in Table 5 $\langle 85\text{JCS(P2)1839} \rangle$. This increase does not impose a regular trend on the conformer ratio; the change in polarity seems to be more important than steric effect in determining conformational behavior. The results of dipole moment and Kerr constant measurements indicated that 23% of 2-pivaloylfuran exists as a nonplanar (*E*)-conformer in CCl_4 $\langle 84\text{JST(116)377} \rangle$. The angle of twist of the carbonyl group from furan ring should be small ($15^\circ \pm 15^\circ$). In the electronic absorption and phosphorescence spectra of 2-acetylfuran it has been found that the (*E*)-conformer is the major species in nonpolar media, while the (*Z*)-conformer is almost the predominant one in a polar medium $\langle 92\text{BCJ2067} \rangle$. The presence of rotational isomers has also been confirmed by the relatively broad vibrational fine structure observed in photoelectronic spectra of 2-carbonyl furans ($\text{R} = \text{H}, \text{Me}, \text{F}, \text{Cl}, \text{Br}, \text{OH}$) $\langle 90\text{CJC747} \rangle$. Electron diffraction study of the molecular structure and conformation of gaseous 2-furoyl chloride determined that at 359 K 69.8% was the (*E*)-conformer $\langle 85\text{JST(130)255} \rangle$. In 2-acetyl-5-methyl-3-phenylfuran (**9**) the molecular structure determined by x-ray methods for solid state and by NMR lanthanide-induced shift (LIS) measurements in solution indicates that it possesses a transoid structure of (*E*)-type $\langle 92\text{MI 205-02} \rangle$.

The situation appears different in furan-3-carbaldehyde (**40**). Thus, while in furan-2-carbaldehyde the equilibrium between conformers is rather sensitive to external effects, analysis of long-range stereospecific NMR coupling constants $^3J_{\text{C,H}}$, in solvents of different polarity, shows a very small solvent dependence of the conformer ratio for furan-3-carbaldehyde. The less polar (*E*)-conformer predominates in the equilibrium mixture and increases slightly with increasing solvent polarity. This trend has also been confirmed by similar analysis performed on the corresponding 4-iodo derivative, where, however, the (*Z*)-conformer is present to a larger extent $\langle 87\text{OMR(25)804} \rangle$. Theoretical calculations at *ab initio* level $\langle 88\text{JCS(P2)1501} \rangle$, employing a 6-31G* basis set, predict the (*E*)-conformer to be more stable than the (*Z*)-one by $0.75 \text{ kcal mol}^{-1}$ (3.14 kJ mol^{-1}). The relative stability of these conformers has been attributed to a balance between conjugative interaction between the formyl group and the ring and local electrostatic interactions between atoms of the formyl group and those of the ring. Introduction of solvent effect in the calculations has also confirmed the experimental trend of stability. Microwave spectrum, gas-phase IR spectrum and dipole moment measurements have led to the same conclusions regarding conformational equilibrium $\langle 92\text{ACS923} \rangle$. The introduction of correlation effects in the *ab initio* calculations has not improved the calculated energy difference between conformers. Nuclear Overhauser effect (NOE) measurements, combined with MM2 calculation, on the same substrate $\langle 91\text{T6427} \rangle$ have predicted that the proportion of (*Z*)-conformer is high enough (14% at room temperature) to give line broadening effects due to (*E*)-(Z) exchange. Carbon-13 NMR signals at variable temperature yielded a free energy of activation of $8.3 \text{ kcal mol}^{-1}$ (34.7 kJ mol^{-1}). ESR spectra show that the (*E*)-conformation is also adopted by the σ -radical obtained by H-abstraction from the moiety of furan-3-carbaldehyde $\langle 91\text{T6427} \rangle$.

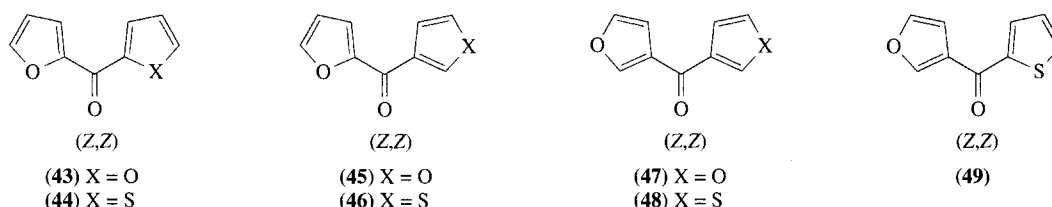


The conformational properties of 2- and 3-anisoyl derivatives of furan ((**41**)-(42)) have been investigated in the solid state and in solution $\langle 87\text{JCS(P2)1443} \rangle$. In the solid state the rings are distorted with respect to the carbonyl plane; the angle of twist in the 2-substituted compound is higher for *p*-anisoyl than for the heterocyclic ring ($\theta = 160.6^\circ$, $\gamma = 158.4^\circ$ (**41**)), while an almost equal degree of

twist is observed in 3-substituted compounds ($\theta = 152.1^\circ$, $\gamma = 151.4^\circ$ (**42**)). The orientation of the carbonyl and furan ring is of the (*E*) type. Analysis in solution has been performed with the NMR LIS method (^1H and ^{13}C). The formation by (**41**) of a chelate complex with the lanthanide alters the conformational information obtained by the LIS method, but analysis of the NMR chemical shifts indicates that a conformational equilibrium between the (*Z*)- and (*E*)- forms, close to that of the corresponding aldehydes, should be present. For (**42**) the (*E*)- form is the preferred one, and the exclusive conformation in solution, while the rings are distorted with an angle of twist of the heterocyclic and phenyl rings with respect to the carbonyl plane near to 30° . Theoretical calculations at *ab initio* level employing a minimal basis set yield very small differences in stabilities between the two conformers, (*E*)- and (*Z*)-, which are not fully planar and have a certain degree of twist with respect to the carbonyl plane for both rings.



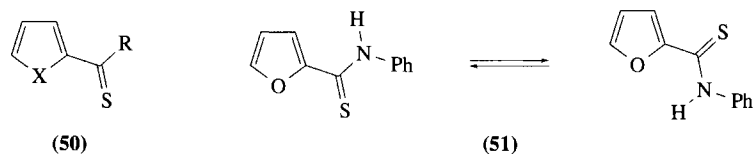
Conformational preferences of difuryl and furyl thienyl ketones ((**43**)–(**49**)) have been studied in the solid state and in solution employing several techniques (x-ray, NMR LIS, and *ab initio* calculation) <89JCS(P2)1741>. MO *ab initio* calculations, employing the 3-21G or 3-21G* basis set for the ground-state conformers of all the molecules considered, show that the solid-state structures are very similar to those calculated for the conformers with the lowest energy content, and the calculated conformer populations are in qualitative agreement with measurements in solution. As a general trend, the solid-state structures, the preferred conformations found in solution, and those calculated for the free molecule show that the preferred orientation of one heterocyclic ring with respect to the carbonyl group is of the type found in the corresponding formyl and acetyl derivatives and is maintained in all the diheteroaryl ketones. For the molecules having conformers of similar energy content and different dipole moment, it was demonstrated on the basis of the classical theory of the solvent effect that the equilibrium mixture of conformers can change in solvents of different polarity.



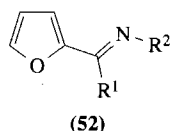
The radical anions of (**43**) and (**44**) have been examined by electron spin resonance spectroscopy <86JCS(P2)1851>. The ESR spectrum of the radical anion of (**43**) has been interpreted in terms of the presence of both (*E,E*)- and (*Z,E*)-conformers and the activation parameters for the interconversion have been determined.

Studies on conformational behavior of different C=X substituents on the furan ring have also been carried out. Closely related to acylfuran derivatives are C=S derivatives, for which little experimental evidence is available. The ^{13}C chemical shifts of the C=S and C=O groups in a series of heterocyclic derivatives (**50**) are found to be linearly dependent only for compounds with similar structure <85RTC9>. The (*Z*)-orientation is preferred as a general rule, but the angle of twist of the C=S plane relative to the heterocyclic or phenyl ring is usually higher than for the corresponding ketones, owing to the larger steric requirements of sulfur <85RTC9, 87AHC(41)75>. A conformational study on compounds (**51**) using various techniques (UV, IR, ^1H NMR, dipole moments, and CNDO/2 calculations) supplies evidence for a nonplanar structure and for free rotation of the thioamide group around the C—C bond. The value of the dipole moment in carbon tetrachloride and in benzene indicates an equal population of both conformers <89BSB327>. Nothing is known about furanthioaldehyde, but an ESR study on rotational conformers of furan-2- and 3-thioaldehyde radical anions, in comparison with the corresponding aldehyde radical anions, is now available

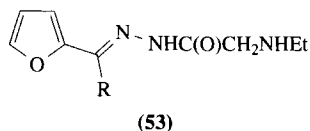
<91JOC6337>. In the radical anions of furan having the CHO or CHS moiety in position 2, it has been found that the two rotational conformers (*E*)- and (*Z*)- are present at the equilibrium with the (*Z*)-conformer always preferred. The aldehyde radical anion exhibits the same conformational preferences as the corresponding furan-2-carbaldehyde. For 3-derivatives it has therefore been proposed that for the radical anion the (*E*)-conformer should correspond to the more stable conformer, by analogy with the corresponding aldehydes.



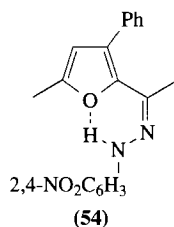
The imines derived from furan-2-carbaldehyde and 2-acetylfuran show the problem of (*E*)-, (*Z*)-isomers relative to the C=N bond. A ^1H and ^{13}C NMR study on some *N*-alkylimines (**52**) has been performed <87SA(A)1055>. NMR spectra show that these imines exist only as a single (*E*)- form. The same conclusion has arisen from analysis of electronic absorption spectra of related compounds ($\text{R}^1 = \text{H}$, $\text{R}^2 = \text{Me}$) with the aid of molecular orbital calculations <84BCJ844>.



For hydrazone derivatives (**53**), ^1H NMR spectra measured in solvents of different polarity indicate a nearly equal amount of (*E*)- and (*Z*)-isomers relative to the C=N bond <82OMR(20)26>. The activation parameters of the thermal isomerization process were measured, and the results are in line with a lateral-shift type mechanism, also supported by theoretical calculations on a model compound.

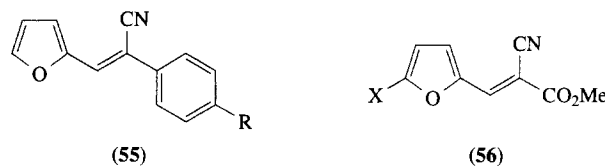


In 2,4-dinitrophenylhydrazones (**54**), in contrast, the (*E*)-conformer is predominant (70%) and its stability has been explained by the possible formation of an intramolecular hydrogen bond between the N—H proton and the furanic oxygen atom <92MI 205-02>. The presence of an intramolecular hydrogen bond has also been supported by the crystal structure of ethyl α -(*p*-tolylhydrazono)-2-furopropionate <88AX(C)1252>.

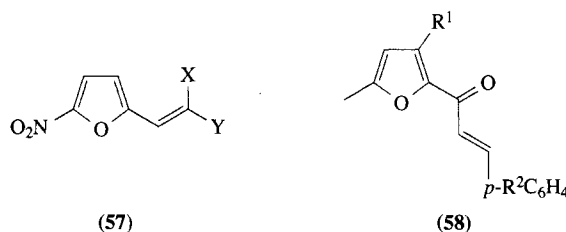


The possibility of (*Z*)/(*E*) isomerism around a double bond is also present in furylethylene derivatives. For acrylonitrile derivatives (**55**), ^1H NMR, UV, and IR spectra indicate that the (*E*)-isomer is the preferred conformation in solution <86JHC1747>. ^{13}C NMR chemical shifts and $^3J_{\text{C,H}}$ measured for 5-substituted derivatives (**56**) <90ZC99> confirmed that these molecules assume the (*E*)-conformation. They have also shown that in the reaction of 5-dimethylaminofuran-2-carbaldehyde

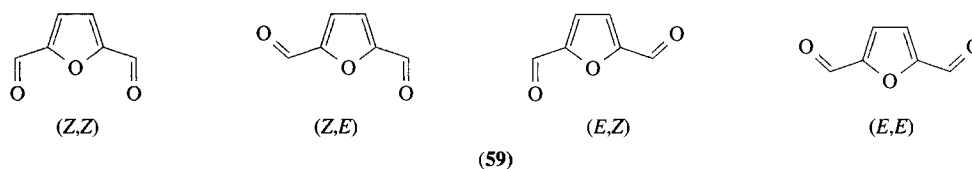
with the methyl ester of cyanoacetic acid only the (*E*)-isomer is produced <89AX(C)533>. The crystal structure of this derivative was shown as (5).



The possible conformation in 5-nitro-2-furylethylenes (57) has been studied through ^1H and ^{13}C NMR spectral data <86CCC889>. Geometric arrangements ((*E*)- or (*Z*)-) of the trisubstituted ethylenes were adduced from vicinal coupling constants $^3J_{\text{C,H}}$ for the carbon atom at the functional group attached to the double bond and the ethylene proton. For preferential orientation of the furan ring toward the double bond of the side chain, it was found that the preferred conformation (*s-cis* or *s-trans*) is dependent on the substituent on the double bond. Furanic analogues of chalcones (58) can exist in several conformational situations depending on the mutual orientation of the carbonyl group, the oxygen atom of the furan ring, and the double bond. The structure of (58) has been determined by IR, UV, ^1H NMR, and ^{13}C spectroscopy <89RRC1131> and the preferred conformation is that shown.

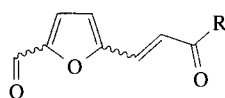


The presence on the furan ring of several substituents, each of which can give rise to rotational isomerism, increases the number of possible conformations. Furan-2,5-dicarbaldehyde represents a classic example. In principle four conformations are possible for this compound (59). Analysis of the electric dipole moments of furan-2,5-dicarbaldehyde in solvents of low dielectric permittivity enabled the relative populations of the conformers to be determined <89JST(196)227>. Experimental data suggest that the compound exists as a mixture of mixed (*E,Z*)/(*Z,E*)- and (*E,E*)-conformers with (*E,Z*) + (*Z,E*) = 0.68 and 0.96, (*E,E*) = 0.32 and 0.04, according to the solvent. Molecular orbital AM1-calculated enthalpies of all the conformers confirm the presence of the mixtures with populations of (*E,Z*) + (*Z,E*) = 0.38 and (*E,E*) = 0.60. Another conformational analysis of the same derivative, obtained by an experimental dipole moment determination combined with theoretical MNDO calculations <89H(29)657>, indicates the presence of a conformational equilibrium in which the (*E,Z*)-conformation has the biggest contribution, and that the two aldehyde groups are not equivalent. Experimental confirmation has been performed for two model reactions: mono-protection with MeOH/TsOH and aldolic condensation.

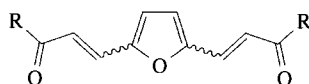


In 2,5-disubstituted derivatives (60) and (61), the number of possible conformations amount to 8 for (60) and 10 for (61). Conformational analysis of these derivatives has been carried out employing experimental dipole moment determinations together with semiempirical calculations (MNDO and AM1) <92JST(253)179>. This indicates that compounds (60; R = Me, OMe) and (61; R = Me) exist as an equilibrium mixture of the possible conformers with different populations, while (61; R = OMe) exists in only one conformation (62).

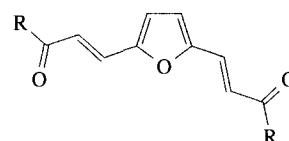
For 2-phenylfuran, electronic absorption spectra <84BCJ844> suggest a planar or nearly planar conformation as for 2,5-diphenylfuran, while for 2,4-diphenylfuran it should be more distorted than that of the monophenyl derivative. A theoretical analysis (*ab initio* level using a minimal basis set)



(60)

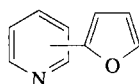


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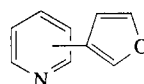


(62)

of conformational behavior <88G753> has determined the planar conformation for 2-phenylfuran and a *gauche* conformation for 3-phenylfurans at the absolute minimum of the potential energy curve. For furylpyridines (63) and (64), the analysis of calculated and experimental dipole moments <83CJC334>, together with UV spectral data and acidity constants, indicate a *trans*-planar geometry as the preferred conformation for 2-(2-furyl)pyridine. The calculated dipole moments for 4-(2-furyl)pyridine and 4-(3-furyl)pyridine are independent of the angle between the planes of the two rings and consistent with the experimentally determined values. For the other isomers of the series the two rings should not be coplanar.

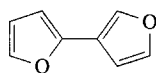


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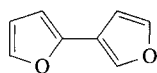


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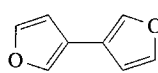
Nonfused biheterocycles like bifurans have become a very interesting subject of study because of their close relation to organic polymers derived from 5-membered aromatic heterocycles (e.g., polyfurans and polythiophenes). Hence, the knowledge of the structure and electronic properties of the dimers can be of great interest as a first approach to those of the corresponding polymers. A theoretical *ab initio* approach to molecular conformation of bifuran ((65)–(67)) and furylpyrroles ((68)–(71)) <87JPC545> is available. Results of calculations in the minimal basis set indicate that for all derivatives the conformational minima corresponding to a planar conformation of the two rings are those shown as structures (65) to (71). For 2,2'-derivatives the conformation of the two rings is opposite: 2,2'-bifuran assumes a *trans* conformation, 2,2'-furylpyrrole a *cis* conformation. Augmentation of the basis set does not change the conformational preferences and the relative stabilities of conformers, except for (67), (69), and (71), for which it determines a *gauche* out-of-plane equilibrium conformation. Although there is no experimental evidence for the geometric structure of (65), the anticoplanar conformation is in agreement with solid-state geometries obtained from x-ray diffraction data for 2,2'-bithiophene <68AX(B)467> and has been commonly accepted as a starting point for analysis of the structure and electronic properties of polyfuran <88JCP(88)386, 88SM109, 90JST(219)397, 91JCS(F2)3009, 91JPC9746, 91MI 205-01, 91SM789, 93JCP(98)769, 93SM(57)4467>. A coplanar structure has been found to be the most stable conformation for unit cells for poly(furylene vinylene) (72) <92JST(254)243, 93CPH(175)283> and phenylene-furan copolymers (73) <92MM3591, 93MM2095>.



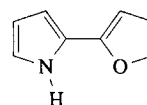
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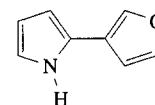
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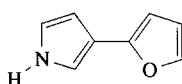
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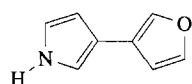
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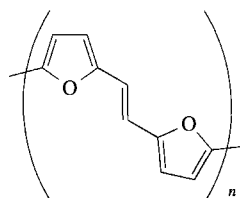
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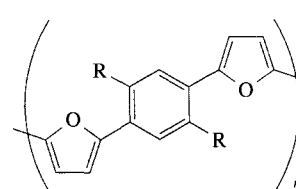
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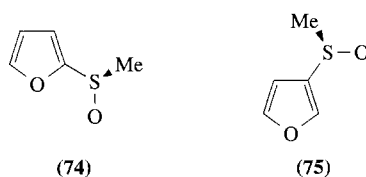
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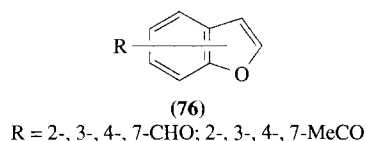
(73)

Methylsulfinyl derivatives of furan show a rotational isomerism similar to that observed in acyl derivatives. For 2-methylsulfinyl furan (74) and 3-methylsulfinyl furan (75), theoretical calculations at *ab initio* level <91JST(228)71> indicate two conformational minima corresponding to the two orientations of the S—O bond with respect to the heterocyclic oxygen (labeled O,O-*cis* and O,O-

trans) with small differences in energy content. The O,O-*trans* planar form is the most stable conformer for the 3-derivative, while for the 2-derivative the most stable conformer corresponds to an O,O-*cis* form highly twisted ($\text{O—C—S—O} = 62.8^\circ$). An approximate prediction of the behavior of conformational equilibrium for these molecules in solution has been attempted in the framework of the reaction field theory. For (74) an increase in solvent polarity seems to cause stabilization of a third conformer of the O,O-*cis* type with the S—O bond twisted on the opposite side of the ring plane ($\text{O—C—S—O} \cong 60^\circ$). These compounds, together with their derivatives substituted on the furan ring, have also been studied in solid state (x-ray diffraction) $\langle 91\text{JST}(246)99 \rangle$ and with various NMR techniques in solution, namely, ^1H , ^{13}C , ^{17}O chemical shifts $\langle 91\text{JST}(246)81 \rangle$, stereospecific long-range coupling constants $^nJ_{\text{C,H}}$, LIS, nuclear Overhauser effect, and spin-lattice relaxation times (T_1) $\langle 91\text{JCS}(P2)269 \rangle$. Experimental results consistently support theoretical predictions of the conformational behavior of these compounds.



The conformational behaviors of formyl and acyl derivatives of benzo[*b*]furan are also available. Conformational analysis on (76) was carried out by NMR methods, using ^1H , ^{13}C , long-range proton-proton coupling constants, and LIS simulation $\langle 84\text{JCS}(P2)1479 \rangle$. For 2- and 7-derivatives the results of this study provide evidence of solvent-dependent (*E*), (*Z*)-conformational mixtures, with the (*Z*)-form predominating in solvents with higher polarity. The presence of the same substituents in the 3- and 4-positions gives compounds with predominantly (*Z*)-conformation, and no change is caused by solvents. From comparison of the ^1H chemical shifts (in various solvents) of the 2-trifluoroacetylbenzo[*b*]furan and those of the corresponding 2-acetyl derivative, and on the basis of the empirical interpretation of the experimentally measured $^5J_{\text{H,F}}$ coupling constant, a predominantly (*Z*)-conformation has been tentatively assigned to these derivatives $\langle 84\text{OMR}(22)197 \rangle$. The conformational properties of *p*-methoxybenzoylbenzo[*b*]furans ((25), (26)) were studied in the solid state (x-ray analysis) and in solution (^1H , ^{13}C , and LIS simulation) $\langle 87\text{JCS}(P2)351, 87\text{JCS}(P2)1443 \rangle$. In the solid state the benzo[*b*]furan and phenyl rings are distorted with respect to the carbonyl plane. The orientation of the carbonyl and heterocyclic oxygens is of (*E*)- type in (26) and (*Z*)- type in (25), where packing forces provide a probable explanation for the adoption of the more polar (*Z*)-conformation. In solution, LIS simulations for (26) show a distorted form, with the two rings twisted respectively inward and outward with respect to the carbonyl plane by 30° . For (25), LIS simulation is not representative because of the formation of a chelate complex with a lanthanide atom; nevertheless, the NMR chemical shifts indicate that an equilibrium between (*E*)- and (*Z*)-forms should be present. The distortion from coplanarity of the rings with the carbonyl groups has been attributed to the steric effect.



2.05.3.3 Structure and Conformation in Hydrofurans

The structural properties of tetrahydrofuran have long been investigated with an eye to its important role as a structural unit of carbohydrates and biological molecules. Accurate bond lengths and angles have not easily been obtained because the ring is flexible and undergoes free pseudorotation. Table 6 lists the experimental bond lengths and angles together with the puckering amplitude q , defined by $q^2 = \sum z_i^2$, where the z_i are the Cartesian displacements of the atoms of the ring from their mean plane $\langle 75\text{JA}1354 \rangle$. As a proof of the molecular pliability along the angular coordinate, only three principal bond lengths (C—O, C—C, and C—H) have been obtained from electron diffraction structural analysis, and the radial distribution could not be resolved in

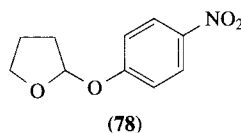
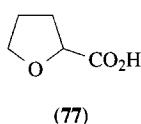
terms of any static conformation $\langle 69\text{ACS}2748, 69\text{T}3045 \rangle$. A symmetric twisted conformation has been demonstrated for the ground state of the molecule by a single-crystal x-ray structure analysis $\langle 83\text{AG(E)}410 \rangle$, and a more detailed structure analysis, performed for deuterated species by high-resolution neutron powder diffraction (HRNPD), has confirmed the C_2 conformation of solid tetrahydrofuran $\langle 92\text{AX(C)}301 \rangle$. Experimental estimates of q cover a fairly wide range of values, the lower limit being found for the solid phase. Analysis of the ^1H NMR spectrum in the nematic phase, jointly with the determination of the pseudorotation potential using a theoretical model (ELS), has determined an equilibrium puckering amplitude of 39 pm $\langle 90\text{MP}429 \rangle$, very close to the value of 38 pm determined by electron diffraction $\langle 69\text{T}3045 \rangle$.

Table 6 Experimental bond lengths, angles, and puckering amplitude (q) for tetrahydrofuran (C_2).

Bond length (pm)			Bond angle (deg)			q (pm)	Method	References
$O(1)-C(2)$	$C(2)-C(3)$	$C-H$	1	2	3			
142.8	153.6	111.5	110.5	106.5	101.8	38.0	Electron diffraction	69T3405
142.8	153.8	111.0	111.6	105.6	101.9	36.5	Electron diffraction	69ACS2748
143.5	153.1	109.0	109.8	106.7	101.9	35.5	X-ray 148 K	83AG(E)410
143.8	151.6	109.7 ^a	109.9	106.4	102.6	34.3	HRNPD ^b 5 K	92AX(C)301

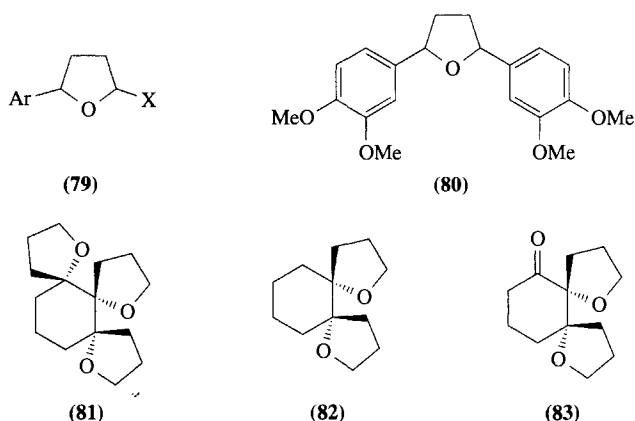
^aMean value. ^bHigh-resolution neutron powder diffraction.

Substituents on the ring have small influence on pseudorotation but may introduce conformational preferences. The dimensions of the ring in (*R*)-tetrahydro-2-furancarboxylic acid monohydrate (**77**), determined by the x-ray diffraction $\langle 90\text{AX(C)}106 \rangle$, are similar to those of tetrahydrofuran $\langle 83\text{AG(E)}410 \rangle$, but the torsion angles indicate that the twofold symmetry of that compound no longer exists in (**77**). Likewise in (**78**) the tetrahydrofuran adopts a distorted envelope conformation $\langle 89\text{AX(C)}239 \rangle$. A model has been proposed which assumes that the pseudorotational potential in 5-membered rings is given by the combination of contributions from the unsubstituted ring, from the individual substituents, and from interactions between pairs of substituents $\langle 88\text{JCC}188 \rangle$. The application of this model to the potential calculated by MM2 force field for *cis*- and *trans*-2,5-dimethoxytetrahydrofuran, together with a pseudorotational analysis by vicinal proton spin-spin coupling constants $^3J_{\text{H,H}}$, explains the conformational behavior of these compounds.



The substituents reveal their control on the conformational preferences of the tetrahydrofuran moiety by contributions of different steric and stereoelectronic effects. Conformational analysis on a series of aryl tetrahydrofuran derivatives in solution (^1H NMR spectra) and in solid state (x-ray), jointly with MM2 calculations of the pseudorotational potential $\langle 88\text{T}429, 88\text{T}2203, 89\text{T}3371 \rangle$, has determined the conformational preferences in these substrates. In fact, none of the studied molecules is rigid. The tetrahydrofuran ring may exhibit two or only one conformation of minimum energy. For instance, in (**79**) these conformations of minimum energy comply with the tendency of the aryl group to be equatorial and the polar X group to be axial $\langle 89\text{T}3371 \rangle$. These two effects (steric for aryl and anomeric stereoelectronic for X) are strongly opposed, but neither prevails. The equatorial preferences of the aryl group have also been found in the x-ray crystal structure of *cis* and *trans* isomers of (**80**) $\langle 86\text{JMC}1917 \rangle$. A detailed analysis of variable-temperature NMR (by means of 2D measurements) and crystal structure data $\langle 92\text{JOC}3947 \rangle$ has instead shown that the stereoelectronic *gauche* effect is of major importance in dictating the major conformation adopted in a series of spirocyclic tetrahydrofurans (**81**)–(**83**).

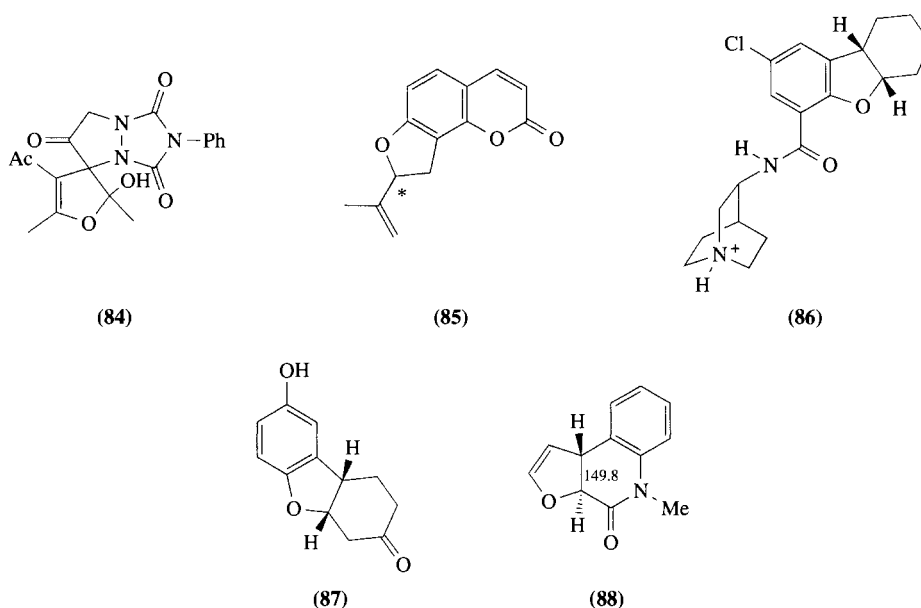
Furanose rings possess considerable conformational flexibility. The configurations assumed by the rings are important stereochemical characteristics of the three-dimensional structure of DNA and RNA molecules. Statistical analysis of x-ray data has determined the probabilities of alternative conformations of the furanose ring in nucleosides and nucleotides and the free-energy difference $\langle 87\text{AX(C)}275 \rangle$. However, steric effects alone cannot entirely account for the conformational behavior of various furanose moieties in (modified) nucleosides, and the *gauche* and anomeric effects are



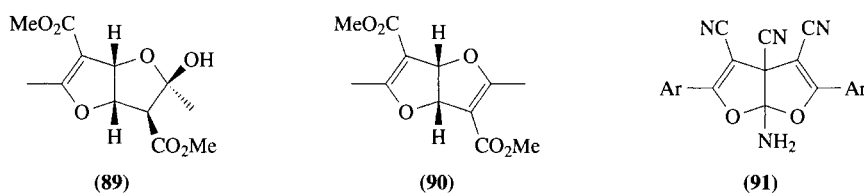
employed for this purpose. The manner in which the *gauche* and anomeric effects may drive the pseudorotational equilibrium in the pentafuranose moiety of nucleosides has been examined by ^1H NMR spectra $\langle 93\text{JA}9734 \rangle$. A generalized Karplus equation linking $^3J_{\text{H,H}}$ coupling constants to the corresponding proton–proton torsion angles between two parameters A and B has been used. The two correlation parameters have been derived from x-ray structural data and from theoretical *ab initio* calculations. The values of ΔH° and ΔS° for the pseudorotational equilibrium have also been derived and ΔH° dissected into various stereoelectronic effects (*gauche* versus anomeric) of exocyclic substituents on the pentafuranose moiety.

The 2,3-dihydrofuran ring is nonplanar with the C-2 methylene group out of the plane of the other ring atoms. Microwave spectra $\langle 86\text{JST}(147)255 \rangle$ support such a structure and indicate a barrier to ring inversion of 1.12 kJ mol^{-1} . From the ground-state inertial data a dihedral angle of 17° is obtained. Another one-dimensional ring-puckering potential function, obtained using previously determined experimental data and molecular structures from molecular mechanics (MM2) for calculating accurate kinetic (inverse reduced mass) functions, gives an equilibrium value of 21.5° for the dihedral angle $\langle 87\text{JSP}(124)369 \rangle$. Similar values for the ring-puckering potential (1.17 kJ mol^{-1}) and for the dihedral angle (20°) have also been obtained from microwave spectra of 2-methyl-4,5-dihydrofuran $\langle 85\text{JSP}(112)26 \rangle$. The ring is flexible and tends to remain distorted in the solid state also. In *spiro*-dihydrofuran-pyrazolidinones (**84**), x-ray structure reveals that the 5-membered rings deviate very slightly from planarity $\langle 89\text{T}7929 \rangle$. The preferred conformation for the three pentatomic rings is a twisted envelope with pseudo- C_2 symmetry. The corresponding axes pass through N-2 for the triazole and pyrazole and C-2 for the furan ring. The furan ring appears to be the most puckered among the three heterocyclic rings. In the furanocoumarin derivative (**85**), the coumarin moiety is planar while the furan ring has a C^* -envelope conformation with a maximum value for the dihedral angle of -13.76° $\langle 93\text{MI} 205-01 \rangle$. Crystal structures of hexahydrobenzofuran derivatives (**86**) exhibit normal values of bond lengths and angles $\langle 93\text{AX}(C)1551 \rangle$. The benzene ring is planar, while the dihydrofuran ring is distorted, with a torsion angle of 31.9° between the furanoic oxygen atom and the marked carbon atom common to benzene and furan rings. The dihydrofuran/cyclohexane ring fusion is *cis* (axial and equatorial) and the cyclohexane is only slightly distorted from perfect chair geometry by this linkage. The same type of ring junction has been found in derivative (**87**), but the cyclohexanone ring assumes a twist-boat conformation $\langle 91\text{JOC}6180 \rangle$. The x-ray crystal structure of the product formed by reduction of (**87**) indicated that the cyclohexane ring adopted a chair conformation with an equatorial hydroxyl group. Derivative (**88**), obtained by photocyclization of furan-2-carboxanilide, presents a very short bond (149.8 pm) in the dihydrofuran ring $\langle 87\text{JOC}3178 \rangle$ and a torsional angle associated with the H atoms of the dihydrofuran ring of 160° . These two protons show very large ^1H NMR spectra of the vicinal coupling constants and this has been attributed to the short bond distance.

Little is known about compounds containing two fused furan rings. The structure of furo[3,2-*b*]furan derivatives (**89**), and in particular the stereochemistry, has been assigned by ^1H and ^{13}C NMR and mass spectra and confirmed by single-crystal x-ray analysis $\langle 88\text{JOC}2807 \rangle$. Acid-catalyzed dehydration of (**89**) affords furo[3,2-*b*]-(**90**) in small percentage. The assignment of the structure of (**90**) has been based upon its origin and particularly its remarkably simple NMR spectra, which showed three singlets in the proton spectrum and six singlets in the proton-decoupled carbon spectrum $\langle 88\text{JOC}2807 \rangle$. Similarly, NMR spectra (^1H and ^{13}C) of (**91**) suggest perfect geometric



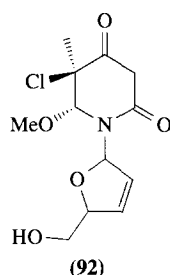
symmetry in solution, while in the solid state $\langle 88\text{JOC}5341 \rangle$ the molecule does not present a pseudo-mirror plane: each half is nearly situated in a plane, the dihedral angle being about 119.5° , and both 5-membered rings are planar. However, while in the right-hand moiety the furan and phenyl rings and the nitrile group are nearly planar, in the other moiety the same groups are farther from planarity. This non-symmetric molecular geometry in the solid state is probably a requirement of the molecular arrangement in the crystal.



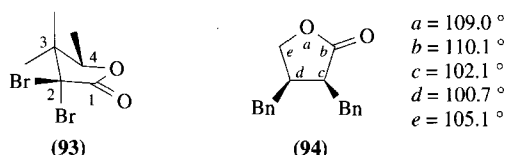
The molecular structure of 2,5-dihydrofuran has been determined both by gas-phase electron diffraction $\langle 84\text{JA}20 \rangle$ and by microwave spectroscopy $\langle 87\text{CPH}(115)103, 93\text{JSP}(160)158 \rangle$. The ring equilibrium configuration has been established as strictly planar. Theoretical calculations employing different basis sets $\langle 93\text{JSP}(160)158 \rangle$, and molecular mechanics (MM2) $\langle 90\text{JPC}1830 \rangle$, also predict the planar structure with C_{2v} symmetry as the equilibrium conformation. The ring-puckering motion of 2,5-dihydrofuran has been studied in detail by far-infrared spectroscopy $\langle 84\text{JA}20 \rangle$ and microwave spectroscopy $\langle 87\text{CPH}(115)103, 93\text{JSP}(160)158 \rangle$. A single-minimum one-dimensional potential function of the quadratic-quartic type has been estimated from the ring-puckering vibration by use of both sets of spectroscopic data. Theoretical calculations and molecular mechanics do reasonably well in predicting the barriers to planarity and in describing the ring-puckering in this molecule. The hydrogen-bond heterodimer formed by 2,5-dihydrofuran with hydrogen fluoride has been identified and characterized by means of its infrared and microwave spectra $\langle 87\text{JST}(162)31 \rangle$. The geometric parameters for the heterodimer, determined under the assumption of unchanged monomer geometries, demonstrate that the heterodimer has C_s symmetry and a pyramidal configuration at oxygen. X-ray analysis of **(92)** reveals that two molecules are present in the asymmetric unit $\langle 93\text{AX}(C)246 \rangle$, both the molecules exhibiting *O-endo* puckering; the oxygen atom is 16 and 18 pm, respectively, out of the plane defined by the four carbon atoms of the furan ring: this puckering reduces steric interactions among substituents, which assume an equatorial orientation.

2.05.3.4 Furans with Exocyclic Double Bonds

Structural study of some butyrolactones substituted in position 2 has been carried out using various techniques (x-ray, IR, and NMR) $\langle 84\text{JST}(118)277 \rangle$. Crystallographic data analysis reveals



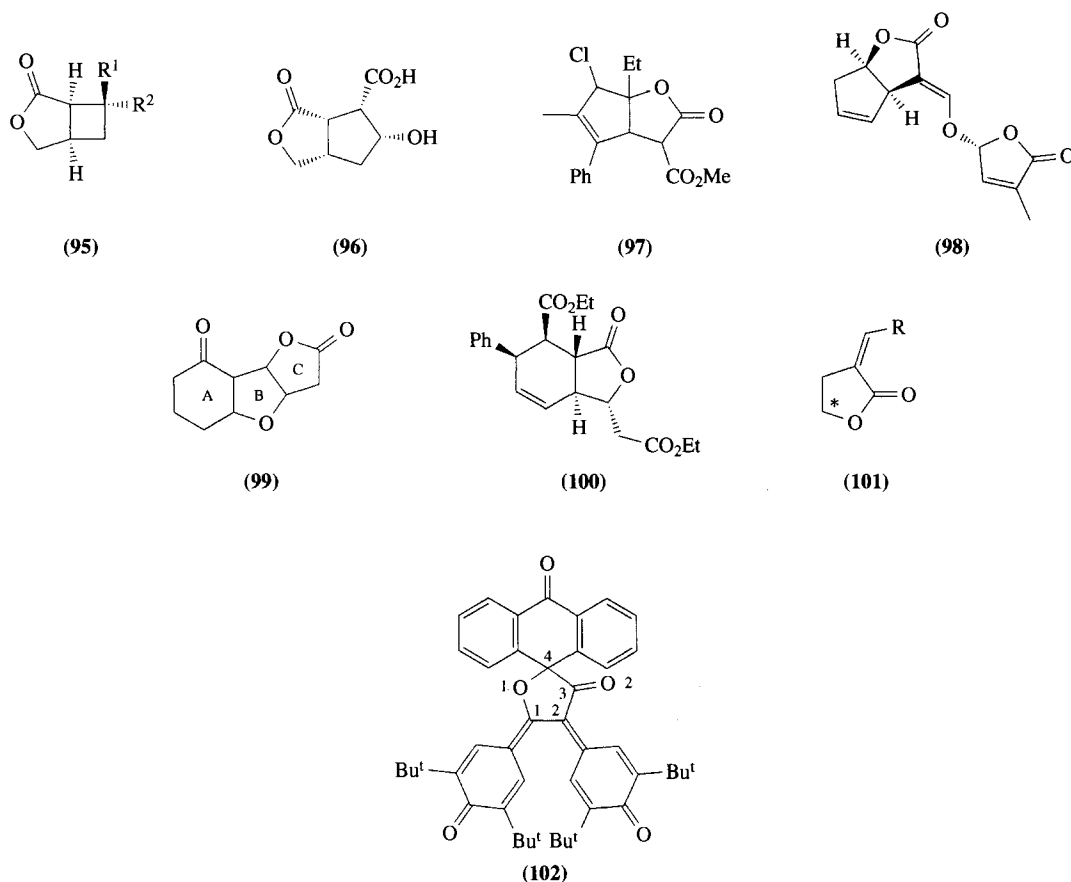
that in the solid state 2,2-dibromobutyrolactone adopts an “envelope” structure (93) with atoms O-1, C-1, C-2, C-4 describing a plane, whereas C-3 lies out of this plane. Spectroscopic IR data relative to (CH₂) have been interpreted for the dissolved state in terms of rigid or exchanging envelope forms, according to substituents. For 2- and 3-halogenated derivatives, quantitative analysis of IR spectra shows a pseudo-axial predominance in apolar solvents and the NMR spectra confirm this. A very similar envelope conformation for the lactone ring has been found in the *cis*-3,4-dibenzyl derivative (94) <90AX(C)872>. The internal bond angles of the lactone ring are significantly less than the values associated with *sp*³ and *sp*² hybridized carbon centers. The internal bond-angle compression about atoms C-1 through C-4 in turn induces a broadening of the external bond angles. Related molecules were found to exhibit a similar geometric trend <84JCS(P2)489, 89AX(C)297>.



In a condensed system the lactone ring may change its conformations. Accordingly, in *endo* ($R^1 = \text{CN}$, $R^2 = \text{H}$) and *exo* ($R^1 = \text{H}$, $R^2 = \text{CN}$) derivatives (95) the lactone ring assumes configurations intermediate between an envelope and a half-chair <84AX(C)1950>. In both compounds the cyclobutane ring is attached to the lactone ring in a *cis* manner. All rings are nonplanar but are flatter in the *exo* than in the *endo* form. An envelope conformation for the lactone ring is present in the molecular structure of (96) where the cyclopentane ring assumes a twist conformation and the ring junction is still of *cis* type <87LA811>. The *cis* junction is also present in (97) and (98). The dihedral angle between the cyclopentane and furan ring amounts to 74° in (97) <88AX(C)1443> and 116.9° in (98) <92MI 205-01>. The 5-membered ring in (99) adopts a conformation intermediate between half-chair and b-envelope <89AX(C)898>. The junctions between all the rings are *cis*, while the angles between the best planes through the A/B, B/C, and A/C rings are 6.7° , 117.0° , and 112.8° , respectively. The X-ray structure for derivative (100) corroborates the stereochemistry assigned by NMR spectroscopy of a *trans* junction between the rings <88JOC231>. The furanone ring in derivatives (101) deviates slightly from planarity but, in contrast to γ -butyrolactone, the largest displacement from the best plane has been found for the carbon atom indicated by * <90CCC674>. The configurations (*E*)- and (*Z*)- for the substituent ($R = p\text{-MeC}_6\text{H}_4\text{SO}_3$, MeSO_3 , $\text{C}_6\text{H}_5\text{CO}_2$) with respect to the double bond determined in the solid state are in agreement with the assignments made on the basis of ¹H and ¹³C NMR spectra.

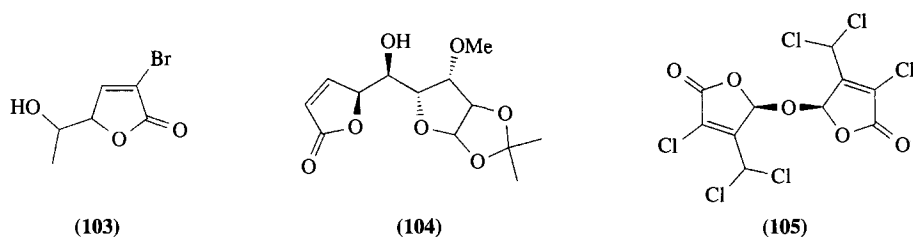
Dihydrofuran-3-ones (isomeric with γ -butyrolactone) are uncommon. In spiro-3-furanone (102) the furanone ring has the atoms C-1, C-2, C-3, and O-2 almost coplanar and lying in the mean plane of the quinoid rings <93TL5559>. The compound is intensely colored owing to a low-energy UV absorption band ($\lambda_{\text{max}} = 682 \text{ nm}$ in CHCl_3 solution) evidently resulting from an electronic transition of the extended π -electron system. The low-energy transition may reflect destabilization of the ground state due to twisting of the quinoid rings (the dihedral angle between the planes of these rings is 45.6°). Additional conjugation involving the carbonyl group, as well as a lone pair of electrons on O-1, may also contribute to the low transition energy. The contribution of the anthraquinoid moiety to conjugation is probably negligible, since it is nearly orthogonal to the mean plane of the furanone ring.

It has been determined by microwave spectroscopy that the heavy atoms of γ -crotolactone are strictly coplanar at equilibrium and that the ring-bending mode is governed by an essentially quadratic potential-energy function <81JCS(F2)2191>. A factor that favors the planar ring is the

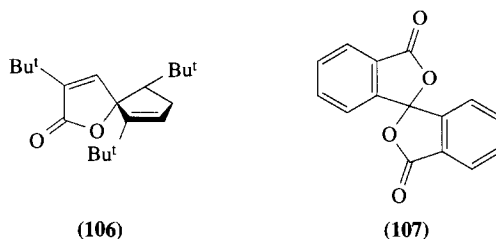


conjugation of the C=O group with the endocyclic double bond. This is evident from the value of $\mu_a = 4.726$ D ($1 \text{ D} = 3.336 \times 10^{-30} \text{ C m}$) in γ -crotonolactone, considerably greater than the $\mu_a = 4.04$ D of γ -butyrolactone; given that the angle between the C=O bond and the a -axis is essentially identical in these two molecules, the difference presumably arises from conjugation in the former $\langle 81\text{JCS}(F2)2191 \rangle$. Planarity of the lactonic ring has also been found in the crystal structure of the *erythro* stereoisomer of (103), where the maximum deviation from the mean plane of the ring is 0.3 pm at the ring oxygen atom $\langle 90\text{JHC}183 \rangle$. The lactone ring also maintains its planarity in (104), and the bond distances and angles are in the normal ranges for this type of molecule $\langle 88\text{AX}(C)159, 90\text{T}5807, 91\text{MI} 205-02 \rangle$. X-ray study of the single available diastereoisomer of anhydride (105) has shown it is the *dl*-diastereomer $\langle 90\text{MI} 205-01 \rangle$. However, in the particular lattice in which it crystallized it lacked the expected molecular symmetry of a *dl*-diastereomer. This is evident, for example, in comparing anomeric torsion angles: $\text{O}(1)-\text{C}(5)-\text{O}(7)-\text{C}(5') = -65^\circ$; $\text{O}(1')-\text{C}(5')-\text{O}(7)-\text{C}(5) = -78.3^\circ$. The 5-membered rings appear to be nonplanar; C-5 is displaced 130 pm below the plane of $\text{O}(1)-\text{C}(2)-\text{C}(3)-\text{C}(4)$ whereas C-5' is 330 pm below the plane of $\text{O}(1')-\text{C}(2')-\text{C}(3')-\text{C}(4')$; in the molecule there is essentially an oxygen-dominated surface, involving O-2, O-1, O-1', and O-2', and an opposite, chlorine-dominated surface involving all the chlorine atoms. Embedded below the chlorine surface is O-7.

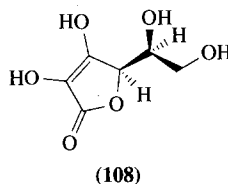
X-ray structures are available for the spiroderivatives (106) $\langle 89\text{HCA}811 \rangle$ and (107) $\langle 92\text{JCS}(P1)1769 \rangle$.



All bond angles were found to be normal, with the two planes of the *spiro*-system almost perfect, and at an angle of 90.2° to each other in (107).



Vitamin C (ascorbic acid) (108) is a substance of paramount importance. The structural features obtained with the MNDO method are in qualitative agreement with the x-ray data <92JST(256)271>. More specifically, the 5-membered ring is almost planar and the side chain is very flexible, possessing three local minima with low intermediate barriers. The molecule adopts one of these minima in the crystal. Conformational analysis of (108) has also been carried out in aqueous solution employing ^1H and ^{13}C NMR spectra and molecular mechanics (MM2) <86JST(142)387>.



2.05.4 DIPOLE MOMENTS

One of the earliest physical methods employed in the study of heteroaromatic systems was the determination of dipole moments. Table 7 lists the dipole moments of a number of simple derivatives in addition to those reported in CHEC-I. The experimental values for the furan derivatives are those utilized in conformational analysis and discussed in Section 2.05.3. For hydrofuran derivatives the dipole moments reported are those determined from microwave spectra (Stark effect) for the ground state of the ring-puckering mode.

Table 7 Dipole moments at 25°C for furan derivatives.

Substituent	Dipole moment, μ (D)	Phase	Reference
2-CHO	3.52	Benzene	89JST(196)227
3-CHO	2.77	Gas	92ACS923
2-Pyvaloyl	2.90	CCl_4	84JST(116)337
(51)	4.03	Benzene	89BSB327
2,5-Diformyl	3.27	Benzene	89JST(196)227
	3.16	Benzene	89H(29)657
(60) R = Me	2.13	Benzene	92JST(253)179
(60) R = OMe	3.21	Benzene	
(61) R = Me	3.54	Benzene	
(61) R = OMe	2.22	Benzene	
(63) 2-pyridyl	1.37	Benzene	83CJC334
(63) 3-pyridyl	2.42	Benzene	
(63) 4-pyridyl	3.04	Benzene	
(64) 2-pyridyl	2.27	Benzene	
(64) 3-pyridyl	2.23	Benzene	
(64) 4-pyridyl	2.35	Benzene	

1 D = 3.336×10^{-30} C m.

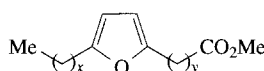
2.05.5 SPECTROSCOPIC CHARACTERIZATION

2.05.5.1 Proton and ^{13}C NMR Spectroscopy

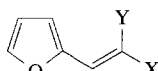
Nuclear magnetic resonance has provided a large amount of structural information on furan derivatives. NMR spectral parameters (^1H and ^{13}C chemical shifts, ring interproton coupling constants, direct and long-range ^{13}C and ^1H coupling constants) for furans and their benzo derivatives have been collected in CHEC-I. The effect of substituents on ^1H chemical shifts has also been discussed exhaustively and the correlation for ^1H chemical shifts in 2-substituted furans is examined. Considering the substituent effects as additive, the chemical shift for di- and trisubstituted furan derivatives can be calculated approximately by employing the chemical shift of monosubstituted derivatives and the substituent additivity constant $\langle \text{B-84MI 205-01} \rangle$.

Carbon chemical shifts show much the same features as the proton shifts and correlations have been found among them. Extensive quantitative correlations have been established between the shifts of corresponding nuclei in different heterocycles. In a multivariation investigation of ^1H and ^{13}C NMR shifts of 2- and 3-substituted furans, thiophenes, selenophenes, and tellurophenes $\langle 90\text{MRC397} \rangle$ the effects of substituents on chemical shifts has been studied by means of principal component analysis (PCA) and partial least squares (PLS) analysis. Data analyzed by the PCA approach showed that for both series (2- and 3-substituted compounds) the substituents are grouped into four classes depending on the heteroatom present in the ring, and that the substituent-heteroatom interaction has the same nature for all the rings. PLS analysis of the data sets enabled prediction of the chemical shift values of a different ring having the same substituents.

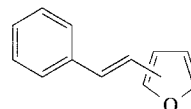
The effect of the furan nucleus as substituent on ^1H and ^{13}C chemical shifts has made possible the characterization of the positional isomers of the 2,5-disubstituted C_{18} furanoid fatty ester (**109**) $\langle 86\text{JCS(P2)1275} \rangle$. Carbon-13 chemical shifts turn out to be more responsive for unambiguous identification of the positional isomers. Substituent constants for the furyl group, together with those for other heteroaroyl groups, on β -vinyl carbon have been derived for a series of ethylenes (**110**) bearing heterocyclic substituents $\langle 89\text{T4103} \rangle$ and β -heteroaroyl styrenes (**111**) $\langle 90\text{JCS(P2)645} \rangle$.



(109)

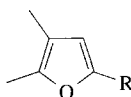


(110)



(111)

Much structural information has been obtained by examining data from ^1H and ^{13}C resonance experiments side by side, though ^{13}C data are sometimes more informative. Thus, for instance, ^1H and ^{13}C spectra of some 2-substituted 4,5-dimethylfurans (**112**) have been reported $\langle 92\text{MRC1141} \rangle$. The assignments of the carbon signal have been based on the characteristic splittings of signals and on the values of long-range $J_{\text{C,H}}$ coupling constants in proton-decoupled ^{13}C NMR spectra. Analysis of the substituent effects on the chemical shift values together with the value of $J_{\text{C,H}}$ has also enabled the identification of geometric isomers of some derivatives. The information derived from NMR data, together with other chemical findings, forms the basis for determining the structures of very large molecular systems. In this way structures are elucidated for furanoterpenes $\langle 92\text{JCR(S)366} \rangle$, furanosesquiterpenes $\langle 83\text{AJC371, 90P527} \rangle$, furanoid norditerpenes $\langle 86\text{MRC646} \rangle$, furanoid diterpenes $\langle 93\text{LA325} \rangle$, furanoid diterpene glycosides $\langle 93\text{LA491} \rangle$, and β -dihydroagarofuran sesquiterpenoids $\langle 91\text{MRC650} \rangle$.



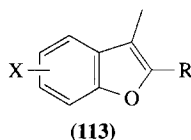
(112)

Nuclear magnetic resonance spectroscopy has been widely employed in the study of rotational isomer phenomena produced by substituents lacking rotational symmetry. The preferred conformations in solution have been determined by various NMR techniques, essentially: (i) analysis of ^1H and ^{13}C chemical shifts regarding the orientation of the substituent; (ii) analysis of stereospecific long-range coupling constants $^nJ_{\text{H,H}}$ and $^nJ_{\text{C,H}}$; (iii) relaxation parameters such as NOE and spin-lattice relaxation time (T_1); (iv) LIS experiments at ^1H and ^{13}C frequencies. The results obtained

with one or more of these procedures have been reported already and discussed together with other experimental evidence (Section 2.05.3).

Information about molecular dimensions may be obtained by liquid-crystal NMR spectroscopy. Molecular structure determined thus for furan <83OMR(21)143, 85MRC725, 89JST(196)1> is in fair agreement with the results derived from the microwave spectrum for benzo[*b*]furan <82OMR(18)157> and for dibenzofuran <84OMR(22)547>. For the latter compounds, deviation from planarity has been observed in agreement with the crystal structures.

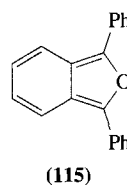
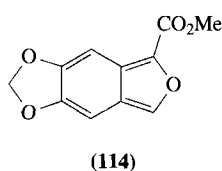
A structural analysis by ^{13}C NMR and mass spectrometry of a series of dimethoxycarbonyl-2,3-benzo[*b*]furan derivatives has shown the importance of electronic and steric interactions in these compounds <83SA(A)429>. From analysis of ^{13}C chemical shifts, benzo[*b*]furan seems not to behave as a perfect heterocyclic derivative, but rather as a styrene derivative. The importance of the *peri* type of steric effect is also evidenced. A study of selective heteronuclear NOE enhancements in benzoheterocycles <88MRC511> (including 3-methylbenzo[*b*]furan derivatives (113)) found that the differences in the magnitude of negative indirect heteronuclear NOE enhancements between benzo-fused pentagonal and hexagonal rings could be dependent on ring size. The magnitude of indirect heteronuclear enhancements in benzo[*b*]furans is correlated with the presence of a *peri* proton at C-4.



Little data is available for benzo[*c*]furan derivatives (Table 8). Proton and ^{13}C NMR spectra of compounds (35) and (114) have been examined <86JOC3973>. In both the derivatives the furanoid moiety is much like furan in its ^{13}C behavior, while the remaining carbon atoms (C-4 to C-7) belong to the cyclohexadiene moiety and can be considered as pairs (C-4,C-7 and C-5,C-6). Proton and ^{13}C NMR data are also available for 1,3-diphenylbenzo[*c*]furan (115) <88JCS(P2)31, 92RRC1285>. The dianion derived from (115) by metal reduction has been characterized by NMR measurements <88JCS(P2)31>. The proton and ^{13}C spectral parameters depend on the alkali metal used for the reduction and show, moreover, that the electron transfer reaction induces hindrance of the free rotation about the carbon-carbon bond of the phenyl substituents.

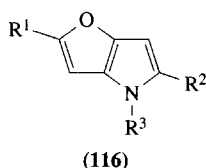
Table 8 Carbon-13 NMR chemical shifts (ppm) for benzo[*c*]furan derivatives.

Compound	C-1	C-3	C-4	C-5	C-6	C-7	C-3a	C-7a	Reference
(35)	117.6	139.5	91.2	148.7	151.5	93.1	121.9	130.9	86JOC3973
(114)	134.4	138.3	92.7	147.9	150.9	94.0	122.7	127.4	
(115)	144.2	144.2	120.5	125.6	125.6	120.5	122.7	122.7	88JCS(P2)31
(115)	143.67	143.67	120.10	125.05	125.05	120.10	122.12	122.1	92RRC1285

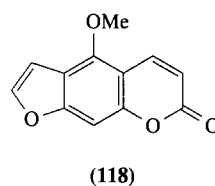
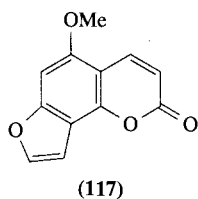


A systematic study is available on the ^{13}C NMR spectra of furo[3,2-*b*]pyrroles (116) <90MRC830>. The chemical shifts and $J_{\text{C,H}}$ values of C-2 and C-3 of the furan ring fall within the usual range measured for 2- and 3-substituted furans or benzofurans. The largest effect from substituents in position 2 has been observed at C-2, C-3, and C-6a. Carbon 6a, with the character of a *para* position is much more responsive to the substituent effect than C-3a.

The effects of furan substituents on the coumarin nucleus have been estimated in some angular (117) and linear (118) furocoumarins <84P697>. The effect of the furan ring on ^1H methoxyl signals is similar to its effect on protons in the same position but an additional downfield shift is observed.



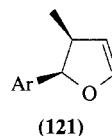
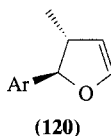
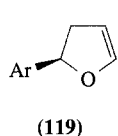
The differences in the methoxyl chemical shifts thus provide a simple method for distinguishing between an angular and a linear furocoumarin. Spectral ^1H and ^{13}C data for some 3,4-fused furocoumarins have also been reported recently <91MRC631>.



2.05.5.2 Proton and ^{13}C NMR Spectra of Modified Furans

2.05.5.2.1 Hydrofurans and their benzo derivatives

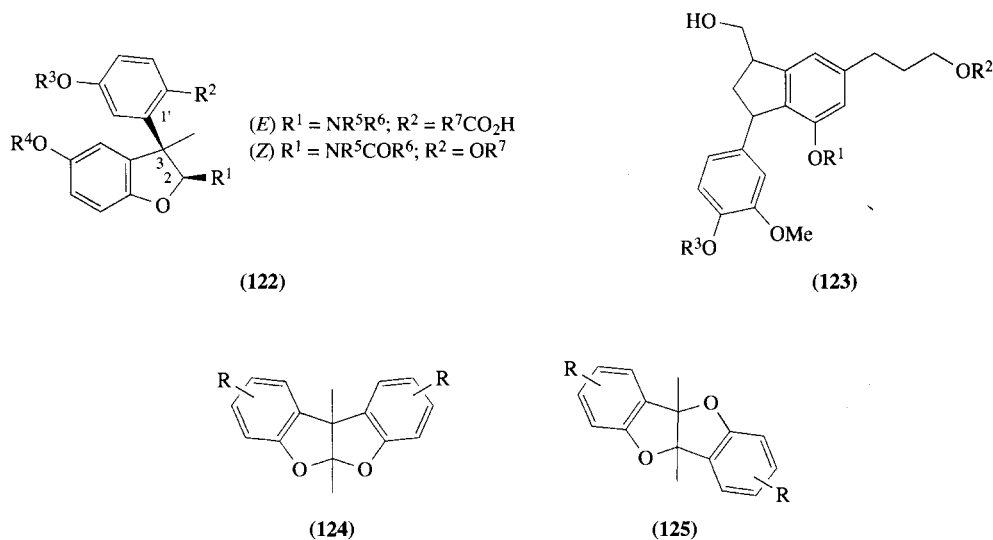
Carbon-13 NMR spectra of some 2,3-dihydrofurans ((119)–(121)) with the aromatic group symmetrically and unsymmetrically substituted have been measured <84OMR(22)636>. Differences observed in the ^{13}C chemical shift values of C-2, C-3, and Me, together with ^1H chemical shifts of H-2, Me, and the coupling constant $J_{2,3}$ turn out to be distinctive parameters for the characterization of the *trans* (120) and *cis* (121) isomers. Restricted rotation of the bulky aromatic groups of (121) has been observed when these groups have substituents in both *ortho* positions. The variations in linewidths of the ^{13}C signals have been employed to deduce barriers to rotation.



Proton NMR analysis has been employed for structure determination of dihydrofurans obtained as reaction intermediates. It has thus evidenced the formation of *cis*- and *trans*-2,5-dichloro-2,5-dihydrofuran, *trans*-2,3-dichloro-2,3-dihydrofuran, and *cis*-2,3-*trans*-4-*cis*-5-tetrachlorotetrahydrofuran in the addition of molecular chlorine to furan in carbon disulphide <84G1>. The structures of the six adducts isolated in the reaction of methyl 2-furoate with acetyl nitrate, in their (*Z*)- and (*E*)-configurations, have been established <84JOC1636>.

Reference data for the constitutional structure and stereochemistry determination of dihydrobenzofurans are available. In derivatives (122) the assignment of the (*E*)- and (*Z*)-configurations is based on comparison of the ^{13}C NMR chemical shifts for the two stereochemical sites (i.e., 3-Me, and C-1' in the (*E*) and (*Z*) series) <85MRC988>. Further, the chemical shifts of atoms attached to C-3 in the two diastereomeric series serve as strong evidence that the steric compression shift between sp^2 and sp^3 carbons and nitrogen leads to a low-frequency shift in a γ -*cis* arrangement of the atoms compared with a γ -*trans* arrangement. The steric compression in the γ -*cis* arrangement has been confirmed by x-ray analysis. Proton and ^{13}C NMR data have been reported and utilized for deducing structural information on some dihydrobenzofuran lignans (123) <83OMR(21)119, 93MRC692>.

The sites of fusion of the furan ring with a phenyl ring may be localized using the ^1H NMR spectra <90MRC867>. From comparison of ^1H spectra of benzofuran and its dihydrobenzofuran derivatives it has been observed that the benzene proton *ortho* to the furan oxygen undergoes a larger change in chemical shift than those for the other protons. The structures of benzofurobenzofurans ((124) and (125)) have been determined <84SA(A)189> and the stereoelectronic effects of substituents have been determined.



2.05.5.2.2 Tetrahydrofuran derivatives

Most of the NMR studies on tetrahydrofurans in solution have been devoted to determining the preferential conformations of the flexible tetrahydrofuran ring with reference to substituents on the ring and the conformations of the substituents themselves when they may assume different orientations. The change in conformation induced by alkyl substituents does not seem to affect the ^{13}C spectra as much as expected, since substituent increments remain roughly additive (see CHEC-I). A correlation has been proposed between the overall ^{13}C chemical shielding ($\Sigma\delta^{13}C$) and the steric energy (computed with the MM2 force field) for dialkyl-substituted tetrahydrofurans <92JST(265)225>. Comparison of the differences between the overall ^{13}C chemical shielding of the diastereoisomers with the differences in their steric energies shows that the diastereoisomers with lower energy are relatively the less shielded.

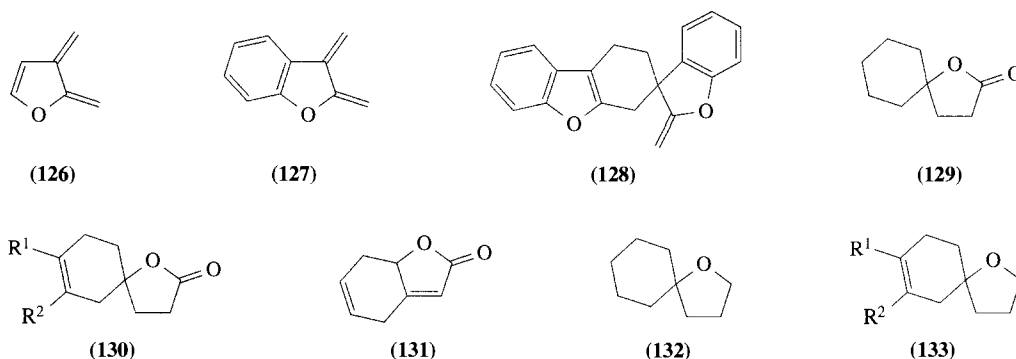
The methodology employed in the structural and conformational analysis of substituted tetrahydrofurans, as for furanose sugars, has already been described.

The main problem is the correct assignment of ^1H and ^{13}C NMR shifts and a clear J pattern. Assistance in solving this problem has come with the advent of a whole series of new methods, such as polarization transfer, multiple quantum coherence, and a variety of two-dimensional spectroscopic methods. These methods, which rely heavily on the availability of high-field spectrometers and fast, powerful computers, have produced a qualitative change in NMR by enabling the detailed study of large and complex molecules. It has also become feasible to establish the stereochemical and conformational relationships in substituted 1,2:5,6-di-*O*-isopropylidene- α -D-allofuranose derivatives <92CAR(229)245, 93CAR(244)49>, β -D- and α -L-arabofuranosides <92JA3710, 93CAR(248)81>. Ribonucleosides <90JA7373>, erythronucleosides <92JOC1772>, and 2'-deoxyribonucleosides <93JOC5513> ^{13}C -enriched at C-1' of the furanose ring have been prepared and studied by ^1H and ^{13}C NMR spectroscopy. The value of direct spin-spin coupling $^1J_{C-1',H-1'}$ appears to depend on base structure. Information about the ring conformation has also been deduced from the value of $J_{C,C}$ for erythronucleosides <92JOC1772> and the apiofuranose ring <87CAR(166)85>.

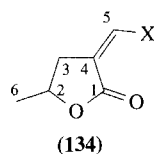
2.05.5.2.3 Furans with exocyclic double bonds

2,3-Dimethylene-2,3-dihydrofuran (126) and 2,3-dimethylene-2,3-dihydrobenzofuran (127) have been prepared by flash vacuum pyrolysis of substituted furylmethyl <81JA6691, 86JA4138> and benzofurylmethyl esters <86JOC4208>. Since these compounds dimerize at room temperature, very pure monomers have been obtained utilizing a cold trap. Low-temperature NMR spectra have been used to determine the structures of (126), (127), and the dimerization products. The major product from (127) is the [4 + 2] spirodimer (128), an unstable compound whose structure it has not yet been possible to confirm by ^1H and ^{13}C spectra. ^{13}C NMR data are available for a series of lactones and ethers containing the spiro[4,5]decane ring system ((129)–(133)) <93MRC608>. In spirolactones (129)–(131) the *spiro* ring junction carbon absorption depends on the presence or absence of sp^2 carbon

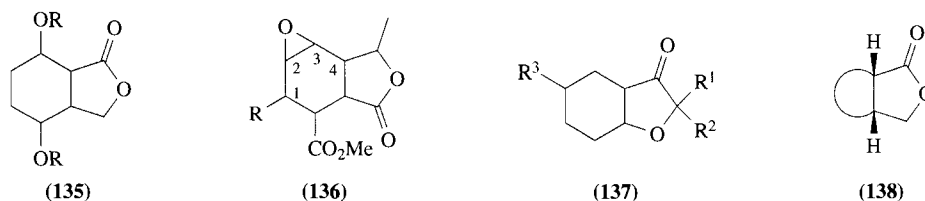
atoms in each ring moiety. Moreover, the C-10 and mainly C-6 chemical shifts are strongly influenced by the presence of unsaturation at the pentagonal ring.



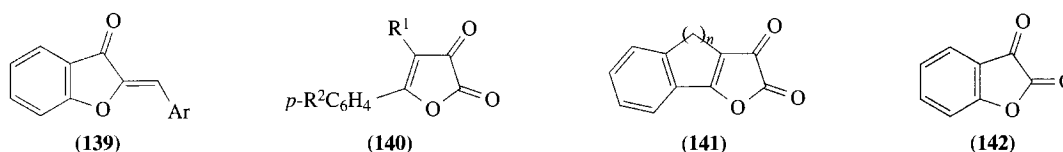
Configurations of the isomeric sulfonates and carboxylates of (134) have been derived from ^1H and ^{13}C NMR spectra <85CCC348>. The chemical shifts of the vinyl protons and their coupling constants in the ^1H NMR spectra have enabled determination of the (*E*)- and (*Z*)-configurations, confirmed by the value of ^{13}C shifts of C-1 and C-5. Analysis of the values of $^4J_{\text{H,H}}[\text{H-2,H-3; H-2,H-3}']$ and $^3J_{\text{C,H}}[\text{C-6,H-3; C-6,H-3}']$ suggests that the ring may assume a sole conformation with an axial H-3 atom and an equatorial methyl group.



The structure of the perhydrobenzofuran derivatives (135) has been derived for comparison with a proposed structure of "garlicin" <92LA383>. Proton NMR spectroscopic studies for $\text{R} = \text{COMe}$ and $\text{R} = \text{COCF}_3$ enable the determination of the relative configuration at the chiral centers, because all ^1H signals are well separated at 400 MHz. ^1H NMR at 300 MHz has made possible the determination of the structure of the isomers (136) obtained through epoxidation of unsaturated lactones <89T4213>. In these determinations the values of $J_{\text{H-1,H-2}}$ (<1 or 2.8–3.5 Hz) and $J_{\text{H-3,H-4}}$ (3.1 or <0 Hz) according to stereochemical relationship are important. A complete spectral analysis has enabled the determination of the configuration of a series of *cis*-benzofuran-3-ones (137), and of the corresponding alcohols and their acetates <85BSF(2)931>. The *cis* junction between the rings is made evident by ^1H NMR data. The cyclohexane ring adopts a chair conformation slightly flattened owing to the *cis* junction, and the 5-membered ring, with the exception of 5- β -*t*-butyl derivatives, prefers a conformation with the ether bond in an axial position. The prevalence of the isomers with a *cis* junction has also been emphasized by NMR data for perhydrocyclododeca[*b*]furan derivatives (138) <91CJC521>.



Carbon-13 NMR chemical shifts for a variety of aurones (139) are reported and discussed in order to determine how the substituent effects can be used for structural elucidation <83H2203>. Substituent effects seem not to influence the ^{13}C chemical shifts of furan-2,3-dione derivatives (140) and (141) <91BSF393>. The modification of the substituent in the *para* position of the phenyl ring (140) or the strain induced by the bridge (141) do not greatly affect the chemical shifts of the carbon atoms of the furandione ring.



2.05.5.3 Oxygen-17 NMR Spectra

Despite its unfavorable NMR properties, the ^{17}O nucleus has attracted considerable interest at molecular level, since its chemical shifts represent a discriminating probe for structural and molecular properties. A recent monograph covers a large range of applications of ^{17}O spectroscopy to organic chemistry (B-91MI 205-04).

Examination of ^{17}O chemical shifts of several 2-substituted and 2,5-disubstituted furans (85MRC985) has demonstrated the additivity of substituent effects by plotting the ^{17}O chemical shifts of 2,5-disubstituted furans versus those of the 2-substituted analogues. Failure of the electronic character of substituents in determining ^{17}O chemical shifts is found, because they do not correlate with Hammett constants. Such a lack of correlation is known for ^{13}C *ortho* chemical shifts in aromatic systems. A good correlation is obtained by plotting ^{17}O chemical shifts versus a parameter Q of the substituent—a parameter which accounts for the paramagnetic contributions and is a function of the polarizability and ionization potential of the substituent. The cyano- and methylfurans, however, do not follow the Q relationship.

In a study of some 5-membered heterocycles (furan and isoxazole methyl derivatives) (84OMR(22)55) it was found that the ^{17}O chemical shifts are mainly determined by the p -electron density on the oxygen atom and are also affected by alkylation at different positions of the ring system, which gives rise to a b effect similar to those observed for simple ethers. The effect of the annelation of a benzene ring to a furan results in a distinct shielding of the ether oxygen, and this effect is additive (87MRC489). This has been shown for furocoumarins and related compounds and the interaction effects can be interpreted in terms of steric and electronic effects.

Furan-2,3-dione derivatives (140), (141), and (142) have been examined by ^{17}O NMR spectroscopy (92MRC837). For the unstrained compound (140), the ^{17}O chemical shifts of the carbonyl oxygens of aryl monosubstituted furan-2,3-diones reflect the electron-donating and electron-withdrawing properties of the p -phenyl substituents. This is demonstrated by the good correlation between ^{17}O chemical shifts and the Hammett constants of the substituents or the total charge density at oxygen atom calculated by AM1. No correlations are obtained for the oxygen atom of the ring. For compounds (141) and (142) no conclusive information can be obtained.

2.05.5.4 Mass Spectrometry

The employment of mass spectrometry in conjunction with gas chromatography represents one of the most powerful analytical tools: it makes it possible to determine even trace amounts of organic materials. The signals detected by the spectrometer are the result of a series of unimolecular fragmentations and several structural features of the original molecule are often retained in the fragment, thus enabling deduction from the fragmentation pattern of the molecular structure.

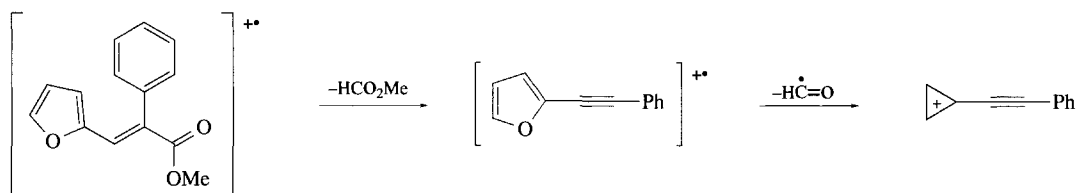
The application of mass-spectroscopic techniques to carbohydrates and other oxygen heterocycles and stereochemical considerations have been examined in a review (87AHC(42)335). The influence of structure on the fragmentation mechanisms of some stereoisomeric furanosides derivatives has also been determined (92OMS(27)1114). The principal fragmentation pathways for furan and benzofuran have been reviewed in conjunction with other heterocycles (B-85MI 205-01), while mass-spectral data can be obtained from a published registry (B-89MI 205-02).

Mass spectrometry has been applied to the study of the mechanism of isomerization and fragmentation of the molecular ions in the gas phase. A combined analysis of experimental and theoretical results has been utilized to study the mechanism of the unimolecular losses of CO and C_2H_2 from furan radical-cations (85NJC25). Theoretical calculations (MNDO and *ab initio* level) of the energy of various $\text{C}_4\text{H}_4\text{O}^+$ isomers and of the potential energy curves from the isomerization reactions enable interpretation of the observed spectra. The electron impact and collisional activation mass spectra, together with MNDO calculations, have been employed to study mutual interconversion of the molecular ions $\text{C}_6\text{H}_6\text{O}^+$ of 2-methylfuran, 3-methylfuran, and 4H-pyran before their fragmentation to C_5H_5^+ ions (86OMS(21)459). In agreement with experimental results,

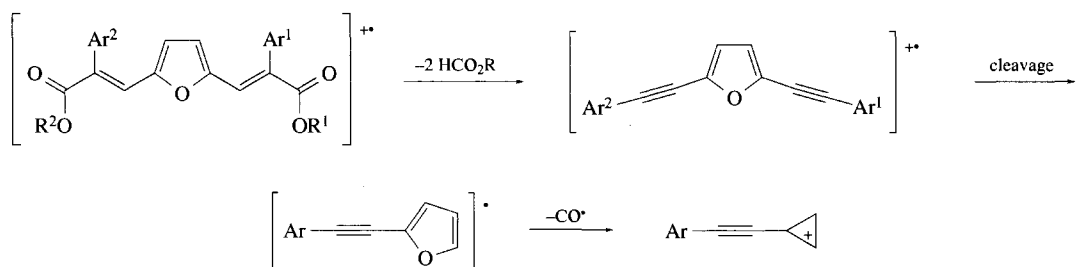
the MNDO calculations of the minimum-energy reaction path reveal that 2- and 3-methylfuran ions favor a rearrangement into pyran ions before fragmentation into furfuryl ions.

The alkylation of 2-methylfuran has been studied by mass spectrometry under chemical ionization conditions <88OMS(23)370>. The experimental results show that alkylation occurs predominantly at the *b* position; moreover, MNDO calculations show that this reaction is under thermodynamic control.

The literature contains very little information about the mass spectra of furan compounds substituted with an unsaturated chain. The fragmentation mode of some 2-styryl- and 2,5-di-styrylfuran derivatives has been studied <92OMS(27)615>. Each styryl substituent has an α -carboxylic acid or methyl carboxylate substituent. The molecular ion is the base peak for styryl and distyryl derivatives and the distyryl species show less fragmentation than the styryl species. An important fragmentation pathway for both these derivatives, in addition to simple bond cleavage, is the loss of formic acid or methyl formate, presumably forming acetylenic fragments (Schemes 1 and 2).



Scheme 1

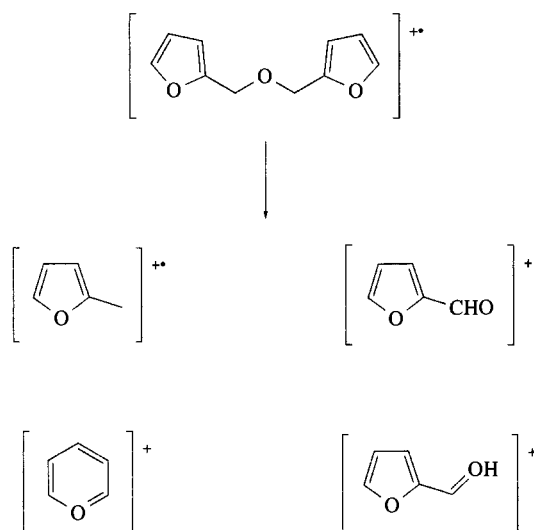


Scheme 2

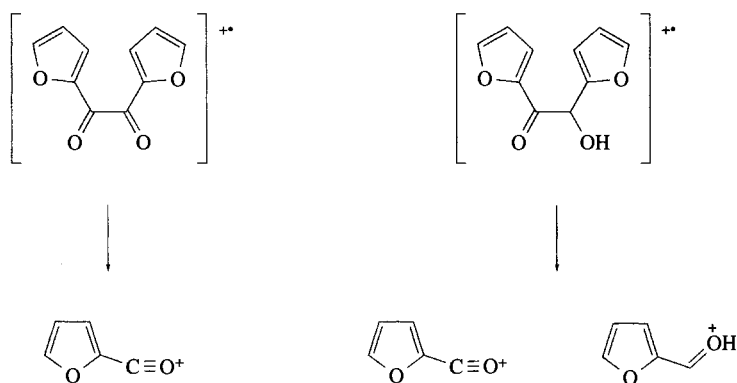
A study of the dissociation behavior observed during thermal and electron impact-induced fragmentation has been carried out for compounds containing “bridges” between two or three furan nuclei <91MI 205-03>. The behavior is quite similar in the case of oxabismethylenefuran; an oxidation–reduction has been proposed for the radical-induced fission of the ether bridge, resulting in the aldehyde, the corresponding alcohol, and the methylfuran. Mass-spectral data could be explained according to the fragmentation pathway of Scheme 3. The behavior of the dicarbonyl bridge is comparable. Fural shows very simple thermal and induced fragmentation (Scheme 4). The dominant process in both the latter cases is the cleavage of the dicarbonyl group resulting in two furfural molecules on pyrolysis and the corresponding cation in the mass spectra. The formation of an acylium cation has also been seen as the product of the principal fragmentation pathway for methoxycarbonylbenzofuran (Scheme 5). The loss of two molecules of CO gives rise to the dehydrotropylium ion <83SA(A)429>.

2.05.5.5 Photoelectronic Spectroscopy

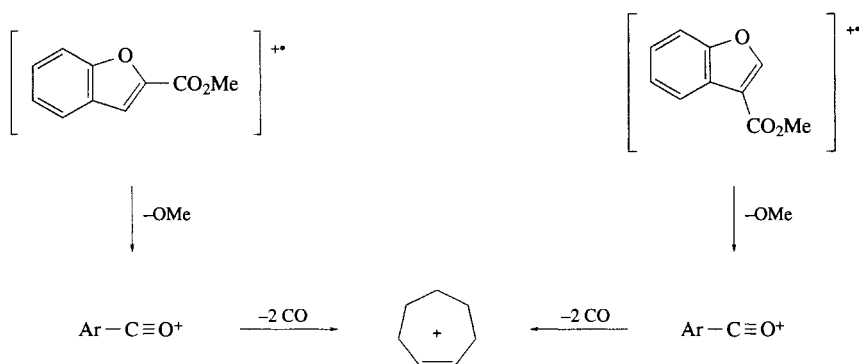
Photoelectronic spectra furnish direct information on the structure of the molecular energy levels. The assignment of the spectra is greatly helped by the availability of accurate calculations. Parallel to experimental development have been the increasingly sophisticated theoretical treatments that include higher levels of electron correlation. In the high-resolution synchrotron photoelectron spectra of furan, besides the attribution and significant insight into the dominant character of the valence orbitals, several ionization peaks attributed to inner valence orbitals and correlation states have been clearly resolved <92CPH(164)283>. The orbital assignments have been confirmed by cross-section ratios. In addition, two classes (tightly bound and less tightly bound) of a_1 -type molecular orbitals and of b_2 -type molecular orbitals have been distinguished.



Scheme 3



Scheme 4

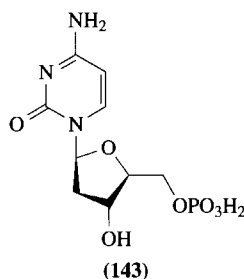


Scheme 5

Photoelectronic spectra of a series of six carbonylfurans (**39**; R = H, Me, F, Cl, Br, OH) and 2-furonitrile have been interpreted by comparison within the series and by differences in the relative effects of substituents (90CJC747). The peaks at lower ionization energy have been assigned to photoionization from π -orbitals of the furan rings, orbitals localized on the carbonyl group, and orbitals of the carbonyl substituents, in agreement with MO calculations. Within the series of

compounds it has been found that the inductive effect of the carbonyl group and its substituents is the predominant one. The determination of the electronic structure of a compound is of considerable interest, whether in order to obtain more in-depth knowledge of its inner chemical structure or to aim at a specific view, as in designing conducting polymer materials. Hence the photoelectronic spectra of bifurans and thienylfurans <92JES143> and of the first two oligomers of furan <91JPC9746> have been recorded and assigned. The data for oligomers of furan have been extrapolated to polyfurans.

The employment of photoelectronic spectroscopy in conjunction with results from *ab initio* MO calculations has provided a valence orbitals description of the structure of the neutral and anionic forms of the nucleotide (**143**), the simplest among all the common nucleotides occurring in DNA and RNA <90JA538>. The electronic structure enables conclusions to be drawn about the distribution of the products in the electrophilic attack of nucleotides. This is supported by alkylation patterns in methylation and ethylation of DNA and RNA.



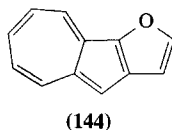
2.05.5.6 Electronic Spectra

Although this technique has seldom been utilized in the 1990s, the assignment of electronic spectra has not yet been clarified unambiguously even for molecules as simple as furan. The difficulties stem from the appearance of a number of strongly allowed Rydberg series, which overlap with some broad and diffuse bands usually assumed to arise from $\pi-\pi^*$ valence-type excitation. Progress in these studies has been made by comparing the vibronic structure of selected band systems with those of the corresponding photoelectron bands <92JST(273)133> and by employing *ab initio* multiconfigurational second-order theory (CASPT2) <93JA6184>.

Simple substituted benzofurans show a marked dependence of the first electronic absorption band on the position as well as the nature of the substituent <86ZN(A)1425>. In agreement with the results of configurational analysis, a simple model has been developed based on the molecules-in-molecule approach that quite satisfactorily reproduces the characteristic dependence of the UV spectra of these compounds on the substituent position.

Application of electronic spectroscopy to conformational analysis of some furan derivatives has been reported.

The phosphorescence and fluorescence spectra of compound (**23**) have been recorded <89CB1119>. The electronic and fluorescence spectra have been measured for azuleno[1,2-*b*]furan (**144**), which is an iso-*p*-electron system of benz[*a*]azulene and presents anomalous fluorescence from the second excited singlet state, as does azulene <87SA(A)1377>. Fluorescence spectral properties of a series of 2-(4-substituted phenyl)benzofurans have been investigated to determine their applicability as organic reagents for analysis <85BCJ2192>.



2.05.5.7 Infrared Spectra

The IR spectra are widely registered and reported in papers for their relatively rapid qualitative analysis and molecular determination, but no complete vibrational assignments are ever reported.

The relevant quantities needed for this purpose are the force constants, which may be determined for small molecules and transferred to large systems. However, purely experimental determination of force fields is extremely difficult even for the smallest molecules and transferability is limited.

Theoretical determination of force fields may frequently be of assistance. The calculated frequencies are, however, dependent on the level of theory employed. Calculated *ab initio* harmonic frequencies for furan are sufficiently accurate to help in the assignment of experimental frequencies <88JPC1739> as well as the vibrational overtones <91JPC7659>. In a reinvestigation of the vibrational spectrum of tetrahydrofuran all the normal modes have been identified and assigned in terms of symmetry coordinates with the guidance of the computed *ab initio* spectrum <93JPC7844>.

Application of a scale factor to force constants calculated with the AM1 semiempirical Hamiltonian enables the vibrational frequencies of large molecules to be predicted <88JCP7295>. The method has been applied to benzene and furan to predict the vibrational spectra of benzofuran. For this compound the vapor-phase far-infrared spectra gave evidence of the coplanarity of the two rings <84JCP(81)4281>.

IR spectroscopy is particularly helpful in detecting the presence of hydrogen bonds. Raman and IR spectra of furan-3,4-dicarboxylic acid <86MI 205-01>, furan-2,3-dicarboxylic acid <88MI 205-02>, and its potassium salts in aqueous solution show the existence of a very strong intramolecular hydrogen bond, with a resulting chelate ring in the furan plane.

Vibrational spectroscopy has been applied to the study of molecular structure and physical properties of conjugated polyfurans <92CPL(191)419, 93JCP(98)769, 93SM(57)4467>. Vibrational spectra provide simultaneous and selective information on the structure of a polymer and on its charge distribution. Interpretation of experimental data is performed with the assistance of lattice dynamics calculations.

2.05.6 THERMODYNAMIC ASPECTS

2.05.6.1 Melting and Boiling Points

The melting or boiling point is often one of the first properties to be determined in the attempt to identify an unknown compound. A computer-assisted method has been proposed for predicting the normal boiling point for new compounds of furan, tetrahydrofurans, and thiophenes <91JCI301>. The method employs structural parameters that are calculated directly from the structures for a given set of compounds. These parameters are then correlated with observed boiling points using multiple linear regression analysis techniques to obtain linear predictive equations (QSPR). These equations have been found to yield good results for a wide variety of furan- and tetrahydrofuran-containing compounds. The models developed indicate that the boiling point is a function of the size and shape of the molecule as a whole, as well as of the type of functional groups present in the molecule and does not show specific divisions based on the parent ring.

2.05.6.2 Gas Chromatography

The specificity and sensitivity of gas chromatographic techniques have enabled the separation and measurement of compounds in complex mixtures which hitherto had been impossible. Many methods have been developed for the determination of environmental contaminants, among them polychlorinated dibenzofurans (PCDF), whose toxicity is well known and whose presence has been reported in water as well as in fly-ash from incinerators. A relationship between gas chromatographic retention index and structure of all the possible chlorinated isomers of dibenzofuran has been proposed <92MI 205-03>. The structures were encoded in the form of descriptors. Four types of descriptors were calculated: topological, electronic, geometric, and atom-based descriptors. Regression equations modeling gas chromatographic retention for nonpolar, moderately polar, and polar stationary phases show good agreement between the experimental data and the calculated values. Another type of relationship has been reported: the gas chromatographic retention index has been correlated with physical properties for four classes of compounds, among them polychlorinated dibenzofurans <91ANC2829>. The physical properties employed are molecular polarizabilities, ionization potentials, and dipole moments calculated using AM1 methods.

2.05.6.3 Aromaticity

Aromaticity is undoubtedly one of the central concepts of organic chemistry and in particular of heterocyclic chemistry. The concept has a twofold aspect which has long been discussed: first a qualitative aspect for defining the characteristic of an aromatic compound, then a quantitative one for defining a numerical scale or measure of aromaticity. While the first aspect has long been realized, many attempts have been made to obtain a quantitative measure of aromaticity. For this purpose various criteria have been proposed that are related to physiochemical properties that are considered as a manifestation of aromaticity. The chief criteria employed are energetic, geometric, and magnetic ones. In some cases not all these criteria are satisfied concurrently (87JA2902). A statistical analysis of the several parameters employed in attempting to quantify aromaticity has pointed to the existence of two different types of aromaticities, designated as 'classical' and 'magnetic' (89JA7). These two types of aromaticity are orthogonal and this may account for the discordance evidenced (87JA2902). In the discussion of aromaticity in general, two review papers stand out, one concise (91H(32)127) and one comprehensive (93AHC(56)303), devoted to the problem of heteroaromaticity. The present discussion continues by examining the application of aromatic criteria to furan and benzofuran.

Furan, like benzene, has six π -electrons and is therefore considered to be aromatic. Together with benzene and other heterocyclic compounds, it has often been chosen as a reference compound in defining and determining aromaticity. Mulliken population analysis of the π -electron distribution in furan, pyrrole, and thiophene points to a greater aromaticity of pyrrole and thiophene relative to furan (81JST(85)163). This has been attributed to the size of the heteroatom, which determines the degree of delocalization of electron density in the ring. A similar trend has been found to determine the delocalization energy of the π -electrons by calculating the energy effects associated with fragment π -orbital interactions using the quantitative PMO treatment (88JST(163)173). Besides the three previously mentioned heterocycles, cyclopentadiene and benzene are included in the series. The most important interactions (stabilizing and destabilizing) have been determined in order to assess the degree of aromaticity of these compounds. The delocalization energy calculated with PMO and the orbital energy method correlates very well with experimental resonance energy.

The problem of σ - π separability and the influence of both contributions on the structure of 6- and 5-membered heterocycles has been investigated with respect to the behaviour of σ - and π -electrons (90JA6772). In neutral systems the π -electrons prefer localized structures, whereas the δ -electrons prefer delocalized structures. While in 6-membered rings the σ -electrons dominate, generate delocalized structure, and enforce delocalization of π -electrons, so originating the aromatic properties of such ring systems, in 5-membered heterocycles no dominance of either π or σ parts can be found. Consequently, in 5-membered heterocycles the equilibrium structure is a compromise and the aromaticity is less pronounced.

Another criterion of aromaticity is based on the estimation of the delocalization stabilization from the differences for the bond separation and superisodesmic reactions (88JA4204). On the basis of superisodesmic reactions all the 5-membered rings with the heteroatom belonging to group 6 (X = O, S, Se, Te) exhibit significant stabilization due to delocalization, with thiophene being much more delocalized than furan.

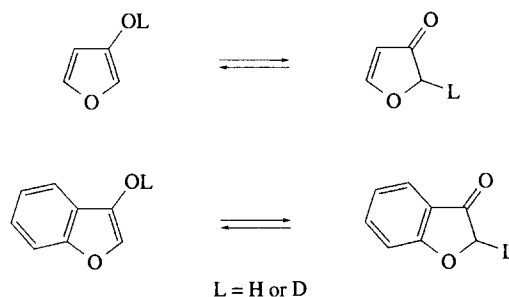
"Relative hardness," defined as the difference between the values of the absolute hardness (HOMO-LUMO gap) for a given molecule and for the corresponding acyclic reference structure, has been proposed as an index to measure aromaticity (89JA7371). According to this index benzo[*b*]furan is predicted to be aromatic, and furan to be nonaromatic. The difference between the energy of the lowest natural *p*-localized orbital (NLMO or Boys localized) and the corresponding energy of the lowest π -delocalized molecular orbital (MO) has been associated with effective resonance energy and proposed as a criterion for measurement of aromaticity (90IJQ843). This criterion has been applied to a set of heteroaromatic compounds and good correlation found with χ^{mcl} (nonlocal (π ring current) contribution to the out-of-plane component of the magnetic susceptibility tensor) and with other theoretical measurements of aromaticity.

Aromaticity indices based upon structural properties have been proposed. The Pozharskii index (85CHE717) is the average of the fluctuations of all the ring bonds evaluated by the bond-order values. Bird's index (85T1409) is a dimensionless quantity reflecting the degree of statistical uniformity of the bond orders in the aromatic ring. The Jug index (83JOC1344) corresponds to the bond order of the weakest ring bond. This assumption is justified on the ring current concept: the most significant breakdown in cyclic ring current occurs specifically in the vicinity of the weakest ring bond. Therefore, the aromaticity should be higher as the order of this bond increases. All these indices have been re-examined and a fairly complete data set is reported for 5- and 6-membered heterocycles

<90JPR853> and for their benzo-fused derivatives <90JPR870>. The problem of the interrelation among the different criteria of aromaticity (geometric, energetic, and magnetic) has been tackled by principal component analysis of several measures of aromaticity and leads to the important conclusion that aromaticity is of at least two kinds, namely, classical aromaticity and magnetic aromaticity <89JA7>. These two kinds measure different concepts and some of the common characteristics of aromatic compounds are determined by the one, some by the other, and some by both of these properties.

2.05.6.4 Tautomerism

The keto–enol tautomerism for a series of 3- and 2-hydroxy-substituted 5-membered heterocycles and their benzo derivatives has been studied <89JA5346>. 3-Hydroxyfuran and 3-hydroxybenzofuran exist exclusively in the keto form, the enolic form having been generated in solution by hydrolysis of their trimethylsilyl derivatives. Attempts to prepare solutions of 2-substituted derivatives have been unsuccessful. The equilibrium constants for the keto–enol tautomerism (Scheme 6) have been determined. These equilibrium constants indicate that 3-hydroxyfuran and its benzo analogue are relatively less stable with respect to their keto forms than are 3-hydroxythiophene and 3-hydroxypyrrole and their benzo analogues with respect to their keto forms. This is correlated with a greater degree of aromatic character of thiophene and pyrrole as compared to furan, and of benzothiophene and indole as compared to benzofuran. The successful generation of 2-hydroxy derivatives of thiophene and benzothiophene, unsuccessful for other heterocycles, is presumably another index of the higher aromaticity of thiophene as compared to furan and pyrrole, and of benzothiophene as compared to benzofuran and indole.



Scheme 6

Another type of tautomeric equilibrium is represented by the tautomeric equilibria of sugars. Two-dimensional NMR techniques have been employed to study the tautomeric equilibria of D-fructose and related compounds in different solvents <90MRC283>. The percentages of the three main tautomers of D-fructose (β -furanose, α -furanose, and β -pyranose) have been determined in D₂O and DMSO at different temperatures. The greater percentage of the β -furanose form in DMSO has been attributed to the presence of intramolecular hydrogen bonding.

2.06

Furans and their Benzo Derivatives: Reactivity

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2.06.1 INTRODUCTION

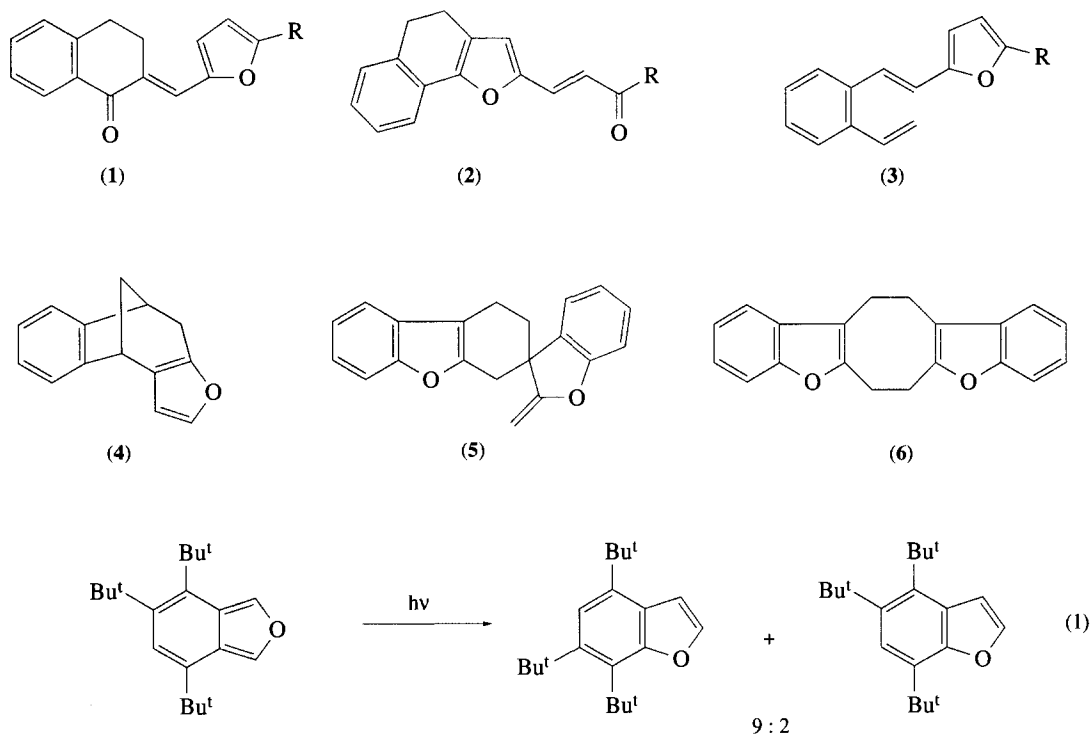
The chemistry of furan and its benzologues and their derivatives have been reviewed from time to time and valuable introductions to the chemistry of these heterocyclic systems can be found in

textbooks by Gilchrist (B-92MI 206-01) and Joule *et al.* (B-95MI 206-01). The synthesis and reactions of Dewar furan have been reviewed (92MI 206-02). A wide variety of cyclopentenones are found in natural products and the synthesis of 1,4-dicarbonyl compounds and cyclopentenones from furans makes the preparation of suitably substituted furans attractive. This topic is the subject of an interesting review (94S867). The use of aromatic heterocycles, including furan derivatives, in the synthesis of natural products (86CRV795, 95COS(2)1), for example, furanosesquiterpenoids (94OPPI), and polyaromatic hydrocarbons involving isobenzofurans (88T2093) have also been reviewed. A number of new routes to furans have been reported including examples based on reactions of but-2-yne-1,4-diol. 3-Tributylstannylfuran can be prepared in two steps by what is effectively the addition of tributylstannane across the triple bond followed by oxidation with pyridinium chlorochromate (85S898). A route to 3-substituted 4-methylfurans starts with the monomethyl ether of but-2-yne-1,4-diol (87JOC2315) and highly functionalized 3-aryl-4-arylsulfonylmethyl-5-methoxyfuran-2-carboxamides in reactions involving the cyclization and methylation of products from the interaction of isonitriles with arylglyoxals and arylsulfonylacetic acids (94LA527).

2.06.2 FULLY CONJUGATED RINGS: REACTIVITY AT RING ATOMS

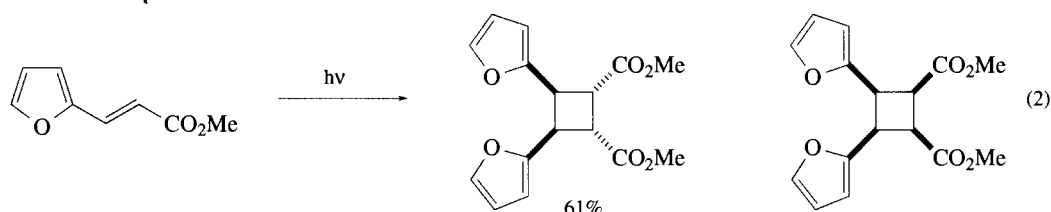
2.06.2.1 Thermal and Photochemical Reactions Involving No Other Species

The photo-rearrangement reactions of a number of furan derivatives demonstrate the stability of the ring system and examples include the conversions of (1) to (2) (90T7831) and (3) to (4) (89H(29)679). The dimerization reactions of 2,3-dimethylenbenzofuran that give rise to the compounds (5) and (6) also demonstrate the stability of the benzofuran ring system. The photo-rearrangement of the very stable 4,5,6- and 4,5,7-tri-*t*-butylisobenzofurans (Equation (1)) give rise to benzofuran derivatives (89TL103, 92BCJ932).



3-Substituted furans can be prepared in a single step by a cascade involving Diels–Alder retro-Diels–Alder reactions of 4-phenyloxazole and alkylacetylenes (83TL2473). More highly substituted furans have also been prepared by the same strategy, for example, 2,5-disubstituted furans have been converted into the related dimethyl 2,5-disubstituted furan-3,4-dicarboxylates by heating the furan with dimethyl acetylenedicarboxylate (88H(27)1115). Flash vacuum pyrolysis has also been used to effect a number of interconversions that result in the formation of furans (86SC1509) and benzofurans (86JOC4208). In the latter case the pyrolysis of (2-methyl-3-benzofuryl)methyl benzoate

gave a 30% yield of a 4.1:1 mixture of two dimers derived from 2,3-dimethylene-2,3-dihydrobenzofuran by 4 + 2 and 4 + 4 reactions respectively. Other examples include the generation of 2,3-dimethylene-2,3-dihydrofuran from 3-chloromethyl-2-methylfuran at 873 K and its subsequent photoisomerization to dihydro-2,3-cyclobutafuran in an argon matrix at 16 K by irradiation at 300 nm. Whereas the irradiation of furylacrylic acid in solution only gave rise to *E*- to *Z*-isomerization, irradiation in the solid state gave the analogues of β -truxinic and α -truxillic acids slowly. On the other hand the photodimerization of methyl *E*-furylacrylate gave good yields (<90JCS(P1)2999> of the products shown in Equation (2).

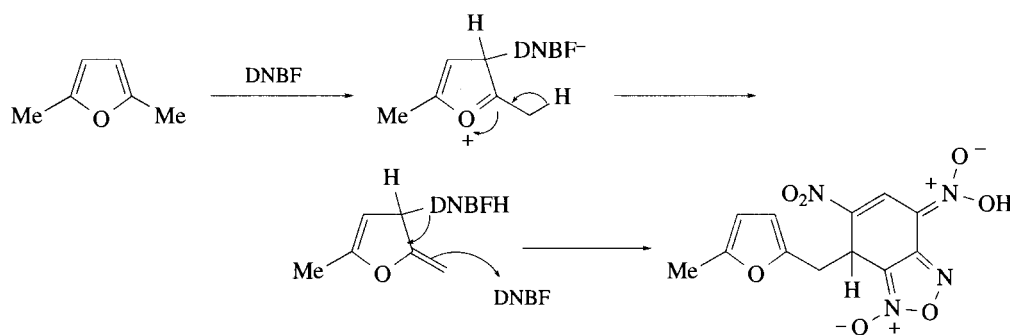


The degenerate photochemical rearrangement using visible wavelength irradiation of 4:7,10:13-diepoxy[15]annulenones has been studied by ^{17}O nuclear magnetic resonance spectroscopy (<91CC1775>). The introduction of the heavy isotope was achieved using acid catalyzed exchange which occurred exclusively at the carbonyl oxygen atom.

2.06.2.2 Reactions with Electrophiles

2.06.2.2.1 Reactivity and directive effects

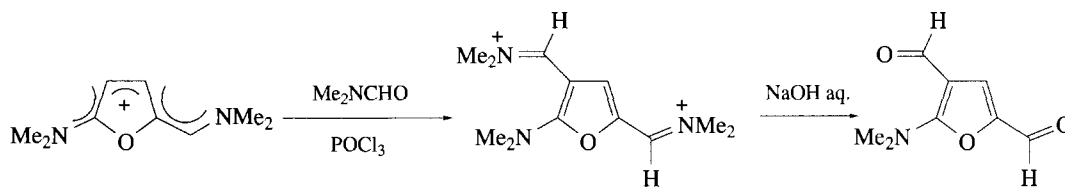
Positional selectivity in electrophilic substitution reactions, including methods of orientation control have been reviewed (<94H(37)2029>). General views concerning the mechanism of electrophilic side-chain reactions of 2,5-dimethyl five-membered ring heterocycles have been reconsidered (<84JOC4363>). 4,6-Dinitrobenzofuroxan (DNBF) and 4,6-dinitrobenzofurazan (DNBZ) take part in reactions of 2,5-dimethylpyrrole, 2,5-dimethylthiophene, and 2-methylfuran carried out in DMSO and behave as conventional electrophiles. Substitution occurs at the unsubstituted β -position. On the other hand the only observed process 2,5-dimethylfuran undergoes is substitution at a methyl group. The proposed mechanism (Scheme 1) is similar to that frequently invoked to explain side chain reactivity, for example, at the benzylic 2-position in 2,3-dialkylindoles.



Scheme 1

The positional reactivity of dibenzofuran in electrophilic substitution reactions depends on the electrophile that is used. Reaction occurs mostly at the 2- and 3-positions but the ratio of the two products varies (<91JOC4671>). The reaction of cyanogen bromide catalyzed by aluminum chloride gives an 80% yield of the 2-substituted product together with 15% of the 3-cyano-derivative (<92ACS312>). Oxidative acetoxylation of dibenzofuran occurs predominantly at the 3-position (~60%) together with attack at the 1-position (~30%) (<92ACS802>). In this latter reaction, the attack by acetate is on the dibenzofuranium radical cation. A good yield of the product has been

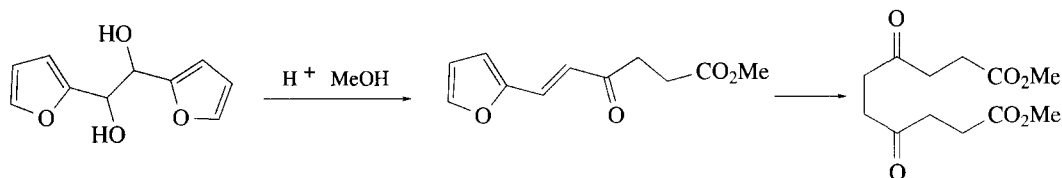
reported in the thallation of furan using phenylthallium(III) 18-crown-6 diperchlorate <92OM4352>. The vinamidium salts such as the example shown in Scheme 2 effectively functions as an enamine in reactions with other electrophiles such as the Vilsmeier reagent <84CCC1600>.



Scheme 2

2.06.2.2.2 Protonation

The basicities at both the α - and β -positions in five-membered heterocycles have been studied by ion cyclotron resonance equilibrium and bracketing experiments on deuteriated substrates. α -Protonation is preferred by 2.8–4.6 kcal mol⁻¹ for both furan and thiophene as compared to 0–2.9 kcal mol⁻¹ for pyrrole <81NJC505> and heteroatom protonation is much less favored than α -protonation. Semiempirical (MNDO) molecular orbital calculations have provided quantitative confirmation of the above conclusions. Proton catalyzed ring opening followed by recyclization is illustrated by the interconversion of furan and selenophene derivatives using either aqueous hydrolysis followed by reaction with hydrogen selenide or directly using hydrogen selenide <83KGS219>. These reactions require protonation at the β -position. Furan derivatives that are unsubstituted at the 2-position are normally polymerized by aqueous acids. On the other hand laevulinic acid derivatives can be obtained even when a 2-position is unsubstituted when a reducible functional group is also present. The redox reactions (known as the Marckwald reaction) occur readily and are exemplified in Scheme 3 <85H(23)667>. The same intermediate furylidene ketone has also been used in a new synthesis of ($\pm$)-11-deoxyprostaglandins <82JCR(S)9> and in a conversion of methyl furfurylidene pyruvate into a substituted cyclopentenone derivative.



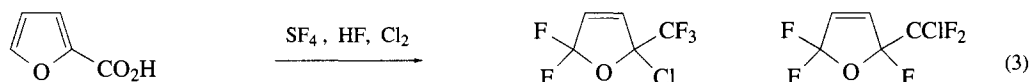
Scheme 3

2.06.2.2.3 Nitration

The nitration of furfural diacetate was discussed in the first edition of *Comprehensive Heterocyclic Chemistry* (CHEC-I) <84CHEC-I(4)599>. Reactions of nitronium acetate with furfural diacetate or methyl 2-furoate resulted in the formation of diastereoisomeric adducts <86JOC3811>. The reactions of the adducts have been compared with the analogous reactions of *ipso* nitronium acetate adducts formed from alkylbenzenes. The deacetoxylation reactions showed large primary kinetic isotope effects and mild acid induced alcoholysis transformed the adducts into 2,5-dialkoxy-2,5-dihydrofurans. In the case of the nitration of furfural in the presence acetic anhydride, 5-nitrofurfural diacetate was formed <92MI 206-03> and the highest yield reported (74%) was obtained using the fuming nitric acid–acetic anhydride system. The nitration of ethyl furan-2-carboxylate with nitronium acetate is successful <83MI 206-01>. A number of studies involving the nitration of benzofuran derivatives showed that electron releasing substituents on the benzenoid ring give rise to products in which either or both rings undergo substitution <82PJC691, 83JHC1175> and a new procedure that uses nitric acid in the presence of tin(IV) chloride in dichloromethane has been used with a series of benzofuran derivatives <83CJC2287>.

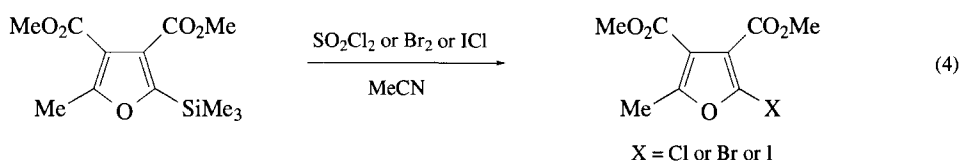
2.06.2.2.4 Halogenation

The major product formed when furan is fluorinated is *syn*-2,5-difluoro-2,5-dihydrofuran <90G749>. On the other hand, furan-2-carboxylic acid is converted by a mixture of hydrogen fluoride, chlorine, and sulfur tetrafluoride into the mixture of products shown in Equation (3) <86BSF974>.

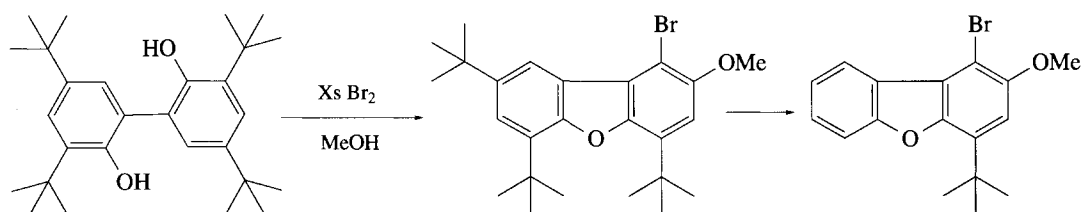


The bromination of furan when carried out in DMF using one or two mole equivalents of bromine gives 2-bromo- or 2,5-dibromo-furan <90SC3371> and a synthesis of the marine natural product bromobekkeride has been reported starting with the bromination at the 4-position of 5-methylfuran-2-carboxaldehyde <89TL1237>. Whereas 2-furylcobaloximes react with bromine and chlorine in acetic acid and chloroform principally by way of cobalt-carbon bond cleavage, ring substitution is faster than cobalt-carbon bond cleavage with 3-furylcobaloximes <84TL3255>.

Electrophilic *ipso*-substitution reactions have been used to control the position of entry of halogens. That the reagent involved is quite strongly electrophilic is indicated by reactions with furylsilanes. Ethyl 5-trimethylsilylfuran-2-carboxylate reacts in acetonitrile with sulfuryl chloride, bromine, and iodine monochloride and gives the expected *ipso*-halogeno-desilylated derivative <91CE699>. Other related silylated furan esters give similar results (Equation (4)) <91JHC853> but in dichloromethane the major product formed by the reaction of ethyl 5-trimethylsilylfuran-2-carboxylate with sulfuryl chloride was shown to be ethyl 2-chloro-3-trimethylsilylfuran-5-carboxylate <92CPB1966>. The conversion of 3,4-bis(trimethylsilyl)furan into 4-iodo-3-trimethylsilylfuran has been used as a method for the controlled introduction of a number of other functional groups and hence to give 3,4-unsymmetrically substituted furan derivatives <94LA29, 94LA689>. The reaction of 2-furyltributylstannane with iodosyl triflate and trimethylsilyl cyanide results in the formation of the di-2-furyliodonium ion <92JHC815>.



A series of bromochloro- and chlorofluoro-2,3-dihydrobenzofurans have been prepared by way of the bromochlorination of benzofuran <83G149> and it is of interest to note that *trans*-2-chloro-3-fluoro-2,3-dihydrobenzofuran undergoes *syn* elimination to give 2-chlorobenzofuran in almost quantitative yield using sodium *t*-butoxide-sodium amide in tetrahydrofuran <87JOC5494>. The bromination of dibenzofuran in the presence of iron powder gave mixtures of polybrominated derivatives with the incorporation up to four bromine atoms <82H(19)2349>. The use of *t*-butyl groups to afford temporary protection of specific positions has been used in the case of dibenzofurans. For example, the reaction of 2,2'-dihydroxy-3,3',5,5'-tetra-*t*-butylbiphenyl with an excess of bromine in methanol gave 1-bromo-2-methoxy-4,6,8-tri-*t*-butyldibenzofuran in a 68% yield. Subsequent reaction of the product with aluminum chloride in toluene at room temperature (Scheme 4) gave 1-bromo-2-methoxy-4-*t*-butyldibenzofuran, the product of partial *ipso*-proto-de-*t*-butylation, in a 74% yield <82JOC4425>.

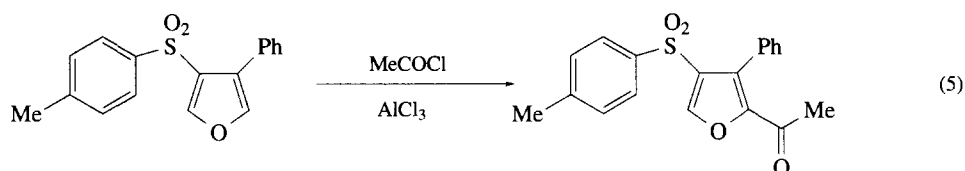


Scheme 4

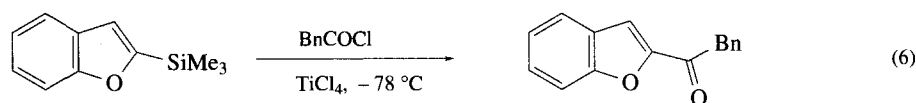
2.06.2.2.5 Acylation

The Vilsmeier formylation reactions of furan and benzofuran and their substituted derivatives have been known for some time and the results are collected in a review <93HOU(E3)36>. The majority of the reactions are carried out at quite low temperatures. However, the reaction involving furan was rather surprisingly reported <57JOC1269> to afford 2-formylfuran in a 64% yield when carried out at 100°C over a 30 h period. 2,5-Dimethylfuran was reported to be resinified by the Vilsmeier procedure but 3-formyl-2,5-diphenylfuran was obtained from 2,5-diphenylfuran in a 40% yield. Using the anhydride, pyrophosphoryl chloride, milder reactions conditions were found to be effective <93T4015>. 2-Formylfuran was obtained in a 71% yield in a reaction conducted at 0°C for 0.75 h and then at 75°C for 0.5 h. <93T4015>. Similarly 2-formyl-5-methylfuran was obtained in a 77% yield in a reaction conducted at between 0°C and room temperature.

A number of 2-acylfuran derivatives have been prepared as potential bioisosteres for 3-methylisoxazoles that show antirhinoviral activity <94JMC4177>. The acetylation reactions of phosphonoalkylfurans <85ZOB2006> proceed efficiently using acetic anhydride and phosphoric acid. 2-Acylfurans have also been prepared using phosphonic acids in reactions where the acyl residue originates from a carboxylic acid that is activated by the interaction with trifluoroacetic anhydride <82SC1121>. The complementary effects of 3-sulfonyl substitution in metallation and electrophilic substitution reactions is demonstrated by reactions of 3-phenyl-4-toluene-*p*-sulfonylfuran <86TL4123>. Whereas metallation occurs at the position adjacent to the sulfonyl residue acetylation using acetyl chloride and aluminum chloride proceeds as shown in Equation (5).

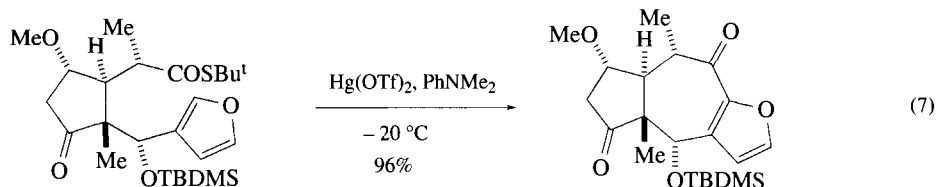


Reactions of furan using half-ester half-acid chlorides, catalyzed by tin(IV) chloride have been reported and give the expected products in moderate yields. Functional group manipulation was then used to effect the synthesis of a series of tetrahydrofuran based dicarboxylic acids that have been previously detected in human urine <85LA1168>. 2-Acybenzofurans are obtained in excellent yields using primary, secondary, and tertiary aliphatic acid chlorides in low temperature Friedel-Crafts acylation reactions using titanium tetrachloride as the Lewis acid <84T621> and the acetylation of a number of benzofuran methyl ethers and other derivatives has also been studied <84BCJ617, 84JHC177>. The deprotonation of, for example, 3-phenylbenzofuranone generally affords products that result from *O*-acylation when allowed to react with an excess of a chloroformate. Treatment of the resulting enol carbonates with dmap in halogenated solvents cause the rearrangement to the *C*-acylated isomers, presumably by way of the *N*-acylpyridinium species <87JOC5425>. Trimethylsilylation at the 2-position in benzofuran is achieved in a quantitative yield by metallation using butyllithium followed by reaction with chlorotrimethylsilane. These reactions (Equation (6)) illustrate the highly electrophilic nature of the reactive intermediates involved in acylation reactions; the lowest yields, not surprisingly, were observed in pivaloylation and phenylacetylation reactions. The reaction of 2,5-dimethylfuran with ethyl cyanoformate that is catalyzed by aluminum chloride results in the formation of a mixture of diastereomeric *ipso* addition compounds <81CJC1395>. In competitive *ipso* substitution reactions using substrates that contain both silyl and stannyl residues with weak electrophiles, the tin group is replaced selectively. The selective introduction of a 2-trimethylsilyl (initially at the 5-position) and a 4-tributylstannyl group in furan by way of 2,3-dibromofuran has been used to achieve the regioselective replacement of the tin residue by an acyl group in a manolide synthesis <88TL1173>. This example illustrates the variation of electron density that can be anticipated from the different electronegativities of silicon and tin.



High yield intramolecular acylation reactions at the 2-position of an appropriately substituted furan thiol ester has been used in natural product synthesis. Model cyclization reactions were carried out using mercury(II) trifluoroacetate <90TL1977>. However, in the step shown in Equation (7), which

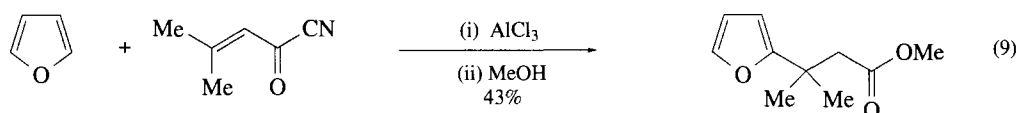
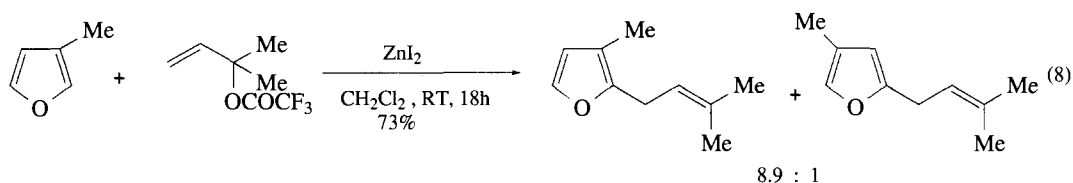
is part of a synthesis of (–)-fastigilin C, the yield of the cyclization product was disappointingly low using mercury(II) trifluoroacetate, presumably due to the nucleophilicity of the trifluoroacetate ion resulting in capture of the nascent acylium ion. Much greater success was attained using mercury(II) triflate as shown in Equation (7) <92JA8349>.

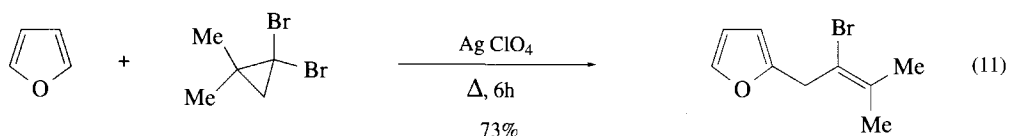
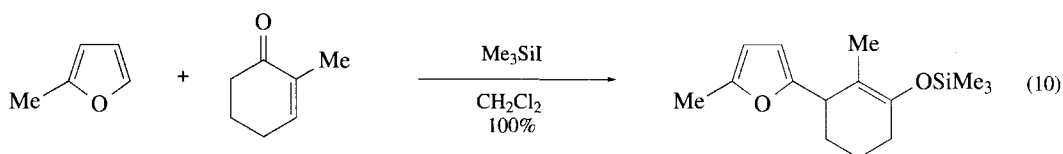


2.06.2.2.6 Alkylation

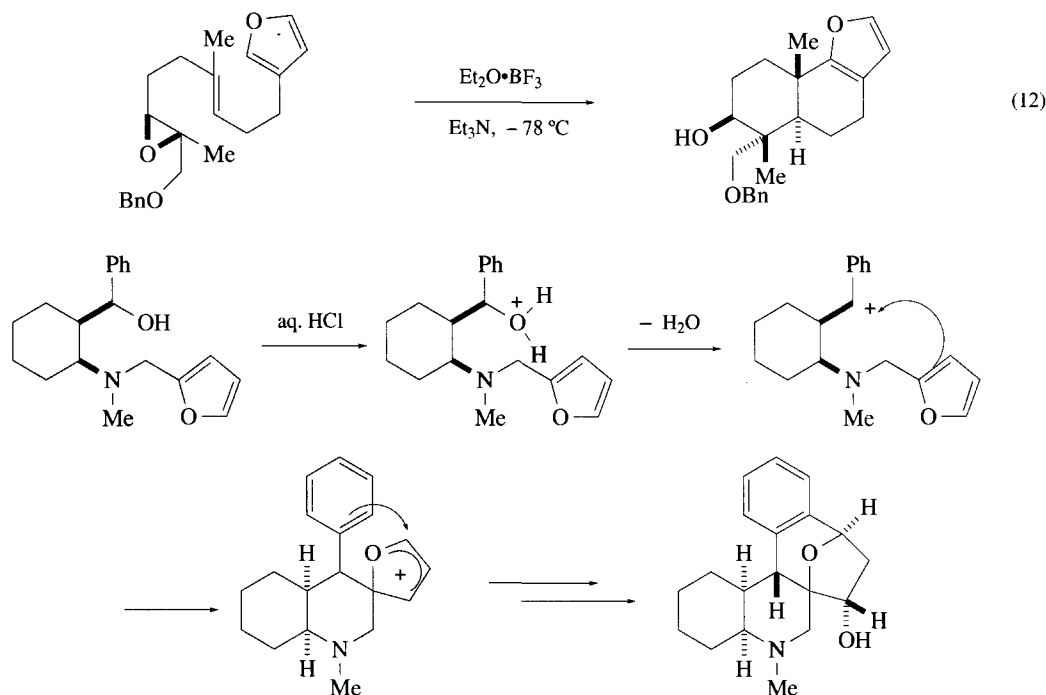
Despite the ease of resinification of furan by Lewis acids, the direct alkylation of furan and substituted furans has been achieved by using recently devised protocols. The formation of *t*-butylfurans has been reported by using a catalytic amount of iron(III) chloride in the presence of a large excess of iron(III) oxide and a source of the *t*-butyl cation. Furan has been converted directly, for example, into 2,5-di-*t*-butylfuran <82CI(L)603>. The iron(III) oxide probably has a dual function: to remove hydrogen chloride and to replenish the iron(III) chloride. 2,5-Di-*t*-butylfuran has also been prepared in 51% yield using *t*-butyl bromide in the presence of silica and sodium carbonate <84JOC4161>. A mixture of furan and 2-hydroxymethylfuran react in the presence of Lewis acids to give oligomers that have been cyclized to furan based calixarenes <93CC1029>. The Lewis acid catalyzed reactions of alkylfurans that result in the substitution by additional *t*-butyl groups does not allow the formation of tetra-*t*-butylfuran. Thus 4-*t*-butyl-2-methylfuran gives 2,3-di-*t*-butyl-5-methylfuran by reaction with *t*-butyl chloride and aluminium chloride and 2-*t*-butyl-5-methylfuran gives 3,5-di-*t*-butyl-2-methylfuran <90CC460>. This latter result is reminiscent of the acylation of 2-*t*-butyl-5-methylfuran using acetic anhydride and zinc chloride which gave 3-acetyl-5-*t*-butyl-2-methylfuran <72JCS(P1)2372>.

New methodologies for the generation of carbon electrophiles involving, for example, treating allylic trifluoroacetates with zinc halides <83AG(E)719, 83HCA828>, also allows reactions to be carried out without polymerization of the furan (Equation (8)) and 1,3-bis(seleno)-propenes have been shown to be valuable precursors of selenium stabilized cations which undergo electrophilic substitution reactions with furan in a highly stereoselective manner <83TL3911>. The alkylation of 2-*t*-butyldimethylsilyloxy-3-methylfuran and allylic bromides has been used in syntheses of furano-sesquiterpenes <90TL5741>. The alkylation with a tertiary alkyl group by effectively carrying out a Michael type of addition to an activated α,β -unsaturated ester is also useful (Equation (9)). The conjugate addition of furans to enones also proceed under mild conditions where polymerization is not a problem and using 2-methoxyfuran provides a route to butenolides. The reaction shown in Equation (10) proceeds by way of the very unstable γ -iodo-enoltrimethylsilyl ether <83TL2727>. The ring opening rearrangement of cyclopropyl halides proceed to generate allyl cation intermediates under stereoelectronic control and the intermediates generated in silver ion induced reactions are trapped efficiently by furan <84BCJ1165> as shown in Equation (11).



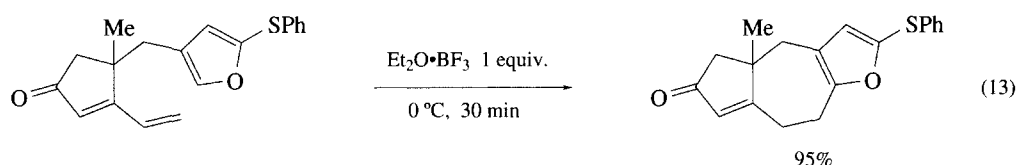


Intramolecular alkylation in which the initiation step involves electrophile induced epoxide ring opening and the termination step involves capture of the carbon electrophile by a furan has been developed ^[83JOC4572] and used in a formal total synthesis of optically active nonracemic (+)-aphidicolin ^[88JOC4929]. The use of a number of protective groups and Lewis acids was investigated before the protocol shown in Equation (12) was found to give satisfactory results. The use of a protected chiral alcohol located α - to an alkene has been used to control the chirality in an alkene cyclization triggered by means of mercury(II) triflate ^[93CPB791]. The cyclization of a chiral ethyl 3-(carbamoyloxy)-7-(3-furyl)-4-methyl-4-heptenoate gave the bicyclic product in 73% yield with essentially complete chirality transfer. A complex series of cyclization reactions that are triggered by the generation of a benzylic cation lead overall to the isomerization product that is shown in Scheme 5 and probably involve the steps that are indicated ^[86JOC5002].

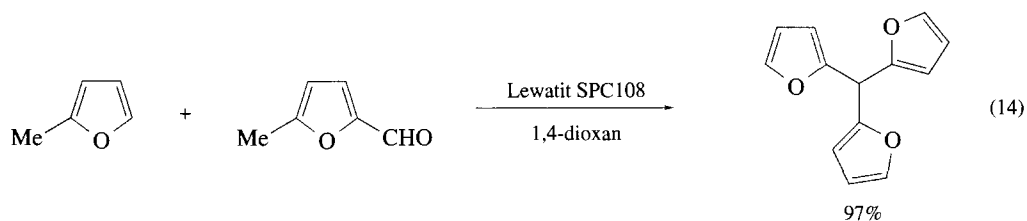


Scheme 5

The competition between Michael addition of α,β -unsaturated ketones and Diels–Alder reactions involving furan and 2-methylfuran is affected by the catalyst used. Methylvinyl ketone was shown to give the alkylation product with furan and 2-methylfuran in the presence of silica gel ^[88TL175]. Bis(alkylated) products have also been obtained in reactions of 2-methylene-1,3-dicarbonyl compounds ^[90H(31)1699]. An intramolecular proton catalyzed alkylation reaction of an α,β -unsaturated ketone provided a straightforward synthesis of norpinguisone ^[90TL4343] and in the example shown in Equation (13) the cyclization reaction involved an $\alpha,\beta,\gamma,\delta$ -dienone ^[94TL4887]. The use of 1,4-dimethoxy-1,4-dimethylcyclohexa-2,5-diene as a masked *p*-xylene equivalent has been reported and results in the formation of 2-(2',4'-dimethylphenyl)furan ^[91T313].

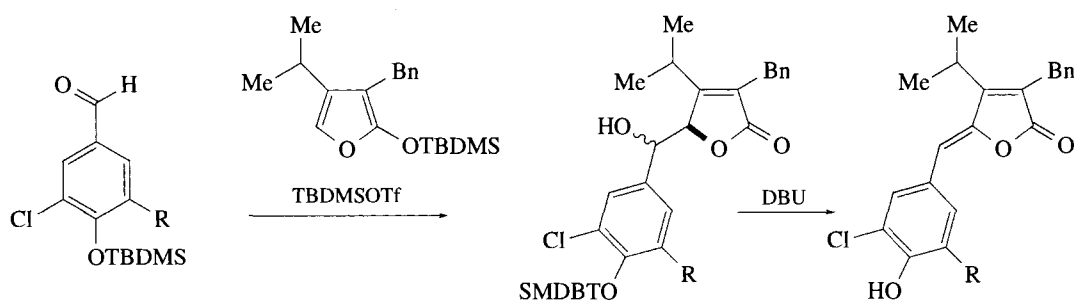
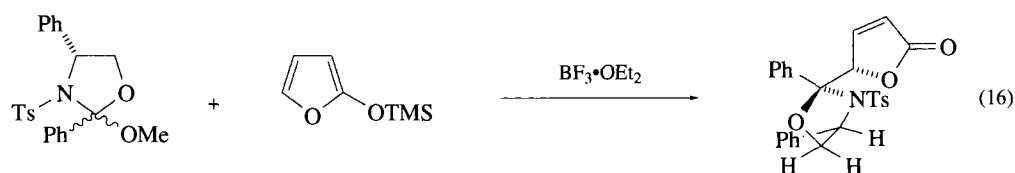
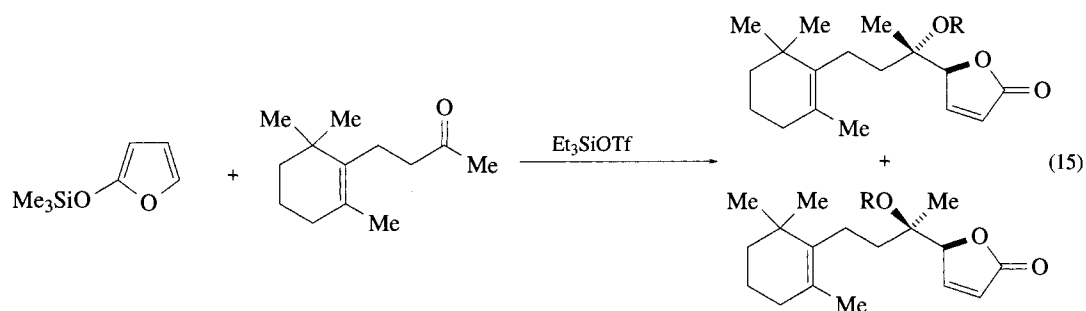


The activation of saturated aldehydes to produce difurylmethane derivatives has been effected using microporous ion-exchange resins in dioxan. The reaction of paraformaldehyde with 2-methylfuran in the presence of Lewatit SPC108, which contains about 10% of water, gave 2,2'-dimethyl-5,5'-difurylmethane in 98% yield, and a reaction of 2-methylfuran with 5-methylfuran-2-carboxaldehyde gave the triarylmethane shown in Equation (14) <89SC3169>. The use of other ion exchange resins has also been reported, for example, in reactions of 2-pentenylfuran with various aliphatic and aromatic aldehydes <82C398>. An effective method has been described for the synthesis of unsymmetrical difurylmethanes and difurylalkenes by way of phosphorus substituted 2-furylmethanol derivatives <85TL6399>. The alkylation of 2-methylfuran with methyl chloromethoxyacetate at room temperature in THF using zinc chloride as the Lewis acid gave the expected methyl diarylacetate in a 46% yield, evidently the first formed product—methyl α -methoxy- α -5-methylfurylacetate—reacts further via a benzylic type cation. The use of bis(trimethylsilyl)acetamide as a proton scavenger then allowed methyl α -methoxy- α -5-methylfurylacetate to be isolated, albeit in only a 25% yield <91TL6171>. Cyclohexa-2,5-dienone derivatives function as masked electrophiles that are activated with boron trifluoride etherate and react with furan to give 2-aryl furan derivatives <87JCS(P1)1771>.

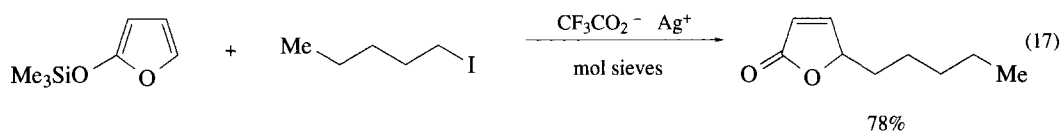


2-Trialkylsilyloxyfuran derivatives are protected butenolides and may be alkylated to afford a range of 4-substituted butenolides using an aldehyde activated by a Lewis acid. A good recent example is the efficient four step synthesis of neopatulin from 4-*t*-butyldimethylsilyloxymethyl-2-*t*-butyldimethylsilyloxyfuran and 2-formyldithiane <94TL5769>. Dicobalt hexacarbonyl complexes of propargyl cations also alkylate 2-trimethylsilyloxyfuran to give butenolides <91H(32)949>. Reactions with *N*-benzylimines give the δ -amino- α,β -unsaturated γ -lactones <92T727>. The *N*-benzylaldimine derived from isopropylidene glyceraldehyde has been converted by that protocol into an iminoalditol after several further steps <92T727>. Perfluoroalkylbutenolides have also been prepared by the reaction of a perfluoroperoxydicarboxylic anhydride with 2-trimethylsilyloxyfuran <93JCS(P1)501>. Using triethylsilyl trifluoromethanesulfonate gave predominantly the *threo*-alcohol whereas tetrabutylammonium fluoride gave reversed diastereoselectivity <87TL4037>. The reaction of the dihydro- β -ionone shown in Equation (15) was used in a synthesis of the ichthiotoxic γ -lactone cavernosine as the racemate <87TL4041>. Once again trialkylsilyl trifluoromethanesulfonates such as triethylsilyl triflate were found to be the best Lewis acids for the activation of the carbonyl group. The generation of heteroatom stabilized carbenium ions, for example, from a chiral 2-alkoxyoxazolidine, and their reactions with 2-trimethylsilyloxyfuran allows the diastereoselective synthesis of butenolides as exemplified in Equation (16) where the major diastereomer is shown <94TA1745>. It is not necessary to separate the diastereomeric oxazolidines since they both give rise to the same planar cation. The first total syntheses of nostoclide I and II have been reported <94TL7897> starting with butenolide and involving as the final steps the aldol condensation and dehydration shown in Scheme 6. Primary alkyl iodides have also been shown alkylate 2-trialkylsilyloxyfuran derivatives <88CC364> in the presence of silver trifluoroacetate, exemplified in Equation (17), a procedure that has been used in the synthesis of the aroma components of whisky and cognac lactones <89HCA1362>. Reactions with allyl halides such as 3,3-dimethylallyl bromide have been used in synthesis, for example, of racemic eldanolide <87TL949>. The alkylation of a masked butenolide at position-3 rather than at position-5 has been achieved using a number of methods, including the use of tin or boron derivatives <88CC1595> and reactions of 2-nitrofuran <88JCR(S)228>. Furan-2-one itself reacts with chloroformates to give good yields of 2-furyl esters in the presence of triethylamine <86SC69>. 2-Furylmethyl

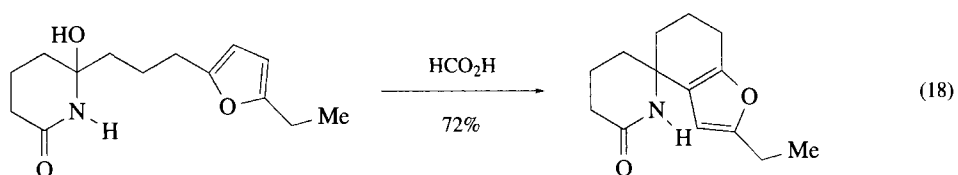
chloride is alkynylated using lithium trialkyl-alkynylboronates which may then be converted into ketones by oxidation with alkaline hydrogen peroxide (93MI 206-01).

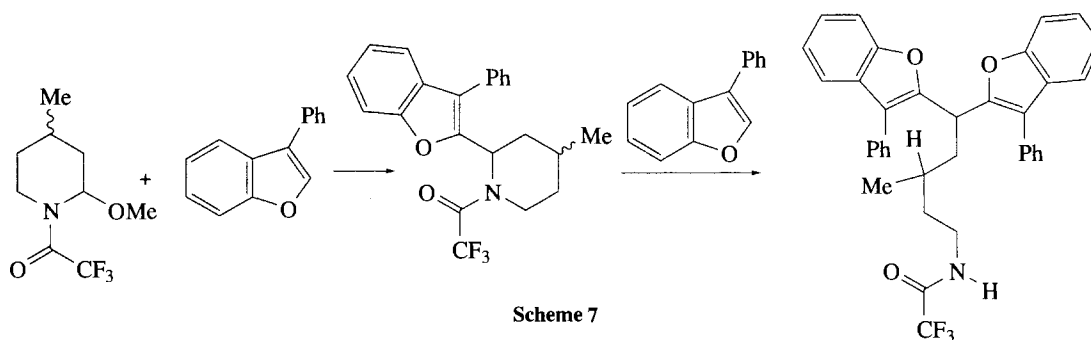


Scheme 6

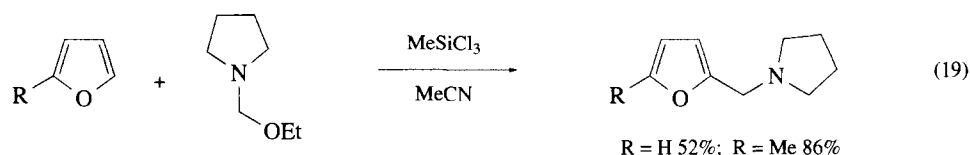


Alkylation reactions involving iminium and acyliminium ions have been reported. Although the latter ions (94JHC679) are more electrophilic than the former, both types undergo reactions with furan and benzofuran derivatives. Intramolecular reactions of acyliminium ions have found their use in natural product synthesis in which imides such as glutarimide are converted into carbinolamides and hence into an acyliminium ion (85TL5347). The step shown in Equation (18) was used in the synthesis of racemic perhydrohistrionicotoxin (87TL2495). Remarkably, the generation of the acyliminium ion from the isomeric mixture of 4-methyl-2-methoxy-1-trifluoroacetyl piperidines shown in Scheme 7 resulted in the isolation of the expected product in low yield together with, as the major product, the bis(benzofuran) derivative (85BSB39). Presumably the second alkylation step proceeds because the protonated first formed product fragments to a lower energy benzylic-type cation.

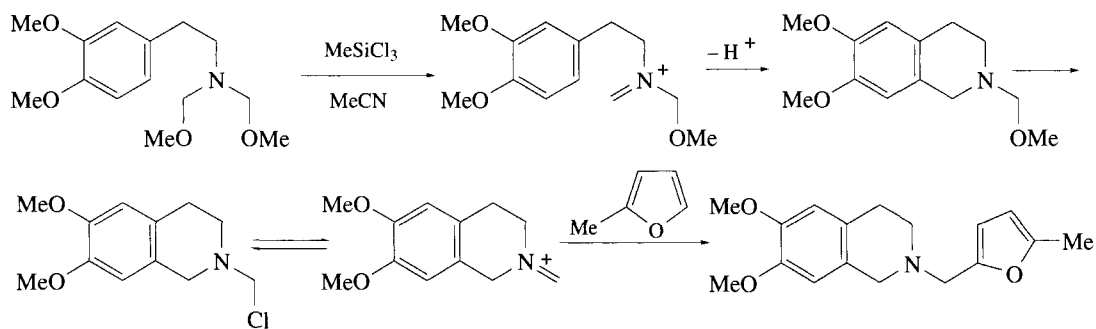




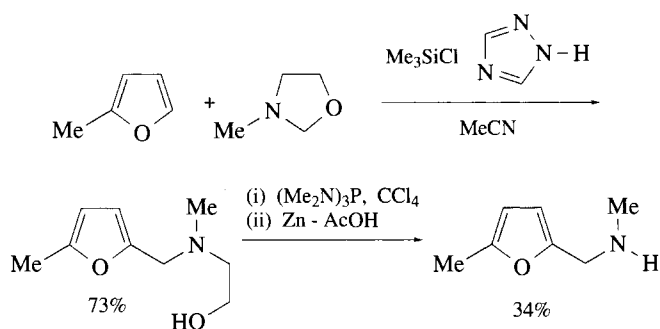
The classical Mannich reaction using 2-methylfuran proceeds efficiently to afford tertiary 2-*N,N*-dialkylaminomethyl-5-methylfuran derivatives. However, most remarkably, it fails using furan itself <59JA1209>. The fact that thiophene reacts with *N,N*-dimethylmethyleniminium chloride suggested that furan should also react with preformed methyleniminium salts and this has been confirmed <88TL2377> in a number of reactions in yields ranging from 48–74%. 2-Methylfuran also gives good yields of the expected tertiary amines with preformed iminium salts. It is possible to activate aminals (bis(*N,N*-dialkylamino)methanes) and aminol ethers (α -*N,N*-dialkylaminomethyl ethers) using a number of electrophiles. With unsymmetrical precursors the conjugate base of the stronger acid is the better leaving group and so aminol ethers are easier to activate than aminals. Satisfactory yields of Mannich bases have been obtained using furan in “*in situ*” reactions with chlorosilane derivatives. For example, using chlorotrimethylsilane and ethoxypyrrolidinylmethane gave with furan and 2-methylfuran the Mannich bases in 52% and 86% yields respectively <88CC1161> as shown in Equation (19). On the other hand, 2-methylfuran did not give a Mannich base using di-*N*-pyrrolidinylmethane in the presence of chlorotrimethylsilane, but did give the expected product using dichlorodimethylsilane. Ethoxypyrrolidinylmethane also gave 2-methyl-5-*N*-pyrrolidinylmethylfuran in 68% yield in a reaction using sulfur dioxide in acetonitrile to produce a reactive electrophile <88TL2997>. Cascade reactions have been carried out using 3,4-dimethoxy- β -phenylethyl-bis(methoxymethyl)amine <90SL617>. The reagent together with 2-methylfuran gave the product shown in Scheme 8 in 65% yield. The reaction evidently proceeds by way of two iminium ions, the first taking part in a Pictet-Spengler reaction, the intramolecular version of the Mannich reaction, and the second in the reaction with 2-methylfuran. The possibility of functionalizing electron rich aromatic compounds using oxazolidines has also been studied. 2-Methylfuran was shown to react with 3-methyl-1,3-oxazolidine in the presence of chlorotrimethylsilane in a reaction which was improved by the addition of a catalytic amount of 1,2,4-triazole <89TL1433>. A similar reaction using *t*-butylchlorodimethylsilane gave the product with the alcohol function protected. Removal of the hydroxyethyl residue by conversion into the chloroethyl group followed by reaction with zinc provides a route to secondary amines as shown in Scheme 9. A number of dihydro-1,3-benzoxazines have been shown to function as precursors to Mannich reagents and give good yields of the expected products in reactions with, for example, 2-methylfuran <94MI 206-01>. Like 5-bromo-1-piperidinylmethylisatin <68JPS1097>, 1-piperidinylmethylbenzotriazole functions efficiently as a Mannich reagent precursor <92T4971> and, using zinc chloride as the Lewis acid, reacts with 2-methylfuran to give the expected product in 87% yield.



Although the formation of secondary amines by way of Mannich reactions is much less well documented than is the case for the formation of tertiary amines, two other methods have been reported which provide access to secondary amines. In addition to the use of benzotriazole in reactions leading to the formation of tertiary amines, it has also been used as an efficient leaving group in reactions leading to the formation of secondary amines <92T4971>, as shown in Equation

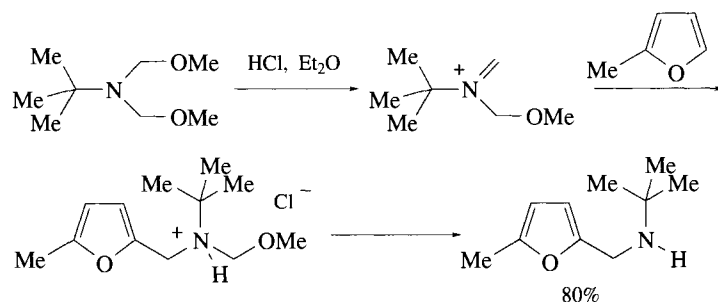
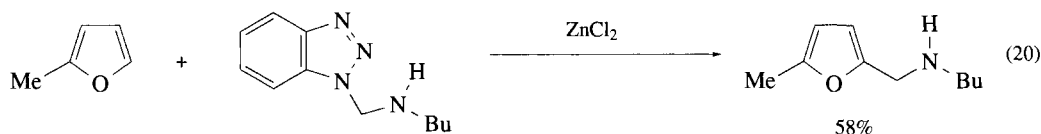


Scheme 8



Scheme 9

(20). The use of alkoxyethyl groups as precursors to iminium salts and simultaneously to protect the amino group, as temporary protection, has also been studied (90TL4229). Iminium salts derived from bis(aminol) ethers by interaction with hydrogen chloride allowed the isolation of secondary amines after hydrolytic removal of the protecting alkoxyethyl group, as shown in Scheme 10. However, good yields of the secondary amines were only really obtained where the other alkyl group was sterically demanding. A route to 2-furylglycine derivatives involves the formation of the *p*-toluenesulfonylimine from methyl glyoxylate and *p*-toluenesulfonylimine which then reacts with the furan derivatives in room temperature reactions (91SL29).



Scheme 10

2.06.2.2.7 Reactions with aldehydes and ketones

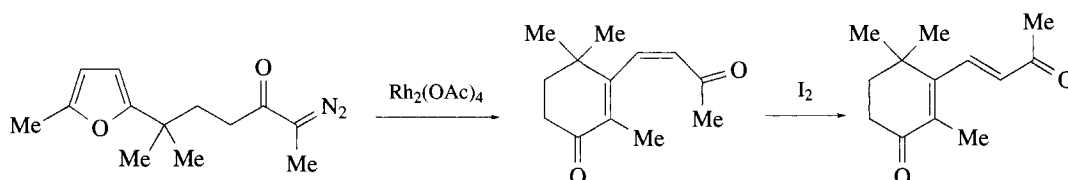
A systematic investigation of the various parameters that affect the condensation of furan with acetone has been reported (85JCS(P1)973). The dramatic yield enhancement for macrocycle formation in the presence of metal ions that has been previously ascribed to a template effect was shown to result from pH effects. Optimization of the reaction conditions allowed the isolation of a number of products in significantly improved yields. In the case of the reaction with cyclohexanone a cyclic tetramer was formed in a single reaction. That was the first example of the one pot formation of a cyclic tetramer. The tetrameric furan–ketone macrocycles do not show any binding interaction with metal ions. However, the tetrahydrofuran–acetone macrocycle does form an isolable 1 : 1 complex with lithium perchlorate. High pressure reactions of furans with carbonyl compounds, including 2,5-dimethylfuran, have been studied (83CC1447). Stereochemical aspects of reactions, for example, of diethyl mesoxalate (89JOC4469), and the factors controlling intramolecular addition reactions (88TL5017), have also been studied. Michael addition reactions of furans, including 2-methoxyfuran (87H(25)399), and benzofurans (94BSF900), have been reported using Lewis acid catalysis.

2.06.2.2.8 Reactions with diazonium salts and diazo-compounds

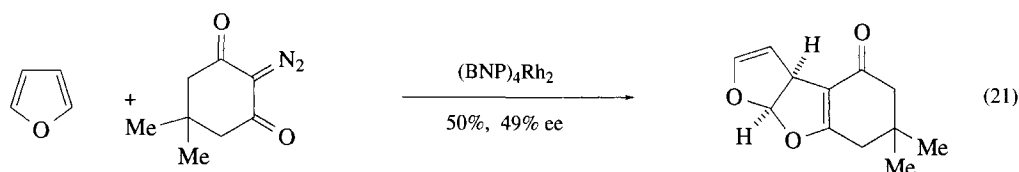
2-Arylfuran-5-carboxaldehyde derivatives can be prepared from furfural and arenediazonium salts (86ZOR2331, 93CCC1905) and 2-arylfuran-3-carboxylic acids are the major products in reactions of furan-3-carboxylic acid (89CCC215). Azo-coupling reactions with simple furan derivatives are uncommon but *ipso* substitution involving the replacement of iminium groups have been reported for 5-(*N,N*-dialkylamino)-2-furfurylidene-*N',N'*-dialkylmethyleiminium salts (88CCC1053) as have Meerwein type reactions of, for example, 5-nitro-2-furylethylene derivatives (83CCC3130).

Both inter- and intra-molecular metal catalyzed reactions of aliphatic diazo-compounds have yielded interesting results. The cyclopropanation of furan using diazomethane in the presence of either copper(I) chloride or bromide leads to the formation of 2-oxabicyclo[3.1.0]hexene in good yield when using an excess of furan (86S1059) together with a small amount of the bis(adduct) in the stoichiometric reaction. The use of rhodium catalysts has also featured intermolecular cyclopropanation reactions using diazoketones and diazoesters (88IZV2833). The reaction of an excess of ethyl diazoacetate with 1,2-di-(2-furyl)ethane and rhodium acetate followed by reaction with boron trifluoride gave a diketo-diester which was converted into corticocin after a number of additional steps (90JOC6203). Rhodium carbene complexes (B-89MI 206-01) are intermediates. Molybdenum carbonyl carbene complexes have also been involved in reactions both where furan rings are formed and where furan rings are involved as part of the reacting system (92BSF579). Reactions of alkynes with chromium carbonyl carbene complexes have also figured prominently in new routes to benzofuran derivatives (87TL1381, 88JA7419, 88OM2346, 88TL2513, 89JOC3625) and in reactions where the major cyclization product is a cyclopentanone fused furan (88TL3403). Aminobenzannulation has been achieved using metathesis reactions of isonitriles with chromium carbene complexes derived from 2- and 3-arylfurans. The known reaction of isonitriles with carbene complexes that result in the formation of, for example, dienylketenimines chromium pentacarbonyl complexes, sets up an electrocyclic process that gives a tautomer of an aminobenzene (92JA8723). Diazoimides, prepared from imides under standard diazo transfer conditions, give transient isomünchenone dipoles on treatment with rhodium(II) acetate which undergo dipolar cycloaddition reactions, for example, across a tethered furan ring system (94JOC7072). The efficiencies of the reactions is dependent on the length and nature of the tether connecting the dipole with the dipolarophile. Acyclic diazoketones or diazo- β -diketones react with furans to give $\alpha,\beta,\gamma,\delta$ -dienals or dienones, but reactions of cyclic diazo- β -diketones using rhodium acetate result in annelation (89HCA756, 91JOC6269). A short synthesis of the naturally occurring dibenzofuran, rusco-dibenzofuran, has been reported in which the key step involved the formation of a 2-isopropenylbenzofuran by the reaction of an *o*-halophenol with copper(I) isopropenylacetylide (83JCS(P1)2927). Diazocyclohexane-2,6-dione also reacted with 2-methylbut-1-ene-3-yne in the presence of rhodium acetate and gave 2-(2-propenyl)-6,7-dihydrobenzofuran-4-(5*H*)-one in 82% yield which was carried on to 5-acetyl-4-hydroxy-2-(2-propenyl)benzofuran (isoeuparin) (94TL6229). Furans with side chains at the 2-position that terminate with a diazomethyl group next to a ketone carbonyl group are converted into 2-cycloalkenones on reaction with rhodium acetate (87HCA1429) and the formyl analogues undergo similar reactions (89JOC299). The reactions proceed by a cyclopropanation—ring opening sequence. This type of rhodium carbenoid chemistry has been used in the synthesis of a number of terpenes (89HCA756) and retinol-carotene fragments (90JOC6203). For

example, (*E*)-4-oxo- β -ionone, a constituent of *Osmanthus* absolute oil, has been synthesised using the route outlined in Scheme 11. The new chiral rhodium catalyst derived from bina-phthol(dirhodium tetrakis(binaphtholphosphate)) has been used in dipolar cycloaddition reactions which proceed with reasonable asymmetric induction as shown in Equation (21) <92TL5987>. Although not strictly relevant to this section it is of interest to note that nitrene insertion has been studied to produce pyrrolofurans by the thermolysis of suitably positioned azides <94CCC473>.

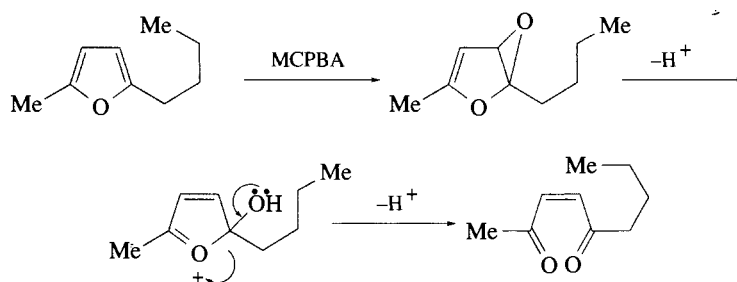


Scheme 11

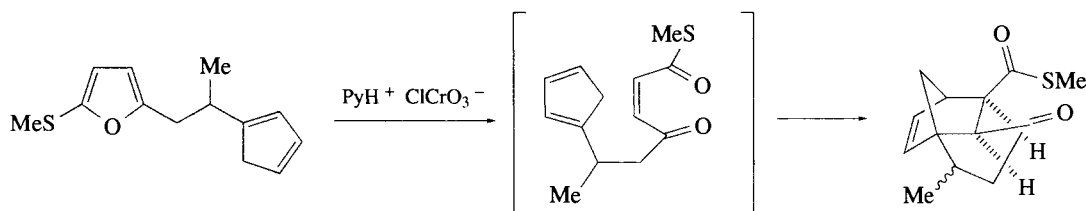
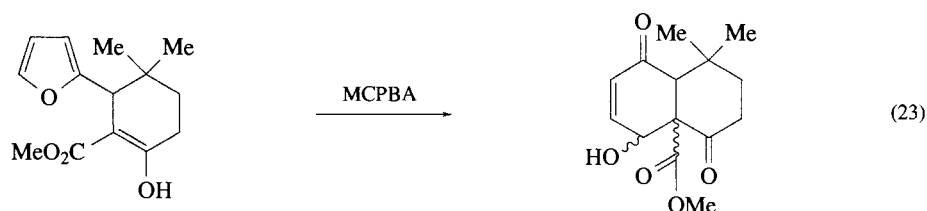
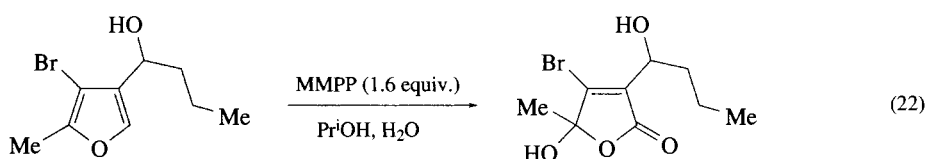


2.06.2.2.9 Reactions with oxidants

The well known electrochemical methoxylation of furan in methanol that results in the formation of 2,5-dimethoxy-2,5-dihydrofuran has been reported using a platinum-solid polymer electrolyte prepared by the deposition of platinum on both surfaces of Nafion 415. The addition of a small amount of bromine improved the efficiency of the reaction <84MI 206-01>. Other examples of the electrochemical oxidation of furan derivatives have been reported <83MI 206-02>, including the concurrent cyanation and methoxylation of methyl substituted furans <87BCJ229>. The oxidation of 2,5-disubstituted furans to give *Z*-1,2-diacylethenes has been reported on a number of occasions using peroxycarboxylic acids and includes the use of the method to introduce a radiolabel into the methyl group in *Z*-3-acetylacrolein <84MI 206-02>. The peracid oxidation of furan systems has been reviewed <90MI 206-01>. The most frequently reported acid used is MCPBA and substrates oxidized contain a range of functionalities <83JOC2606, 84JOC1284, 87TL1203, 87TL2297, 89JOC5186>. It is assumed that the first step involves epoxidation as shown in Scheme 12 <83CL1771>. An important mechanism in the toxicity of furans involves the cytochrome P450 monooxygenase catalyzed activation to form highly electrophilic products within the target tissues. The products are evidently identical to the MCPBA oxidation products obtained from 2- and 3-methylfuran. The unsaturated aldehydes acetylacrolein and methylbutenedial have been identified as conjugates bound to tissue macromolecules in pulmonary and hepatic microsomal systems <84SCI884>. Very high yields of *Z*-1,2-diacylethenes have also been reported <90TL7669> using magnesium monoperoxyphthalate (MMPP) <93MI 206-04>. The oxidation shown in Equation (22) was achieved in high yield using MMPP as part of a synthesis of bromobekerelide, a product isolated from a marine red alga <89TL1237>. An important cyclization step in the synthesis of (–)-fastigilin C <88TL4521, 90TL1977, 92JA8349> has been mentioned previously and in another step the oxidation of the 2-trimethylsilylfuran derivative was achieved in very high yield using peroxyacetic acid <92JA8349>. The ring cleavage of benzofurans have also been reported using MCPBA <94CB941>. Chemoselective oxidation of alkadienylfurans using MCPBA have used the furan residue as a masked dienophile for use in intramolecular Diels–Alder reactions that lead to hydrindenones <85TL1367> and β -dicarbonyl substituted furans can be oxidized to give, as the final product, the aldol derivative (Equation (23)) <93SL163>. The possibility of generating a dienophile in the presence of a diene by the oxidation of a suitably substituted furan has been used to establish a cascade as shown in Scheme 13, <87CC898>, presumably via the intermediacy of the γ -ketothiol ester. This procedure is a development from the previously described oxidation of thio-substituted furans using pyridinium chlorochromate <84S280>.

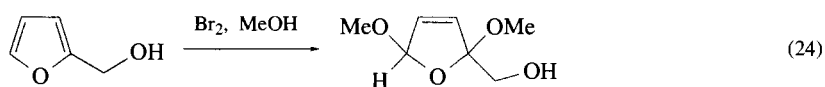


Scheme 12



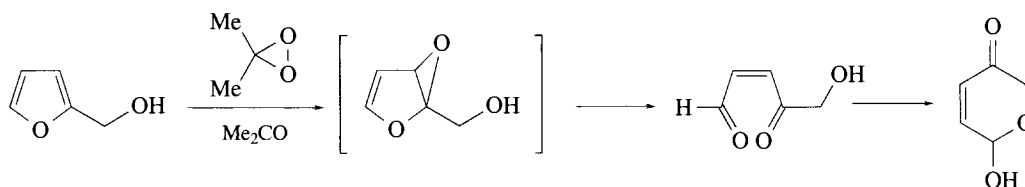
Scheme 13

The oxidative rearrangements of 2-furyl ketones that result in the formation of oxidized spiroacetals were carried out using *N*-bromosuccinimide in aqueous tetrahydrofuran and provide an interesting entry into highly functionalized furan derivatives ^[89JOC2044]. The use of *N*-bromosuccinimide has also been recommended for the oxidation of, for example, 2-hydroxymethylfurans to 2*H*-pyran-3(6*H*)-ones ^[86JOC2725], but others have found the method unreliable ^[91JCS(P1)929]. On the other hand, the well known oxidation of furan by bromine in methanol (Equation (24)) ^[71T1973] to the acetal was found to be more effective ^[91JCS(P1)929]. The oxidation of 2-trimethylsilyloxy-derivatives of furan and benzofuran in the presence of nucleophiles using hypervalent iodine compounds such as iodosobenzene, gives 5-substituted furanones or 3-substituted benzofuranones ^[89TL3019]. 5-Methyl-2-furanone also results from the oxidation of 2-methyl-5-furyl-phenylselenide ^[86SC877].



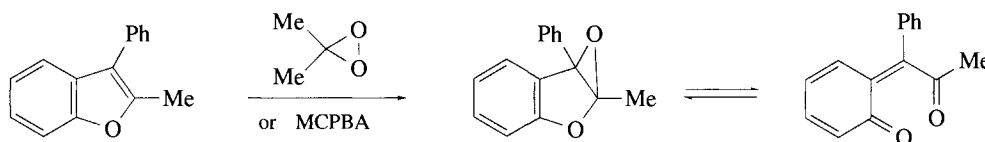
Dimethyldioxirane (DMD) is a mild neutral oxidizing agent and has found use in reactions with furan and benzofuran derivatives. Maleic dialdehyde (*Z*-1,2-diformylethene) is very unstable but is nevertheless formed in high yield and high purity when furan is oxidized with DMD in acetone ^[91CC1553]. One of the most widely studied oxidative transformations of furan derivatives is concerned with the conversion of 2-hydroxymethylfurans into 2*H*-pyran-3(6*H*)-one derivatives and the use of this process in natural product synthesis has been reviewed ^[92OPP97, 93MI 206-02]. An interesting variation is concerned with the oxidation of the trialkylsilyl ethers of 5-hydroxymethyl-

furan-2-carboxaldehyde using *N*-bromosuccinimide in the presence of azo-bis(isobutyronitrile) from which furan-2,5-dicarboxaldehyde can be obtained in good yields <94SC939>. The oxidation of 2-hydroxymethylfuran has been studied using DMD and gave 2*H*-pyran-3(6*H*)-one in high yield, probably via the transient epoxide (Scheme 14), and among the other examples quoted, the dimethyl-acetal from furfuraldehyde was also efficiently converted into the expected product. The epoxidation of menthofuran using DMAD gives both stereoisomeric epoxides which also undergo ring opening to the dicarbonyl compound <92JOC2744>.

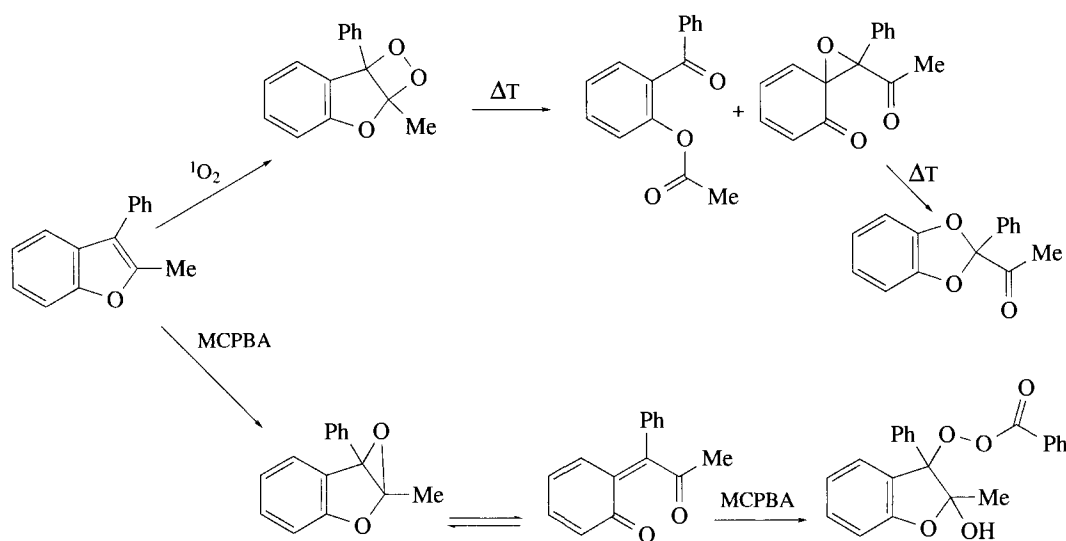


Scheme 14

The reaction of 2,3-dimethylbenzofuran with DMD results in the formation of an epoxide which decomposes below 0°C <92CB231> and the reversible opening of 2-methyl-3-phenylbenzofuran <93AG(E)735> (Scheme 15) explains the difficulty in observing the epoxide in the former case <93JA8603>. Other examples have been reported <94CB941>, including reactions of naphthofurans and furocoumarins with trifluoromethylmethyldioxirane <94LAC689> and the further oxidation of 2-methyl-3-phenylbenzofuran epoxide with DMD, shown in Scheme 16, and the oxodiperoxomolybdenum complex $\{\text{HMPA-MoO}(\text{O}_2)_2\}$ <94T8393>. The cycloadduct formed between methyl furan-3-carboxylate and singlet oxygen undergoes thermal rearrangement to a number of product types including the diepoxide in nonpolar solvents and in basic solvents to a Δ^2 -hydroxybutenolide derivative <85CC124>. 2-Methyl-5-(3'-hydroxypropyl)- and 2-methyl-5-(4'-hydroxybutyl)-furans have been oxidized by ddq to 2-furyltetrahydrofurans and 2-furyltetrahydropyrans respectively <87TL5175>. Oxidative acetoxylation at a benzylic 5-position using ddq has also been used on a number of 2-triisopropylsilyl-3,4,5-tri- and 2-tri-isopropylsilyl-4,5-di-alkylfurans <87TL5403>.

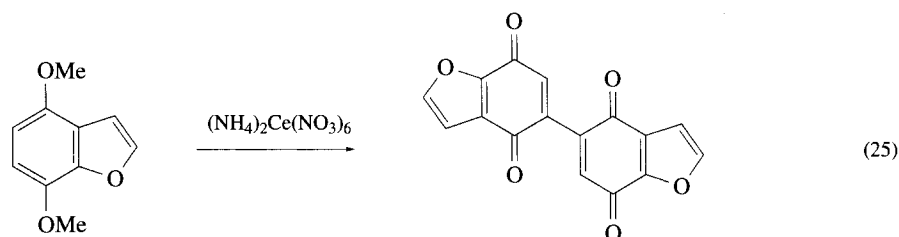


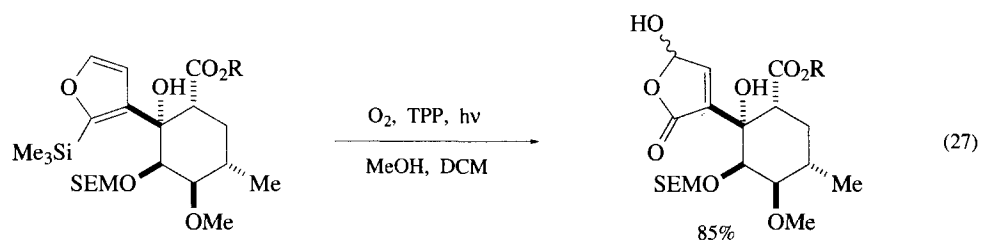
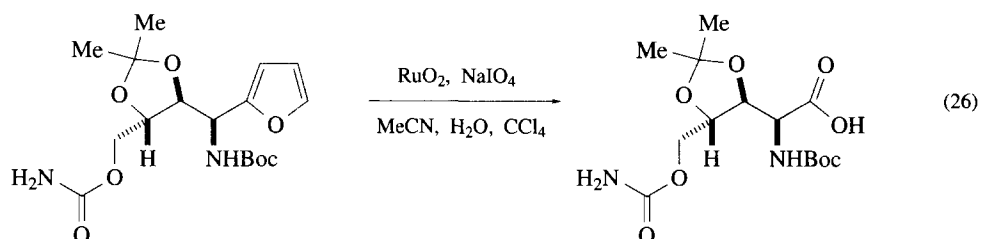
Scheme 15



Scheme 16

Oxidation reactions using metal oxidants, for example, manganese dioxide <94TL3609> and ceric ammonium nitrate <83S1018>, and metallic catalyst systems, also result in epoxidation followed by ring opening of the types illustrated in connection with the reactions of DMD. Khellinone has been prepared in a short synthesis in which the key step is the oxidative methoxylation at position-4 using thallium(III) nitrate or lead(IV) acetate in methanol <85TL1385>. The oxidative demethylation of 4,7-dimethoxybenzofuran using ceric ammonium nitrate results in dimerization, as shown in Equation (25), rather than ring opening of the heterocyclic ring <92H(34)1749>. Oxidative dimerization of furans that carry electron withdrawing groups can also be achieved using palladium(II) acetate <84S255>. Vanadium(V) catalyzed reactions of 2-furylcarbinols with *t*-butyl hydroperoxide <83SC207>, and the kinetic resolution of 2- and 3-furylcarbinols <89JOC2085> have been reported. The ruthenium dioxide–sodium metaperiodate system has also been used to oxidize 2-furylmethanols as part of a route to 2-furfuralcarbonitriles <83T3881>. It is also worth noting at this point that furan can be regarded as a protected form of the carboxyl group. The oxidation of the furan ring to the carboxylic acid can be accomplished using either ruthenium tetroxide or ozone <88JA3929, 89JOC2085, 91TA759, 92TL1411, 93T2641>. In the example shown in Equation (26) the furan was converted into the α -amino acid that has three contiguous chiral centers <93TL5479>. Autoxidation of furan derivatives leading to hydroperoxides <90G471> and photosensitized oxidations <93CL1525> have also been reported. In the latter case a straightforward synthesis of bromobeckerelide was achieved by the photosensitized oxygenation of a suitably substituted 2-trialkylsilylfuran. A number of other examples of this process have been reported and reviewed <87RTC469> and the value of this method is shown in the high yield conversion of the highly functionalized 2-trimethylsilylfuran into the milbemycin fragment <91TL2269> shown in Equation (27) <85TL4625>. Other examples of this transformation have found use in natural product synthesis <86TL4533, 87CC1342, 87TL1191, 88TL1173, 93CL1525>. A long series of investigations of singlet oxygen oxidation reactions involving substituted furan derivatives includes, in the more recent reports, the production of unstable dioxoles and carbonyl oxides <89JCS(P1)241>, including the first direct evidence for 3*H*-1,2-dioxole formation <92JCS(P1)1855>. The same group of workers have also reported the selective formation of 5-hydroperoxyfuran-2(5*H*)ones <94S944>, a one pot synthesis of functionalized *cis*-4-oxoalk-2-enoates <95S439>, and the stereoselective synthesis of functionalized methyl *cis*-3-aryl-2,3-epoxypropanoates <95SL1161>. In the last example, 5-aryl-2-methoxyfuran derivatives were converted into the 5-aryl-5-hydroperoxyfuran-2,2-dimethoxy-2,5-dihydrofurans by dye-sensitized photo-oxygenation reactions carried out in methanol which then gave the epoxy-esters on treatment with sodium hydride in diethyl ether. Singlet oxygen oxidation of a number of 2,4-disubstituted furans also gives 4-substituted-2(5*H*)furanones <91JOC7007> and an example has been reported where the initial endoperoxide was shown to rearrange at -20°C to an isolable dioxetane <93AG(E)80>. The 4 + 2 cycloadduct formed by the photosensitized oxygenation of 2,5-dimethylfuran has been isolated and is reduced by triphenylphosphine to *E*-2,5-dioxohex-3-ene <83AG(E)726>. The endoperoxide was also shown to give a dimeric product that gave rise to 2,3,4,5-bis(epoxy)-2,5-dimethyltetrahydrofuran. The photooxygenation of benzofurans, for example, 2,3-dimethylbenzofuran, to give dioxetanes can be achieved using tetraphenylporphine as the photosensitizer <91LA33> and the deoxygenation of the dioxetane can be carried out using triphenylphosphine to give firstly the benzofuran epoxide and then the benzofuran <94TL6063>. The triphenylphosphine deoxygenation of the singlet oxygen adducts formed with a number of 5-alkyl-furan-2-methanol derivatives has been shown to give, among a number of products, 2-hydroxydihydropyran-3-ones <90H(31)1941>. 3-Alkylfurans can be converted by singlet oxygen, generated using a polymer bound Rose Bengal catalyst, in the presence of a hindered base into 3-alkyl-4-hydroxybutenolides <88JOC2773> with little or none of the isomeric butenolide. This latter method neatly avoids the necessity of using a 2-trialkylsilylfuran. The photosensitized oxidation of 2-methoxy-3,5-diphenylfuran gives the expected cycloadduct, the breakdown of which depends on solvent polarity. One type of product was shown to be a carbonyl oxide and the reaction also provided the first direct evidence for the existence of a dioxole <91JCS(P1)1479>.

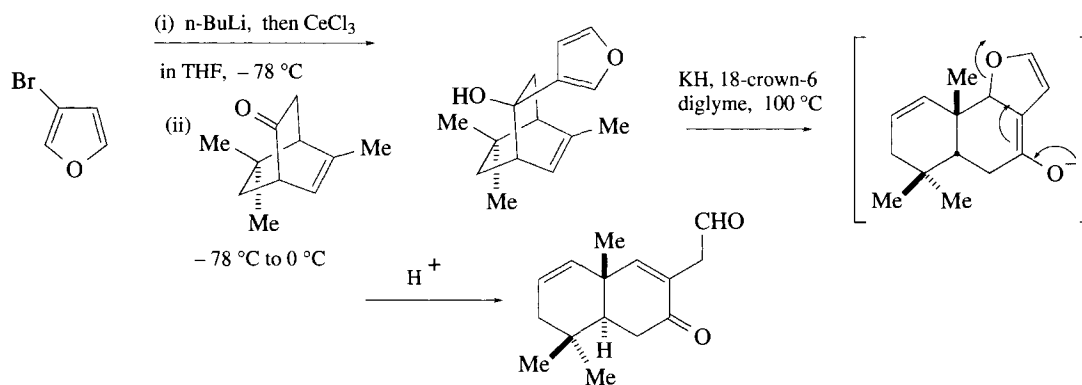




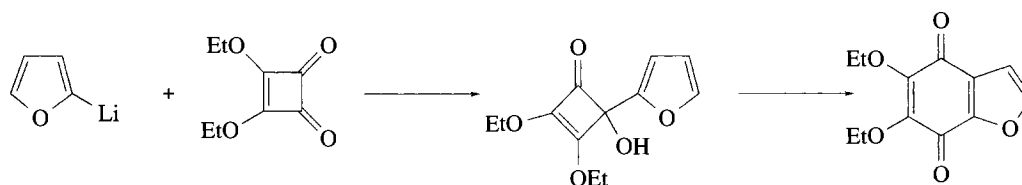
2.06.2.3 Reactions with Nuclear Anion Equivalents

Elaboration of the furan ring has been the subject of a number of investigations involving the use of organometallic reagents, especially furyllithium reagents. The reactions of higher order cuprates $\langle 90\text{SL119} \rangle$ derived from 2-furyllithium have been surveyed $\langle 88\text{TL3045} \rangle$. The coupling of 2-furyllithium with, for example, 5-lithio-2,2'-bifuryl to form the terfuryl is achieved in high yield using copper(II) chloride $\langle 81\text{CB3667} \rangle$. 2-Furyllithium reacts with diethylaluminum chloride to form 2-furyl diethylaluminum which has been used to effect the stereospecific replacement of fluorine in a protected 2- β -fluoroglucose $\langle 88\text{JOC3371} \rangle$. Butenolides have been prepared by the interaction of 2-nitrofuran with Grignard, organocadmium, and organolithium reagents $\langle 90\text{T7497} \rangle$ and substituted Δ^2 -butenolides have been prepared from furan equivalents by regioselective metallation, sulfenylation (and/or silylation), followed by removal of the sulfur group and peracid oxidation $\langle 83\text{TL5835} \rangle$. The conversion of a number of 5-substituted 2-tributylstannylfurans into the acetates of butenolides has been reported using lead(IV) acetate as the oxidizing agent $\langle 88\text{CC560} \rangle$. A high yield preparative scale method for the formation of 2-bromofuran involves the reaction of furan with ethyllithium and then bromine $\langle 89\text{SC1047} \rangle$. 3-Furyllithium, prepared from 3-bromofuran and butyllithium in tetrahydrofuran, only reacts with reactive electrophiles at temperatures below -40°C . In the presence of hexamethylphosphoric amide the reactivity towards weaker electrophiles such as primary alkyl halides is increased and reactions can then be carried out at low temperatures. At higher temperatures transmetallation results in the formation of 2-furyllithium $\langle 90\text{T1199} \rangle$. Bis(2-furyl)zinc has been shown to react with a number of aldehydes in the presence of *N,N*-dibutyl or *N,N*-di-(4-phenylbutyl)ephedrine to give the expected secondary alcohols with enantiomeric excesses of up to 73% $\langle 90\text{JCS(P1)3214} \rangle$. (*S*)-Diphenyl-(1-methylpyrrolidin-2-yl)methanol $\langle 89\text{H(29)2219} \rangle$, quinine $\langle 89\text{TL6427} \rangle$, and chiral β -aminoalcohols $\langle 91\text{JCS(P1)25} \rangle$ have also been used in conjunction with dialkylzinc reagents. The metallation of benzofuran at the 2-position using an alkyl lithium reagent has been used to generate an anion to take part in $\text{S}_\text{N}2$ reactions with, for example, *n*-butyl iodide $\langle 91\text{TL5903} \rangle$. 2-Furylzinc chloride has been in coupling reactions with vinyl and aryl triflates catalyzed by tetrakis(triphenylphosphine)palladium(0) $\langle 90\text{SL47} \rangle$ and also in a reaction that formed part of a route to a novel terphenoquinone where the furan was eventually to be the central ring $\langle 94\text{CL863} \rangle$. The lability of benzylic chlorides is well known as also is the difficulty of handling the derived Grignard reagents. The introduction of a 5-trialkylsilyl substituent not only stabilizes the chlorides but also it allows satisfactory yields of products from Grignard reactions to be isolated $\langle 87\text{TL2281} \rangle$. There are occasions when furyl Grignard and lithium reagents and furylzinc chlorides are unselective or have inappropriate reactivity and the furyl-triisopropoxytitanium reagents may then be worth consideration. An example of the use of a titanium reagent involved a reaction with methyl 1-formylhex-4-ynoate $\langle 91\text{TL903} \rangle$. Conversion of 3-furyllithium into the cerium reagent by addition to anhydrous cerium(III) chloride allowed the addition to an optically pure ketone selectively and in

high yield (Scheme 17). The anionic oxy-Cope rearrangement involving the furan ring gave the keto aldehyde as a result of β -elimination following the [3,3] rearrangement was a key step in the total synthesis of the marine sesquiterpene (+)-pallascensin A (92JOC7118). 2-Furyllithium has also been used in a versatile method for attaching C-glycoside precursors to aromatic rings, including the western end of nogalamycin (83CC896). The 2,9-dioxabicyclo[3.3.1]nonane system, similar to that found in tirandamycin, is efficiently assembled in a method that starts with the lithiation of 2,3-dimethylfuran at the 5-position using *n*-butyllithium in the presence of TMEDA (82TL2243). The preparation of 4-benzoylfuran-2-sulfonic acid was achieved by the sequential reaction of 3-bromofuran with *n*-butyllithium, benzonitrile, and sulfuric acid/acetic anhydride (89JHC1793). Transmetallation reactions involving organotin compounds normally involves reactions of organolithium reagents with chlorotrialkylstannanes. Both 2- and 3-furyllithium have been converted into a number of other organometallic reagents such as silanes (83ZOB1082), stannanes, and germanes (83ZOB383) by interaction with the appropriate metal chloride. It has been shown (92CC1723) that 3,4-bis(tributylstannyl)furan undergoes the reverse reaction to replace one tin residue with lithium using *n*-butyllithium and gives 3-tributylstannyl-4-furyllithium, even in the presence of an excess of *n*-butyllithium. It is expected that the high bond strength of the silicon-oxygen bond means that oxygen to carbon migration will be rare. An example has been reported where the trimethylsilyl ether of 3-hydroxymethylfuran is rearranged in an intramolecular reaction to give 3-hydroxymethyl-2-trimethylsilylfuran on treatment with *n*-butyllithium at 0°C in hexamethylphosphorus triamide (87TL5965). The elaboration of 2-furyllithium to 5,6-diethoxybenzofuran-4,7-quinone proceeds as shown in Scheme 18 (90OS(69)220). The carboxylation of 2-furyllithium with ^{14}C labeled carbon dioxide has been used in a synthesis of a radiolabeled triazolinyfuran (83MI 206-01). We noted the formation of furylarenes in Section 2.06.2.2.6. 2-Furyllithium has also been shown to add to arynes to produce biaryls (93JOC4722). The arynes were generated from aryl triflates by reaction with lithium di-isopropylamide (1.5 equiv.) and a large excess of 2-furyllithium (8–10 equiv.). The reaction of 5-*t*-butoxy-2-furyllithium has been used in a synthesis of a tropone derivative in which the carbonyl group is separated from the cycloheptatrienyliene ring by the furan moiety in the furanone (82CL1895). The planar molecule shows a remarkably small contribution from the dipolar structure in the ground state which suggested that there is only a slight conjugative interaction between the cycloheptatrienyliene residue and the carbonyl group.



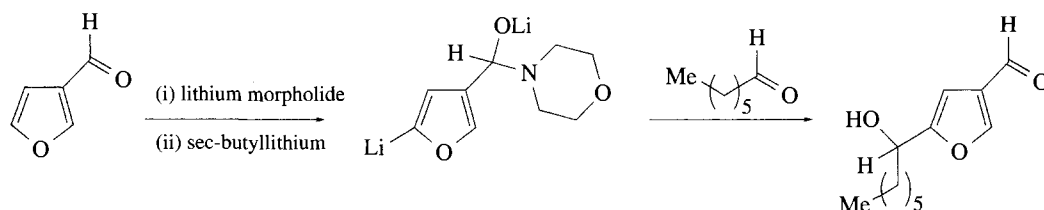
Scheme 17



Scheme 18

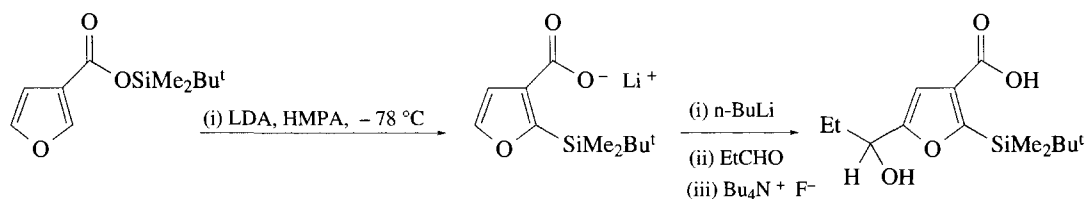
The temporary protection of furancarboxaldehydes as a device to direct metallation has been used in a number of different ways. Lithiation of the diethylacetal of furan-2-carboxaldehyde occurs

at the 5-position <84ZOB1315>: the 2-carboximide function also directs lithiation to the 5-position <94T4149>. Furan-2-carboxaldehyde is converted into the related *N,N'*-dimethylimidazolidine by reaction with *N,N'*-dimethylethylenediamine which then also undergoes metallation at the 5-position <85T3803>. The formyl functionality can then be regenerated under very mild reaction conditions after reaction of the organolithium reagent with an electrophile. The oxazolinyll group, which is at the carboxylic acid oxidation level, directs metallation to the adjacent position so that whereas a 2-furyloxazoline undergoes Friedel–Crafts reactions at the 4-position, metallation allows the introduction of the same functional groups at the 3-position <92T149>. Reactions of the 2-(4,4-dimethyl-1,3-oxazolinyll)-3-furyllithium reagent <82JCS(P1)1343> with other electrophiles and the related 5-trimethylsilyl-2-(4,4-dimethyl-1,3-oxazolinyll)-3-furyllithium reagent <90T2623> has been converted into the zinc reagent and then reacted with a range of aryl iodides and allyl, benzyl, and vinyl bromides. The bis(dimethylamino)phosphate group at the 2-position in furan also directs metallation to the 3-position <86TL1391>. The 3-*p*-methoxyphenylsulfonyl group also directs metallation using *n*-butyllithium to the adjacent 2-position in furan <87TL3241>. Selective protection and subsequent 5-substitution of furan-2-carboxaldehyde using lithium *N*-methylpiperazide <92S954> and furan-3-carboxaldehyde using lithium morpholinide <92JOC3126> followed by metallation at the 5-position and reaction with an electrophile can be achieved as exemplified in Scheme 19.



Scheme 19

A new approach to the synthesis of 2,4-disubstituted furans involves protection of the 2-position in 3-substituted furans such as furan-3-carboxylic acid <91SL33, 91JCS(P1)2600> and 3-furylmethanol <94TL5335> as the 2-*t*-butyldimethylsilyl-derivatives. Metallation at low temperature followed by reaction with an electrophile and removal of the silicon protecting group then gives the required 2,4-disubstituted furan (Scheme 20). On the other hand, if the substituent at position-3 directs lithiation to an adjacent position then the method provides a route to 3,4-disubstituted furans <88TL1247>. The metallation of furan using the very strong base system *n*-butyllithium–potassium *t*-butoxide–TMEDA gives 2,5-dilithiofuran and a reaction with DMF resulted in the formation of 2,5-diformylfuran <88S316>.

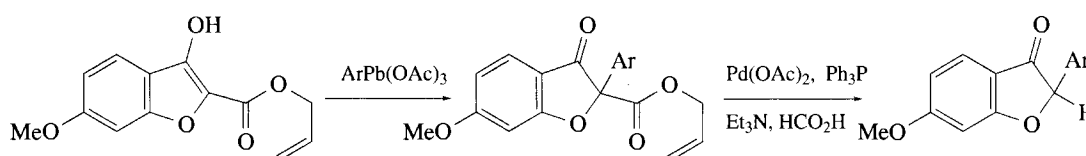


Scheme 20

2.06.2.4 Reactions Catalysed by Metals and Metallic Derivatives

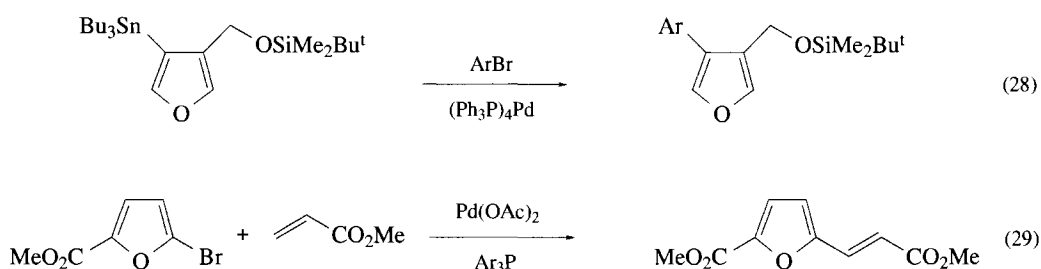
Highly functionalized unsymmetrical furan containing biaryls have been prepared by intramolecular Ullmann reactions <93H(36)1867>. The preparation of 2,2'-bifuran and the terfuran, 2,5-bi-(2'-furyl)furan, has been achieved in good yields by ring connection methodology in which a two furyllithium species was either oxidatively coupled using copper(II) chloride or by the reaction of an iodofuran with a furylcopper reagent <81CB3667>. The functionalization of heterocycles in nickel and palladium catalyzed reactions has been reviewed <91RCR339>. Although the majority of the activity in this area has involved the use of palladium catalysts other species have been involved. For example, the arylation of a benzofuran-3-one at the 2-position, actually in the enolized form, is exemplified in Scheme 21 <90TL6637>. The initial product was then further elaborated by removal of the allyl ester function using palladium(II) acetate–triphenyl phosphine as shown. The coupling

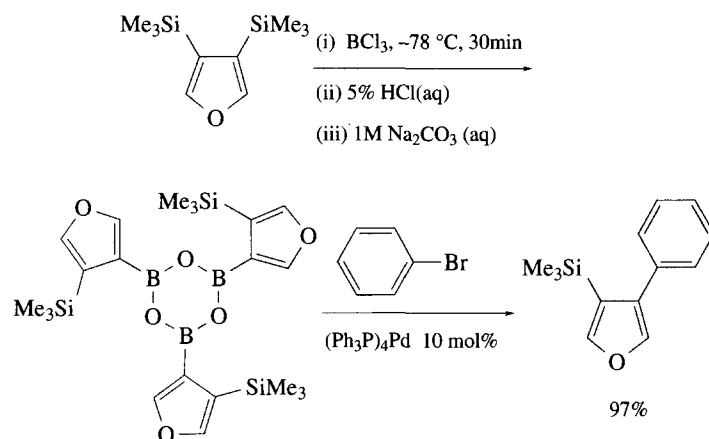
of vinyl iodides with furylzinc species in Heck-type reactions has been shown to proceed effectively under palladium catalysis <89T5283>. Insertion into C—H bonds has been observed in rhodium acetate catalyzed reactions with dimethyl diazomalonate and with furoyldiazomethane to give cyclopropane derivatives by way of carbenoid cycloaddition to 2,3-dimethylbut-2-ene <86IZV1454>. Furan reacts to give the cyclopropanation product with ethyl diazoacetate at 150°C fourteen times more rapidly than the analogous reaction involving naphthalene <88JOC4374>. The direct fluoroalkylation of furan can be carried out in the presence of tetrakis(triphenylphosphine)nickel <89JFC(43)385>. Furan and its 2- and 2,5-dimethyl- derivatives react with a number of Grignard reagents in the presence of a nickel(II) catalyst to give substituted buta-1,3-dienes, normally with retention of configuration <84CC617>.



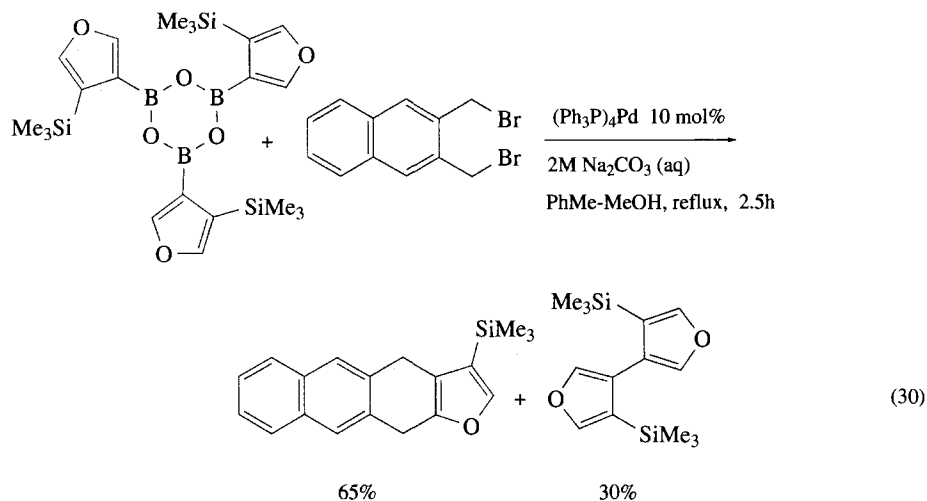
Scheme 21

The alkenylation of 2-formylfuran with methyl acrylate catalyzed by palladium(II) acetate functionalizes the 5-position <84S488>. The use of the different reactivity of tributylstannyl- and trimethylsilyl- residues was mentioned previously in connection with a manolide synthesis. Another step in the same synthesis <88TL1173> involved a palladium catalyzed carbonylative coupling reaction and furan itself has been shown to be carbonylated in the presence of palladium(II) acetate and sodium acetate. In the presence of methanol the methyl ester is isolated and with acetic acid the mixed anhydride results <90JMOC23>. A number of interesting Stille-type coupling reactions involving the replacement of a tributylstannyl group have been reported <91S242, 92CC656, 94ACS279>, including a mild route to 5-arylbutenolides <92SC1627>, and in the generalized example shown in Equation (28), palladium(0) catalysis is involved <91CJC1326>. The palladium catalyzed cross-coupling of a protected 6-hydroxy-2-trimethylstannylbenzofuran with tricarbonyl chromium complexes of protected and unprotected 5-iodoresorcinols has been used in a synthesis of moracin M <87TL2645>. The direct coupling of furan with aryl bromides has also been recorded using tetrakis(triphenylphosphine)palladium <90H(31)1951>. The reverse process, in which a bromofuran is coupled with an alkene or an alkyne, has been shown to be quite general <89SC1745> and is exemplified in Equation (29). Boroxine derivatives, which can be made by reactions involving the interaction of furylsilanes and boron trichloride <93AG(E)432>, and as shown in Scheme 22, undergo Suzuki-type cross-coupling reactions under circumstances where the direct displacement of a silicon residue does not occur. Reactions of 1,2-bis(bromomethyl)arenes with boroxines result in the formation of regiospecifically cross-coupled products together with the dimeric bifuranyls as indicated in Equation (30) <94JOC33>. In the case of an attempted reaction with 2,3-bis(bromomethyl)quinoxaline no cross-coupling was observed and the bifuranyl derivative was isolated in good yield. The *in situ* generation of furylboronates via the furyllithium reagent followed by palladium catalyzed coupling with aryl bromides, benzyl bromide, and aryl triflates and vinyl iodides has been used to functionalize 2-*t*-butyldimethylsilyl-3-hydroxymethylfuran at the 4-position <91TL5881>. 2-Formylfuran-3-boronic acid also undergoes a Suzuki-type of coupling with aryl bromides <89SC1001>. The scope and limitations of *in situ* palladium(0) catalyzed Suzuki-type of cross-coupling reactions has been summarized and also investigated without the isolation of the intermediate arylboronic acids <94JOC6501>.

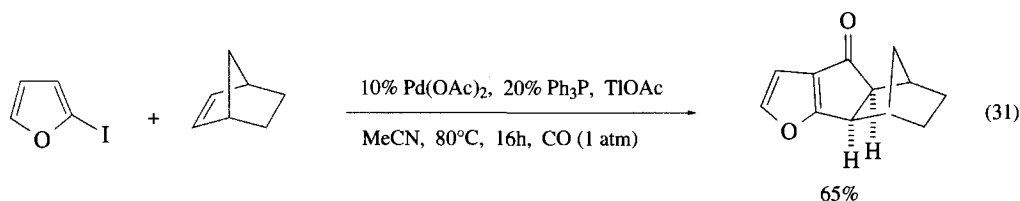




Scheme 22



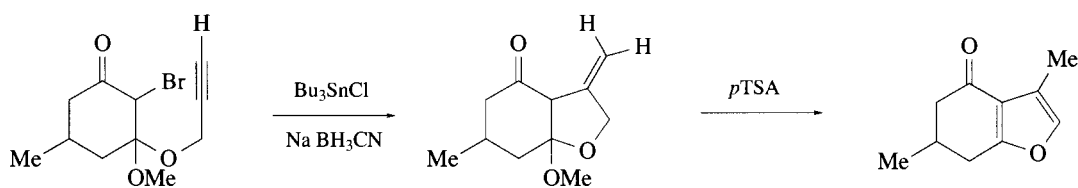
Palladium(II) trisulfonated catalysts have been used, with carbon monoxide under pressure and aqueous reaction conditions to carbonylate, for example, 2-hydroxymethyl-5-formylfuran. 5-Methyl-2-formylfuran was obtained in addition to 5-formyl-2-furylacetic acid <94CC2569>. Palladium(0) catalysts have been shown to effect a number of cycloaddition processes that use carbon monoxide as a one carbon component. Benzofurans have been synthesised by cyclocarbonylation <89TL95> and in the example shown in Equation (31) norbornene gave the tricyclic system in good yield <94TL3197> in a reaction where the palladium(0) catalyst was generated *in situ*.



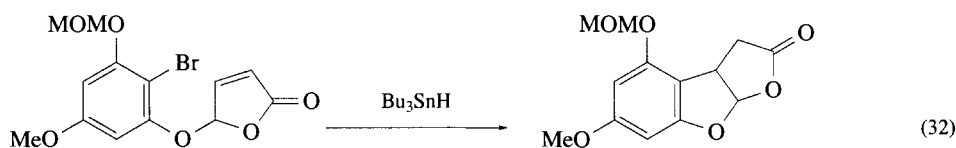
2.06.2.5 Reactions Involving Free Radicals

Radical induced coupling and cyclization reactions involving furan and benzofuran derivatives have found a number of interesting synthetic applications. The regiospecific cross-coupling of Grignard reagents with halofurans is catalyzed by nickel(II) ethane-1,2-bis(diphenylphosphine) dichloride <82JOC1590> and furan derivatives have been synthesised by the radical cyclization of bromoacetals using a procedure derived from the earlier studies that involved the conversion of

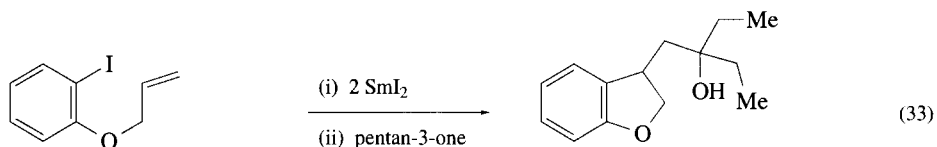
bromoacetals into bicyclic acetals and lactones <83JA3741>. The cyclization shown in Scheme 23 was used in a synthesis of racemic evodone <88TL4995> and is effective for the preparation of a number of 3-acyl-4-methylfurans. The method allows the formation of 2,3-disubstituted furans in reactions where propargyl ether-acetals are converted into the furans by way of a radical generated by reduction of the bromo-derivative in the presence of chlorotributylstannane and sodium cyanoborohydride <87TL6393>. The radical cyclizations of *o*-bromoaryl allyl ethers have been used in the synthesis of pterocarpanes <88CC28> and further to butenolides in formal syntheses of aflatoxins B₁ and B₂ <88TL4685> as indicated in Equation (32). Electrophilic radicals, for example, the ethoxycarbonylmethyl radical, have been generated from bromo- and iodo-derivatives by the auto-oxidation of triethylborane, and homolytic aromatic substitution carried out with, for example, furan <93TL5015>. The *cine*-substitution of 2-nitrofurans that also carry an acyl or alkoxycarbonyl group at the 5-position using anions derived from secondary nitroalkanes give products that are denitrated with tributyltin hydride <87S821>. Sulfones have been shown to undergo radical displacement reactions. For example, the reaction of 4-[(4-methylphenyl)sulfonyl]dibenzofuran reacts with tributyltin hydride and AIBN in toluene to give the corresponding dibenzofurylstannane <93TL7469>.



Scheme 23

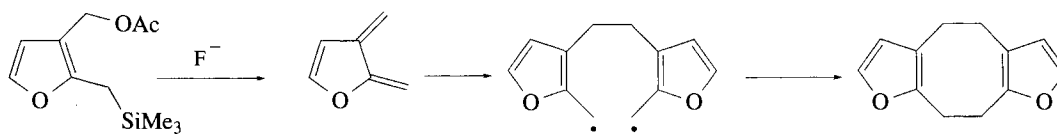


The use of sequence reactions involving samarium(II) iodide, first in a radical process followed by an anionic reaction, has been reported for the synthesis of 2,3-dihydrobenzofuran derivatives <90SL773, 92JA6050>. *o*-Iodophenylallyl ethers undergo cyclization in a 5-*exo-trig* process to a primary radical that is reduced further to an anion which is formally a samarium(III) reagent and which can then undergo reactions with a range of electrophiles as exemplified in Equation (33) by the reaction with pentan-3-one. Allylic acetates have also been used as radical acceptors which can undergo β -elimination of the acetate after reduction of the radical to the anion <91TL1737>. Radical anions have also been generated from 2,5-bis(tricyanovinyl)furan <95JCS(P2)209>. It is unlikely that the nucleophilic substitution reactions of methyl 5-nitrofuran-2-carboxylate and 2-cyano-5-nitrofuran proceed by way of an S_{RN}1 mechanism although the nucleofuge is nitrite and well established electron donors such as thiolate ion are involved <86JHC1179>.

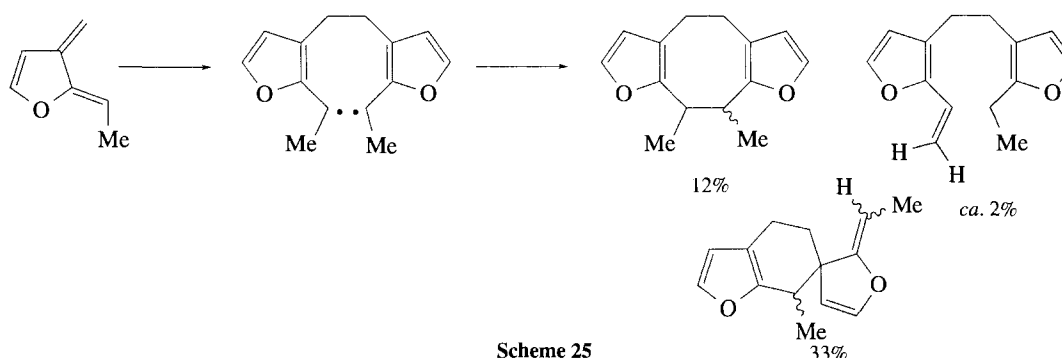


2,5-Dimethylenefuran has been generated by flash vacuum pyrolysis of 5-methyl-2-hydroxymethyl benzoate at 630–650°C and can be captured by a number of reagents such as bromine, acetic acid, and benzenethiol. In the absence of a trapping agent the dimer, 2,2',5,5'-[2.2]-furanophane and a trimer were isolated <92MI 206-04>. 2,3-Dimethylene-2,3-dihydrofuran has been studied in detail and shown to dimerize in high yield <81JA6691> by way of an intermediate diradical at temperatures above –30°C <94JOC2594, 94JOC2613>. The reactive intermediate is best generated by the fluoride ion induced elimination of trimethylsilyl acetate from 3-acetoxymethyl-2-trimethylsilylmethylfuran (Scheme 24). 2,3-Dimethylene-2,3-dihydrofuran has also been generated by the flash vacuum pyrolysis of 3-chloromethyl-2-methylfuran at 550°C and collected at a low temperature. Photolysis in an argon matrix at 16 K converted dimethylene-2,3-dihydrofuran into the isomeric dihydrocyclobutafuran <87AG(E)471>. The mechanistic interpretation of the dimerization process is supported by

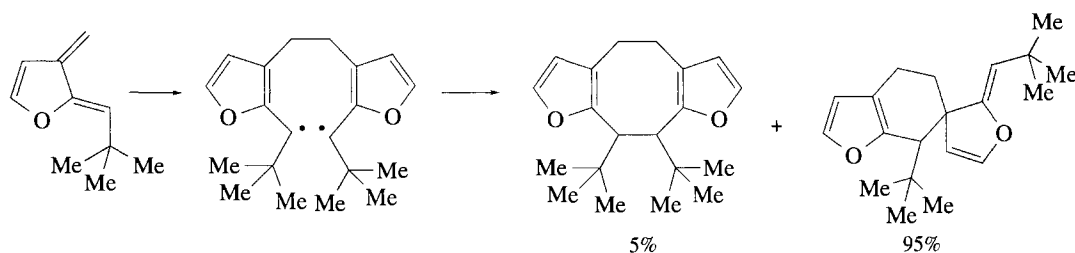
the results of a study of secondary deuterium isotope effects $\langle 86JA4138 \rangle$ and by the effect of additional methyl groups at the 2- and 3- positions and *t*-butyl group substitution on the 2-methine position and at the 5-positions on the furan ring. The rate of dimerization is hardly affected by the bulky substituent at the 5-position and this supports the view that the bulky groups are far apart and therefore that an *exo*-transition state is involved $\langle 94JOC2613 \rangle$. The effect of substituents on the ends of the diene system has also been addressed. A methyl group at the 3-position retards the dimerization process and this is also consistent with the two step mechanism where the rate determining step is the formation of the intermediate diradical $\langle 95JA841 \rangle$. The generation of 2-ethyldene-3-methylene furan results in the formation of the three products shown in Scheme 25 where the minor product clearly arises from the intermediate diradical. In the reaction shown in Scheme 26 the rate of reaction is almost the same as that involving 2,3-dimethylene furan but, as expected, the major product does not arise by head to head dimerization $\langle 94JOC2594 \rangle$.



Scheme 24



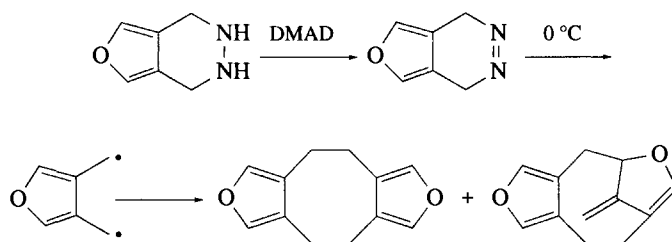
Scheme 25



Scheme 26

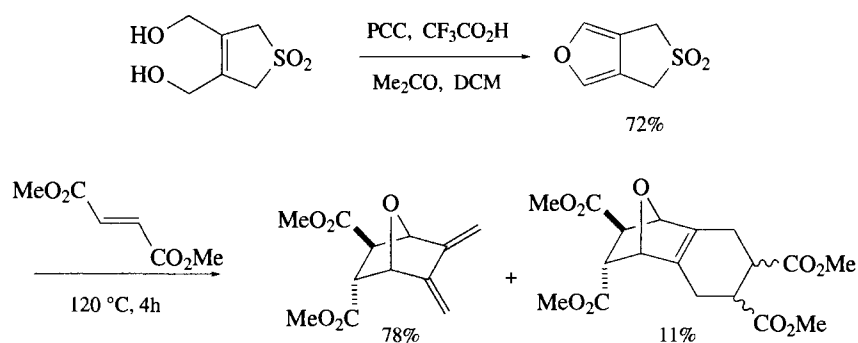
The isomeric 3,4-dimethylene-3,4-dihydrofuran has been described as a singlet diradical $\langle 89JA3659 \rangle$ which adds stereospecifically to electron deficient alkenes. A number of precursors of 3,4-dimethylene-3,4-dihydrofuran have been described which can be used to generate the intermediate at a variety of temperatures. There is a strong correlation between the dienophilic and diylphilic properties of alkenes, for example, in the presence of a dienophile such as fumaronitrile the product is that expected of a Diels–Alder reaction $\langle 89JA8732 \rangle$. A precursor that fragments at low temperatures, 1,4-dihydrofurano[3,4-*d*]pyridazine, can be prepared in over 55% yield in three steps from 3,4-bis(chloromethyl)furan: in the last step the tetrahydro-derivative is oxidized at low temperatures using, for example, dimethyl azodicarboxylate. At temperatures above $0^\circ C$ dihydrofurano[3,4-*d*]pyridazine fragments to 3,4-dimethylene-3,4-dihydrofuran and gives two dimers, one of which is an *anti*-Bredt isomer (Scheme 27). Photolysis of 1,4-dihydrofurano[3,4-*d*]pyridazine at 310–380 nm also gives 3,4-dimethylene-3,4-dihydrofuran $\langle 89JA3659 \rangle$. Absolute rates of dimerization

and cycloaddition of 3,4-dimethylene-3,4-dihydrofuran have been measured by nanosecond time-resolved spectroscopy <88JA4050>.

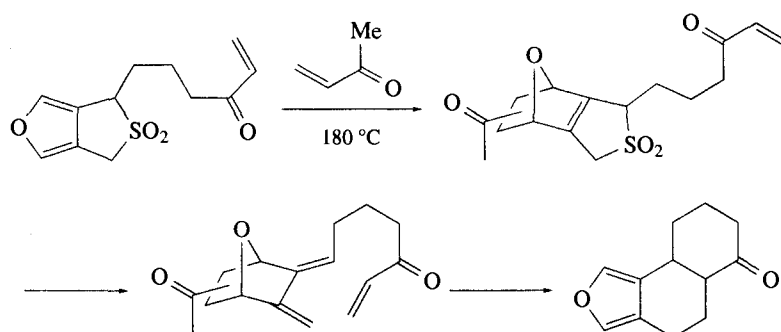


Scheme 27

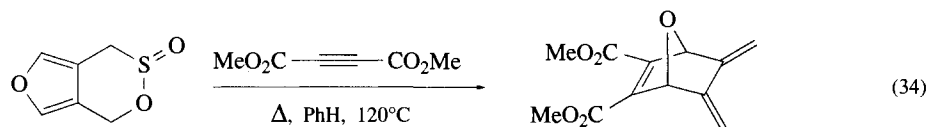
In the majority of the reactions of 3,4-dimethylene-3,4-dihydrofuran with dienophiles, the first reaction involves a conventional Diels-Alder reaction of the furan ring so that the final product may incorporate two molecules of the dienophile <93H(35)57> (Scheme 28) or may result in regeneration of the furan ring system by way of a retro-Diels-Alder reaction. Two other methods have been used to generate 3,4-dimethylene-3,4-dihydrofuran. In the most widely studied method 4*H*,6*H*-thieno[3,4-*c*]furan, the furan fused sulfone can be readily obtained starting with 2,3-dimethylbuta-1,3-diene <93JCS(P1)2263>. The chemistry of heteroaromatic sulfones has been reviewed <94H(37)1417> and the temporary protection of a furan derivative by means of a Diels-Alder reaction-retro-Diels-Alder strategy has been used in the construction of polycyclic systems by means of intramolecular reactions of the dimethylene moiety as shown in Scheme 29 <91CC1765>. Steric congestion has been shown to result from some types of reactions. The alternative approach <95CC2537> involves loss of sulfur dioxide at elevated temperatures from a sulfone which may be prepared by elaboration of 3,4-bis(chloromethyl)furan by reaction with rongalite (sodium formaldehyde sulfoxylate) and is exemplified in Equation (34).



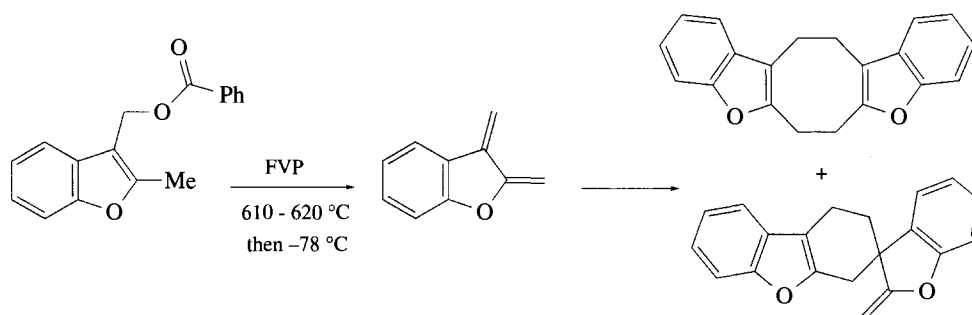
Scheme 28



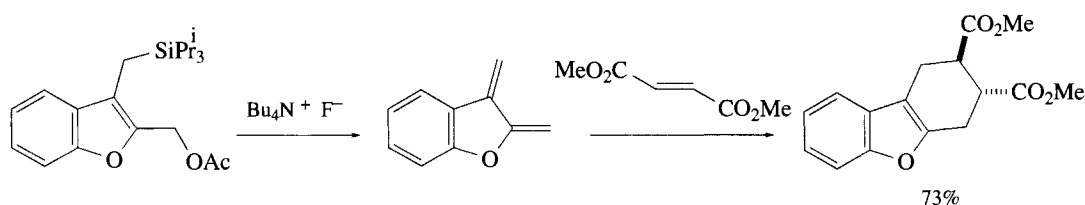
Scheme 29



Although 2,3-dimethyleneindole derivatives have been the most extensively studied of the benzo-five-membered-1,2-dimethylene heterocycles, there have been some successful reports of the generation and reactions of 2,3-dimethylenebenzofuran. The flash vacuum pyrolysis product from 2-methyl-3-benzofurylmethyl benzoate, carried out at 610–620°C, was condensed at –78°C and transferred for study by NMR spectroscopy and peaks were observed that could be assigned to 2,3-dimethylenebenzofuran [\[86JOC4208\]](#). When the pyrolysis product was allowed to warm to room temperature two dimers were isolated in 85% yield and a ratio of 4.1 : 1. The major product (Scheme 30) was shown to be the 4 + 2 dimer rather than the alternative head to head dimer. Further studies have been reported in which the 2,3-dimethylenebenzofuran was generated by fluoride ion induced elimination of tri-*isopropylsilyl* acetate and trapped as shown in Scheme 31 [\[91SL627\]](#). Small amounts of the dimers were formed in the latter reaction and their presence was taken to implicate the diradical character of the xylene.



Scheme 30

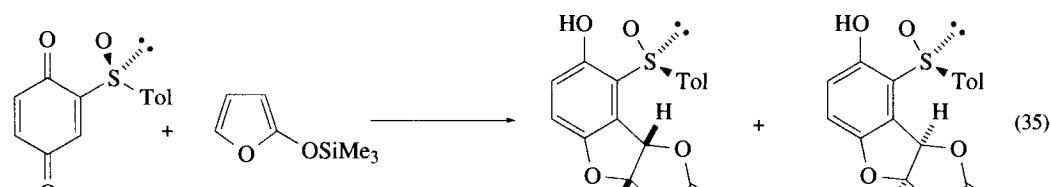


Scheme 31

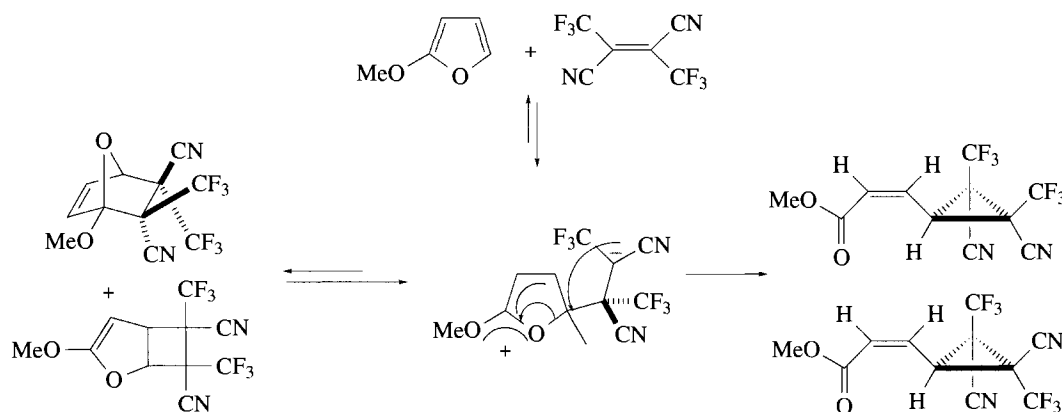
2.06.2.6 Further Cycloaddition Reactions

A very large section in CHEC-I [\[84CHEC-I\(4\)599\]](#) was concerned with Diels–Alder reactions of furan and substituted furan derivatives and 1,3-diarylbenzo[*c*]furans. The previous section has been concerned with the reactions of furan and benzofuran derived 1,2-dimethylene radicals and their cycloaddition reactions and so it is appropriate to link those reactions to what are more formal Diels–Alder reactions by considering cycloaddition reactions of dipolar species and the [4 + 3] cycloaddition reactions of oxyallyl intermediates. 1,3-Dipolar nitrile oxide cycloaddition reactions of the weak dipolarophiles furan and 2-methylfuran [\[85T3519\]](#) give furoisoxazolines which are versatile precursors for a range of novel amino sugar derivatives. The reactions of nitrones with 2-trimethylsilyloxyfuran using trimethylsilyl triflate as a catalyst have been investigated as the first step in a synthesis of bicyclic isoxazolidines [\[94JOC6843\]](#). The reactions were carried out at –20°C in dichloromethane and gave 2(5*H*)-furanone 5-*N*-hydroxymethanamines by way of a catalytic cycle whereby triflate anion desilylated the furan derivative and redeposited the silicon residue on what had been the nitrone oxygen atom. The diastereoselective addition of 2-trimethylsilyloxyfuran to (*S*)-(+)-(*p*-tolylsilyl)-1,4-benzoquinone has been studied [\[95TA263\]](#) and gave the products shown in

Equation (35). High diastereoselectivity has also been observed in the addition of allyltriphenylstannane to optically pure 2-formyl-3-*p*-tolylsulfinylfuran using titanium tetrachloride <94H(38)1751>. Interestingly, tin(IV) chloride gave the other diastereomeric alcohol apparently exclusively. Intramolecular reactions, for example, of nitrile oxides based on derivatives of *o*-(2-furylmethyl-oxy)benzaldehyde, have also been reported <84CL285>. The use of oxyallyl cations in synthesis has been reviewed <86T4611>. Cyclopropanone–oxyallyl intermediates have been prepared by the dehydrohalogenation of α -haloketones using, for example, triethylamine and lithium perchlorate, and react with furan and substituted furans to give 3-oxo-8-oxabicyclo[3.2.1]oct-6-enes <82S976, 87T2533, 87T5431>. The dehalogenation of 2,4-dibromopentan-3-one using copper and sodium iodide in the presence of furan produced the bicyclic derivative in a good yield as part of a synthesis of the tricyclic skeleton of the pseudoguaianolides <88T5151, 92JCS(P1)337>. The regioselectivity of the cycloaddition reactions of unsymmetrical oxyallyl cations towards 2-methylfuran has been studied <84TL1773>. An improved procedure that uses α -methanesulfonyl derivatives of ketones, exemplified in Equation (36), gives the same products after shorter reaction times <86CB524>. Intramolecular [4 + 3] cycloaddition reactions of oxyallyl intermediates have been reported <84CB2580>, including examples where the stereochemistry of the product was found to be independent of the stereochemistry of the initial allyl cation precursor <90TL5981>. The synthetic potential of this chemistry has been explored and an example of an intramolecular reaction that gives a good yield <94JOC1241> when using recently developed technology is shown in Equation (37). A [4 + 2] anionic cycloaddition strategy together with a thermal cyclo-retrograde reaction has been developed for the synthesis of furocoumarins <94TL9617>.



including cyclopentadiene and 1,3-diphenylisobenzofuran <82TL4203>. Reactions of furan with 1,1,1,8,8,8-hexafluoro-4-methoxycarbonyl-4-methylocta-2,5-diyne have been studied and products that arise from the bis(Diels–Alder) reaction involving two molecules of furan followed by complex rearrangements have been isolated and identified <89CC1637>. Reactions of very electron deficient alkenes with donor substituted furans have been reinvestigated <94TL4977>. 2-Methoxyfuran was shown to form an orange charge-transfer complex with *E*-1,2-bis(trifluoromethyl)-1,2-dicyanoethene which faded after several hours and gave rise to a mixture of cyclopropane derivatives as shown in Scheme 32. The two isomeric compounds are readily identified from their NMR spectra since one of them is chiral and the other has a plane of symmetry. The zwitterionic intermediate can regain the aromaticity of the furan ring in a base catalysed prototropic shift that was observed when the experiment was carried out in the presence of pyridine. In a detailed ^{19}F NMR investigation Diels–Alder and 1,2-cycloadducts were observed to be in equilibrium with the zwitter ion. Finally the cyclopropane derivatives were shown to be formed by the irreversible ring opening of the furan ring <94TL4981>. Once again a very large number of papers have been published on Diels–Alder reactions and we will be selective and concentrate in this section on synthetic applications. The section will be further divided into inter- and intramolecular reactions.

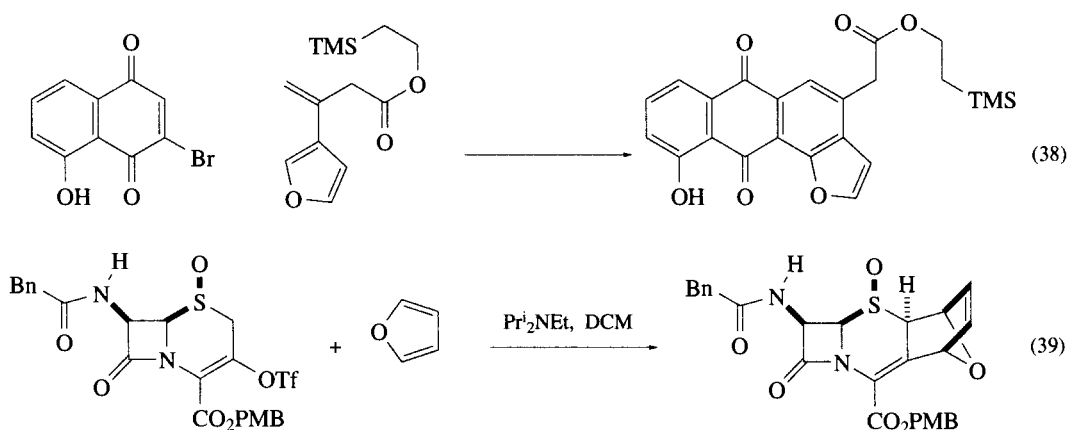


Scheme 32

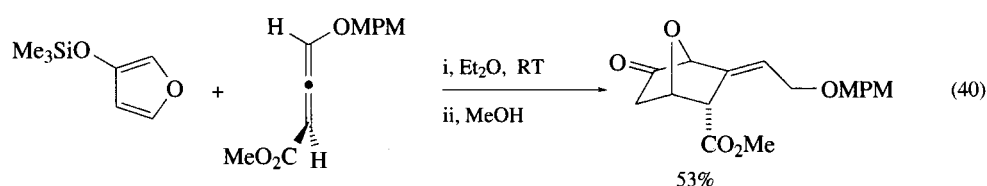
(i) Intermolecular Diels–Alder reactions

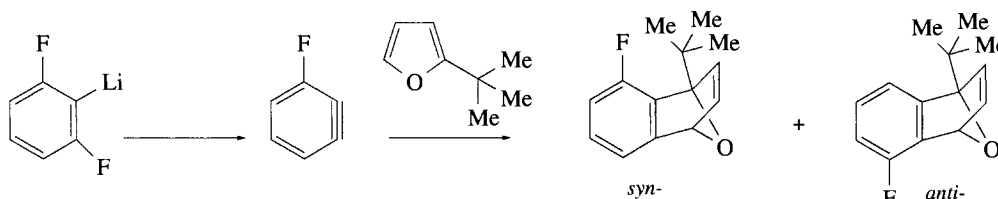
Thermal Diels–Alder reactions of furans are problematic with some dienophiles because of the ease with which they take part in competitive aromatic substitution reactions. Lewis acid catalysis often helps to avoid that problem. The reaction of methyl acrylate with furan proceeds in good yield when catalyzed by boron trifluoride etherate <84BCJ3339>, and was used in a synthesis of ($\pm$) avenaciolide <86BCJ3881>. Clays impregnated with transition metal cations are also effective <94JCS(P1)761>. Copper(I) and copper(II) salts have also been used as catalysts to improve the efficiency of reactions that are known to be difficult using dienophiles such as α -acetoxycrylonitrile and α -chloroacrylonitrile <82HCA1700>. Significant acceleration of the rates of reactions have been recorded in the presence of zinc(II) iodide <82TL5299> and this method has been used in the reaction between 2-methylfuran and 2-chloroacrylonitrile as the first step in syntheses of 2,5-epoxy-6(*E*),8(*E*)-megastigmadiene and 3',6'-epoxycycloaurapten <89H(29)2391, 90MI 206-02>. The use of other nitriles has also been studied, including 3-cyanocyclopropene <90H(31)1895>. Aluminum chloride has been found to enhance the regioselectivity of reactions between butenolides and dienes <87TL3405> and high stereoselectivity has been reported for reactions of buta-1,3-diene with chiral butenolides <88T1711>. The effects of a number of parameters on the position of Diels–Alder equilibria have been investigated using fumaronitrile as the dienophile <94T12125>. Although furan-2-carboxaldehyde is a poor diene the related *N,N*-dimethylhydrazones do take part in reactions with a range of dienophiles including maleic anhydride, maleimides, and fumaronitrile <88JOC1199>. Ethyl *trans*- β -nitroacrylate gives the furan adducts in high yield and were used in part of an investigation into the synthesis of (+)-compactin <86JA5908>. Quinones have been shown to react with furan derivatives to give useful products <87TL4891> and in the example shown in Equation (38) the 2-vinylfuran derivative undergoes the Diels–Alder reaction and is further elaborated into 11-deoxy-daunomycinone and related compounds <82JA7372>. Cephalosporin triflates have also been shown

to undergo reactions with furan in the presence of a base. The regiochemistry of the cycloaddition process is determined by the oxidation state at sulfur in the cephalosporin and is illustrated in Equation (39) <94JOC1606>. Part of a synthesis of ivermectin used an inverse electron demand Diels–Alder reaction of 3,4-dibenzoyloxyfuran with 2-(trimethylsilyl)ethylcoumalate which gave a mixture of *endo*- and *exo*-cycloadducts; the major *endo*-adduct was then taken on in the synthesis <87TL5977>. A number of other examples of reactions where a furan functions as the dienophile have been published <88JA7188, 88JOC5850, 89JOC4134>, including reactions with tropone carried out at high pressure <87BCJ3633>.

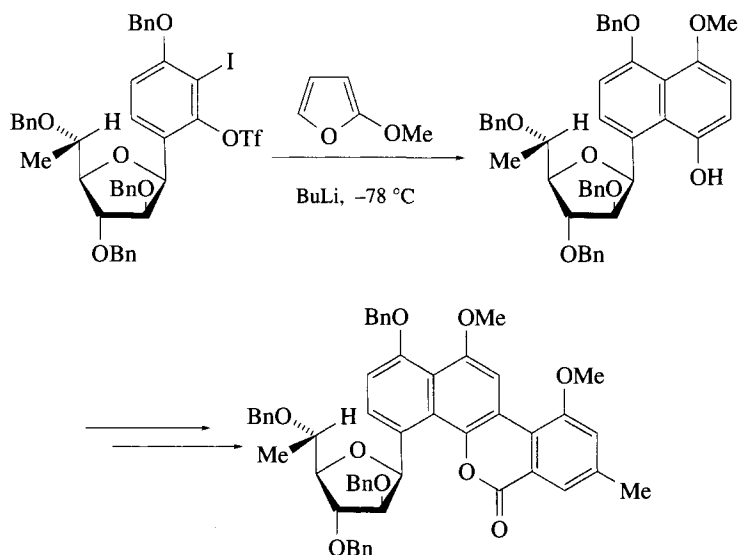


Many of the synthetic applications of furan Diels–Alder chemistry relate to the ease with which the oxygen bridge in the adduct can be manipulated. Citreoviral, a novel metabolite from *Penicillium citreoviride*, was synthesised in racemic form starting with a Diels–Alder reaction of 2,4-dimethylfuran <85CC292> and a synthesis of ubiquinones proceeds by way of the cycloadduct that is formed between trichloroethene and 2,5-bis(trimethylsilyloxy)-3-methylfuran <90HCA790>. Reactions of more complex 2,5-disubstituted furans with dimethyl acetylenedicarboxylate give products that have been converted into highly functionalized phenols and hence into coumarin derivatives <93H1795>, and an approach to the A-ring of 1- α -hydroxyvitamin D started with the reaction between 3-trimethylsilyloxyfuran and the allenic ester shown in Equation (40) <93TL4369>. A novel route to phenanthrene derivatives takes advantage, as the key step, of the bis(Diels–Alder) reactions of a 4,4'-ethenyl-linked 3,3'-bifuryl <94TL4839>. Similarly, much of the work involving arynes has been involved in the synthesis of polycyclic <88AJC235> and macrocyclic systems <82JOC4370>, including reactions where arynes have been generated from bis(dibromo)[2.2]paracyclophanes <94CB2263>. New methods of generating arynes are normally authenticated by forming the adduct with furan and this was the case with the method that involves the reaction of phenylmagnesium bromide with *o*-chloro- and *o*-bromoaryl phenylsulfoxides such as 3-bromo-4-pyridyl phenylsulfoxide <87TL2727>. 3,4-Pyridyne has also been generated from the masked diazonium salt, 3-*N,N*-dimethyltriazolopyridine-4-carboxylic acid <89TL1507>. Anthracene and naphthalene derivatives have been prepared starting with reactions where arynes have been generated from 1,2,4,5-tetrabromobenzene and 2,3,6,7-tetrabromo-1,4,5,8-tetramethylnaphthalene <87T5203>. Benzyne chemistry has also been used together with a radical induced cyclization process <89JOC5073>. Regioselectivity in the cycloaddition reactions of a number of methoxy-substituted benzyne with substituted furans has been studied <91JCS(P1)1571, 91JCS(P1)1581>. The reaction of 3-methoxybenzyne with 3-methoxyfuran was found to be highly regioselective and gave a mixture of the two expected cycloadducts in the ratio of 8.6 : 1.4 <91JCS(P1)1589>. The major product was 2,5-dimethoxy-1,4-dihydro-1,4-epoxynaphthalene. Reactions of 3-fluorobenzyne with a number of 2-alkylfurans lead to mixtures of *syn*- and *anti*-cycloadducts. In the reaction with 2-*t*-butylfuran (Scheme 33) the *syn*- to *anti*-adduct ratio was 9 : 1 <88TL6227>. The aryne reaction involving the regioselective reaction of 2-methoxyfuran, shown in Scheme 34, was an early step in a synthesis of the antibiotic gilvocarcin <92JA3568>.





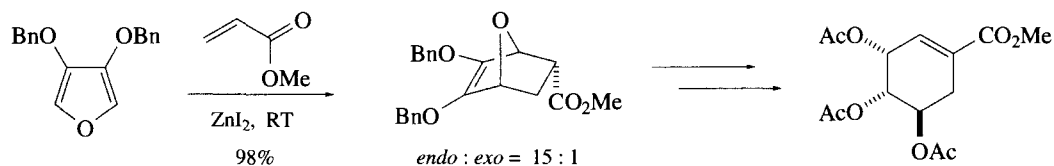
Scheme 33



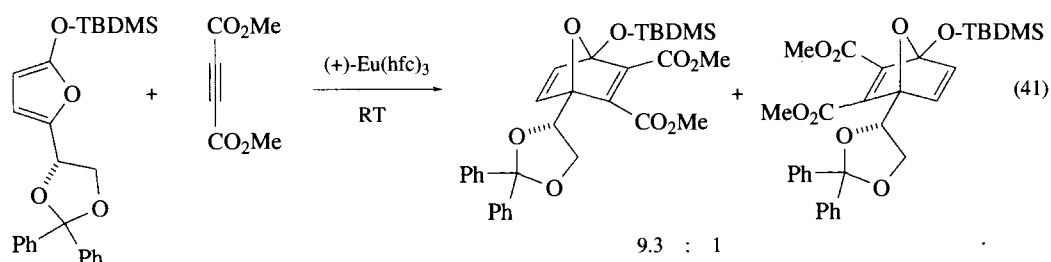
Scheme 34

A major objective in Diels–Alder chemistry relates to the fact that the method can generate four contiguous chiral centers and therefore if the absolute stereochemistry of one center is defined the other three follow (B-84MI 206-03, B-86MI 206-01). The kinetic preference for the formation of the *endo*-adduct in Diels–Alder reactions of alkenes has been reversed by using a cyclic porphyrin trimer to bind a diene and dienophile within its cavity (95AG(E)217). In the reaction of 3-(4-pyridyl)furan with *N*-(4-pyridylmethyl)maleimide the activation energy for the *exo*-reaction is reduced while that for the formation of the *endo*-reaction is increased. Two early routes to the important synthetic intermediate 7-oxabicyclo[2.2.1]hept-5-en-2-one ('naked sugar') (90BSB395, 90SL173, 93T5683) have been devised and the use of both enantiomers in the synthesis of carbohydrate derivatives has been summarized (93T5683). In one (83HCA1865, 87HCA690) an optically pure nonracemic dienophile was used and in the second route a cyanohydrin was resolved using brucine (84HCA1612). Efficient diastereoselective syntheses of 7-oxabicyclo[2.2.1]hept-5-en-2-carboxylate derivatives (86CC771) have been published together with the conversion into carba- α -DL-glucopyranose (92JOC1945). Reactions using (*R*)-5-[(1*R*)-menthyloxy-2-(5*H*)furanone (88JOC1125) provides another approach. A catalytic route to 7-oxabicyclo[2.2.1]hept-5-en-2-one has been described which used (α *S*, β *R*)- β -methyltryptophan in the reaction between 2-bromoacrolein and furan (93TL3979) and which then gave the adduct with an enantioselectivity of 96:4. A large number of syntheses based on this methodology include (+) and (–) methyl nonactate (87HCA690), (+)-lividosamine (92T10603), (+)-castanospermine (91JOC2128), ($\pm$)-cyclophellitol (92TL5243), and the all *cis*-Corey lactone (93JOC5895). An efficient concise synthesis of ($\pm$)-methyl triacetylshikimate, in an overall yield of 60%, outlined in Scheme 35, used 3,4-dibenzyloxyfuran as the diene component (89JCS(P1)2129) and (+) methyl 5-*epi*-shikimate has been prepared using the cycloaddition of furan with menthyl (*S*)-3-(2-pyridylsulfinyl)acrylate as the key step (89S189). Diels–Alder reactions of 2-silyloxyfurans derived from L-ascorbic acid show enhanced diastereofacial selectivity when specific protecting groups are used as indicated in Equation (41) (95TL1283). Other acetals did not give as high a diastereoselectivity and it is of interest to note that the chiral auxiliary and the chiral catalyst work cooperatively. In the case of related acetal having two *p*-methoxyphenyl residues the use of Eu(fod)₃

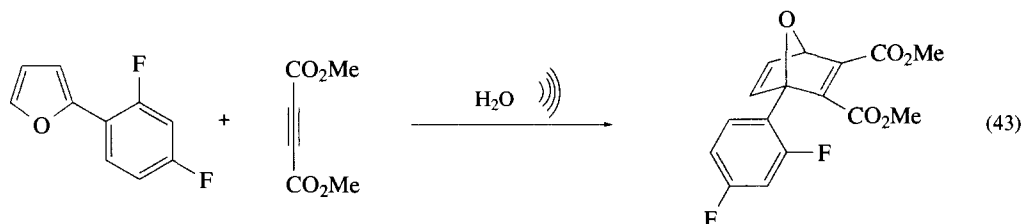
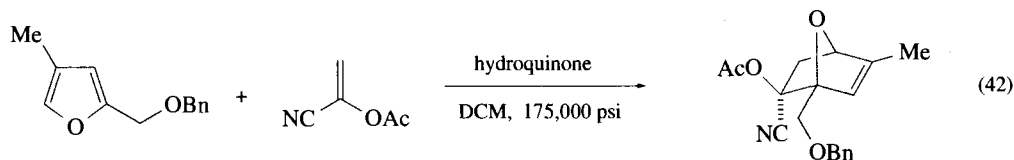
gave a diastereoselectivity of 3.7:1 as compared to 9.3:1 when using the chiral catalyst (+)-Eu(hfc)₃.



Scheme 35



One of the major disadvantages of traditional Diels–Alder methodology relates to the ease with which the retro-reaction occurs in some cases, particularly when the reactions have to be conducted at high temperatures. The reactions of dimethyl acetylenedicarboxylate with a number of 5-substituted 2-trimethylsilylfuran derivatives carried out under reflux conditions proceed to the dimethyl 5-substituted 2-trimethylsilylfuran-3,4-dicarboxylates <88H(27)1115> and dicyanoacetylene has been used in a similar way <93PAC47>. The retro-Diels–Alder reactions that involve the loss of ethene rather than ethyne proceed with lower activation energies and so it is sometimes more convenient to isolate the initial adduct and heat the 1,4-epoxy-1,2,3,4-tetrahydrobenzene derivative <91JFC(55)189>. The transition state in the Diels–Alder reaction occupies a smaller volume than the coreactants and so rates of reactions are significantly increased by methods that produce compression. This can be achieved either by using high pressures <83JOC3830, 83SC537>, including the first examples where a simple 1,4-benzoquinone functions as the dienophile in reactions with furan <83HCA218, 83HCA222>, or by carrying out the reactions in water. Both of these devices have been used in synthesis. The high pressure technique has been used in a large scale synthesis of cantharidin <85JOC2576>, (+)-jatropholones A and B <86JA3040> and, as exemplified in Equation (42), as a key step in an approach to the synthesis of furanoheliangolides <92JOC4512>. Diels–Alder reactions of 2-acetoxy- and 2-methylthio-furan proceed smoothly at room temperature and 15 Kbar <87H(26)2347>. An example of the approach using aqueous reaction conditions and sonication <93H(35)129> is shown in Equation (43).



Reactions where 1,3-diarylisobenzofuran derivatives act as the diene component in Diels–Alder reactions are well known and new routes have been developed, especially to isobenzofurans that are oxygenated in the benzenoid ring <87T3681, 89BSF441>. Diels–Alder reactions of isobenzofurans

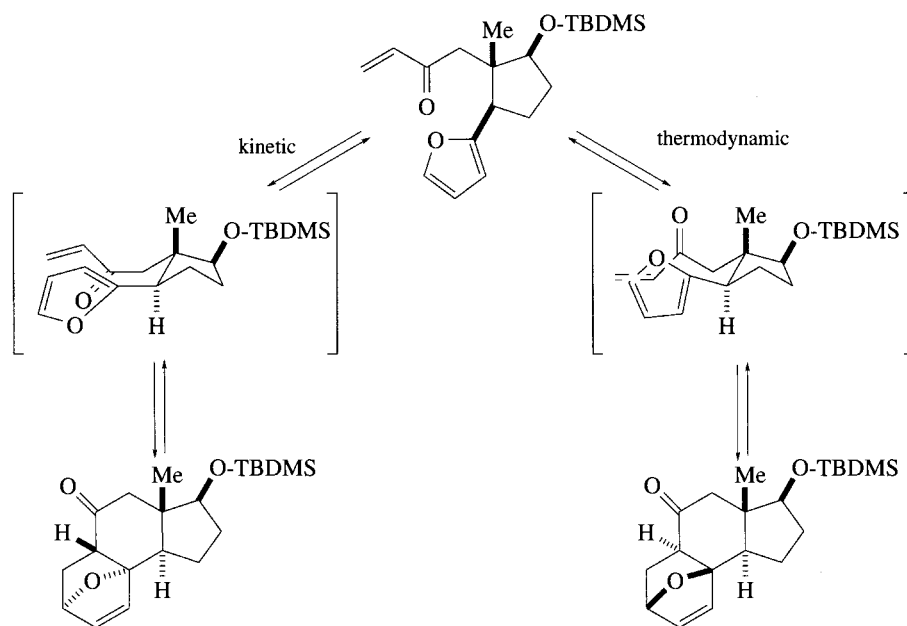
have been used in the synthesis of podophyllotoxins <89JOC4280>. Examples of dienophiles involved in their reactions include benzocyclopropene <87BCJ4141>, 1,7-dehydroquadracyclane <83CB2205>, and 1,2-dichloro-3,3-difluorocyclopropene <88HCA544>. Reactions between 1,1,1-trichloro-3-nitro-2-propene and furan, 2-methyl-, and 2,5-dimethyl- furan have been reexamined and products in addition to the expected Diels–Alder adducts have been found <84JOC4547>.

(ii) Intramolecular Diels–Alder reactions

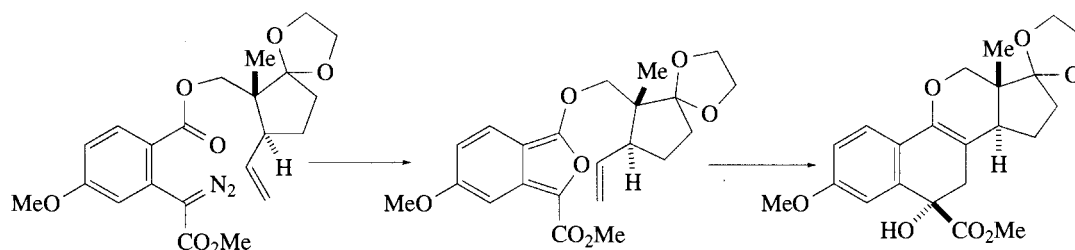
The development of a variety of intramolecular Diels–Alder (IMDA) reactions for use in the synthesis of natural products <80CR63> and the facility with which substituted furans can be assembled has resulted in IMDA reactions of furan derivatives being studied much more extensively since CHEC-I <84CHEC-I(4)599> was published. Various aspects of IMDA reactions have been reviewed <84OR(32)1, 86CRV795, 87CSR187, 90SL186>. Although IMDA reactions are entropically less disfavored than the intermolecular versions they are nonetheless not as simple as it might appear at first. The well known “Alder endo rule” and its modern frontier molecular orbital theoretical interpretation involving secondary orbital interactions, together with steric considerations, serve to explain the kinetic preference for the *endo*-product and the thermodynamic preference for the *exo*-product in intermolecular Diels–Alder reactions. For the IMDA reaction an additional parameter, the ability of the tether that connects the diene to the dienophile to fold into the conformation required in the transition state, has to be considered. A model has been developed that is based on explicit starting material and product conformations using a combination of a conformational search program (WIZARD) and the energy calculating program MM2 <92JA10738>. The reactions provide routes to a wide range of important polycyclic systems, including examples involving isobenzofurans <86H(24)297> and, by construction of a thiophene annelated furan, a benzo[b]-thiophene synthesis <88TL1137>. An interesting approach to the clerodane ring system is provided by an inverse electron demand IMDA reaction where the tethered furan functioned as the dienophile in a reaction with a 1,2,3,4,4a,8a-hexahydronaphthalene-5-carboxylic acid <92AJC2089>. A route to 11-keto steroids was based on a furan IMDA sequence <79SC771> for the construction of rings B,C, and D. The cyclization of the furan derivative shown in Scheme 36 was found to be reversible <82TL3283, 83TL3145, 84BSB1019> and after six days at room temperature there was a 61% conversion to an 8 : 1 mixture of the kinetic and thermodynamic products whereas in benzene at 80°C there was a 50% conversion to a 1 : 9 mixture in favor of the thermodynamic product. It is interesting to note that the free alcohol related to the furan derivative shown in Scheme 36 cyclized exclusively to the kinetic product when it was shaken in water for 10 min <85T4667>. A cascade synthesis of 11-oxasteroids, functionalized at the 6-position, starting with an isobenzofuran synthesis and then an IMDA reaction, is exemplified in Scheme 37 <88TL2045>. A number of pericyclic reactions have also been reported where vinylfurans effectively function as the diene component in IMDA reactions <84JA6735, 86TL947, 87CB355, 88JOC860, 88TL2107>. The methodology has been used in approaches to naturally occurring sesquiterpenes of the furanoeremophilane group <93JCS(P1)2395>. Thermolysis gave the product shown in Equation (44) in almost a quantitative yield by cyclization followed by isomerization of the exocyclic double bond to rearomatize the furan residue by what is effectively a disfavored 1,3 hydrogen shift. As indicated in Equation (44), the reaction proved to be highly stereoselective. IMDA reactions have been included increasingly as key components in the synthesis of natural products, for example, to construct an intermediate tricyclic furan for use in approaches to viridin and related compounds <87CC515>. The dienophiles that have been tethered to a furan include the obvious possibilities: alkynes, allenes, arynes, and alkenes.



One of the early examples of a furan IMDA reaction involved a multistep route to a precursor alkyne that gave a mixture of two diastereomeric cycloadducts as an entry to a functionalized gibbane <82TL3291>. The protocol was extended to a relatively short route to gibberelins, for example ($\pm$)GA5, and made use of an alkyne–furan IMDA reaction. The IMDA reaction, when carried out in benzene at 80°C proceeded slowly and gave a low yield of a 3 : 1 mixture in which the required

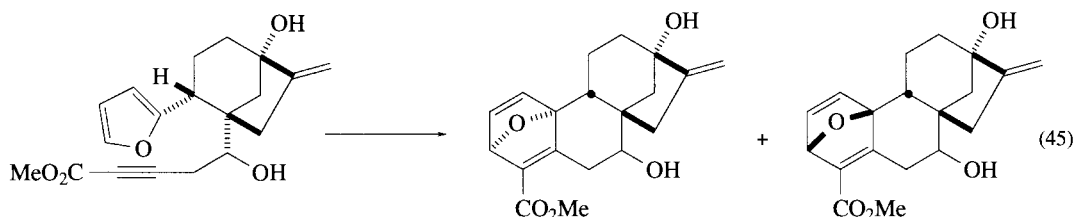


Scheme 36



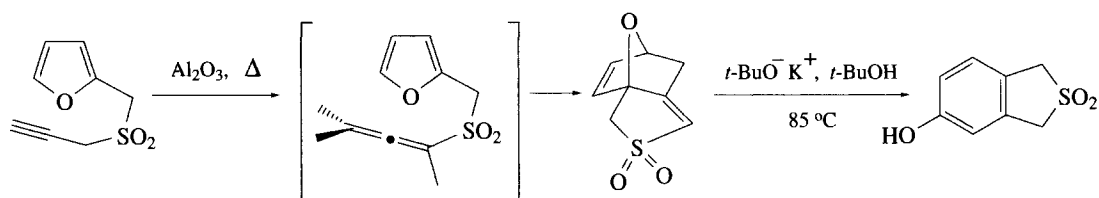
Scheme 37

diastereoisomer, with the oxygen bridge α -, was the major product. The rate of the reaction and the yield was improved when carried out in water in the presence of β -cyclodextrin ^[86TL1731]. Similar reactions, for example, as shown in Equation (45), when carried out in benzene were also found to be slow and not particularly stereoselective. However, like the earlier results, the reactivity and stereoselectivity was improved (45% yield $\alpha:\beta = 6\%:75\%$) when using β -cyclodextrin in water. Interestingly the major, kinetically preferred diastereomer had the opposite configuration of the oxygen bridge (β) ^[88T4605]. In a series of IMDA reactions aimed at the synthesis of phorbols it was found that the use of an acetylenic dienophile residue gave no control over the stereochemistry at the A–B ring junction and the oxygen bridge ^[90CC605, 90CC608]. The IMDA reactions involving furan derivatives using a four carbon tether between the diene and the dienophile units are known to be problematic and a number of solutions have been reported. In the case of an acetylenic dienophile Lewis acid catalysis using methylaluminum dichloride was found to be particularly effective ^[91SL353].

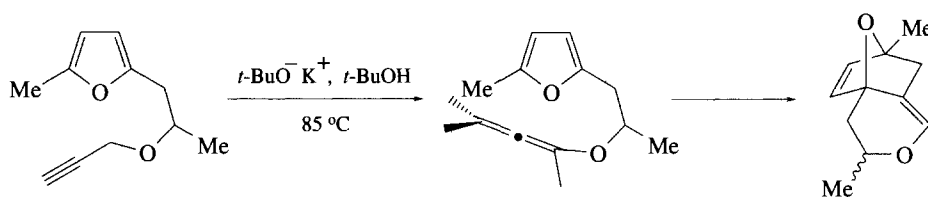


The IMDA product from the propargyl sulfone shown in Scheme 38 proceeds via the allene and was converted subsequently into the benzenoid sulfone under base catalysis ^[92CC735]. Propargylic

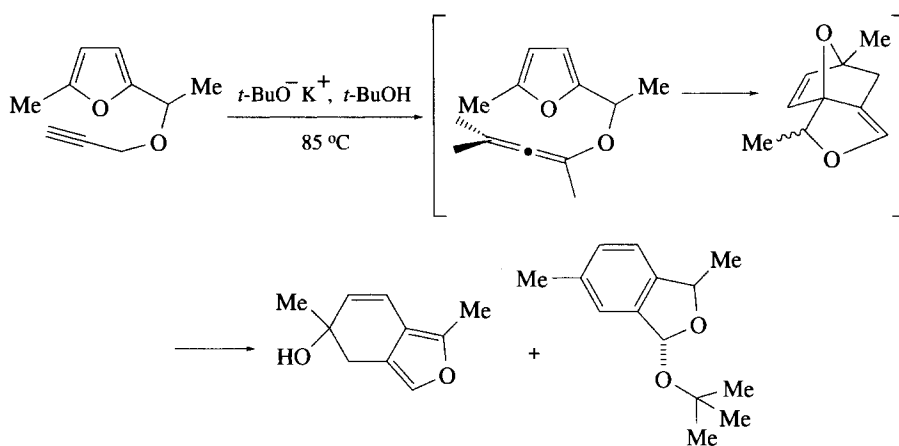
ethers can also be isomerized to the related allenyl ethers using base catalysis and the products that are then obtained in IMDA reactions are highly dependent on the chain length of the tether $\langle 94H(38)1507 \rangle$ as indicated in Schemes 39 and 40. The ring transfer product presumably arises because the initial IMDA adduct is highly strained with the shorter tether and fragments by cleavage of the epoxide bridge. Base catalyzed prototropy then results in the formation of the aromatized product. One of the products shown in Scheme 40 is the typical furan ring transfer product which was also used in an approach to the syntheses of spongiaditerpenes $\langle 94TL5677 \rangle$. Two effective synthetic approaches to $(\pm)$ -euryfuran, one of the drimane marine sesquiterpenoids, have been described, both of which involve furan ring transfer reactions $\langle 94T5645 \rangle$. The protocol has also been used in a synthesis of xestoquinone $\langle 91TL4761 \rangle$ and in chirality transfer processes with moderate to good enantioselectivity $\langle 89CC470, 92TL4967 \rangle$. A similar reaction to that shown in Scheme 40, where the substituent at the 5-position in the original furan ring was the methylthio group, resulted in the formation of the dihydrobenzo[c]furan where the methylthio group had undergone a 1,4-rearrangement as outlined in Scheme 41, possibly involving the fragmentation to a zwitter ion and thiosulfide migration to the oxonium center $\langle 94TL729 \rangle$. A new synthesis of the isoindole ring system also takes advantage of the ring exchange protocol and is illustrated in Scheme 42 $\langle 94CC1535 \rangle$.



Scheme 38

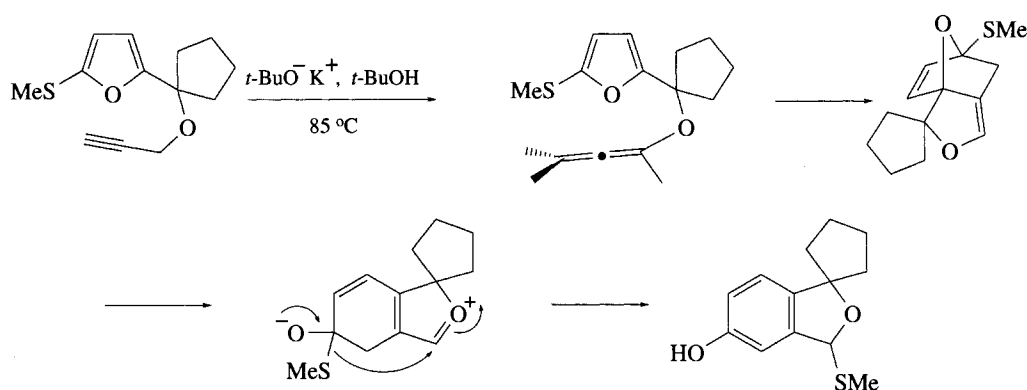


Scheme 39

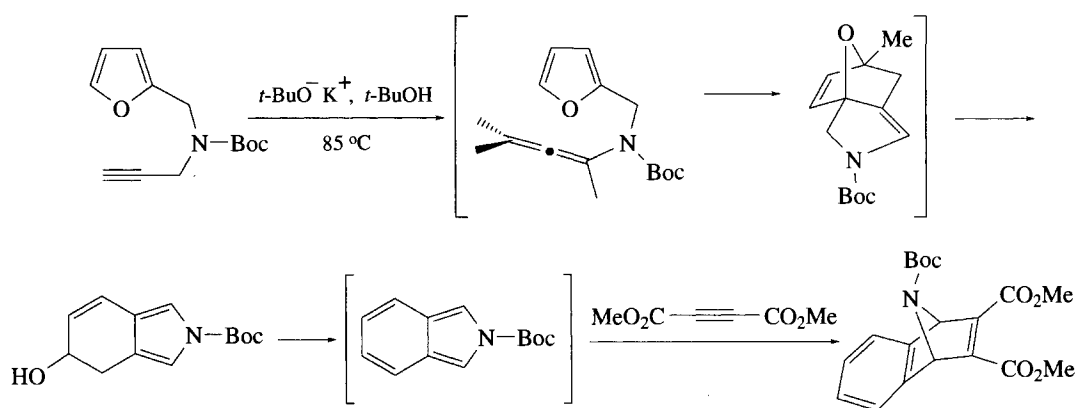


Scheme 40

The IMDA–Grob fragmentation strategy that starts with an allene cycloaddition reaction is illustrated in Scheme 43. The allene–furan cycloadduct was formed together with the other *exo*-adduct which had the opposite configuration at the *i*-propyl group. Partial isomerization of the other product into the thermodynamic product shown and conversion into the 10-membered ketone constituted a formal synthesis of periplanone-B $\langle 88TL6501 \rangle$ since that ketone had been used in an

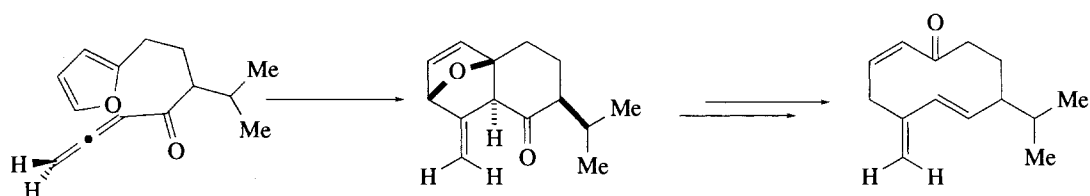


Scheme 41



Scheme 42

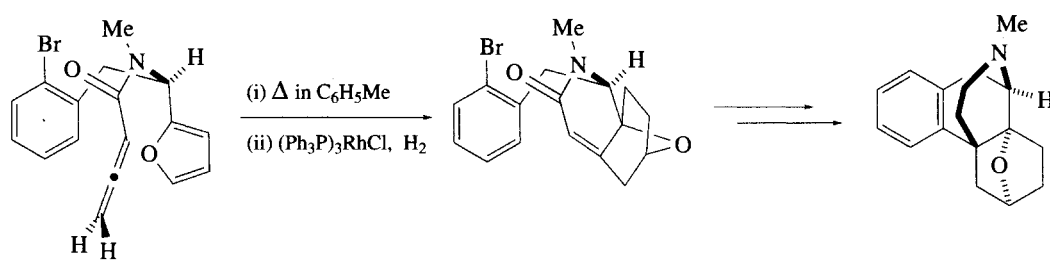
earlier synthesis of periplanone. A cascade allene initiated IMDA-radical cyclization procedure has been described (Scheme 44) for the construction of the morphinan ring system $\langle 89\text{TL}2585, 90\text{SL}384 \rangle$. The IMDA step involved cycloaddition of the terminal double bond of an allenic amide. Subsequent steps involved double bond reduction, radical cyclization, and reduction of the amide carbonyl group.



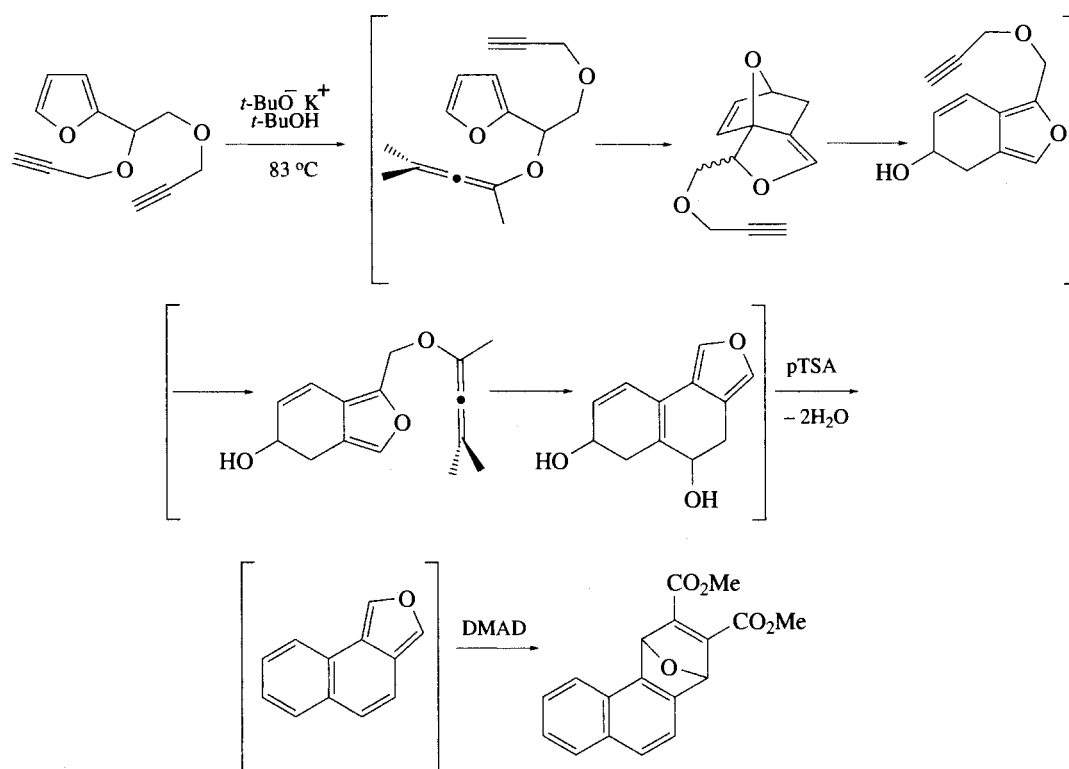
Scheme 43

The ring transfer strategy has been extended (Scheme 45) in a complex sequence of reactions in which a furan was converted into 1,2-naphtho[c]furan using a cascade strategy. A tethered alkyne was first reacted with a furan, possibly being first converted to an allene in a base catalyzed isomerization reaction, and then to the product of ring interconversion then being converted into 1,2-naphtho[c]furan by base catalyzed ring opening, reaction with a second tether, in that case an allene, and acid catalyzed dehydration $\langle 85\text{TL}2689 \rangle$. The unstable product was finally reacted with dimethyl acetylenedicarboxylate in an intermolecular Diels–Alder reaction.

Although arynes are recognized as highly reactive dienophiles there are relatively few reports of IMDA reactions where those reactive intermediates are tethered to a furan. A three or four atom tether linking the aryne precursor to the furan leads to efficient reactions $\langle 86\text{AJC}635 \rangle$. The use of a

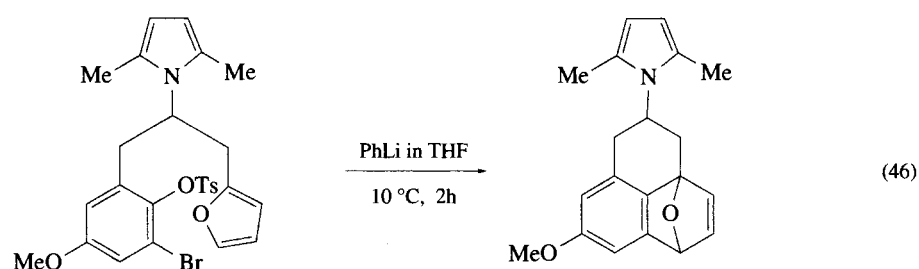


Scheme 44

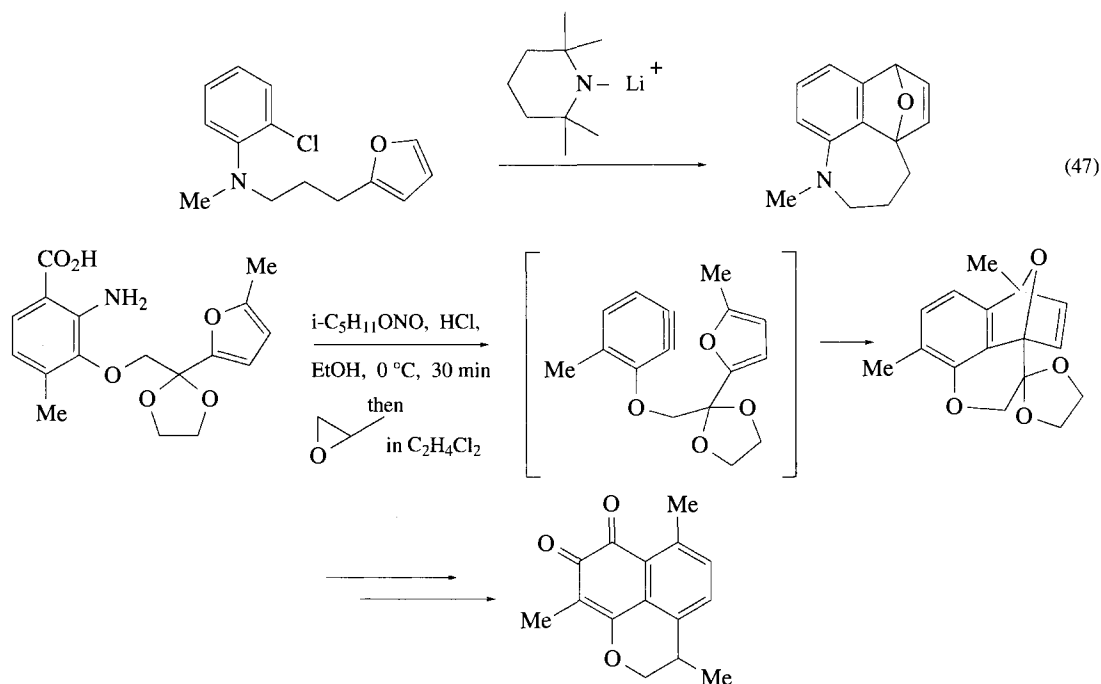


Scheme 45

three atom tether with an amino group protected, most interestingly, as a pyrrole, was used in a synthesis of 2,3-dihydro-1H-phenalene [\(88TL1883\)](#). The key step is shown in Equation (46). The generation of an aryne from an *o*-haloaniline derivative has been used [\(94H\(38\)31\)](#) in a new route to tetrahydrobenzazepines and is illustrated in Equation (47), and intramolecular benzyne cycloaddition reactions have also been used as a key step in the synthesis of the naturally occurring *o*-naphthoquinone mansonone E [\(81TL4877\)](#) (Scheme 46) and mansonones I and F [\(86AJC647\)](#).

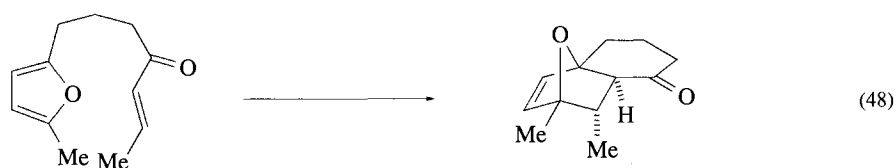


(46)



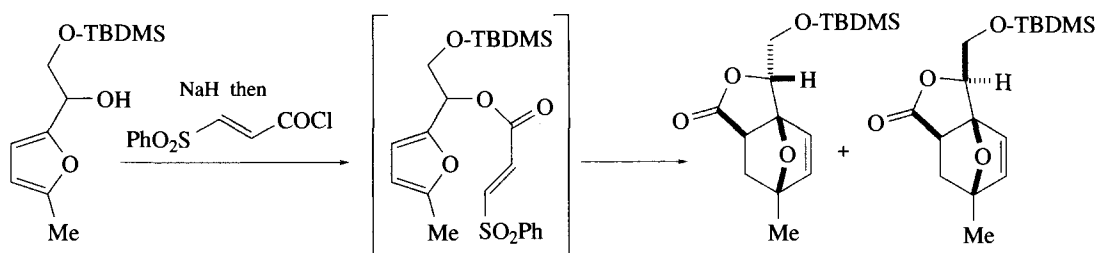
Scheme 46

The effects of a variety of changes in reaction conditions have been reported using the model reaction shown in Equation (48). Other examples with analogues having different substituents at position-5 in the furan and on the dienophile alkene residue, or as part of the tether, show the same effects. A methyl group at position-5 on the furan was shown in one case to result in an increase in the rate of cyclization of about 20 times [\(84JA8327\)](#). Lewis acids such as methylaluminum dichloride [\(91SL353\)](#), 2.0 M calcium chloride [\(87CC419\)](#), and increased pressure, for example, 12.5 Kbar at room temperature [\(89TL1045\)](#), all cause significant rate increases. Remarkably, the yields obtained in IMDA reactions of 5-substituted 2-*N*-furylmethyl-*N*-aryl-*N*-allyl amines give enhanced yields of the cycloadducts, when compared to the yields obtained where the substituent at the 5-position is hydrogen, irrespective of whether the 5-substituent is electron releasing or electron withdrawing [\(87JHC1787\)](#).

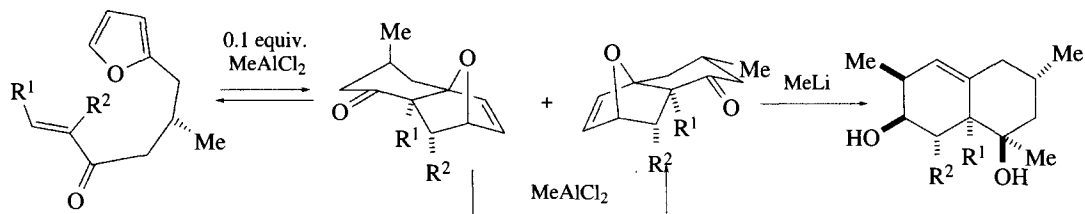
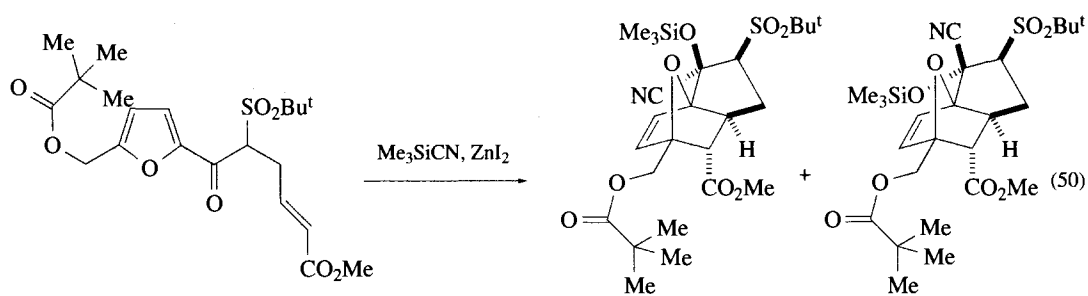
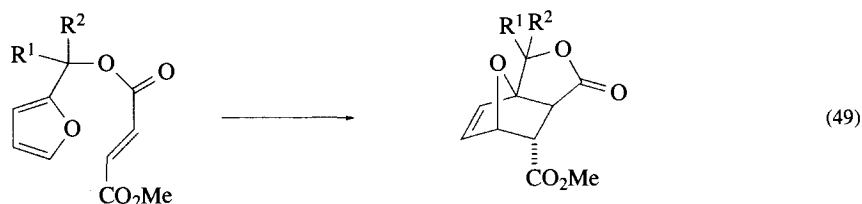


The effects of substituents on the furan [\(85JOC1770\)](#) and adjacent to the dienophile moiety have also been studied [\(88TL2493\)](#). An IMDA reaction that uses an (*E*)-2-phenylsulfonylacrylate as the dienophile component has been described [\(88TL6059\)](#) as part of a potential route to the benzofuran residues in the milbemycins and avermectins. The high reactivity of the electron depleted dienophile moiety results in a rapid reaction at room temperature to give the two diastereomers shown in Scheme 47. The IMDA reaction of the fumaric acid derivative shown in Equation (49) showed substantial rate increases when both R^1 and R^2 were alkyl groups due to higher populations of the reactive rotamers [\(88TL2429\)](#), the so called 'reactive-rotamer' effect, and also in the more polar solvent systems [\(89JA5469\)](#). The key experiments that allowed the possibility of angle compression to be studied and rejected in favor of the reactive rotamer effect involved the cyclobutane derivative [$R^1-R^2 = (CH_2)_3$] in Equation (49). A similar effect has been noted in a study of remote electron withdrawing substituents in the IMDA reactions of the 2-sulfone substituted 1-furyl-4-pentenyl-1-ols [\(94T6767\)](#), and an example is shown in Equation (50). The rates of the reactions and the product yields were observed to increase as the steric demands of the alkyl group on the sulfone was

increased. The presence of an appropriate electron withdrawing group such as methoxycarbonyl resulted in the system becoming very reactive and cyclization then occurred at ambient temperature. The photooxidation reactions noted in Section 2.06.2.2.9 that result in ring cleavage have been used with 5-alkyl-2,2'-difurylalkanes to generate an intramolecular dienophile by using the generation of singlet oxygen in the presence of methanol using Rose Bengal as the sensitizer (90TL7201). The success of the reactions depends on the regioselectivity in the oxidation step and, as expected, the disubstituted furan ring is converted into the hydroperoxide at a faster rate than the monosubstituted ring. The position of the equilibria in the IMDA reactions were found to vary in the presence of various catalysts and, as in the case of a number of IMDA reactions, zinc iodide gave the highest yields of the cycloadducts. This methodology has been elaborated to allow the stereoselective synthesis of decalin derivatives. High enantiomeric excesses have been obtained IMDA reactions leading to decalin derivatives, illustrated in Scheme 48, using optically pure starting materials in thermodynamically controlled reactions catalyzed by methylaluminum dichloride (94TA141).



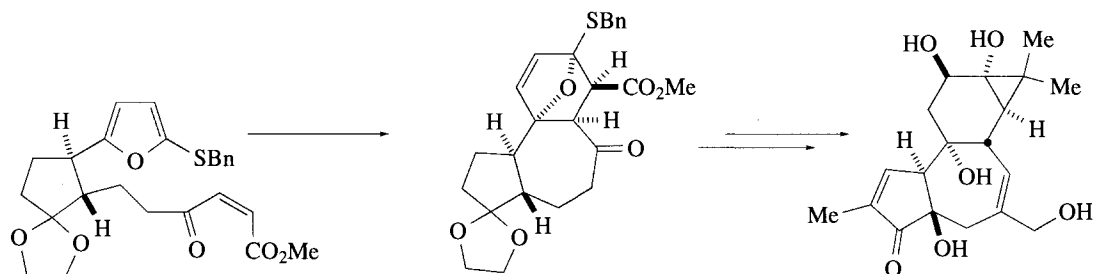
Scheme 47



Scheme 48

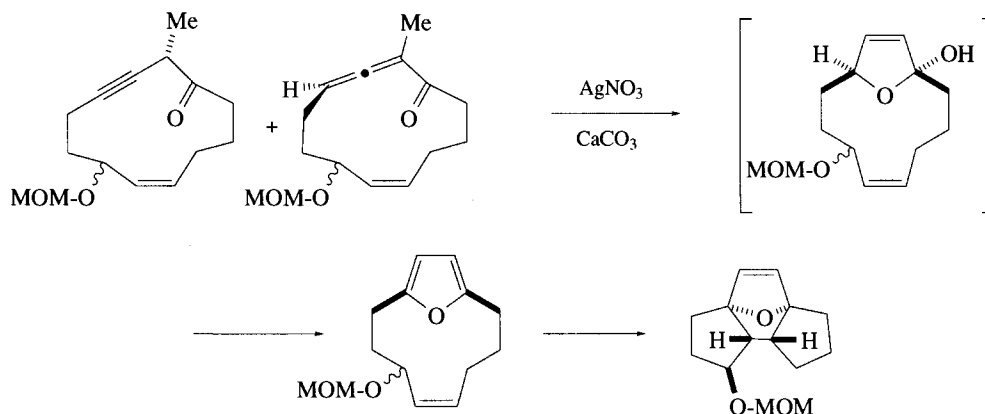
The approaches to the carbocyclic system related to phorbol which were developed using an acetylenic dienophile (*vide ut supra*) have been followed by an IMDA reaction of the 5-ben-

zylthiofuran derivative shown in Scheme 49 (91CC527). The modification of the earlier approach led to a number of important improvements to the protocol. The benzylthio-substituent at the bridgehead permitted easy cleavage of the oxygen bridge. In addition the new procedure, that included the use of high pressure reaction conditions, led to a significantly improved yield and stereoselectivity.



Scheme 49

Part of a program directed towards the synthesis of biologically active cembranoids involved the preparation of 2,5-furanocycles (92JOC3387). Remarkably, it was found that treatment of a mixture of the propargylic and allenic ketones with a catalytic amount of silver nitrate and calcium carbonate in aqueous acetone gave on TLC analysis two spots one polar and one less polar. The polar and less polar spots gave way to two less polar spots and eventually to one less polar spot which was identified as being due to the tricyclic product of the stereospecific transannular Diels–Alder reaction as shown in Scheme 50. Presumably the reaction between the furan moiety and what would normally be regarded as an unreactive alkene fragment relates to the relief of the strain energy associated with the furan macrocycle.

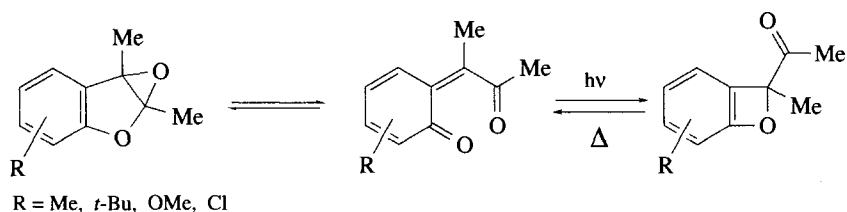


Scheme 50

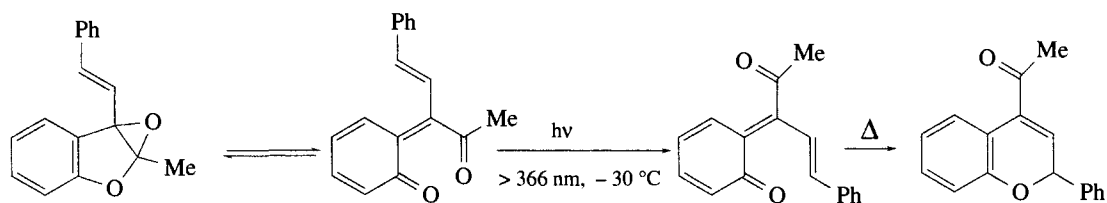
2.06.2.7 Photochemical Reactions

The photosensitized oxidation of furans has been discussed in section 2.06.2.2.9 in connection with the synthesis of butenolides. The epoxidation of 2,3-dimethylbenzofuran and 2-methyl-3-phenylbenzofuran using DMD and the thermal ring opening to quinonemethides was also discussed in section 2.06.2.2.9. The photochemical reactions of the quinonemethides derived from a number of 2,3-dimethylbenzofurans have been studied and shown to undergo 2 + 2 cycloaddition to give thermally unstable benzoxetes as shown in Scheme 51 (94CB1115). The quinonemethides can be intercepted in intermolecular 4 + 2 cycloaddition reactions with, for example, ethyl vinyl ether. The possibility of intercepting a quinonemethide in an intramolecular reaction has been addressed by studying the reactions of 2-methyl-3-styrylbenzofuran (94CB433). The ring opening of the 2,3-epoxide occurs regiospecifically to give the wrong diastereomer for cyclization. However, photoisomerization at low temperature led to the isolation of 4-acetyl-2-phenyl-2(*H*)chromene as shown

in Scheme 52. The photooxidation of tetrabromofuran in solution in benzene gave dibromomaleic anhydride but in the solid state tetrabromofuran-2-one was obtained (87AJC1137).

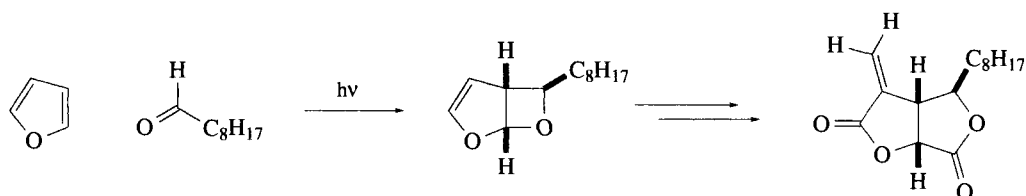


Scheme 51



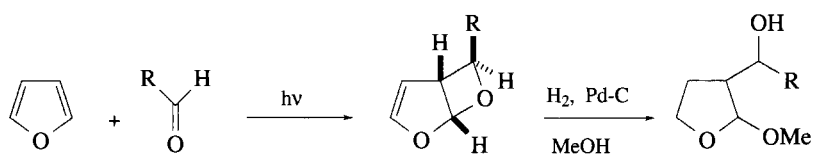
Scheme 52

The 2 + 2 cycloaddition reaction of an alkene with carbonyl compounds, the Paterno-Büchi reaction, has been investigated using furan and a number of aldehydes, and provides a method for the addition of an enolate equivalent and gives access to *threo*-aldol products and hence by further elaboration to a variety of highly functionalized systems with a good degree of stereochemical control (83JA660). This protocol was exemplified in the synthesis of the bis(tetrahydrofuran) moiety of the mycotoxin, asteltoxin (83JA6723) and also in a total synthesis of racemic avenaciolide (84JA7200) that started (Scheme 53) with the photocycloaddition reaction of nonanal to furan. The importance of the method is apparent because, not only can the reaction be carried in high yield and with complete stereochemical control, but also the reaction can be carried on a multigram quantities of materials. A wide variety of compounds have been studied including aromatic carboxaldehydes (90CB357), together with furan and substituted furan derivatives. Reactions using furfural diacetate and the ethylene acetal of furan-2-carboxaldehyde in reactions with benzophenone (84CCC71) and 2-acetylfuran (92JCS(P1)1081) gave, as the predominant product, the most highly substituted oxetane but the photocycloaddition of butyl glyoxylate to 2-tributylstannyl furan, and a silicon substituted furan, resulted in reversed regiochemistry (88TL6689). Reduction of the products in methanol give 2-methoxy-3-substituted tetrahydrofuran derivatives (Scheme 54) which can be carried on to α -alkylidene- γ -lactones (84JP1839). Acid catalyzed oxygen exchange occurred exclusively at the carbonyl oxygen in 4 : 7, 10 : 13-diepoxy[15]annulenone, effectively a cyclic bifurylthene linked to a penta-1,4-dien-3-one residue. That allowed the degenerate photochemical rearrangement of 4 : 7, 10 : 13-diepoxy[15]annulenone to be studied by ^{17}O NMR spectroscopy (91CC1775).



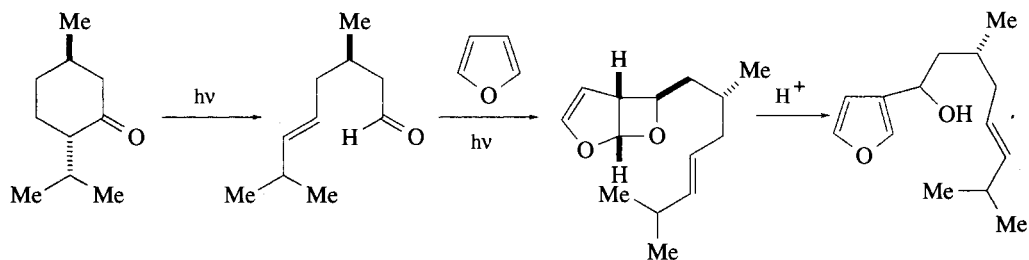
Scheme 53

Paterno-Büchi reactions using chiral alkyl glyoxylates have been investigated but only low optical purities were observed (82T1447). The photochemical reaction between ketones such as menthone proceed by an initial Norrish type-I cleavage followed by the Paterno-Büchi reaction which allows the isolation of 3-furlycarbinols after acid catalyzed isomerization as shown in Scheme 55 (82T1453). Esters (89JOC140) and aryl thioamides (89TL44221) have also been shown to undergo 2 + 2 photochemical cycloaddition reactions and in the latter case provide a route to 3-furyl aryl ketones as exemplified in Scheme 56. Intermolecular 2 + 2 cycloaddition reactions of alkenes with furan

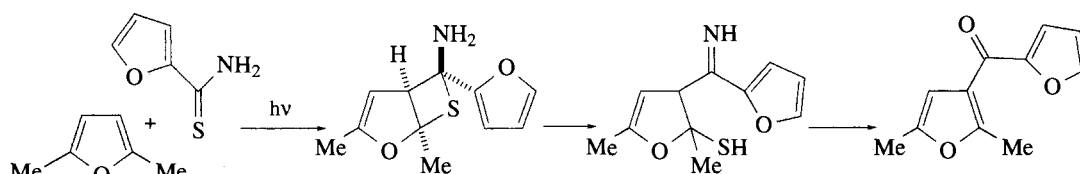


Scheme 54

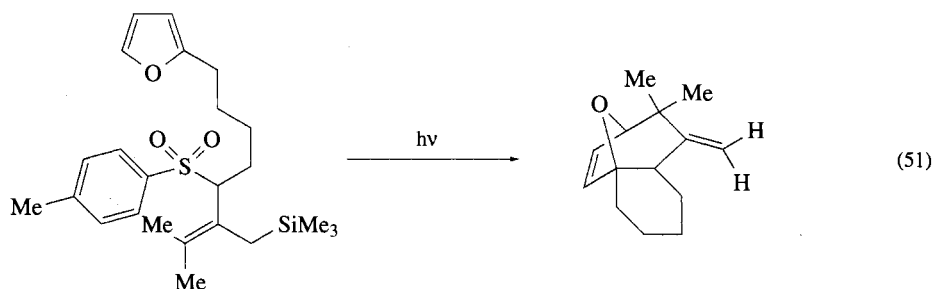
<89JOC135> and benzofuran <83CC501> derivatives as well as intramolecular variants <89H(29)679> have also been studied as illustrated in Equation (51) <93TL5381>.



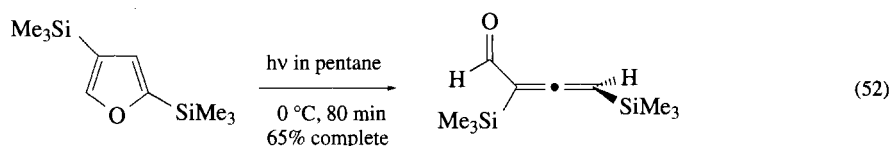
Scheme 55



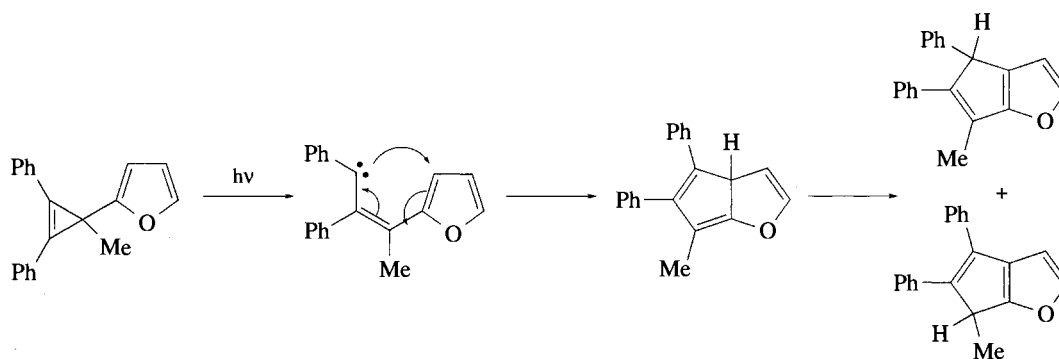
Scheme 56



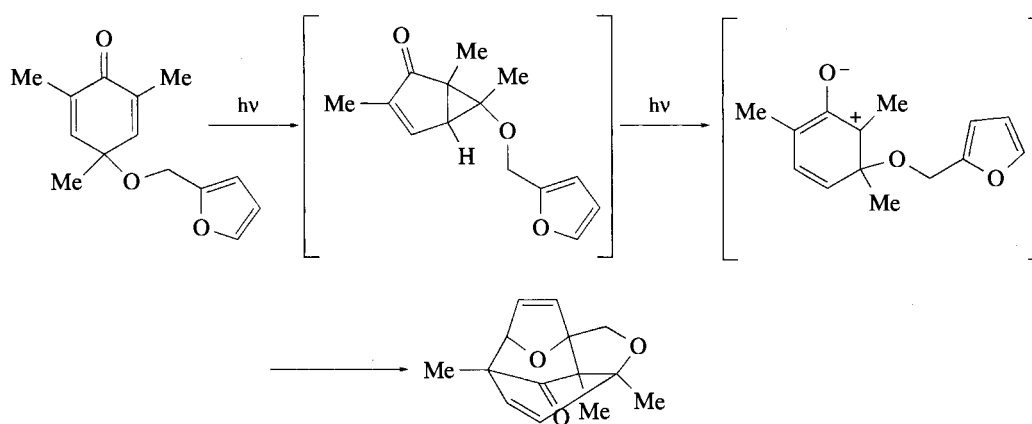
The photochemistry of a number of heterocyclic 3-substituted cyclopropenes has been studied including 3-methyl-3-(2-furyl)-1,2-diphenylcyclopropene <83JCS(P1)915>. The rearrangement proceeds from the $\pi-\pi^*$ singlet state of the cyclopropene which undergoes ring opening, as shown in Scheme 57, to a vinylcarbene and then by an electrocyclic reaction to a transient intermediate which can give the observed products by 1,3- or 1,5-sigmatropic hydrogen shifts. In another photorearrangement the 4,4-disubstituted cyclohexadienone gave the bridged furan adduct shown in Scheme 58 <85CC785> and the remarkable rearrangement of 2,4-bis(trimethylsilyl)furan <83JA6316> is shown in Equation (52).



The photochemical arylation reactions of furans and thiophenes have been reviewed <89G419>. The irradiation of 3- and 5-bromo-2-formylfuran in aromatic solvents has been shown to give the

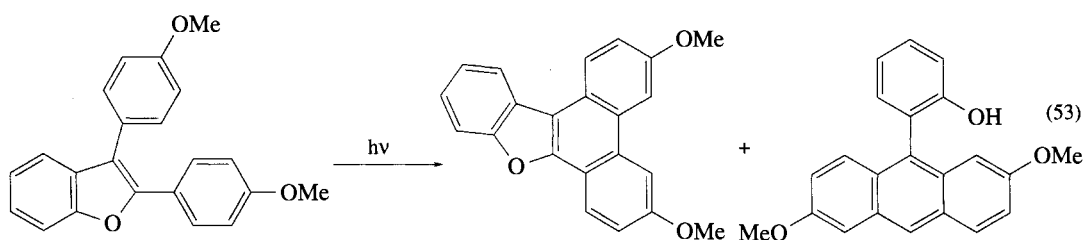


Scheme 57

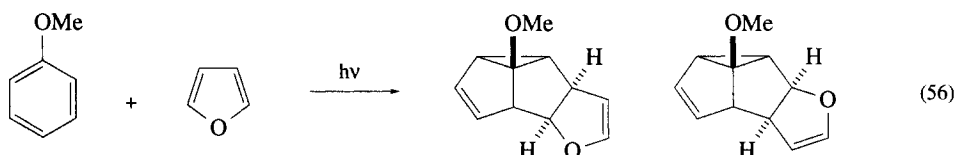
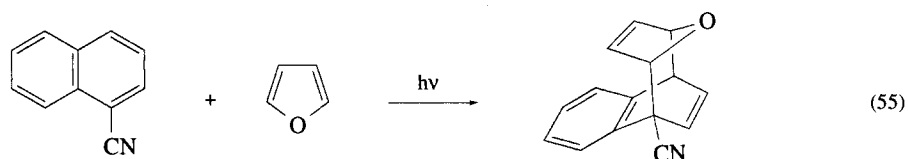
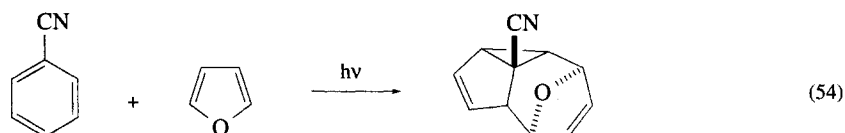


Scheme 58

3- and 5-aryl-2-formylfurans in good yields and 4,5-dibromo-2-formylfuran and 4,5-dibromo-2-acetylfuran gave the 4-bromo-5-aryl derivatives (85JCS(P1)1285). Other 2-acyl-5-arylfurans have been prepared in the same way while irradiation in either diethyl ether or di-isopropylamine resulted in the intermediate radical abstracting a hydrogen atom (85T3441). Coupling reactions have been reported in which 5-bromo- or 5-iodo-2-formylfurans react with alkenes such as indene (89H(29)1331), 2-vinylthiophene (90JOC4019), naphthalene (86H(24)1575), and styrene (93G129). On the other hand reactions of 2-furylallyl acetate and a number of iodofuran derivatives, including 5-iodo-2-acetyl-, 5-iodo-2-formyl-, and 5-iodo-2-methoxycarbonylfuran do not couple at the alkene residue on photoirradiation but give the 5,5'-disubstituted-2,2'-bifurys (94JCS(P1)1245) as a result of aryl-aryl coupling. The allylic alcohols do not couple but give the photoreduction products of the halofurans. The photochemistry of furancarboxanilides has been shown to give a number of product types, including compounds derived from the initial cyclization products, (3,4-dihydro-(2,3-dihydrofuro)-quinolin-2-(1*H*)ones) (87JOC3178). The related photocyclization reaction of 2,3-bis(*p*-methoxyphenyl)benzofuran was shown to give the expected phenanthrene derivative and in addition the novel rearrangement product, 2,6-dimethoxy-9-(2-hydroxyphenyl)anthracene (84CC613) shown in Equation (53).



Photocycloaddition reactions of furans to carbocyclic aromatic compounds have been studied in some detail. The reaction mode and the regiochemistry of the addition reactions are very dependent on the substituents on the arene. Photoadducts formed in 2 + 3, 4 + 3, and 4 + 4 processes have all been identified. Empirical rules that allow the rationalization of the modes of photo-reactions of arenes with alkenes have been presented $\langle 85T2405 \rangle$ and orbital symmetry and polarity considerations have been summarized in connection with the types of intermediates involved $\langle 86T6011 \rangle$. Unlike the photoaddition reactions of alkenes to benzene derivatives, the ease of electron transfer between the addends is not the dominant factor. Both phenylacetylene and benzonitrile undergo regio- and stereospecific 2,6, 2',5'-photocycloaddition to furan, illustrated in Equation (54) for the benzonitrile reaction, to give the 4 + 3 cycloadducts $\langle 85CC1057, 90JCS(P1)931 \rangle$. The directing influence of the nitrile substituent was considered to result from the stabilizing effect of the cyano group on the developing negative charge in the polarized S_1 state of the benzenoid ring formed on the approach of the S_0 1,3-diene $\langle 94JCS(P2)247 \rangle$. The single 4 + 4 photocycloadduct that is formed between 1-cyanonaphthalene and furan was mentioned in CHEC-I $\langle 84CHEC-I(4)599 \rangle$. A single crystal x-ray structure determination has established the structure as the *endo* isomer $\langle 79BCJ1634 \rangle$, as shown in Equation (55). Furan behaves like an enol ether towards photoexcited anisole and gives a pair of *exo*-dihydrosemibullvalene derivatives shown in Equation (56).

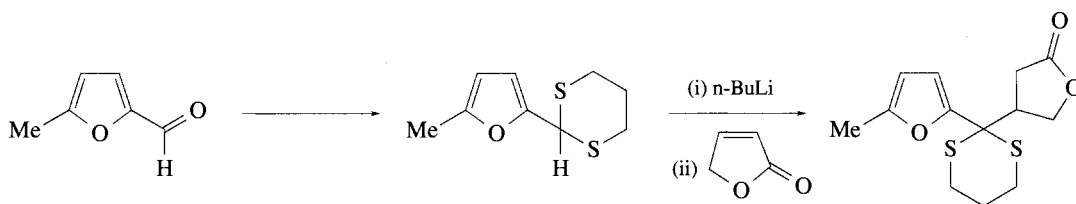


2.06.3 REACTIVITY OF SUBSTITUENTS

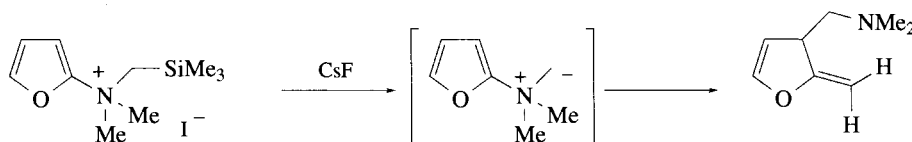
2.06.3.1 Alkyl and Substituted Alkyl Substituents

The oxidation of a 2-methyl group to the corresponding carboxylic acid has been reported using potassium permanganate for a number of 1-methyl-2-(5'-methylheteroaryl-2')-benzimidazoles including 1-methyl-2-(5'-methylfuryl-2')-benzimidazole $\langle 82KGS1089 \rangle$, and the oxidation of 2-(hydroxymethyl- ^{14}C)furan with γ -manganese dioxide was used in a preparation of 2-[^{14}C]formylfuran $\langle 86MI 206-02 \rangle$. The preparation of a series of 4-substituted 5-(*N,N*-dimethylamino)-2-formylfurans involved the addition of primary and secondary amines to furfurylideneammonium salts $\langle 86CCC573 \rangle$. A convenient route to a number of 3-alkyl and 3-alkenylfuran derivatives has been developed whereby a lithium dialkylcuprate or a diallylcuprate was used to displace toluene-*p*-sulfonate from furan-3-methyl toluene-*p*-sulfonate $\langle 83JOC1106 \rangle$. NBS continues to find use for the preparation of benzylic type bromides, for example, in the benzofuran series $\langle 83KGS1460 \rangle$ and 2,5-dichloromethylfuran has been used as a precursor for the preparation of a highly conductive high molecular weight poly(2,5-furylene vinylene) $\langle 87CC1113 \rangle$. A number of reactions starting from 2-bromomethyl-3-benzoyl-5-acetoxybenzofuran have been reported and include its conversion, via the hydroxymethyl derivative, to 2-formyl-3-benzoyl-5-acetoxybenzofuran $\langle 83KGS178 \rangle$. Radical-nucleophilic

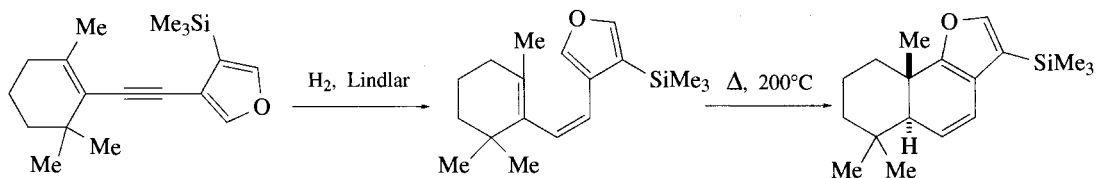
substitution ($S_{\text{RN}}1$) reactions of a number of α -substituted 2-methyl-5-nitrofuran derivatives have been reported. One report was concerned with the reactions of the anion from 2-nitropropane which gave products derived by initial attack by carbon ($S_{\text{RN}}1$) and attack by oxygen ($S_{\text{N}}2$) $\langle 85\text{JCR}(\text{S})1814 \rangle$ while the other report $\langle 87\text{JOC}2796 \rangle$ was concerned with the reactions of the anions derived from 2-nitropropane and 2-methylmalononitrile on 2-(1-chloro-2,2-dimethylpropyl)-5-nitrofuran. The ring chain isomerism of *N*-(2-hydroxyalkyl)nitrones that can be made from furan-2-carboxaldehyde and its 5-dialkylamino- and 5-nitro-derivatives has been studied by using the acylation reactions using diphenylborinic acid, isocyanates, as well as carboxylic acids $\langle 83\text{CB}27 \rangle$. 2-Furylcarbene, generated from the tosylhydrazone has been described as singlet species $\langle 90\text{H}(31)115 \rangle$. Deprotonation at the benzylic position in 5-trimethylsilyl-2-phenylthiomethylfuran using *n*-butyllithium, followed by reaction with an electrophile and desulfurization, allowed overall functionalization of 2-methylfuran after finally removing the trimethylsilyl group $\langle 85\text{TL}6225 \rangle$. Alternatively, the same overall objective was achieved via the diphenylthioacetals. The conversion of furan-2-carboxaldehyde and a number of its derivatives into the related dithianes has allowed reactions of the acyl anion equivalent to be carried out. In the example shown in Scheme 59 the conjugate addition of the anion to furan-2(5*H*)-one was successful $\langle 94\text{JCS}(\text{P}1)45 \rangle$. An interesting method of generating the ylides derived from quaternary salts that are related to Mannich bases depends on the desilylation of trimethylsilylmethyl salts $\langle 92\text{JOC}5419 \rangle$. The rearrangement reaction shown in Scheme 60 is typical. A number of transformations of furfurylidene derivatives have been reported $\langle 83\text{CCC}459, 89\text{SC}3175 \rangle$ including the example shown in Scheme 61 which was used as the basis of a synthesis pallexensin A $\langle 87\text{SC}1655 \rangle$. The thermal electrocyclic step followed by a 1,5-sigmatropic shift of the hydrogen was shown to proceed more easily if the trimethylsilyl group was removed before the thermolysis was carried out. The trimethylsilyl group was used to prevent hydrogenation of the furan ring during the reduction of the acetylenic residue. Anionic [2,3]-sigmatropic rearrangement reactions of benzofurylmethyl allyl ethers have also been reported $\langle 84\text{ZOR}842 \rangle$.



Scheme 59



Scheme 60

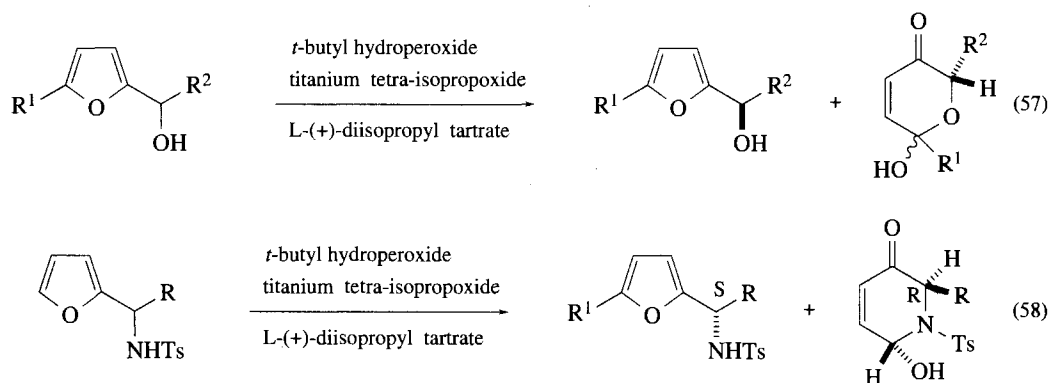


Scheme 61

The reaction of 2-methyl-3-furoic acid with two equivalents of *n*-butyllithium generates a dianion of which the 2-methylene anion is the more reactive and was found to give good yields of 2-substituted 3-furoic acids by interaction with a range of electrophiles $\langle 92\text{JCS}(\text{P}1)2729 \rangle$.

The nucleophilic ring opening reactions of furyl-2-oxirane by lithium aluminum hydride and sodium borohydride give markedly different results. The former reagent gave a 2:1 mixture of the secondary:primary alcohols while the latter reagent gave a 20:1 mixture of the primary:secondary alcohol $\langle 90\text{H}(31)1997 \rangle$. The kinetics of the uncatalyzed methanolysis of furyl-2-oxirane have been

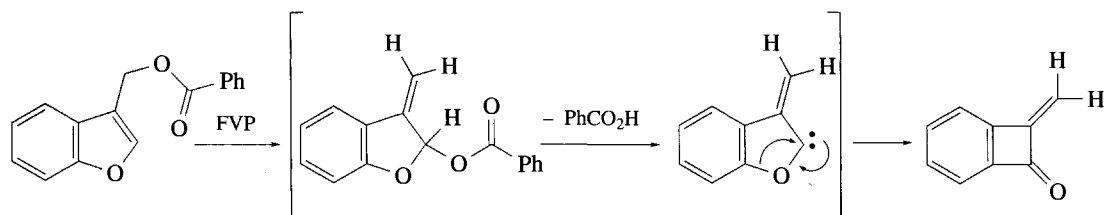
studied in order to gain information on the mechanism of ring opening reactions <94T5555>. The reduction of β -furyl- α,β -unsaturated ketones using magnesium in methanol <91TL4183> and the addition of diethyl phosphite <85ZOB2010> and tris-(dimethylamino)phosphine <94CCC2109> to vinyl-furans that have terminal electron withdrawing groups have all been reported. Synthetic intermediates that are valuable for the synthesis of rethrolone and prostaglandin related compounds have been prepared from a series of 2-furylhydroxymethylphosphonates <87CC907>. Chiral 2-furylcarbinols have been obtained by the enzyme catalyzed hydrolysis of a number of different esters of phenylacetic acid using penicillin acylase <89TL3057>. The kinetic resolution of furylcarbinols has been achieved by stereoselective oxidation using the Sharpless reagent system <88H(27)2107, 88JOC1586, 90JCS(P1)639> as indicated in the generalized Equation (57), and in similar reactions a series of secondary 2-furylamines have been resolved as shown in Equation (58) <93T2641>.



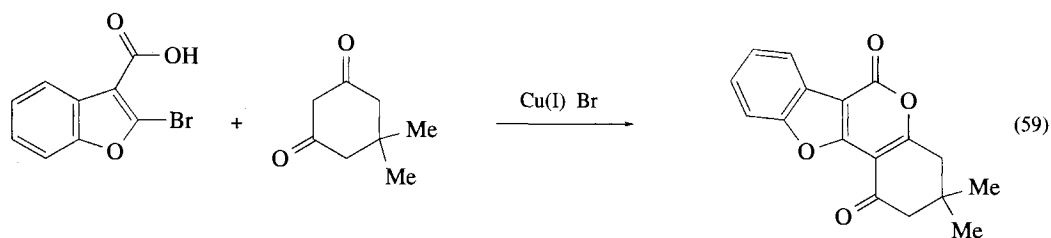
R = alkyl, allyl, or ninyl

2.06.3.2 Carboxylic Acids and their Reactions

The oxidation of 2-formylfuran to furan-2-carboxylic acid in yields of up to 92% has been reported using permanganate generated *in situ* by the anodic oxidation of manganese dioxide in sodium hydroxide using a cell with no diaphragm <93MI 206-03>. Derivatives of furylacetic acid have been prepared by the interaction of β -ketoesters such as methyl cyclohexanone-2-carboxylate with the cation that can be generated from 2,5-dihydro-2,5-dimethoxyfuran using zinc chloride <89T4471>. The related furyl derivatives of β -diketones can be made in an analogous way from β -diketones <88JOC5540>. 2,3,5-Tris(trifluoromethyl)furan has been prepared by first oxidizing ethyl 5-acetoxymethyl-2-trifluoromethylfuran-3-carboxylate to 2-trifluoromethylfuran-3,5-dicarboxylic acid with nitric acid and the dicarboxylic acid was converted into the final product by treatment with sulfur tetrafluoride in hydrogen fluoride <84ZOR846>. A number of furopyridine-2-carboxylic acids have been made via the related 2-lithio derivatives and conversion into the 2-formyl-furopyridines and other derivatives <87JHC373>. The interaction of alkyl lithium reagents with the lithium salt of furan-3-carboxylic acid has been shown to result in ketone formation, for example, 4-benzyloxybutyllithium gave 4-benzyloxy-1-(furan-3'-yl)pentan-1-one <84AJC619>. In a reaction that is effectively the reverse of the previous example, 2-furyllithium was allowed to interact with the lithium salts of carboxylic acids and provided an alternative ready access to 2-furyl ketones <95JOC8>. A number of benzofurancarboxylic acids have been shown to form dianions on reaction with lithium diisopropylamide which then react with electrophiles at carbon <82TL4127>. Benzofuran-2,3-dicarboxylic acid is decarboxylated thermally to benzofuran-3-carboxylic acid <84JA2696>. Reduction to 3-hydroxymethylbenzofuran and conversion into the benzoate ester gave a suitable precursor for flash vacuum pyrolysis which gave 2-methylenebenzocyclobutenone as shown in Scheme 62. Decarboxylation of a dicarboxylic acid was used in a route to dicyanodifurylmethane that used the same procedure that was found to be valuable for the preparation of 1,9-dicyanodipyrromethane from the related dicarboxylic acid <94SL939>. The reaction of dimedone with 2-bromobenzofuran-3-carboxylic acid in the presence of copper(I) bromide followed by lactone formation has been developed as a new route to a modified coumestane ring system <93SC2973>, outlined in Equation (59).



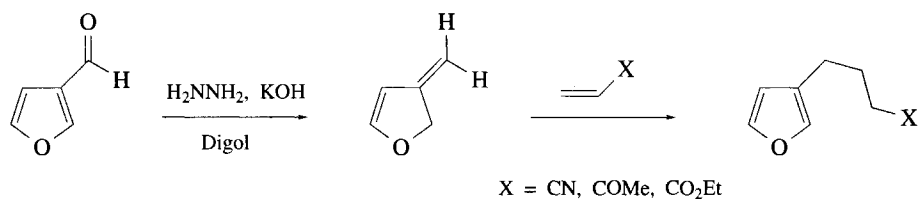
Scheme 62



5-Methylfuran-2-acetic acid has been made by a route that involved a degradation of diethyl 5-methyl-2-furylmalonate $\langle 84\text{MI } 206-04 \rangle$ and furylacrylates and acrylic acids have been prepared in moderate yields by the palladium catalyzed Heck reactions of ethyl acrylate with bromofurans $\langle 94\text{H}(38)759 \rangle$. Furyl and benzofuryl acrylates have also been made by coupling methyl acrylate with the appropriate acid chlorides using palladium acetate at 130°C in the presence of *N*-ethylmorpholine $\langle 89\text{JHC}597 \rangle$. Other alkenes such as acrylonitrile, styrene, and methyl vinyl ketone were also subjected to the decarbonylative coupling reactions. Bromofuran-2-carboxylic acids have been debrominated using Raney copper–aluminum alloy in sodium deuterioxide dissolved in deuterium oxide $\langle 88\text{JCR}(\text{S})136 \rangle$.

2.06.3.3 Acyl Substituents and their Reactions

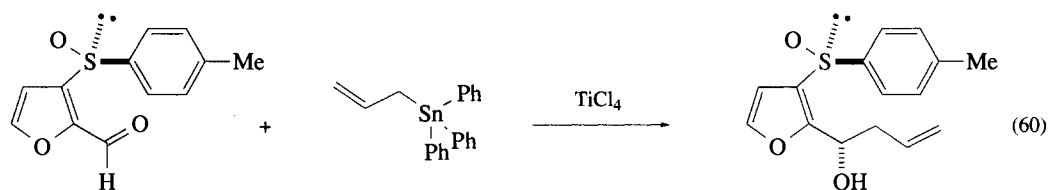
A new palladium on carbon–potassium carbonate catalyst is activated by irradiation at 254 nm and has been used to decarbonylate 3-formylfuran to give pure furan and carbon monoxide $\langle 83\text{CL}1403 \rangle$. The best conditions for the conversion of 2-formylfuran into the epoxide using the sulfur ylide method have been established $\langle 87\text{BSF}1073 \rangle$ and by catalytic isomerization to the 2-furylacetaldehyde $\langle 91\text{SC}1735 \rangle$. The confusion in the literature concerning the reactions of 2-formylfuran with aniline have been addressed and it has been established that the different products arise as a result of different molar ratios of reactants $\langle 90\text{AJC}655 \rangle$. The hydrolysis of 5-nitro-2-formylfuran diacetate proceeds easily when conducted in the presence of a clay catalyst and microwave irradiation, while a clayzic catalyst is useful for the condensation of active methylene compounds with 5-nitro-2-formylfuran $\langle 94\text{JCR}(\text{S})146 \rangle$. Wittig–Horner reactions using 2,5-diformylfuran have been carried out to produce pyranilidene- and thiopyranilidene-furans as potential precursors of organic metals $\langle 94\text{CL}255 \rangle$. The Huang–Minlon reduction of 3-formylfuran very surprisingly gave 3-methylene-2,3-dihydrofuran: the product was shown to undergo ene reactions with a number of electron depleted alkenes and provides a route to functionalize the 3-position in furan as shown in Scheme 63 $\langle 93\text{TL}5221 \rangle$. 3-Formylfuran has also been used as a precursor in a 5,6-cyclohexannelated benzofuran synthesis $\langle 90\text{H}(31)2219 \rangle$.



Scheme 63

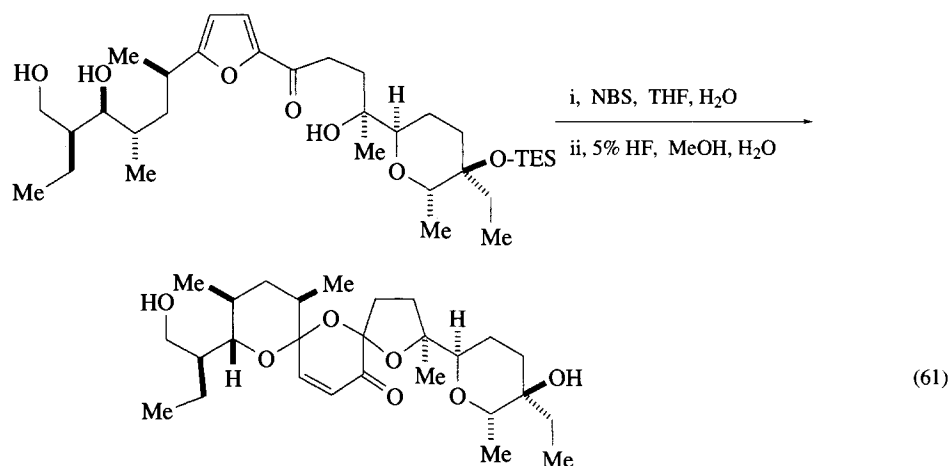
It is interesting to note that intramolecular control in the diastereoselective addition of a number of organometallic reagents to furancarboxaldehydes has been studied $\langle 94\text{TA}1203 \rangle$. The use of an

adjacent chiral sulfoxide residue to generate the diastereoselectivity then allowed the optically pure nonracemic alcohols to be isolated after removal of the auxiliary. The addition of allyltriphenylstannane to the optically pure 3-*p*-tolylsulfinylfuran-2-carboxaldehyde shown in Equation (60) in the presence of titanium tetrachloride gave the alcohol shown with high diastereoselectivity, whereas a similar reaction carried out in the presence of tin(IV) chloride resulted in the exclusive formation of the other diastereomer <94H(38)1751>.



Coupling reactions of organomanganese reagents with 2- and 3-furoyl chloride in the presence of copper or iron catalysts have been used to prepare a number of naturally occurring ketones in high yields <92TL5245> and aryltrialkylstannanes have been shown to arylate 2-furoyl chloride in the presence of a palladium catalyst <92CC1440>.

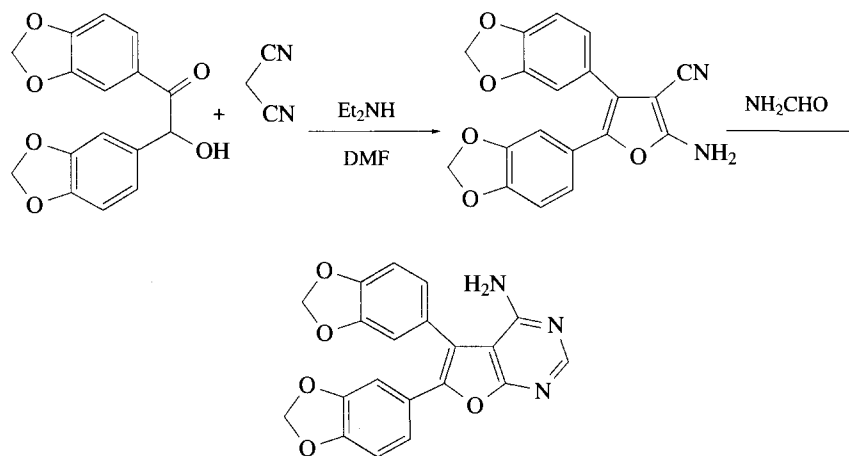
The condensation of 5-nitro-2-acetylfuran with a number of aromatic aldehydes has been used to prepare a range of 5-nitro-2-furylpropenones some of which were screened and showed significant activity against specific bacteria <86MI 206-03>. The preparation of furacyl halides presents an interesting problem because of the ease of nuclear halogenation. Two methods have been developed for the trimethylsilylation of 2-acetylfuran using either lithium hexamethyldisilazide <88SC727> or chlorotrimethylsilane and a base and DMF <91SC11>. The trimethylsilylenol ethers were then used in reactions with bromine or copper(II) chloride or *N*-chlorosuccinimide. Isomaltol (2-acetyl-3-hydroxyfuran) a nonenzymatic browning product gives an orange-red dimer, *E*-2-[1-(3-hydroxy-2-furanyl)ethylidene-(2*H*)-furan-3-one], in reasonable yield when treated with toluene-*p*-sulfonic acid <86CAR(146)107>. A key step in a synthesis of the polyether antibiotic Salinomycin was the oxidative rearrangement of a 2-acylfuran shown in Equation (61) to the core unit a 1,6,8-trioxadispiro-[4.1.5.3]pentadec-13-ene derivative <94SL417>.



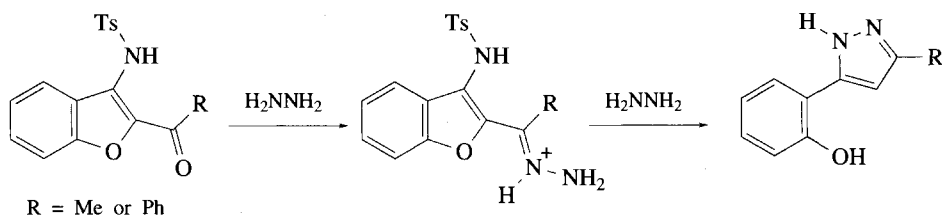
2.06.3.4 Heteroatom-linked Substituents and their Reactions

The instability of simple aminofurans was pointed out in CHEC-I <84CHEC-I(4)599>. 2-Substituted 5-nitrofurans have been reduced to the 5-aminofurans by an electrochemical method <91TL631>. Work showed that although catalytic reduction gives 2-aminofurans in low yields they can be trapped <82JOC2483> using ethyl ethoxymethylenecyanoacetate or ethoxymethylenemalononitrile <93JHC113>. The 2-aminofuran behaves as a dieneamine and substitution occurs at the 2-position. Quaternary ammonium salts, can be prepared, for example, from 2-bromo-5-nitrofuran and trimethylamine <83CCC1885>. The salts undergo nucleophilic displacement reactions with azide and cyanide ions in aqueous solutions. It is of interest to note that 2-amino-3-cyano-4,5-bis(methyl-

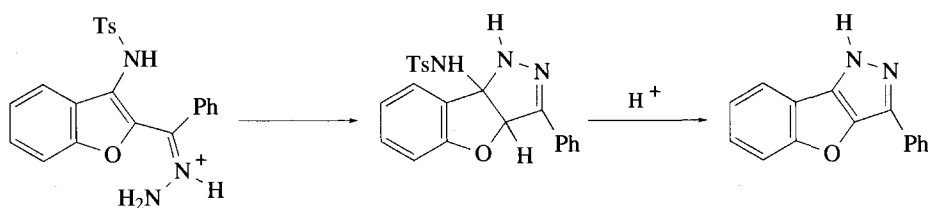
enedioxybenzyl)furan was prepared by the route shown in Scheme 64 <84CCC1788> and was converted into the furopyrimidine by the reaction with formamide. 2-Acetyl- and 2-benzoyl-3-amino-benzofurans failed to form hydrazones with hydrazine and its *N*-methyl and *N,N*-dimethyl derivatives. The related 2-acetyl- and 2-benzoyl-3-*N*-*p*-toluenesulfonylaminobenzofurans did give the hydrazones easily <84JHC937>. The hydrazones undergo further reactions as shown in Scheme 65 to give, for example, 5-(2-hydroxyphenyl)pyrazoles as a result of furan ring cleavage. Alternatively, hydrazone cyclization can result in the formation of benzofuopyrazoles as shown in Scheme 66.



Scheme 64



Scheme 65



Scheme 66

3-Ethoxycarbonylamino-2-benzofurancarbonyl azide undergoes a Curtius rearrangement in the presence of alcohols to give carbamates <84IJC(B)132> and 2-trimethylsilyloxyfuran is desilylated with methyllithium and then gives carbonates after reactions with alkyl chloroformates <87SCI035>.

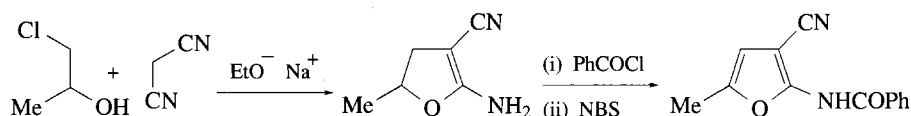
2.06.3.5 Saturated and Partially Saturated Rings

2.06.3.5.1 2,3-Dihydrofurans and dihydrobenzofurans

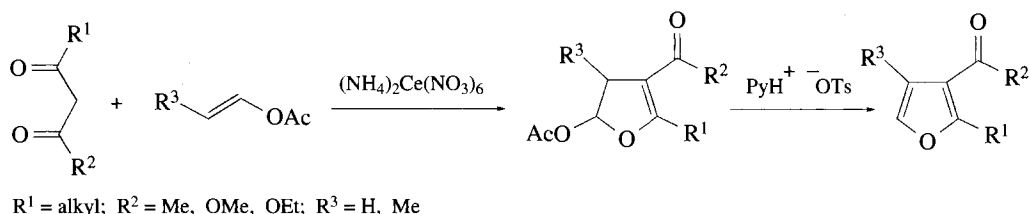
The conversion of dihydrofurans and their benzologues into the aromatic compounds has been the subject of a large number of investigations. For example, the treatment of benzo[c]furanone

with lithium diisopropylamide and chlorotrimethylsilane provides a convenient way of generating 1-trimethylsilyl-3-trimethylsilyloxyisobenzofuran in a one pot procedure <92TL2769>. 3-Halo-benzo[b]furan formation by elimination of hydrogen halide from a large number of 2,3-dihalo-2,3-dihydrobenzofurans has been the subject of an extensive mechanistic investigation <83JA6114> and the oxidative deselenation of 2-phenylselenomethyl derivatives of both 2,3-dihydrofuran and its benzologue give the aromatized heterocycles <93JOC1349>. The rates of the thermal racemization of 2,3-cyclopropabenzofuran have been reported <93TA479>. 2-Amino-4,5-dihydrofuran-3,4-dicarbonitriles are thermally converted into the 2-aminofuran-3-carbonitriles by the loss of hydrogen cyanide <84JCS(P1)2009>. The reverse process by which furans are reduced to dihydro- and tetrahydrofurans has been studied using lithium in ammonia <84AJC1303>.

Malononitrile reacts with 2-chloroethanol, or 1-chloro-2-propanol or 2-methyloxirane, in the presence of sodium ethoxide to give 2-amino-3-cyano-4,5-dihydrofuran, or its 5-methyl homologue, and the benzamido derivatives were converted into the corresponding furans by oxidation using *N*-bromosuccinimide as exemplified in Scheme 67 <85MI 206-01>. The 2-amino-3-cyano-4,5-dihydrofuran derivatives have also been converted into a number of other ring systems, for example, pyrrolidinones and cyclopropanes <89H(29)5> and, in the case of the 5-aryl derivatives, cyclopropanes <90CHE828>. In addition, ring fragmentation reactions have been reported from the interaction of sodium trichloroacetate with 2-*N,N*-dialkylamino-3-cyano-4,5-dihydrofuran derivatives in dimethoxyethane <92LA1109>. The oxidative addition of enol esters to β -diketones generates dihydrofurans which are aromatized with pyridinium tosylate, as shown in the general outline in Scheme 68, to give 2,3,4-trisubstituted furans <88SC1841>. The cyclization of allyl derivatives of β -diketones using iodine provides a route to 2,3-dihydrofurans (Scheme 69) which can then be dehydroiodinated and isomerized to 2,4-disubstituted furans <88TL4987>. Base catalyzed isomerization of an exocyclic double bond to give an aromatized derivative has also been reported <90H(31)1417>.



Scheme 67

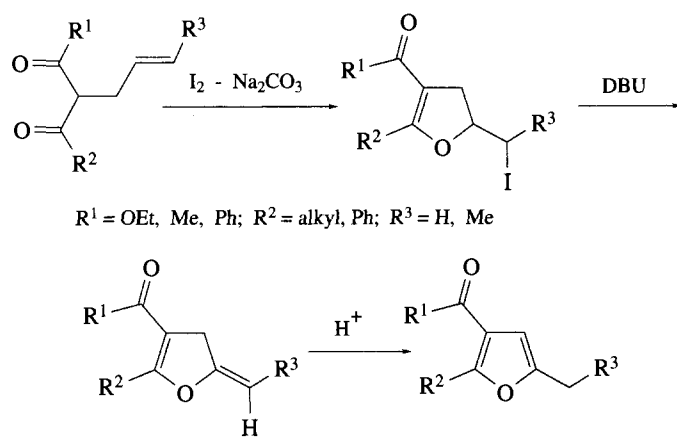


R¹ = alkyl; R² = Me, OMe, OEt; R³ = H, Me

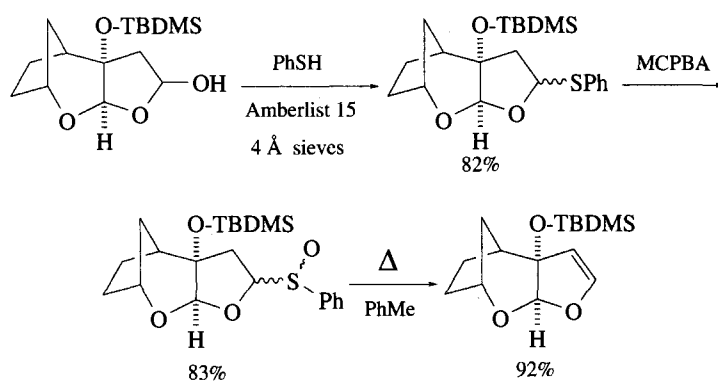
Scheme 68

The 2,3-dihydrofuran sub-unit is present in a number of natural products such as clerodin and azadirachtin and as a consequence some effort has been expended in the search for efficient ways of constructing highly functionalized 2,3-dihydrofurans. The conversion of the lactol into the 2,3-dihydrofuran, shown in Scheme 70, represents a part of the azadirachtin molecule <87TL221>. Whereas the sulfoxide shown in Scheme 70 was isolable, the selenoxide, formed by the oxidation of the monoseleno acetal that was obtained from the lactol and illustrated in Scheme 71, gave the dihydrofuran directly <88TL2179>.

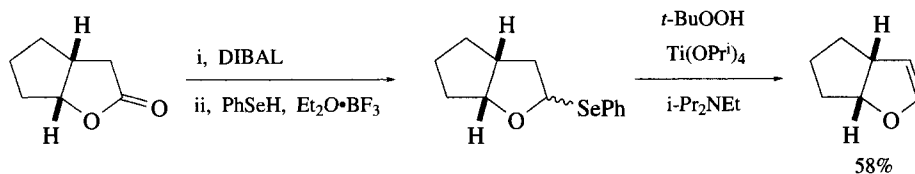
The use of a palladium catalyst in the addition of secondary amines such as morpholine across the enol ether double bond of 2,3-dihydrofuran provides a route to tetrahydrofuran based aminol ethers <92CC1552>. Stille type coupling reactions have been reported which allow the conversion of 2-tributylstannyl-4,5-dihydrofuran into 2-aryl and 2-alkenyl substituted derivatives <90TL6077> and Heck type reactions have also been carried out on 2,3-dihydrofuran using aryl iodides (Scheme 72) <90JOC407> and aryl triflates <91JA1417, 92TL1485>. Reactions with a number of cycloalkenyltriflates have also been carried out and high enantioselectivity was reported in the presence of a palladium BINAP-*N,N,N',N'*-tetramethyl-1,8-diaminonaphthalene catalyst system, exemplified in Equation (62) <93TL2505>.



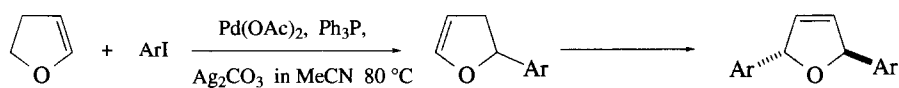
Scheme 69



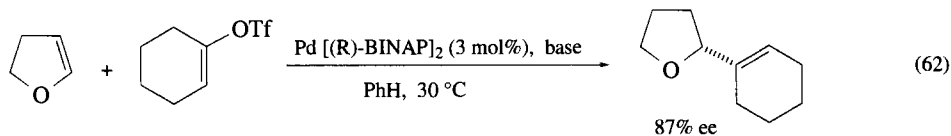
Scheme 70



Scheme 71



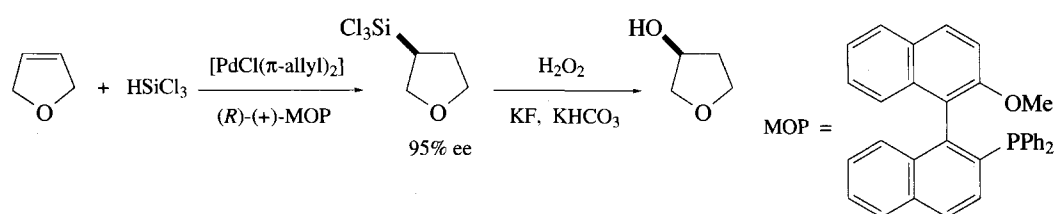
Scheme 72



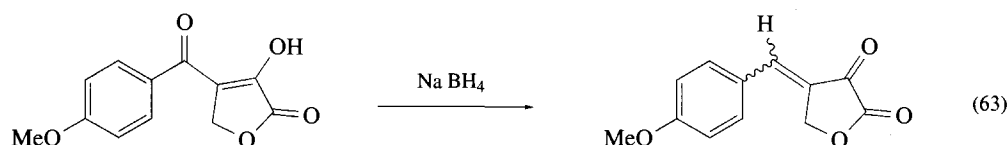
The epoxidation of enol ethers is well known to be difficult to control but 2,3-dihydrofuran has been successfully epoxidized by using dimethyldioxirane $\langle 88JA7929 \rangle$. 2,3-Dihydrofuran reacts with singlet oxygen to give a dioxetane and 2,5-dihydrofuran-2-hydroperoxide $\langle 91JOC4017 \rangle$. The latter compound decomposes thermally to furan and hydrogen peroxide while the dioxetane ring opens to 2-formylethyl formate. 2,3-Dihydrofuran is metallated by *n*-butyllithium to give 4,5-dihydro-2-furyllithium. Ring opening reactions have also been reported from the products of the reactions of 4,5-dihydro-2-furyllithium with Grignard reagents $\langle 91CC1595 \rangle$. Treatment of 4,5-dihydro-2-furyllithium with an alkyl halide and then with a Grignard reagent and bis(triphenylphosphine)nickel dichloride resulted in the overall conversion of 2,3-dihydrofuran into a number of derivatives of 4,4-disubstituted but-3-ene-1-ol $\langle 92JCS(P1)3419 \rangle$. A number of Paterno-Büchi reactions have been reported involving, for example, benzophenone and 4-methylene- γ -butyrolactone $\langle 89LAC869 \rangle$. In reactions conducted under argon the normal reaction was observed but in the presence of oxygen 1,3,4-trioxan derivatives were formed.

2.06.3.5.2 Addition reactions of 2,5-dihydrofurans and butenolides

Reactions of 2,5-dihydrofurans have also been widely investigated. The hydrosilylation of 2,5-dihydrofuran with trichlorosilane in the presence of the bis(π -allyl)palladium chloride complex and (R)-(+)-MOP proceeds with high enantioselectivity and provides a route to the optically pure nonracemic 3-hydroxytetrahydrofuran as shown in Scheme 73 $\langle 93TL2335 \rangle$. 3-Trimethylsilyl-2,5-dihydrofuran reacts with dichlorocarbene, generated in a two phase catalytic system to give both of the possible products of insertion into the C—H bonds at the 2- and 5-positions as well as the cycloadduct $\langle 84JOM(265)237 \rangle$. Stille coupling reactions of 3-tributylstannyl- and 4-tributylstannyl-2(5*H*)-furanones have been reported $\langle 92TL7049 \rangle$. A number of highly substituted 2(5*H*)-furanones have been converted into furans either by reduction using di-isobutylaluminium hydride followed by dehydration or by dehydration following a reaction with a Grignard reagent $\langle 88KGS313 \rangle$. The reduction of a number of arylated 2(5*H*)-furanones have been reported using the borane dimethylsulfide complex $\langle 88S68 \rangle$ and a variety of 4-aryl-3-hydroxy-2(5*H*)-furanones has been investigated using different reducing agents $\langle 86JHC199 \rangle$. Sodium borohydride, for example, reduces the aryl carbonyl group which then dehydrates as shown in Equation (63). The *trans*-addition of mercury(II) nitrate across the double bond of 2,5-dihydrofuran evidently proceeds by way of the intermediacy of a mercurinium intermediate $\langle 88JHC1035 \rangle$. Treatment with sodium hydroxide gave the nitroalkene which with ammonia gave the *trans*-addition product and then after catalytic reduction *trans*-3,4-diaminotetrahydrofuran.

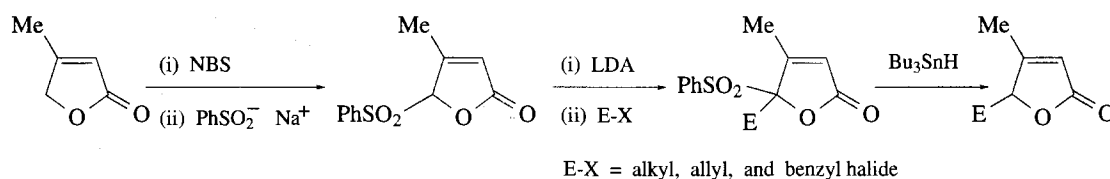


Scheme 73

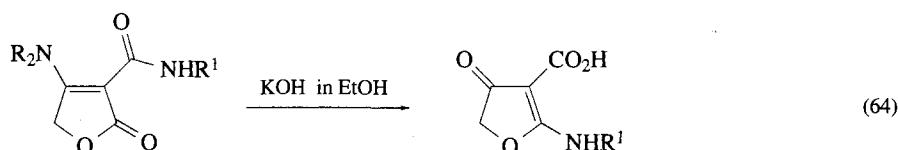


The functionalization of the 5-position in 4-methyl-2(5*H*)-furanone has been achieved by using the phenylsulfone group as a temporary adjunct to activate the 5-position as shown in Scheme 74 $\langle 89CL1391 \rangle$ and also in a similar manner by means of the thioethyl residue $\langle 88JOC3330 \rangle$. The 5-bromo-5-alkyl-2(5*H*)-furanones can be hydrolyzed to the corresponding 5-hydroxy-5-alkyl-2(5*H*)-furanones and the dehydrated to the 5-alkenyl-5-alkyl-2(5*H*)-furanones $\langle 90TL5517 \rangle$. The vinylogous

ureas (4-dialkylamino-3-alkylaminocarbonyl-2(5*H*)-furanones) undergo the interesting rearrangement on hydrolysis that is shown in Equation (64) <88HCA783>.



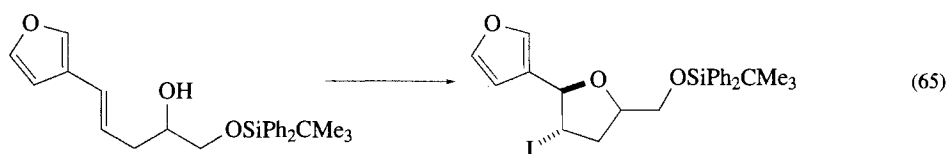
Scheme 74



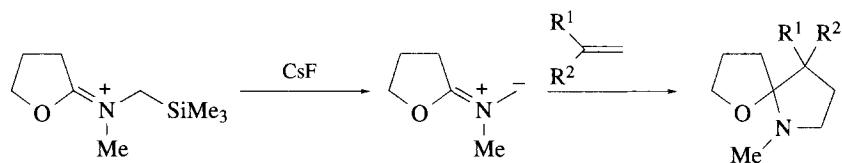
The Diels–Alder cycloaddition reactions of 5-alkyl-2(5*H*)-furanones with cyclopentadiene form the starting point for a protocol that allows the alkylation at the 3-position <88JOC4393>. The adducts are deprotonated using lithium diisopropylamide, the anions alkylated with an alkyl halide, and finally the products are heated in order to carry out the retro-Diels–Alder reaction that gives the 5-alkyl-2(5*H*)-furanones together with cyclopentadiene. Similarly, Diels–Alder reactions between furan and 2(5*H*)-furanones have been elaborated to give chiral 5-methoxycarbonylmethyl-2(5*H*)-furanones, the (*S*)-enantiomer of which is a naturally occurring marine product <87TL5819>. Asymmetric 1,3-dipolar cycloaddition reactions have been carried out using 5-menthyloxy-2(5*H*)-furanones <88TL5317>. A series of reactions with 2,5-dihydrofuran and cyanates has given cycloadducts that can be hydrogenated in a two step process to the 3-alkyl-4-hydroxytetrahydrofurans and hence by using vinyl acetate and a lipase to chiral 3-alkyl 4-hydroxytetrahydrofurans and their acetate esters <93JOC2173>. Enzymatic hydrolyzes of 3-alkyl-4-acetoxytetrahydrofurans provide an alternative source of chiral 3-alkyl 4-hydroxytetrahydrofurans <90JCS(P1)2359> and monoesters have been obtained in high optical purity by the enzymatic hydrolysis from *meso*-2,5-dialkyl-2,5-bis(hydroxymethyl)tetrahydrofuran diesters <90TL445>. Finally we may note that L-ascorbic acid and its D-enantiomer have been converted into the enantiomeric 4-hydroxytetrahydrofuran-2-ones <87S570>.

2.06.3.5.3 Tetrahydrofurans

A number of interesting approaches to the synthesis of tetrahydrofurans have appeared since CHEC-I <84CHEC-I(4)599>. The stereocontrolled iodoetherification shown in Equation (65) also provides ready access to the corresponding dihydrofuran <93TL7579>. γ -Bromo-primary amides cyclize to protonated imine salts using silver fluoroborate <89TL6571> and in the case of the analogous secondary amides an iminium salt is formed. In the example shown in Scheme 75 the iminium salt was desilylated to an ylide and hence by reactions with alkenes to spiro-pyrrolidino-tetrahydrofurans <89TL6573>. 4-Hydroxybut-1-ene derivatives are cyclized using a palladium–copper(II) catalyst in the presence of ethyl orthoacetate in methanol to give 2-methoxy-4,5-tetrahydrofuran derivatives <89CL737>.



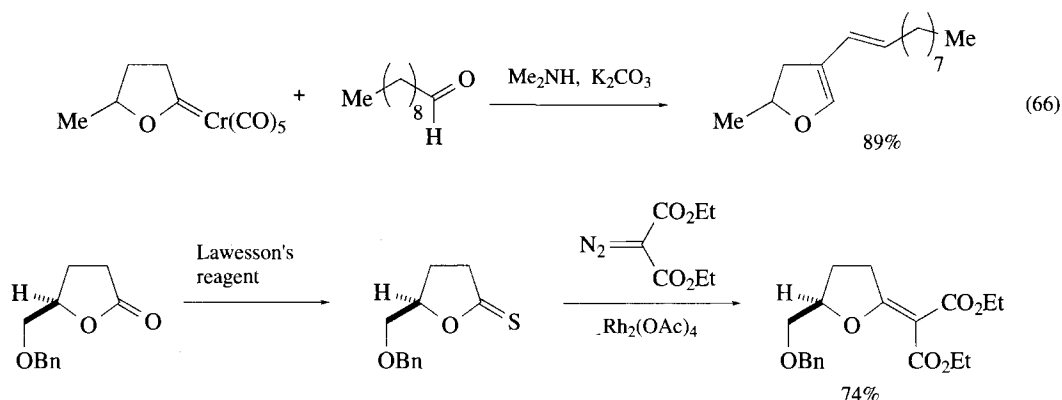
The deprotonation of tetrahydrofuran-2-carboxylic acid with two equivalents of lithium diisopropylamide allows additional functionality to be introduced at the 2-position <92TL6783>. In the case of the reactions of the dianion with ketones, the 2- β -hydroxytetrahydrofuran-2-carboxylic acids that are formed can be converted into the exocyclic methylene derivatives by a process that



Scheme 75

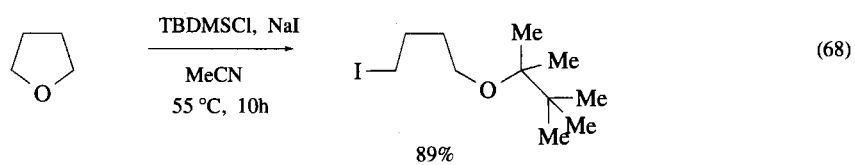
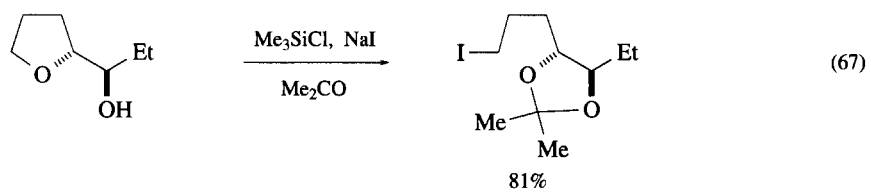
involves the loss of carbon dioxide and water. The phenylsulfonyl group has been shown to be displaced from the 2-position in tetrahydrofuran by the organometallic reagent formed by the interaction of a Grignard reagent with zinc chloride (90SL749).

The increasing use in synthesis of pentacarbonyl(alkoxycarbene)chromium complexes is reflected in their use in connection with the preparation of conjugated dienes. The conversion of a tetrahydrofuran carbene complex by displacement of the carbene ligand and conversion into a conjugated diene by reactions with aliphatic aldehydes that have α -hydrogen atoms is illustrated in Equation (66) (91CC437). The reverse of that reaction, in which a rhodium carbene complex is reacted with a chiral tetrahydrofuran-2-thione, provides a route to 2-acylmethylene tetrahydrofurans in good yields (87S1116), as exemplified in Scheme 76. The yields obtained were much better than those achieved using earlier procedures, for example, from lactone acetals and active methylene compounds.



Scheme 76

The thermal ring opening reactions of the isomeric 2,3-4,5-bis(cyclopropa)furans to 2,3-dihydrooxepine have been studied (93CB485). The *syn*-isomer undergoes the isomerization reaction at 130°C whereas the *anti*-isomer only undergoes the ring opening at 370°C . The use of silicon reagents in ring opening reactions of tetrahydrofuran (82SC253) and its derivatives has been reported. Tetrahydrofuran is converted by bromotrimethylsilane into 4-bromotrimethylsilyloxybutane in 82% yield in a reaction conducted in the neat reagent after being heated under reflux for 40 h (81S383). Similarly, 2-acetoxymethyltetrahydrofuran gave 5-bromo-2-trimethylsilyloxypropyl acetate in 87% yield. The reaction time is significantly reduced in reactions using chlorotrimethylsilane and sodium iodide in acetonitrile and in addition good regioselectivity was observed when cleaving 2-substituted tetrahydrofurans: 2-methyltetrahydrofuran gave predominantly the primary iodide (87BSF505). In the reaction shown in Equation (67) which was carried out using acetone as the solvent the acetal was isolated in good yield. In a development of that method chloro-*t*-butyldimethylsilane has been used in place of chlorotrimethylsilane, as shown in Equation (68), and the TBDMS group then survives subsequent reactions with a wide range of nucleophiles (88S56). Highly regioselective ring opening reactions of tetrahydrofuran derivatives have been studied using chlorotrimethylsilane and sodium iodide (87BSF505). 2-Methyltetrahydrofuran is opened predominantly to give the primary iodide, 5-iodopentan-2-ol but, not surprisingly, the reaction involving 3-methyltetrahydrofuran is less selective. Lithium 4,4'-di-*t*-butylbiphenylide has also been used to cause the ring opening of tetrahydrofuran at -80°C in the presence of boron trifluoride etherate (91JA1866).



2.07

Furans and their Benzo Derivatives: Synthesis

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Universität Kiel, Germany

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2.07.1 INTRODUCTION

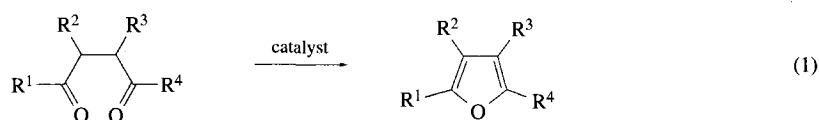
The chemistry of furans and benzo[*b*]furans has been a field of active research for a long period. There are several reasons for this activity. A number of furan derivatives have achieved considerable technical importance (e.g., furfural), and it has been found that the furan ring – both in its native as well as in its reduced form – occurs in a number of natural products. These compounds can exhibit remarkable pharmaceutical activity. The chemistry of furans and benzofurans has been reviewed several times in considerable detail <B-50MI 207-01, B-51MI 207-01, B-51MI 207-02, B-53MI 270-01, 66AHC(7)377, B-74MI 207-01, 79COC(4)713, 80AHC(30)135, 82AHC(30)167, 84CHEC-I(4)657>. Other aspects of this field are available <86CRV795, 88T2093, 89PHC(1)126, B-89MI 207-01, 90PHC(2)87, 91PHC(3)109, 92PHC(4)95, 93PHC(5)129, 93MI 207-02, 94PHC(6)129, 95PHC(7)130>. A general treatment of the synthesis of five-membered heterocycles has appeared <84CHEC-I(4)89>.

2.07.2 FURANS

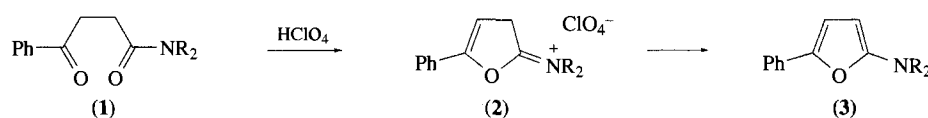
2.07.2.1 Ring Synthesis from Acyclic Compounds

2.07.2.1.1 Cyclization of 1,4-diketones and related compounds

The preparation of furans by acid-catalyzed ring closure of 1,4-diketones (Paal–Knorr method) (Equation (1)) <1884CB2756, 1885CB299, 1885CB367> is of general applicability <69RCR389>, but may fail with sterically hindered diones <50JA5754>.

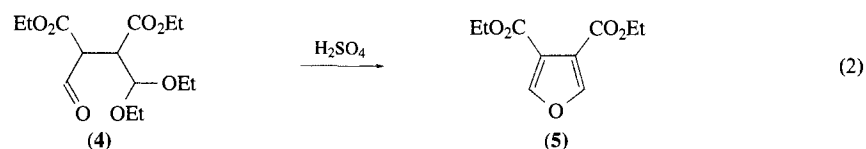


As acidic catalysts sulfuric acid <89SC2101, 91JHC225>, hydrochloric acid, polyphosphoric acid <84S593>, *p*-toluenesulfonic acid, oxalic acid <52CB457>, Amberlyst 15 <73S209>, acetic anhydride/zinc(II)bromide, and others are used. Yields are generally good to high. If β -aroylpropionamides (1) are used as starting material, after treatment of the iminium salt (2) with triethylamine the corresponding 2-aminofurans (3) are obtained (Scheme 1) <73JCS(P1)2523>.

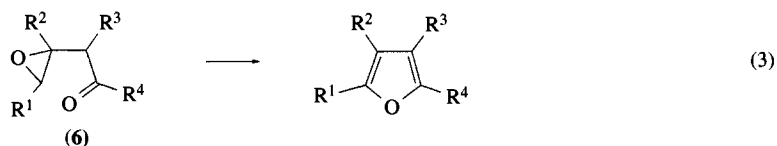


Scheme 1

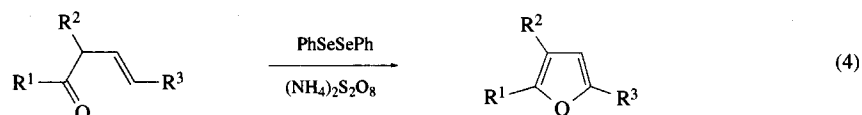
2-Aminofurans are also obtained by cyclization of δ -ketonitriles <90MI 207-01>. The 1,4-diketone may also be used in protected form, e.g., as ketal. 3,4-Diethoxycarbonylfuran (5) is obtained from the acetal (4) with sulfuric acid in 68% yield (Equation (2)) <54JOC1671>.



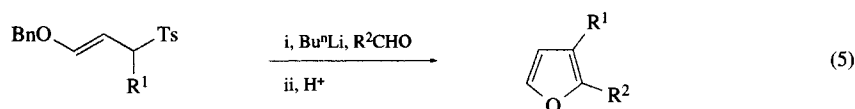
Further examples have been described <73JOC2361, 86JOC2351>. Oxiranes of type (6) behave as masked 1,4-dicarbonyl compounds. The cyclization to furans (Equation (3)) can be accomplished either by acids <77S838, 81SC365, 83H(20)593, 83S997, 84TL1061, 88SC677> or bases <71JOC3011, 84TL4383>, or simply by heating without catalysts <63OSC(4)649>.



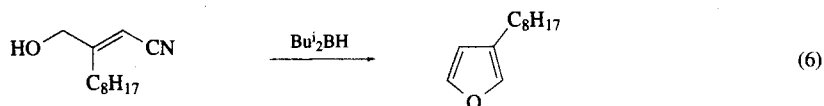
α -Substituted β,γ -unsaturated ketones can be converted to 2,3,5-trisubstituted furans with diphenyldiselenide– $(\text{NH}_4)_2\text{S}_2\text{O}_8$ (Equation (4)) <94SL373>.



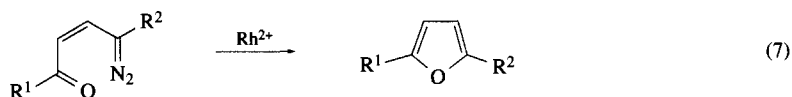
1-Benzyloxy-3-tosylalkenes can be used as enal β -anion equivalents. After deprotonation with Bu^nLi , reaction with aldehydes, and work up under acidic conditions 2,3-disubstituted furans are obtained (Equation (5)) <93TL7487>.



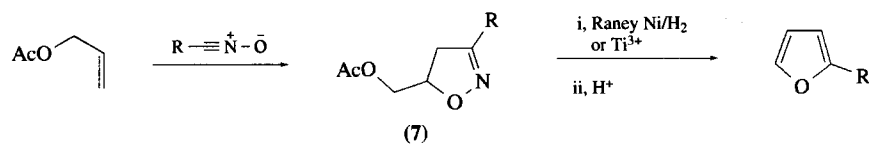
On reduction with bis(2-methylpropyl)borane, γ -hydroxyacrylonitriles yield the corresponding aldehydes, which cyclize spontaneously to give furans (Equation (6)) <81TL2187>.



4-Diazo-1-oxoalkenes with *Z*-configuration on treatment with rhodium catalysts yield furans (Equation (7)). This reaction can be envisaged as an electrocyclic reaction of the corresponding carbene <83JOC3813, 90TL6835> (see also Section 2.07.2.2.3). This route is also useful in the benzo[*b*]- and benzo[*c*]furan series (see Sections 2.07.3 and 2.07.4).

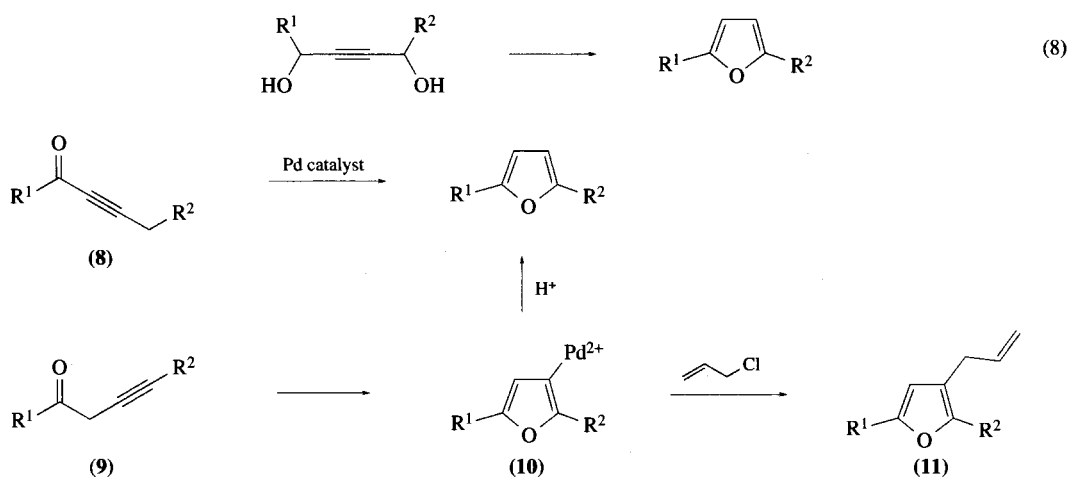


Dihydrooxazoles of type (7), which are in turn available by addition of nitrile oxides to allyl acetate, are reduced to β,γ -dihydroxyketones (or protected derivatives thereof). On treatment with acids, furans are obtained (Scheme 2) <82ACS(B)1, 83T2247, 87ACS(B)426, 88BCJ2133, 90H(31)597, 92H(33)73, 93JCR(M)328, 93JCR(S)58, 94H(38)641>.

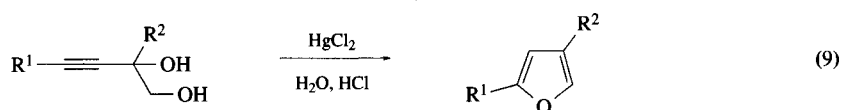


Scheme 2

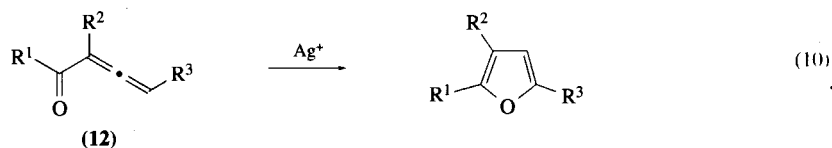
1,4-Dihydroxyalkynes may be considered as masked 1,4-dicarbonyl compounds (Equation (8)). This isomerization–dehydration reaction leading to furans can be accomplished by Pd catalysts <93CC764>. Alkynylketones of type (8) and (9) can be cyclized by Pd(0) and Pd(II) catalysts <86TL4893, 87S1022, 91JOC5816>. The intermediate (10) can either be hydrolyzed or trapped with electrophiles like allyl chloride to give 3-substituted alkenylfurans (11) (Scheme 3). In this way, 3-allyl-2-hexyl-5-methylfuran (11; $\text{R}^1 = \text{Me}$, $\text{R}^2 = \text{C}_6\text{H}_{13}$) could be obtained in 70% yield <91JOC5816>. 1,2-Dihydroxyalkynes on treatment with HgCl_2 yield furans (either directly or after treatment with acids) (Equation (9)) <55ZOB81, 77RZC249>.



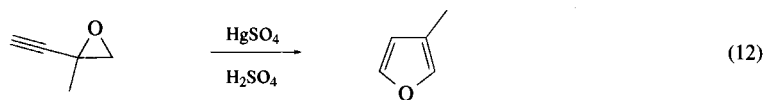
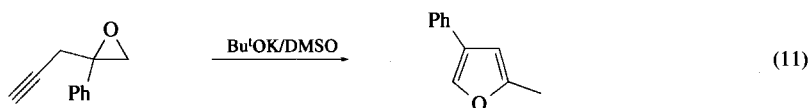
Scheme 3



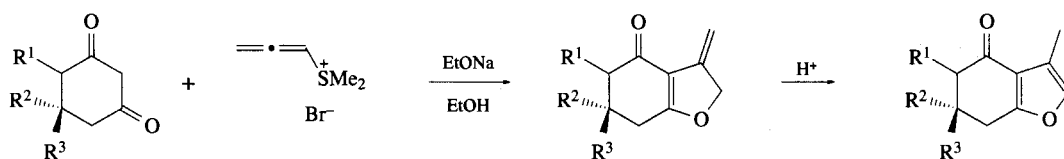
In quite a similar manner 4-ketoallenes (**12**) can be transformed by metal catalysts (Ag(I), Rh(I)) to furans; 2,3-disubstituted and 2,3,5-trisubstituted furans (Equation (10)) are available with good yields <82T1413, 90JOC3450, 91JOC960>, as are 2,5-bridged furans (furanophanes) <92JOC3387>.



Cyclization of the corresponding hydroxyallenes yields dihydrofurans <93JOC7180>. Furans with a similar substitution pattern can be obtained by cyclization of alkynyl- or alkadienyloxirans (Equations (11) and (12)) <69JCS(C)12, 72JOU2286, 72JOU2289, 72ZOR2237, 72ZOR2240, 76RTC75, 76TL2387, 83JCR(M)1625, 83JCR(S)167, 92JA1450>.



Fused 3-methylfurans are readily obtained by the reaction of an allenic sulfonium salt and the enolate anion of a cyclic dicarbonyl compound (Scheme 4) <93JOC3960>.

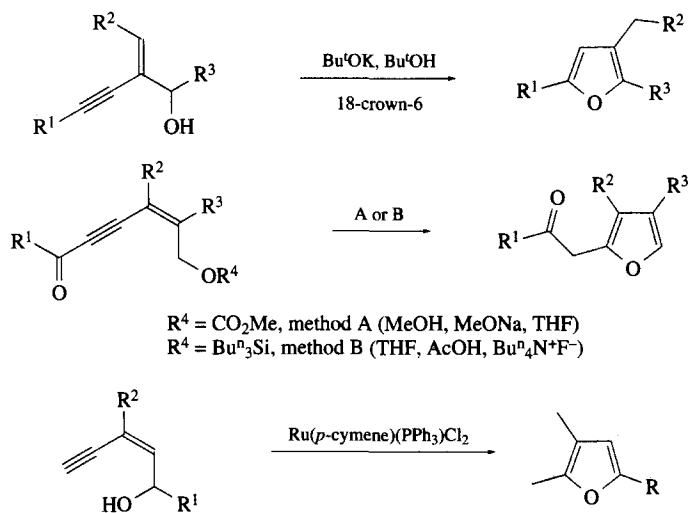


Scheme 4

It has been reported that the cyclization of dimedone with monosubstituted alkynes in the presence of Hg(OAc)₂ yields tetrahydrobenzo[c]furans <93ZOR1067>.

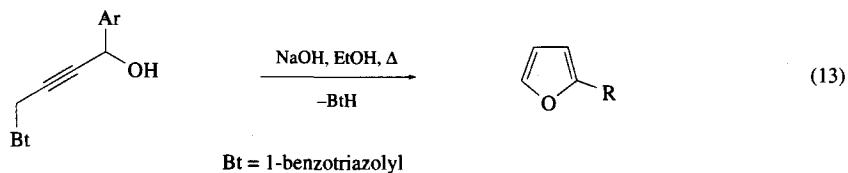
Hydroxyalkynoates can be cyclized to furans with base (Scheme 5) <93JOC3435, 93JOC3602>.

Furans containing a functional group at C-5 can also be obtained by cyclization of (*Z*)-pent-2-en-4-yn-1-ols under neutral conditions in the presence of Ru(*p*-cymene)(PPh₃)Cl₂ (Scheme 5) <94CC493>.

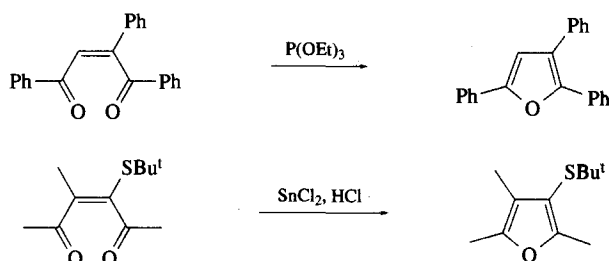


Scheme 5

Base-assisted cyclization of 1-[3-[hydroxy(substituted methyl)]propargyl]benzotriazoles, which are available from lithiated 1-propargylbenzotriazole and aromatic aldehydes, gives 2-arylfurans (Equation (13)) <93JOC3038>.

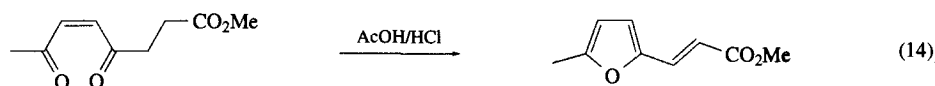


If 1,4-dioxoalkenes are used as starting materials, the cyclization to furans includes a reduction step (Scheme 6). This can be achieved by tin(II), diphosphorus tetraiodide (P₂I₄), or phosphites (P(OR)₃). Yields seem to be moderate to excellent <65JOC2284, 74TL4179, 79JOC494, 85JHC495>.

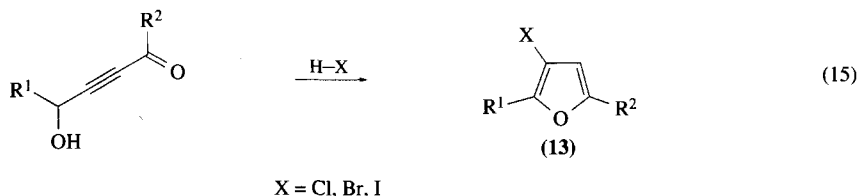


Scheme 6

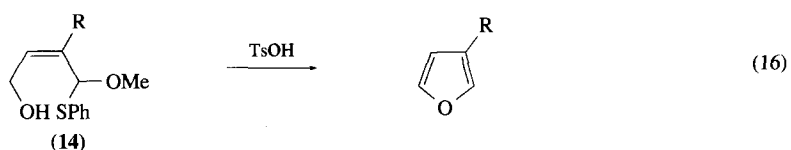
2-Alkenylfurans are accessible from suitably substituted 1,4-dioxoalkenes by treatment with acids (Equation (14)) <59CCC530>.



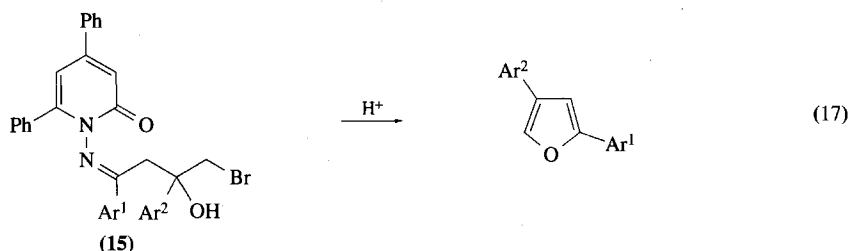
A versatile route to 3-halogenofurans starts with 4-hydroxy-1-oxoalkenes or their protected analogues. On treatment with $H-X$ ($X = Cl, Br, I$) furans of type (13) are obtained (Equation (15)) <89HCA447>; *cis*-alkenes are intermediates in this reaction.



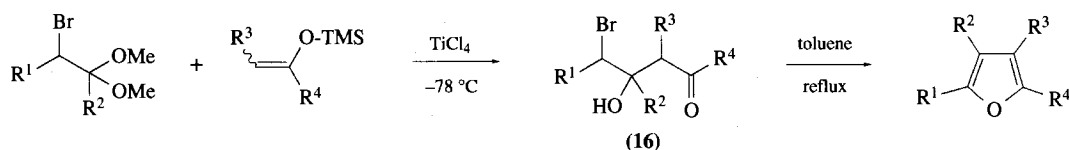
3-Substituted furans have been prepared by acid-catalyzed cyclization of protected 4-hydroxyalkenes of type (14), which are quite easily available from methoxy(phenylthio)methane; both (*Z*)- and (*E*)-isomers of (14) gave the furan (Equation (16)) <83CL1909>.



An efficient regioselective synthesis of 2,4-diarylfurans is accomplished by cyclization of 4-bromo-3-hydroxyketoimines of type (15) (Equation (17)), which were generated *in situ* by reaction of the corresponding pyridone with Bu^nLi -phenacylbromide <83S49>; yields are good to excellent.

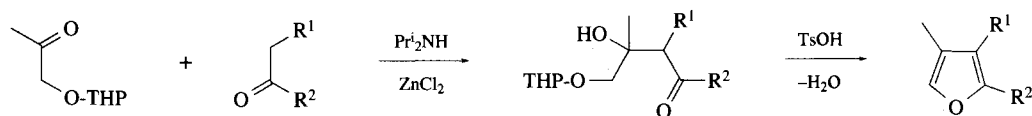


β -Alkoxy- γ -bromoketones (16), which in turn are available in good yields from a $TiCl_4$ -promoted reaction of α -bromoacetals with silyl enol ethers, can be cyclized to furans in refluxing toluene; yields are moderate to good (Scheme 7) <75CL527>.



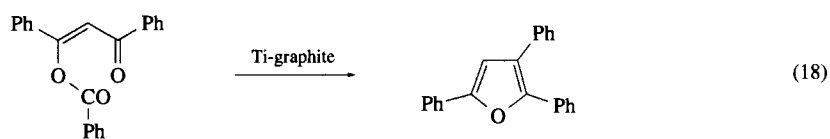
Scheme 7

A base-assisted condensation of carbonyl compounds with a protected α -hydroxyketone yields partially protected β,γ -dihydroxyketones, which can be cyclized to furans (Scheme 8) <84JCS(P1)91>.

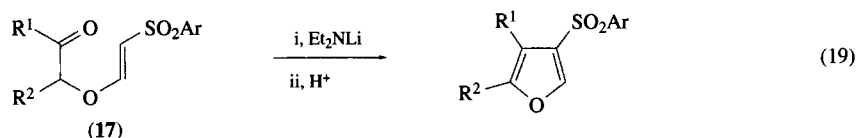


Scheme 8

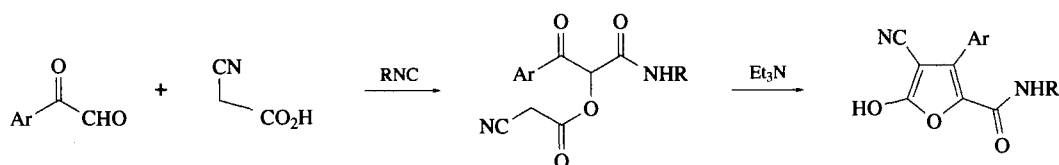
Furans can also be obtained by reductive cyclization of β -acyloxy- α,β -unsaturated ketones (Equation (18)) <92T5991>.



Treatment of arylsulfonyl ethenyl oxoethyl ethers of type (17) with base yields 3-arylsulfonylfurans (Equation (19)) <87TL4123>.

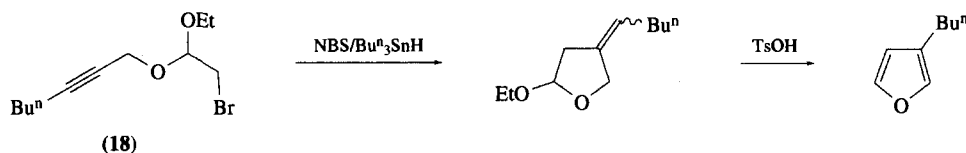


In a Passerini-type reaction, treatment of arylglyoxals with cyanoacetic acid and isocyanides yields 3-aryl-2-cyanoacetoxy-3-oxopropionamides. Cyclization of these compounds under basic conditions gives 2-hydroxyfurans (2-furanones), albeit in low yield (Scheme 9) <93S783>.



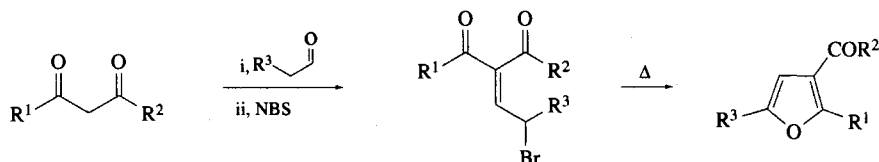
Scheme 9

The radical-initiated cyclization of alkynylacetals of type (18) yields alkylidenetetrahydrofurans, which on treatment with acids give furans (Scheme 10) <83JA3720, 90T7901>.



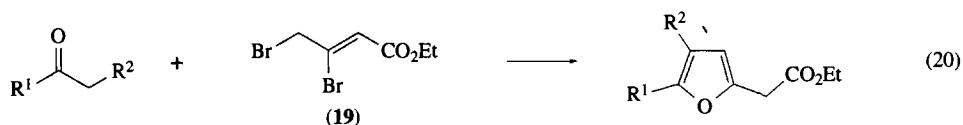
Scheme 10

Knoevenagel condensation of 1,3-dicarbonyl compounds with subsequent NBS bromination yields allyl bromides, which are converted thermally to 3-acyl- or 3-alkoxycarbonyl furans (Scheme 11) <78JOC4596>.



Scheme 11

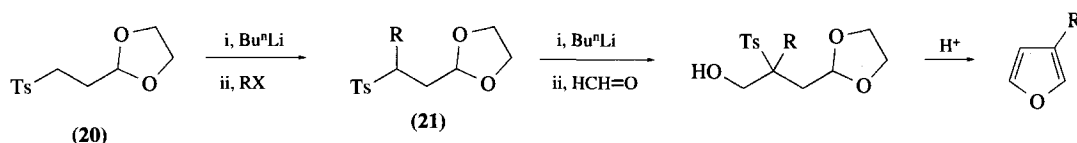
The reaction of a carbonyl compound with a doubly functionalized alkene (19) yields trisubstituted furans (Equation (20)) <80S52, 92JOC1161, 92JOC1170>.



2-Aminofurans are available from a similar route (Equation (21)) <73ZOB1809, 73ZOR1834, 78JOC3821>.

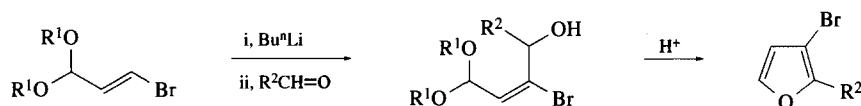


A protected arylsulfonylpropanal (e.g., (20)) can be used as starting material for 3-substituted furans. Introduction of an alkyl (aryl) group and hydroxymethylation yields a protected γ -hydroxypropanal (21), which after treatment with acids gives the corresponding furans (Scheme 12) <78BCJ930, 78H(10)105, 79CL709, 79H(12)1467>.



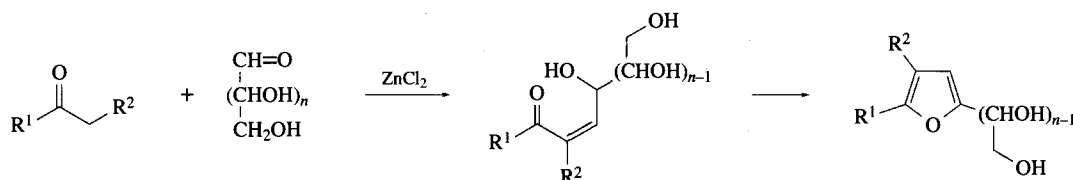
Scheme 12

3-Halogenofurans with a substituent in position 2 are available by a similar route. A protected β -halogenoacrolein is transformed to a γ -hydroxypropenal *O,O*-acetal, which after treatment with acids yields a halogenofuran (Scheme 13) <85JOC4872>. Phenylthiofurans can be prepared quite analogously <86TL139>.



Scheme 13

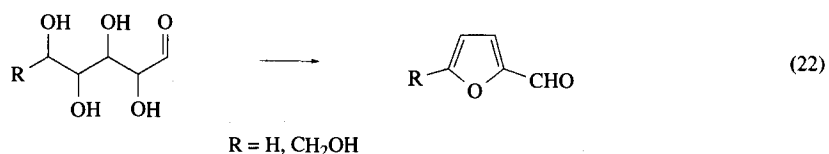
When carbonyl compounds with an active methylene group are condensed with sugars (pentoses, hexoses), furans with a polyhydroxylated side chain are obtained; possibly γ -hydroxy- α,β -unsaturated ketones are intermediates (Scheme 14) <82BSF(2)176>.



Scheme 14

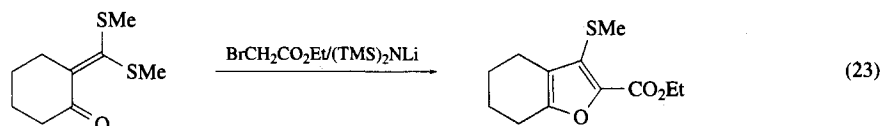
2.07.2.1.2 Transformation of carbohydrate precursors

The formation of furfural (furan-2-carbaldehyde) from pentoses and pentosanes has long been known and is of considerable technical importance <B-89MI 207-01>. The mechanism of this reaction has been studied in some detail <72JOC1606, 86H(24)1373>. Hexoses (e.g., fructose) are starting materials for the preparation of 5-hydroxymethylfuran-2-carbaldehyde (Equation (22)) <82CL617, 83CAR(122)59, 87BSF855, 88CI(L)228, B-91MI 207-01, B-91MI 207-02, B-92MI 207-01, 94SC939>. In the presence of hydrochloric acid (or other chlorinating agents), 5-chloromethylfuran-2-carbaldehyde is obtained <82CL617, 92S541>.

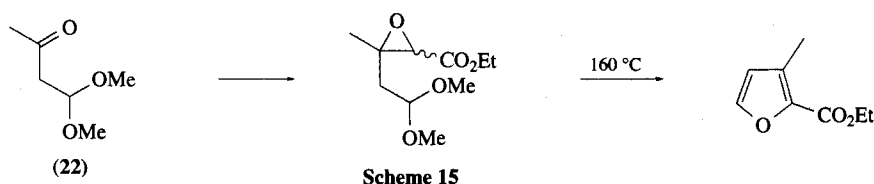
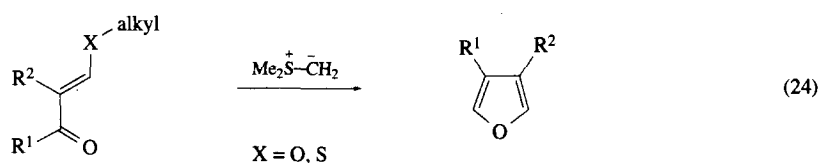


2.07.2.1.3 From α,β -unsaturated ketones and related compounds (4 + 1)

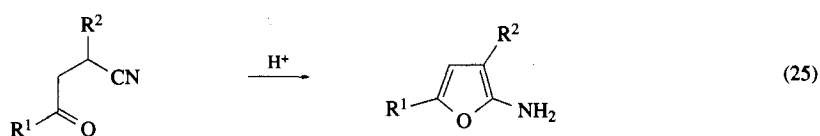
β,β -Bis(methylthio)- α,β -unsaturated ketones react with ethyl bromoacetate in the presence of base to give 3-methylthiofurans (Equation (23)) <89T7631>; alkyl oxirans are intermediates.



In a similar sequence, α,β -unsaturated ketones with a donor group in the β -position react with sulfonium ylides <73JA250, 84JOC3819, 89JOC2777>. This method is especially suited for the preparation of 3,4-disubstituted furans (Equation (24)). The Darzens reaction of a partially protected 1,3-dicarbonyl compound (e.g., (22)) yields an oxiran, which can be cyclized to a furan either thermally or under basic conditions (Scheme 15) <56JOC102, 63OSC(4)649, 86SC431>.

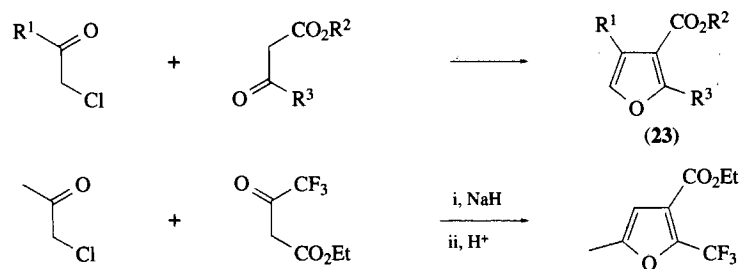


A cyanide anion may serve as an one-carbon equivalent. β -Halogenoketones react with potassium cyanide to give a β -ketonitrile; these compounds cyclize under acid conditions to give 2-aminofurans (Equation (25)) <82S513, 83H(20)803>.



2.07.2.1.4 From ketones and related compounds (3 + 2)

The base-catalyzed reaction of β -ketoesters with α -halogenoketones (Feist-Benary reaction) <02CB1545, 11CB493> yields 3-alkoxycarbonylfurans (23) (Scheme 16). Usually the reaction starts with an aldol condensation; if in the first step an alkylation of the β -carbonyl compound takes place, the reversed regiochemistry is observed <68JHC95>.

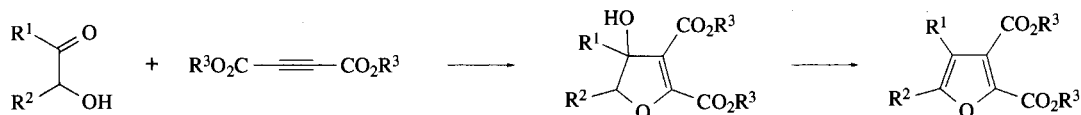


Scheme 16

3-Alkoxy-carbonylfurans are also available by the reaction of β -ketoesters with acyloins in the presence of zinc(II) chloride (Equation (26)) <65JCS5984, 80CB699, 85SC1191>.

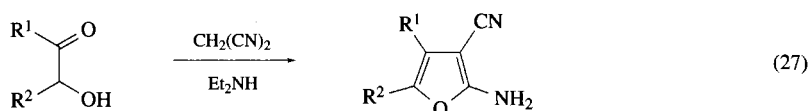


Acyloins may react with dialkyl ethynedicarboxylates to give 3-hydroxy-2,3-dihydrofurans, which after elimination of water yield furans (Scheme 17) <77JHC745, 91TL4687>.

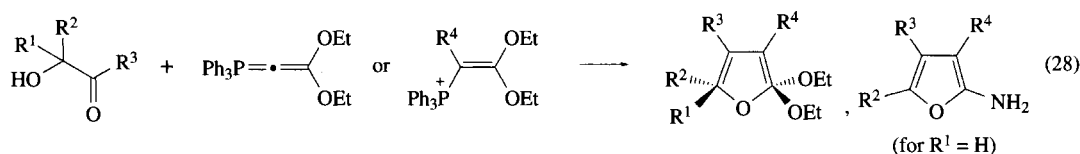


Scheme 17

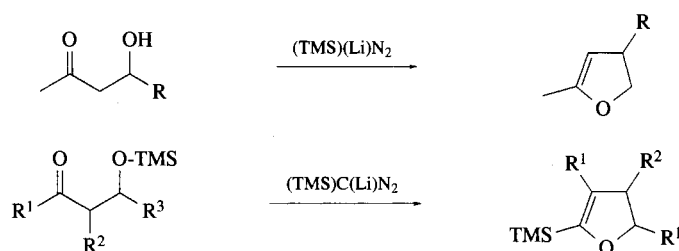
2-Aminofuran-3-nitriles are available by base-catalyzed condensation of acyloins with malononitrile (Equation (27)) <66CB1002, 80CCCC1581, 83CCC3140>.



Depending on substituents the reaction of α -hydroxyketones with (2,2-diethoxyvinylidene)-triphenylphosphorane or (2,2-diethoxyvinyl)triphenylphosphonium tetrafluoroborate yields 2,2-diethoxy-2,5-dihydrofurans or 2-ethoxyfurans (Equation (28)) <94ZN(B)389>.



Treatment of β -hydroxyketones or β -trimethylsilyloxyketones with trimethylsilyldiazomethyl-lithium yields 2,3-dihydrofurans, which can be dehydrogenated to furans with manganese dioxide (Scheme 18) <94SL461>. Obviously a carbene is an intermediate in the first step of the reaction.

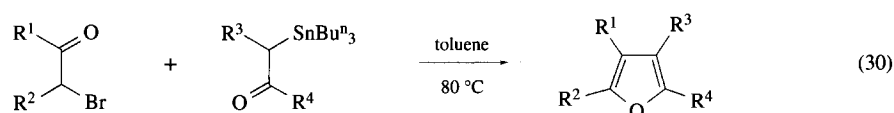


Scheme 18

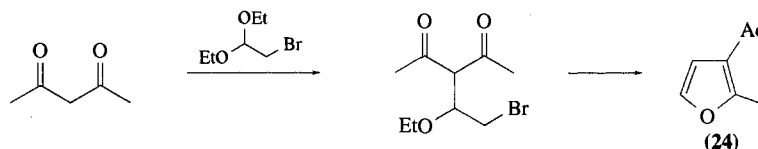
The reaction of β -ketoesters with 1-acetoxy-1,2-dibromoethane yields 2-substituted 3-alkoxy-carbonylfurans (Equation (29)) <90SC1923>.



Mono- through tetrasubstituted furans can be prepared by the reaction of α -bromoketones with α -stannylated carbonyl compounds (Equation (30)) <83CC989>.

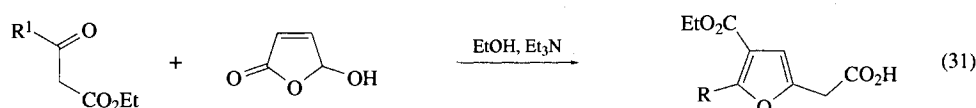


Treatment of β -dicarbonyl compounds with 1,1-dialkoxy-2-bromoethane in the presence of trimethylsilyl trifluoromethanesulfonate with subsequent heating of the resulting intermediates (toluene, reflux) yields 2-substituted 3-acylfurans (**24**) (Scheme 19) <88G73>.

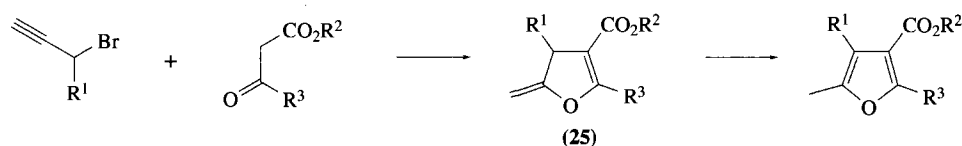


Scheme 19

Heating of β -ketoesters with 5-hydroxy-5H-furan-2-one in the presence of Et_3N gives 3-alkoxy-carbonylfurans (Equation (31)) <88OPP173>.

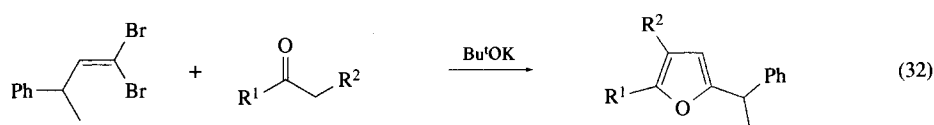


Treatment of the sodium salts of β -ketoesters with 3-halogenoalkynes in the presence of Cu(II) yields alkylidenefurans (**25**), which isomerize under acid conditions to furans (Scheme (20)) <78S581>.

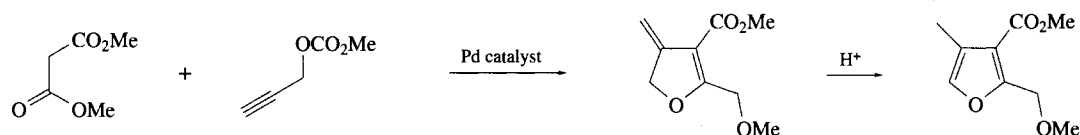


Scheme 20

Di- and trisubstituted furans can be obtained from 1,1-dibromo-3-phenyl-1-butene and various aldehydes and ketones (Equation (32)). The reaction proceeds via dehydrobromination, $\text{S}_{\text{N}}2'$ -substitution, and a tandem Claisen rearrangement-cyclization <93SL905>.



A Pd-catalyzed annulation of methyl 4-methoxyacetoacetate with methyl 2-propynyl carbonate yields a dihydrofuran, which can be converted directly to a furan (Scheme 21) <93S1109>.

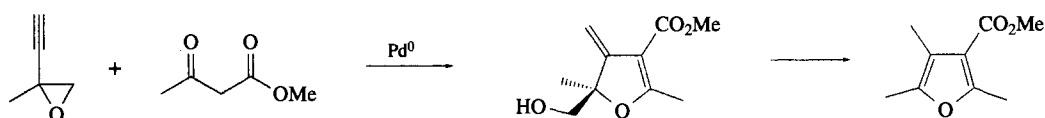


Scheme 21

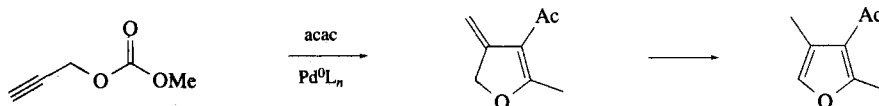
Alkynyloxirans can be coupled with β -ketoesters using Pd(0) catalysts; under mild conditions the resulting alkylidenefurans lose formaldehyde to give furans (Scheme 22) <87JOM(334)225>.

The Pd(0) -assisted coupling of 1,3-dicarbonyl compounds can also be accomplished with alkynyl esters; the 3-alkylidene-2,3-dihydrofurans are isomerized to furans (Scheme 23) <87JOM(334)225>.

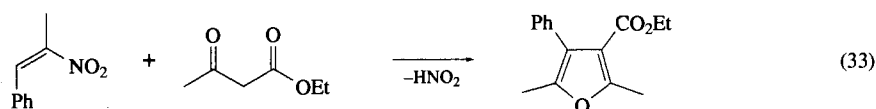
Michael addition of 1,3-dicarbonyl compounds to nitroalkenes with subsequent cyclization and loss of HNO_2 yields furans (Equation (33)). 4,5-Annulated furans are also available by this route <57CB1215, 59LA(626)71, 78JCS(P1)1144, 80JOC2945, 83IJC(B)914, 83SI027>.



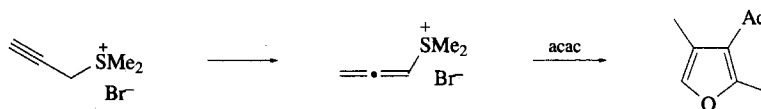
Scheme 22



Scheme 23

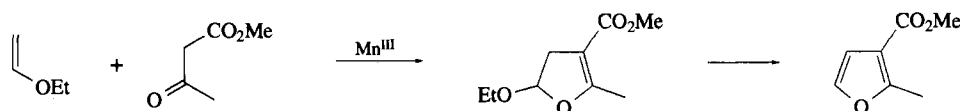


Alkynyl-substituted sulfonium salts react with carbonyl compounds in the presence of base to give 2,3,4-trisubstituted furans. The first step in this reaction is an alkynyl-allenyl isomerization (Scheme 24) <73JCS(P1)65, 90H(31)1003, 93JOC3960>.



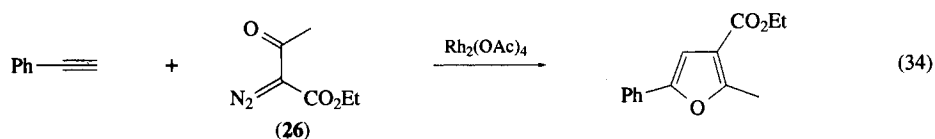
Scheme 24

Both 3-alkoxycarbonyl- and 3-acylfurans can be prepared by oxidative coupling of enol ethers with 1,3-dicarbonyl compounds in the presence of Mn(III) and subsequent treatment of the resulting 2,3-dihydrofurans with acids (Scheme 25) <87CL223, 88SC1841, 93S833>.

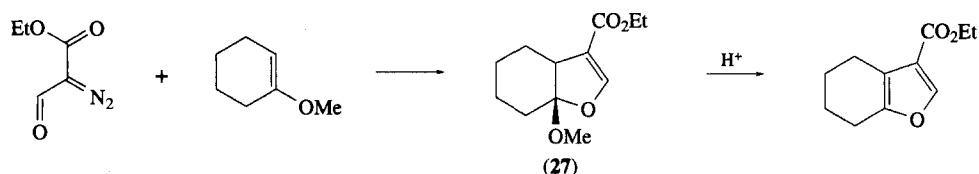


Scheme 25

The decomposition of α -diazocarbonyl compounds of type (26) with transition metal catalysts (e.g., $\text{Rh}_2(\text{OAc})_4$) in the presence of monosubstituted alkynes can lead to furans (Equation (34)) <88JOC2699, 88T3343>.

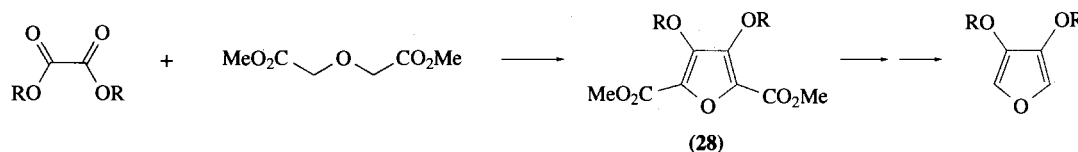


As expected, the decomposition of α -diazocarbonyl compounds in the presence of enol ethers yields 2,3-dihydro-2-alkoxyfurans (e.g., (27)), which can be transformed to the corresponding furans (Scheme 26) <78HCA430, 90JOC4975>. The $\text{Rh}_2(\text{OAc})_4$ -catalyzed reaction did not succeed in this case <90JOC4975>.



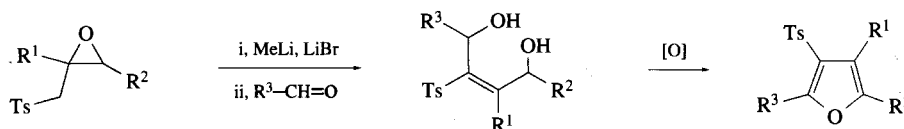
Scheme 26

2,5-Dimethoxycarbonyl-3,4-dialkoxyfurans (**28**) can be prepared by the condensation of oxalic esters with bis(alkoxycarbonylmethyl)ethers (Scheme 27). Hydrolysis of the ester function with subsequent decarboxylation (Cu(II) catalyst, heat) yields 3,4-dialkoxyfurans <91ACR22>.



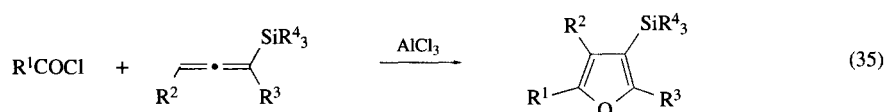
Scheme 27

The oxidation of 1,4-dihydroxybut-2-enes (pyridinium chlorochromate; MnO_2/H^+), which are in turn available from arylsulfonylmethyloxirans, yields furans (Scheme 28) <89JOC1491>.

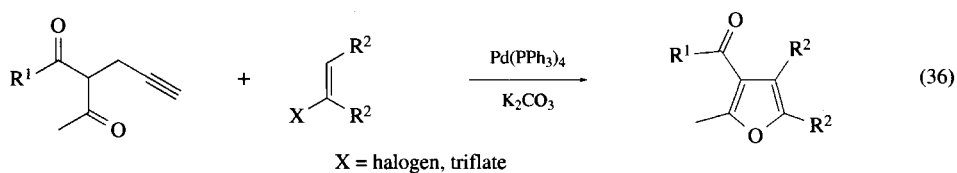


Scheme 28

A regioselective [3 + 2] annulation strategy has been reported for a wide variety of furans. Reaction of acid chlorides with trialkylsilyl-substituted allenes under the influence of AlCl_3 (CH_2Cl_2 , -20°C) yields the desired compounds (Equation (35)). The intramolecular variant of this method has also been described <89JA4407>. 2,3,5-Trialkyl-2,5-dihydrofurans are available in a one-step synthesis from alkenes and aldehydes using $\text{CF}_3\text{CO}_2\text{H}$ as the acidic reagent (Prins reaction) <93ZOR748>.

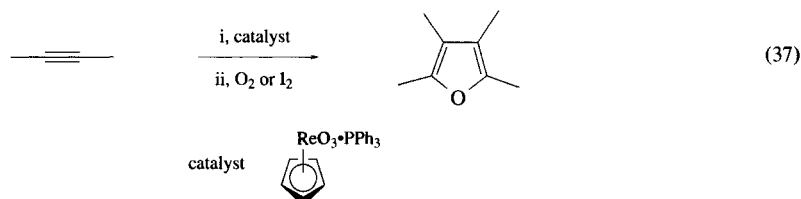


A Pd-catalyzed annulation of 2-propargyl-1,3-dicarbonyl compounds with vinylic or aryl triflates or halides in the presence of potassium carbonate yields 2,3-disubstituted 3-acylfurans (Equation (36)) <93TL2813>.



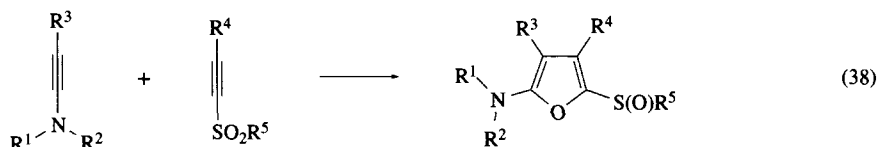
2.07.2.1.5 From miscellaneous precursors

Tetramethylfuran is available in nearly quantitative yield by cyclocondensation of but-2-yne with a transition metal catalyst in the presence of oxygen or iodine (Equation (37)) <74JCS(D)169, 86JA8271>.

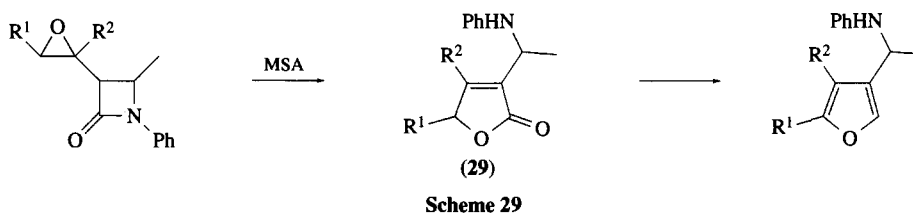


Butadiene and other alkadienes can be oxidized to furans using copper catalysts ($\text{CuCl}_2/\text{CuCl}$) <81USP4268421>, silver molybdate/ Al_2O_3 <81USP4293444>, or $\text{PdCl}_2/\text{In}(\text{OH})_3$ in the presence of AIBN

<82JAP(K)81113774>. A versatile synthesis of 2-amino-5-sulfinylfurans consists of the reaction of *N*-(1-alkynyl)amines with sulfonylalkynes (Equation (38)) <87CB71, 88CB2163>. Obviously a Pummerer-type rearrangement is involved.



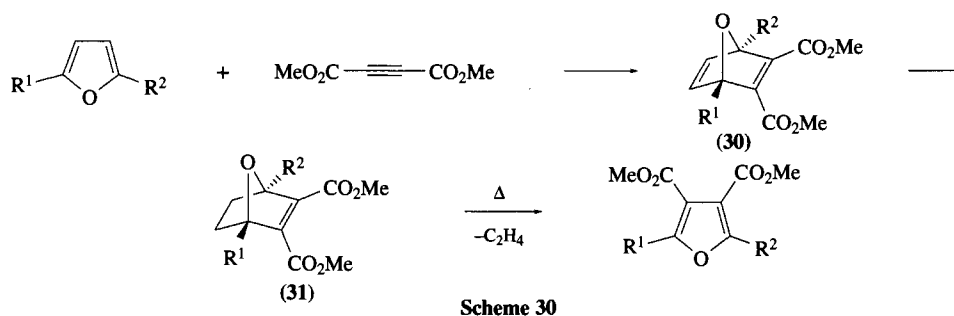
The acid-catalyzed ring opening–ring closure reaction of an azetidinone yields a dihydrofuranone (29), which can be converted to a furan using either a reduction–dehydration step or reaction with phenyllithium and subsequent dehydration (Scheme 29) <82JCS(P1)257>.



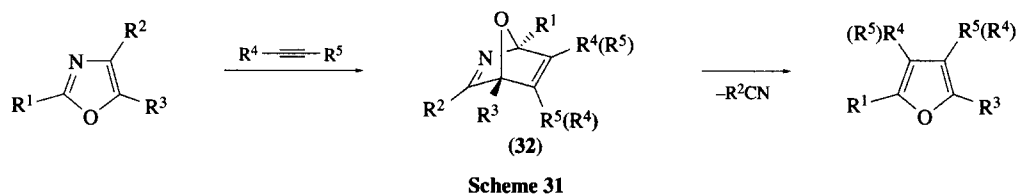
2.07.2.2 Ring Transformations

2.07.2.2.1 From five-membered heterocycles

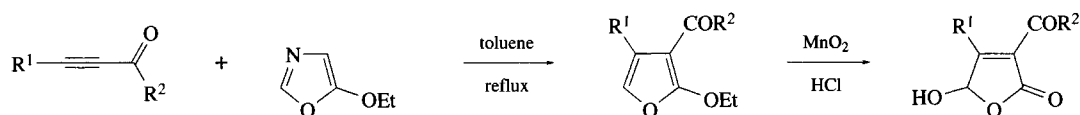
Furans react with electron-deficient alkynes giving 7-oxabicyclo[2.2.1]heptadienes (30). Whereas it is possible to induce thermally a retro cycloaddition <62JOC3520, 89CC1238>, normally these intermediates are reduced at the less hindered side, yielding 7-oxabicyclo[2.2.1]heptenes (31) (Scheme 30). Elimination of ethylene (Alder–Rickert reaction <37CB1354> yields substituted furans (Scheme 30) <68T4501, 85JHC1233, 87BSF131>.



Cycloadducts of 7-oxabicyclo[2.2.1]heptadienes with nitrile oxides or tetrazines may undergo a cycloreversion giving furans and isoxazoles (pyridazinones) <72CC211, 74TL2163>. Oxazoles react with alkynes to give 2-aza-7-oxabicyclo[2.2.1]heptadienes (32), which lose a nitrile spontaneously by a cycloreversion reaction yielding furans (Scheme 31).

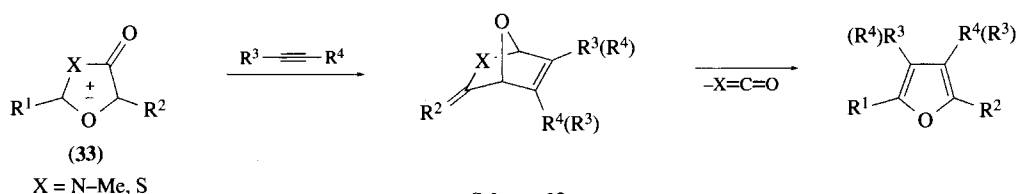


The oxazole acts as an electron-rich diene. With activated alkynes the reaction can even occur at room temperature. In the case of unsymmetrically substituted alkynes, generally both regioisomeric furans are obtained <70JA4340, 70JCS(C)552, 79SC789, 81LA668, 81TL1727, 83TL2473, 85CPB3670, B-87MI 207-01, 89CC1238, 93MI 207-01>, but alkynones react with 5-ethoxy-4-methyloxazole to give only one isomer <94TL3609, 94TL3613>. This method is useful for the regioselective synthesis of 4-hydroxybutenolides (Scheme 32).



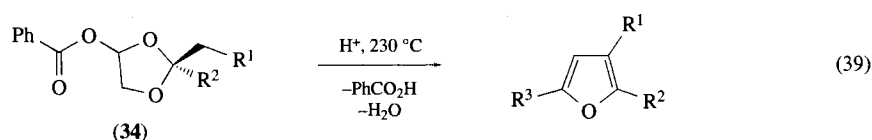
Scheme 32

The intramolecular variant of this sequence yields 2,3-disubstituted furans <81JOC2065, 84JA5585, 89TL6607, 91JA5384>. Both mesoionic 1,3-oxazolium-4-olates (**33**; X = N—CH₃) and 1,3-oxathiazolium-4-olates (**33**; X = S) react with activated alkynes to give furanes. Yields are moderate to good (Scheme 33) <75CL21, 78CB3336, B-84MI 207-01, 85CB785, 86CB2317, 89CB1081>.

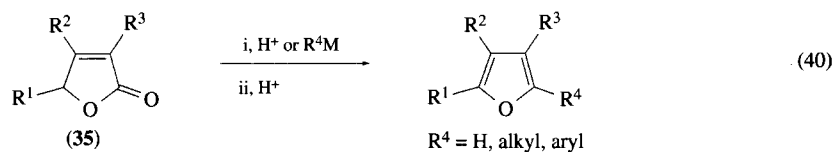


Scheme 33

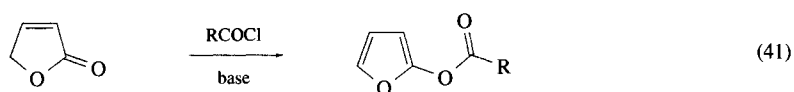
In an interesting sequence, 4-benzoyl-1,3-dioxolanes (**34**) are transformed at higher temperature under the influence of acids into furans (Equation (39)) <76AG718, 76AG(E)682>.



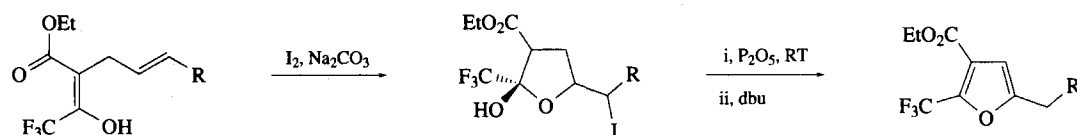
Furanones <75JOC542, 75S67, 76CRV625, 78FOR133> are suitable precursors for furans. Reduction of 5H-furan-2-ones (**35**) ([[(CH₃)₂CHCH₂]₂AlH, (CH₃OCH₂CH₂O)₂AlH₂⁻Na⁺, NaBH₄) or treatment with organometallic reagents (R⁴MgBr, R⁴Li) with subsequent elimination of water yields furans (Equation (40)) <66JCS(C)377, 74IJC(B)1144, 76SC331, 77TL2869, 78H(10)87, 82JCS(P1)257>.



Treatment of 5H-furan-2-one with electrophilic agents (acid chlorides, etc.) yields O-protected 2-hydroxyfurans (Equation (41)) <83S85, 86SC69>; O-silylated derivatives are prepared similarly <77CL167>. If 3-furanones are used as starting material, the corresponding 3-silylated derivatives are obtained <76H(4)1663, 80TL2891>.

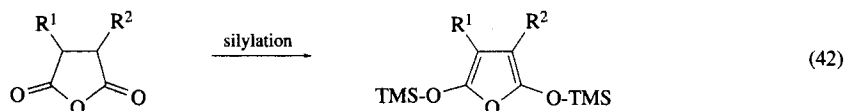


Trifluoromethylated furans (**35**) are available via iodolactonization of γ -substituted ethyl trifluoroacetoacetates; tetrahydrofurans are intermediates in this reaction (Scheme 34) <93JCS(P1)2787>.



Scheme 34

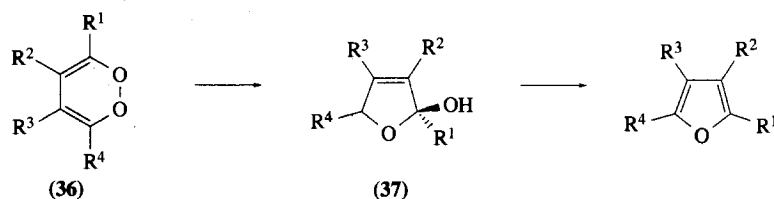
Treatment of succinic anhydrides with silylating agents (TMS-Cl/ZnCl₂/Et₃N; (TMS-OSO₂C-F₃/Et₃N) yields 2,5-bis(silyloxy)furans (Equation (42)) <80TL3423, 87LA839, 90HCA790, 90TL5741>.



(42)

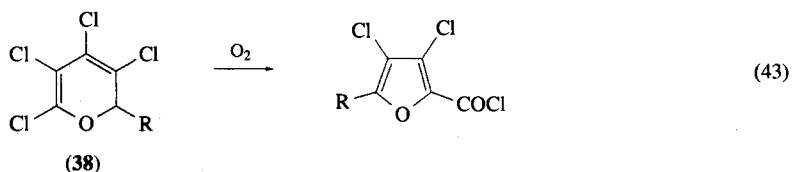
2.07.2.2.2 From six-membered heterocycles

3,6-Dihydro-1,2-dioxines (**36**), which are available from dienes and ¹O₂ <B-85MI 207-01> can be transformed into furans (Scheme 35). The rearrangement (to **37**) and the elimination of water is usually effected by treatment with base. In some cases Co(II) and Fe(II) catalysts have been used <76TL4363, 77JOC1900, 77SC119, 82ACS(B)31, 89JOC3475>.



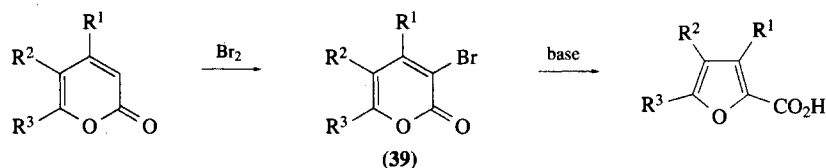
Scheme 35

Halogenated 2*H*-pyrans (**38**) on treatment with dry oxygen are transformed to 2-chloro-carbonylfurans (Equation (43)) <75LA240>.



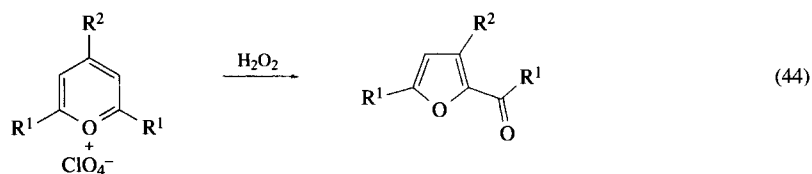
(43)

3-Bromopyrones (**39**), which are available from pyrones with bromine, on treatment with base yield furancarboxylic acids (Scheme 36) <62JCS5297, 68JCS(C)769, 73JCS(P1)1130, 75H(12)129, 80JOC1524>.

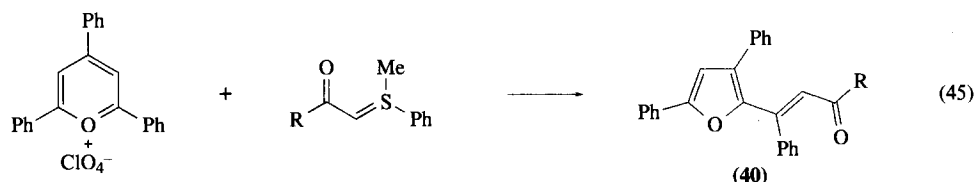


Scheme 36

The oxidation of pyrylium salts with hydrogen peroxide gives furans (Equation (44)) <69OPP63>.

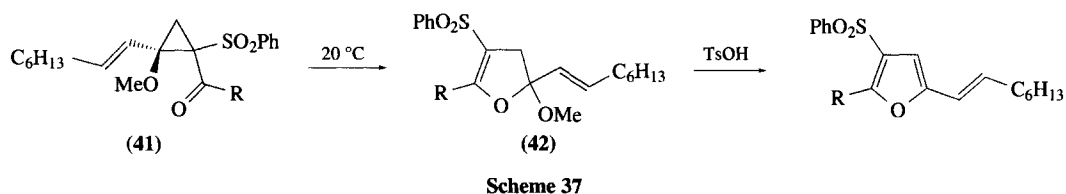


2(3-Oxo-1-propenyl)furans (**40**) are formed in high yields on treatment of pyrylium salts with acyldenesulfuranes (Equation (45)) <75JCS(P1)2489>.



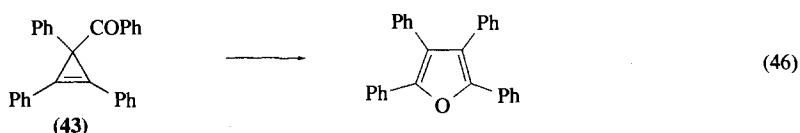
2.07.2.2.3 From three-membered rings

Acylcyclopropanes (**41**) can be transformed to dihydrofurans (**42**) either thermally or photochemically <69JOC2301, 73CJC1487, 84CC1542>. If suitable leaving groups are present, furans are formed on treatment with acids (Scheme 37) <93TL7583>.

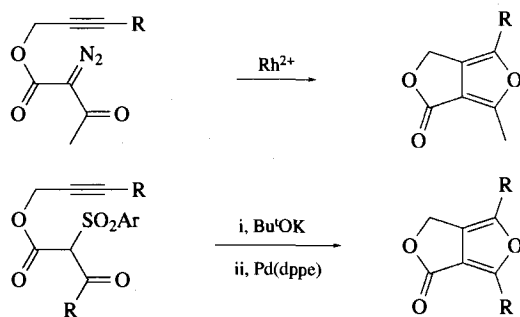


Scheme 37

It has been observed that acylcyclopropanes (**43**) can give furans under similar conditions (Equation (46)) <75T2495, 78TL1015, 88T6345, 91JOC6971, 92JA363, 94AG1881, 94AG(E)1797>.

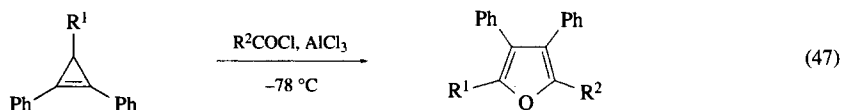


It is an open question whether carbenes are involved in the photochemical rearrangement of acylcyclopropanes <92JA6603>. Treatment of diazoketones with rhodium catalysts generate a keto-carbene (ketocarbenoid), which can be added to an alkyne to give furans (Scheme 38) <92T5385, 93JOC21>. α -Sulfonylalkylketones react similarly (Scheme 38) <94SL447>.

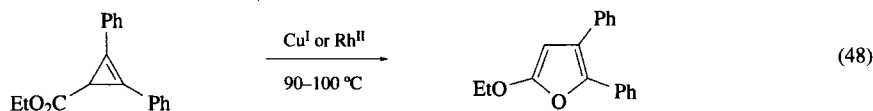


Scheme 38

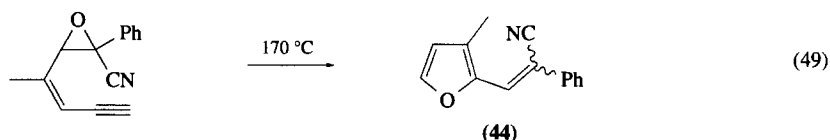
The reaction of cyclopropanes with acyl chlorides, which yields furans (Equation (47)), is of some generality <87SC263>.



On heating 2,3-diphenyl-1-ethoxycarbonylcyclopropene with Cu(I) or Rh(II) catalysts, 2,3-diphenyl-5-ethoxyfuran is obtained in 90% yield (Equation (48)) <90HCA1233>.



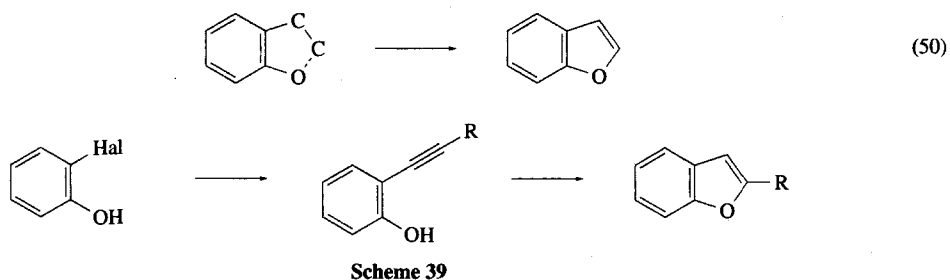
Electrocyclic ring opening of oxiranes – either thermally or photochemically – yields carbonyl ylides, which can be trapped with alkynes to give dihydrofurans. If 1-buten-3-ynyl-oxiranes are used as starting materials, the corresponding ethenylfurans are obtained (Equation (49)). This sequence, which has been studied quite intensively by Eberbach and co-workers, can also be used to prepare annulated derivatives <93CB975>.



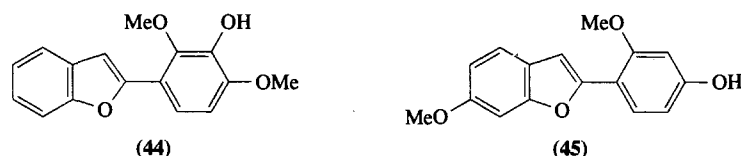
2.07.3 BENZO[b]FURANS

2.07.3.1 Ring Closure by Formation of the O—C(2) Bond

The ring closure between the oxygen atom and a suitably substituted C—C bond (Equation (50)) forms the basis for a wide variety of syntheses of benzo[b]furans. The *ortho*-substituent of the phenol may be an unsaturated (C≡C—R, CH=C(Hal)Ar, CH₂—CH=CH₂, CH₂=C=CH—CH₂—COR) or a saturated group (CH₂—CH₂OH, oxiranyl). Heating of *o*-alkynylphenols, which are available from *o*-halogenophenols and alkynes – without or with catalysts (e.g., Pd(PPh₃)₂(OAc)₂/LiI/2,4,6-trimethylpyridine) – yields benzofurans (Scheme 39).

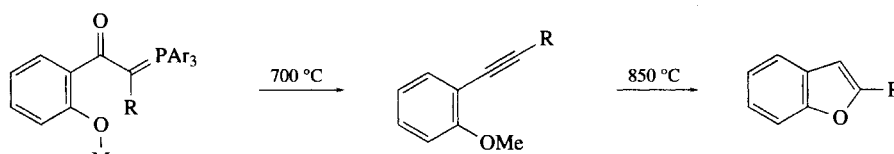


Both coupling and ring closure can be done in one step <63JOC3313, 66JOC4071, 69JA6464, 69JOC1974, 71TL3311, 72CB1943, 85JCS(P1)2443, 86S320, 86S749, 89H(29)1255, 89TL1597>. Pterofuran (44), vignafuran (45) and other naturally occurring 2-arylbenzofurans have been synthesized by this method <76JCS(P1)1514, 77JCS(P1)802, 79JOC4710>.



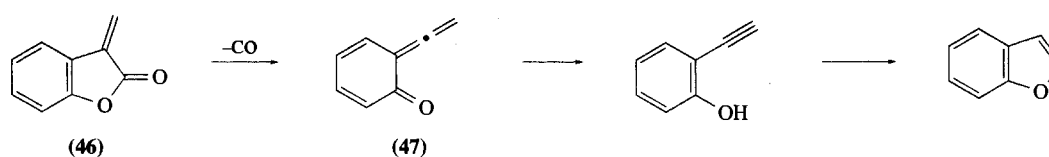
The preparation of 2-(1-naphthyl)benzofurans can be accomplished in yields up to 80% if the intermediates are isolated and purified <77TL419>. The cyclization of 2-alkynylphenols or

2-alkynylphenol ethers (Scheme 40) can also be effected by flash vacuum pyrolysis (FVP) <85JOC158, 87TL3717>.

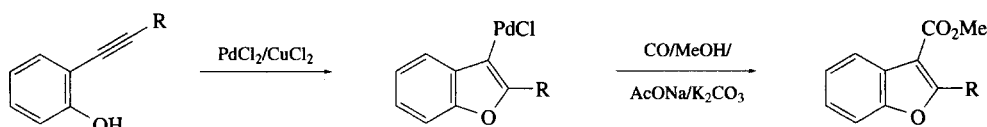


Scheme 40

Flash thermolysis converts the methylene lactone (46) to benzofuran (Scheme 41) <81TL3597>. Probably ketoketenes (47) are intermediates in this reaction. If the Pd-catalyzed cyclization is conducted in the presence of CO/CH₃OH the corresponding 3-methoxycarbonyl-substituted benzofurans are obtained (Scheme 42) <89H(29)1013>.

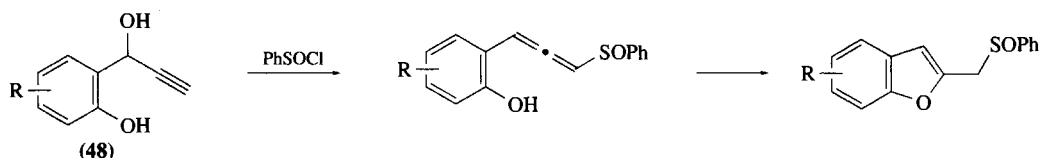


Scheme 41



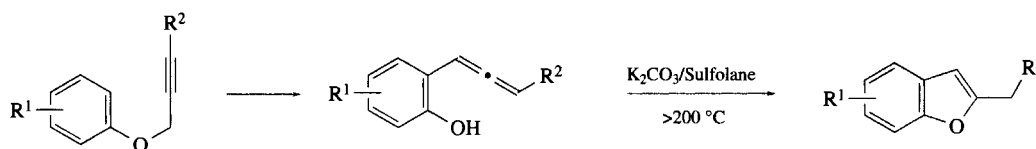
Scheme 42

Reaction of alkynylcarbinols of type (48) with phenylsulfinyl chloride leads to sulfinate esters; the cyclization yields benzofurans (Scheme 43) <92SL597>.



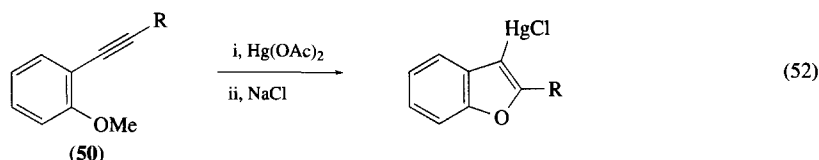
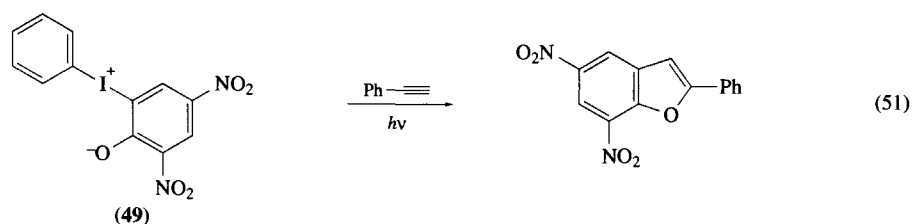
Scheme 43

The Claisen rearrangement of propargylarylethers yields allenes, which cyclize in the presence of base to give benzofurans (Scheme 44) <73HCA1457, 73HCA2981, 73JOC3832, 81JCS(P1)1417, 82JCS(P1)107, 83TL5023>.

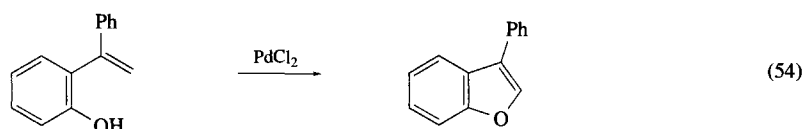
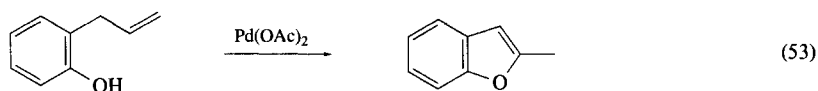


Scheme 44

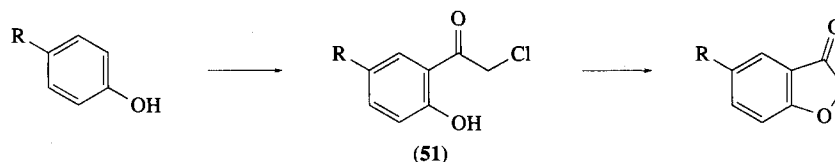
On irradiation in the presence of alkynes the stable zwitterion (49) yields 5,7-dinitrobenzofuran (Equation (51)) <86JOC3453>. In an intramolecular alkoxymercuration reaction phenyl ethers of type (50) are cyclized with mercury(II) acetate to give mercurated benzofurans (Equation (52)) <84JA4218, 86JOC3453>.



The cyclization of *o*-allylic or vinylic phenols can be effected by palladium catalysts (Equations (53) and (54)) <73TL739, 75BCJ1533, 75G1151, 76BCJ3662, 77S122, 78CC687, 81JA2318, 85JOC1282>.

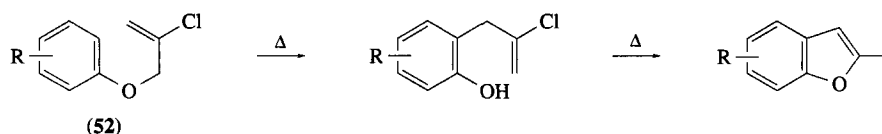


Both linear and angular annulated 2-methylfurobenzopyranones can be prepared by this route <94BCJ1972>. Benzofuran-3-ones (3-hydroxybenzofurans) are available by an intramolecular nucleophilic substitution reaction (Scheme 45), (Auwers–Pohl reaction).



Scheme 45

The starting material (51) can be prepared conveniently either by Friedel–Crafts acylation or by a Fries rearrangement. Quite a number of natural products have been synthesized using this methodology (griseofulvin, furanochromes, khellin derivatives, isopiminellins) <48JA1970, 56JCS2322, 51JA5765, 54JCS429, 55JOC813, 55JA1623, 59JOC2000, 72AJC543, 73IJC989, 79JHC799>. The reaction of phenols with 2,3-dichloropropene yields allyl phenyl ethers (52). A thermally induced rearrangement with subsequent cyclization (either in a one-step or in a two-step reaction) gives 2-methylbenzofurans (Scheme 46) <74CC174, 76JCS(P1)1, 80AJC1817>.

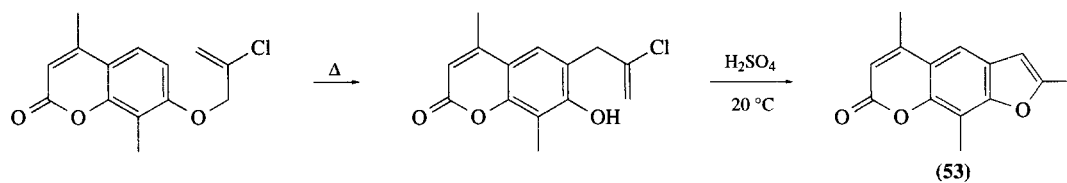


Scheme 46

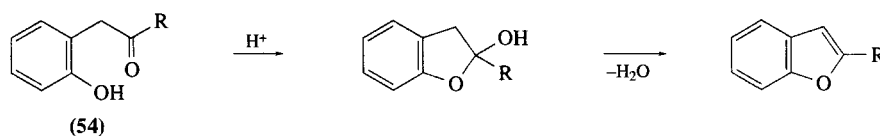
4,5',8-Trimethylpsoralen (53) has been prepared by this method (Scheme 47) <79JOC2176>. The acid-catalyzed cyclization of carbonyl compounds of type (54) yields benzofurans (Scheme 48).

2-Acyloxy-1-bromomethylarenes (55) can be transformed to 2-alkylbenzofurans with Cr(II)-Cl₂/BF₃·OEt₂ in tetrahydrofuran; the first step of this interesting reaction seems to be a 1,4-transposition <86CC171, 87T5841>.

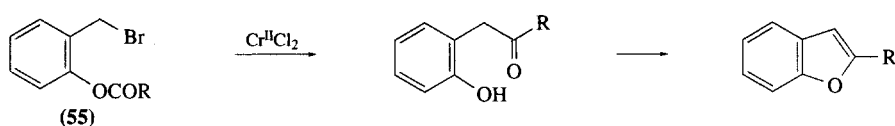
Phenol ethers can also be used as starting material for the synthesis of benzofurans (Scheme 50) <56ACS1422, 70BSB235, 78JOC3063, 80JOC3994, 80S236, 83JCS(P1)2423, 85CC1543>.



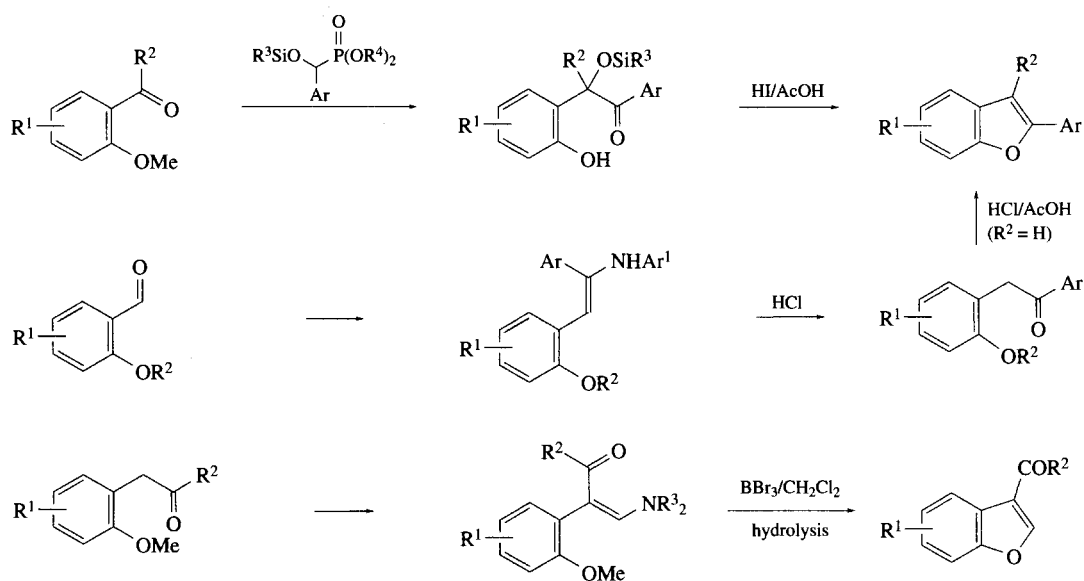
Scheme 47



Scheme 48



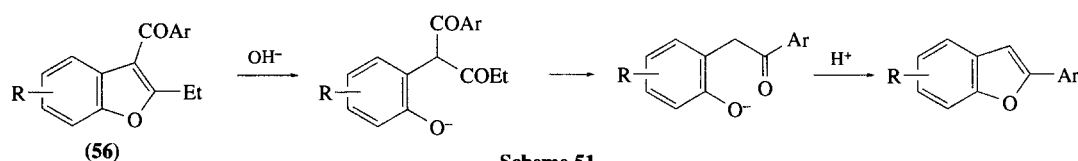
Scheme 49



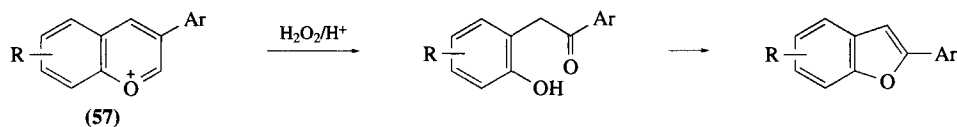
Scheme 50

Base-catalyzed ring opening of 3-arylbenzofurans (**56**) with subsequent acid-catalyzed ring closure yields 2-arylbenzofurans (Scheme 51) <77JHC867>. Compounds of this type can also be obtained by treatment of benzopyrylium salts (**57**) with hydrogen peroxide in an acidic medium (Scheme 52) <77TL419, 79TL1109>.

Under acidic conditions (H_3PO_4 , AcOH , $\text{ZnCl}_2/\text{AcOH}$, BF_3/AcOH , HCl/EtOH , H_2SO_4),

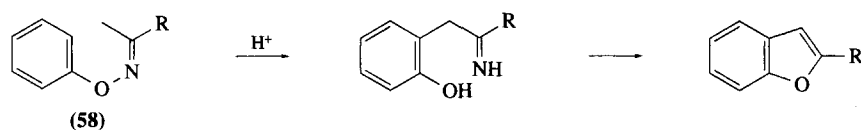


Scheme 51



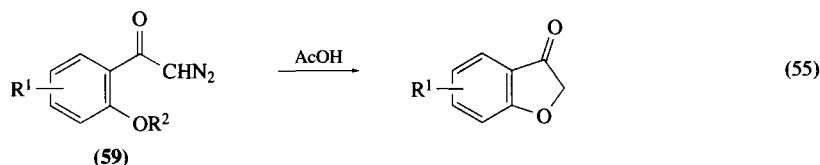
Scheme 52

O-arylketoxyimes (**58**) give 2-substituted benzofurans. This reaction (Scheme 53) resembles quite closely the Fischer indole synthesis <67JHC413, 67TL859, 67TL2867, 73CHE26, 73KGS31, 76MI 207-01, 79JOC2176, 85CC417>.



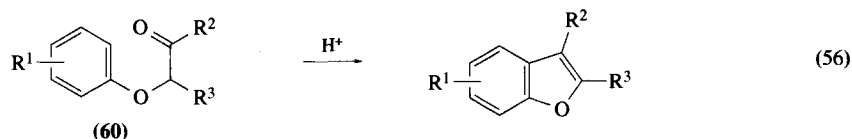
Scheme 53

A O—C(2) ring closure can also be effected by treatment of diazo carbonyl compounds of type (**59**; R² = H, CH₃) with acetic acid (Equation (55)) <73JIC989>.

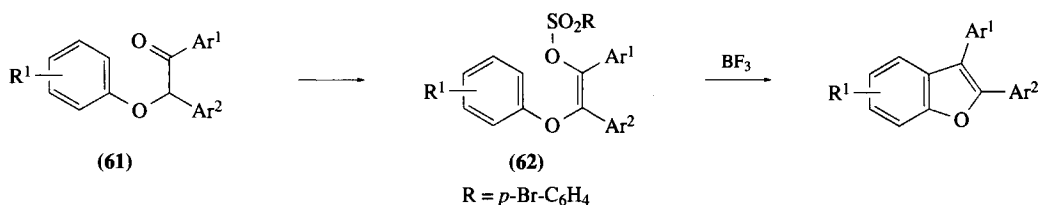


2.07.3.2 Ring Closure by Formation of the Ring-C(3) Bond

The acid-catalyzed cyclization of α -aryloxy carbonyl compounds of type (**60**) has proved to be of great value for the synthesis of a wide variety of benzofurans (Equation (56)) <61JA3096, 70BCJ1976, 70BCJ3496, 70BSF1029, 70BSF1037, 70BSF2253, 70BSF3601, 72TL237, 94JL221>.



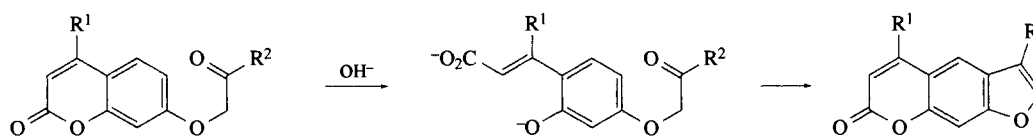
It has been observed that 3-arylbenzofurans may rearrange to 2-arylbenzofurans under the conditions of cyclization <58JCS822, 77TL419>. If aryloxyalkanones of type (**61**) are treated with *p*-bromobenzenesulfonyl chloride, the corresponding vinyl esters (**62**) are obtained (Scheme 54). These compounds can be cyclized to 2,3-diarylbenzofurans with BF₃ <72JCS(P1)216>.



Scheme 54

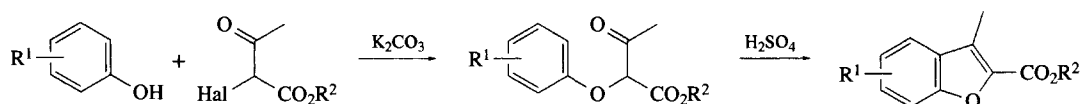
2,3-Diarylbenzofurans can also be obtained by treatment of phenols with benzoin and subsequent ring closure of the α -aryloxyketones with sulfuric acid <76ZN(B)1138, 77ZN(B)1084, 81JIC697>. If the

C(2)-ring closure occurs in the *para* position to a hydroxy group, the cyclization can be achieved by bases (Scheme 55) <72TL237>.



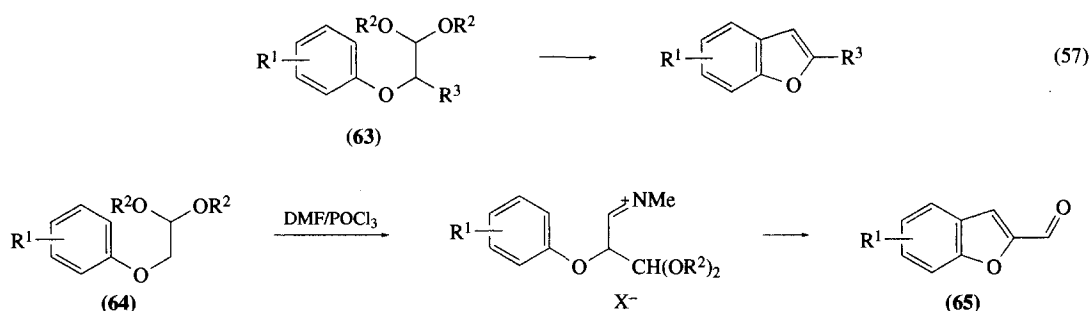
Scheme 55

Phenols react with α -halogeno- β -ketoesters to give aryloxy compounds. Cyclization with concentrated sulfuric acid yields 2-alkoxycarbonylbenzofurans (Scheme 56) <63OSC(4)590>.



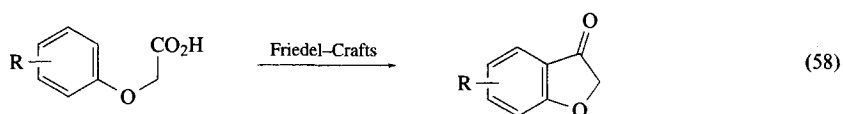
Scheme 56

α -Aryloxyaldehydes are often used as protected derivatives (acetals); these compounds (**63**) are easily available from α -halogenoacetals and phenols <57BJ188, 73BSF2355, 74BCJ1263, 84JCS(P1)1479, 89SC257>. The reaction conditions for the cyclization step (Equation (57)) may be crucial <72JCS(P1)556>. If the cyclization of 2-aryloxy-1,1-dialkoxyethane (**64**) is conducted in the presence of DMF/ POCl_3 , 2-formylbenzofurans (**65**) are obtained (Scheme 57) <86JHC1715>.



Scheme 57

Aryloxyacetic acids cyclize under Friedel–Crafts conditions to give benzofuranones (Equation (58)) <25LA254, 68JCS(B)745, 85JHC1243>.

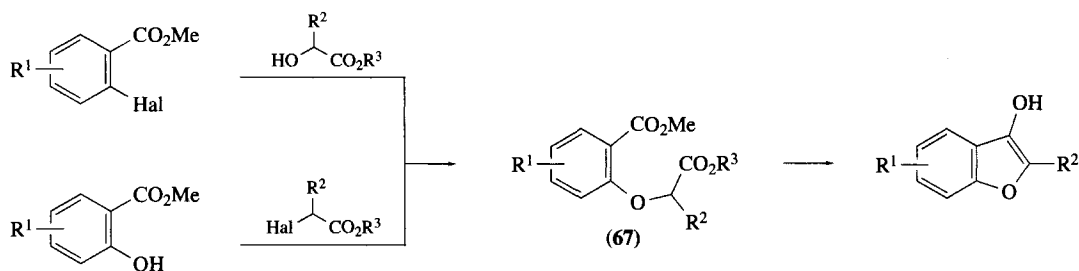


If *N,N*-disubstituted amides are used as starting material, the corresponding 3-dialkylamino-benzofurans are obtained <73JOC1746>. Suitably substituted allyl phenyl ethers can be cyclized to 2,3-dihydrobenzo[*b*]furans <94H(38)177>.

2.07.3.3 Ring Closure by Formation of the C(2)—C(3) Bond

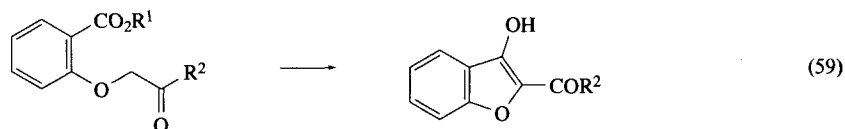
Dieckmann condensation of activated methylene compounds of type (**67**), which can be prepared either from the corresponding halogenated arenes or by etherification of phenols, yields benzofuran-3-ones (3-hydroxybenzofurans) (Rössing synthesis) (Scheme 58) <1899CB1867, 58JA5808, 60AJC95, 62HCA2241, 62JOC586, 67JCS(C)869, 71AJC1883, 72AJC545, 86JHC549, 86JHC665>. A general access to benzofurans is accomplished by the reaction of 2-acyl-1-hydroxy-arenes with α -halogeno carbonyl compounds and subsequent ring closure (Rössing synthesis) <1884CB2988, 09CB901, 10CB2155, 48HCA75,

50JCS3206, 51JA872, 58JA5808, 62HCA2241, 69JOC2311, 71AJC1883, 71JCS(C)2834, 73BCJ1772, 73BSF2355, 74BCJ485, 75BCJ488, 75BCJ1249, 83BCJ2762, 83BCJ2767, 85BCJ2821).

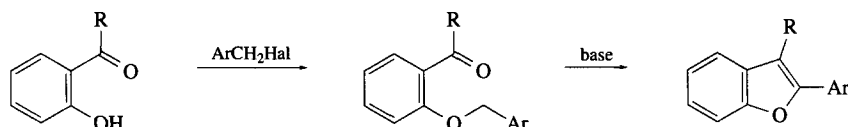


Scheme 58

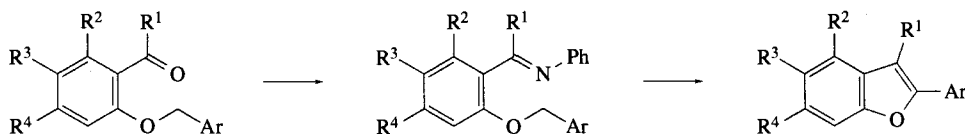
2-Acyl-3-hydroxybenzofurans can also be obtained by this route (Equation (59)) <55JA1623, 84S1068>.



2-Arylbenzofurans are available by similar methods (Scheme 59) <62T1311, 69TL4139, 71LA(749)68, 72AJC545, 74LA523, 75BCJ1249, 82LA1836, 85BCJ2192>.



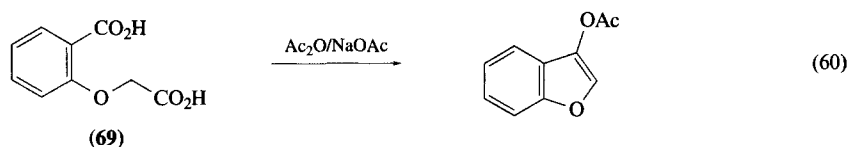
Scheme 59



Scheme 60

In some cases it seems to be convenient to prepare the imines (68) in advance <74LA523>.

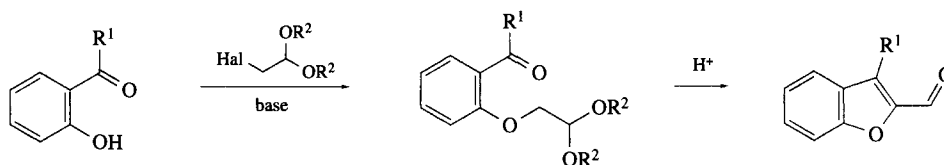
The cyclization of the corresponding acids (69) with acetic anhydride/sodium acetate yields 3-(O-acetoxy)benzofurans <71BSF2072, 76CR(C)993>.



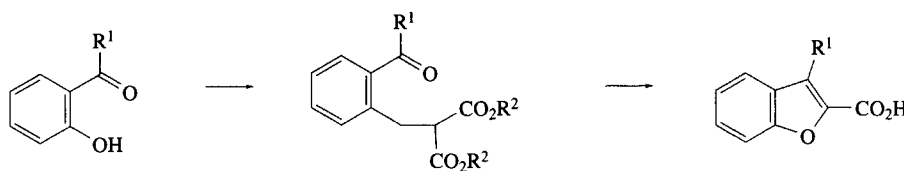
On treatment of 2-acylphenols with unprotected (e.g., chloroacetone) or protected 2-halogenalkanals with subsequent acid-catalyzed ring closure, benzofuran-2-carbaldehydes are accessible (Scheme 61) <00LA237, 39JA2705, 46ZOB1877, 56JOC1458, 57JCS2593, 60CB54, 62T1311, 69TL4139, 72AJC545, 81IJC(B)1053, 86JHC1277, 87SC341>.

In the methodology of Tanaka and co-workers, *o*-acylphenols are treated with bromomalonate; after condensation, saponification, and decarboxylation the corresponding benzofuran-2-carboxylic acids are obtained (Scheme 62) <51JA872, 72JCS(P1)556, 82LA1836, 85JHC1377>.

A C(2)—C(3) bond formation can also be accomplished by an intramolecular [2 + 2] cycloaddition reaction. 2-Acylaryloxyacetic acids (70) on treatment with acetic anhydride/sodium acetate yield

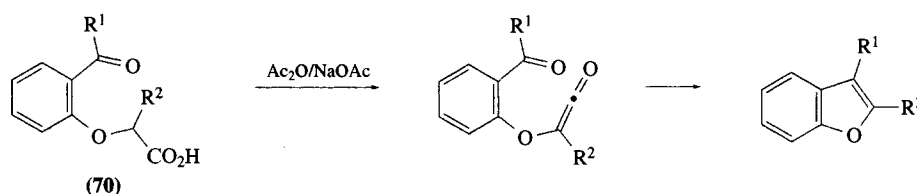


Scheme 61



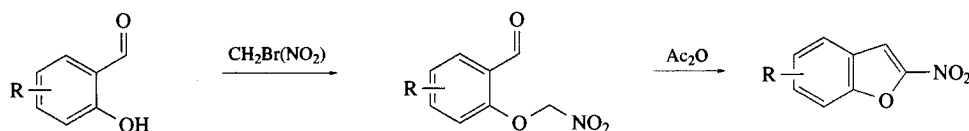
Scheme 62

benzofurans (Scheme 63). A ketene has been proposed as an intermediate <88JHC969, 92MI 207-03>. The course of the reaction may depend on the conditions employed <87JHC965>.



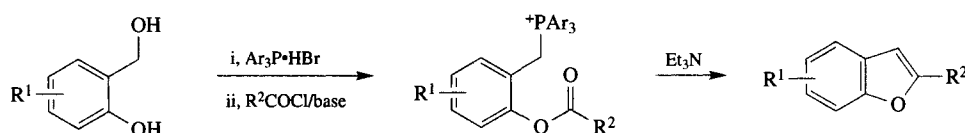
Scheme 63

The ketenes can also be generated from the acid chlorides (Et_3N) or on treatment of the acids with $p\text{-TsCl}/\text{Et}_3\text{N}$ <86JOC2145, 87JOC3457, 87S395>. 2-Nitrobenzofurans are available from o -hydroxybenzaldehydes by reaction with bromonitromethane and subsequent ring closure with acetic anhydride (Scheme 64) <70BSF3740, 72BSF4158, 83MI 207-01, 84CPB4260, 84S978, 85S1074, 88CPB1336>.



Scheme 64

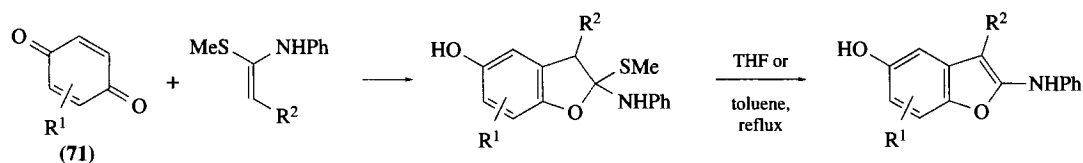
The C(2)—C(3) ring closure can also be effected by an intramolecular Wittig reaction (Scheme 65) <79TL2145, 81T2867, 88CB2259>.



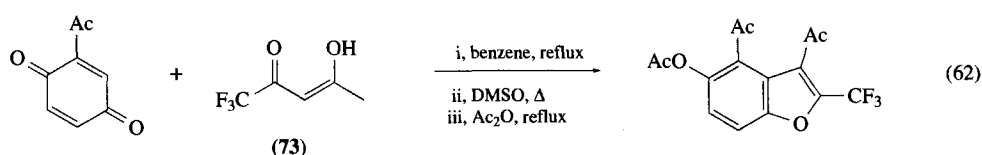
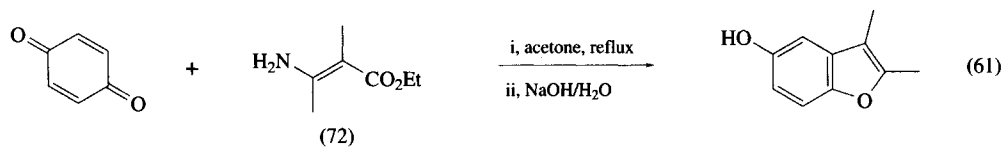
Scheme 65

p -Benzoquinones are suitable starting materials for the synthesis of benzofurans. Treatment of (71) with 1-(N -methylanilino)-1-methylthioether in refluxing tetrahydrofuran or toluene yields a 2-aminobenzofuran (Scheme 66) <87JCS(P1)1501>.

β -Amino- α,β -unsaturated carboxylic esters (72) and enols of 1,3-diketones (73) react similarly (Equations (61) and (62)) <53JCS1262, 87CB1791, 88CB1841>.

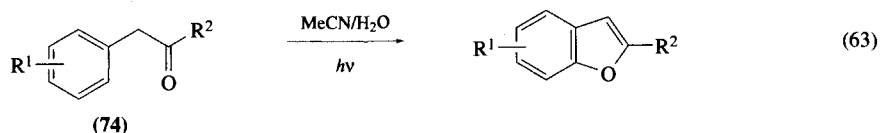


Scheme 66

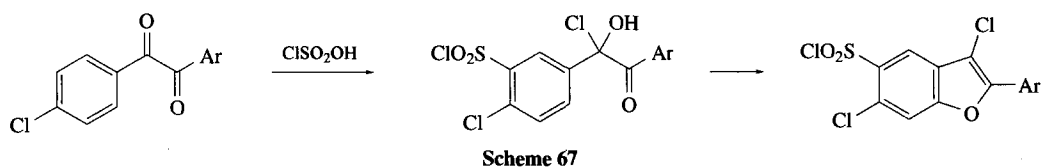


2.07.3.4 Ring Closure by Formation of the Ring—O Bond

It has been reported that aryl-substituted alkanones of type (74) can be cyclized photolytically to benzofurans (Equation (63)). This synthesis does not seem to be of preparative importance <89TL1867>.



Treatment of benzils with chlorosulfonic acid yields 2-arylbenzofurans (Scheme 67) <87JHC117>.



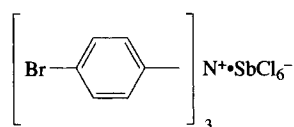
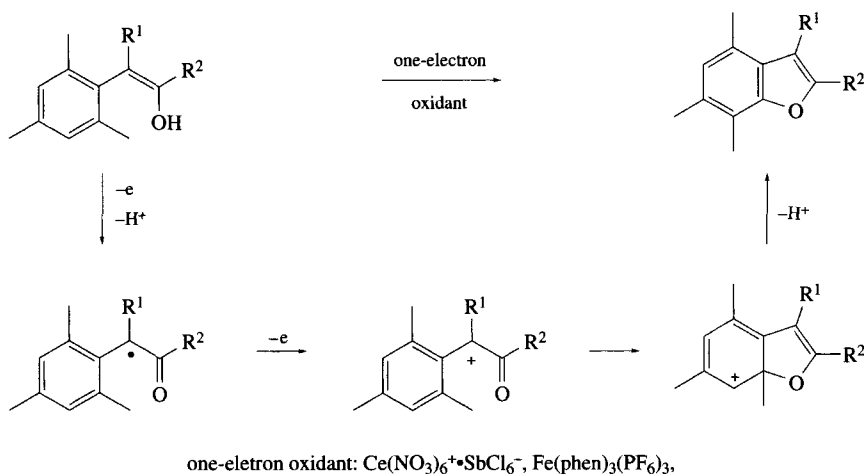
Scheme 67

4,6,7-Trimethylbenzo[*b*]furans are formed by a one-electron oxidation of mesityl-substituted enols (Scheme 68) <94JPR325>.

2.07.3.5 From Furans and Dihydrofurans

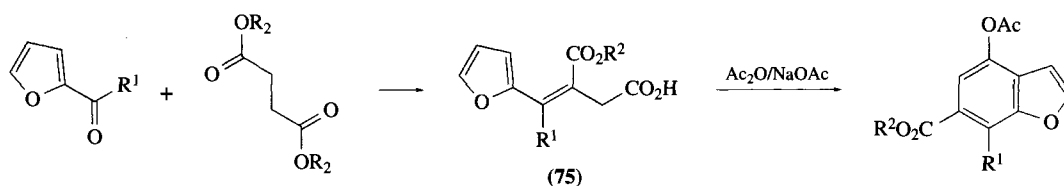
Benzofurans are also available from suitably substituted furans by cyclization and generation of the benzene ring. Stobbe condensation of 2-acylfurans with succinic esters yields a carboxylic acid (75), which on treatment with acetic anhydride/sodium acetate gives a 4-acetoxybenzofuran (Scheme 69) <72JPR213>.

Electrophilic ring closure of alkylidenefuranones of type (76) (Δ^{βγ}-butenolides), which are available from furan-2-carbaldehyde and γ-keto acids, with AlCl₃ yields 4-arylbenzofuran-6-carboxylic acids (Scheme 70) <77JPR689>.

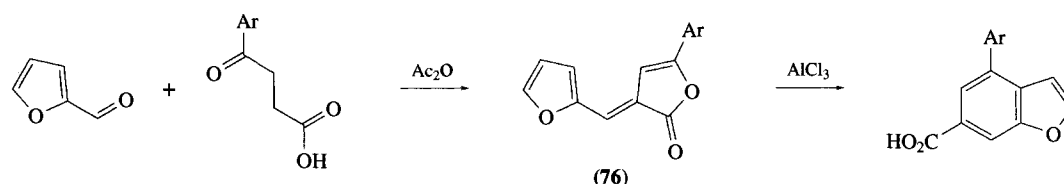


Scheme 68

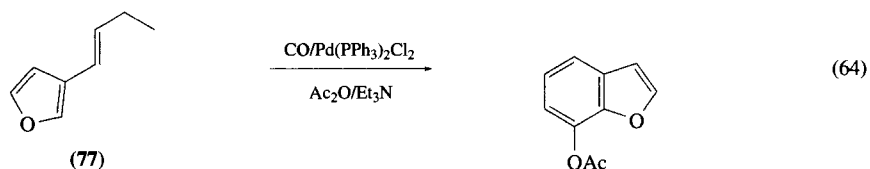
A Pd-catalyzed cyclocarbonylation of 3-acetoxy-1-(3-furyl)-propenes (**77**) in the presence of acetic anhydride yields 4-acetoxymethyl-2-benzofurans (Equation (64)) <89TL95>.



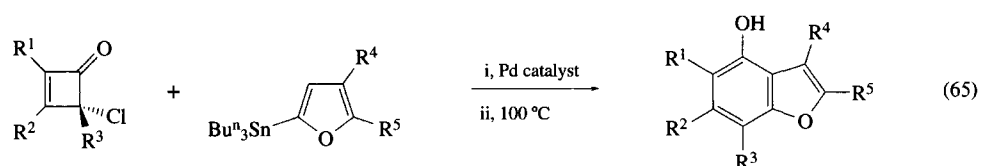
Scheme 69



Scheme 70

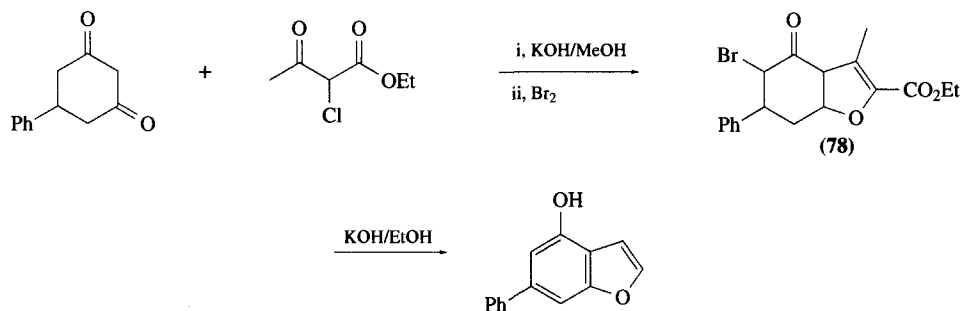


4-Chloro-2,3-disubstituted cyclobutenones undergo a Pd-assisted cross coupling with 2-stannylated furans; subsequent heating to 100°C gives 4-hydroxybenzo[*b*]furans (Equation (65)) <93JOC3550>.



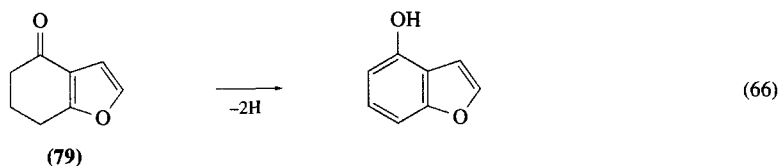
2.07.3.6 Miscellaneous Syntheses

The benzene ring of benzofurans can also be built up by elimination reactions from suitably substituted precursors. Treatment of (78), which is available from 1,3-diketo-5-phenylcyclohexane and ethyl α -chloroacetoacetate and subsequent bromination with base, yields a 4-hydroxybenzofuran (Scheme 71) <76CHE733, 76KGS879>.

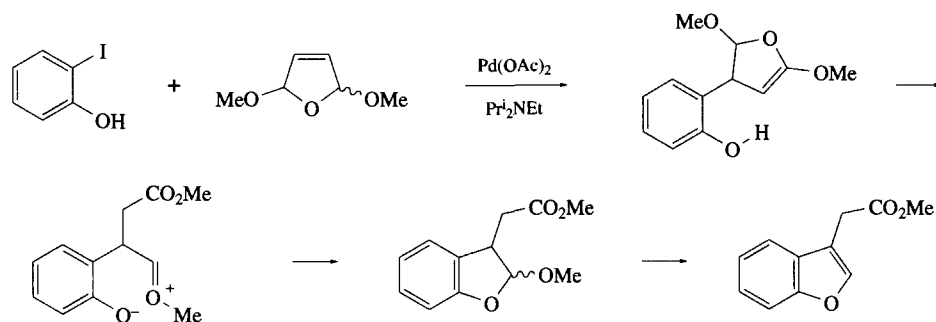


Scheme 71

4-Hydroxybenzofurans are available from 4-ketotetrahydrobenzofurans (79) by dehydrogenation (Equation (66)) <55CB271, 73AJC201, 77CHE831, 77KGS1036, 86SCI635, 88JOC4135>.



Heck reaction between 2-iodophenol and 2,5-dimethoxy-2,5-dihydrofuran yields methyl 3-benzo[*b*]furanylacetate in one step <94H(38)1745>.



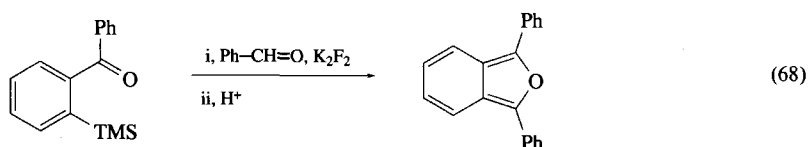
Scheme 72

2.07.4 BENZO[C]FURANS

2.07.4.1 From *o*-Diacylarenes, *o*-Hydroxyalkyl(aryl)acylarenes, 1-Hydroxyphthalans, *o*-(1-Diazoalkyl)acylarenes, and Related Compounds

Reduction of *o*-diacylarenes with subsequent treatment of the reaction mixture with acids provide a convenient route to 1,3-diarylbenzo[*c*]furans (Scheme 73); zinc dust/NaOH and complex

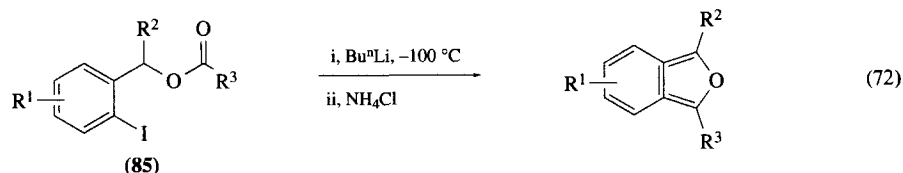
Scheme 73

$$\begin{array}{ccc}
 \text{Ar}^1 & & \\
 \parallel & & \\
 \text{C}_6\text{H}_4\text{Br} & \xrightarrow[\text{iii, H}^+]{\begin{array}{l} \text{i, Bu}^\text{t}\text{Li} \\ \text{ii, Ar}^2\text{CH=O} \end{array}} & \text{Ar}^1 \\
 & & \diagup \quad \diagdown \\
 & & \text{O} \\
 & & \text{Ar}^2
 \end{array} \quad (67)$$
[illegible]

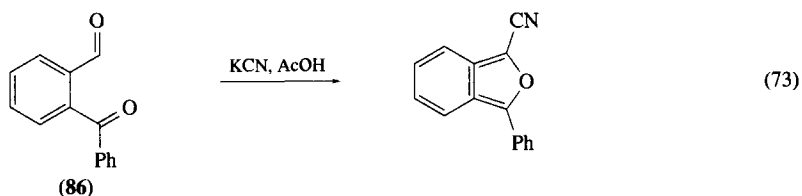
(83) (70)

(84) $\xrightarrow{H^+}$ (71)

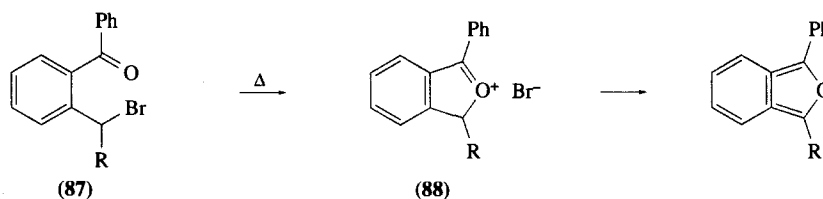
The reaction of *o*-bromobenzylalcohol with BuⁿLi/benzonitrile yields a benzophenonimine, which on treatment with acids gives 1-phenylbenzo[*c*]furan (trapped with dienophiles) <88JOC2942>. Iodine-metal exchange in esters of type (85) and subsequent intramolecular reaction of the metalated arene with an ester carbonyl group yields a hydroxyphthalan. Under acidic conditions the corresponding benzo[*c*]furans are generated (Equation (72)) <92CC164>.



An interesting synthesis, which could be of some generality, starts from *o*-formylbenzophenone (86). Treatment of this compound with KCN in acetic acid yields 1-cyano-3-phenylbenzo[*c*]furan (Equation (73)) <70TL707>.

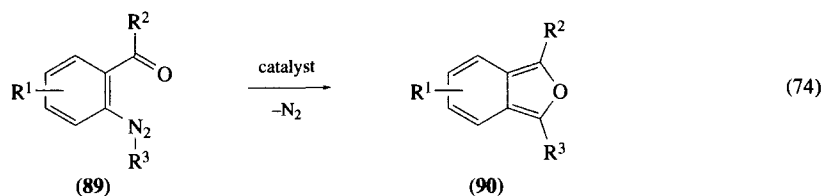


In an intramolecular nucleophilic substitution reaction, bromoketones of type (87) yield arylbenzo[*c*]furans; clearly oxonium salts of type (88) are involved (Scheme 74) <76JCS(P1)336>.



Scheme 74

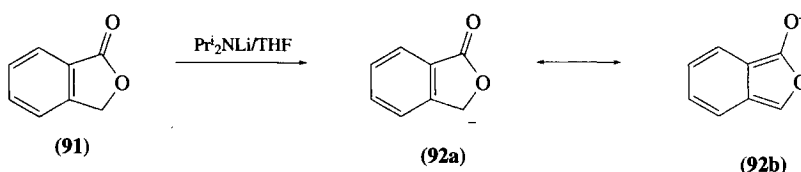
A convenient route to benzo[*c*]furans, which has been extended to a great variety of substituted derivatives, starts with diazo compounds of type (89). Nitrogen can be extruded either by light or by metal catalysts to give a carbene or a carbenoid, which undergoes cyclization to (90) (Equation (74)) <73JA7402, 76CL287, 77LA116, 83TL2945, 86H(24)297, 86JOC3325, 87TL4279, 88TL2045, 89H(29)1003, 89H(29)1243, 93ZN(B)213>.



2.07.4.2 From Phthalides

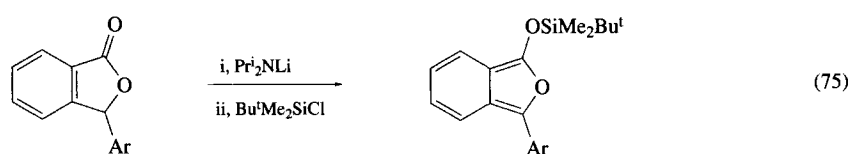
Phthalides are versatile starting materials for a wide variety of benzo[*c*]furans. Deprotonation of the parent compound (91) with strong bases (e.g., Pr₂NLi) results in the generation of a deep red-

orange solution, which contains the corresponding anion. It can be considered as the anion of 1-hydroxybenzo[*c*]furan (**92b**) (Scheme 75).

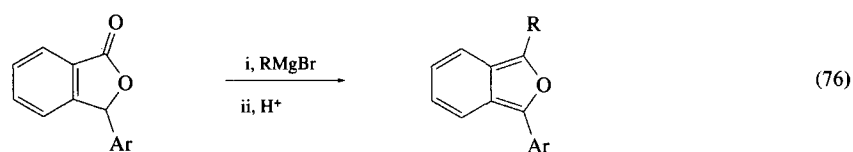


Scheme 75

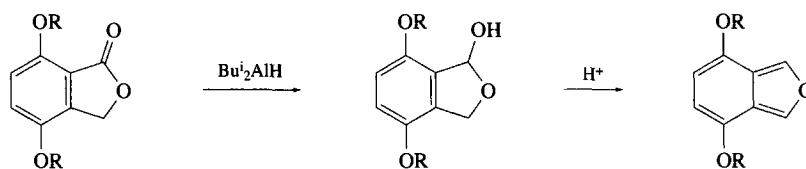
Generation of the corresponding anion of 3-arylphthalides with subsequent *O*-silylation yielded *O*-silylated benzo[*c*]furans, which were trapped with dienophiles (Equation (75)) <84CL1263, B-89MI 207-02>.



Treatment of arylphthalides with Grignard reagents or other organometallics with subsequent acid-catalyzed dehydration yields benzo[*c*]furans (Equation (76)) <61JOC2630, 62JA2008, 63BSF229, 69JOC538, 70TL1409, 74JOC3648, 78JCR(M)4944, 78JCR(S)408, 92AM(4)562, 92AM(4)564, 92AM(4)649>.



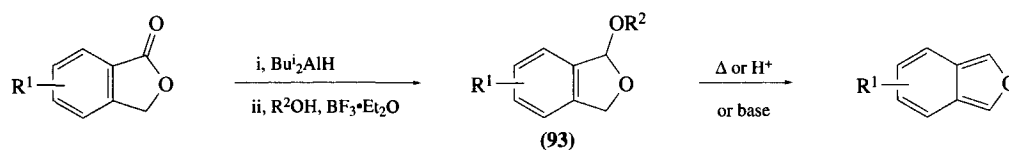
It is possible to reduce phthalides to phthalanols (1-hydroxyphthalans), which on treatment with acids yield benzo[*c*]furans (Scheme 76) <78MI 207-01, 84T4597, 85CJC735, 86CB1876>.



Scheme 76

If deuterated complex metal hydrides are used, 1-deuteriobenzo[*c*]furans are obtained <85JOC5902, 88T2093>.

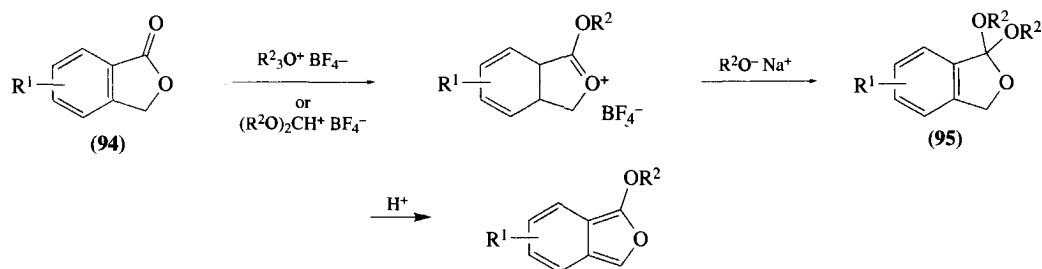
The reduction of phthalides with subsequent treatment of the resulting phthalanols with $\text{ROH}/\text{BF}_3 \cdot \text{Et}_2\text{O}$ yields 1-alkoxy-1,3-dihydrobenzo[*c*]furans (**93**) (Scheme 77). The elimination of R^2OH can be accomplished thermally, with acids, or with bases (e.g., Bu_2NLi , benzene) <79JA1196, 82JOC4011, 83JOC2237, 83JOC3869, 84JOC304, 86JOC3849>.



Scheme 77

Phthalides are also versatile precursors for the preparation of 1-alkoxybenzo[*c*]furans. *O*-Alkylation of (**94**) with trialkyloxonium tetrafluoroborate or diethoxymethylum tetrafluoroborate with

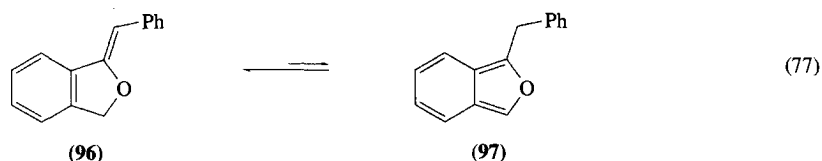
subsequent treatment of the oxonium salts with sodium alcoholates yields 1,1-dialkoxy-1,3-dihydrobenzo[*c*]furans (**95**). The acid-catalyzed elimination of the carbinol R^2OH yields benzo[*c*]furans (Scheme 78) <80CJC2573, 80JOC4061, 83JOC2237, 83JOC3869, B-89MI 207-02>.



Scheme 78

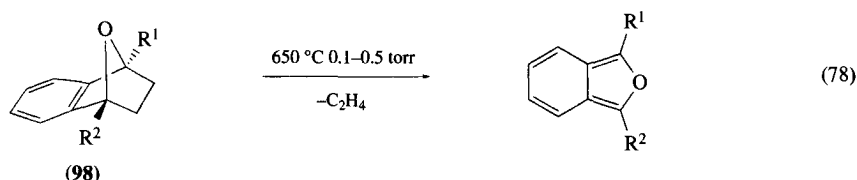
2.07.4.3 From Benzylidenephthalans

In solution there is an equilibrium between 1-benzylidenephthalan (**96**) and 1-benzylbenzo[*c*]furan (**97**) (Equation (77)). Although (**97**) cannot be detected either by NMR or by UV spectroscopy, it can be trapped with dienophiles, when heated in the presence of acids <74JOC3648, 74TL2603, 80JOC1817, 81JOC4083, 88JOC1841>.



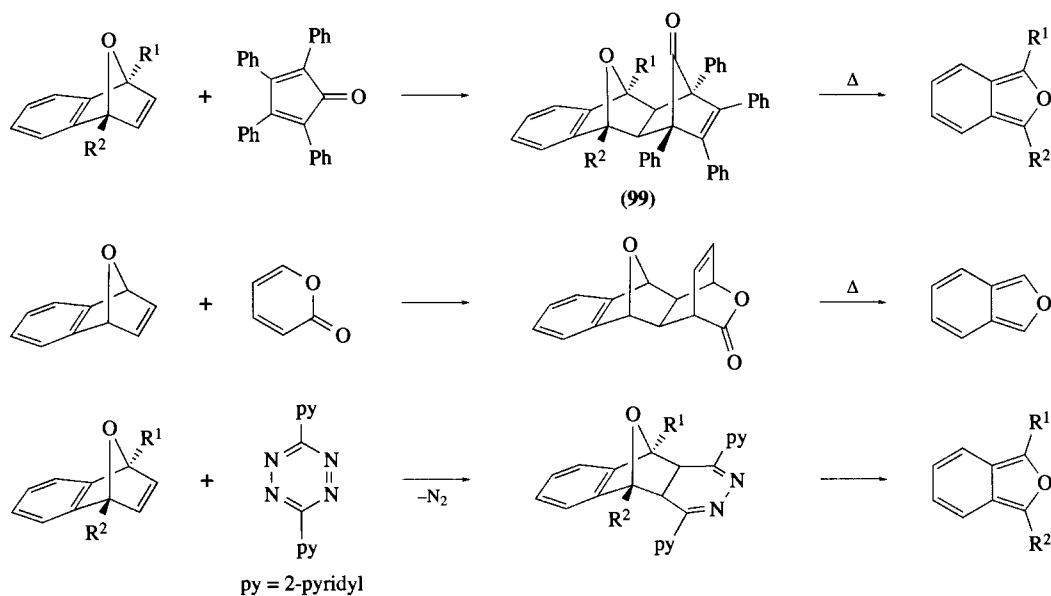
2.07.4.4 From Benzo-7-oxabicyclo[2.2.1]heptenes, Benzo-7-oxabicyclo[2.2.1]heptadienes, and Related Compounds

Flash vacuum pyrolysis of benzo-7-oxabicyclo[2.2.1]heptenes (**98**) yields benzo[*c*]furans (Equation (78)). This represents a versatile methodology for the preparation of the parent compound and other unstable 1-alkyl- and 1,3-dialkylbenzo[*c*]furans <71TL2337, 72CC347, 76TL2507, 80TL3831, B-81MI 207-01, 82RTC317, 82RTC365, 88AJC235>.

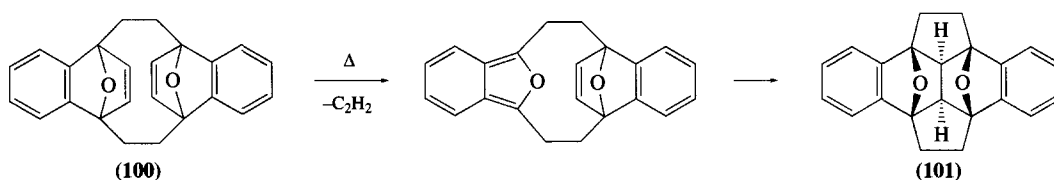


If benzo-7-oxabicyclo[2.2.1]heptadienes are used as starting materials, in an initial step a Diels–Alder reaction with an electron-deficient diene is performed to give 7-oxabicyclo[2.2.1]heptenes (e.g., (**99**)), which can be transformed by a retro Diels–Alder reaction to benzo[*c*]furans (Scheme 79) <64JA2081, 65CJC1599, 69TL5231, 71JA2346, 73CL335, 75BCJ1661, 76JCS(P1)1647, 76JCS(P2)81, 77JOC4170, 77TL3537, 81AJC1223, 81CC942, 82AJC757, 82CC1195, 84AJC1699, 84TL4833, 91AJC1275, 91TL1889, 92AJC1035, B-92MI 207-02>.

On heating compound (**100**), ethyne is eliminated to give (**101**) via a benzo[*c*]furan (Scheme 80) <82JA6125>.

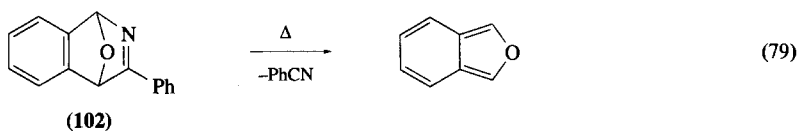


Scheme 79



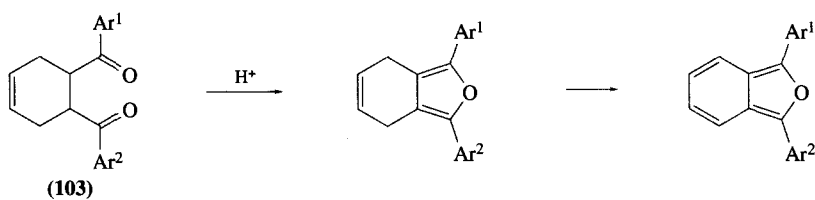
Scheme 80

A very convenient route to benzo[c]furan has been reported by Rickborn and co-workers. Heating (102) in benzene yields the parent compound, which can be trapped by dienophiles (Equation (79)) <88JOC5595>.



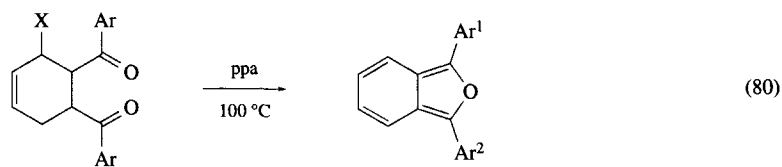
2.07.4.5 From 1,2-Diaroylcyclohexenes and 1,2-Diaroylcyclohexadienes

1,2-Diaroylcyclohexenes (103) are available by Diels-Alder reactions of butadiene with 1,2-diaroylethenes. Cyclization of (103) with acids (H_2SO_4 , H_3PO_4) <39JA2083, 40JA1233, 40JA2038, 43JA1283, 64T195> followed by dehydrogenation yields benzo[c]furans (Scheme 81) <49JCS1555, 65BSF2342, 74BSF2105, 75CR(C)961, 76CR(C)555, 78JHC793>.

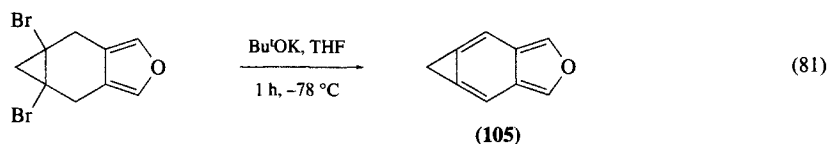
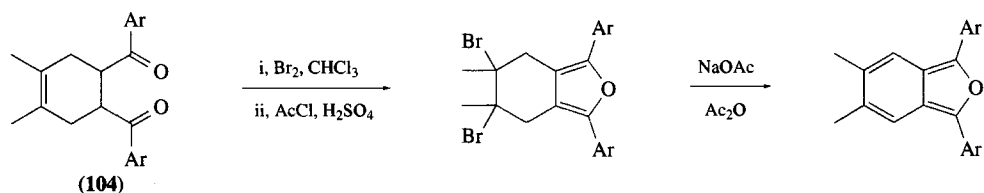


Scheme 81

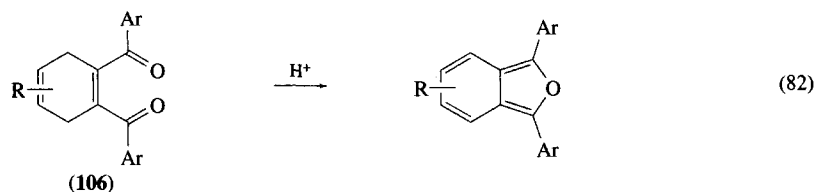
If a leaving group is present (Equation (80), $X = OSiR_3$, OR , $OCOR$, NR_2), the benzo[c]furans are obtained in one step <80S131, 81TL1697>.



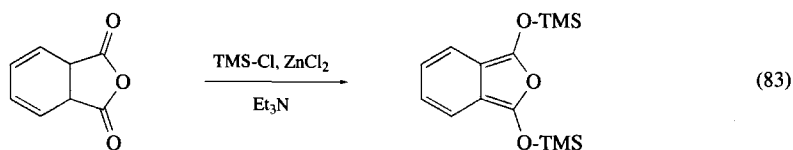
Treatment of diaroilcyclohexenes of type **(104)** with bromine and subsequent cyclization (MeC-OC₂H₅, H₂SO₄) and dehydrobromination (NaOAc/Ac₂O or other bases) yields benzo[*c*]furans (Scheme 82) <39JA2083, 40JA1233>. Cycloprop[*f*]benzo[*c*]furan (**(105)**) is prepared similarly (Equation (81)) <87TL4217>.



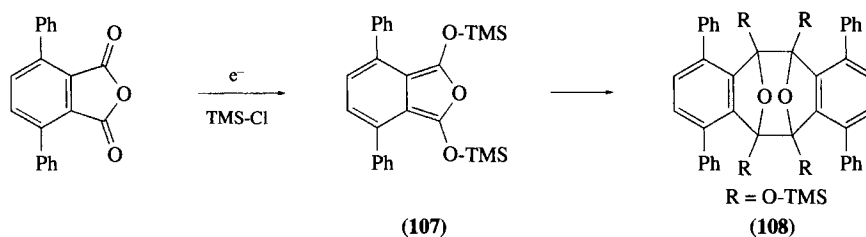
1,2-Diaroilylcyclohexadienes of type **(106)** can be converted to benzo[*c*]furans by treatment with acids (Equation (82)) <60LA(639)61, 71JOC1048, 76CZ291, 78JCR(M)4944, 78JCR(S)408>. A photochemical ring closure of **(106)** has also been reported <92JCR(S)372>.



1,3-Bis(trimethylsilyloxy)benzo[*c*]furan can be prepared from 1,2-dihydrophthalic anhydride by silylation (Equation (83)) <84TL2981>.



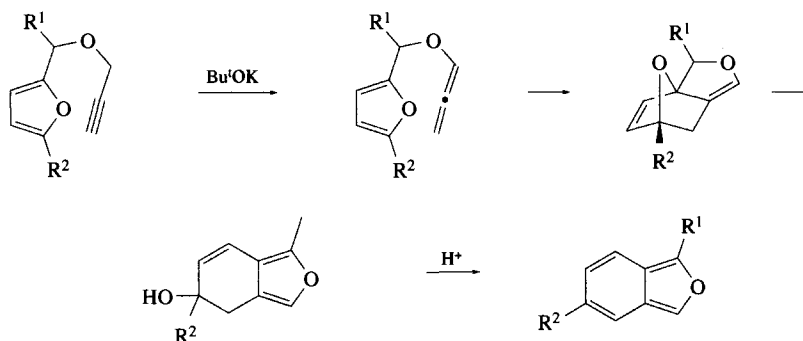
The electrochemical reduction of 3,6-diphenylphthalic anhydride in the presence of TMS-Cl yields benzo[*c*]furan (**(107)**), which dimerizes immediately to give **(108)** (Scheme 83) <81TL3497, 84AG596, 84AG(E)622>.



Scheme 83

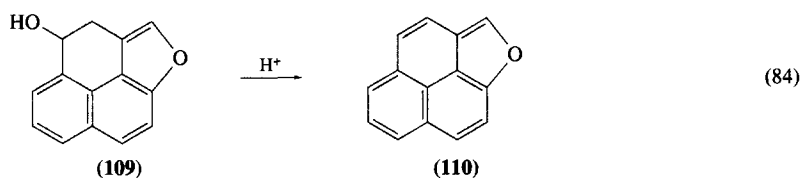
2.07.4.6 From Furans

Kanematsu and co-workers succeeded in preparing benzo[*c*]furans by an intramolecular Diels–Alder reaction of suitable furans (Scheme 84) <85TL2689, 87JOC2040, 89CC470>.

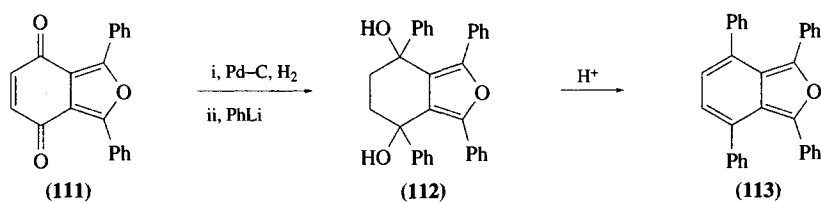


Scheme 84

Dehydration of (109) yields a phenanthro[1,9-*a-b,c*]furan (110) (Equation (84)) <88JCS(P1)3169>.



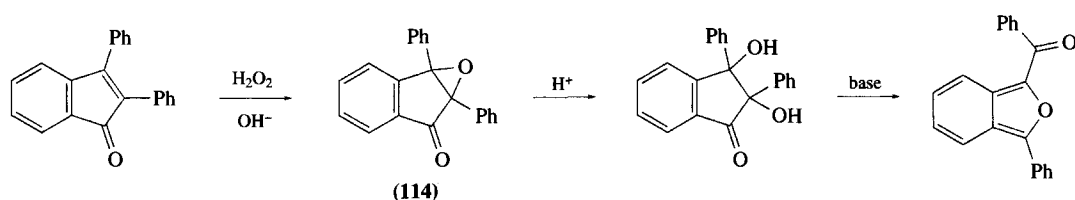
4,7-Dihydrobenzo[*c*]furan-4,7-diones (e.g., (111)) can be converted to benzo[*c*]furans. Catalytic reduction of (111) and treatment of the resulting product with phenyllithium yields a diol (112), which can be dehydrated to (113) (Scheme 85) <73BSF1154>.



Scheme 85

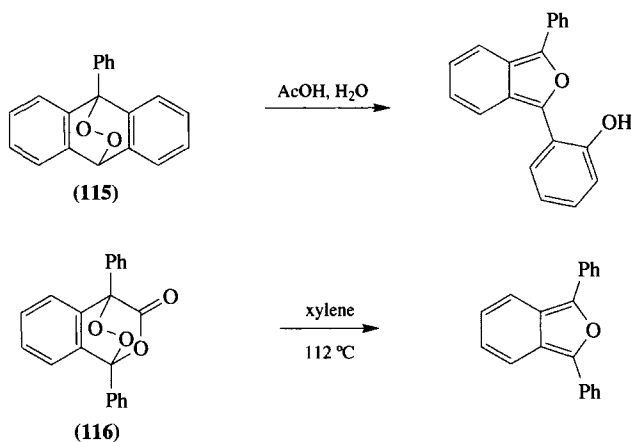
2.07.4.7 Miscellaneous Syntheses

1-Benzoyl-3-phenylbenzo[*c*]furan can be prepared by ring opening of epoxide (114) with acids and subsequent treatment of the resulting diol with base (Scheme 86) <48JA2069, 66JA4942>.



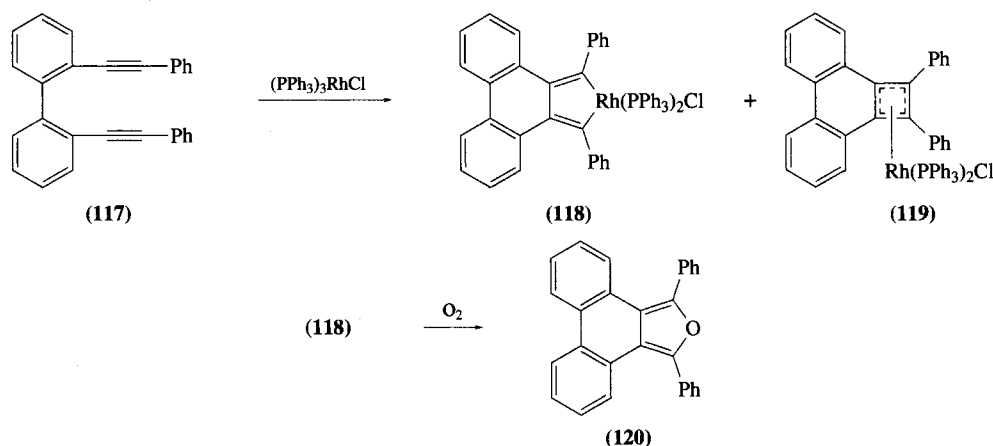
Scheme 86

Peroxides (**115**) and (**116**) either on treatment with acids or by thermolysis yield benzo[*c*]furans (Scheme 87) <74BSF2521, 82JA1041>.



Scheme 87

An interesting route for the preparation of a phenanthro[9,10-*c*]furan (**120**) has been described by Müller and co-workers. Treatment of the diyne (**117**) with $(\text{PPh}_3)_3\text{RhCl}$ yields complexes (**118**) and (**119**). The oxidation of (**118**) with oxygen gives (**120**) (Scheme 88) <71TL521, 72LA(758)16, 74S761>.



Scheme 88

2.07.5 DIHYDRO- AND TETRAHYDROFURANS

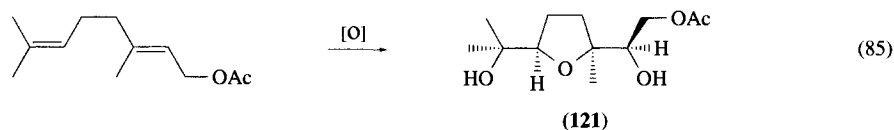
2.07.5.1 Introduction

Substituted dihydro- and tetrahydrofurans occur frequently as subunits of many biologically important natural products <B-82MI 207-01, 87T3309, B-88MI 207-01, 90T3321, 91COS(1)579, 92T8545>. Polyether antibiotics (monensin, ionomycin, nigericin, lasalocid A, etc.) in particular have attracted considerable attention because of their ability to transport metal ions across lipid bilayers. The construction of di- and tetrahydrofurans with well-defined stereochemistry remains a current challenge in organic synthesis <91JA9868, 92JOC6198, 93JOC809, 93TA1711, 93TL6251, 94TL2517>. In the following subsections progress in this field is described.

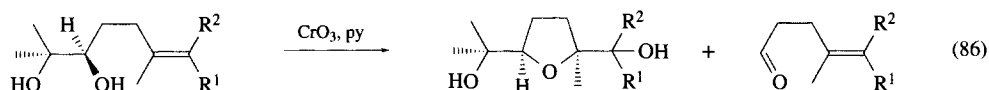
2.07.5.2 Ring Closure by Formation of the O—C(2) Bond

In 1965, Klein and Rojahn reported that 1,5-dienes are converted into tetrahydrofurans when treated with potassium permanganate <65T2353>. This oxidative cyclization leads specifically to *cis*-

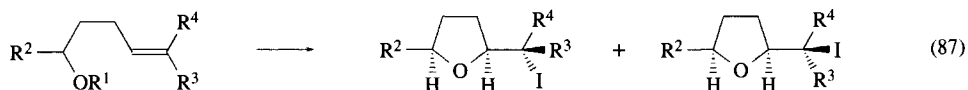
2,5-disubstituted heterocycles; for example, geranyl acetate yields (**121**) (Equation (85)). The reaction has been investigated in considerable detail <79CC918, 79JA4396, 80TL3531, 81JA940, 87T3309, 93TA1711>.



1,5-Dienes can also be cyclized in a mercuration–demercuration process <71OCS7>. In extension of this work it has been found that 5,6-dihydroxy alkenes can also be converted to *cis*-2,5-disubstituted tetrahydrofurans by treatment with Cr(VI) (Equation (86)); it was established that the presence of both hydroxy groups is essential <74MI 207-02, 82TL727, 88TL3171>.



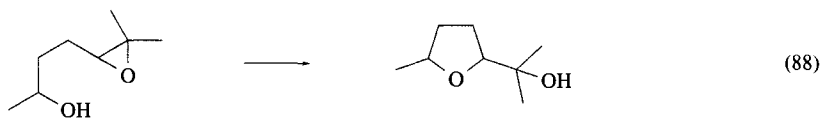
Halocyclization of γ,δ -unsaturated alcohols and the corresponding *O*-alkylated derivatives also gives tetrahydrofurans (Equation (87)) <81JA3963, B-84MI 207-02, 85TL1943, 87T3309, 92TL1747>.



γ -Hydroxyallenes may also react with iodine in the presence of potassium carbonate or sodium bicarbonate to deliver the corresponding tetrahydrofurans <92TL3969>.

An O—C(2) ring closure can also be effected by sulfenoetherification <87CC1280>; by phenylselenoetherification <85TL1333, 87CAR(171)1, 91JOC5245, 91T859, 93PHC(5)129>; by a mercuration reaction <74JHC771, 76JHC349, 78JHC225, 81TL2259, 87TL1023, 90JA5290>; oxidatively with Pd(OAc)₂/Cu(OAc)₂/O₂ <76TL1821>; with BuOOH/VO(acac)₂ <78JA2933, 78TL2741, 88CL1847, 90JOC5088, 91JA7613>, with MCPBA <91T4143>; by oxidative cyclization of 1,3-dioxolanes <84JA2641>; by reductive cyclization of γ,δ -ketoepoxides <88JOC1082>; and from 1,5-dienes after 1,3-dipolar cycloaddition of nitrile oxides followed by treatment with an electrophile (e.g., I₂, Br₂, PhSeBr) <90JOC283>. Iodo- and phenylselenocyclization is also possible with β,γ -unsaturated alcohols <92JA1084>.

γ -Hydroxyepoxides on treatment with acidic catalysts yield tetrahydrofurans (Equation (88)) <80JA7981, 85JA1691, 87JA1696, 87T3309, 87TL3075, 87TL5205, 88JOC1082, 89TL5799, 91JA9369>. This methodology has been found to be of value for the total synthesis of solamin and reticulatacin <94JCS(P1)1975>.

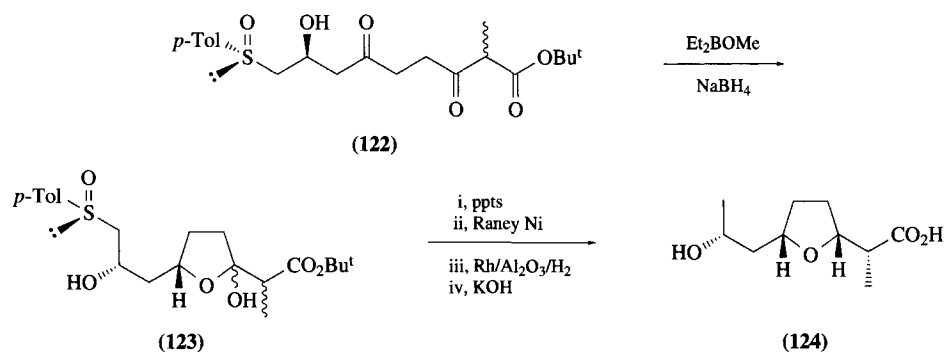


Cyclization of homoallylic alcohols possessing a sulfone group at the 3-position with base (KH, Bu^tOK) yields tetrahydrofurans <85TL4455, 92TL695>. The epoxidation–cyclization sequence can also be applied to β,γ -unsaturated alcohols <89TL5915>. Cyclization of appropriate 1,4-diols (or derivatives thereof) may also give tetrahydrofurans <74JCS(P1)1943, 75JCS(P1)1191, 80JA2118, 81JOC3361, 81S474, 88TL2927, 90SL423, 91T9179, 92CL195>.

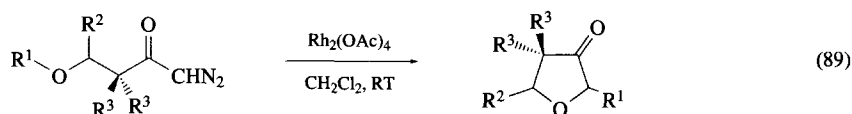
An enantioselective synthesis of (–)-(2*R*,3*R*,6*S*,8*R*)-*epi*-nonactic acid (**124**) is shown in Scheme 89. The reduction of (**122**) with diethylmethoxyborane/sodium borohydride yields a *syn*-diol (not isolated), which cyclizes spontaneously to the lactol (**123**). Pyridinium *p*-toluenesulfonate-catalyzed dehydration, desulfurization with Raney nickel, Rh/Al₂O₃-catalyzed hydrogenation, and hydrolysis of the ester group affords (**124**) <94JOC3898>.

Several other examples of the reduction–cyclization of γ -hydroxyketones have been reported <84JA5304, 87CAR(171)141, 89JOC610, 89T3139, 91HCA397, 92TL2557>.

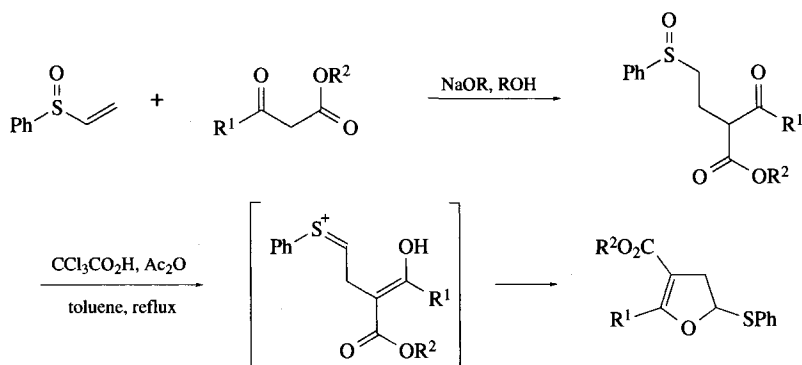
The rhodium acetate-catalyzed decomposition of 1-diazo-4-(benzyloxy)butan-2-one and related compounds offers a way to substituted tetrahydrofuranones; a Stevens [1,2]-shift of a cyclic oxonium ylide is involved in this reaction (Equation (89)) <92JOC3479>.



Scheme 89

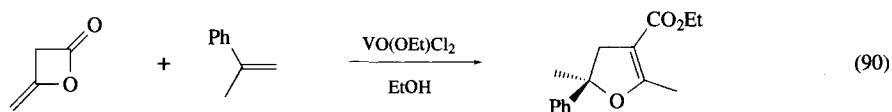


2,3-Dihydrofurans can be prepared efficiently by a Michael addition of β -ketoesters to alkenyl sulfoxides followed by a Pummerer rearrangement (Scheme 90) <92JCS(P1)945>.

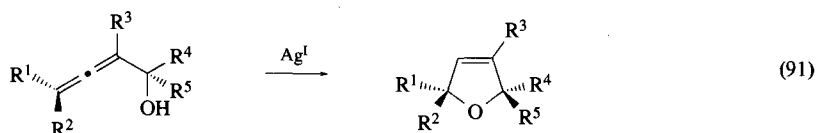


Scheme 90

Diketene undergoes a vanadium-induced reaction with styrenes yielding 3-alkoxycarbonyl-substituted 4,5-dihydrofurans <91JOM(407)C1>.



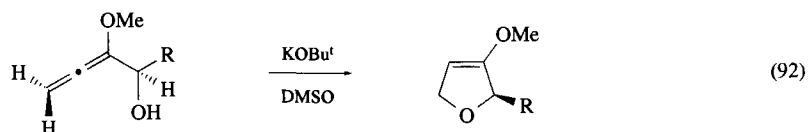
Dihydrofurans can also be prepared by a Ag(I)-catalyzed cyclization of allenols (Equation (91)) <74T289, 79S743, 84CC804, 90JOC2995, 94JOC324>.



Acid-, base-, and Hg(II)-catalyzed cyclizations of allenols and other compounds have also been reported <59AK475, 61CB948, 68BCJ1493, 69CR(C)241, 69RTC609, 69RTC845, 70CR(C)1808, 72BSF720, 72BSF1946, 90JOC2995>.

Base-induced cyclization of methoxyallene carbinols, which are available from lithiated methoxyallenes and carbonyl compounds, yields 2,5-dihydro-3-ethoxyfurans (Equation (92)) <69RTC609,

91SL179, 94JOC67). These compounds are valuable precursors for the preparation of 3(2*H*)-dihydrofuranones.



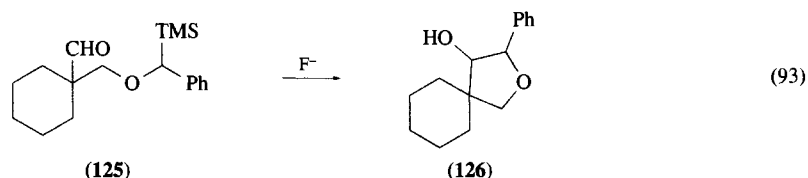
O-Silylated allenylcarbinols are readily cyclized to 2,3-disubstituted tetrahydrofurans (91SL869). Tetrahydrofurans are also obtained when γ -hydroxyketones are treated with silanes (89T3139).

2,3-Dihydrofurans are available from γ,δ -alkenylketones by cyclization with iodine or ppa (90H(31)1417, 90SL673).

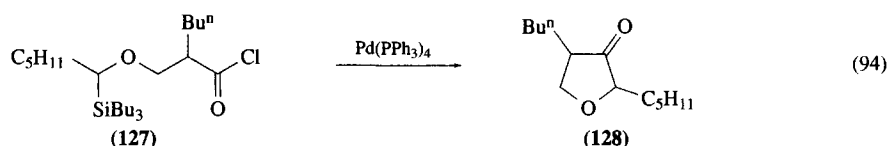
For further synthetic routes to tetrahydrofurans see (93TA1711).

2.07.5.3 Ring Closure by Formation of the C(2)—C(3) Bond

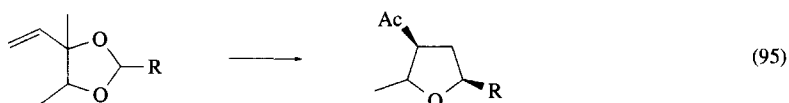
Several methods have been described for the preparation of tetrahydrofurans by a C(2)—C(3) ring-closure reaction. The fluoride-induced cyclization of (125) yields (126) (Equation (93)) (90JA7438).



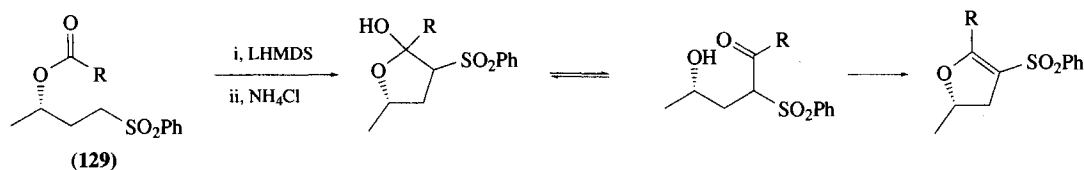
Treatment of (127) with tetrakis(triphenylphosphine)palladium(0) gives (128) (Equation (94)) (90JA7438).



The stannic chloride-catalyzed rearrangement of allyl acetals to tetrahydrofurans (Equation (95)) has been studied extensively (91JA5354, 91JA5365, 91JA5378).



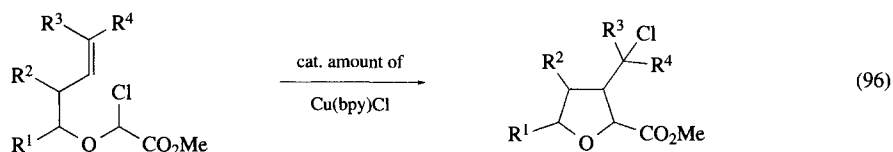
Deprotonation of sulfones of type (129) with lithium(hexamethyldisilyl) amide (LHMDS) and subsequent intramolecular acylation yields lactols, which are in equilibrium with the corresponding γ -ketocarbinals. Dehydration of this mixture with *p*-toluenesulfonic acid gives 2,3-dihydro-3-sulfonylated furans (Scheme 91) (94JOC2014).



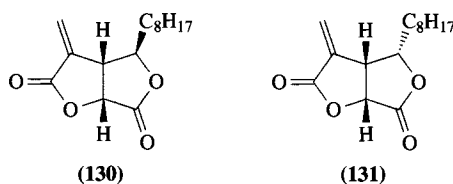
Scheme 91

The transition metal-catalyzed cyclization of 2-(3-alken-1-oxy)-2-chloroacetates gives good yields of 3-(1-chloroalkyl)-2-tetrahydrofuran carboxylic esters (Equation (96)). The stereochemical course

of this radical reaction shows a preference for the formation of 2,3-*cis*-substituted tetrahydrofurans <91TL3123, 92JCS(P2)1529, 93TA425, 94JOC1993>.



This method represents an improvement of the Bu_3SnH -mediated radical cyclizations of the corresponding phenylsulfides <91JA2335, 91TL1491>. Avenaciolide (**130**) and isoavenaciolide (**131**), two fungal antimetabolites from *Aspergillus avenaceus*, have been prepared by this route <94JOC1993>.



For references and reviews of radical cyclizations including atom-transfer methods see <B-86M1 207-01, 88S489, 91CRV1237, 91JOC2746, 91COS(4)715, 92JOC1682, 92T1637, 92T3945, 92TL5141, 93JOC464, 93JOC2360>.

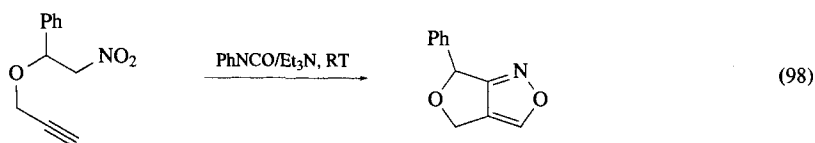
2.07.5.4 Ring Closure by Formation of the C(3)—C(4) Bond

2-Iodo- and 2-bromoethyl allyl ethers undergo a highly diastereoselective diethylzinc-mediated palladium-catalyzed ring closure producing *trans*-4,5-disubstituted lactols (Equation (97)) <94TL8349>. Related chromium(II)- and cobalt(I)-mediated ring closures have been reported <90SL44, 92JOC1740, 92SL943, 92TL2851, 93T4559>.

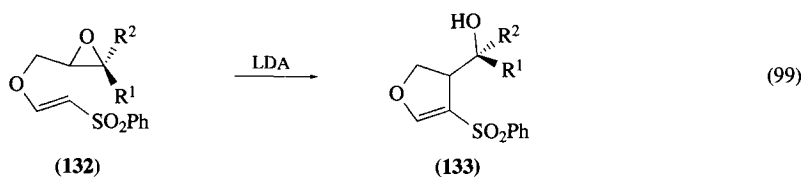


A radical promoted cyclization of the corresponding alkynyl ethers has also been described <87TL6393>.

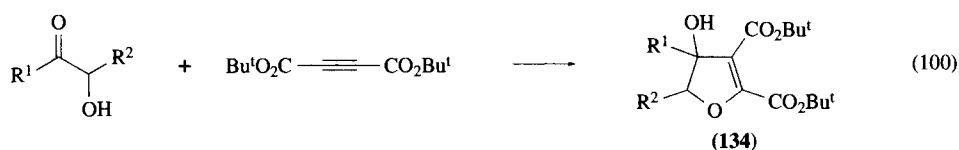
The intramolecular nitrile oxide–alkene cycloaddition reaction of propargylic ethers offers a route to dihydrofuroisoxazoles (Equation (98)) <94JOC3783>.



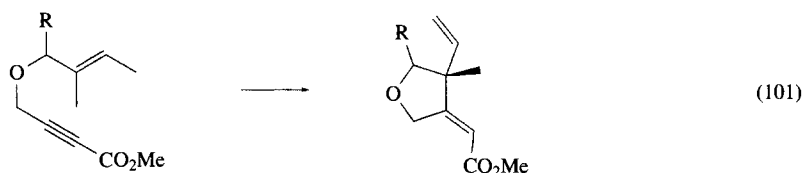
The conversion of (**132**) to (**133**) is initiated by a vinylic deprotonation (Equation (99)) <91JOC3556>.



The condensation of α -hydroxyketones with di-*t*-butyl ethyne dicarboxylate yields (Equation (100)) (**134**) <91TL4687>.



The intramolecular ene-reaction of allyl propargyl ethers gives tetrahydrofurans (Equation (101)) <87CL2347>.



2.07.5.5 Ring Contraction of Tetrahydropyrans

The formation of tetrahydrofurans by ring contraction of tetrahydropyrans offers an attractive route to tetrahydrofurans. This methodology has been investigated extensively by Bartlett and co-workers. In an initial step a γ,δ -unsaturated alcohol is treated with 2,4,4,6-tetrabromo-2,5-cyclohexadienone (TABCO), yielding a bromotetrahydropyran. The ring contraction is induced by silver tetrafluoroborate in acetone (Scheme 92) <84JA2668>. It is possible to improve this reaction sequence by using Tl(III) ions as electrophiles <85JOC2416>.

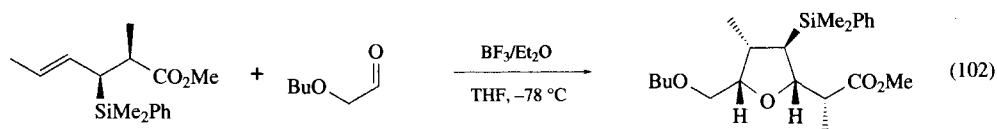


Scheme 92

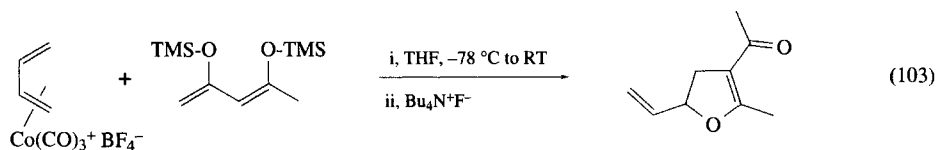
2.07.5.6 Miscellaneous Syntheses

It is well known that di- and tetrahydrofurans can be obtained by inter- and intramolecular 1,3-dipolar cycloaddition reactions of carbonyl ylides with alkynes and alkenes. This methodology has also been shown to be of value in the preparation of a furanophane <92TL57>.

The addition of chiral (*E*)-crotylsilane to α - (and β -) alkoxy aldehydes provides a convenient route for the diastereoselective synthesis of tetrasubstituted tetrahydrofurans (Equation (102)) <91JA9868>.

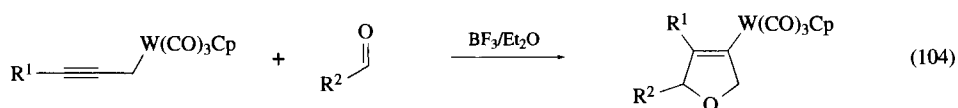


Dihydrofurans are also available by the addition of enolate dianion derivatives to (μ^4 -diene) $\text{Co}(\text{CO})_3$ tetrafluoroborates (Equation (103)) <88JOC2114>.

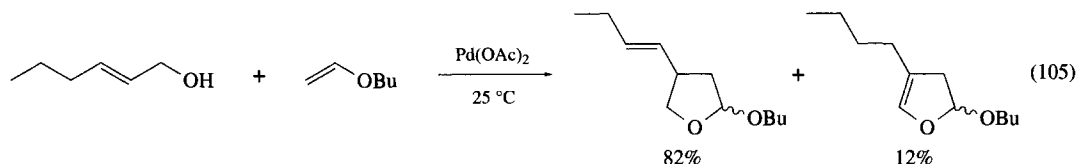


4-Acyldihydrofurans have been prepared in variable yields by an oxidative coupling of β -dicarbonyl compounds with alkenes and dienes <82IZV2077, 85BCJ1922>.

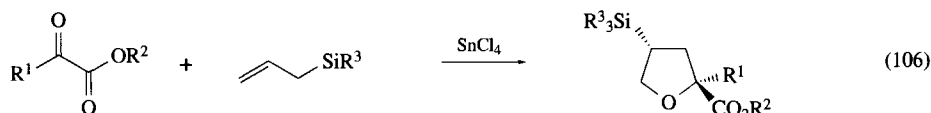
Dihydrofurans can be prepared in good yields (>80%) by the reaction of tungsten μ^1 -propynyl compounds with aldehydes in the presence of BF_3 etherate in cold toluene (-40°C) (Equation (104)) <92CC934>.



Reaction of allylic alcohols with vinyl ethers in the presence of palladium(II) acetate affords dihydro- and tetrahydrofurans (Equation (105)). Yields are moderate to good <89BCJ2050>.

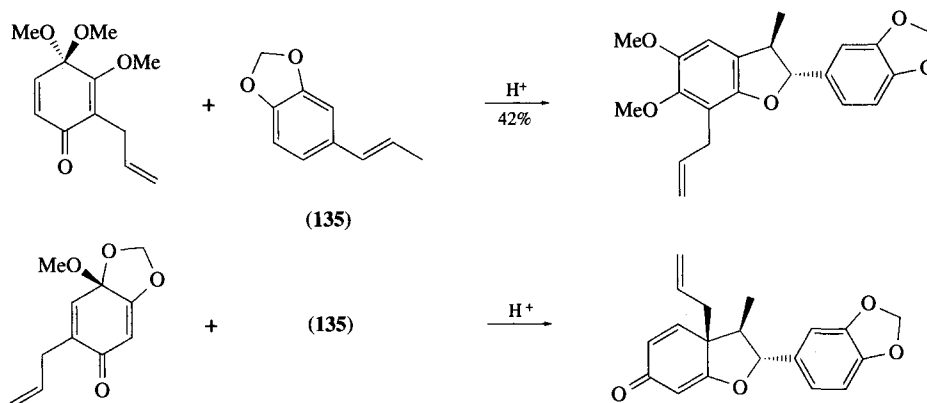


The tin(IV) chloride-promoted [3 + 2] cycloaddition of allylsilanes and α -ketoesters provides a versatile diastereoselective route to silyl-substituted tetrahydrofurans via 1,2-silyl migration (Equation (106)) <94CL627, 94TL8401>.



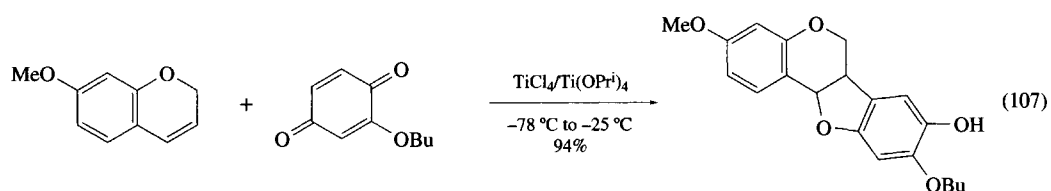
2.07.6 DIHYDROBENZOFURANS

For the preparation of 2,3-dihydrobenzo[*b*]furans, several strategies have been developed. The acid-catalyzed cycloaddition of styrenes (e.g., (135)) with quinone monoketals is suitable for the synthesis of both 2,3-dihydrobenzofurans and the quinonoid derivatives (Scheme 93) <77JA8073, 78JOC3717, 83TL5011, 88JCS(P1)2305>.

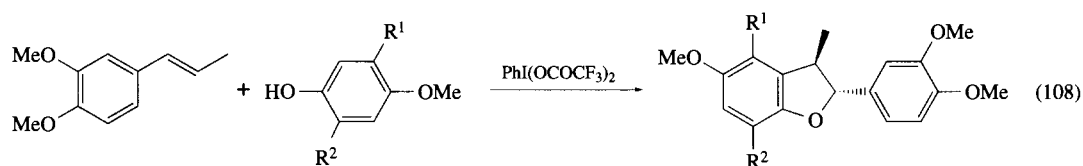


Scheme 93

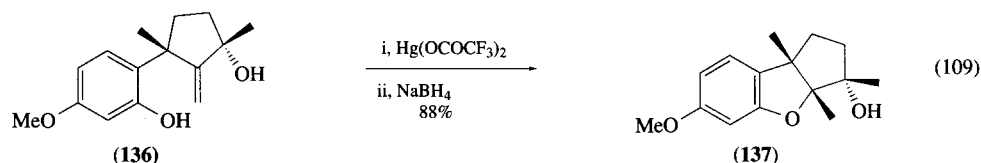
The Lewis acid-catalyzed addition of benzoquinones with styrene derivatives yields dihydrobenzofurans (Equation (107)). This method has also been used for the preparation of pterocarpanes <88JA7931, 89CC1573, 90JOC1248, 90JOC5810>. [2 + 2] Cycloadducts may appear as by-products.



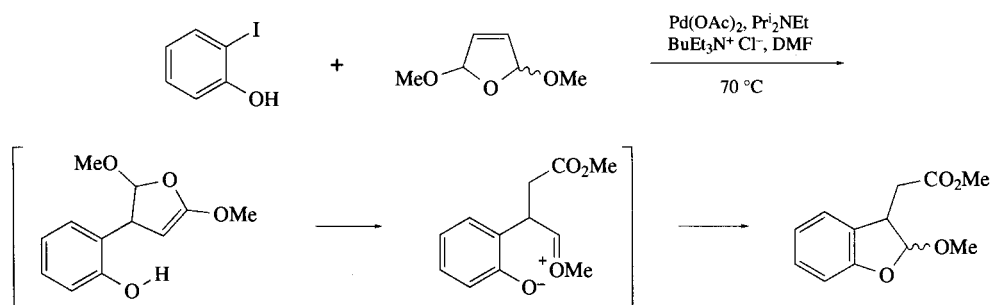
Trans-2,3-dihydrobenzofurans are obtained stereoselectively when *p*-methoxy-substituted phenols are oxidized with iodobenzene bis(trifluoroacetate) in the presence of electron-rich styrene derivatives (Equation (108)) <91JOC1979>.



The cyclization of (136) to (137) can be effected by an oxymercuration–reduction procedure (Equation (109)) <94JOC74>.

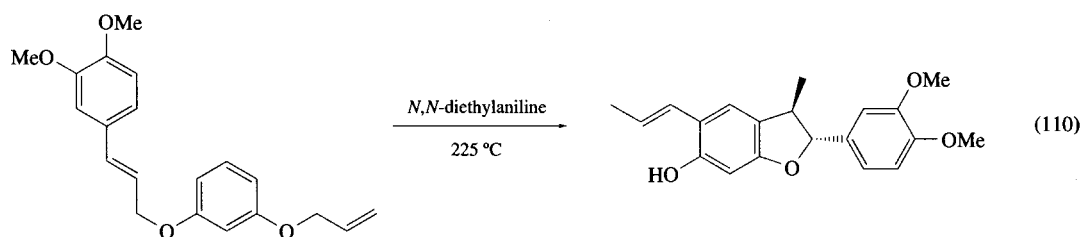


The Heck reaction <85-BMI 207-02> of iodophenol with 2,5-dihydro-2,5-dimethoxyfuran yields a 2,3-dihydrobenzofuran (Scheme 94) <94H(38)1745>.



Scheme 94

It has been reported that 2,3-dihydrobenzofurans are obtained by an abnormal <72HCA1625> Claisen rearrangement (Equation (110)) <86TL309>.



2.08

Furans and their Benzo Derivatives: Applications

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2.08.1 INTRODUCTION

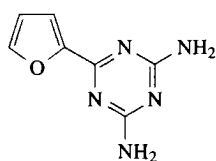
This chapter describes various applications of furan and benzofuran derivatives since 1982. The subject matter concentrates on the actual use of molecules that contain one or more of the following ring systems: a furan ring, dihydro- and tetrahydro furan ring, 2(5*H*)- and 3(2*H*)-furanones, five-membered anhydrides, benzofurans, isobenzofurans, dibenzofurans, benzofuranones, benzoanhydrides, polyarylfurans (and derivatives therefrom), and their hydro derivatives. This chapter is not an exhaustive review of the literature since 1982 due to the large number of references found on this topic and the page limitation imposed upon this chapter. Thus, what appears is a selection. There were too many references to simple compounds like tetrahydrofuran, maleic anhydride, succinic

anhydride, and phthalic anhydride, so for the most part, they are not discussed in detail. The reader is referred to *Chemical Abstracts* for references related to these compounds. It was very difficult to distinguish whether compounds described in patents had an actual application or were being listed as the subject of claim. Therefore, molecules whose claim of application would be of general interest have been included. Again, due to space restrictions biologically active molecules isolated from natural sources have not been included.

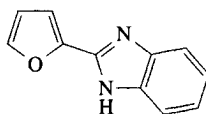
2.08.2 FURAN DERIVATIVES

2.08.2.1 Agrochemical Bioregulators

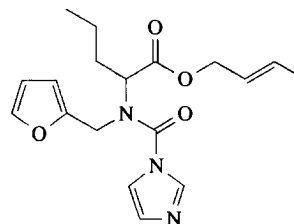
Some monosubstituted aryl and alkyl furans have exhibited beneficial properties. Furyl triazine (1) was shown to have detrimental effects on larval survival when introduced into adult female house flies, *Musca domestica* <85MI 208-01>. It was found that, although females deposit eggs and the eggs hatch, the larvae fail to pupate. The results were dependent upon when the eggs were treated with (1) and the concentration injected. The distribution, elimination, and metabolism of ¹⁴C-labeled (1) has also been studied on the common house fly <86MI 208-01>. Ring opening of the furan moiety to yield a saturated dihydroxy derivative appears to be the major metabolic pathway. Furyl triazine (1) suppressed the population of *T. granarium* <91MI 208-01>. Larvas treated with (1) provided adults with small legs and incomplete wing pads. The solubility of the widely used fungicide fuberidazole (2) in water-surfactant systems was measured as a function of pH to help determine how it degrades in the environment. <92MI 208-01> Finally, a mixture of furan (3), the salt of kasugamycin, white carbon, lauryl sulfate, calcium ligninsulfonate, and clay (20 : 1 : 20 : 3 : 2 : 54 wt. parts) were shown to protect rice seeds from infection by *Gibberella fujikuroi* and *Pseudomonas glumae* <91JAP(K)03133907>.



(1)



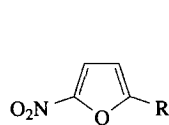
(2)



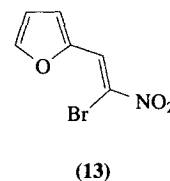
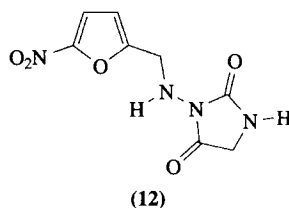
(3)

A variety of 2-nitrofuran derivatives have shown interesting activity. 2-Nitrofuran (4) has been used to control barnacles and mussels at levels as little as 0.1 ppm <83JAP(K)58015902>. Venters *et al.* has tested a variety of 5-substituted-2-nitrofuran derivatives <90MI 208-01>. Thus, furan (5) was shown to be an effective agent against houseflies and rice weevils, nitrile (6) was active against bean spider mites, carbonitrile (7) was a plant growth regulator and herbicide, and compounds (8) and (9) were fungicides. In addition, 40 other derivatives of 5-nitrofuran have been prepared and assayed for their fungicidal activity <90MI 208-02>. Silyl-containing amides of acid (10) showed the best success against *Phytophthora* on tomato and gray rot of beans. Furan (11) gave >90% inhibition of five species of fungi; a spray of as little as 0.3% gave good control of blue mold of tobacco in pot experiments <85MI 208-02>. Furdantin (12) has been shown to act as a chemosterilant on the lesser bandicoot rat <82MI 208-01>. An improved synthesis of 2-(2-bromo-2-nitroethenyl)furan (13) has been reported, since it has been shown to be active against a wide variety of microbes <89MIP8911793>. Synergistic effects have been noted when furan (13) is mixed with other compounds. Thus, a mixture of (13) with 2-bromo-2-nitropropane-1,3-diol is an effective industrial microbicide and algicide <91USP5045104>, while with glutaraldehyde or didecyltrimethylammonium chloride the mixture controls the bacteria *K. pneumoniae* in aqueous systems, such as cooling waters and in paper manufacturing <92USP5158972, 92USP5158973>.

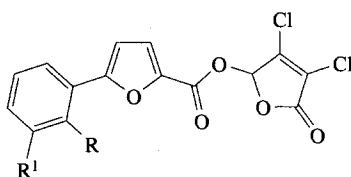
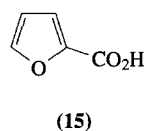
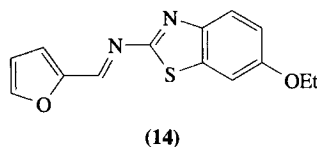
Derivatives of furan-2-carbaldehyde and 2- and 3-furoic acid have shown interesting properties. The copper complex of compound (14) possesses herbicidal activity <89MI 208-01>. The complex was more active than the parent compound (14). 2-Furoic acid (15) increased hypoxic stress resistance in wheat, thus increasing the yield and quality of the crop <92JAP(K)04342507>. The furyl furanones (16)–(18) <87CZP246434> and indanylfuran (19) <87JAP(K)62249966> have shown promise as antifungal agents. The latter exhibited 100% control of *Rhizoctonia solani* at 12.5–100 ppm. Furan (20) is a



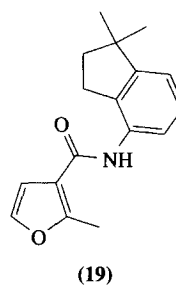
- (4) R = H
 (5) R = CH=CH-CF₃ (E)
 (6) R = CH=CH-CN (E)
 (7) R = CN
 (8) R = CH=CH-CHO (E)
 (9) R = CH=NOH (E)
 (10) R = CH=CH-CO₂H (E)
 (11) R = CH=C(CN)-CO₂Et (E)



seed-disinfectant fungicide when mixed with *N*-methyl-2,3-dichloromaleimide <85EGP218261>. A formulation of 30% maleimide and 10% (20) applied as a seed dressing, at 4 g kg⁻¹, synergistically controlled *H. gramineum* on barley.



- (16) R = H, R¹ = NO₂
 (17) R = Cl, R¹ = H
 (18) R = R¹ = H

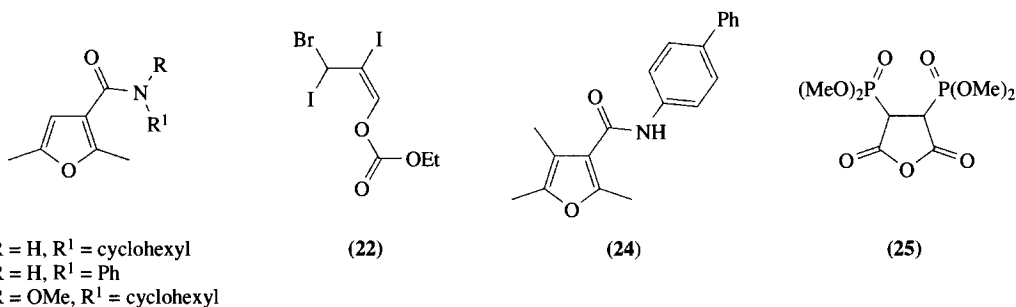


Furan (21) when mixed with Sanplas (22) acted as a wood preservative and inhibited the growth of *Coriolus* <93JAP(K)05112406>.

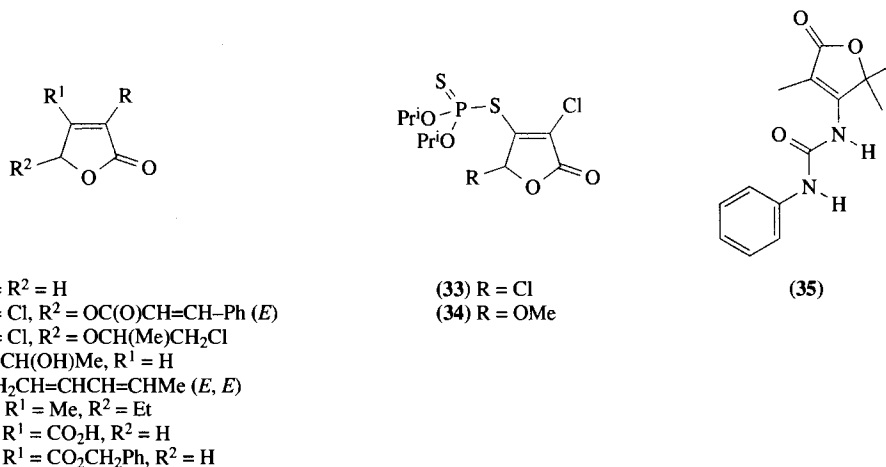
Compound (23) has exhibited some very interesting synergistic effects. Mixtures of (23) with 1,1,1,2,6,7,7,7-octachloro-4-oxaheptane when added to plywood adhesives were effective in controlling termites <87JAP(K)62016402>. Mixing (23) with isopropargyl derivatives <86JAP(K)61091109> or 2-iodo-*N*-phenylbenzamide <84GEP(O)3316007> provided good fungicidal wood preservatives. Adding phenyl salicylate to (23), when used as a wood preservative, retarded photodegradation <86JAP(K)61060676>. Finally, structure-activity relationships of a variety of mono-, di-, and trimethyl-furan carboxamides have been tested against the succinate dehydrogenase complex (SDC) activity in mitochondria isolated from sporidia of a wild-type strain and a moderately carboxin-resistant mutant strain of *U. maydis* (corn smut). Furan (24) had the highest affinity for the mutant SDC <88MI 208-01>.

Maleic anhydride was the active ingredient in a synergistic microbicide; 15 mg ml⁻¹ of maleic anhydride controlled the growth of slime in paper manufacturing <89JAP(K)01221302>. In addition, industrial microbicides containing both maleic acid and maleic anhydride have been useful for controlling microbial growth in water and prevented slime formation in textile lubricating oils, paints, adhesives, latex, glues, leather, lumber, paper goods, bamboo, and in paper manufacturing <89JAP(K)01050803>. The dimethylphosphono derivative (25) is an excellent insecticide. *Musca domestica* was completely killed when exposed to a film of 0.01% of (25) <87EGP246023>.

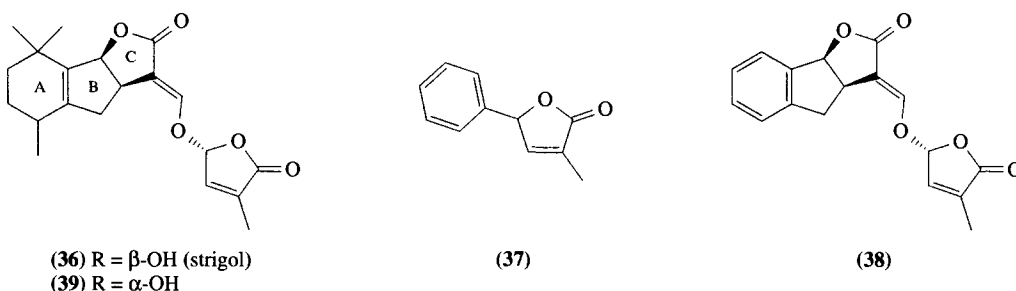
A number of furanones have been reported to exhibit interesting properties. Many papers have been published describing the ability of 2(5*H*)-furanone (26) to stimulate seedling vigor, enhance root and foliage growth, and increase the grain yield of corn <83MI 208-01, 88MI 208-02, 89MI 208-02,



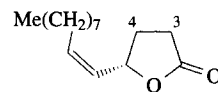
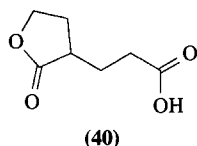
90MI 208-03, 91MI 208-02). Two 3,4-dichloro derivatives of 2(5*H*)-furanone have been used as fungicides with seeds. Compound (27) compared favorably with the fungicide Vitavax <88CZP248215>, while (28) was successful with rye seeds <88CZP254669>. Furanone (29) was shown to be secreted by *Trichoderma harzianum*, which inhibits the growth of microorganisms <92P485>. Compound (30) was a powerful fly attractant <81JAP(K)81133201> and furanones (31) and (32) showed inhibition of the microbe *Pyricularia oryzae* <90JAP(K)02255669, 91JAP(K)03099058>. The dithiophosphate derivatives (33) and (34) are aphicides <90URP1578133>, while the methylated derivative (35) (and others) have been shown to be good herbicides <88JAP(K)63174983>.



A series of analogues of strigol (36), a potent weed seed germination regulator, have been prepared and tested. It has been shown that the A, B, and C rings of strigol are not necessary for germination stimulation of *Striga* and *Orobancha* seeds. This is illustrated by the simple compound (37) exhibiting almost the same activity as the complex molecule (38) <92MI 208-02>. Pepperman and Cutler have prepared a variety of 5-alkoxy-3-methyl-2(5*H*)-furanones and screened them for plant growth regulating activity. Interestingly, strigol did not inhibit the growth of etiolated wheat coleoptiles, but epistrigol (39) did at 1 mM concentrations <91MI 208-03>. 5-Hydroxy-3-methyl-2(5*H*)-furanone was shown to stimulate the germination of dormant cheat and nondormant perennial ryegrass and sorghum seeds, while 5-allyloxy-3-methyl-2(5*H*)-furanone showed a similar activity against witchweed as strigol did. In general, the 5-alkoxy-3-methyl derivatives of butenolide were less effective in stimulating seed germination than the multiring systems <88MI 208-03>.

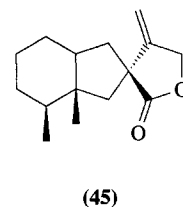
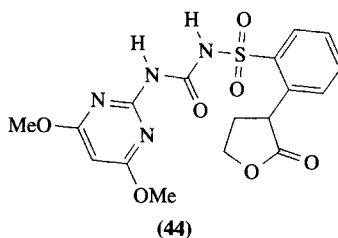
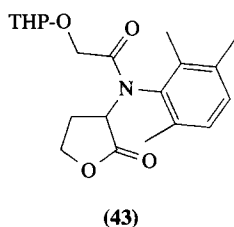


A variety of tetrahydro-2-oxofurans have exhibited beneficial properties. Lettuce seeds when treated with acid (40) showed almost no germination <91JAP(K)03251505>. A combination of the sex attractant lactone (41) with a PEP–eugenol mixture caught significantly more *Popillia japonica* than either alone. More male beetles were trapped during the period of heavy adult emergence <81MI 208-01>. The structure of (41) is similar to the female sex pheromone (42) which has an additional double bond in the C(3)—C(4) position. The latter has been used to assess the population of the Japanese beetle (*J. japonica*) in the Azores <88MI 208-04>.

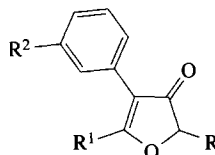


(42) Double bond at C3–C4

Compound (43) has been shown to possess antifeedant activity against *Phytophthora infestans* on tomato plants <85MIP544086>, while (44) controlled nut sedge (*Cyperus rotundus*) and barnyard grass (*Echinochloa crus-galli*) <86EUP178101>. Bakkenolide A (45) showed very good antifeedant activity against a variety of insects <84MI 208-01>.



A variety of 3(2*H*)-furanone derivatives have been synthesized and tested for activity. Sandmann and Boeger have performed structure–activity studies on 26 derivatives of (46) for inhibition of formation of colored carotenoids in cultures of *Synechococcus*. The most active compound was found to have structure (47) <93ZN(C)312>. Other groups over the past ten years have been studying various analogues of compound (46) for their herbicidal activity and a few patents have been granted. Compounds (47)–(52) have shown good herbicidal activity. Compound (47) controlled spurred anoda and prickly sida and partially controlled yellow foxtail with no phytotoxicity to peanuts and cotton <93USP5180415>. Compounds (48) and (51) were active against crabgrass, wild oats, lambsquarter, and mustard <86USP4568375, 86USP4568377>, while mustard and pigweed was controlled by (49) <86USP4568378>. Furanones (50) and (52) were active against a variety of common weeds <85USP4537623, 87MI 208-01>. In addition, (52) possesses potent bleaching activity and is postulated to act through inhibition of carotenoid biosynthesis. Peanuts and sorghum are tolerant to postemergence applications, while potatoes, barley, peas, safflower, and cucumbers show some degree of tolerance to (52).



(46) R = Ph, R¹ = NHEt, R² = CF₃

(47) R = Ph, R¹ = NHMe, R² = CF₃

(48) R = OEt, R¹ = NMe₂, R² = CF₃

(49) R = Ph, R¹ = NH-cyclopropyl, R² = Ph

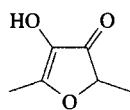
(50) R = 2-thienyl, R¹ = NH₂, R² = CF₃

(51) R = Ph, R¹ = NHMe, R² = CO₂Et

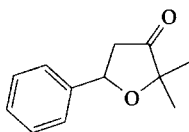
(52) R = Ph, R¹ = CH₂NH₂, R² = CF₃

Compound (53) acts as a cockroach attractant. Baits containing (53), food and insecticide were tested on a variety of common cockroaches. This mixture showed more activity than the control bait (COMBAT) <89EUP319757>. On the other hand, compound (54) acted as a cockroach repellent. It was more active than eugenol, and the effect lasted for greater than five days <89JAP(K)01242503>.

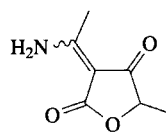
Finally, the 2,4-dione (**55**) controlled a variety of perennial weeds including broadleaved perennials <90EUP351020>.



(53)

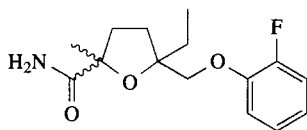


(54)

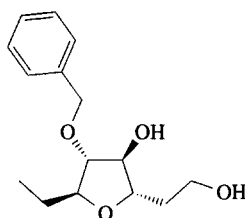


(55)

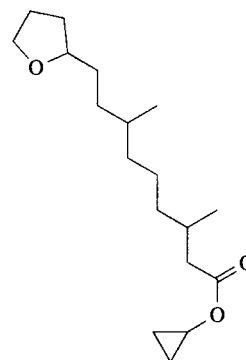
A few tetrahydrofuran-containing molecules have exhibited useful properties. Compounds (**56**) and (**57**) have shown herbicidal activity against a number of common weeds such as barnyard grass, Johnson grass, yellow foxtail, yellow millet, and blackgrass <86USP4594094, 88EUP264978>. Tetrahydrofuran (**58**) has shown insecticidal activity when applied to eggs and pupae of a variety of insects <88MI 208-05>. The tetrahydrofuranyl ether of 8-hydroxymenthyl is a long-lasting insect repellent. When applied to the arms of humans, mosquitoes were repelled even six hours later. This was 55% better than the control compound (**60**) <92JAP(K)04210685>. Compound (**59**) may also be useful as an antifouling agent since it was shown to repel mollusks.



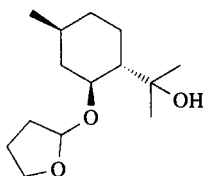
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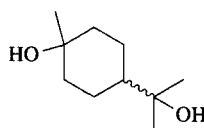
(57)



(58)



(59)

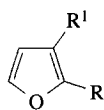


(60)

2.08.2.2 Fermentation and Bioindustrial Chemistry

A number of furan derivatives have been used or detected in the fermentation and bioindustrial area. A nutrient substrate for growing yeast has been prepared by hydrolyzing plant raw material and steam distilling the remains to provide a furfural (**61**)-containing condensate, which was subsequently treated with ozone, and a hydrolysate. Mixing this ozone treated furfural condensate with the hydrolysate increased the yield of the yeast. <90URP1558983>. Furfural has also been degraded to methane and carbon dioxide under anaerobic conditions. Methane was produced at a 80% yield of the theoretical expected biogas when furfural was continuously added to a continuously stirred tank reactor containing the biomass pretreatment liquor <91MI 208-04>. 2-Acetoxyfuran (**62**) has been isolated from the volatiles of various invert sugars. It is suggested that the detection of (**62**) in wines could be used as a measure of whether inverted sucrose solutions have been added to wine <83MI 208-02>. Disulfide (**63**), which has a burnt odour, was reduced in whisky (from grain spirit) by passing oxygen through the solution. Bubbling nitrogen gas increased the concentration of disulfide (**63**). The reduced burnt smell was confirmed by a nosing panel <88BRP2204878>. Furans (**64**)–(**67**)

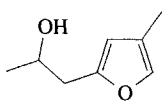
were produced by fermentation of *Streptomyces* strains DSM 4349, 4355, 4200, and 4211 and were found to be active against *staphyl*- and *streptococci* <89EUP337152>.



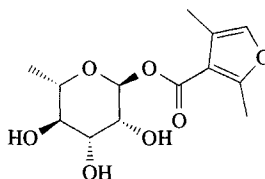
(61) R = CHO, R¹ = H

(62) R = OAc, R¹ = H

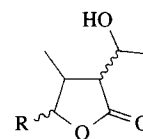
(63) R = Me, R¹ = SMe



(64)



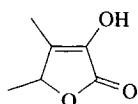
(65)



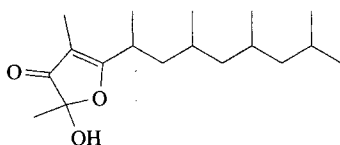
(66) R = OAc

(67) R = H

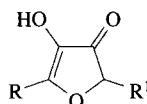
Sotolone (68) is the key substance in the aroma of sugar cane and was detected in botrytized wine. When sotolone was added to normal wine, a sweet, honey-like flavour was detected <84ABC2707>. Wine tasters have also associated the presence of sotolone (68) with an aftertaste of a nutty flavour <92MI 208-03>. Furanone (69) was produced by a shake-culture of *Scedosporium* sp. SPC-15549 and shown to inhibit acyl CoA-cholesterol acyltransferase with an ID₅₀ of 0.29 µg ml⁻¹ <92JAP(K)04356475>. Furanones (70) and (71) have been found to be useful as taste and flavour enhancers for food. The yeast *Zygosaccharomyces rouxii* ATCC 13356, when cultured in a medium containing sucrose, salts, the sodium salt of D-xylulose 5-phosphate, and the barium salt of D-ribose 5-phosphate produced a mixture of furanones (70) and (71) <91JAP(K)03183490>. Finally, it has been reported that *Bacillus thermolysin*, a metallo-protease which is used in the preparation of peptide sweeteners, is acylated with maleic anhydride (72) to improve its heat stability <90JAP(K)02286084>.



(68)

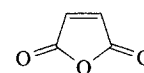


(69)



(70) R = Me, R¹ = Et

(71) R = Et, R¹ = Me



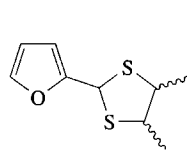
(72)

2.08.2.3 Food and Feed Chemistry

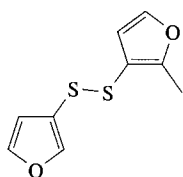
A number of furan derivatives have been used in the food and feed industry, mainly as taste enhancers. Three reviews have appeared in the literature since 1982 on the use of furans in this area. The first two deal with specifications, safety data, and so on, of furanones and sulfur containing furans, respectively, used in flavouring materials <83MI 208-03, 84MI 208-02>. The third discusses the use of furan derivatives as fragrant additives in food products <87MI 208-02>. Furfural (61) has been shown to be a major component of the furans essential for the formation of important coffee aroma constituents <89MI 208-03>. In addition, when furfural is mixed with aspartame, the mixture tastes like sucrose <90JAP(K)02276553>.

Dithiolane (73) at a concentration of 2.5 ppm deepened the chicken taste and added a light onion aftertaste when added to chicken broth <85USP4515968>, while disulfide (74) exhibited a meaty flavor <90MI 208-04>. 2-Undecylfuran (75) has been detected in tonka beans and it has been proposed that furan (75) be used as an indicator for the detection of illegal tonka bean products in food <91MI 208-05>. Furanone (53) has been detected as a volatile component in strawberries, pineapples, grapes, and wines. It has also been used a food additive, in racemic form, since it has a strawberry flavor <83MI 208-04>; however, furan (53) has been reported to induce micronuclei chromatid exchanges in germ cells of male mice and therefore may not be a safe food additive <92MI 208-04>. Furanone (76) has also been isolated from strawberries, and only the racemic form possesses the taste of strawberries. The individual enantiomers have different odor and sensory characteristics <92MI 208-05>.

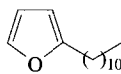
Furanones (70) and (71) have been used as artificial sweeteners in beverages <86JAP(K)61035761> as a sweetener enhancer with sucrose <92JAP(K)04008264>. Furanones (77) helped remove the unpleasant odor of goat milk, while not reducing the nutritional value of the milk <86MI 208-02>. Finally, some five-membered lactones have been detected or used in the food and feed industry. Lactone (78), which imparts a caramel-like sweet odor, has been detected in nonfermented sausages



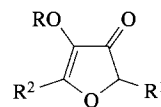
(73)



(74)

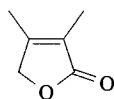


(75)

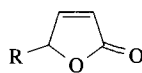


- (53) R = H, R¹ = R² = Me
 (76) R = R¹ = R² = Me
 (70) R = H, R¹ = Me, R² = Et
 (71) R = H, R¹ = Et, R² = Me
 (77) R = H, R¹ = C₁₋₆, R² = H, Me, or Et

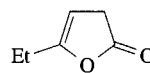
stored at 10°C <93MI 208-01>. Lactones (79)–(81) inhibit the growth of food microorganisms such as *Salmonella infantis*. Thus, 0.1% of (79) inhibited the growth of salmonella in a trypticase soy broth at pH 5 (37°C) <83EUP72202>.



(78)



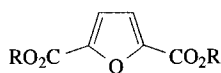
- (79) R = CH=CH₂
 (80) R = Et



(81)

2.08.2.4 Textiles and Fibers

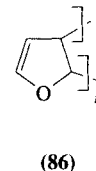
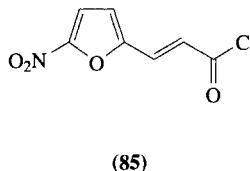
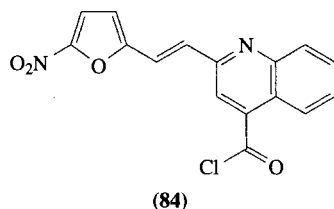
Maleic anhydride (72) has been used extensively in the textiles and fibers industry. Some representative examples are given below. Cellulosic fabrics (i.e., cotton knits) have been treated with a mixture of maleic anhydride and a polyoxyalkylene co-polymer to improve hygroscopicity and softness with good washfastness <92JAP(K)04174774>. Hygroscopic, flat yarns have been prepared for use with electrical cables, water-retaining sheets for agricultural goods, and medical instruments by sandwiching a polypropylene extrudate between two extrudates of maleic anhydride-grafted linear polyethylene <89JAP(K)01156578, 89JAP(K)01162874>. High density fibers with high bending strength have been prepared by spinning polypropylene (30 parts), maleic anhydride modified polypropylene (20 parts), cellulose pulp (45 parts), and silicone rubber (5 parts) at 120°C <91JAP(K)03224713>. Maleic anhydride and Glauber's salt (sodium sulfate decahydrate) are useful additives for dyeing wool. Wool, dyed with a mixture of Palatine Fast Blue, maleic anhydride, a wetting agent and Glauber's salt at 100°C for 1–1.5 h provided fibers with blue shades which were no more sensitive to alkali than raw wool <90MIP150368>. Polypropylene fibers with improved dyeability were prepared by spinning isotactic polypropylene with 1–25% aminotriazole containing copolymers. These copolymers were prepared by the condensation of furans (82) or (83) with hydrazine hydrate (containing water) in the presence of various aliphatic or aromatic monocarboxylic acids and ε-aminocaproic acid <81CZP192298>.



- (82) R = H
 (83) R = Me

Cellulose esters have been prepared by the esterification of cellulose hydrate fibers with acid chlorides (84) and (85). The resulting fibers exhibited antimicrobial properties <85MI 208-03>. To make environmentally friendly paper substitutes, weather degradable fibers have been prepared by melt spinning poly(2,3-dihydrofuran) (86) at 285°C. When the spun polymer was used with poly-(vinyl alcohol) fibers in a paper substitute, the paper showed marked degradation after 40 d of weathering <91JAP(K)03206113>. Finally, carbon fiber strength was improved by impregnating pitch-

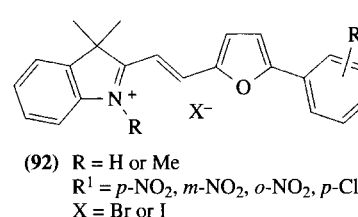
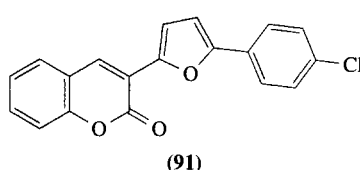
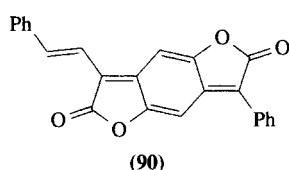
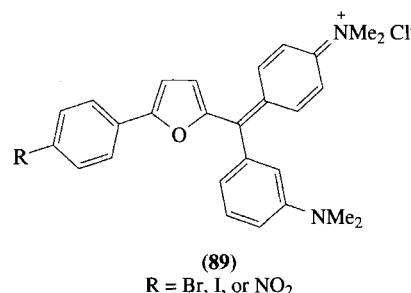
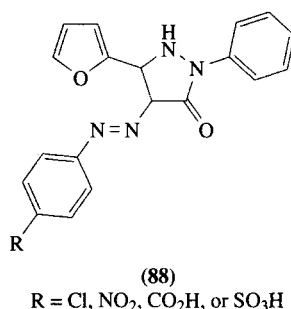
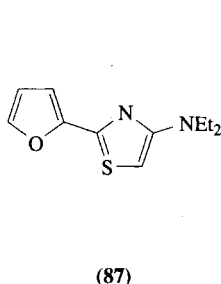
based carbon fibers with a 90 : 10 mixture of furfuryl alcohol and furfural-modified furfuryl alcohol resin containing 2% $\text{BF}_3 \cdot \text{EtNH}_2$. The tensile strength of the fiber increased from 11.0 g denier⁻¹ for untreated carbon fibers to 14.2 g denier⁻¹ <88EUP251596>.



2.08.2.5 Dyes and Photosensitizers

A few furan rings have been incorporated into molecules which have shown useful properties as dyes and photosensitizers. Furan (87) was reacted with diazotized *p*-nitroaniline to produce a reddish blue dye which has been used to color polyester fibers <80GEP(O)2910806>. Polyamide fibers were dyed yellow to brown shades by furans (88) <88MI 208-06>, while furans (89) provided dyes ranging in colour from blue-green to violet <86MI 208-03>. Some furan derivatives have been used to dye polyesters. The highly conjugated lactone (90) provided a fast red shade <88BRP2205324> while the furyl coumarin derivative (91) was found to enhance the whitening effect of polyesters <85DP331>.

Finally, the furan salts (92) have been reported to be photosensitizers, which enhance the photo-sensitive properties of poly(vinylcarbazole). Unfortunately, no data were given <85MI 208-04>.



2.08.2.6 Cellulose, Lignin, Paper, and Other Wood Products

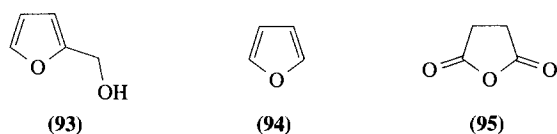
A number of interesting property changes are possible in wood and paper products by the addition of furan-containing compounds during manufacturing processes. Furfuryl alcohol (93) has been used numerous times, along with other additives, in the preparation of particleboard. Particleboard made from the flakes of *Abies concolor*, when oxidized with nitric acid and then treated with a cross-linking agent consisting of ammonium lignosulfonate, furfuryl alcohol (93), maleic anhydride (72), and water (in a ratio of 4.2 : 1.8 : 1.0 : 5.2 per 100 g of wood) gave a constant thickness swelling of approximately 14–16% in a 2 h boiling test. A similar treatment with hydrogen peroxide failed the boiling test <83MI 208-05>. However, if paraformaldehyde was used in place of maleic anhydride, good strength properties and resistance to swelling were observed <82MI 208-02>. Flakes from a

Douglas fir tree, when treated with hydrogen peroxide and a cross-linking agent consisting of ammonium lagnosulfonate, furfuryl alcohol (**93**), maleic acid, and ferric chloride produced boards with strength properties and boiling resistance comparable to or better than those obtained from phenolic resin binders. <82MI 208-03>. Evenly colored black birch veneer was produced when the wood was impregnated with 10% furfuryl alcohol (**93**) then immersed in an aqueous *p*-toluenesulfonic acid solution for 5 s and dried at 170°C <87JAP(K)62160202>. It has been claimed that the properties of some phenolic resins can be improved by the addition of either furfuryl alcohol (**93**) or poly(vinyl alcohol) to the phenol during the polymerization process; however, no details were provided <88MI 208-07>.

A mixture of sulfuric acid and furan (**94**) was found to increase the uniformity of wetting of cotton husks, which resulted in a fourfold decrease of the acidity value of the raw material. In addition, the yield of furfural increased by approximately 5% <85MI 208-05>.

Maleic anhydride (**72**) has been used extensively in the paper and wood industry. There have been numerous reports in which maleic anhydride is added to the emulsions used for the sizing of paper <88MIP145090, 90MIP2011990, 91JAP(K)03234892>. Generally, emulsions containing some maleic anhydride (among other additives) give paper showing good Stoeckigt sizing. Enhanced water-proofing and frictional properties of wrapping paper have been reported when the paper is treated with an emulsion containing a mixture of ricinoleic acid, linoleic acid, and maleic anhydride <83MIP27736>. The impact strength of modified wood is increased by adding triethylene glycol dimethacrylate containing styrene and 2–5 wt.% maleic anhydride. It was reported that the processability of the wood was also improved <82URP975400>. The dimensional stability of wood and weather resistance is improved by adding anhydrides to the hydroxy groups present on the outer layer of wood and then treating the wood with epoxy compounds at 60–150°C. Thus, a piece of Japanese cypress was dipped into molten maleic anhydride containing *N,N*-dimethylbenzylamine at 100°C for 3 h and then treated with epichlorohydrin at 120°C for 3 h. The resulting wood showed improved stability and discolored to an ocher color after 800 h exposure in a Sunshine weatherometer. Untreated wood turned reddish brown <88JAP(K)63054204>. Wood impregnated with maleic anhydride and glycerol in water or alcohol, followed by hot pressing gave improved plywood with excellent gloss, surface smoothness, water resistance, dimensional stability, and durability <89JAP(K)01226302>.

Interestingly, maleic anhydride has been used in the ink industry both to prepare inks and to deink paper. Printing inks giving sharp images and high gloss have been prepared by heating rosin (100 parts) with maleic anhydride (1–20 parts) at 150–250°C, adding diprimary diamines and heating to 130–250°C, followed by adding polyols and heating to 200–300°C. A mixture of the above resin, linseed oil, and mineral oil was used as a printing ink <90GEP(O)3837520>. A deinking agent was obtained by the reaction of a wood rosin–ethylene oxide/propylene oxide (24:16) block co-polymer adduct with maleic anhydride <92JAP(K)04202880>. Succinic anhydride (**95**) has also been used to prepare a deinking agent for use with recycled paper by treating succinic anhydride with ethylene oxide, propylene oxide, and 12-hydroxystearic acid <92JAP(K)04202881>.

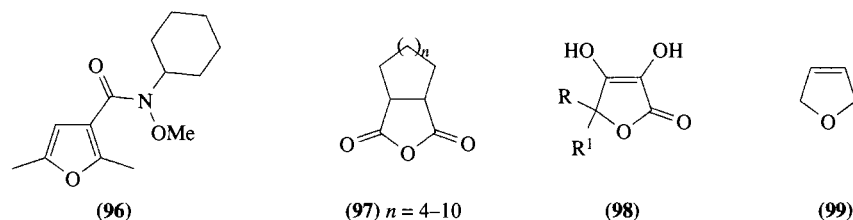


Maleic anhydride has been polymerized with a variety of alkenes to form copolymers. These copolymers have found wide use in the paper industry as sizing agents for paper. For example, copolymers of maleic anhydride with styrene <84USP4440884>, C_{12–40} monoalkenes <93GEP(O)4133123>, phthalic anhydride–propylene glycol copolymer <90JAP(K)02142814>, norbornene <84EGP210695>, diisobutylene <84USP4481319>, and α -monoalkenes <83EGP159077> have been reported.

Furan (**96**), when added to paints, provided good wood preservatives, which had good wood penetration and temperature stability <86GEP(O)3610189>. Thus, wood destruction by termites was reduced <83GEP(O)3130675>, and attack by *Coniophora puteana*, *Poria monticola* or *Coriolus versicolor* was prevented <83GEP(O)3139252>. Cycloalk(en)ylsuccinic anhydrides have good dispersibility and sizing effects on paper <88JAP(K)63227894>. Thus, handsheets sized with cyclohexenylsuccinic anhydride had a sizing degree of 36 s, versus 27 s when sized with dodecenylsuccinic anhydride. Furanones (**98**), where R¹ or R² is any substituent that does not attack the furanone ring, reduces the brightness reversion of high-yield wood pulps which have been bleached with Na₂S₂O₄ <89USP4871423>. Finally, a two-stage emulsion polymerization of butadiene (25 parts), styrene (49

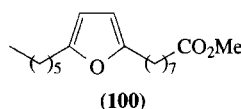
parts), methyl methacrylate (15 parts), acrylonitrile (8 parts), itaconic acid (2 parts), and acrylic acid (1 part) with 2,5-dihydrofuran (**99**) (2.5 parts) gave latexes with good adhesion and printability on paper <91JAP(K)03227304>.

Finally, a review has been published on the manufacturing of furan and furan derivatives from biomass and papermaking wastes <87MI 208-03>.



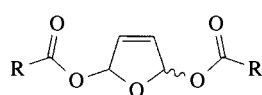
2.08.2.7 Industrial Organic Chemicals, Leather, Fats, and Waxes

Only three compounds were found to be useful in this category. Furan (**100**) has shown good antioxidant properties for fats and oils. Thus, when 1% (by weight) of furan (**100**) is mixed with methyl linoleate and heated to 45°C for 2 d, the amount of oxidation of methyl linoleate was reduced <89JAP(K)64003192>. Succinic anhydride (**95**) has been used to prepare a flux for soldering. A mixture of succinic anhydride (**95**), tetrahydrophthalic anhydride and pyromellitic anhydride when combined with trimethylolpropane and heated for 1 h at 150–160°C gave a poly(half)ester. When this ester is mixed with hydrogenated rosin and isopropanol a soldering flux was obtained with good working properties <92JAP(K)04200992>. Finally, good manganese and cobalt catalysts are obtained when a limonene/maleic anhydride co-polymer or β -pinene/maleic anhydride copolymer are complexed with Mn^{2+} or Co^{2+} ions. The catalysts effectively oxidized *n*-hexadecane to *n*-hexadecanoic acid <93MI 208-02>.

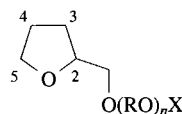


2.08.2.8 Surface Active Agents and Detergents

A few furan derivatives have found a use in the detergent industry. A copolymer (1:1) of $MeO(C_2H_4O)_9CH_2CH=CH_2$ and maleic anhydride (**72**) (20 000 ppm) when mixed with potassium nitrate (1100 ppm), magnesium sulfate (520 ppm), calcium dihydrogen phosphate (310 ppm), and ammonium sulfate (140 ppm) provided a good cleaning agent for plants and trees <90JAP(K)02067400>. A dispersant was obtained for α -ferric oxide and titanium dioxide when 1-heptene/maleic anhydride co-polymer and polyethylene glycol (mol. wt. 2000) were heated to 70°C and cooled to room temperature. The dispersant showed a higher affinity than conventional sulfonated petroleum salts, fatty acid calcium salts, or partially oxidized polyethylene wax <89JAP(K)01004228>. Oxidized co-polymers of furan and maleic anhydride have been reported to have a good sequestering capacity for calcium ions. The co-polymers have found a use as filters in detergent compounds when used for laundering and general cleaning <91USP5061396, 92USP5087682>. Diacyloxydihydrofurans (**101**) are activators for inorganic peroxygen compounds. Thus, the bleaching power of a laundry detergent containing hydrogen peroxide was enhanced by the addition of 2,5-diacetoxy-2,5-dihydrofuran (**102**) <91GEP(O)4015859>. Finally, mixtures containing $\geq 50\%$ of furan derivatives (**103**) (double bonds at C_{2-3} and C_{4-5}), dihydrofuran derivatives of (**103**) (double bond at C_{2-3} , C_{4-5} , or C_{3-4}) or tetrahydrofurans derivatives (**103**) with 0.1–30% surfactants have been found useful for the defluxing of printed circuit boards <91JAP(K)03062898>.



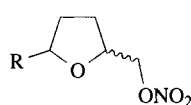
(101) R = C₁₋₈ alkyl, phenyl
(102) R = Me



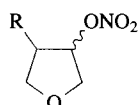
(103) R = C₂₋₄ alkylene
X = H, alkyl, acyl
n = 0-10

2.08.2.9 Fossil Fuels, Derivatives, and Related Products

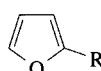
A number of furan derivatives have been used as additives in the fuel and oil industry. Tetrahydrofuran (104) has been found to improve ignition in ethanol-based diesel fuels <83GEP(O)3227493>. In a similar manner, the cetaine number of diesel fuels is increased by the addition of 0.05–0.5 wt.% of furans (105)–(107) <83USP4405335, 83USP4406665, 85USP4522630>. Not surprisingly, maleic anhydride has been used as an additive in fuels. A mixture of maleic anhydride (72), Jeffamine M-300 (a polyoxyalkylene monoamine), and Duomeen T (a hydrocarbyl polyamine) have been found to inhibit engine octane requirement increase (ORI) and carburetor deposit formation <89USP4865621>, while copolymers of maleic anhydride with C₂₋₄₀-alkenes have been added to Otto fuels as anticorrosion-antiwear additives <89GEP(O)3730885>. Furan (94) or branched C₁₋₃-alkyl derivatives promote flame propagation for internal-combustion engines <93GEP(O)4225420>. Furfuryl alcohol (93), furfuryl amine (108), and esters of furoic acid (109) are effective antiknock agents (at 0.1 M levels) in unleaded gasolines <82USP4339245>. Finally, succinic anhydride dimers (110) have been added to diesel and heating fuels to improve the low-temperature fluidity of the fuels. Thus, the reaction product of epoxidized C₂₄₋₂₈-alkenes, di(hydrogenated tallow) amine, and (110) when added to a base fuel gave a pour point of –40 °C <89USP4834776>.



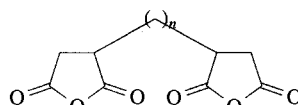
(104) R = H
(105) R = ONO₂



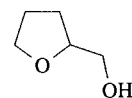
(106) R = H
(107) R = ONO₂



(93) R = CH₂OH
(108) R = CH₂NH₂
(109) R = CO₂Me or CO₂Et



(110) n = 32–36



(111)

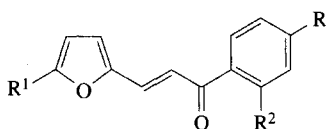
Maleic anhydride has also been used as an additive for oils. Various effects by the addition of maleic anhydride to the oils have been reported (or claimed) and are listed below. Asphaltene flocculation in unstable heavy oils is decreased by the addition of maleic anhydride in the presence of an amine <90FRP2633935>. An effective corrosion inhibitor for iron metals was obtained when the Diels–Alder adduct between maleic anhydride and 2,3-poly(allocimene) was added to petroleum oil or grease <90USP4946626>. A mixture of polyamine dispersants, (prepared by the mixing of polyisobutylene substituted phenol, formaldehyde, tetraethylenepentamine, and oleic acid) and maleic anhydride is effective in maintaining a high level of dispersancy in lubrication oils. In addition, fluorohydrocarbon seals were observed not to deteriorate <90USP4940552>. Maleic anhydride was grafted onto an ethylene–propylene copolymer and then added to a lubricating oil (2.0 wt.%). Testing in a 2.3 L 4-cylinder Ford Ranger truck engine showed the additive to have a dispersancy and seal compatibility superior to a commercial polyisobutenylsuccinimide additive <92USP5162086>. Lastly, viscosity-index improvers for lubricating oils have been produced by grafting maleic anhydride onto Shellvis 200 <90EUP364058> or an ethylene/propylene copolymer <89EUP336716>.

The addition of furfuryl alcohol (93) and formaldehyde to assist with the cross-linking of the water soluble acrylamide/sodium 2-acrylamido-2-methylpropane sulfonate/*N*-vinyl-2-pyrrolidone copolymer provides not only long-term stability of the resulting gel in subterranean formations having an ambient temperature of 93 °C, but also delays the gelling process giving the fluid more time to travel down the wellbore <91USP5043364>. Finally, the addition of furfuryl alcohol (93), furfural (61), or tetrahydrofurfuryl alcohol (111) to a raffinate (obtained from the selective purification of petroleum oils in the presence of Al–Co–Mo or Al–Ni–Mo catalysts, followed by deparaffination) prior to hydrotreatment provided an improved lubricating oil basestock <88URP1432089>.

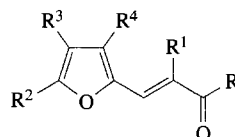
2.08.2.10 Essential Oils and Cosmetics

Polymers have been used extensively in the essential oil/cosmetic industry. A few examples are provided herein. Maleic anhydride has been polymerized with $\text{CH}_2=\text{CHCH}_2\text{O}(\text{C}_3\text{H}_6\text{O})_5(\text{C}_2\text{H}_4\text{O})_{15}\text{Me}$ to provide a copolymer, which when mixed with floral perfumes, propylene glycol, ethanol, water, gelatin, and sucrose gave a transparent air freshner gel with good physical properties <91JAP(K)03197531>. Similar gels prepared from various alkene/maleic anhydride copolymers have also been used in the manufacturing of pet repellents and detergents <89JAP(K)01297484>. Nonaqueous sunscreen oils have been prepared by mixing a 1-octadecene/maleic anhydride copolymer with Lipovol MOS-70, benzyl alcohol, propylparaben, octyldimethyl PABA, benzophenone, homomenthyl salicylate, octyl *p*-methoxycinnamate, silicone oil, and fragrance. The sunscreens exhibited a high sun-protection factor <90USP4940574>. A neutralized cross-linked maleic anhydride/ C_{1-5} alkyl vinyl ether copolymer is the gel forming agent in some shaving gels. Thus, a mixture of 0.2–5.0% gel forming agent, 3–15% soap and 80–97% water provided a useful shaving gel. <91USP5034220>. Finally, a nail polish has been prepared (colored or not colored), which is not toxic to skin, by mixing poly(tetrahydrofuran) (8 g), nitrocellulose (12 g), dibutyl phthalate (4 g), camphor (2 g), and a 2:1 mixture of butyl acetate and ethyl acetate (up to 100 g). The poly(tetrahydrofuran) (mol. wt. 110000) was prepared by the polymerization of tetrahydrofuran in the presence of silver hexafluoroantimonate under a nitrogen atmosphere <83GEP(O)3243291>.

A variety of highly conjugated furan derivatives (112) and (113) have been prepared and used as UV absorbers in sunscreens. Unfortunately, no data was provided on how active the compounds were as UV absorbers <87JAP(K)62158206, 88USP4740369>.

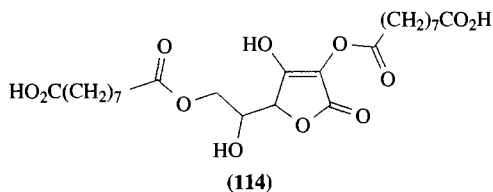


(112) R = H, Me, OH, or OMe
R¹ = H or Me
R² = H or OH

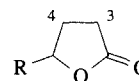


(113) R = alkyl, alkenyl, aryl, alkylaryl, or OR⁵
R⁴ = alkyl, aryl, alkenyl, aryl, or alkylaryl
R¹–R⁴ = H, Me, CH₂CH₂OH, OMe, OEt, OPh

A derivative of vitamin C, furan (114), has been prepared, which exhibited better skin-lightening effects than vitamin C itself. Furan (114) was applied to skin as a mixture of glycerin, pyrrolidonecarboxylic acid, pyridoxin·HCl, ethanol, dipropylene glycol, poly(oxyethylene) hydrogenated castor oil, and water <88JAP(K)63198674>. Lactones (115) and (116) have been shown to stimulate hair growth in synergism with skin peripheral vasodilators, such as vitamin E nicotinate, and capronium chloride. The mode of action is believed to be by the inhibition of testosterone-5 α -reductase <88JAP(K)63275514>.



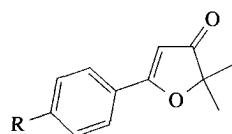
(114)



(115) R = alkyl, double bond at C₃–₄
(116) R = alkyl

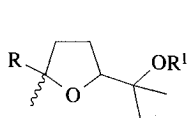
Compound (117) has a long-lasting hay-like odor and has been used to create a fragrance having a rose-like odor. A large number of chemicals have been mixed to produce the rose-smelling fragrance. The chemicals are listed to give the reader an idea of the large number of chemicals involved in the preparation of some fragrances. Thus, furanone (117) (40 g) was mixed with 960 g of a composition of chemicals. This latter composition was made up of 2-phenylethanol (200 g), geraniol (50 g), heliotropin (20 g), citronellol (10 g), nerol (100 g), hydroxycitronellal (30 g), methylphenylcarbinyl acetate (25 g), geranium oil (10 g), linalool (30 g), benzyl acetate (35 g), benzyl alcohol (20 g), rosephenone (10 g), rhodinol (280 g), rose oil (10 g), β -ionone (50 g), benzyl salicylate (40 g), cyclopentadecanolide (30 g), and guaiawood oil (50 g). As can be seen above, the furan compound plays a minor role in the creation of the fragrance of rose <88JAP(K)63313719>. Furanones

(118) have been used as UV absorbents in cosmetics and plastics, by effectively absorbing UVA light <90JAP(K)02164817>.

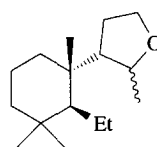


(117) R = H
(118) R = OC₁₋₄ alkyl

Tetrahydrofurans (119) and (120) have been used as additives in perfumes to create a citrus-like floral aroma <87JAP(K)62270573, 89JAP(K)01261384>. Similarly, tetrahydrofuran (121) has been added to perfumes in Russia. Unfortunately, no data regarding the aroma of compound (121) was provided <85URP1169971>.

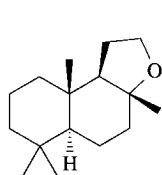


(119) R = Et, R¹ = Me or Et
(120) R = vinyl, R¹ = H

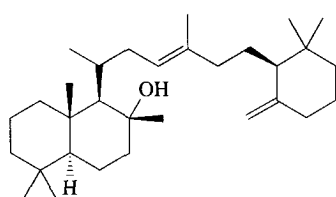


(121)

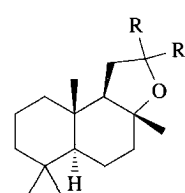
A large number of papers have appeared in the last 12 years regarding the search for suitable analogues of Ambrox (122), which is reported to be major compound responsible for the woody scent with unique fixative properties indicative of the ambergris-type odorants <B-82MI 208-04>. Ambrox (122) is presumably formed from the oxidation of (–)-ambrein (123), which is present in the gut of the blue sperm whale *Physeter macrocephalus* L. <90PAC1377>. The need in the perfume industry for ambergris-type odorants, coupled with the almost worldwide ban on whaling has led to a plethora of papers related to Ambrox (122) and derivatives thereof. Ambrox (122) and tetrahydrofurans (124) and (125) have been produced by the microorganism *Hyphozyma roseoniger*. The latter two compounds have demonstrated an ambergris-type odor <86SAP8504306, 89USP4798799>. Russian workers have prepared tetrahydrofuran (126) and used it in perfume solutions <83URP1049490>. Finally, an elegant enantioselective total synthesis of (–)-9-epi-ambrox (127) has been achieved by Paquette and Maleczka using an anionic oxy-Cope rearrangement as the key step in the synthesis <91JOC912>.



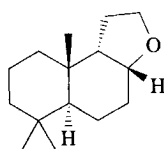
(122) Ambrox



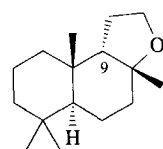
(123) Ambrein



(124) R + R¹ = O
(125) R = Me, R¹ = H



(126)

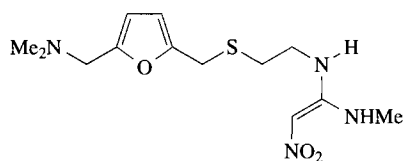


(127) (–)-9-Epi-Ambrox

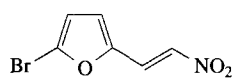
2.08.2.11 Pharmaceuticals

A wide variety of pharmaceuticals have been prepared which incorporate a five-membered ring containing one oxygen atom. A number of compounds have been reported which contain a furan

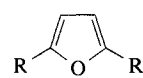
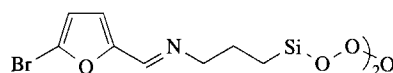
ring. Ranitidine (**128**) has been mixed with either tartaric or citric acid and complexed with bis-muth(III) to provide salts which are very effective for the treatment of gastrointestinal disorders <90BRP2220937, 93MIP9317679, 93EUP533281>. It has been reported that the antimicrobial activity of a variety of drugs can be extended beyond the expected shelf life when the drugs are mixed with furan (**129**) and a carrier solvent comprising of propylene carbonate and/or ethylene carbonate <92USP5122301>. Some 5-substituted-2-furoic acid derivatives (**130**)–(**132**) have shown some pharmaceutical potential. Thus, furan (**130**) has exhibited antiinflammatory and analgesic properties <84EUP123546>, furan (**131**) was effective as an antiseborrhea agent via a topical application <92GEP(O)4033563>, and furan (**132**) was very useful as a snake bite antidote. The latter furan (**132**) was effective when topically applied onto the hemorrhages caused by various snake venoms in mice <82USP4347255>. Interestingly, polysilasesquioxane (**133**) has demonstrated useful antitumor properties <85JAP(K)60163930>, while *N*¹-(2-furanyl)-5-fluorouracil (**134**) has been shown to be useful for the treatment of basaloma <91URP1697814>.



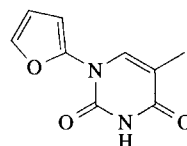
(128)



(129)

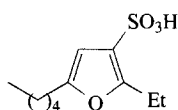
(130) R = *p*-ClC₆H₄, R¹ = CO₂H(131) R = -(CH₂)₁₃Me, R¹ = CO₂H(132) R = CF₃, R¹ = CH=C(SH)CO₂H

(133)

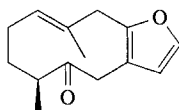


(134)

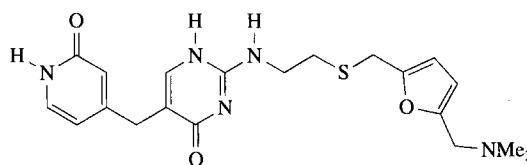
Furyl sulfonic acid (**135**) was isolated from earthworms (*Lumbricus rubellus*) and found to be useful as an anticoagulant, a blood platelet aggregation inhibitor, an ulcer inhibitor, and a vasoconstriction inhibitor. Thus, furan (**135**) showed a 100% inhibition of collagen-induced blood platelet aggregation in human blood at a concentration of 1.1×10^{-1} M <88JAP(K)63005088>. Furano-germenone (**136**) was isolated from a ginger extract and found to be an excellent antiulcer agent. Thus, when furan (**136**) was given orally to mice (500 mg kg⁻¹) and the mice immobilized, placed in water and subjected to stress, gastric ulcers did not appear <86JAP(K)61227523>. Donetidine (**137**) has been shown to be an effective agent against gingivitis or periodontitis by topical administration to gingival tissues in the mouth. Donetidine is believed to act as a H₂ histamine antagonist <93MIP9320815, 94USP5294433>.



(135)



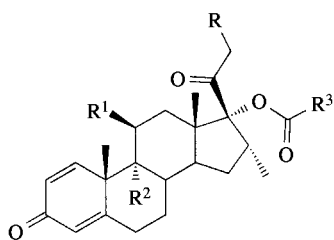
(136)



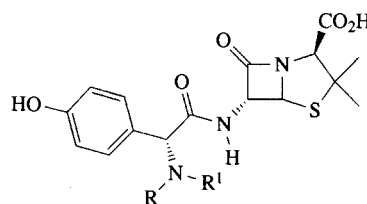
(137)

A steroid and three penicillin derivatives containing a furan ring have exhibited some interesting properties. The Shering Corporation has reported that the steroid derivative (**138**) possessed greater antiinflammatory activity than Valisone (**139**) in the croton oil ear edema test in mice <84USP4472393>. The stability of amoxycillin (**140**) was enhanced by treatment of (**140**) with furfural to give furan derivative (**141**). The bactericidal activity was not affected by the addition of the furfural moiety <82USP4327105>. Furans (**142**) and (**143**) are reported to be improved oral cephalosporin-type antibiotics when administered at the rate of 100–3000 mg d⁻¹ <85GEP(O)3500090>.

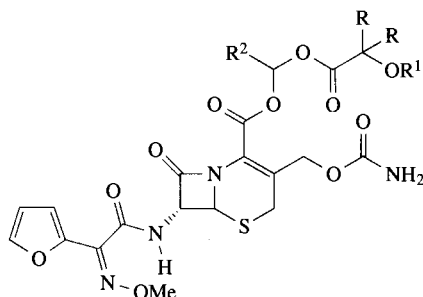
The final three compounds containing a furan ring also possess two or more complex heterocyclic rings. Janssen has patented both barmastine (**144**) and the citrate salt of noberastine (**145**) as antihistaminic agents <85EUP144101, 92EUP464927>. The citrate salt of noberastine (**145**) exhibited superior light stability compared to noberastine and noberastine dimaleate. The hydrochloride salt



- (138) $R = R^1 = R^2 = \text{Cl}$, $R^3 = 2\text{-furyl}$
 (139) $R = R^1 = \text{OH}$, $R^2 = \text{F}$, $R^3 = (\text{CH}_2)_3\text{Me}$

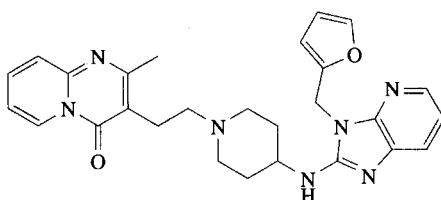


- (140) $R = R^1 = \text{H}$
 (141) $R + R^1 = \text{CH}-(2\text{-furyl})$

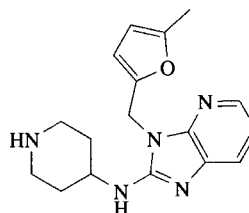


- (142) $R = \text{H}$ or $\text{C}_1\text{-C}_4$ alkyl, $R^1 = \text{C}_1\text{-C}_4$ alkyl, $R^2 = \text{H}$
 (143) $R = R^1 = R^2 = \text{Me}$

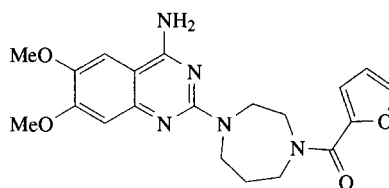
of furan (146) was reported to prevent ventricular tachycardia or ventricular fibrillation in animals at 0.5 mg kg^{-1} . The compound was administered by preparing a capsule containing furan (146) (70 parts), mannitol (300 parts), corn starch (450 parts), lactose (200 parts), calcium stearate (10 parts), and hydroxypropyl cellulose (30 parts) <87EUP209089>.



(144)



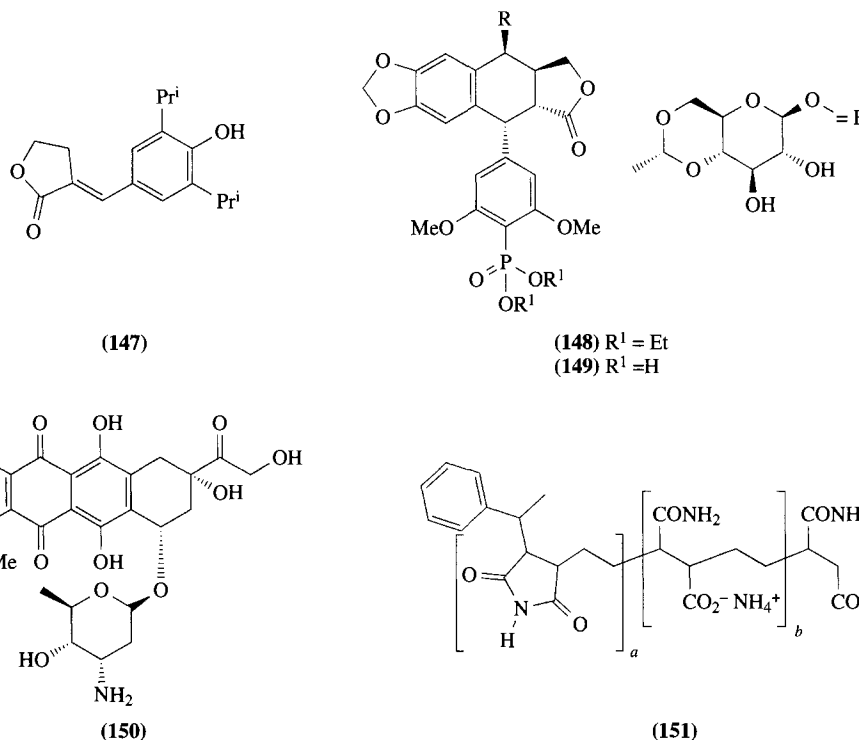
(145)



(146)

Two lactones have shown promise in the pharmaceutical industry. γ -Butyrolactone (147) and its salt was reported as an allergy inhibitor showing very little side effects <85JAP(K)60011415>. Etoposide diethylphosphate (148) was effective for the treatment of small cell lung cancer and refractory testicular tumors. The diethyl ester lost very little potency over an eight-week period when heated to 50°C <93EUP537555>. Interestingly, the prodrug etoposide 4-phosphate (149) can be delivered to a tumor site by the administration of a tumor-specific antibody-enzyme conjugate. Thus, mice with H3347 tumors were treated with an alkyl phosphatase enzyme covalently linked to the monoclonal antibody L6.96.5 through a thioether linkage followed by etoposide 4-phosphate (149). Six out of eight mice showed complete tumor regression and the prodrugs were apparently less toxic than the alkylated drugs <89EUP302473>. The half-amide : half-amide copolymers obtained from ethylene and maleic anhydride derivatives ($a : b = 1 : 2-5$) decreased the cytotoxic side effects of adriamycin (150). No lethality in mice was shown after 30 d when the mice were treated with 1700 mg of (151) per kg

of mouse concomitantly with 21 mg of adriamycin (**150**). Mice treated with 21 mg kg⁻¹ of adriamycin died within 5 d in the absence of the co-polymer (**151**) <90EUP393575>.

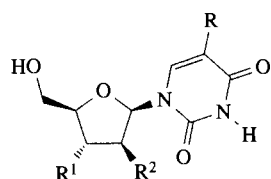
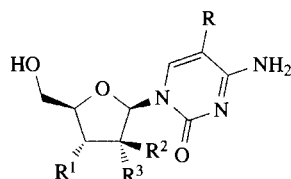
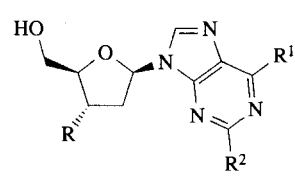
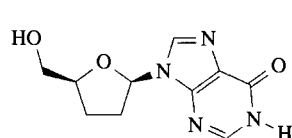


Since the report in 1986 that 3'-azido-3'-deoxythymidine (AZT, now called zidovudine) (**152**) was effective against human retroviruses <86GEP(O)3608606>, a plethora of ribonucleoside derivatives have been prepared and their pharmaceutical properties studied and reported. Only a few representative examples (**152**)–(**164**) are discussed here; space limitations do not allow for a comprehensive overview.

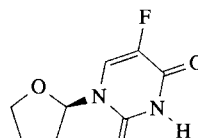
Over 195 patents have been filed since 1986 in which zidovudine (**152**) is mentioned. Potent side effects preclude the widespread use of zidovudine against the AIDS virus. In addition to having activity against the AIDS virus, zidovudine has activity against the feline leukemia virus and gram-negative bacterial infections <86EUP199451, 86EUP217580>.

Alovudine (**153**), sorivudine (**154**), and stavudine (**155**) possess antiviral activity <88EUP254268, 88EUP273277, 91EUP439117>. In addition, monoclonal antibodies to sorivudine (**154**) have been prepared by the hybridoma method and used in radioimmunoassays and enzyme-linked immunosorbent assays for detecting (**154**) <91EUP439117>. Fialuridine (**156**) has exhibited very good activity against herpes infections when given concomitantly with acyclovir <92MIP9209287>. Fialuridine (**156**) and fialitabine (**157**) were effective for the treatment of hepatitis B infections <93EUP565412>. Thus (**156**) at 0.1 mg kg⁻¹ decreased the hepatitis B viral DNA serum levels by 88% in patients. The triphosphate analogue of compound (**158**) showed good activity against human and Woodchuck hepatitis viruses <93USP5246924>. The geminally substituted difluoro compound gemcitabine (**159**) was active against the herpes simplex virus <84BRP2136425>, while the 2',3'-dideoxy derivative zalcitabine (**160**) was active against the HTLV-III virus <89EUP311100>. Purine derivatives (**161**) and (**162**) exhibited anticancer and anti-HIV activity, respectively <92MIP9210198, 93MIP9314103>. Compound (**163**) has been used successfully in combination with other drugs against strains of HIV-I which are resistant to reverse transcriptase inhibitors such as zidovudine, pyridinone, and foscarnet. Combinations of drugs were found to be more effective than the drugs used singly <94MIP9402149>. Finally, it has been shown that a mixture of compound (**164**) and uracil effectively delivers 5-fluorouracil to cancer tissues in warm-blooded animals <82USP4328229>.

A mixture of alfuzosin hydrochloride (**165**) and diltiazem (**166**) act synergistically as an effective antihypertensive agent. Thus, rats treated with combination of 30–160 mg of diltiazem and 0.1–10 mg of alfuzosin showed a decrease in blood pressure which was more than that shown by the drugs alone <86FRP2570275>. Alfuzosin hydrochloride (**165**) has also reduced the pressure at the neck of the urinary bladder by 44% in patients with neurological dysuria <86EUP204597> and stimulated hair

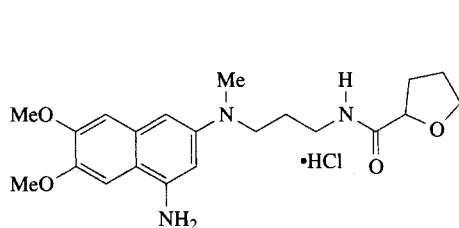
(152) R = Me, R¹ = N₃, R² = H (AZT)(153) R = Me, R¹ = F, R² = H(154) R = CH=CHBr (*E*), R¹ = R² = OH(155) R = Me, R¹ + R² = double bond(156) R = I, R¹ = OH, R² = F(157) R = I, R¹ = OH, R² = F, R³ = H(158) R = Me, R¹ = OH, R² = F, R³ = H(159) R = H, R¹ = OH, R² = R³ = F(160) R = R¹ = R² = R³ = H(161) R = OH, R¹ = NH₂, R² = Cl(162) R = F, R¹ = NMe₂, R² = NH₂

(163)

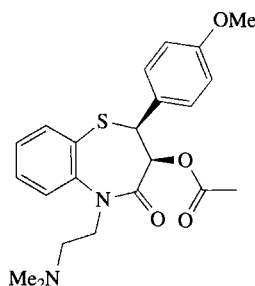


(164)

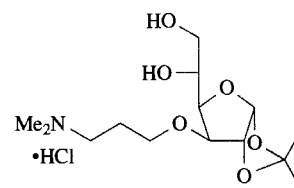
growth <88BRP2197589>. A hair-growth lotion contained alfuzosin hydrochloride (0.3 g), 13-*cis*-retinoic acid (0.025 g), ethanol (95 g), and propylene glycol (4.675 g). A formulation of amiprilose hydrochloride (167), cellulose, starches, and magnesium stearate provided good antirheumatic activity. A preferred dose of 2–200 mg kg⁻¹ day⁻¹ of amiprilose hydrochloride is recommended <89JAP(K)01042436>.



(165)

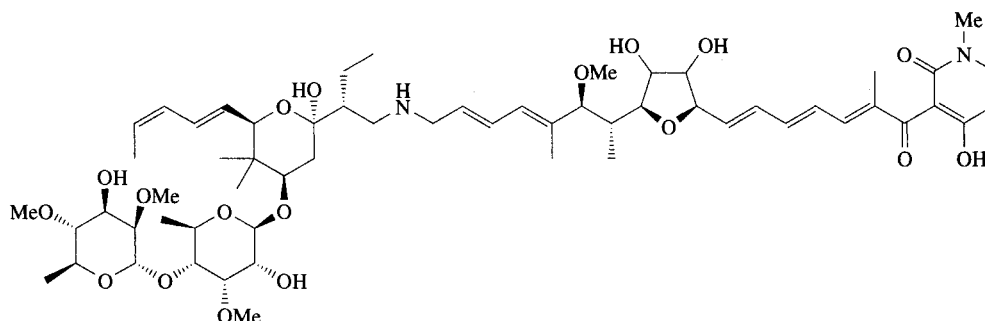


(166)



(167)

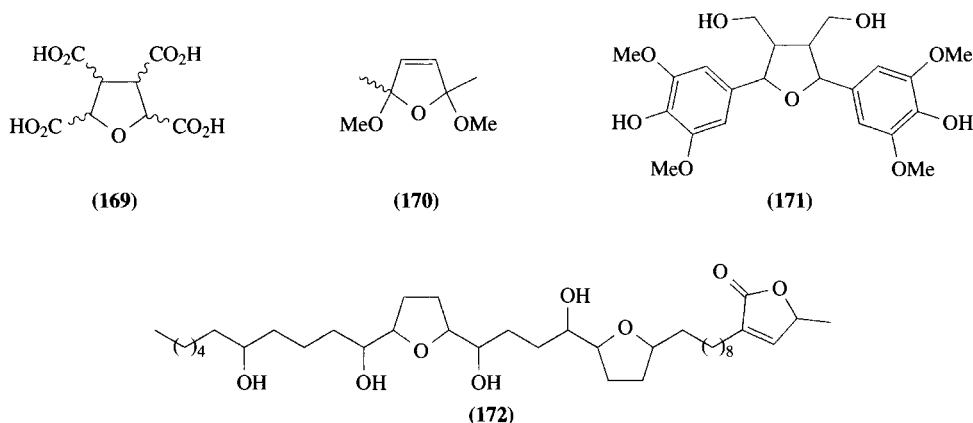
Elfamycin (168) is an antibiotic produced by *Streptomyces lactamdurans* NRRL 3802 and possesses growth promoting activity. A suitable feed additive was formulated by adding the sodium salt of efrotomycin (735 mg) in ethanol (5 ml) to corn cob grits (8.265 g) followed by solvent evaporation and a coating of the solids with 10% tristearin (glyceryl tristearate) <88SAP8706440>.



(168)

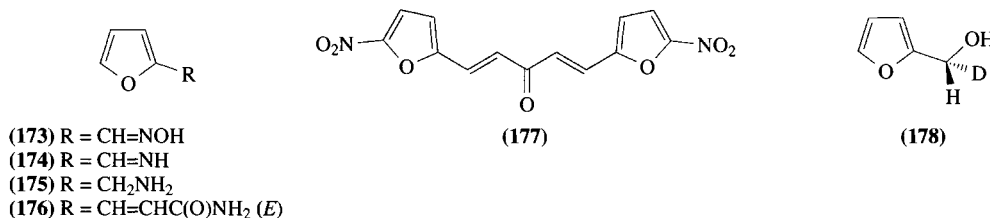
A dental cement has been prepared by mixing an acrylic acid–maleic acid copolymer 7500 (44 parts), water (44 parts), and tetrahydrofuran-2,3,4,5-tetracarboxylic acid (169) (12 parts). This mixture (1 g) when mixed with dental cement powder (1.4 g) had a setting time of 4–6 min and a crushing strength of 1490 kg cm⁻² as compared with 4–8 min and 700 kg cm⁻² for regular cement

<81BRP2076847, 82JAP(K)82126406>. 2,5-Dimethoxy-2,5-dimethyl-2,5-dihydrofuran (**170**) has been used as a disinfectant. Furan (**170**) was active against *Staphylococcus aureus*, *Pseudomonas aeruginosa*, and *Candida albicans* in minimum inhibitory concentrations of 250, 1000, and 1000 ppm, respectively <83GEP(O)3143048>. The lignin derivative (**171**), when given orally to mice at 30 mg kg⁻¹, showed a 64% inhibition of gastric secretion <91JAP(K)03081272>. Finally, the bis(tetrahydrofuran) containing compound (**172**) exhibited anticancer activity against the murine leukemia cell L1210 with an ED₅₀ of 0.5 µg ml⁻¹. Mice treated with compound (**172**) at levels of up to 0.5 g kg⁻¹ d⁻¹ showed no lethal side effects <91JAP(K)03041076>.



2.08.2.12 Miscellaneous

Compounds (**173**)–(**176**) have been added to nitrogen fertilizers, at 2–4%, to inhibit nitrification <91URP1673579>. During the manufacturing of the fodder additive furan (**177**) explosive by-products are formed, which can be removed by a 20% NaOH wash <85MIP34451>. Furan (**178**), prepared by the asymmetric reduction of [1-²H]-furfural, has been used as a precursor in the synthesis of both (*R*)- and (*S*)-[2-²H]-glycine <88T5597>.



Maleic anhydride (**72**) when mixed with triphenylbismuthine in a ratio ranging from 1 : 1 to 1 : 1.5 provided a curing catalyst for a propellant composite comprising an oxidizer, isocyanate curing agent, and a hydroxy-terminated polymer binder. The resulting composite showed improved modulus and/or stress values for use with propellants <91MI 208-06>.

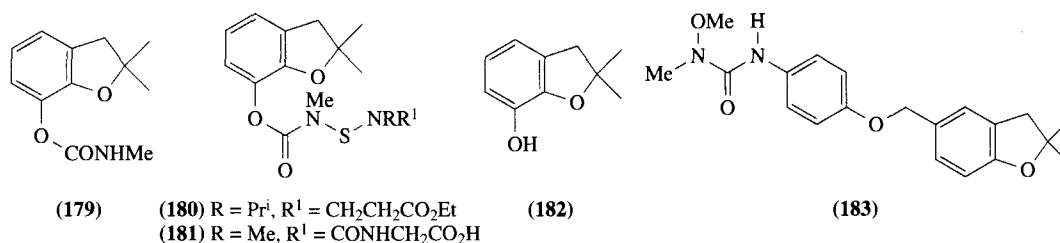
A peptide, conjugated using 1-ethyl-3-(3-dimethylaminopropyl)carbodiimide hydrochloride to a maleic anhydride-derived lysozyme, provided a polyclonal antibody with increased cross-reactivity with maleimidobenzoyl-*N*-hydroxysuccinimide ester-derived hemocyanin <89MI 208-04>. A sandwich enzyme immunoassay for rabbit IgG has been developed. Thus, the enzyme labeling of a goat antibody to rabbit IgG with β-D-galactosidase was possible using the crosslinker *N*-(γ-maleimido-butyryloxy)succinimide <83MI 208-06>.

2.08.3 BENZOFURAN DERIVATIVES

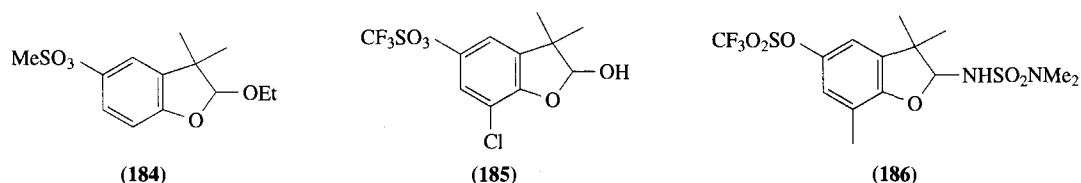
2.08.3.1 Agrochemical Bioregulators

There is a great deal of activity in the synthesis of 2,2-dimethylbenzofurans such as Carbofuran (**179**). Carbofuran is a systemic insecticide active against flies, aphids, larvae, and mosquitos.

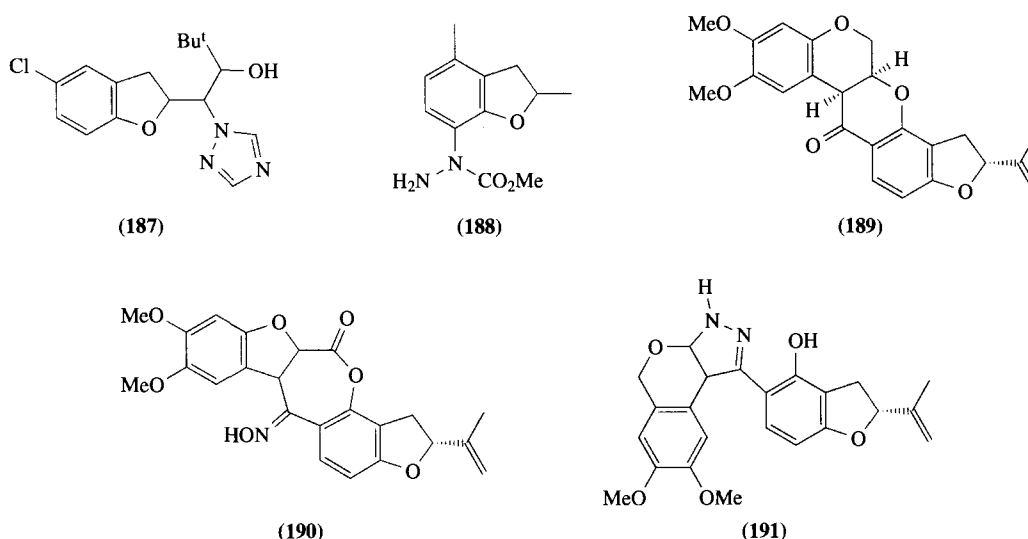
<64NEP6407316> A similar example is the insecticide Benfuracarb (**180**) <83JAP(K)5826804>. Derivative (**181**) is a water-soluble analogue of Benfuracarb active against bean aphid, southern armyworm, and tobacco caterpillar among others <86USP4605667>. Hydroxybenzofuran (**182**) and its 3-keto derivative are components in a mixture that regulated plant growth <84MIP8403419>. Urea (**183**) gave control of barnyard grass <88EUP293839>.



Several 3,3-dimethylbenzofurans exhibit pesticidal activity. Ethofumesate (**184**) is an herbicide for the control of peas, mustard, flax, corn, oats, and ryegrass <69GEP(O)1926139>. Hemiacetal (**185**) gave high preemergent control over four weeds without damaging rice seedlings <92JAP(K)04368376>. Triflate (**186**) gave 100% preemergent control of several weeds in the protection of rice crops with less damage <92JAP(K)04235178>.

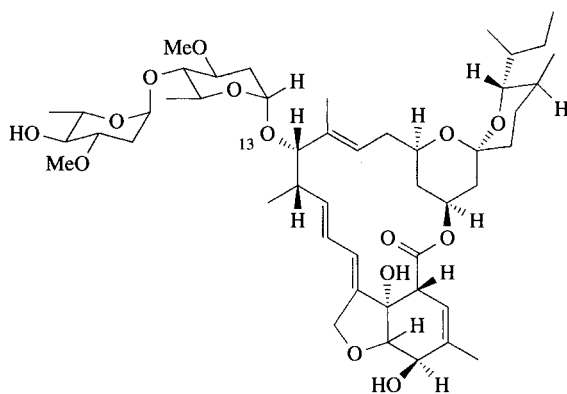


Triazole (**187**) acts as a plant growth regulator found to be effective in reducing the height of summer rape <83GEP3139250>. Hyrazide (**188**) and a related diazine were found to be more effective against spotted spider mites (on bean) than parathion <86USP4587259, 85USP4550121>. Rotenone (**189**) is a natural insecticide found in derris root <B-71MI 208-01>. Oxime (**190**), derived from β -rotenone, and another analogue (**191**) have analgesic activity in addition to insecticidal activity <88JAP(K)63243087, 88JAP(K)6383088>.

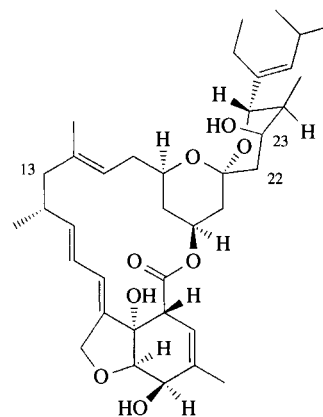


In 1976, Merck, Sharp, and Dohme discovered the Avermectins, a class of macrolides which contain a benzofuran unit as part of the macrolide ring <90PAC1231, 91CSR211>. A dihydro-derivative, Ivermectin (**192**) (22,23-Dihydroavermectin B_{1a}), has emerged as one of the most useful anthelmintics currently available <77GEP(O)2717040, 89CBR692>. It kills a range of parasites in cattle, sheep, pigs, and horses and also has insecticidal activity against lice and mites. Its 13-*epi* isomer is reported to be just as potent but less toxic <92JMC3873>. Closely related are the milbemycins, many of which

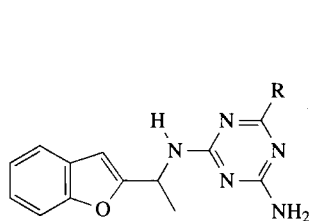
exhibit similar biological activity. One example discovered in this class is Nemadectin α (**193**), isolated from *Streptomyces cyaneogrius* ssp. *noncyanogenus* <87CC402>. The development of milbemycin analogues is a very active area and has been reviewed <91CSR211>. Variations include RNHAr-CH₂CH₂S— <91EUP430884> or aryl ether <91EUP444964> substituents at the 13-position (where the disaccharide is located in Ivermectin). Another example (13-F, 23-C=NOH) prevented the pupation of housefly larvae <91EUP423445>.



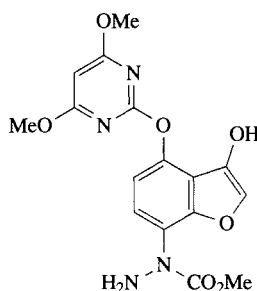
(192) Ivermectin

(193) Nemadectin α

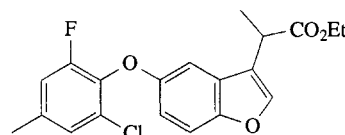
Triazine (**194**) is a herbicide useful in protecting rice paddies <87EUP216259>. Another example (**195**) gave complete control of *Cyperus serotinus* and *Echinochloa crusgalli* without crop damage <89JAP(K)01246279>. Hydroxybenzofuran (**196**) is a preemergent herbicide active against *Echinochloa crus-galli*, *Monchoria vaginalis*, and other weeds <90JAP(K)0477487>. Phenoxybenzofuran (**197**), active against nine weeds, is a postemergent herbicide used to protect rice <92BRP2253847>.



(194) R = Cl
(195) R = CHCl₂

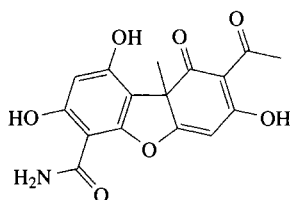


(196)

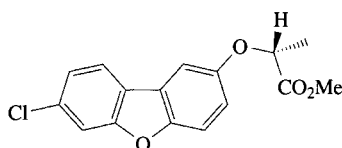


(197)

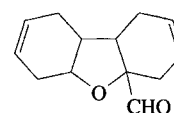
Dibenzofuran (**198**) protected paprika from damage by *Botrytis cinerea* <90GEP(O)3825840>. Another dibenzofuran (**199**) is a herbicide that gave 85% control over *Galium aparine* <91GEP(O)4021503>. R-11 (**200**) is best known as an insect repellent, though the original work also mentions utility as a plasticizer and a bactericide <57USP2795592>.



(198)



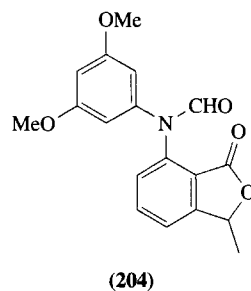
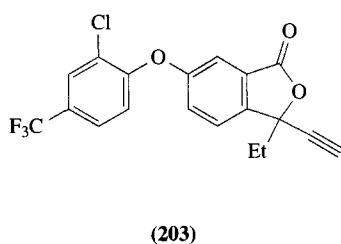
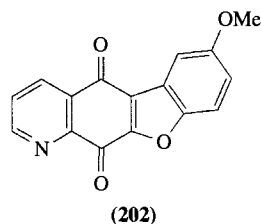
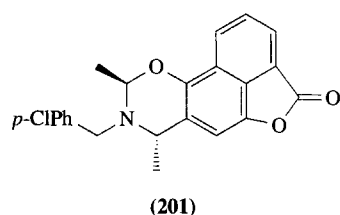
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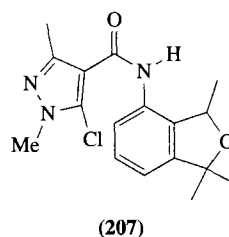
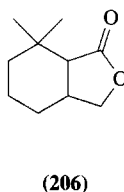
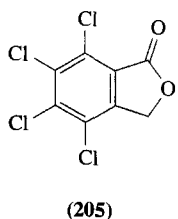
(200)

Lactone (**201**) is an antifungal compound active against yeast and also has antileukemic activity <91PHA742>. Quinone (**202**) is a bactericide <91CCC706>. Phenoxyphthalide herbicide (**203**) gave 95–100% preemergent herbicidal effect on five weeds <87EUP306095>. Pyrimidine derivative (**204**) gave

preemergent and postemergent control of barnyard grass, pigweed, goosefoot, and umbrella sedge <93JAP(K)0592971>.



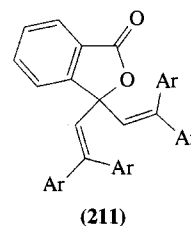
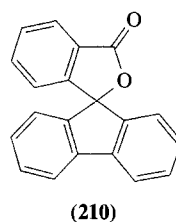
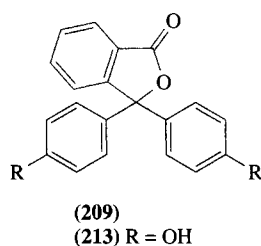
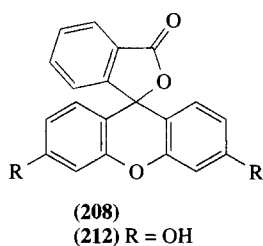
Tetrachlorophthalide (**205**) has been used in a fungicidal composition that controlled *Piricularia oryzae* on rice <83JAP(K)59154506>. Dimethylhexahydrophthalide (**206**) is an useful insect repellent. With its tetrahydro analogue, it was found to be twice as effective against mosquitoes as *N,N*-diethyl-3-methylbenzamide (DEET) <91CP2022491>. Compound (**207**) is an agrochemical fungicide which gave control of *Rizoctonia solani* <89EUP315502> and is a synergistic antimicrobial which controlled *Piricularia oryzae* in rice <91JAP(K)03284601>.



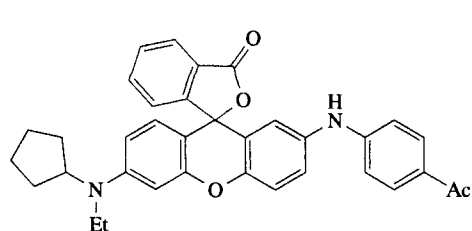
2.08.3.2 Dyes and Photographic Materials

Phthalide derivatives are of major importance in the dye industry, particularly in the area of recording material color formers. Pressure-sensitive carbonless copy paper and thermal recording paper are typical applications. Five principal structural classes have been developed extensively: the xanthene dyes (fluorans (**208**)), 3,3-diarylphthalides (**209**), spirofluorenes (**210**), 3,3-bis(diarylethylene)phthalides (**211**), and 3-substituted phthalides <84MI 208-03>. Some of these structures are common to many familiar acid/base indicators, dyes, biological stains, or laser dyes such as fluorescein (**212**) and phenolphthalein (**213**) <B-91MI 208-07>.

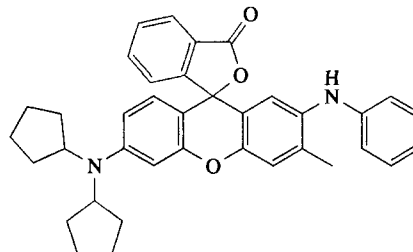
Fluoran (**214**) gave a dark violet image on contact with silica gel and a black image on bisphenol



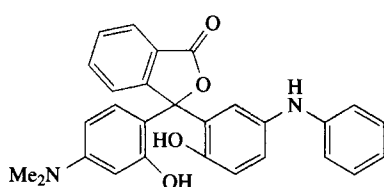
A <84JAP(K)5915452>. Fluroan (**215**) <93JAP(K)05221142> and its “acyclic” counterpart (**216**) <92JAP(K)04191093> have both been patented for used in recording materials. Diaryl phthalide (**217**) and spirofluorene (**218**) are electron-donating couplers in thermal and pressure-sensitive papers <86JAP(K)6122076>.



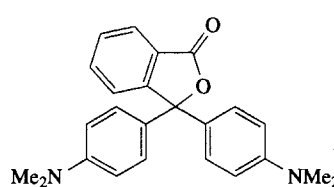
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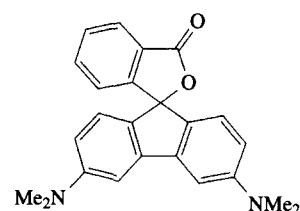
(215)



(216)

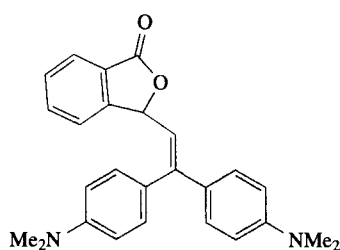


(217)

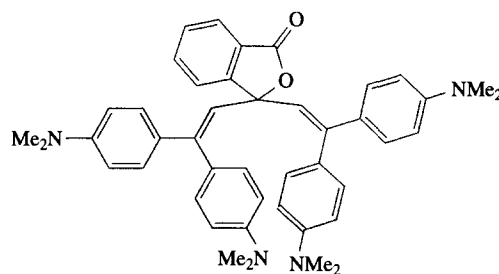


(218)

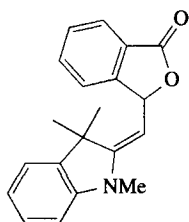
Monosubstituted phthalide (**219**) was used in a pressure-sensitive copy paper <93JAP(K)05177827>, while disubstituted phthalide (**220**) was used in thermal recording paper <88JAP(K)6362777>. Indole (**221**), a less common example, gave an intense yellow color in a composition used for preparing pressure-sensitive copy paper <91EUP437185>. Phthalide (**222**) is a color former in heat-sensitive recording materials <88GEP3727201>. Compound (**223**), which incorporates a benzophenylene system, develops a grey-green color in contact with a clay-coated developing layer <88BRP2192637>.



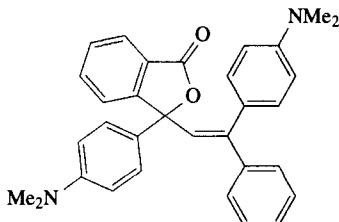
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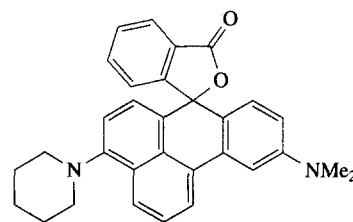
(220)



(221)



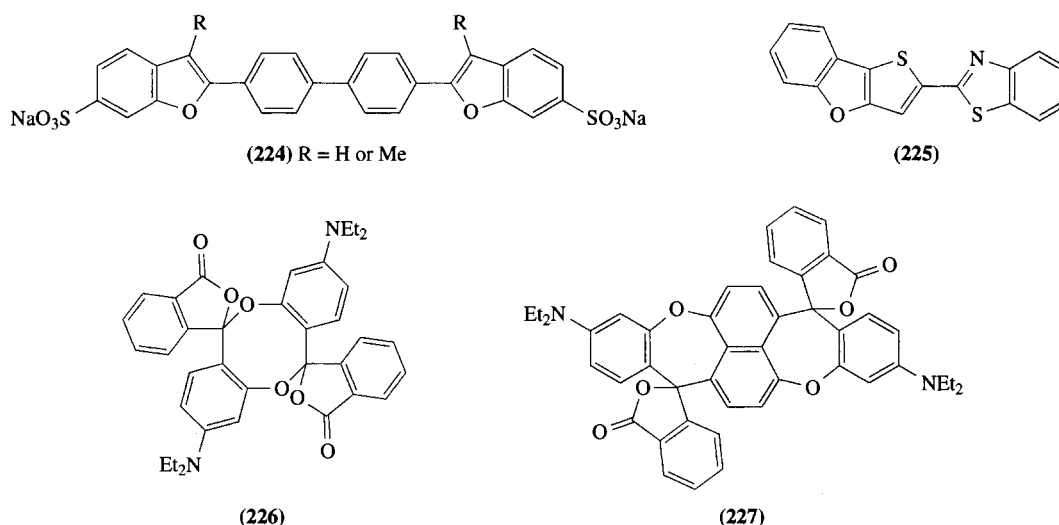
(222)



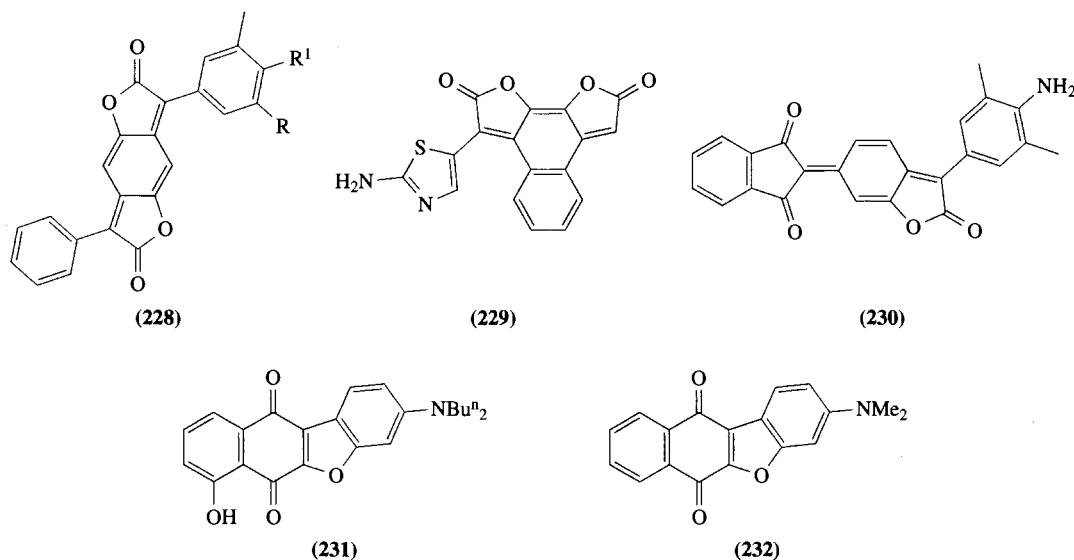
(223)

Bis(sulfo benzofuranyl)biphenyls such as (**224**) have been used as fluorescent brighteners in stable bleach solutions <90EUP395588>. Some examples (R = H) survived much longer than stil-benedisulfonic acid derivatives <89GEP(O)3900651>. Derivatives of polyheterocycle (**225**) were used to whiten polyester textiles <83GEP(O)3311092>. In combination with zinc salicylate and 2-phenylindole,

bis(phthalide) (**226**) was used as a dye coating on facsimile paper <90EUP395588>. Bis(oxepane) (**227**) has been patented for use in recording materials <89JAP(K)01211591>.

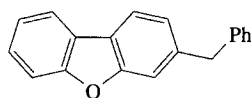


Benzodifurandiones (**228**) and naphthodifurandione (**229**) <92EUP574118> are polyester dyes. Compound (**228**; R = H, R¹ = O-allyl) dyed aromatic polyester dyes a fast, bright red <86EUP182507>, while another example (R = Et, R¹ = NH₂) dyed polyesters blue <93EUP575056>. Indandione (**230**), of similar structure, is a dye in a dye-diffusion thermal transfer printing process <93EUP535842>. Benzofuranoquinones find application as dyes in liquid crystal mixtures. Hydroxyquinone (**231**) had high dichroic property with good photoresistance <92JAP(K)04290880>. Analogue (**232**) was developed as a dichroic dye useful for liquid-crystal displays in electronic devices <91JAP(K)03264575>.

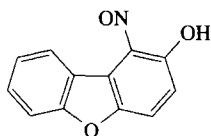


Benzyl dibenzofuran (**233**) was used in a thermal recording material <92JAP(K)0485078>. Nitrosodibenzofuran (**234**) is a dye intermediate <89JAP(K)01193261>. A dialkylaminodibenzofuran has been employed as a charge-transporting agent in electrophotographic photoconductors <90JAP(K)02178670>. Bisazo compound (**235**) is a charge-generating agent in an electrophotographic photoreceptor <93JAP(K)05173344>. Diphenylisobenzofuran (**236**) and several substituted derivatives have also been used in this way <88JAP(K)6360453>. Bibenzofuranyl (**237**) is used as a dye in thermal recording material <93JAP(K)05294977>.

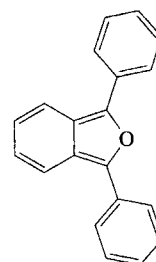
Benzofuranone (**238**) is part of a color developer for thermal recording materials which colors at higher temperature and are particularly good for multicolor recording. The compound is an alter-



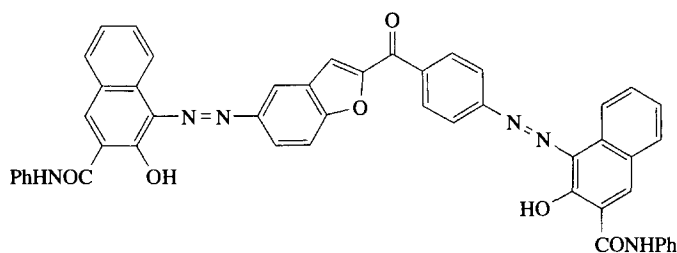
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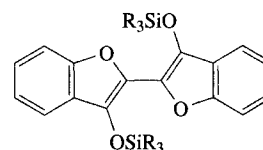
(234)



(236)

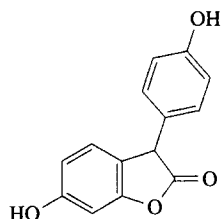


(235)

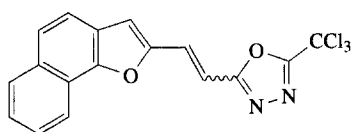


(237)

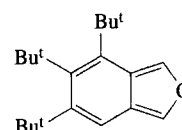
native to bisphenol A <88JAP(K)6317083>. Naphtho[b]furan (**239**) is used to prepare presensitized lithographic plates <93JAP(K)0534904>. Tri(*t*-butyl)isobenzofuran (**240**) has been patented as a photochromic substance useful in preparing optical recording materials <87JAP(K)62201880>.



(238)



(239)

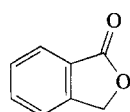


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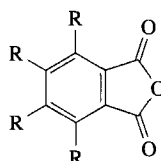
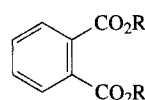
2.08.3.3 Polymers

Phthalide (**241**) has been used as an additive used to prepare polyesters with low molten viscosity <91JAP(K)03200859>. Phthalic anhydride (**242**), prepared by the oxidation of *o*-xylene, is of major importance to the chemical industry with 1992 production over 400000 tonnes <93CEN38>. It is used as a monomer in the preparation of alkyd resins (a polyester with glycerol and other polyols) and thermosetting polymers (with maleic anhydride and polyols). Another important application is in the production of dioctyl phthalate (**243**), which is used as a plasticizer for PVC (1991 production, 123000 tonnes). Over 400 references pertaining to the use of phthalic anhydride in polymer compositions have appeared since 1982. Tetrabromophthalic anhydride (**244**) has been used to prepare flame-retardant bisphthalimides as polymer additives <90JAP(K)02145568>.

Trimellitic acid anhydride (**245**), which has an additional carboxyl group available for cross-

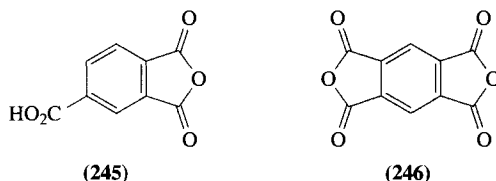


(241)

(242) R = H
(244) R = Br

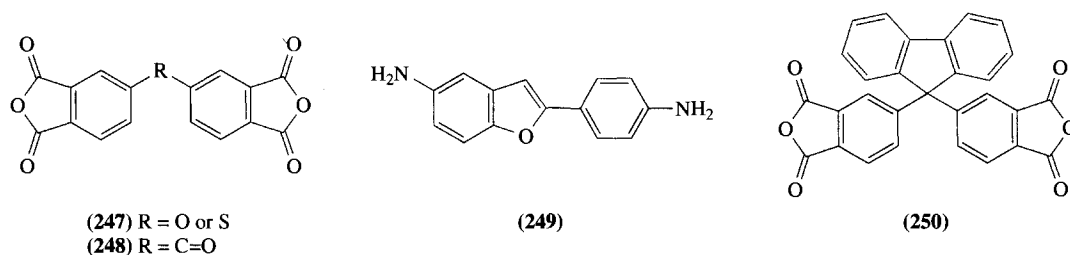
(243) R = 2-ethylhexyl

linking, has also found wide application in the polymer area <91JAP(K)0361954>. In addition, it is also used to prepare bisanhydrides useful as polyimide monomers. Pyromellitic dianhydride (**246**) has been used often, for example, in the preparation of membranes for liquid separation <91EUP418883>.

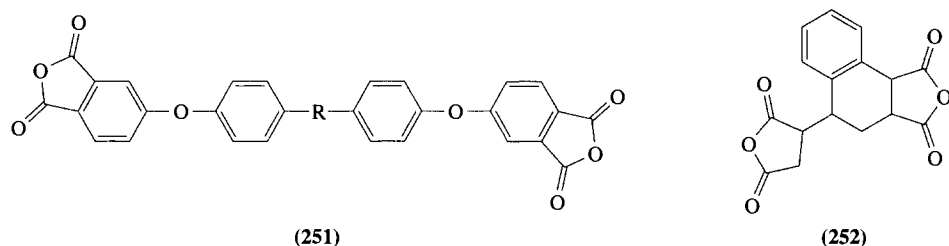


A series of hydro- and bicyclic derivatives of phthalic anhydride have been employed as hardeners in epoxy resins used for optical recording disks <88JAP(K)6383701>, and in a urethane composition to prepare a wire coating with good insulating, thermal shock resistance and adhesion <86JAP(K)61200164>.

A wide variety of bisanhydrides have also been popular as monomers, especially for polyimide preparation. A particularly interesting example (for the purposes of this review) involved combination of bisanhydrides such as (**247**) and (**248**) with benzofuranamine (**249**). The resulting polyimides had good glass transition temperatures and solvent resistance <88EUP264836>. Fluorenic bisanhydride (**250**) has been developed as a polyimide monomer <91USP5061809>. There are many examples of the use of bisanhydride (**248**). One example, in which the anhydride was polymerized with an aminosiloxane, gave a polyimide with good adherence to silicon wafers <90MI 208-05>. A hexabromo derivative of (**247**; R = S, CH=CH, others) has been used in ABS plastic to improve flame retardancy <93EUP549021>.



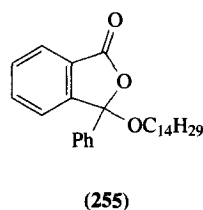
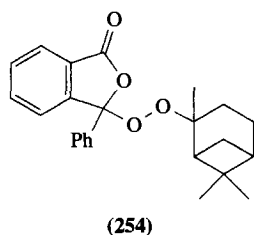
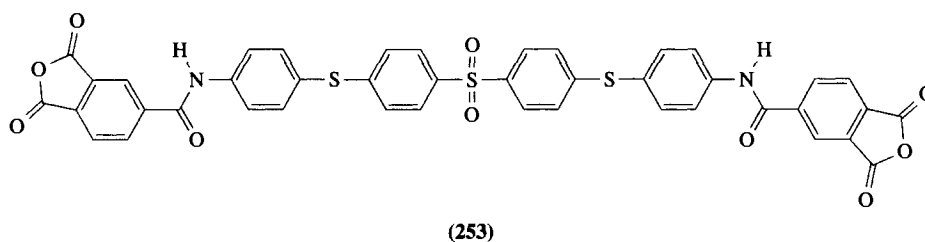
More elaborate examples of bisanhydrides include (**251**; R = CPh₂) which, when polymerized with aryl diamines, gave polyether-polyimides useful for the manufacture of heat and moisture resistant optical disks <90JAP(K)0251527>. Another series (X = spirocyclohexyl or *p*-C(Me)₂PhC(Me)₂) were used to prepare heat-resistant polyether-polyimides that are useful for electronic parts <89JAP(K)01230637>. Bisanhydride (**252**), prepared from styrene and maleic anhydride, is useful as a hardener for epoxy resins, and as a polyester and polyimide monomer <91JAP(K)03264574>.



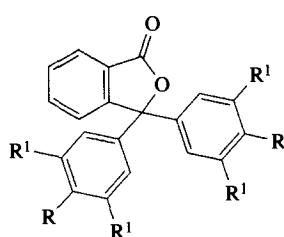
Trimellitic acid anhydride (**245**) was used to prepare sulfone (**253**). Not surprisingly, polymers prepared with this monomer have high thermal stability <89EUP315992>.

Peroxide (**254**) was developed as a cross-linking and vulcanization agent and also serves as a vinyl polymerization initiator <90JAP(K)02117670>. Pseudoester (**255**) is a photopolymerization initiator <89JAP(K)01180857>.

Phenolphthalein (**213**) and its derivatives have found application in the polymer area. Phenolphthalein itself (**213**) and substituted derivatives were polymerized with 2,6-dichlorobenzonitrile to form heat-resistant polycyanoaryl ethers <89JAP(K)01225631>. A tetrabromo derivative (**256**) was polymerized with epichlorohydrin to form flame-retardant epoxy resins. The phthalide-derived



polymers had better flame retardancy than those formed from 3,3',5,5'-tetrabromobisphenol A <84JPS(A)1707>. The fluorinated derivative (257) has enhanced solubility in fluoropolymers making it a useful additive in coating materials <93JAP(K)0543569>. An alkylated phenolphthalein (258) was polymerized with tere- and isophthaloyl chlorides to form a polyester <87USP4652608>. A phenolphthalein homologue (259) has been used to prepare polyurethanes in reaction with 2,4-toluenediisocyanate <88MI 208-08>. Compound (260) has been used to prepare polyamides and polyimides which form flexible, transparent films <94JPS(A)423>.

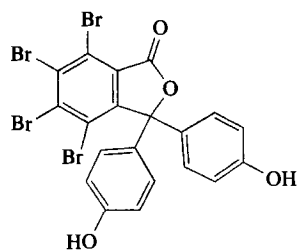


(213) R = OH, R¹ = H

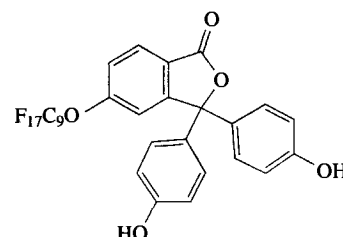
(258) R = OH, R¹ = Me

(259) R = CH₂CH₂OH, R¹ = H

(260) R = -OC₆H₄NH₂ (*para*), R¹ = H

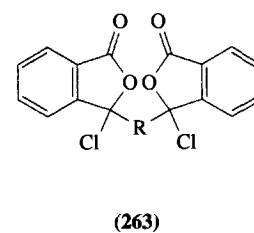
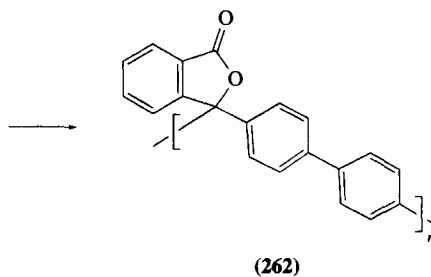
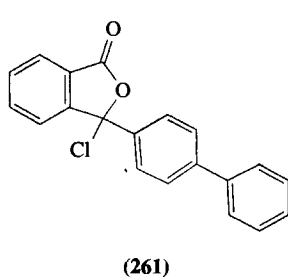


(256)



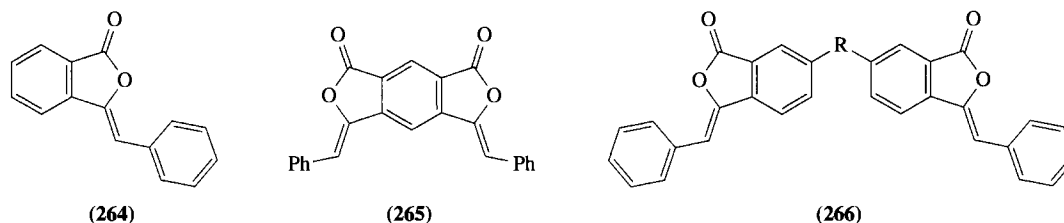
(257)

Chlorophthalide (261) undergoes homopolymerization to form an interesting polyphthalide (262) <90MAC1477>. The monomer was prepared in two steps from phthalic anhydride and biphenyl followed by reaction with SOCl₂. Metal halides such as SbCl₃ or InCl₃ were used as catalysts for the Friedel-Crafts polymerization <84MI 208-04>. A bifunctional example (263; R = PhOPh (*para*), PhSPh (*para*), dibenzofurandiyl) was highly reactive in electrophilic polymerization and could also be polymerized with bisphenol A <90DOK(311)127>.



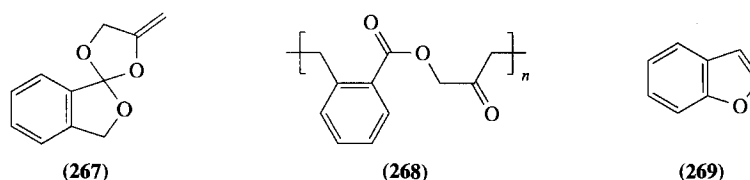
Several arylidenephthalide derivatives have been used in various polymers. Benzaldehyde itself (264) was found to act as a UV stabilizer in aromatic polyesters and polycarbonates

<87GEP(O)3601900>. Benzodifuran (**265**) was reacted with 4,4'-(diaminophenyl)ether and a diacid to form a polyimide-polyamide copolymer <90USP4918153>. Bisbenzalphthalide (**266**; R = CO-PhOPhCO (*para*)) was condensed with diamines to give a variety of polyimides <83JPS(A)1241, 84JAP(K)5927884>.

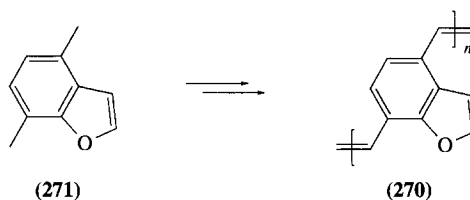


Methylene-1,4,6-trioxaspiro[4,4]nonane (**267**) (BMTN), an orthoester synthesized from phthalide and epichlorohydrin, underwent radical ring-opening polymerization to form an aromatic polyketoester (**268**) with high thermal stability <83JPS(A)353>.

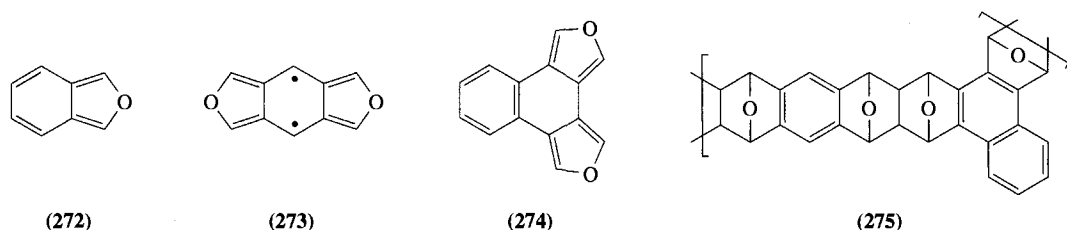
Despite its similarity to styrene, benzofuran (**269**) does not see extensive application in the polymer area. It has been used as an electron-rich vinyl monomer <86PB405> and copolymerized with maleic anhydride (**73**) <82MI 208-05>. It appears in a review on electroanodic polymerization <87CLY673>.



Interest in the preparation of conducting polyphenylenes led to the development of poly(4,7-benzofuran vinylene) (**270**). It was prepared from 4,7-dimethylbenzofuran (**271**) and was found to have a band gap of 2.76 eV. The corresponding thiophene polymer had a slightly larger band gap of 2.92 eV <94JPS(A)65>.



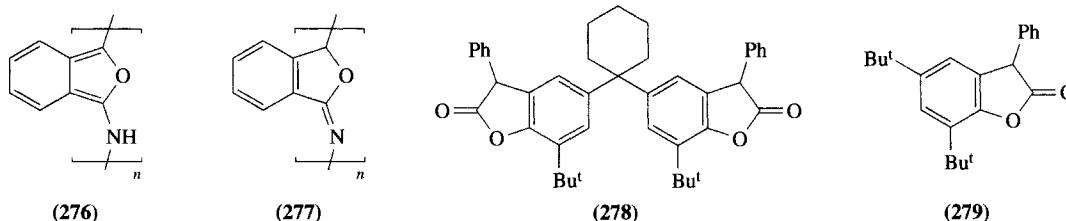
Isobenzofuran (**272**) (benzo[*c*]furan) is a reactive polyene and is unstable at room temperature. There are no examples of a poly(isobenzofuran). Two related species, the theoretical bis(isobenzofuran) (**273**), and naphthobis(furan) (**274**) have been used to prepare an intriguing Diels-Alder ladder polymer (**275**) <92MAC2829>.



Polymerization of *o*-cyanobenzaldehyde has been reported to give poly(nitriloisobenzofuran-1,3-diyl) (**276**), which has good thermal stability. The proposed structure is startling, given the instability of isobenzofuran, particularly when electron-donating groups are present. A more plausible structure may be tautomer (**277**) <82MI 208-06>.

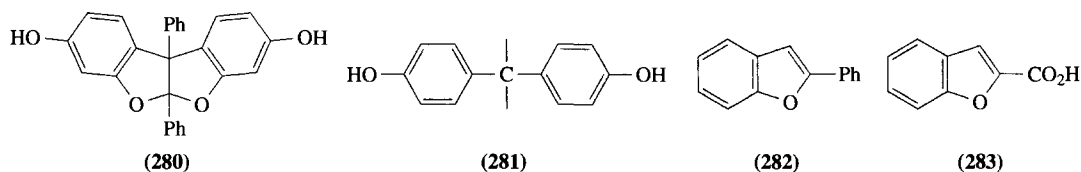
Bislactone (**278**) is a stabilizing agent used to improve the melt index of polypropylene <93BRP2257140>. Other 3-phenyl-2-benzofuranones (**279**) are useful polymer additives for their anti-

oxidant properties <93EUP543778>, for instance, in the manufacture of optical recording disks <86JAP(K)61138648>.



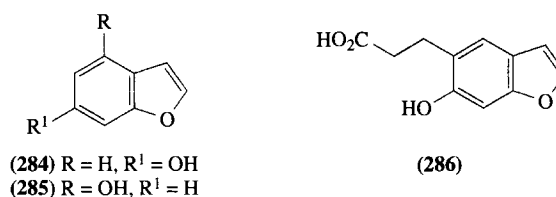
Diol (**280**) has been used as an alternative to bisphenol A in the preparation of thermoplastic polyethers with good fire- and heat-resistant properties <89EUP329029>. It has also been used as a comonomer with bisphenol A (**281**) in a polymerization with 4,4'-difluorobenzophenone <89EUP329029>.

Phenylbenzofuran (**282**) was used to protect polyphenylene ether resins against damage from UV radiation <87USP4665112>. As a nucleating agent, benzofuran-2-carboxylic acid (**283**) was found to improve the properties of an ethylene/4-methyl-1-pentene copolymer <84JAP(K)59197446>.



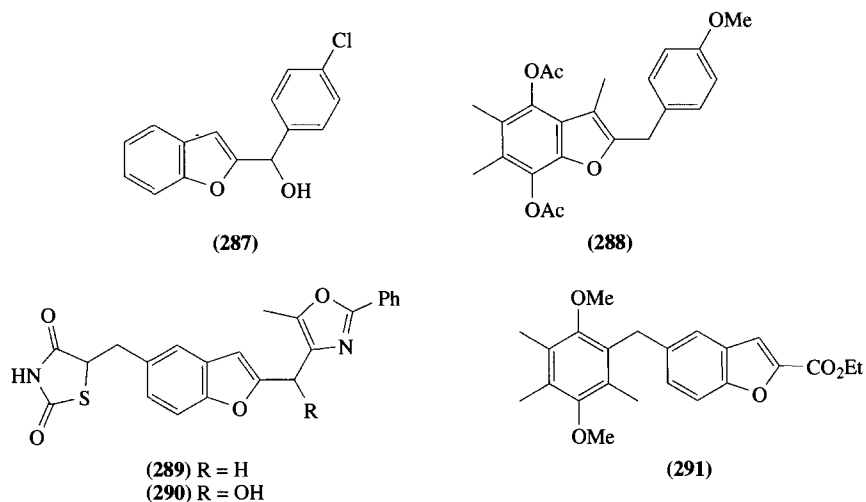
2.08.3.4 Pharmaceuticals

Hydroxybenzofuran (**284**) has found application in the treatment of melasma as a topical skin depigmentation agent <93EUP524108>. An isomer (**285**) has been used as a coupling agent for oxidative hair dyes <92EUP506549>. Acid (**286**), extracted from *Cnidium monnieri*, reportedly enhanced sexual activity in mice. It has been patented for use in the treatment of physical and mental stress <92JAP(K)0459733>.

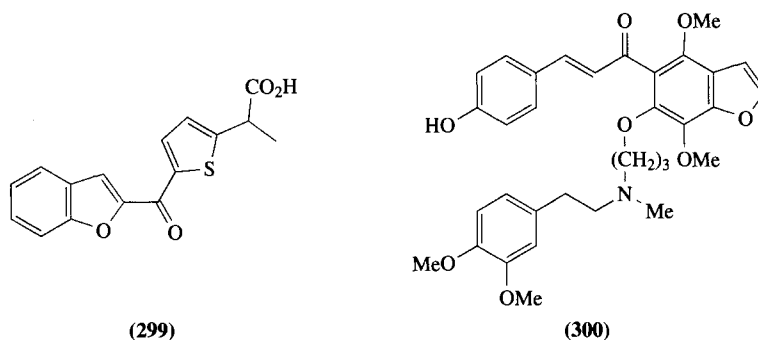
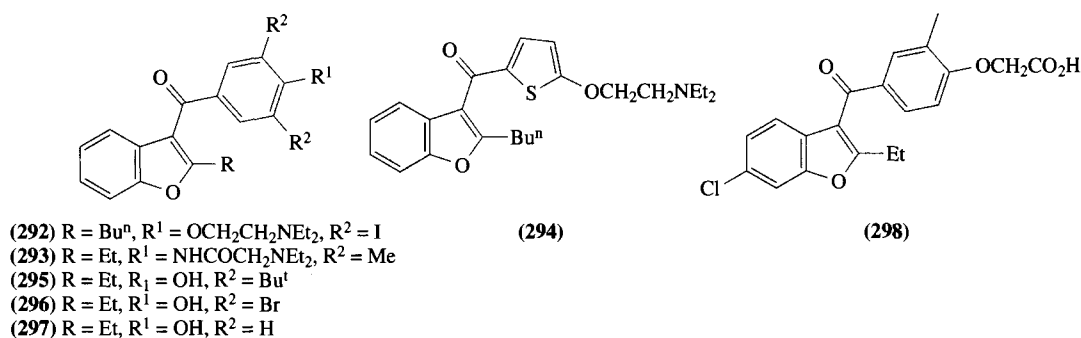


Cloridarol (**287**) has use in the treatment of lipidemia and as an anticoagulant. It was found to be less toxic than the similarly active clofibrate <81GEP(O)3029421>. Benzyl derivative (**288**) is an inhibitor of leukotriene biosynthesis <88EUP295851>. Oxazolidine (**289**) is a promising hypoglycemic; it was found to lower blood sugar levels in obese mice. The benzofuran system was found to greatly enhance the activity of various alternative analogues, phenyl for example <91JMC1538>. A similar example (**290**) has been patented as an antidiabetic and hypolipemic <89EUP299620>. Ethyl ester (**291**) is an inhibitor of arachadonic acid-induced platelet aggregation, potentially useful in treating a variety of cardiac ailments <92JAP(K)04225972>.

A host of aroylbenzo[b]furans have medical applications. Amiodarone (**292**) is an antiarrhythmic and antiischemia drug <85BEP900138> and has also been patented as a component in a skin wrinkle cream <92USP5118707>. Analogues of Amiodarone are (**293**) <89USP4831054> and thiophene (**294**) <90MIP9002743> which have similar activity. Another example (**295**) is an antiarrhythmic which was superior to lidocaine in animal tests <90USP4975458>. Other benzofuran drugs have similar structures; benzobromarone (**296**) is a uricosuric drug <57BEP553621>, while benzarone (**297**) is a capillary protectant <61USP3012042>. Compound (**298**) promotes the excretion of uric acid and is useful for



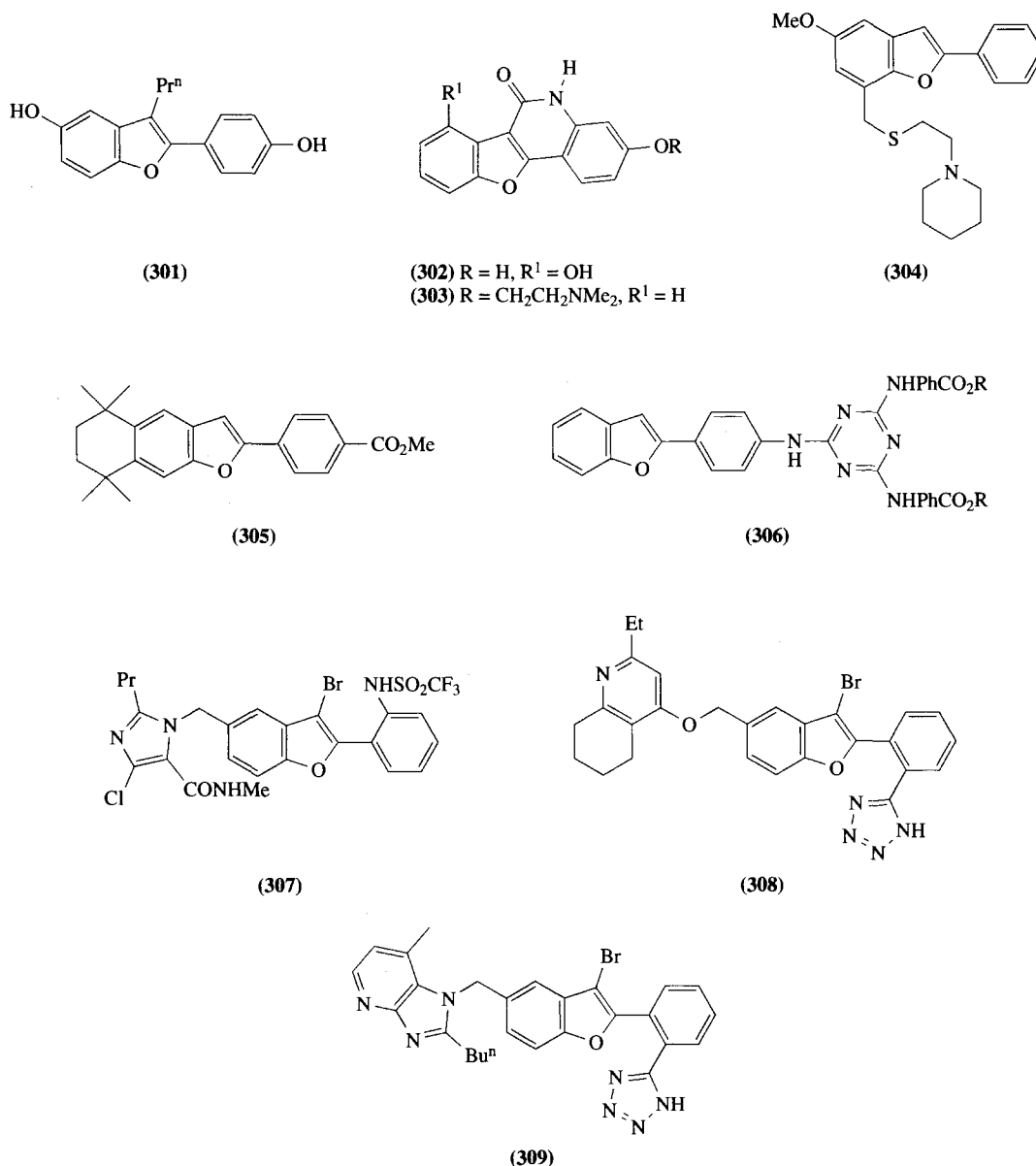
treating gout <91JAP(K)03261778>. Thiophene (299) has analgesic activity <87FRP2600065>. Compound (300) is an antihypertensive and cardiovascular agent <89EUP303920>.



Hydroxybenzofuran (301) is an estrogen inhibitor <83GEP(O)4117512>. Quinolinones (302) and (303) are bone growth stimulants useful in the treatment of osteoporosis <89JAP(K)01242585, 90EUP370760>. Sulfide (304) and its analogues are bladder control agents <89EUP306226>. Ester (305) has been used to treat dermatological conditions <88FRP2614618>, while the triazinyl derivative (306) finds use as a sunscreen <93EUP570845>.

Several synthetic polyheterocyclic compounds are angiotensin II antagonists. Bromobenzofuran (307) is reportedly useful in the treatment of cognitive disorders <92EUP514216>. Other examples are (308) <92EUP520724> and (309); the latter is potentially useful for the treatment of coronary and circulatory diseases <93EUP546449>.

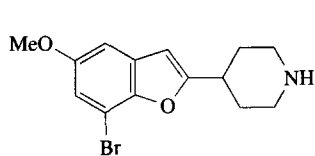
Medical applications of a series of amine benzofuran derivatives have been explored. Brofaromine (310) has been developed as a nootropic for the treatment of degenerative nervous system diseases <92EUP499586>. Etofuradine (311) is an antitussive and antihistamine <68BEP704705>. Coumazoline



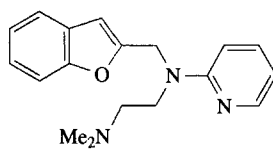
(312) is a peripheral vasoconstrictor useful as a nasal decongestant <72GEP(O)2203373>, while the related faroxan (313) exhibits K channel blocking activity for the treatment of diabetes <92MIP9205171>. Prifuroline (314) is an antiarrhythmic with low toxicity <86AF1761>. Pyridarone (315) is an antidepressant <74GEP(O)2423835> as is befuraline (316), which also possesses analgesic properties <74GEP(O)2240665>. Becliconazole (317) is antimycotic and antibiotic with activity against *Staphylococcus aureus* and *Pseudomonas aeruginosa* <88EUP257171>.

Carocainide (318) is a cardiac drug that reversed ouabain-induced ventricular tachycardia in dogs <78GEP(O)2730593>. Enadoline hydrochloride (319) is an analgesic that also has diuretic and cerebrovascular activity <90EUP393696>. In mice, amide (320) was found to protect the liver from acetaminophen-induced damage <93JAP(K)05320169>.

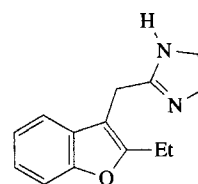
Many carboxylic acid derivatives of benzofurans have important activity. Benfurodil hemisuccinate (321) is a cardiotonic and vasodilator <65FRP1408721>, while sulfonamide (322) and furacrinic acid (323) are antihypertensives <72GEP(O)2227423, 89MI 208-05>. A pyridinyl derivative (324) (sodium furegrelate) is an inhibitor of thromboxane synthetase <83EUP69521>. Several compounds have analgesic activity. Raxofelast (325), for example, is an antiinflammatory and antioxidant <90BRP2221463>. Sodium tifurac (326) was found to be more effective than aspirin in mice <85EUP132130>. Furaprofen (327) is an analogue of the well-known Ibuprofen <82GEP(O)3026402>.



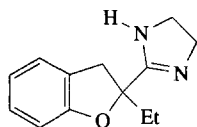
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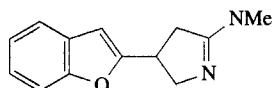
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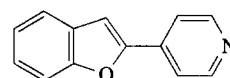
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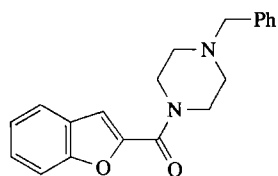
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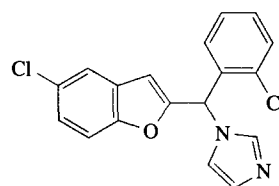
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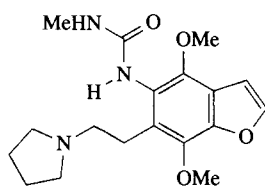
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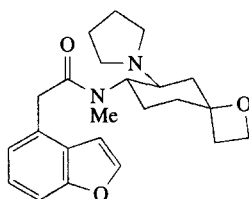
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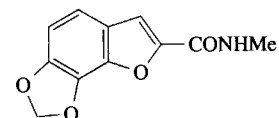
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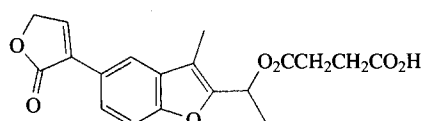
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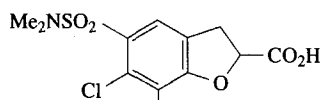
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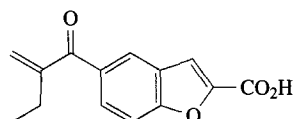
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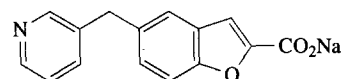
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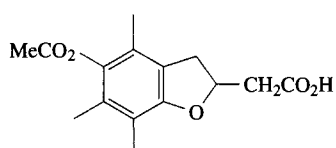
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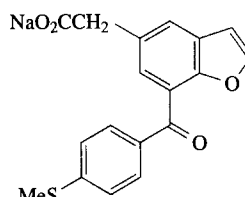
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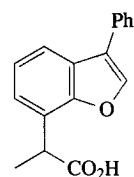
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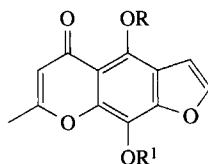


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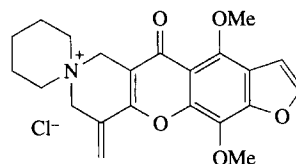
Great interest has been taken in khellin (**328**) and its derivatives. Khellin, which has lipid-altering activity and is a coronary vasodilator, is isolated from the seeds of *Ammi visnaga* <83JOC3863>. Like khellin, iprocrolol (**329**) is a coronary vasodilator <71FRP2077694>. Analogs such as butocrolol (**330**) are antiarrhythmics and effective local anesthetics <75FRP2232311>. Azaspirium chloride (**331**) is a khellin derivative that offers the advantage of water solubility <71GEP(O)2019570>. Compound (**332**) <83USP4412071> and bis(khellin) (**333**) <90MIP9002129, 90JMC2685> have both been developed for the treatment of atherosclerosis.



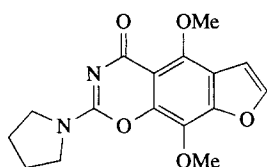
(**328**) R = R¹ = Me

(**329**) R = H, R¹ = OCH₂CH(OH)CHNHPri

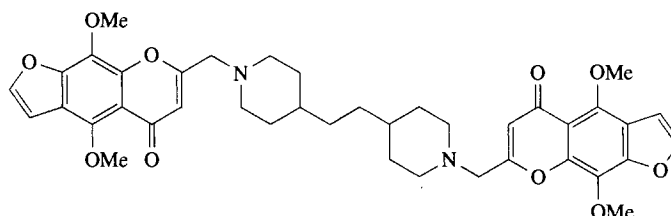
(**330**) R = H, R¹ = OCH₂CH(OH)CHNHBu¹



(**331**)

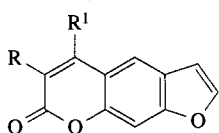


(**332**)



(**333**)

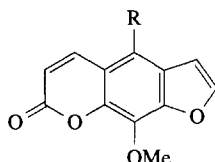
Psoralen (**334**) and its derivatives exhibit a wide range of activity <86MI 208-04>. Methoxsalen or xantotoxin (**335**) and ester (**336**), have been used in the treatment of psoriasis and other skin disorders <83SZP638978>. The psoralen derivative (**337**) has been patented for the prevention and treatment of retroviral infections (HIV-1 or HIV-2) <93FRP2691629>. Sulfonamide derivatives (**338**) show antimicrobial activity superior to methoxsalen <88JIC422>. Trioxsalen (**339**) is a synthetic psoralen that offers skin protection against UVB erythema and is an orally active tanning and pigmentation agent useful in the treatment of vitiligo <59USP2889337>.



(**334**) R = R¹ = H

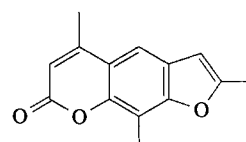
(**336**) R = CO₂Et, R¹ = H

(**337**) R = H, R¹ = OMe



(**335**) R = H

(**338**) R = SO₂NHPh

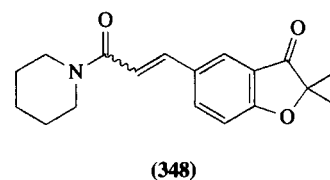
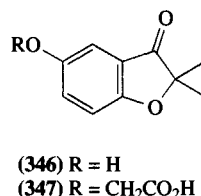
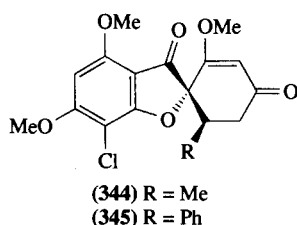
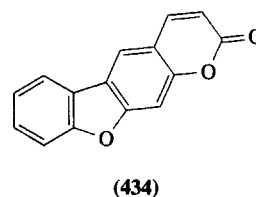
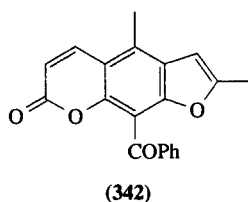
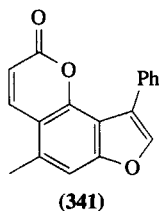
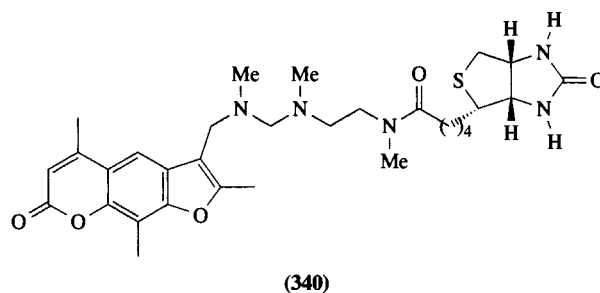


(**339**)

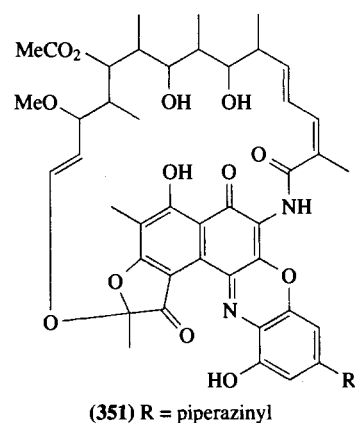
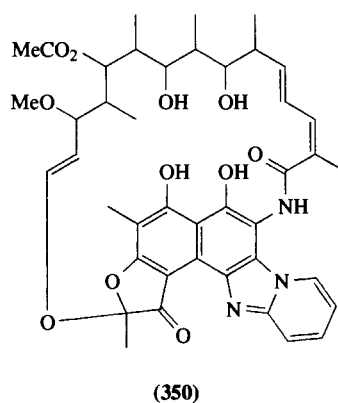
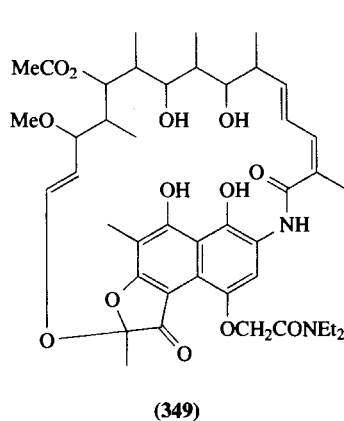
The photochemical activity (phototoxicity) of several psoralen derivatives has potential application in several areas. Biotin derivative (**340**), for example, is a neoplasm inhibitor; it inhibited the growth of lymphocytes by 99% in the presence of near UV light <87MIP8705805>. Compounds (**341**) and (**342**) both displayed photochemical antibacterial activity <87MI 208-04>. Benzopsoralen (**343**) is a DNA photoreagent <86JHC1405>.

Griseofulvin (**344**) is an antifungal antibiotic <89JAP(K)01258668>. Phenyl analogues (**345**) have also been found to be active <92MI 208-06>. Spirofurone (**346**) is an antiulcerative which gave 48% inhibition of gastric acid secretion in rats <79EUP3084>; analogues (**347**) and (**348**) share this activity <89JAP(K)01246273, 89JAP(K)01246279>.

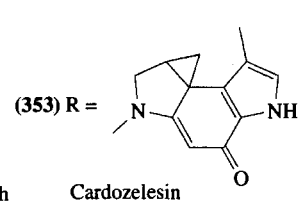
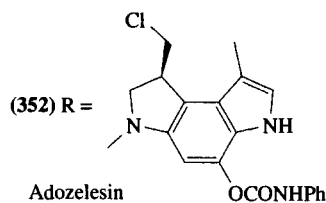
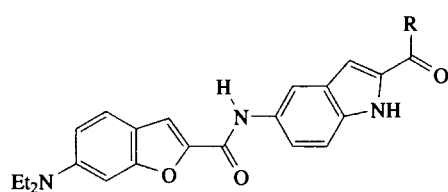
The rifamycins, isolated from the fermentation broths of *Streptomyces mediterranei*, are an important class of antibiotics which have a naphtho[2,1-*b*]furan system as part of a macrolide. Rifamide (**349**) (Rifamycin B amide) was reported to be active against gram-positive bacteria and *Mycobacterium tuberculosis* <63BEP632770>. Many analogues have been prepared, presenting a broad spectrum of antibiotic activity. An example is rifaximin (**350**), which incorporates a fused benzimidazole <81BEP888895>. Another analogue (**351**) was more active against *Mycobacterium*



tuberculosis than rifampicin <91JAP(K)03101681>. Prepared analogs of Rifamycin S have been reported to lower blood cholesterol levels <92EUP475896>.

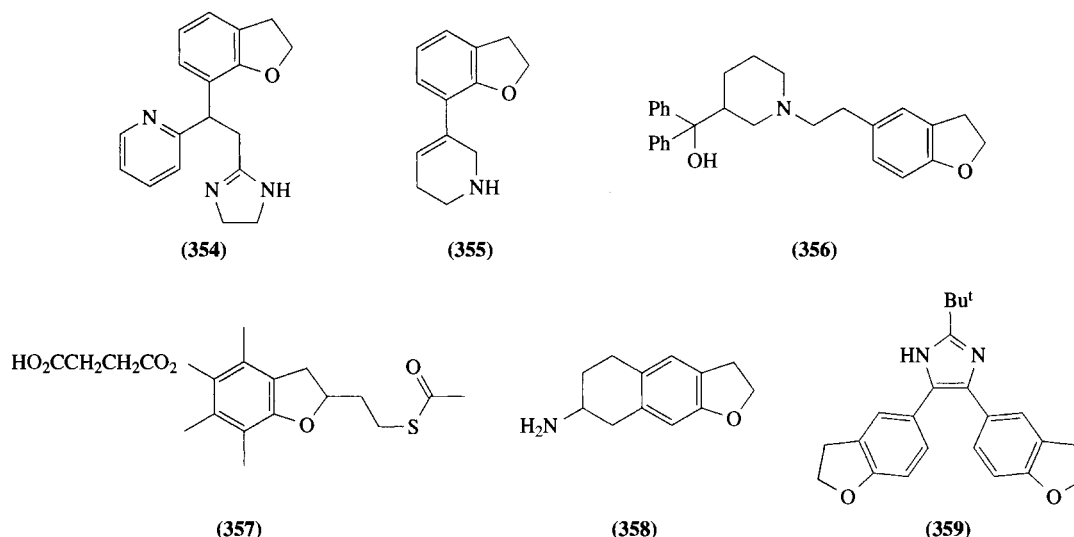


Upjohn has developed two antineoplastic drugs that are analogues of CC-1065. Adozelesin (352) and carzelesin (353) show antileukemia activity in mice <88MIP8804659>.

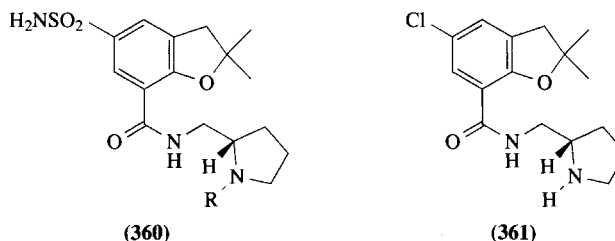


Pyridine (354) is a hypoglycemic shown to reduce blood glucose levels in starved rats <91MIP9100862>. Piperidine (355) is an antihypertensive, psychotropic agent <90EUP398413>.

Pyrrolidine (356) is an anticholinergic agent <91MIP9110651>. Thioester (357) is an antioxidant, mucolytic and antischemic agent which gave significant protection against carbon tetrachloride-intoxicated liver lipoperoxide formation in rats <91EUP413668>. The D-form of amine (358) is a dopaminergic agent <88EUP286515>. Bisbenzofuran (359) is an antiinflammatory and analgesic; tested for the inhibition of edema in rats. It showed greater potency than Ibuprofen <91JAP(K)03157383>.



Amides (360; R = Buⁿ and R = CH₂CH₂CH₂Ph) are psychotropic drugs useful in the treatment of nerve and mental disorders such as schizophrenia, dystrophy, and others <91MIP9117144, 92JAP(K)04364181>. An earlier example (361) was patented as an antiemetic for cancer chemotherapy patients <89EUP314483>.

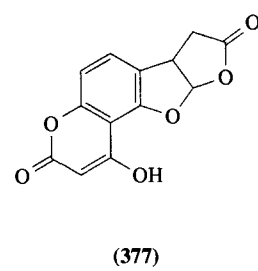
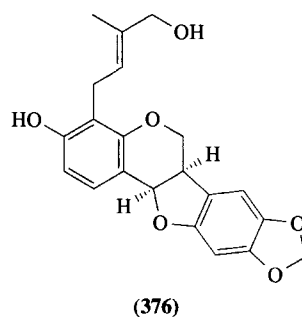
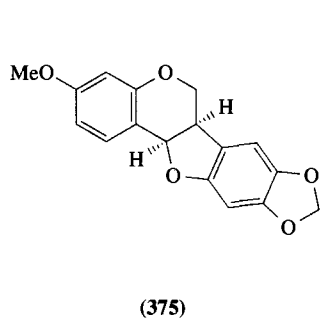
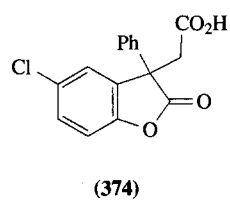
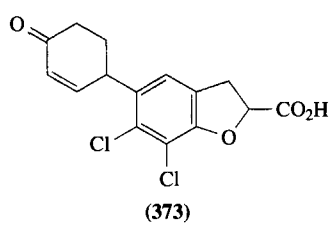
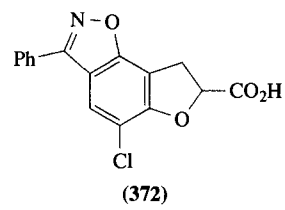
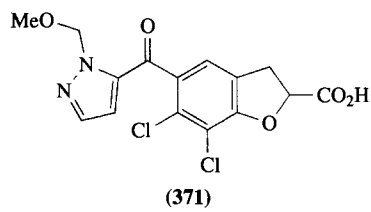
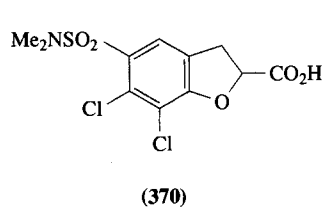
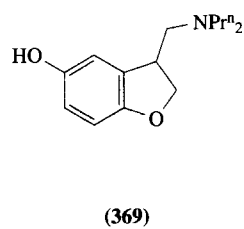
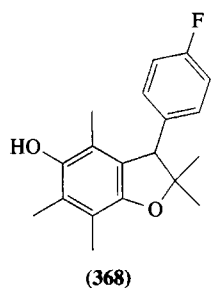
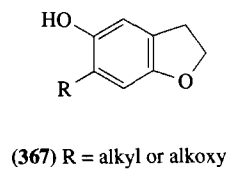
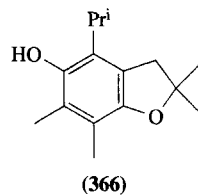
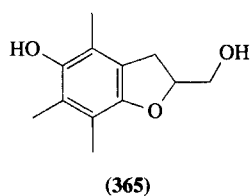
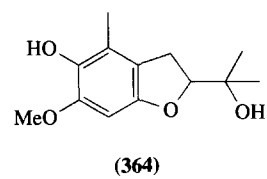
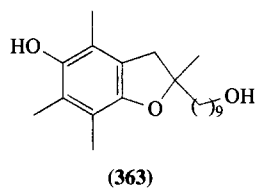
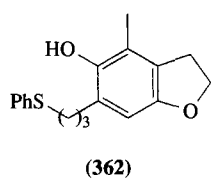


The antioxidant activity of 5-hydroxybenzofurans makes them useful in the inhibition of biochemical oxidations. Sulfide (362) is an inhibitor of leukotriene B₄ biosynthesis <90BRP2221680>. Long-chain alcohol (363) was found to be a better inhibitor of lipid autooxidation than vitamin E <93JAP(K)05178848>. Alcohols (364) and (365) are antioxidants; the former tested against luminol-induced chemiluminescence <90EUP398142> and the latter in an *in vitro* test against the oxidation of ethyl linolate <88JAP(K)6388173>. Alkylated benzofuran (366) inhibited Fe-ADP-adriamycin complex-induced liposome oxidation <90JAP(K)02121975>. A series of compounds (367) has been evaluated as inhibitors of leukotriene biosynthesis <89JMC1006>. Two other examples have medicinal application: fluoride (368) is a cardiovascular and antiallergy agent <88EUP273647> and amine (369) is useful for the treatment of central nervous system disorders <88EUP281261>.

In a study of structure-activity relationships, it was found that the (+)-enantiomer of (370) was responsible for its uricosuric activity <87CPB3195>. Alcohol (371) and acid (372) are also diuretics <89JAP(K)01246277, 92CPB2597>. Ketone (373) was developed for the treatment of brain injury <87USP4654365>, while lactone (374) is a drug for the treatment of hypoxia <87USP4654365>.

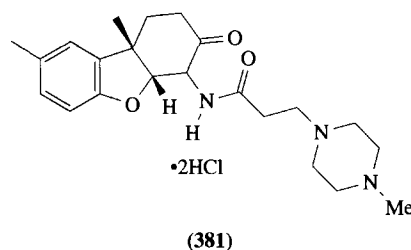
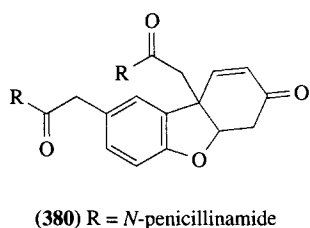
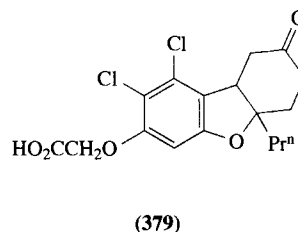
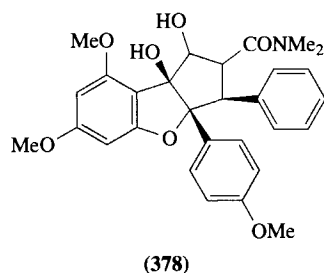
Cabnegrin A-I (375) and its regioisomer cabnegrin A-II are orally active antidotes against snake venoms, isolated from a South American plant. They are members of the pterocarpin (376) family <83EUP89229>. Compound (377) is an aflatoxin analogue which has modest anticoagulant activity <90JIC917>.

Rocaglamide (378) is a natural product isolated from the roots and stems of *Aglaia elliptifolia* Meer. (Meliaceae). It displays antileukemic activity against P388 lymphocytic leukemia in mice and

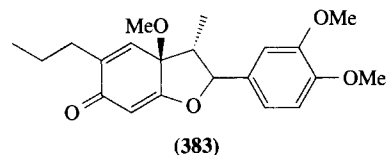
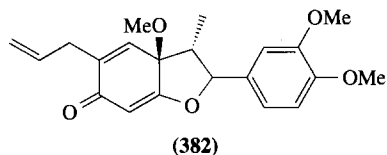


has *in vitro* activity against KB cell carcinoma <82CC1150>. Several analogues derived from (378) have also been prepared and evaluated for their activity <85USP4539414>.

Oxyacetic acid derivative (379) was developed for use in treating brain trauma <88USP4769370>. A similar ring system is a component in a new penicillin derivative (380), which was active against *Staphylococcus aureus* 209P <92JAP(K)04187689>. Taziprinone hydrochloride (381) is an antitussive <75GEP(O)2518289>.



Kadsurenone (382) is a highly specific agonist of platelet-activating factor with a K_i of 5.8×10^{-8} M. It is found in the Chinese herb *Piper futokadsura* (heifenteng) <85JAP(K)6097972, 86TL309>. Similarly, active analogue (383) and other derivatives have been patented <87SAP8606273>.



Several prostaglandin analogues of PGI_2 incorporate benzofurans, for example (384) and (385) <87JCS(P1)1825, 88MI 208-09>. Another such derivative, beraprost (386), is an inhibitor of platelet aggregating factor <82EUP100449>, and analogue (387) has antiulcer activity <87EUP232776>.

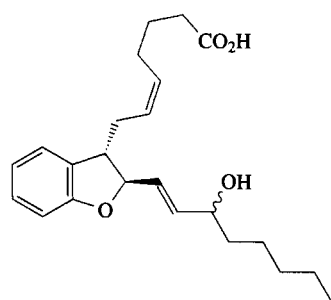
A series of Gossypol derivatives were tested for inhibitory activity of aldose reductase. Lactone (388) showed the best inhibition <91JMC3301>. Clofurac (389) has analgesic, antipyretic activity and inhibits blood platelet aggregation, edema, and inflammation <76GEP(O)2608697>.

The well-known phenanthrofurans such as codeine, morphine, and heroin (390; R = Me, OH, and CH_3CO , respectively) are very important narcotic analgesics, heroin being famous for its abuse. Many derivatives have been developed as antinarcotics and/or appetite suppressants. Examples are (391) <92JAP(K)04342529> and (392) <87JAP(K)62283976>. The morphine-like compound (393) had a high affinity for opiate receptors <89EJM385>, while (394) had analgesic properties and was a narcotic antagonist <89URP869288>.

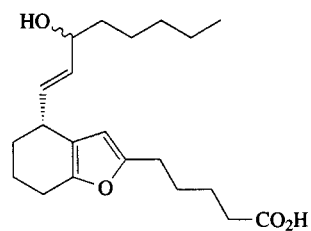
A variety of polycyclic benzofurans have anticancer activity. Dibenzofuranol (395) <88EUP257701>, quinone (396) <92JAP(K)04139177>, dichloride (397) <90MIP9008142>, and aminoalcohol (398) <91EUP447703>, have all been prepared as anticancer agents. Indole (399) has antitumor and antiviral activity <90KFZ44>. Hydroxylamine (400) is a lipoxygenase inhibitor, useful as an antiinflammatory <88USP4769387>.

Azabenzofuran (401) (as the hydrochloride) is a platelet-activating factor receptor antagonist and antitumor agent <91USP4992428>.

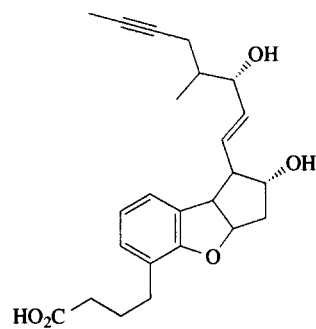
For an apparently simple compound, butylphthalide (402) exhibits a surprising range of activity. Along with its 4,5-dihydro derivative, it was found to have anticonvulsant activity <84MI 208-05> and it has been used in skin cosmetics <92JAP(K)0469314>. With its analogues, (402) has also been used as part of a stress-controlling fragrance <90JAP(K)02184683>, and it reportedly enhances the activity of



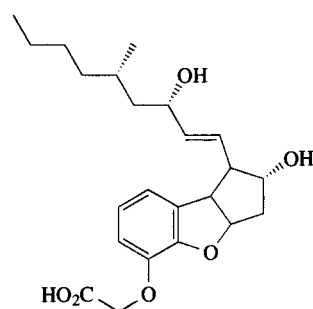
(384)



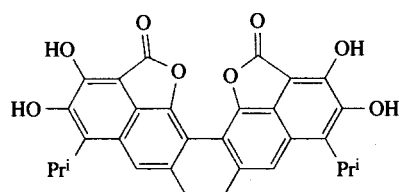
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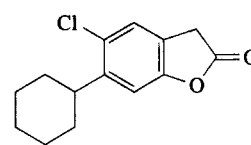
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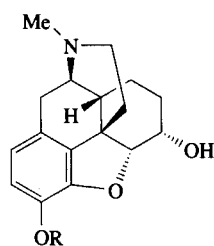
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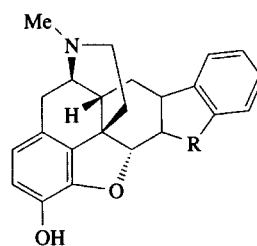
(388)



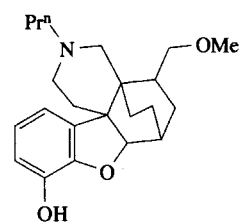
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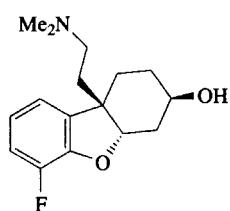
(390)



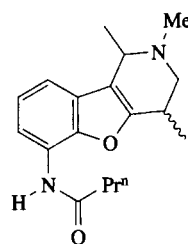
(391) R = NH or O



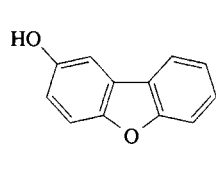
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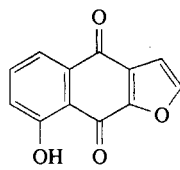
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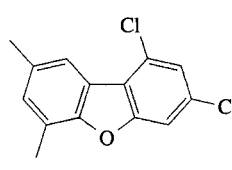
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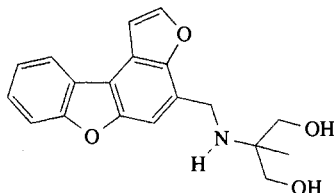
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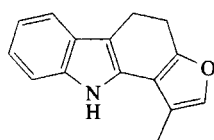
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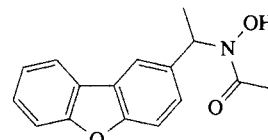
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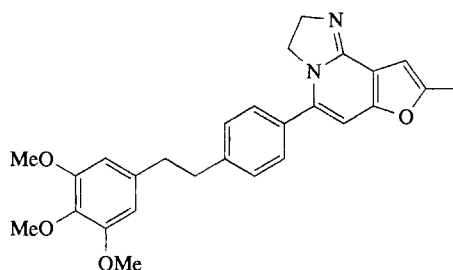
(398)



(399)



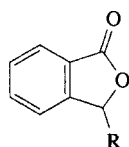
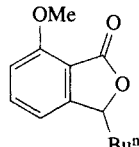
(400)



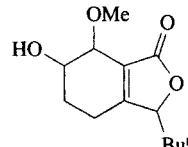
(401)

acetyl cholinesterase <93JAP(K)05247022>. Addition of a methoxy group to the 4-position gives a compound (403) which is a prostaglandin $F_{2\alpha}$ inhibitor <89JAP(K)01199958>. Tetrahydrophthalide (404) is useful in the treatment of Alzheimer's disease <92JAP(K)04334378>.

Another alkylphthalide (405) was an active ingredient in a hair tonic that increased the amount of anagen-phase hair in volunteers over a six-month period <91JAP(K)03135907>, while isoamylphthalide (406) is found in a dentifrice active against periodontal disease <92JAP(K)0469325>.

(402) R = Buⁿ

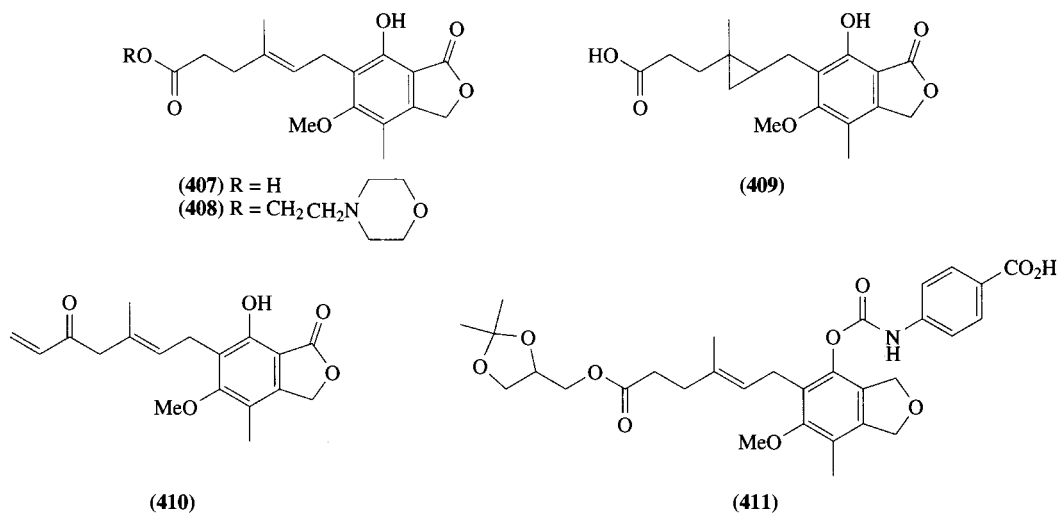
(403)



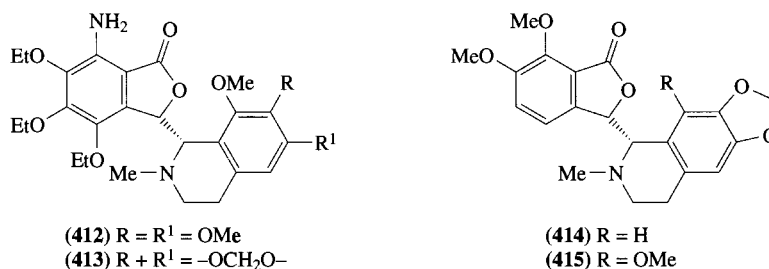
(404)

(403) R = (CH₂)₇Me(406) R = CH₂CH₂CHMe₂

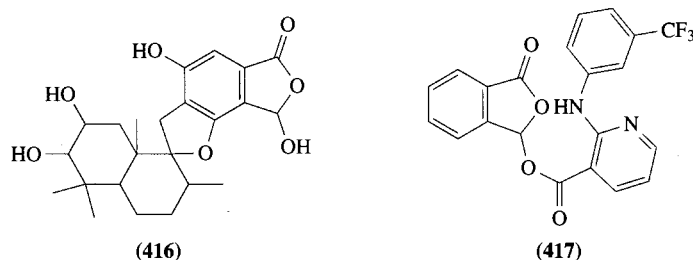
Mycophenolic acid (407), a phthalide produced by *Penicillium brevi-compactum*; *P. stoloniferum*, has antibiotic, antitumor, and immunosuppressive activity <69BRP1157099>. A derivative, Mycophenolate mofetil (408), has been applied to the problem of transplant rejection as an immunosuppressive agent <93ANY(685)309> and has been reported to have antiinflammatory and virucidal activity <88USP4727069>. Several side-chain variants of mycophenolic acid (409) have been prepared, and a study of their structure-activity relationships has appeared <90JMC833>. A very similar compound, vinyl ketone (410), was isolated from *Hericium erinaceum*. It was found to possess activity as a uterocervical cancer inhibitor <91JAP(K)03157379>. Several derivatives, such as (411), have been applied to the treatment of rheumatoid arthritis <87USP4686234>.



Altoqualine (**412**) and tritoqualine (**413**) are antihistamines and antinausea agents <59BRP873935> related to the alkaloids hydrastine (**414**) and noscapine (**415**).

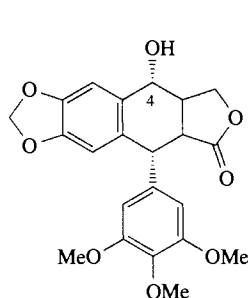


Lactol benzodifuran (**416**), a sesquiterpene isolated from *Stachybotrys* sp. K76, was found to be useful for the treatment of noninsulin-dependent diabetes <91JAP(K)03169811>. Lactol ester talniflumate (**417**) is an analgesic and antiinflammatory <78BEP858864>.

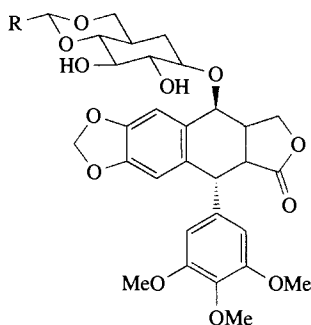


Podophyllotoxin (**418**) (sometimes referred to as VP-16), isolated from *Podophyllum peltatum*, and its diastereomers continue to be of substantial interest owing to their antineoplastic activity <B-80MI 208-01>. As a topical antiviral, podophyllotoxin was shown to be effective in the treatment of genital warts <87MI 208-05, 89MI 208-06>. Etoposide (**419**) <71SZP514578> and teniposide (**420**) <68SAP6607585> are semisynthetic derivatives that are also antineoplastics <68SAP6607585, 71SZP514578>. Podophyllotoxin and its esters have shown significant activity against P-388 lymphocytic leukemia <83JPS1158>. Many more derivatives of podophyllotoxin have been prepared. Hydroxyalkyl (**421**) <91JAP(K)03127792>, thioester (**422**) <92MI 208-07>, amino (**423**) <90MIP9009788>, and benzylamino (**424**)

<91JMC3346> derivatives are typical examples. In many instances, the compounds (421)–(424) showed greater activity than either compounds (419) or (420).

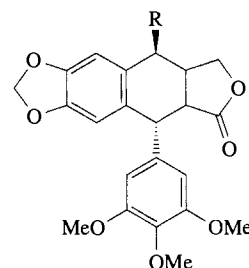


(418)

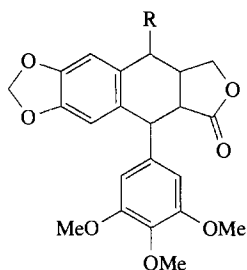


(419) R = Me

(420) R = thiophene-2-yl

(421) R = CH₂CH(OH)CH₂OH(422) R = S-(C=O)-cyclohexyl-NH₂(423) R = NHCH₂CH₂OH(424) R = NHCH₂C₆H₄-F (*para*)

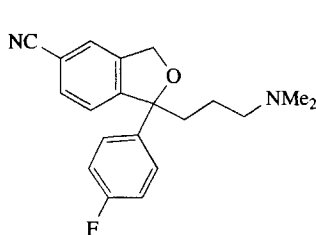
Justicidin A (425) and justicidin B (426) are known piscicidal agents <70BRP1178341>. Analogues have been patented for use in the treatment of osteoporosis <92JAP(K)04211609>.



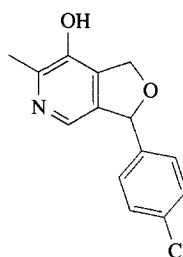
(425) R = OMe

(426) R = H

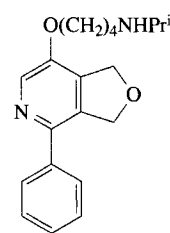
Phthalan citalopram (427) and its analogues are antidepressants <77GEP(O)2657013>. Cicletanine (428), an antihypertensive, was found to have diuretic activity in rats and was a hypolipemic in rabbits <82BEP891797>. Barucainide (429) is a novel antiarrhythmic agent <81GEP(O)3008522, 90MI 208-06>.



(427)



(428)

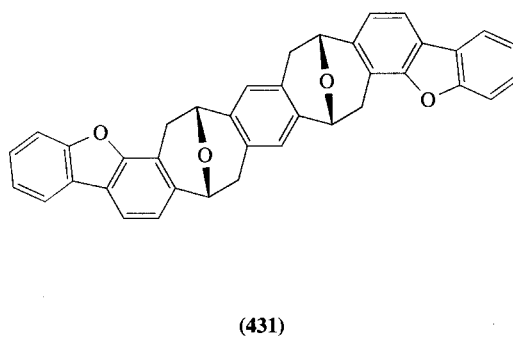
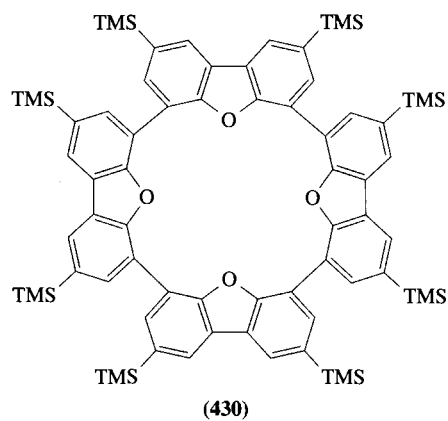


(429)

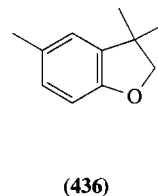
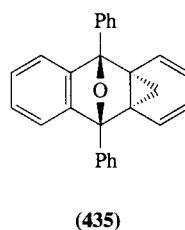
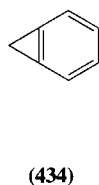
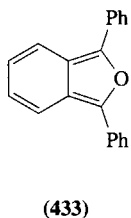
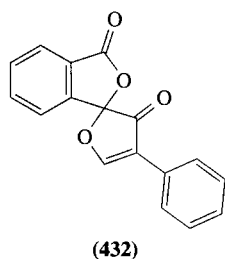
2.08.3.5 Miscellaneous

Cram has prepared a host system consisting of four linked dibenzofurans (430). One variation was found to form a complex with guanine in methanol <92JA10775>. Bisdibenzofuran (431), a derivative of Kagan's ether, is a molecular tweezer that binds to trinitrobenzene <90JA5655>.

The spirolactone fluorescamine (432) reacts with primary amines to form highly fluorescent products making it a useful analytical reagent for the fluorometric determination of primary amines <72JA5927>. Diphenylisobenzofuran (433) continues to be useful as a trap for singlet oxygen and reactive alkenes. An example is its reaction with cyclopropabenzene (434), which is postulated to



occur via a diradical mechanism to produce **(435)** <91TL593>. Benzofuran **(436)** has a musk odor and is used as a perfume component <90FRP2634203>.



2.09

Thiophenes and their Benzo Derivatives: Structure

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2.09.1 INTRODUCTION

Heterocyclic compounds are divided into heteroaromatic and heteroalicyclic derivatives. In heteroaromatic compounds (as in carbocyclic aromatics), each atom in the ring is in the same plane and each ring is associated with $(4n + 2)\pi$ -electrons.

Thiophene is an aromatic compound. Its structure can be assumed to be derived from benzene by replacement of two annular CH groups with sulfur. The sulfur atom in this five-membered ring acts as an electron donating heteroatom by contributing two electrons to the aromatic sextet and thiophene is thus considered to be an electron-rich heterocycle. The chemistry of thiophene and other five-membered heterocycles, for example pyrrole and furan, are consequently dominated by this effect.

The fact that the lone pair on sulfur contributes to the aromaticity is seen in the lower dipole moment of thiophene as compared to its saturated analogue tetrahydrothiophene (0.52 D vs 1.90 D) <72JA8854>. In thiophene, as in most of the other five-membered heterocycles and their saturated analogues, the dipole is directed from the ring towards the heteroatom. The only exceptions to this are pyrrole and *N*-alkylpyrroles where the nitrogen atom is the positive end of the dipole.

Although thiophene is aromatic and assumed to be derived from benzene, the bond length equivalence seen for the C—C and C—H bonds in benzene is not found here. In thiophene, as will be discussed later, the C—S and C—C bonds are all different in length as are the C—H bonds.

The numbering in thiophene (1) starts at the sulfur atom and continues around the ring. This numbering system will generally be followed throughout the text. The C₂ and C₅ carbon atoms are also designated as C_α; the C₃ and C₄ carbons as C_β. Substituted thiophenes are named similarly to substituted benzenes. Some of the more common radicals (2–5) are shown in Figure 1. Protonation of thiophene leads to three cationic species (6–8) none of which are aromatic. There are two dihydro derivatives, 2,3- (9) and 2,5-dihydrothiophene (10) and a single tetrahydrothiophene (11). Alternatively, (9) and (10) are also known as Δ²- and Δ³-dihydrothiophene or 2- and 3-thiolane where the number indicates the position of the double bond. The benzo derivatives benzo[*b*]- (12) and benzo[*c*]-thiophene (13) are considered analogues of naphthalene while dibenzothiophene (14) is related to phenanthrene.

Thiophene was first discovered in 1882 by Victor Meyer. It is a colorless liquid with a boiling point of 84.4°C (760 torr) and a melting point of –38.3°C <83KO(22)965>. Although it is generally thought of to have an odor similar to benzene, pure thiophene, when distilled from copper (II) chloride is practically odorless. It is highly flammable, moderately toxic, immiscible with water and soluble in organic solvents. Some of the physical properties of thiophene have been listed in Table 1.

Thiophene was first synthesized by Meyer <B-1888MI 209-01, 83KO(22)965> by ring closure of succinic acid with phosphorus pentasulfide. Although thiophene is found in deposits of lignite, coal, and crude oils, its extraction from these sources is not feasible. A summary on the investigation of thiophene and thiophene derivatives occurring in petroleum, oil shales, and coals has been covered by Galpern in *Thiophene and Its Derivatives* <85HC(44/1)325>. Laboratory procedures involve heating anhydrous sodium succinate with phosphorus trisulfide under a carbon dioxide stream. The crude thiophene is condensed, steam distilled, and purified <43OSC(2)578>. Industrial processes involve continuous vapor-phase techniques that use C₄ raw materials and sulfur compounds in the presence of metal oxide catalysts. For example, thiophene has been prepared in high yield from furan and hydrogen sulfide at 300–400°C <66GEP1228273>. Other C₄ precursors include but-1-ene, butadiene, and butanol, with carbon disulfide as the sulfur source <73GEP2225443>. These processes afford higher yields of thiophene by eliminating formation of thiophene tars, a problem generally encountered in some of the earlier methods. These latter processes are also used for the formation of 2- and 3-alkyl thiophenes.

In addition to being naturally present in coal tar and shale oil, thiophene derivatives have also been found in plants and fungi. In most cases, these compounds are derived from C₁₀ or longer conjugated acetylenic precursors. Thiophene formation is presumed to arise from addition of hydrogen sulfide (or its biological equivalent) to the C—C triple bond. A multistage process is proposed since thioenol ethers have also been simultaneously detected. A survey of naturally occurring thiophenes has been published by Bohlmann and Zdero <85HC(44/1)261>.

Thiophene has also found widespread use in the synthesis of biologically active species <91HC(44/4)397>. Since thiophene is structurally and electronically similar to benzene, thiophene analogues of biologically active benzene derivatives could possess similar activities. Additionally, the presence of a heteroatom could alter the metabolic effect and perhaps be a better therapeutic drug. Being an electron-rich aromatic system, thiophene is expected to be superior to benzene in

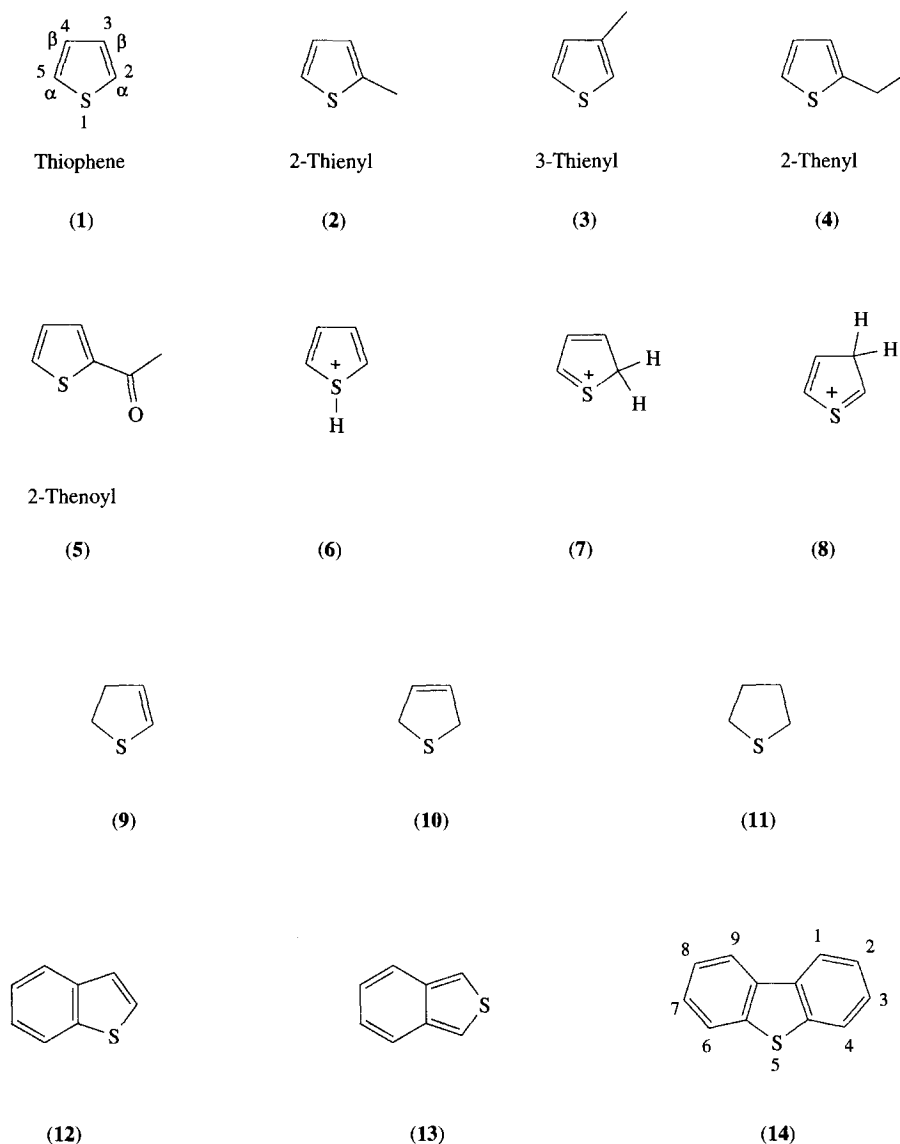


Figure 1 Thiophene and some common derivatives.

certain cases. Thus, biologically active thiophene derivatives contain thiophene either as the central ring or as part of a central fused ring system. Thiophene containing drugs have been used in the therapy of the central nervous system, metabolic, and infectious diseases and also as pharmacodynamic and agricultural agents.

Several books and reviews are available on the chemistry of thiophenes. Much of the work up to 1950 has been documented by Hartough (52HC(1), B-54MI 209-01). Over the past three decades, Gronowitz has either authored or edited a variety of articles dealing with thiophene. Some of them have appeared in the *Advances in Heterocyclic Chemistry* series, (63AHC(1)1) and supplemented periodically in *Organic Compounds of Sulfur, Selenium, and Tellurium* (B-72MI 209-01, B-75MI 209-01, B-77MI 209-01, B-79MI 209-01), and also in a five-part series entitled *Thiophene and Its Derivatives* in the Weissberger series *The Chemistry of Heterocyclic Compounds* (85HC(44/1), 86HC(44/2), 86HC(44/3), 91HC(44/4), 94HC(44/5)). Much of the work up to 1980 has been documented in *Comprehensive Heterocyclic Chemistry* (CHEC-I) and consequently, the work here concentrates on the chemistry of thiophene since then.

Table 1 Physical properties of thiophene <94MI 209-01>.

Physical property	Value	Physical property	Value
Melting point	-38.3°C	Critical temperature	579.4 K
Boiling point	84.4°C	Critical pressure	5.69 mPa
Flash point	-1.11°C	Critical molar volume	219 cm ³ mol ⁻¹
Dipole moment	0.55 D	Diamagnetic susceptibility	57.2 ($-\chi_m \times 10^6$)
Dielectric constant (16°C)	2.76	Enthalpy of fusion (-39.4°C)	-1.19 kcal mol ⁻¹
Refractive index	1.5289 ²⁰	Enthalpy of vaporization (25°C)	8.30 kcal mol ⁻¹
Density	1.6049 ^{20/4} g cm ⁻³		
Heat capacity	123.8 J mol ⁻¹ K ⁻¹	Vapor pressure (mm Hg)	Temperature (°C)
Surface tension (25°C)	30.68 mN m ⁻¹	10	-10.9
(50°C)	27.36 mN m ⁻¹	40	+12.5
Thermal conductivity (25°C)	0.199 W m ⁻¹ K ⁻¹	100	+30.5
(50°C)	0.195 W m ⁻¹ K ⁻¹	400	+64.7
(100°C)	0.186 W m ⁻¹ K ⁻¹	760	+84.4

The large number of reports pertaining to the synthesis of thiophene and its derivatives corroborates the continuing intrigue and importance of these compounds in biology, chemistry, industry, and medicine <88H(27)1731, 91FOR87, 91HC(44/4)397>.

Amongst the derivatives of thiophene, the chemistry of thiophenium salts and S,C- and S,N-thiophenium ylids has also been reviewed <89AHC(45)151> as has the chemistry of the thiophene 1-oxides, 1,1-dioxides, and sesquioxides <85HC(44/1)572>. The chemistry of benzo[b]thiophenes has been reviewed by Scrowston <81AHC(29)171> and there is a study by Gronowitz on the isomeric dithienothiophenes <94JHC641>.

Interest in thiophene has also been extended to the study of oligomers of thiophene and their derivatives. Interest in this arises due to its wide range of photobiological effects <91T8443, 93MI 209-01> and as alternatives to inorganic materials in the synthesis of a class of organic conducting polymers <B-86MI 209-01, 92JPS(A)1891>. These electron-rich conjugated oligomers have π -electron systems similar to the linear *cis,trans*-polyenes. These oligomers display high electrical conductivity in the oxidized form and possess nonlinear optical properties <B-86MI 209-01, B-87MI 209-01, B-91MI 209-01>. Polythiophene is attractive in that it is stable in air and when some of the hydrogens are substituted by alkyl or phenyl groups, it is soluble in most organic solvents <86SM169>. The interesting behavior of this class of conducting polymers has attracted many theorists and experimentalists to investigate and understand their electronic properties. Results of *ab initio* studies on some of these thiophene oligomers will be discussed briefly in the next section (see Section 2.09.2)

2.09.2 THEORETICAL METHODS

2.09.2.1 General

Molecular orbital (MO) calculations have been used to obtain properties of molecules, ions, and radicals, some of which include bond distances, bond angles, heats of formation, ionization energies and dipole moments. Early methods such as *Hückel Molecular-Orbital* (HMO) calculations <B-87MI 209-02> treated the σ and π electrons separately. The advent of powerful computers and the development of software packages has enabled MO calculations to be carried out by including all electrons in a molecule. These calculations are termed *ab initio* which actually means "from first principles" <B-85MI 209-01, B-86MI 209-02>. Initial interest in the *ab initio* study stemmed from its ability to include the sulfur *d*-orbitals in its calculations and determine to what extent these orbitals played a role in the reactivity and property of sulfur heterocycles. As we shall see later, not all calculations are significantly affected by the inclusion of the *d*-orbitals.

Cheaper and quicker methods involve semiempirical calculations. The CNDO method was first used by Pople *et al.* in 1965 <65JCP(43)S129, 65JCP(43)S136>. Dewar is credited with the development of the modified intermediate neglect of differential overlap (MINDO/3) method <75JA1285>; the modified neglect of diatomic overlap (MNDO) method <77JA4899> and the Austin model 1 (AM1) <85JA3902>. Since then, there have been many modifications of the above semiempirical methods.

Results from initial studies found in the literature have been covered in *CHEC-1*. In addition, an excellent review by Henriksson-Enflo <85HC(44/1)215> has also covered literature up to 1981; the reader is encouraged to consult them for complete details on prior work.

2.09.2.2 The Role of *d*-Orbitals on Sulfur

Initial MO calculations on thiophenes assumed that only the $3p_z$ -atomic orbital of sulfur contributed to the π -electron MO wave function <35JA2086> and thus treated it similar to its iso- π -electronic counterparts pyrrole and furan. The fact that thiophene is more aromatic than either of these two led Schomaker and Pauling <39JA1769> to believe that *d*-orbitals could participate in the hybridization. Bielefeld and Fitts later calculated the total energy of thiophene by employing a basis set in the LCAO approximation which included the $3d_\pi$ and $4d_\pi$ functions on sulfur <66JA4804> and compared the results with two other LCAO MO approximations. The first considered only the $2p_z$ orbitals on the carbon atom and the second included the $3d_{xy}$ and $3d_{yz}$ atomic orbitals on the sulfur atom. The Pariser–Parr–Pople (PPP) calculations indicate that the $3d_\pi$ -orbital participates to only a small extent. High level *ab initio* <70CC319, 72TCA171> calculations later showed that in actuality, the effect of the $3d$ -orbitals was negligible. The difference in the total energies calculated by these methods was attributed to the selection of the basis set which seems to be more important than the inclusion of $3d$ -orbitals.

Although the population of the $3d$ -orbital is very small, its inclusion in the *ab initio* calculation causes noticeable changes in the populations of the atoms, especially on the adjacent carbon atoms. Gelius *et al.* <72TCA171> showed the population of $3d$ -orbitals on sulfur to be 0.143 electrons in the $3d_\sigma$ -orbital and 0.038 in the $3d_\pi$ -orbital which are much smaller than the value of 0.24 and 0.138 electrons obtained by Clark from CNDO calculations <68T2663>. A comparison of the populations on sulfur and on the adjacent carbon atoms is shown in Table 2.

Table 2 Atomic populations on sulfur and adjacent carbon atoms in thiophene.

Method	Basis on sulfur	Population in 3d on S		Population in 2p on C _α		Ref.
		3d _σ	3d _π	2p _σ	2p _π	
PPP	$3p_z, 3d_{xz}, 3d_{yz}$		0.050			66JA4804
CNDO	$3s, 3p, 3d$	0.24	0.14	1.96	1.06	68T2663
<i>Ab initio</i>	Double ζ			2.135	1.109	72TCA171
	Double ζ, H_{2p}			2.076	1.105	72TCA171
	Double $\zeta, 3d$	0.143	0.038	1.976	1.085	72TCA171
	Double $\zeta, 3d, H_{2p}$	0.143	0.037	1.184	1.080	72TCA171

Depending upon the basis set, the gross charges on sulfur and on the other atoms vary to a much greater extent than the population. When no polarization function is included in the basis set, the charge on sulfur is found to be +0.482 <72TCA171>. This is reduced to +0.016 when the $3d$ -orbitals on sulfur are included and to +0.001 when the $2p$ -orbitals on hydrogen and the $3d$ -orbitals on sulfur are included. This decrease is larger than the corresponding $3d$ -population. The effect of the $3d$ function is to increase the populations in the sulfur $3s$ - and $3p$ -orbitals; thus it acts as a polarization function rather than as a valence orbital. This is in contrast to compounds where sulfur is bonded to more electronegative atoms such as in SO_4^{2-} and SF_6 <71TCA368, 71TCA590> where the $3d$ -level is stabilized and available for additional bonding. Table 3 compares the charges on the various atoms in thiophene as obtained from various calculations with basis sets that do, and do not, include the sulfur $3d$ -orbitals.

Total dipole moments depend on contributions from: (i) net atomic charge densities; (ii) atomic s - p polarizations obtained from mixing s - and p -orbitals on each atom; and (iii) atomic p - d polarizations obtained from the sulfur $3p$ - and $3d$ -orbitals. Thus quantum chemical methods such as INDO which do not include *d*-orbitals in their approximations would not compare favorably with experimental values obtained for compounds containing sulfur. Inclusion of the $3d$ -orbitals in the basis set usually leads to a lowering of the dipole moment. For example, *ab initio* calculations employing a double zeta basis set on sulfur, gives a dipole moment of 0.96 D which is lowered to 0.62 D when the basis set includes $3d$ -orbitals on sulfur and $2p$ -orbitals on hydrogen <72TCA171> which is much closer to the experimental value of 0.55 D <53JCS1622>. Dipole moments calculated by various methods are discussed in more detail in Section 2.09.2.5.

2.09.2.3 Total Energies

The stability of a molecule is usually determined by its total energy from which one can determine heats of formation, resonance energies, and localization energies. In most cases, *ab initio* methods

Table 3 Gross charges on the different atoms in thiophene as calculated by various methods.

Method	Basis on sulfur	Gross charges on the atoms					Ref.
		S	C _α	C _β	H _α	H _β	
PPP	3p _z	0.21	−0.06	0.04			66JA4804
	3p _z , 3d _{xz} , 3d _{yz}	0.16	−0.09	0.01			66JA4804
CNDO	3s, 3p	0.13	−0.05	−0.07	0.03	0.03	68T2663
	3s, 3p, 3d	−0.04	−0.06	−0.02	0.05	0.03	68T2663
<i>Ab initio</i>	Double ζ	0.48	−0.57	−0.18	0.27	0.24	72TCA171
	Double ζ, H ₂ p	0.49	−0.44	−0.06	0.15	0.11	72TCA171
	Double ζ, 3d	0.02	−0.25	−0.24	0.25	0.23	72TCA171
<i>Ab initio</i>	Double ζ, 3d, H ₂ p	0.00	−0.11	−0.12	0.12	0.10	72TCA171
	Minimal	0.14	−0.24	−0.17	0.17	0.26	72TL4165
	Minimal, 3d	0.04	−0.17	−0.16	0.16	0.15	72TL4165
	Minimal, 3d, 3s	0.03	−0.17	−0.17	0.16	0.15	72TL4165

have been used to determine the total energy of thiophene. It is imperative that all electrons be considered in the calculations, hence theories involving only π -electrons (HMO, PPP) or all-valence electrons (EHT, CNDO, INDO, etc.) cannot generally be employed. However, as shown by Dewar and co-workers <67JA3089, 69JCP(50)1262, 75JA1285>, some of these can be parameterized to give good energy values.

As discussed previously in Section 2.09.2.2, inclusion of the 3d-orbitals in the basis set fails to show any significant difference in the total energy. Clark <68T2663> obtained only a 1.77% energy lowering by including the 3d-orbitals in his calculations and 2.02% when the 4s- and 4p-orbitals were also included. Clark and Armstrong <70CC319> obtained an energy decrease of 74 kcal mol^{−1} when the 3d-functions on sulfur were included in the basis set. By contrast, Gelius *et al.* <72TCA171> obtained an energy decrease of 18.8 kcal mol^{−1} when they included the 3d-functions on sulfur and 14.4 kcal mol^{−1} when the 2p-functions on hydrogen were included. The two functions were found to be independent of each other. The total energy calculated by Gelius *et al.* was lower than that by Clark and Armstrong due to the fact that a larger set of contracted Gaussians were used in the former case. Thus here the basis set is more important than is the inclusion (or exclusion) of the 3d-function of sulfur.

A comparison of the total energy of thiophene obtained by various methods is given in Table 4. We see from the Table that CNDO calculations which use only the all valence electrons give higher energy values (less negative) than the *ab initio* methods which includes all electrons in its approximations. The total energy calculated by employing the spin-coupled approach is lower in energy than that obtained from SCF calculations by 37.5 kcal mol^{−1} (0.06 a.u.) <89JCS(P2)263>, a trend also observed with pyrrole and furan with energy lowerings of 39.4–43.7 kcal mol^{−1}.

Calculations at the STO–3G level on the protonation of thiophene and chlorothiophenes show energy lowerings of 232–250 kcal mol^{−1} <84BCJ1312>. The calculations show that a chlorine atom at the β -position (**16**) lowers the energy to a greater extent than at the α -position (**15**) while protonation at the α -position (**7**), lowers the energy 11.7 kcal more than at the β -position (**8**), consistent with previous study <83BCJ2208> where only α -protonated products have been observed. Thus protonation of 2-chloro-, 2,3-dichloro- and 2,5-dichlorothiophene occurs at the 5-position only. With two chloro substituents, the 2,4-derivative is the most stable one, whilst amongst the cations, the 2-*H*,3,5-dichlorothiophenium cation (**17**) is the most stable one.

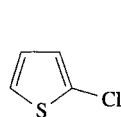
Quantum chemical investigations have also been carried out on the 1,2-proton shift in protonated five-membered heterocycles <84CHE966>. As the proton travels round the ring, the intermediates are either at the local minima or in a transition state and consequently, the change in energy can be calculated. Calculations for thiophene by the SCF–MO–LCAO method in the MINDO/3 approximation and nonempirically in the OST–3GF* basis both indicated protonation at the α -position to be more favorable, in line with results observed above.

2.09.2.4 Charge Distribution

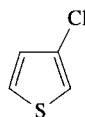
In Section 2.09.2.2, it was reported that, although inclusion of the sulfur 3d-orbitals in the calculations showed negligible changes in the 3d_σ and 3d_π populations on the sulfur atom, the gross charges on all atoms showed large variations. Table 5 lists the variations in charges on sulfur and

Table 4 Total energy of thiophene obtained via various methods.

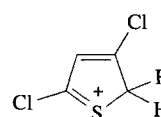
Method	Basis on sulfur	Total energy (a.u.) ^a	Ref.
CNDO	3s, 3p	−507.148	68T2663
CNDO	3s, 3p, 3d	−516.149	68T2663
CNDO	3s, 3p, 3d, 4s, 4p	−517.338	68T2663
<i>Ab initio</i>	Minimal	−550.417	70CC319
	Minimal, 3d	−550.535	70CC319
<i>Ab initio</i>	Double ζ	−550.923	72TCA171
	Double ζ , H ₂ p	−550.946	72TCA171
	Double ζ , 3d	−550.976	72TCA171
	Double ζ , 3d, H ₂ p	−550.999	72TCA171
<i>Ab initio</i>	Minimal	−550.075	72TL4165
	Minimal, 3d	−550.144	72TL4165
<i>Ab initio</i>	Minimal, STO-3G	−545.087	85JCS(P2)97
<i>Ab initio</i>	Minimal, STO-3G	−545.092	77G55, 79JA311
<i>Ab initio</i>	Minimal, STO-3G	−545.092	84BCJ1312
<i>Ab initio</i>	Split valence, 4-31G	−550.599	80JCC348
<i>Ab initio</i>	4-31G + d	−550.662	82JA1375
<i>Ab initio</i>	Spin-coupled	−546.497	89JCS(P2)263
	SCF	−546.437	89JCS(P2)263
<i>Ab initio</i>	SCF	−551.064	82MP649
<i>Ab initio</i>	HF	−551.077	76JES179
<i>Ab initio</i>	3-21G	−548.473	89JST(186)101
	3-21G*	−548.515	89JST(186)101
<i>Ab initio</i>	TZVP-SCF	−551.361	92ZN(A)203
	TZVP-CI	−551.922	92ZN(A)203

^a 1 a.u. = 2625 kJ mol^{−1} = 627 kcal mol^{−1}.

(15)



(16)



(17)

on the carbon atoms obtained when the 3d-orbitals were, and were not, included in the calculations. From the Table we see large variations in the charges on the atoms from one calculation to another and even within the same methods themselves. For example, by HMO calculations, the charge on sulfur varies from 0.12 to 0.75 while in the PPP method, the charge varies from 0.09 to 0.36, and between 0.0 to 0.49 by *ab initio* methods.

Table 5 Variation in gross charges on sulfur and on carbon atoms in thiophene as calculated by various methods.

Method	Variation in gross charges			Ref.
	S	C _α	C _β	
HMO	0.12 to 0.75	0.02 to −1.13	0.07 to −0.22	35JA2086, 58JA5543 52BCJ179 61JCP(34)389
PPP	0.09 to 0.36	−0.02 to −0.13	0 to −0.07	63T157, 70ACS23 65CJC1569, 70TCA49
CNDO	0.13 to −0.05	−0.05 to −0.06	−0.01 to −0.07	68T2663
CNDO/2	−0.06	−0.005	0.038	89CHE874
MNDO	0.10	−0.06 to −0.15	−0.004 to −0.09	86IJQ1599, 89CHE874
<i>Ab initio</i>	0 to 0.49	−0.07 to −0.57	−0.05 to −0.24	72TCA(27)171, 76JES179, 72TL4165, 79JA311, 77G55, 80JCC348

For the *ab initio* methods even a change in the basis set causes large changes in the atomic charges although, if a large basis set is used, charges calculated with $\langle 72TCA171 \rangle$ and without $\langle 76JES179 \rangle$ d-orbitals are about the same. There is, however, a trend that is observed in almost all cases. Most

calculations show the sulfur atom to bear a positive charge and the carbons to bear a negative charge with the α -carbon atom to be a bit more negative.

Calculations using the CNDO method gave a positive value for the sulfur atom (0.136) when the d -orbitals of sulfur (method I) were not included in the calculations $\langle 68T2663 \rangle$. When the basis set included the sulfur $3d$ -orbitals (method II) the value calculated was -0.0358 , and -0.0514 when the $4s$ - and $4p$ -orbitals were also included (method III). Calculations via method I show the sulfur atom to be a σ electron acceptor and π electron donor. Since the latter effect is greater, the net charge is positive. The σ inductive effect is felt on the adjacent carbon atoms rendering them σ electron deficient while the C_β carbon atoms are relatively unaffected and thus approximately neutral with respect to the σ electrons. Inclusion of the d -orbitals in the calculations (method II) leads to a net negative charge due to the drift of the σ and π electrons towards the sulfur atom. The σ charge mainly results from the $3p_x$, $3p_y$, and $3d_{xy}$ orbitals with smaller contributions from $3d_{x^2-y^2}$ and $3d_z$, implying that the d -orbitals are at least as important in σ bonding as in π bonding. Further changes in the orbital populations due to the inclusion of the $4s$ - and $4p$ -orbitals (method III) are very small.

Charges on five-membered heterocycles calculated using the MNDO approximation are generally greater than those by CNDO/2 $\langle 89CHE874 \rangle$. For *N*-methylpyrrole, the charge on the nitrogen atom differs by a factor of 5–6 while in thiophene, the two methods differ not only in value but also in sign. By comparison, CNDO calculations without the $3d$ -orbitals affords a positive value. Similarly, the charge on the β -carbon atom calculated by the CNDO/2 is found to be $+0.038$, contrary to the other methods where the charges are negative.

2.09.2.5 Dipole Moments

The experimental value of the total dipole moment as measured in the gas phase is found to be 0.550 ± 0.040 D $\langle 53JCS1622 \rangle$. In sulfur-containing compounds, the p - d polarization also contributes to the total dipole moment value. Thus depending on the method employed, a large variation is expected for thiophene (see Table 6).

Table 6 Dipole moments of thiophene obtained from various calculations.

Method	Basis on sulfur	Dipole moment (D)	Ref.
Experimental		0.55	53JCS1622
HMO		2.50	52BCJ179
PPP		0.50	63T157
	$2p$	-0.18	66JA4804
	$2p, 3p, 3d$	0.68	66JA4804
	$2p, 3p, 3d, 4p$	0.72	66JA4804
	$3d$	0.63	87CHE454
CNDO	$3s, 3p$	0.84	68T2663
	$3s, 3p, 3d$	2.26	68T2663
	$3s, 3p, 3d, 4s, 4p$	1.84	68T2663
CNDO/S	$3s, 3p, 3d$	1.23	85CPB3077
CNDO/2	$3s, 3p, 3d$	0.42	83JST(94)115
CINDO/SHIFT/UV	$3s, 3p$	1.94	83JST(94)115
	$3s, 3p, 3d$	1.78	83JST(94)115
	$3s, 3p, 3d, C_{3d}$	1.24	83JST(94)115
INDO/S		2.71	83JST(94)115
MNDO		0.90	83MI 209-01
		0.90	86IJQ1599
MINDO/3		2.22	80IJQ797
MP2	6-31G(+ sd + sp)	0.45	92JPC(96)7301
<i>Ab initio</i>	Double ζ	0.96	72TCA171
	Double ζ , H_{2p}	0.97	72TCA171
	Double ζ , $3d$	0.61	72TCA171
	Double ζ , $3d$, H_{2p}	0.62	72TCA171
	HF	0.65	76JES179
	Split valence, 4-31G	0.38	80JCC348
	Minimal	-0.29	82MP649
	Minimal, $3d$, H_{2p}	0.07	82MP649
	Minimal, $3d$, H_{2p} , C_{3d}	-0.07	82MP649
	3-21G	1.34	89JST(186)101
	3-21G*	0.75	89JST(186)101
	SCF/6-31G(+ sd + sp)	0.91	92JPC(96)7301

The fact that dipole moments are generally overestimated at the Hartree–Fock level is shown by the relatively high value of 1.34 D obtained by employing the 3–21G basis set <89JST(186)101>. When the *d* polarization function on sulfur is included (the 3–21G* basis set), the value of the dipole moment drops to 0.75 D. *Ab initio* calculations that are closest to the experimental value are those obtained by Gelius *et al.* <72TCA171> (0.61 D) and von Niessen *et al.* <76JES179> (0.65 D). The slight overestimation in the latter case is expected when wave functions calculated with a double ξ basis set do not use polarization functions. Amongst the semiempirical methods, calculations by the PPP method <63T157, 87CHE454> are generally in good agreement with the experimental value.

The HMO method <52BCJ179> and MINDO/3 <80IJQ797> calculations gave very high dipole moments. For the MINDO/3 method, when the *d*-orbitals are neglected, the large polarization term causes an overestimation of the total dipole moment. Inclusion of the 3*d*-orbitals in the basis set should show improvement in the value. A similar overestimation is also observed in the MNDO method for thiophene and for a variety of other sulfur compounds which arises due to the non-inclusion of the 3*d* atomic orbitals <83MI 209-01, 86IJQ1599>.

CNDO calculations gave the best values when the *d*-orbitals were excluded from the basis set <68T2663>. The dipole moment of 2.26 D obtained when the sulfur *d*-orbitals are included is lowered to 1.84 D when the 4*s* and 4*p* are also included and to 0.895 D when only the 3*s* and 3*p* are taken into account. The larger values calculated by the PPP method <66JA4804> when the sulfur 3*d*- or the 4*p*-orbitals are included is not surprising, since inclusion of the sulfur 3*d*-orbitals in the calculations leads to a drift of the σ and π electrons towards the sulfur atom (see Section 2.09.2.4). As a result, μ_π becomes smaller in magnitude and less negative making the net dipole moment more positive. For the CNDO method, inclusion of the sulfur 3*d*-orbital changes the contribution from the charge density from -1.153 D to $+0.582$ D. Although the *p*–*d* polarization factor is negative, it is still not large enough to offset the change and hence the large value of 2.26 D in this case. When the *d*-orbitals are excluded from the basis set <66JA4804>, a negative value is obtained indicating the dipole is directed away from the heteroatom.

A comparison of results from INDO/S and CNDO/2 calculations <83JST(94)115> show that the value concerning the charge densities are nearly equal in both cases and in good agreement with the experimental value. For thiophene, contributions from the *s*–*p* and *p*–*d* polarizations are opposite in sign and much higher than the charge density. However, since the INDO/S approximation does not include the sulfur *d*-orbitals, there is no contribution from the *p*–*d* polarization function and thus the net dipole moment calculated is much larger than the 0.412 D obtained from CNDO/2. From CINDO/SHIFT/UV (the spectroscopic version of CNDO/2) calculations, the results obtained are generally high, due to the large difference in the two polarization contributions. The dipole moment calculated from charge densities alone (0.40 D) is in good agreement with the experimental value. Inclusion of *d*-orbitals on both sulfur and carbon reduces the dipole moment from 1.935 D to 1.236 D, but this is still much higher than that obtained by experimental or by some of the other methods. However, one must realize that the CINDO/SHIFT/UV method is mainly parameterized for spectroscopic properties such as electron transitions.

With the diffusely polarized 6–31G(+*sd* + *sp*) basis set, a better correlation is obtained with MP2 values than with SCF values <92JPC7301>. With this basis set, the MP2 level tends to overestimate the experimental value for oxygen but underestimates sulfur and selenium. The SCF overestimates in all cases.

Brouckère *et al.* <82MP649> have obtained satisfactory results for the dipole moments of furan, pyrrole, and dihydrothiophene from *ab initio* SCF calculations. For thiophene, the value differs considerably. With no polarization function, or when an extra *d*-function on sulfur is included, negative values are obtained. The SCF calculated dipole moment is sensitive to the basis set used. For example, in this case, the dipole moment as calculated without the *d*-function on carbon was found to be $+0.066$ D while that using a smaller basis set and no carbon *d*-function gave a value of 0.62 D <72TCA171>, which is in close proximity to the experimental value. Reasonable results can be obtained as long as the basis set is not too large.

2.09.2.6 Orbital Energies-Ionization Potentials

Orbital energies have generally been used for predicting and assigning ionization potentials. As a general rule of thumb, the orbital energy can be (according to Koopmans' theorem <33MI 209-01>) assumed to be the negative of the ionization potential, i.e., $\varepsilon = -\text{IP}$. The photoelectron spectrum

of thiophene has been studied by Eland $\langle 69IJM471 \rangle$ and by Derrick *et al.* $\langle 71IJM(6)177 \rangle$ and x-ray electron spectroscopic studies by Gelius *et al.* $\langle 72TCA171 \rangle$. The assignments by Derrick *et al.* were made based on experimental data, comparison of spectra with similar compounds and on extended Hückel and *ab initio* calculations but the order has been questioned. The assignments of the first seven bands made by Derrick *et al.* $\langle 71IJM(6)177 \rangle$ were of the order $1a_2(\pi)$, $2b_1(\pi)$, $1b_1(\pi)$, $6a_1$, $4b_2$, $5a_1$, $3b_2$. *Ab initio* calculations $\langle 72TCA171, 76JES179 \rangle$ are generally in good agreement with the experimental values as are semiempirical, although the latter could also be due to the choice of parameters selected. A selection of recent references and some key earlier references dealing with the application of semiempirical and *ab initio* methods for the calculations of ionization potentials and electron spectra is given in Table 7.

Table 7 References to various semiempirical and *ab initio* calculations of ionization potential for thiophene and its benzo derivatives.

Method	Thiophene	Benzothiophene	Dibenzothiophene
Experimental	69IJM471, 79CRV77, 71IJM(6)177, 82JCS(P2)539	56JCS3438, 69IJM471	56JCS3438, 73JCS(F2)1155
ETH	71IJM(6)177		
PPP	66JA4804		
CNDO	68T2663, 76JCP(64)3021, 74TCA(33)19, 83JST(105)375		
CNDO/S	74TCA19, 85CPB3077, 83ACH97, 86IZV2251		
INDO/S	83JST(105)375	83JST(105)375	83JST(105)375
MNDO	83JCC84, 86IJQ1599, 85JCS(P2)97		
<i>Ab initio</i>	69BCJ933, 72TCA171, 76JES179, 85JCS(P2)97, 70CC319, 75JCS(P2)974, 83JST(105)375, 89JST(186)101	83JST(105)375	83JST(105)375

In most cases, the assignments of the first two orbitals are unambiguous. The band at 12 eV initially assigned by Derrick *et al.* to be the $2b_1(\pi)$ band was later shown to arise from σ orbitals. *Ab initio* calculations $\langle 69BCJ933, 75JCS(P2)974, 76JES179 \rangle$, while supporting the existence of the σ orbitals, generally differ on the amount of σ orbitals present between the π_1 and π_2 orbitals. Thus while for Gelius *et al.* the order was $1a_2(\pi) < 2b_1(\pi) < 6a_1(n) < 4b_2 < 1b_1(\pi)$, calculations by von Niessen *et al.* $\langle 76JES179 \rangle$ indicated only the $6a_1$ orbital to lie between the two $b_1(\pi)$ orbitals. Subsequent experiments observed the He^{II}/He^I changes in the photoelectron spectrum, and were able to reassign the band at 12.5 eV to that from the $1b_1(\pi)$ orbital $\langle 82JCS(P2)539 \rangle$. Based on the results observed here, the ordering is assigned as $1a_2(\pi)$, $2b_1(\pi)$, $6a_1(n)$, $1b_1(\pi)$, $4b_2$, $5a_1$, $3b_2$ which is in agreement with that of von Niessen *et al.* $\langle 76JES179 \rangle$. *Ab initio* calculations obtained by employing the STO-3G basis set tend to underestimate the first few ionization potentials.

CNDO calculations show that the MO energies are not affected by the inclusion of the *d*-orbitals $\langle 83JST(105)375 \rangle$. A modified version of the CNDO/S method used to interpret the UPS spectra of pyrrole, furan, and thiophene show there to be only one σ level which is strongly localized on the sulfur atom and is thus an *n*-type $\langle 83ACH97 \rangle$. Calculations were obtained by three methods; the first excluded the *d*-orbitals, the second included the sulfur *d*-orbitals, while the third included both the sulfur and carbon *d*-orbitals. The values are in good agreement and the assignments the same in all three cases indicating that the *d*-orbitals on sulfur do not affect the orbital energies to any significant extent as they do some of the other molecular properties; a trend also observed by Poirier *et al.* $\langle 89JST(186)101 \rangle$. A small decrease in the ionization energy observed in going from the 3-21G to 3-21G* basis set indicates the negligible effect of the *d*-orbitals in ionization potential calculations in contrast to some of the other electronic properties.

MINDO/3 calculations $\langle 80IJQ797 \rangle$ compare relatively well with the experimental values and correlate better than those obtained by Hartree-Fock *ab initio* calculations $\langle 76JES179 \rangle$. MINDO/3 does tend to underestimate the binding energy of upper level σ orbitals, but the other ionization potentials are calculated to within 0.5 eV of the experimental values and from *ab initio* calculations obtained via the many-body approach. The CNDO/S and INDO/S calculations give results for the low binding energy region but tend to overestimate the high binding region. For the CNDO/2 and INDO methods, the eigenvalues need to be corrected when used for photoelectron calculations.

2.09.2.7 Excitation Energy–UV Spectra

Quantum chemical calculations on the electronic spectrum of compounds are generally carried out by separate calculations on the ground state and on each excited state. Most of the initial calculations on thiophene have been carried out by the PPP method, although calculations by CNDO and INDO have also been done. Results obtained prior to 1981 are discussed in detail in the review by Henriksson-Enflo <85HC(44/1)215>.

In most of the earlier calculations, the *d*-orbitals on sulfur were omitted. Although the values differ due to the various approximations and parameters employed, most results assign the weak peak at ca. 5.33 eV to the in-plane polarized $\pi \rightarrow \pi^*$ transition and the band at 5.62 eV to the out-of-plane $\pi \rightarrow \pi^*$ transition. The strong peak observed at 6.6 eV in the experimental spectrum is assumed to consist of two transitions, one of which is in-plane and the other, an out-of-plane transition. Low lying triplets of A_1 and B_1 symmetries have also been found in the calculations.

Inclusion of the sulfur *3d*-orbital helps in lowering the $^1A_1$ and $^1B_1$ transitions of the second band from 7.1 and 7.3 eV to 6.9 and 6.6 eV, respectively, <66JA4804> which is closer to the experimentally calculated value to 6.6 eV <85HC(44/1)215>. However, in both PPP <66JA4804> and in the CNDO <74TCA19> methods, the presence of the *d*-orbitals in the calculations leads to changes in the ordering of the assignments and thus do not agree any more with the experimentally observed polarizations. PPP calculations for the first band give values of 5.7 and 5.2 eV for the singlet $^1A_1$ and $^1B_1$ transitions. The difference in the two drops to 0.1 eV when both the *3d*- and *4p*-orbitals on sulfur are added to the basis set, but the assignments are reversed. When only the *3d*-orbital is included, assignments are the same but the energies drop to 5.0 and 4.9 eV, respectively. With the CNDO method <74TCA19>, from calculations obtained without the *d*-orbitals, the transition with the lowest energy (ca. 5.4 eV) was assigned to the $^1B_2$ transition, while calculations that included the *d*-orbital of sulfur assigned the value at 5.0 eV to an $^1A_1$ transition.

PPP calculations also show four triplet states for thiophene. When only the *3p*-orbital is employed in the basis set, the assignments are 3.3, 4.0, 5.4, and 6.0 eV which correspond to the $^3B_1$, $^3A_1$, $^3A_1$, and $^3B_1$ transitions, respectively. When the *3d_{xy}* and *3d_{yz}* orbitals are included, the four transitions are calculated to be at 3.2, 3.1, 5.5, and 5.7 eV, respectively. Inclusion of the *4p_z* orbital alters the values to 4.0, 4.0, 5.5, and 5.8 eV. The two lowest energies are extremely close to the experimental values of 3.90 and 3.96 eV obtained by excitation measurements <52MI 209-01>.

The CINDO/SHIFT/UV method <83JST(94)115> which is the spectroscopic version of the CNDO/2 method, has been used to investigate the function of *d*-orbitals. Inclusion of the sulfur *3d*-orbitals and sulfur and carbon *d*-orbitals in the calculations does not affect the lower energy transitions but tends to lower the higher energy transition. The pattern observed from the CNDO/S calculations with and without the *d*-orbitals are similar to those obtained elsewhere <83ACH97>. When the *d*-orbitals are excluded from the calculations, the energies of the first three $\pi \rightarrow \pi^*$ transitions were found to be comparable to those calculated by the INDO/S and CINDO/SHIFT/UV methods <83JST(94)115>. However, in the latter case, the fourth absorption band is calculated to be about 0.5 eV lower in energy than the experimental value. Transition energies of heterocyclic compounds containing the second-row elements which are calculated by the CNDO/S method show better correlation with experimental values when the *spd* basis set is employed than with those that exclude the *d*-orbitals <85CPB3077>.

Amongst the methods investigated, the results obtained by the CNDO/S <85CPB3077> and INDO/S <83JST(94)115> methods coincide the best with experimental values. CNDO/SHIFT/UV calculations afford good interpretation of the optical spectrum for thiophene and its benzo analogues. The calculations also show that the *d*-orbital contributions are negligible since the low energy transitions mainly arise from the *p*-atomic orbitals.

Interest in the application of heterocyclic oligomers in various fields has resulted in computational chemistry playing a major role in predicting whether particular oligomers could possess the required properties or not. The PPP model with configuration interaction (PPP-CI) has been used to predict the long wavelength excitation energies, that is, for estimating band gaps (E_g) for several planar oligomers including those of thiophene and benzo[*c*]thiophene <87MM2023>. There is good correlation between the calculated values and experimental data, although they are slightly overestimated. This is encouraging since it enables prediction of E_g for polymeric cases of hypothetical infinite chain length. Although the PPP-CI method has limitations, it is suitable for calculating π -symmetry electron transitions for conjugated systems, it is readily available and provides an inexpensive first approximation for experimentalists interested in the design and synthesis of polymers possessing certain properties.

2.09.2.8 Vibrational Frequencies

The vibrational spectrum of a compound is usually calculated by harmonic force field calculations <B-55MI 209-01>. For thiophene, frequencies obtained from these calculations <69ACS3139, 69ACS3407, 69JSP(31)451> are in closer proximity than those by MINDO <77JA1685> and vary from experimental frequencies by less than 20 cm⁻¹ (see Table 8).

Table 8 Experimental and theoretical vibrational frequencies (cm⁻¹) of thiophene.

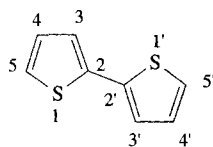
Assignment	Exptl <65SA689>	Force field <69ACS3139>	MINDO <77JA1685>	MP2 <88JPC1739>
A ₁ C—H str	3126	3127	3561	3328
C—H str	3098	3104	3501	3302
C=C, C—C, ring str	1409	1427	1677	1471
C=C str, CCH bend	1360	1364	1348	1420
CCH bend in-plane	1083	1072	1058	1111
CCH bend C—C str	1036	1030	988	1079
C—S str	839	844	778	885
Ring def. in-plane	608	642	460	628
A ₂ C—H wag out-of-plane	898	893	802	822
C—H wag out-of-plane	683	698	712	596
Ring def. out-of-plane	565	565	435	516
B ₁ C—H str	3125	3129	3558	3325
C—H str	3086	3089	3490	3287
C=C, ring str	1504	1532	1729	1542
CCH bend in-plane	1256	1254	1100	1296
CCH bend in-plane	1085	1069	970	1114
C—S str	872	857	774	909
Ring def. in-plane	763	745	605	780
B ₂ C—H wag out-of-plane	867	865	795	768
C—H wag out-of-plane	712	712	699	680
Ring def. out-of-plane	452	450	315	447
Assignment	SCF <69ISM3491>	SCF <92JST(259)181>	ACPF <92JST(259)181>	QCFF/PI + CISD <94JCP(100)2571>
A ₁ C—H str	3125	3130	3135	
C—H str	3092	3099	3095	
C=C, C—C, ring str	1421	1422	1415	1420
C=C str, CCH bend	1359	1356	1361	1352
CCH bend in-plane	1077	1077	1088	1089
CCH bend C—C str	1023	1027	1032	1006
C—S str	829	833	837	825
Ring def. in-plane	605	604	603	556
A ₂ C—H wag out-of-plane	908	901	878	1092
C—H wag out-of-plane	703	697	726	846
Ring def. out-of-plane	550	575	564	491
B ₁ C—H str	3123	3126	3132	
C—H str	3078	3085	3083	
C=C, ring str	1504	1504	1499	1536
CCH bend in-plane	1262	1262	1251	1310
CCH bend in-plane	1076	1074	1084	1061
C—S str	870	865	872	936
Ring def. in-plane	757	761	753	677
B ₂ C—H wag out-of-plane	877	878	873	1086
C—H wag out-of-plane	687	692	696	728
Ring def. out-of-plane	452	446	451	398

Harmonic vibrational frequencies have also been calculated by *ab initio* methods at the MP2 level with a double ζ basis set with one set of polarization functions (DZP) <88JPC1739>. Although the correlation is better than with those from MINDO calculations, the results are not nearly as good as those from force field calculations. For the high-frequency in-plane stretches, the theoretical values are about 6.5% higher. At the other end of the spectrum, the difference is smaller which is partly due to the anharmonicity of these modes. At the MP2 level the out-of-plane vibrations for both A₂ and B₂ symmetry are lower than the experimental frequencies obtained by gas- and liquid-

phase IR spectra and liquid Raman spectra <65SA689>. This is attributed to the fact that the prediction of harmonic frequencies for bending modes are lower than the experimental fundamentals for aromatic systems. Flexible basis sets which include polarization functions could circumvent this problem.

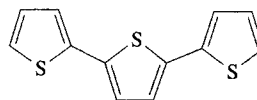
Systematic *ab initio* studies were carried out with two basis sets on the vibrational spectra of thiophene and its oligomers <92JST(259)181>. The first basis set was applied at the SCF level and the second at the averaged couple pair functional (ACPF) level. For thiophene, the latter showed an excellent agreement with the experimental values <65SA689, 66JSP(19)188> with deviations of up to 10 cm^{-1} for the in-plane frequencies (see Table 8).

The computed harmonic force fields for thiophene and its oligomers have been scaled as well. The same SCF scaling factors that were used for the thiophene, were used for 2,2'-bithienyl (18) and its oligomers <92JST(259)181>. For bithienyl, the frequencies of the three conformers, for example *syn*, *anti-gauche*, and *anti*, have also been calculated and compared with the solid-state Raman and IR frequencies <90SM299, 91JCP(94)4637>. Previous vibrational frequency calculations have also been studied at the SCF level by Cui *et al.* <91SM3491> but only the minimal basis set (STO-3G) employed. The differences in frequency between the *anti* and the *anti-gauche* conformers are less than 5 cm^{-1} in most cases <92JST(259)181> which is not unexpected since there is only a small energy difference between these two conformations. The difference between the *anti-gauche* and *syn* form is much greater. Although the calculations do not correlate extremely well, the agreement is acceptable. The scaling factors afford a good description of the vibrational spectrum and consequently have been used to determine the vibrational spectra of the antiplanar form of terthiophene (19) and polythiophene.



2,2'-Bithienyl

(18)



(19)

Negri and Zgierski <94JPC(100)2571> have employed semiempirical calculations involving the quantum consistent force field/ π electron configuration interaction singles diffraction (QCFF/PI + C-ISD) method and *ab initio* calculations using the complete active space self-consistent field (CASSCF) and HF/6-31G* methods. At higher frequencies, the in-plane modes calculated by the semiempirical method agree quite well with the experimental values. At lower frequencies, the discrepancies are larger. This is not unexpected since the C—C—C angle bending is usually not well described by QCFF/PI + CISD for five-membered rings <91CPL(179)131, 93JCP(99)3721>. Large differences are also observed for the out-of-plane C—C—H bending modes for A_2 and B_1 symmetries.

The QCFF/PI + CISD force field method has also been used to calculate vibrational frequencies of the in-plane modes for the deuteriated isotopomers of thiophene. The results compare relatively well with experimental values. Reliable results here have enabled application of this method to obtain vibrational frequencies for thiophene oligomers <94JPC(100)2571>. Vibrational frequencies for the bi-, ter-, and tetra-oligomers have been calculated by QCFF/PI + CISD, HF/3-21G*, and MP2/3-21G methods. Other theoretical studies on the vibrational dynamics of polythiophenes can be found in the following references <91JCP(94)957, 91JCP(94)965, 91JCP(95)4783>.

2.09.2.9 Bond Lengths and Angles

The optimized geometry of thiophene, its derivatives, iso- π -electronic heterocyclic partners, and other Group 16 containing heteroatoms has been calculated by both semiempirical and theoretical methods. Some of the calculations are discussed here and the results summarized in Tables 9 and 10.

Overall there is reasonably good agreement between the calculated bond lengths and the experimental results obtained by microwave spectroscopy <61JSP(7)58>. In most cases, the difference between the experimental and calculated bond lengths are less than 0.04 Å and the angles are within

Table 9 Bond lengths (in Å) of thiophene as calculated by various methods^a.

Method	C—S	C(2)=C(3)	C(3)—C(4)	Ref.
Experimental	1.714	1.370	1.423	61JSP(7)58
MP2	1.713(−1)	1.385(+15)	1.423(0)	88JPC1739
QCFF/PI + CISD	1.711(−3)	1.378(+8)	1.453(+30)	94JCP(100)2571
3-21G*	1.722(+8)	1.348(−22)	1.438(+15)	86PAC75
SCF/3-21G*	1.723(+9)	1.347(−23)	1.437(+14)	88JA4204
3-21G*	1.723(+9)	1.347(−23)	1.438(+15)	89JST(186)101
6-31G*	1.726(+12)	1.345(−25)	1.437(+14)	86PAC75
HF/6-31G*	1.726(+12)	1.345(−25)	1.437(+14)	94JCP(100)2571
4-31G + d	1.726(+12)	1.348(−22)	1.432(+9)	82JA1375
MM2	1.726(+12)	1.346(−24)	1.457(+34)	85TL2403
CI-SD(basis I)	1.728(+14)	1.363(−7)	1.434(+11)	92JST(259)181
SCF(basis I)	1.729(+15)	1.350(−20)	1.437(+14)	92JST(259)181
TZVP	1.729(+15)	1.344(−26)	1.435(+12)	92ZN(A)203
SCF(basis II)	1.732(+18)	1.356(−14)	1.435(+12)	92JST(259)181
STO-3G	1.732(+18)	1.335(−35)	1.454(+31)	84BCJ1312
STO-3G	1.732(+18)	1.335(−35)	1.454(+31)	86JCP(84)4228
STO-3G*	1.733(+19)	1.323(−47)	1.484(+61)	88JST(163)173
CASSF/3-21G*	1.734(+20)	1.361(−9)	1.445(+22)	94JCP(100)2571
MNDO	1.693(−21)	1.367(−3)	1.454(+31)	85JCS(P2)97
MNDO	1.692(−22)	1.368(−2)	1.456(+33)	89CHE874
MNDO	1.692(−22)	1.368(−2)	1.457(+34)	86JCP(84)4228
MNDO	1.692(−22)	1.368(−2)	1.458(+35)	86IJQ1599
ACPF(basis II)	1.745(+31)	1.378(+8)	1.442(+19)	92JST(259)181
ACPF(basis I)	1.753(+39)	1.360(−10)	1.433(+10)	92JST(259)181
MINDO/3	1.757(+43)	1.346(−24)	1.462(+39)	80IJQ797
3-21G	1.797(+83)	1.335(−35)	1.448(+25)	89JST(186)101

^aValue in parenthesis corresponds to difference between experimental and theoretical value $\times 10^{-2}$.**Table 10** Bond angles (in °) of thiophene calculated by various methods.

Method	C-S-C	S-C-C	C-C-C	Ref.
Experimental	92.17	111.47	112.45	61JSP(7)58
3-21G	89.2	111.7	119.3	89JST(186)101
MM2	90.3	113.2		85TL2403
STO-3G	90.4	112.7	112.1	84BCJ1312
ACPF(Basis I)	90.5	111.9	112.9	92JST(259)181
TZVP	91.1			92ZN(A)203
4-31G + d	91.2	111.8	112.6	82JA1375
SCF(Basis I)	91.2	111.9	112.5	92JST(259)181
ACPF(Basis II)	91.2	111.9	112.5	92JST(259)181
CASSF/3-21G*	91.2	112.1	112.3	94JCP(100)2571
HF/631G*	91.4	111.8	112.5	94JCP(100)2571
SCF(Basis II)	91.3	111.8	112.6	92JST(259)181
SCF/3-21G*	91.4	111.9	113.4	88JA4204
3-21G*	91.4	111.9	122.2	89JST(186)101
CI-SD(Basis I)	91.5	111.9	112.4	92JST(259)181
MP2	92.3	111.6	112.2	88JPC1739
MNDO	93.1	112.0	111.4	86IJQ1599
MNDO	93.2	111.9	111.5	89CHE874
QCFF/PI + CISD	93.8	110.8	112.3	94JCP(100)2571
MINDO/3	94.3	108.4	114.5	80IJQ797

3°. In almost all cases, the lengths of the C—S and the C—C bonds are overestimated while that of the C=C bond is underestimated. Thus the C—S—C angles calculated by these methods are usually smaller than the experimental values while the X—C(1)—C(2) angles are generally larger than the experimentally determined values but the difference is usually less than 1°.

MNDO calculations do not tend to follow this trend (83MI 209-01, 85JCS(P2)97, 86IJQ1599, 89CHE874). While there is excellent agreement for the C=C bond (difference of less than 0.003 Å), these calculations also tend to underestimate the C—S bond due to the noninclusion of the 3d atomic orbitals which result in smaller sulfur atomic orbitals and hence shorter C—S bonds. Consequently, the C—S—C angles obtained from MNDO calculations are about 1° larger than experimental.

Although MINDO/3 calculations <80IJQ797> follow the trend observed by the other methods, and all calculated bond lengths are within 0.04 Å of the experimental value, the differences here are usually higher than most of the other methods. The larger difference in the bond lengths also leads to larger discrepancies in the bond angles which differ from experimental values by 2–3°. MINDO/3 studies on other five-membered heterocycles correctly predict the C—X bond length to be in the order thiophene > pyrrole > furan, and the order of the C—X—C bond angle to be pyrrole > furan > thiophene.

Amongst the *ab initio* methods, the best correlation between theoretical and experimental values are those obtained by the MP2 method <88JPC1739>. The double zeta valence with one set of polarization function (DZP) basis predicts the bond lengths to within 0.016 Å and the angles to less than 0.7°. This is a tremendous improvement over other *ab initio* calculations especially those with the 3–21G basis set where the C—S bond is overestimated by 0.08 Å due to the absence of *d*-orbitals on sulfur in the 3–21G basis set <89JST(186)101>. Inclusion of the *d*-orbitals in the basis set (3–21G*) reduces the C—S bond by about 0.07 Å. A similar shortening is also observed for C—S single and double bonds in other acyclic sulfur compounds <88JA4204, 89JST(186)101>. Although there is a large difference in the C—X bond for thiophene, the difference between the 3–21G and 3–21G* calculations decreases in going from sulfur to tellurium and thus a better correlation results between the two methods. The reason for this is that, unlike the sulfur 3–21G basis set, the selenium and tellurium 3–21G basis sets already contain *d*-orbitals which are occupied and hence the extra *d*-function is not a polarization function but merely a supplement to the basis set. Similarly, the C—S—C bond angle as calculated with the 3–21G basis set is smaller than the experimental value by about 3° and is reduced to less than 1° when the *d*-orbitals are included. The opposite trend is observed from STO–3G <84BCJ1312> and STO–3G* <88JST(163)173> calculations where the bond lengths calculated without the inclusion of the sulfur *d*-orbitals (STO–3G) are better.

Ab initio calculations of ³³S nuclear quadrupole coupling constants have been carried out on a variety of sulfur compounds using a TZVP basis set <92ZN(A)203>. The equilibrium geometric parameters calculated by this method follows the trend of the other *ab initio* methods and the values are within the acceptable limits.

Molecular mechanics is known to be a reliable and quick method for determining structures, energies, and properties of a variety of compounds. Calculations involving the MM2 force field method, has usually been limited to conjugated hydrocarbons <84TL2297>. This approach has now been extended to compounds containing nitrogen, oxygen, and sulfur atoms <85TL2403>. The bond lengths follow the pattern of *ab initio* calculations where the C—S bond length is overestimated. Agreement is good between the experimental and calculated bond lengths and angles for thiophene, as well as for some of the other heterocyclic systems tested.

The properties of oligomers of thiophene at the ground and excited states have been investigated by semiempirical and *ab initio* methods. Semiempirical calculations for the ground state of thiophene involve the QCFF/PI + CISD method <94JCP(100)2571>, while *ab initio* calculations are carried out at the Hartree–Fock/6–31G* and the CASSF/3–21G* levels <94JCP(100)2571> and also at the SCF and the ACPF levels, with (basis set I) and without (basis set II) the sulfur 3*d*-orbitals <92JST(259)181>.

The molecular geometry of thiophene obtained by the QCFF/PI + CISD method is in good agreement with experimental and with the other *ab initio* methods. Upon excitation to the ¹B₂ state, this method is able to predict geometry changes but tends to underestimate the angle changes; a problem previously observed in the calculations for cyclopentadiene <93JCP(99)3721>. Incidentally, while *ab initio* methods predict an elongation of the C—S bond for both the ¹B₂ and ²A₁ excited states, the semiempirical method predicts a shortening for the latter state. Other than that, results from the QCFF/PI + CISD method for the ²A₁ excited state agree well with those from CASSF/3–21G*.

The optimized structures of the *syn*, *anti*, and *anti-gauche* conformers of 2,2'-bithienyl calculated at the SCF level do not deviate too much from that of the monomer <92JST(259)181>. The bond lengths of the *anti* and the *anti-gauche* conformers differ from each other by less than 0.002 Å while the angles differ by only 0.2°. The intermolecular C(2)—C(2') bond length is only 0.03 Å longer than the ring C(3)—C(4) bond. HF/3–21G* calculations <94JCP(100)2571> for the *anti*-planar conformer agree extremely well with the experimental values <58ACS1671>. The MP2/3–21G calculations, in general, tend to overestimate the C—X bond in five-membered rings. This is especially pronounced here, where the C—S bonds are overestimated by 0.1 Å. Addition of *d*-orbitals on sulfur should shorten the calculated bond lengths and lead to better approximations.

Optimized planar geometries of the all-*anti* terthiophene and tetrathiophene have also been determined by SCF <92JST(259)181> and by QCFF/PI + CISD and HF/3–21G* <94JCP(100)2571> methods. No significant changes are observed upon the oligomerization of thiophene except that

the inter ring C—C bond lengths tend to decrease as the chain length increases, in line with that observed for oligomers of pyrrole and in linear polyenes.

For chlorinated thiophenes, the C_β—Cl and the C_β—H bonds calculated at the STO-3G level are longer than the C_α—Cl and C_α—H bonds <84BCJ1312>. The interior angles of the carbon atoms bearing the chlorine substituents are also larger than the corresponding angles in thiophene. The S—C—C angle in 2-chlorothiophene (**15**) is 113.8° vs. 111.47° in thiophene, while the C—C—C angle in 3-chlorothiophene (**16**) is 113.7° vs. 112.45° in thiophene. For the 2*H*-thiophenium cation (**7**), the C(3)—C(4) and C(5)—S bonds are shorter than in thiophene while all others are longer. Deprotonation of thiophene also leads to an increase in the length of the bond linking the deprotonated carbon atom with the adjacent atoms albeit by a smaller amount than the 0.08–0.18 Å increase observed during protonation. MNDO calculations <89CHE874> predict that deprotonation at C(2) leads to an increase in the C—S bond by 0.013 Å and in the C(2)—C(3) bond by 0.011 Å as compared to the neutral molecules. In general, increases between 0.02–0.04 Å were obtained for the other five-membered heterocyclic compounds as well as their benzo analogues.

2.09.2.10 Other Molecular Properties

The effect of the inclusion of the 3*d*-orbitals of sulfur in semiempirical and *ab initio* calculations has been reported. One of the factors affected by the inclusion of the 3*d*-orbitals is the dipole moment. *Ab initio* calculations generally show a decrease when the 3*d*-orbitals are included. This change is caused by a reduction of the atomic charges resulting in almost nonpolar bonds. Quadrupole moments behave similarly under these conditions <72TCA171>. Due to the enhanced electron density around the sulfur atom, the expectation value $\langle Z^2 \rangle_{\text{cm}}$ is lowered (assuming the *y* and *z* axes are in the molecular plane and that the *z* axis bisects the C—S—C angle). The greatest effect is observed in the value of Q_{zz} which increases from 0.8×10^{-26} to 1.3×10^{-26} esu cm². Calculations performed by the one-particle approximation gave a value of 1.2×10^{-26} esu cm² <76JES179>.

Other *ab initio* calculations of one-electron observables <82MP649> also show smaller changes in the quadrupole moment as compared to the dipole moment. As before, Q_{zz} is closest to the experimental value when the basis set includes one extra *d*-function on sulfur. The quadrupole moments Q_{xx} , Q_{yy} , and Q_{zz} in this case, are between those calculated without any polarization function and those calculated without the *d*-function on carbon. With the 6-31G(+*sd*+*sp*) basis set <92JPC7301>, the MP2 and SCF values do not compare as favorably as results predicted by Gelius *et al.* although relatively large experimental errors makes it difficult to make accurate comparisons <72TCA171>.

Contrary to the effect observed for the quadrupole moments, the effect of the 3*d*-orbitals on the diamagnetic susceptibility tensor is negligible <72TCA171, 82MP649>. There is good agreement between the experimental values and theoretical calculations <76JES179>.

Gelius *et al.* <72TCA171> have also carried out MO-SCF-LCAO calculations for the quadrupole coupling constants for ³³S in thiophene, but were unable to compare the results with any experimental data at that time. However, since similar calculations for the quadrupole coupling constants for ³³S in SO₂ were in good agreement with experimental data, they were confident that the results obtained for thiophene were acceptable.

Ab initio calculations of the ³³S nuclear quadrupole coupling constants (NQCC) have been carried out using the TZVP basis set <92ZN(A)203>. For bivalent sulfur compounds, there is a large reduction in the π-NQCC when one goes from the acyclic dimethyl sulfide to the aromatic thiophene. As before, there is no MW data available to compare the results, but the results obtained here are in good agreement with those obtained above by Gelius *et al.* <72TCA171>. In most cases, the calculated electric field gradients correlate well with the experimental NQCC, including the ³³S atomic quadrupole moment, implying good predictability with the TZVP basis set.

2.09.2.11 Miscellaneous

Didehydroheteroarenes (or heteroarynes) and tetrahydroheteroarenes are generated by removal of one and two pairs of vicinal hydrogen atoms respectively from the parent heterocycle. While the chemistry of benzyne and of the heteroarynes have been summarized in several reviews, much less is known about the tetrahydroheteroarenes. Theoretical calculations involving the didehydroheteroarenes have been mainly semiempirical in nature <64TL1577, 69JA2590, 77CC539, 84JA5256>.

The earliest report on a systematic *ab initio* study on these systems was by Radom *et al.* in 1986 <86PAC75>. Amongst the five-membered didehydroheteroarenes, the didehydrothiophenes have been studied the most. These exist either as the 2,3- (**20**) or the 3,4-didehydrothiophene (**21**) isomer. *Ab initio* calculations at the CASSCF/3-21G level show (**20**) to be 0.23 kcal mol⁻¹ lower in energy than (**21**). Bond lengths calculated by the above method for the two isomers as well as for tetrahydrothiophene (**22**) are listed in Table 11. In all cases, the C—S bond is longer than in thiophene. Although the 3,4-didehydro isomers of furan and thiophene show similar behavior, there is a substantial difference in the 2,3-didehydro isomers of the two. For furan, the C(3)—C(4) bond is predicted to be quite long (ca. 1.516 Å) while in 2,3-didehydrothiophene (**20**), it is 1.424 Å, which is quite similar to that found in thiophene. Calculations on (**22**) show it to be a stable molecule, unlike tetrahydrofuran where calculations predict spontaneous rupture of the C(3)—C(4) bond.

Table 11 Predicted bond lengths (in Å) for di- and tetrahydrothiophenes.

Thiophene	C ₂ S	C ₃ S	C ₂ C ₃	C ₄ C ₅	C ₃ C ₄
Thiophene ^a	1.714	1.714	1.370	1.370	1.423
2,3-Didehydro	1.743	1.780	1.235	1.355	1.424
3,4-Didehydro	1.771	1.771	1.342	1.342	1.259
Tetrahydro	1.786	1.786	1.220	1.220	1.491

^aExperimental values <61JSP(7)58>.



(20)

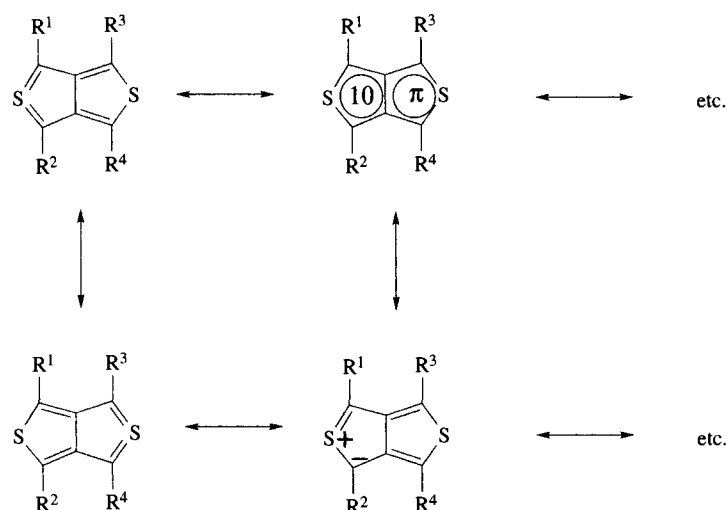


(21)



(22)

The thieno[3,4-*c*]thiophene system is an intriguing 10 π -electron system consisting of two fused thiophene units. This nonclassical thiophene can exist in many isomeric forms (see Scheme 1) and is thus of synthetic and theoretical interest. The parent compound (R = H) is unstable and only a few derivatives have been synthesized. Information regarding the theoretical and synthetic aspects of this system can be found in <83JA1705, 83JA1979, 87BCJ1981, 88JA1793, 94JOC2223> and the interested reader is encouraged to refer to them.



Scheme 1

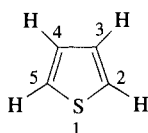
Semiempirical and *ab initio* calculations have also been carried out to determine aromatic energies of thiophene and other heterocycles. This will be discussed later in the section on aromaticity.

2.09.3 EXPERIMENTAL STRUCTURAL METHODS

2.09.3.1 Molecular Structure

Highly accurate molecular geometries for five-membered heterocycles have been obtained by microwave spectroscopy. Bak *et al.* <56JCP(25)892, 60ACS1865, 61JSP(7)58> have studied the microwave spectra of thiophene, deuteriothiophene, and ^{13}C -labeled thiophene. The molecular structure has also been determined by electron diffraction <70AX(B)1010> and the geometry as obtained by these two methods is listed in Table 12. The results from MW spectroscopy described here <61JSP(7)58> are an improvement over earlier results <56JCP(25)892>. The C—C distances here are closer to the benzenoid C—C bond length. Within limits of error, the C(2)—C(3) bond length and the C(3)—C(4) bond length are symmetrically displaced with respect to the benzene C—C bond. The measured C—S bond is almost midway between a pure single and a pure double bond; its double-bond character is estimated to be 40%. Results from electron diffraction compare reasonably well with MW spectral analysis except for the C—H bond lengths which are somewhat smaller than that determined from MW spectroscopy.

Table 12 Bond lengths and angles for thiophene from MW spectroscopy^a and electron diffraction^b.



Bond	MW spectroscopy	Electron diffraction
<i>length (nm)</i>		
C—S	1.714 ± 0.0014	1.717 ± 0.004
C=C	1.370 ± 0.0017	1.368 ± 0.004
C—C	1.423 ± 0.0023	1.424 ± 0.002
C(2)—H	1.078 ± 0.0015	1.071 ± 0.015
C(3)—H	1.081 ± 0.0014	1.071 ± 0.015
<i>angle (°)</i>		
C(5)—S—C(2)	92.1 ± 0.1	91.9 ± 0.3
S—C(2)—C(3)	111.5 ± 0.2	111.9 ± 0.03
C(2)—C(3)—C(4)	112.5 ± 0.2	
S—C(2)—H	119.8 ± 0.8	121 ± 3
C(3)—C(4)—H	124.25 ± 0.1	120 ± 6

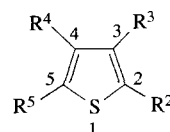
^a<61JSP(7)58>. ^b<70AX(B)1010>.

The molecular structure of thiophene has also been determined by liquid crystal LC NMR spectroscopy <84MP779, 88MCLC267> as well as electron diffraction (ED) and rotational spectroscopy. All three methods have their limitations and thus are generally used for only simple models. The fact that each method complements the other was made use of in a determination of the r_x structure of thiophene by a combined analysis of the three methods <88JST(178)227>. The ED data is useful for determining the absolute size and positions of the heavy atoms. It is however unable to properly locate the hydrogen atom. As a result, the C—H bond distance appears too large. Furthermore, the C—C and C=C bond lengths and the C—S—C bond angle do not agree well with results from other methods.

From LC NMR coupling constants, the location of the hydrogen atoms can be determined. However, since this method does not give information about the absolute size, a few structural parameters are fixed. By combining the two methods, the deviations from MW spectroscopy are smaller <61JSP(7)58> and this is further improved by addition of rotational spectroscopy data.

A search of the Cambridge Crystallographic Database (updated in 1993 ca. 120000 entries) for thiophene not fused to another ring yielded 546 hits. While it is impossible to list all, a few compounds will be discussed here and their geometries are summarized in Table 13.

Although the molecular structure of thiophene has been actively investigated <61JSP(7)58, 70AX(B)1010, 84MP779, 88JST(178)227, 88MCLC267, 88JA4204>, there have not been many reports on that of the methyl derivatives. The molecular structure of 2,5-dimethylthiophene has been determined by gas electron diffraction (GED) and the results compared with *ab initio* calculations carried out

Table 13 Molecular geometries of various substituted thiophenes.

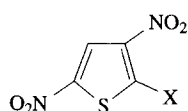
R^2	R^3	R^4	R^5	r_{12}	r_{23}	r_{34}	r_{45}	r_{15}	θ_{215}	θ_{123}	θ_{234}	θ_{345}	θ_{451}	Ref.
Me	H	H	Me	1.736	1.372	1.438	1.372	1.736	92.6	110.7	113.0	113.0	110.7	93JST(301)107
H	Bu ^t	Bu ^t	H	1.78	1.39	1.66	1.39	1.78	90.0	117.0	108.0	108.0	117.0	80CC922
Bu ^t	Bu ^t	Bu ^t	Bu ^t	1.720	1.388	1.499	1.387	1.720						92TL5947
4-NO ₂ C ₆ H ₄	Ph	Ph	4-NO ₂ C ₆ H ₄	1.724	1.381	1.429	1.381	1.724	93.0	110.7	112.8	112.8	110.7	86AX(C)363
CN	CN	CN	CN	1.69	1.40	1.40	1.34	1.71	89.1	113.7	110.8	111.8	114.3	68AX(B)725
Cl	NO ₂	H	NO ₂	1.71	1.37	1.41	1.36	1.71	90.4	111.7	113.9	109.8	114.2	88JHC177
SO ₂ Ph	NO ₂	H	NO ₂	1.706	1.366	1.403	1.342	1.709	89.9	111.1	114.7	109.0	115.4	88JHC177
NH ₂	CO ₂ Et	Ph	H	1.725	1.396	1.452	1.353	1.733	91.2	112.0	111.5	111.9	113.3	91AX(C)1748
CO ₂ H	H	H	H	1.693	1.362	1.414	1.363	1.701	92.0	111.8	112.4	111.9	111.8	62AX913
CO ₂ H	H	H	H	1.693	1.362	1.41	1.363	1.701	92.0	111.8	112.4	111.9	111.8	62AX737
CO ₂ H	H	H	H	1.707	1.369	1.414	1.350	1.695	91.6	111.5	112.0	112.3	112.5	89MI 209-01
COCH ₃	OH	H	H	1.730	1.381	1.431	1.351	1.710	91.4	111.4	111.9	112.4	113.0	69ACS2031
COCH ₃	H	H	Br	1.72	1.37	1.44	1.34	1.72	91	112	112	111	114	70RTC(89)392
CO ₂ H	COCH ₃	H	H	1.718	1.406	1.433	1.376	1.717	93.1	110.3	112.6	112.3	111.8	72BSB319
COCH ₃	CO ₂ H	H	H	1.723	1.381	1.397	1.343	1.699	91.8	110.8	111.5	114.3	111.5	72BSB319
H	CO ₂ H	COCH ₃	H	1.693	1.346	1.454	1.356	1.698	91.7	111.9	113.1	109.3	113.9	72BSB319

at the 3-21G* level <93JST(301)107>. For the equilibrium state, GED results show one of the C—H bonds of the methyl substituent to be *cis* with respect to the S—C bond of thiophene, contradicting STO-3G* results which indicate one of the bonds to be *trans*. This contradiction is considered to arise due to the deficiency in the basis set. However, the STO-3G* calculations parallel that of the STO-3G calculations on 2-methylthiophene <79JA311>. A GED study of 2-methylthiophene should be able to determine the stable configuration of this. The C(2)—Me distance is smaller than a normal C—C single bond and quite close to the C—C bond length in nonconjugated alkanes. The C—S bond length here is larger than in thiophene as is also the C—S—C angle. In general, the STO-3G* calculations reproduce most of the experimental differences between 2,5-dimethylthiophene and thiophene.

The presence of bulky groups adjacent to each other leads to variations from normal trends due to steric interaction. This is more so in six-membered rings than in five-membered heterocycles. For example, x-ray results of 3,4-di-*t*-butylthiophene indicate that the ring is almost planar <80CC922>. However, results do show appreciable internal strain in the molecule. The C(3)—C(4) bond length is 1.667 Å as compared to 1.423 Å in thiophene. The two substituents are also pushed away from each other, the C(3)—C(4)—Bu^t angle being 133° as compared to about 124° in other cases.

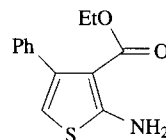
The x-ray structure of tetra-*t*-butylthiophene has also been determined <92TL5947>. The ring is not planar any more, the torsional angle being 16.2°. The torsional angle between the 3- and 4-groups is 44° while that between the 2- and 3-substituents is 26.8°. The C—Bu^t bonds lengths here are also longer (ca. 1.567 Å) as compared to 1.49 Å in 3,4-di-*t*-butylthiophene <80CC922>. The twisting between the *t*-butyl groups and in the ring releases most of the strain and thus the ring bond lengths are smaller than the di-*t*-butyl derivative and hence closer to thiophene. In tetra-*t*-butyl-1,1-dioxide, the ring becomes nonaromatic and bond alternation is much more pronounced. Although the C=C bond length decreases, all other bond lengths increase as does the torsional angle between the *t*-butyl groups at the 3- and 4-position <92TL5947>.

X-ray analysis show the phenyl rings in 2,5-bis(4-nitrophenyl)-3,4-diphenylthiophene are twisted —72.0° from the thiophene plane, while the angle between the nitrophenyl rings and thiophene is 26.6° <86AX(C)363>. In 2,4-dinitrohalobenzenes, the nitro groups are rotated with respect to the benzene rings. In five-membered ring compounds, the distance between adjacent substituents is larger which tends to reduce the interaction between the halo and the nitro group. In 2-chloro-3,5-dinitrothiophene (**23**), the thiophene ring is almost planar <88JHC177>. The 5-nitro substituent is rotated by 1° which increases to 8.3° when the chloride atom is substituted by a phenylsulfonyl moiety (**24**). This relatively small deviation is common for 4-unsubstituted 5-nitrothiophenes <79CHE1201>. The dihedral angle for 3-nitrophenyl is higher with it being 17.5° for (**23**) and 21.0° for (**24**) as compared to 40° in benzene. The strain is further released by a deformation which causes the C(3)—C(2)—X angle to increase to 129.9° and 132.2°, respectively.



(23) X = Cl

(24) X = SO₂Ph



(25)

In ethyl 2-amino-4-phenylthiophene-3-carboxylate (**25**), steric repulsion between the ethoxy group and the phenyl ring is reduced by the phenyl ring being twisted out of the plane by an angle of 70.2°. The phenyl ring is also pushed away as is seen by the larger C(3)—C(4)—Ph angle <91AX(C)1748>.

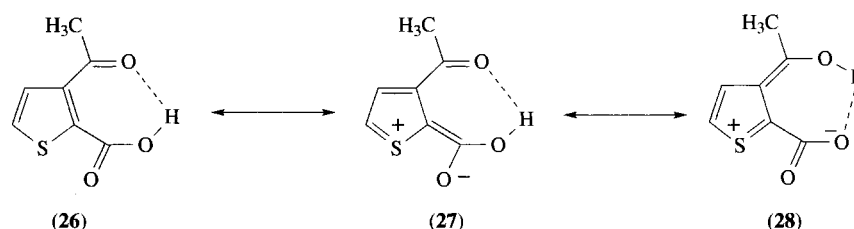
Initial determination of the molecular structure of 2-thiophenecarboxylic acid was not accurate due to poor resolution in the projections <58G229>. Subsequent refinements were in good agreement with each other (see Table 13) <62AX737, 62AX913>. Although the molecule loses its symmetry due to the substituent at the 2-position, the S—C bond lengths are approximately equal as are the two C=C bonds. The molecule is practically planar except for the sulfur atom which lies about 0.03 Å out of the plane. The carboxyl group is pulled towards the heteroatom making the S—C(2)—COOH angle smaller than the C(3)—C(2)—COOH angle.

A reinvestigation on 2-thiophenecarboxylic acid <89MI 209-01> confirms previous reports and also determines the dihedral angle between thiophene and the carboxyl group to be 1.49°. Furthermore,

the C=O bond length here is determined to be 1.235 Å which is longer than the 1.201 Å previously reported (62AX737). 2-Thiophenecarboxylic acid is intermolecularly linked via hydrogen bonding and the pair forms a centrosymmetrical dimer.

The crystal structure of 2-acetyl-3-hydroxythiophene has been investigated in order to determine the amount of hydrogen bonding in the solid phase (69ACS2031). The x-ray structure showed the carbonyl oxygen to be *cis* with respect to the sulfur atom. Thus the two oxygen atoms within the molecule are too far apart (4.27 Å) to bond intramolecularly. The distance between the hydroxyl group in one molecule and the carbonyl oxygen in another is much closer (2.64 Å) and thus capable of hydrogen bonding. The carbonyl oxygen in the first molecule is similarly bonded to a hydroxyl group of a third molecule; thus a chain is formed.

3-Dimensional x-ray analysis has been used to determine the x-ray structure of the three *ortho*-acetylthiophenecarboxylic acids (72BSB319). In all cases the carbonyl groups are in the same plane as the ring. For 3-acetyl-2-thiophenecarboxylic acid (26) the bond length of the C(2)=C(3) bond is 1.406 Å which is 0.036 Å longer than the corresponding bond in thiophene. The O—H bond length here is determined to be 1.52 Å while the distance between the acetyl oxygen and the carboxylic hydrogen is 1.06 Å, implying strong intramolecular hydrogen bonding. In fact, its positioning hints at a possible enol character. This is not surprising since the molecule could also exist in two other tautomeric forms (27) and (28) (Scheme 2).

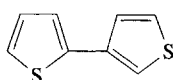


Scheme 2

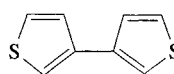
In 2-acetyl-3-thiophenecarboxylic acid, the C(2)=C(3) bond is shorter than in the previous case but the carboxylic group is angled further away from the acetyl group. This is seen in the O—H bond lengths where the bond length of the carboxylic O—H is 1.26 Å while that between the acetyl oxygen and the carboxylic hydrogen is 1.37 Å indicating less hydrogen bonding here and thus less influence from the resonance structure analogous to (28).

Tetrachlorothiophene-1,1-dioxide is nonplanar, with the sulfur atom being displaced from the plane by 0.087 Å. The ring is also deformed as is seen by the difference in the two S—C and C=C bond lengths. Due to lack of conjugation between the SO₂ group and the double bonds, there is noticeable bond alternation here with the C=C bond length being ca. 1.33 Å while the C(3)—C(4) bond length is 1.476 Å. The four C—Cl bond lengths are all different and range from 1.682 to 1.696 Å (93AX(C)1197).

Interest in the chemistry of polythiophenes has stemmed from their potential as organic conducting polymers (B-86MI 209-01). Theoretical studies (see Section 2.09.2) have been carried out on these compounds to predict their properties, stabilities, etc. The simplest polythiophene is bithienyl, which could exist in the isomeric forms 2,2'-(18), 2,3'-(29), or 3,3'-(30). Furthermore, the molecules can rotate round the intermolecular C—C bond and thus each isomer can possess various conformations. Initial studies indicated that, in the solid phase, the molecules are planar and have the *anti* conformation (68AX(B)467). In the gas phase the two molecules are twisted around the central bond by approximately 34° (58ACS1671). The molecule is slightly distorted with the C(2)—C(3)—C(4) angle being about 9° smaller than the C(3)—C(4)—C(5) angle. For 2,3'-bithienyl (29), the conformation could not be obtained due to the disorder in the crystals. Similarly, from the x-ray of 3,3'-bithienyl (30), it was not possible to determine if the *anti* conformation holds for all the molecules. The C—S bond lengths in bithienyl do not differ much from that in thiophene and consequently, the thienyl substituent does not alter the geometry of thiophene to any extent.



(29)



(30)

X-ray studies on the structure of 3,3'-dimethoxy-2,2'-bithienyl show the molecule to lie on a crystallographic center of symmetry and the two rings to be in the *anti* form <88AX(B)509>. Comparison of the experimental results with *ab initio* calculations show extremely good agreement. Although x-ray shows the molecule to be planar, MNDO calculations indicate it to be 17 kcal mol⁻¹ more stable when the two rings are at 90° to each other <77JA4899>. A similar result was also observed for 3,3'-dihydroxy-2,2'-bithienyl where the nonplanar forms were found to be more stable. However, *ab initio* calculations show that in the nonhydrogen-bonded conformation, the planar form is more stable. Since the hydrogen atom of the hydroxyl group in the nonbonded case points in the same direction as the methyl group in the methoxy case, one can assume that the x-ray structures are correct and that MNDO calculations are not accurate in this case.

In 4,4'-dimethoxy-2,2'-bithienyl, molecules in different halves of the unit cell have different geometries. For cases not involving H atoms, the differences in bond lengths and angles are up to 0.014 Å and 0.8°, respectively, while for H-related parameters the differences are up to 0.1 Å and 4–5° <89JST(196)171>.

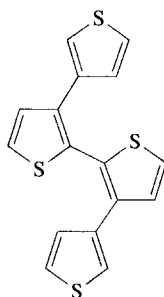
Contrary to 2-bromothiophene where the S—C(2) bond is longer than the S—C(5) bond <70AX(B)1010>, the x-ray analysis of 5,5'-dibromo-2,2'-bithienyl shows the S—C bonds to be of equal length (within experimental error) <88AX(C)562>. The C—S—C angles in the monomer and dimer are approximately the same as are the S—C(2)—C(3) and the S—C(5)—C(4) angles. The major difference is between the C(2)—C(3)—C(4) and C(3)—C(4)—C(5) angles with the latter being smaller by about 2.5°. This occurs since both a thienyl substituent as well as a 2-bromo substituent cause the C(2)—C(3)—C(4) angle to be 9° to 5° smaller than the C(3)—C(4)—C(5) angle. The small difference here is due to the fact that the substituents are on opposite sides and thus tend to cancel each other.

While the 5,5'-dibromo derivative is planar in the solid phase <88AX(C)562>, the 5,5'-dinitro derivative exhibits a small dihedral angle of 3.3° <80MI 209-01> while in 3,5,5'-tribromo-2,2'-bithienyl it is 4.6° <88AX(C)1800>. In the monosubstituted ring, the C(2)=C(3) bond length is 0.08 Å longer than the C(4)=C(5) bond length, while in the disubstituted ring, they are the same. The C(2)—C(3)—C(4) and the C(3)—C(4)—C(5) bond angles are the same in the monobromo substituted ring while in the other ring the latter is smaller by 6.3°.

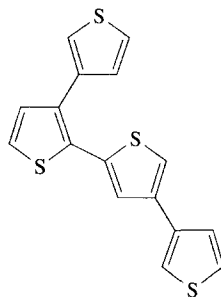
In 5-dimethylamino-5'-nitro-2,2'-bithienyl the dihedral angles between the two rings is 3.7°, between the dimethylamino group and the ring 4.5°, and between the nitro group and the ring 2.1° <93AG(E)719>. The presence of donor-acceptor substituents in the molecule and planarity leads to extended conjugation within the molecule as seen by the distinct equalization of the C—C bond lengths. Thus all the C—C ring bond lengths are between 1.36 and 1.40 Å.

Although 3,3'-bithienyls are planar <68AX(B)467> substituents at the 2,2';4,4'-positions lead to restricted rotation and as a result, these compounds are not planar <75CS205>.

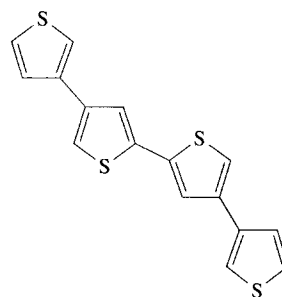
Unlike the 2,2'-bithienyls, 3,3';2',2'';3'',3'''-tetrathiophene (**31**) is not planar with the angles between the 3,3'-rings being 20° while that between the 2',2''-rings is 110° <88H(27)1391>. In the plane of the terminal thiophene rings, two orientations of sulfur were observed in a ratio of 2:1 indicating the presence of two other isomers (**32**) and (**33**).



(31)



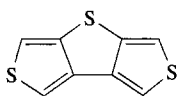
(32)



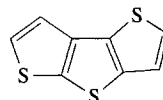
(33)

In addition to polythiophenes, fused thiophenes also possess interesting optical and electronic properties. The x-ray structure of dithieno[3,4-*b*;3',4'-*d*]thiophene (**34**) shows each thiophene ring to be practically planar, the maximum displacement being 0.006 Å <88AX(C)545>. The dihedral angles between the central ring and the two outer rings are 1.2° and 2.4°. The C—S bond lengths for the central ring are about 0.05 Å longer than those of the outer rings. This difference is less so in the fused thiophene (**35**) <83JCP(87)2317>. In both cases, the differences in the C—C bond lengths

for the central rings are much smaller than for the other rings indicating a different bond order in the central ring and thus enhanced aromaticity.



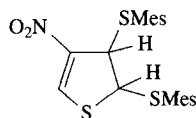
(34)



(35)

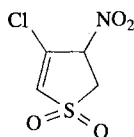
The x-ray crystallographic search for dihydrothiophenes revealed 42 hits. In most of the compounds, the thiophene ring was fused to a cyclic compound. X-ray studies of some dihydrothiophene-1,1-dioxides are also available. In most cases, the x-ray studies were used to determine the configurations of the substituents at the 2,3-position (i.e., *cis* or *trans*).

2-Mesitylthio-4-nitrothiophene is synthesized from 3,4-dinitrothiophene via a 2,3-dihydro intermediate <85JCS(P2)1291>. The regiospecificity here is opposite to that in other heterocycles. The intermediate is of interest to assess the stereochemistry of the above reaction. The x-ray structure of *trans*-2,3-bis(mesitylthio)-4-nitro-2,3-dihydrothiophene (**36**) shows the molecule to be puckered, the torsion angles being between -28° and $+23^\circ$. The conformation is thought of to be a half-chair with a pseudo twofold axis through C(5) and the center of the C(2)—C(3) bond; a conformation also observed in other 2,3-disubstituted 2,3-dihydrothiophenes <80JCS(P2)1764>, as well as in *spiro*-2,3-dihydrothiophene <81CB1074>. The nitro group is planar with the C(3)—C(4)—C(5)—S plane of the molecule with the C(5)—H being bent away from the nitro group to allow for hydrogen bonding with the oxygen atom. The S—C(2) bond length (1.824 Å) is much longer than the S—C(5) bond length (1.710 Å), while the shorter bond length of C(3)—C(4) as compared to C(2)—C(3) arises due to conjugation between the nitro group and the thiophene sulfur atom.



(36)

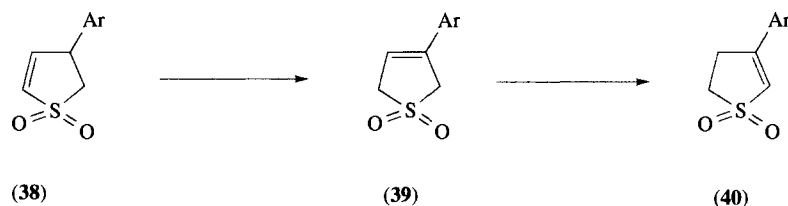
The x-ray structure of 3-chloro-4-nitro-4,5-dihydrothiophene-1,1-dioxide (**37**) indicates the molecule to be planar within 0.03 Å, with the nitro group being twisted at an angle of 88.9° <84JGU1058>. The two S—C bond lengths are different; the difference depending on the substituents on C(3) and C(4). The S—O bond lengths as well as the C=C bond length here are smaller than those found in the 3-aryl derivative (**40**) <80T1667>. The C—Cl bond here is 0.03 Å shorter than a comparable vinyl chloride bond and occurs due to the interaction of the unshared electron pair of the halogen with the unoccupied 3d-orbitals of the sulfur atom through the C(2)=C(3) bond.



(37)

4-Aryl-4,5-dihydrothiophene-1,1-dioxides (**38**) undergo base-catalyzed isomerization to 3-aryl-2,5- (**39**) and 3-aryl-4,5-dihydrothiophene-1,1-dioxide (**40**) (Scheme 3). Although 2,5-dihydrothiophene-1,1-dioxide is more stable than its 4,5-isomer, the presence of a substituent at the β position, shifts the equilibrium to the right <80T1667>.

In (**38**), the two rings are twisted at an angle of 35° , while in (**40**) they are planar. The sulfur atom is at the apex of a distorted tetrahedron in both cases. The C—S bonds in (**40**) are shorter than in (**38**) due to the extended conjugation between the aryl group and the C=C in (**40**). This conjugation also explains the bond length of 1.42 Å for the C—C bond between the two rings which is considerably shorter than a normal C—C single bond. Although there is not a great deal of difference between the geometries of the two molecules, the biggest difference lies in the orientation of the phenyl group with respect to the five-membered ring.



Scheme 3

For the fully saturated heterocycle tetrahydrothiophene, 344 hits were obtained. Many of these references involved the structure of tetrahydrothiophene with metal complexes and also as perhydrobenzothiophenes. The variation is too great and thus will not be discussed here.

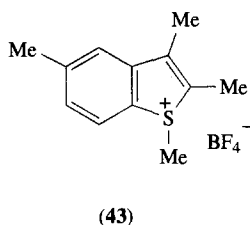
A comparison of the molecular geometries of some of the simple derivatives of benzo[*b*]thiophene is listed in Table 14. In benzo[*b*]thiophene, both rings are planar. Introduction of a substituent on the thiophene ring usually causes the two rings to be inclined to each other at about 1.0° $\langle 74\text{AX}(\text{B})2058, 84\text{AX}(\text{A})\text{C}277 \rangle$. The average C—S bond length is longer than in thiophene with the S—C(2) bond being the longer of the two. The C=C bonds in the five-membered ring which is fused to the benzene ring is longer by 0.044 Å. The C(2)=C(3) bond found here is also shorter than that in thiophene.

In ethyl 3-amino-5-nitrobenzo[*b*]thiophene-2-carboxylate, the two rings are inclined at 2.18° to each other $\langle 91\text{MI } 209-01 \rangle$. In the ester functionality, the carbonyl oxygen is *cis* to the sulfur atom and the NH bond lengths of the amino group indicate one hydrogen to be intramolecularly bonded to the oxygen of the OEt group and the other to be intermolecularly bonded to the carbonyl oxygen.

The x-ray structure of 3-acetoxybenzo[*b*]thiophene-2-carbaldehyde (**42**) which is formed by the acylotropic rearrangement of (**41**) has been determined $\langle 85\text{JOU}784 \rangle$ and is discussed in the section on tautomerism.

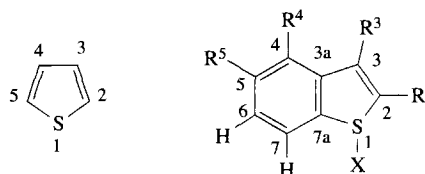


For benzo[*b*]thiophene-1,1-dioxide, its 2-bromo, and 2,3-dimethyl derivatives $\langle 87\text{AX}(\text{C})2421 \rangle$ as well as for the 2- and 3-methyl analogues $\langle 88\text{AX}(\text{C})498 \rangle$, the bond lengths and angles are approximately similar to their unoxidized precursors, the largest difference being in the C(2)=C(3) bond which is much smaller in the dioxide. As before, the two rings are tilted towards each other at about 1° . The geometries are also in good agreement with 1,2,3,5-tetramethylbenzo[*b*]thiophenium tetrafluoroborate (**43**) $\langle 81\text{JCS}(\text{P}2)266 \rangle$. The bonds about the sulfur atom are pyramidal in nature with the methyl group being out of the plane of the molecule. In comparison to benzo[*b*]thiophene derivatives (see Table 14), the salt has longer C—S and C(3)—C(3a) bonds but a shorter C(2)=C(3) bond indicating more bond alternation here due to the lack of delocalization.

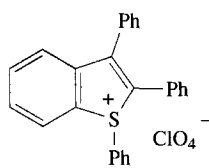


In the thienyl salt (**44**) $\langle 93\text{T}5055 \rangle$, the Ph—S⁺ bond length is shorter than that for Me—S⁺ indicating it to be a relatively stable molecule. As in the other salts, the bonds around sulfur are arranged pyramidally.

A search for x-ray studies on dibenzothiophenes reveal 32 hits from the crystallographic database.

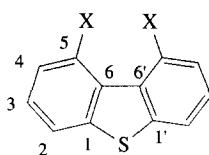
Table 14 Molecular geometries from x-ray studies of some benzo[*b*]thiophene derivatives.

<i>X</i>	<i>R</i> ²	<i>R</i> ³	<i>R</i> ⁴	<i>R</i> ⁵	<i>r</i> ₁₂	<i>r</i> ₂₃	<i>r</i> _{33a}	<i>r</i> _{3a7a}	<i>r</i> _{17a}	θ _{217a}	θ ₁₂₃	θ _{233a}	θ _{33a7a}	θ _{3a7a1}	Ref.
			Thiophene		1.714	1.370	1.423	1.370	1.714	92.1	111.5	112.5	111.5	92.1	61JSP(7)58
–	Me	H	H	H	1.772	1.382	1.485	1.386	1.741	92.4	111.7	111.4	113.3	111.3	85AX(C)929
–	Me	Me	H	Br	1.754	1.355	1.446	1.399	1.741	91.7	112.4	112.4	112.9	110.7	74AX(B)2058
–	Cl	Cl	H	H	1.758	1.341	1.418	1.398	1.765	90.0	112.0	115.1	111.1	111.7	86AX(C)696
–	H	H	Ph(CH ₂) ₂	NO	1.712	1.326	1.417	1.397	1.696	91.5	113.4	112.2	112.0	110.9	82AX(B)685
–	CO ₂ Et	NH ₂	H	NO ₂	1.749	1.376	1.447	1.404	1.724	91.3	112.9	111.4	112.5	111.9	91MI 209-01
–	CHO	OCOMe	H	H	1.739	1.365	1.419	1.409	1.736	90.9	111.7	114.8	110.0	112.6	85JOU784
			Compound 41		1.755	1.483	1.464	1.392	1.750	91.2	112.1	109.6	112.8	114.3	85JOU784
O ₂	H	H	H	H	1.757	1.323	1.467	1.391	1.579	93.0	110.7	114.8	112.3	109.3	87AX(C)2421
O ₂	Me	H	H	H	1.771	1.319	1.472	1.388	1.751	92.7	109.4	115.6	112.5	108.7	88AX(C)498
O ₂	H	Me	H	H	1.746	1.321	1.480	1.395	1.765	93.1	111.7	113.8	112.6	108.8	88AX(C)498
O ₂	Me	Me	H	H	1.766	1.335	1.480	1.401	1.757	94.1	110.2	114.2	113.2	108.3	87AX(C)2421
O ₂	Br	H	H	H	1.770	1.320	1.458	1.383	1.770	92.2	111.3	113.8	113.8	108.8	87AX(C)2421
Me	Me	Me	H	Me	1.779	1.340	1.468	1.389	1.768	92.2	111.0	113.2	113.6	109.2	81JCS(P2)266
Ph	Ph	Ph	H	H	1.790	1.346	1.471	1.400	1.762	92.1	111.1	113.4	113.2	109.9	93T5055



(44)

The x-ray structure of dibenzothiophene (**45**) was first determined by Schaffrin and Trotter <70JCS(A)1561>. Although each of the rings are planar, the molecule is slightly bow shaped with the dihedral angles between the planes being 0.4° and 1.2° . As for benzo[*b*]thiophene, the C—S bond length at $1.740 \text{ \AA}$ is longer than in thiophene as are the C(1)—C(6) and C(1')—C(6') bond lengths. The x-ray structure of dibenzothiophene-1,1-dioxide <68AX(B)981> is quite similar to dibenzothiophene.



(45) X = H

(46) X = NMe₂

Octafluorodibenzothiophene would be expected to be distorted due to the larger size of the fluoro atom. Although the fluoro substituents at C(5) and C(5') are closer than the sum of the accepted van der Waals radii, there is very little change in the molecular geometry <88PS(35)67>. The C—S—C angle is only slightly smaller than in dibenzothiophene (89.8° vs. 91.5°) and the C(5)—C(6)—C(6') angle increases to 131.5° due to the larger fluorine atoms. There is a slight relief in strain since the two fluorine atoms are displaced by $-0.011 \text{ \AA}$ and $+0.011 \text{ \AA}$ from the plane of the molecule.

1,9-bis(Dimethylamino)dibenzothiophene (**46**; X = NMe₂) belongs to a group of compounds known as proton sponges. X-ray structure analysis shows excessive steric strain in this molecule as a result of bulky groups in the 5,5'-positions <88TL1905>. As a result, the torsion amounts to 21.3° and the C(5) and C(5') atoms deviate from the mean plane by 0.33 and $3.0 \text{ \AA}$. This causes the nitrogen atoms to be $28.6 \text{ \AA}$ apart. Protonation causes a relief in the strain. The torsion angle is reduced to 7.7° as is the N···N distance due to the formation of the N···H···N bond which is almost linear (ca. 175°).

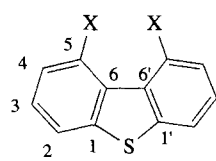
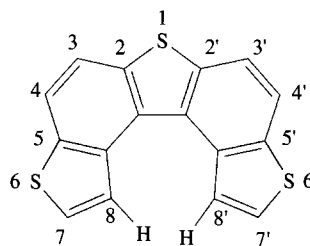
With arylthio groups at the 5,5'-positions (**47**; X = SAr), the central ring is distorted due to the steric repulsion of the substituents <92H(33)101>, the distance between the sulfur atoms being $3.012 \text{ \AA}$. The aryl substituents are *anti* and are close to the dibenzothiophene ring.

Although the x-ray structures of carbohelicenes have been extensively studied, those of the heterohelicenes have received less attention. The x-ray structure of trithia[5]heterohelicene (**48**) <84AX(C)1039> shows the terminal thiophene rings to be planar but possess a dihedral angle of 36.6° with each other. The three other rings are somewhat twisted, the dihedral angles between the central thiophene and the adjacent benzene rings being 7.9° while that between the benzene ring and the terminal rings is 10.6° . The atoms H(8) and H(8'), H(8) and C(8') are closer than the sum of the van der Waals radii and consequently H(8) and H(8') are displaced out of the plane of the thiophene ring. The S(6)—C(7) bond length is close to the value observed in thiophene while the S(6)—C(5) and S(1)—C(2) bond lengths are longer and closer to those found in dibenzothiophene <70AX(B)1010>. The packing mode of the heterohelicene (**48**) is quite different from that found in carbohelicenes.

2.09.3.2 Molecular Spectroscopy

2.09.3.2.1 Proton NMR spectroscopy

Spectroscopy is an important tool for the structure elucidation of compounds. Modern methods enable determination of conformation, aromaticity, mechanism, and other physical properties. A

(47) X = SA_r

(48)

systematic investigation of mono- and di-substituted thiophenes was carried out by Hoffman and Gronowitz <60AK501, 60AK563> which was later followed by a review of the ¹H NMR spectroscopy of thiophene by Gronowitz <63AHC(1)1>. Since then, other reviews on the spectroscopy of thiophene compounds have appeared, including ones being in CHEC-I <84CHEC-I(4)1, 84CHEC-I(4)713> and in the Weissberger series *The Chemistry of Heterocyclic Compounds* <86HC(44/2)135, 91HC(44/4)1>.

The ¹H NMR spectrum of thiophene (in CDCl₃) consists of two multiplets at 7.18 ppm and 6.99 ppm, the one at lower field being assigned to the α-hydrogens <65SA85>. In thiophene, as in other five-membered heterocycles, the H-2 and H-3 protons are about 1 ppm, downfield as compared to their 4,5-dihydro derivatives indicating the presence of an aromatic ring current. The chemical shifts and coupling constants for thiophene in CDCl₃ and acetone-*d*₆ are listed in Table 15. The vicinal proton couplings here are much smaller than in benzene due to the greater separation of the hydrogen atoms in five-membered rings than in six-membered rings. Similarly, smaller coupling constants are also observed for pyrrole and furan. The chemical shifts of the protons in thiophene are solvent dependent <86BCJ1650>; the H-2 proton resonates at 6.89 ppm and 7.16 ppm in benzene-*d*₆ and in cyclohexane <65BCJ1041, 74JOM(77)49> and at 7.46 ppm in acetone-*d*₆ <68ICA12>.

Table 15 Proton NMR spectral data for thiophene^a.

	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃	Acetone- <i>d</i> ₆
H-2	7.20	7.18	7.34	7.35	7.46
H-3	6.96	6.99	7.12	7.14	7.14
<i>J</i> _{2,3}	4.8	4.90	4.95	5.0	4.6
<i>J</i> _{2,4}	1.0	1.04	1.05	0.9	1.1
<i>J</i> _{2,5}	2.8	2.84	2.85	3.0	3.6
<i>J</i> _{3,4}	3.5	3.50	3.50	4.1	2.8
Ref.	60AK563	65SA85	83BCJ2463	68ICA12	68ICA12

^aChemical shifts in ppm, coupling constants in Hz.

The presence of a methyl or an alkyl substituent at the 2-position shifts the other protons upfield by 0.2–0.4 ppm. A similar effect is also observed with electron-donating substituents viz. OMe, OBu^t, and NH₂ <60AK539>. Substituents at the 3-position also cause the protons to shift upfield. With electron-donating substituents, the H-2 hydrogens are found more upfield than the H-4 hydrogens. For example, in 3-aminothiophene, the H-2 proton appears at 5.95 ppm while the H-4 hydrogen is found at 6.43 ppm. Electron-withdrawing substituents at the C-2 position cause substantial downfield shifts of the H-3 and H-5 protons while the H-4 proton chemical shift is hardly affected. For 3-substituents, the H-2 and H-4 protons are shifted downfield.

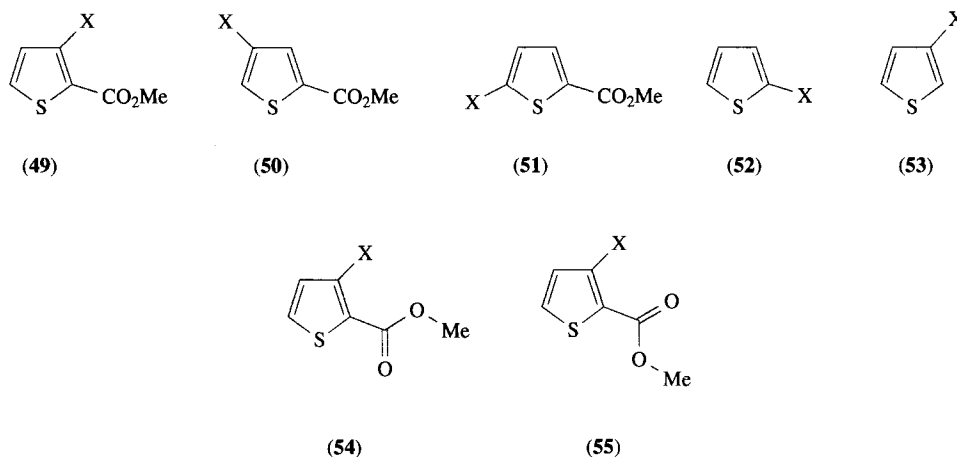
For mono- and di-substituted derivatives, the coupling constants for most compounds were as follows: *J*_{2,3} (or *J*_{4,5}) 4.9–5.8 Hz, *J*_{2,4} (or *J*_{3,5}) 1.2–1.7 Hz, *J*_{2,5} 3.2–3.6 Hz, *J*_{3,4} 3.4–4.3 Hz <60AK501, 60AK563>. For thiophenes containing only alkyl substituents the coupling constants are relatively close to those found in the parent molecule with values being typically about 4.9, 1.3, 2.7, and 3.3 Hz, respectively <74JOM(77)49, 86HC(44/2)135>. In the presence of strongly electropositive or electronegative substituents, values outside this range are observed; a *J*_{3,4} of 6.0 Hz is observed for 2-amino-5-nitrothiophene <81JHC851>.

The chemical shifts and coupling constants of a large number of mono-, di-, and tri-substituted thiophene derivatives have been reported <86HC(44/2)135>.

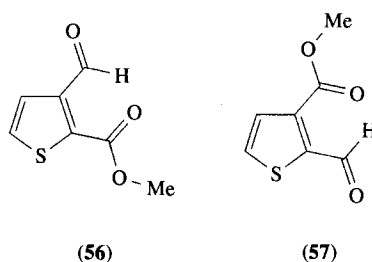
In benzo[*b*]thiophene, the H-2 and H-3 protons resonate at 7.33 ppm and 7.22 ppm <66BCJ2316>

which is slightly downfield from that observed for thiophene, while for benzo[*c*]thiophene, the H-1, H-3 protons appear at 7.63 ppm <76JCS(P2)81>.

The ^1H NMR spectra of methyl 3-, 4-, and 5-substituted 2-thiophenecarboxylates (**49–51**) has also been obtained to compare their substituent effects with those of 2- and 3-substituted thiophenes (**52**) and (**53**) <83BCJ2463>. In most cases, a good correlation is observed in the chemical shifts of the protons in (**49–51**) and the corresponding compounds (**52**) and (**53**) with the exception of the chemical shifts of H-5 in (**49**) and H-5 in (**53**) or H-3 in (**51**). Although the calculated chemical shifts in (**50**) and (**51**) are in good agreement with experimental values, those of (**49**) show large deviations since the carbonyl group can exist in the *S,O-cis* (**54**) or *S,O-trans* (**55**) orientation due to the presence of a substituent at the 3-position. This is seen in the IR spectrum of these compounds where two carbonyl stretching bands are observed. The coupling constants in compounds (**49–51**) vary linearly with the electronegativities of the substituents.



In methyl 3-formyl-2-thiophenecarboxylate (**56**), the aldehyde proton is observed 0.5–0.6 ppm further downfield than in 2-thiophenecarbaldehyde <63AK265>. This is attributed to the conformation of the ester carbonyl group which is capable of hydrogen bonding with the aldehyde proton and also due to the anisotropy effect of the ester carbonyl group <83BCJ2463>. For methyl 2-formyl-3-thiophenecarboxylate, the aldehyde proton is found at 10.62 ppm implying the conformation depicted in structure (**57**).



In thiophene-1,1-dioxide, the ring is no longer aromatic (see Section 2.09.3.1). Chemical shifts and coupling constants indicate the molecule to possess reasonable diene character <84ZN(B)915>. The ^1H NMR spectrum in CDCl_3 shows the α - and β -protons at 6.64 and 6.38 ppm, respectively; which is about 0.7 ppm upfield from the corresponding resonances in thiophene. In benzene- d_6 these protons resonate 1 ppm further upfield. Similarly, the H-2 and H-3 protons in 4,5-dihydrothiophene resonate at 6.06 ppm and 5.48 ppm while that of the corresponding dioxide are found at 6.66 and 6.81 ppm, respectively.

2.09.3.2.2 Carbon-13 NMR spectroscopy

Carbon-13 NMR studies of thiophenes have been carried out since the 1960s <65JA5333, 68JA3543, 70JPC2765>, and extensive investigations carried out later <75CS76, 86HC(44/2)135, 91HC(44/4)1>. The ^{13}C NMR spectrum of thiophene (in acetone- d_6) shows the C-2 and C-3 carbons at 125.6 and 127.3

ppm, respectively $\langle 65\text{JA}5333, 75\text{CS}76 \rangle$. The signal of the C-2 position is more upfield than the C-3, contrary to the ^1H NMR spectrum where the H-3 proton is more upfield. The larger value of the ^{13}C —H coupling constants for the 2-position as compared to the 3-position (185 Hz vs. 168 Hz) is helpful in the structure elucidation of trisubstituted derivatives. The long range ^{13}C —H coupling constants are also distinctive and vary from 3–10 Hz which again is helpful in structure identification.

The ^{13}C NMR chemical shifts of some substituted thiophenes along with their shifts (relative to thiophene) are listed in Table 16. In most cases, a substituent causes a downfield shift of the carbon except for bromo, iodo, and cyano substituents. Electron-withdrawing substituents generally cause downfield shifts of all the other carbon atoms. Highly electronegative substituents or electron-donating groups cause the largest downfield shift on the ipso carbon atom but the adjacent carbons are upfield as compared to thiophene. For substituents at the 2-position, the C-4 carbon resonances are the least affected, while for 3-substituted thiophenes, the C-4 and C-5 resonances do not change much.

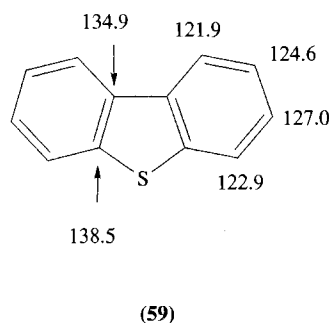
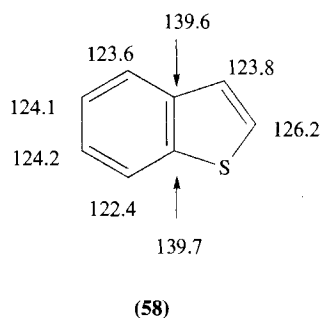
Table 16 Carbon-13 NMR chemical shifts (ppm) of some substituted thiophenes^{a,b}.

Substituent	C ₂	C ₃	C ₄	C ₅
H	125.6	127.3	127.3	125.6
2-Me	139.8(+14.2)	125.9(−1.4)	127.3(0)	123.7(−1.9)
2-F	166.5(+40.9)	108.0(−19.3)	124.7(−2.6)	114.9(−10.7)
2-Cl	129.7(+4.1)	127.4(+0.1)	129.6(+2.3)	125.3(−0.3)
2-NO ₂	151.2(+25.6)	129.9(+2.6)	128.7(+1.4)	134.2(+8.6)
2-OMe	167.4(+41.8)	104.2(−23.1)	125.5(−1.8)	112.4(−13.2)
2-Ph ^c	143.5(+17.9)	123.9(−3.4)	126.5(−0.8)	122.2(−3.4)
3-Me	121.3(−4.3)	138.2(+10.9)	130.1(+2.8)	126.1(+0.5)
3-F	104.2(−21.4)	159.2(+31.9)	117.8(−9.5)	126.4(+0.8)
3-Cl	120.9(−4.7)	125.7(−1.6)	128.1(+0.8)	127.5(+1.9)
3-NO ₂	129.2(+3.6)	149.7(+22.4)	123.1(−4.2)	128.9(+3.3)
3-OMe	97.3(−28.3)	159.8(+32.5)	119.9(−7.4)	125.6(0)
3-Ph ^c	120.1(−5.5)	142.3(+15.0)	126.2(−1.1)	126.2(+0.6)
2,5-di-Me ^d	137.4(+11.8)	125.3(−2.0)	125.3(−2.0)	137.4(+11.8)

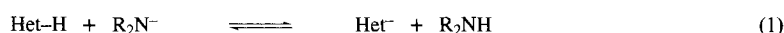
^aValue in parenthesis corresponds to shifts relative to thiophene. ^b $\langle 75\text{CS}76 \rangle$. ^c $\langle 86\text{HC}(44/2)135 \rangle$.

^d $\langle 65\text{JA}5333 \rangle$.

Although the C-2 carbon atom in thiophene is upfield with respect to the C-3 carbon, it is 2.4 ppm downfield to the C-3 carbon atom in benzo[*b*]thiophene $\langle 79\text{OMR}(12)647 \rangle$. The ^{13}C chemical shifts for benzo[*b*]thiophene (**58**) $\langle 76\text{OMR}(8)252 \rangle$ and dibenzothiophene (**59**) are given below.



Carbon-13 NMR spectroscopy has also been used to determine acidity measurements of heterocyclic compounds $\langle 83\text{CC}620, 85\text{CJC}3505 \rangle$. The heterocycle in question is treated with the lithium salt of an amine which is less acidic by about 4 pK units. At equilibrium, the concentration of the four species in Equation (1) is measured by ^{13}C NMR.



The equilibrium constant $K = K_1/K_2$ and thus $\Delta\text{p}K = -\log K = \text{p}K_1 - \text{p}K_2$. Based on these experiments, the proton acidity $\text{p}K_1$ of thiophene is determined to be 33.0, while that of benzo[*b*]thiophene is 32.4. Previously, the proton acidity of thiophene determined in cyclohexylamine was found to be 38.2 $\langle 73\text{JA}6273 \rangle$ indicating that these values are solvent sensitive.

The molecular r_x structure of thiophene dissolved in liquid crystals Merck ZLI 1167 and Phase IV has been determined through the use of ^1H — ^{13}C satellite NMR spectra $\langle 83\text{OMR}(21)143 \rangle$. An analysis of the substituent effect on ^{13}C and ^{17}O NMR chemical shifts of 5-substituted 2-acetylthiophenes has been studied by linear free energy relationships $\langle 93\text{ACS}160 \rangle$. The substituent–ring–probe interactions here are larger than those found in similar benzene derivatives. The data also confirms the fact that the acetyl group conjugates with the heteroaromatic ring (see also 2.09.3.1 and 2.09.4.2).

2.09.3.2.3 Sulfur-33 NMR spectroscopy

The relatively low abundance of ^{33}S (0.74%), the nuclear spin of 3/2 and the low sensitivity (2.3×10^{-3}) as compared to hydrogen, make ^{33}S NMR spectra difficult to study. The ^{33}S NMR spectrum of thiophene was initially studied as a solution in carbon disulfide $\langle 70\text{MI } 209-01 \rangle$ and its chemical shift relative to carbon disulfide was determined to be -220 ± 6 while those of 2- and 3-methylthiophene were -178 ± 9 and -197 ± 26 , respectively.

The ^{33}S chemical shift of neat thiophene (relative to aqueous caesium sulfate) was found to be -119 ppm, while that of its saturated analogue tetrahydrothiophene was -354 ppm $\langle 85\text{JCS}(F2)63 \rangle$. For tetrahydrothiophene-1,1-dioxide (sulfolane) the solvent choice and dilution is important. The ^{33}S chemical shift of neat sulfolane is 36.7 ppm and shifts upfield when the solution is diluted. A shift of 6.5 ppm is observed when the solvent system is changed from water to dioxane and the line widths for 5 mol dm $^{-3}$ solutions of sulfolane in acetone and in water are 16 and 60 Hz, respectively.

The ^{33}S quadrupole coupling constants in thiophene and tetrahydrothiophene have been determined by ^{13}C and ^{33}S relaxation time measurements $\langle 86\text{ACS}(A)218 \rangle$.

2.09.3.2.4 Mass spectroscopy

The 4.4% contribution of the ^{34}S isotope to the $M + 2$ peak and invariably to a (fragment + 2) peak is a good indication of a sulfur-containing compound. Thiophene, along with the other five-membered heterocycles in its group show similar ring fragmentation patterns; the first step being cleavage of the C—S bond (Figure 2). From there, it can proceed by three different pathways to afford three principal peaks: C_3H_3^+ (m/z 39) (**60**), $\text{HC}\equiv\text{S}^+$ (m/z 45) (**61**), and $\text{C}_2\text{H}_2\text{S}^+$ (m/z 58) (**62**).

For thiophene, its molecular ion is the base peak. Although the mass spectrum of thiophene was studied as early as in the 1950s, $\langle 59\text{CCC}1602 \rangle$, it was only recently that the detailed structures of the fragments were determined. The spectra of ^2H - and ^{13}C -labeled thiophenes show that considerable hydrogen and carbon scrambling occurs on electron impact $\langle 69\text{OMS}(2)241, 72\text{AJC}1221, 91\text{HC}(44/4)1 \rangle$.

The fragment ions (**60**) and (**61**) are formed from a common precursor, while formation of (**62**) is presumed to arise from two pathways depending on the beam energy. Initial fragmentation of the C(3)—C(4) bond with concomitant ring closure and loss of alkyne affords (**62**). It is also possible that initial cleavage of the C(2)—C(3) bond is followed by cleavage of the C(4)—C(5) bond which eliminates alkyne and forms (**62**) $\langle 84\text{CHEC-I}(4)713 \rangle$. Furthermore, it is this ion which has the maximum intensity contrary to pyrrole and furan where (**60**) is the most intense peak $\langle 84\text{CHEC-I}(4)1 \rangle$.

A quantum chemical interpretation of the MS fragmentation of organic molecules has been investigated. The interpretation is based on the premise that bond cleavage caused by electron impact is related to their self-consistent π -electron orders $\langle 87\text{CHE}512 \rangle$. The pattern of the spectrum is determined by rearrangements of the intermediate cation radicals or cations. Cyclizations occur via the long-range bond orders which to a certain degree are a measure of its aromaticity. Based on the bond orders, the weakest bond is the C(3)—C(4) bond and hence should fragment first and to a much greater extent than the S—C or the C(2)—C(3) bond. This then forms the divinyl sulfide radical cation (**63**) which cyclizes to (**64**) and then eliminates acetylene to form (**62**) (Figure 3). On the other hand, it is also possible that the bicyclic intermediate (**65**) is formed first which could fragment to form (**66**) and/or (**64**) both of which lose acetylene to form (**62**). Theoretical calculations predict (**62**) to be the major fragment in thiophene while predicting (**60**) to be larger in the case of furan and pyrrole, in line with that observed experimentally.

For 2- and 3-alkyl thiophenes, the base peak at m/z 97 is assigned to the $\text{C}_5\text{H}_5\text{S}^+$ ion which arises from the β -cleavage of the alkyl substituent (Figure 4) and the thiopyrylium structure (**67**) is proposed. The thiopyrylium cation (**67**) is the base peak for long chain alkyl groups at the 2-

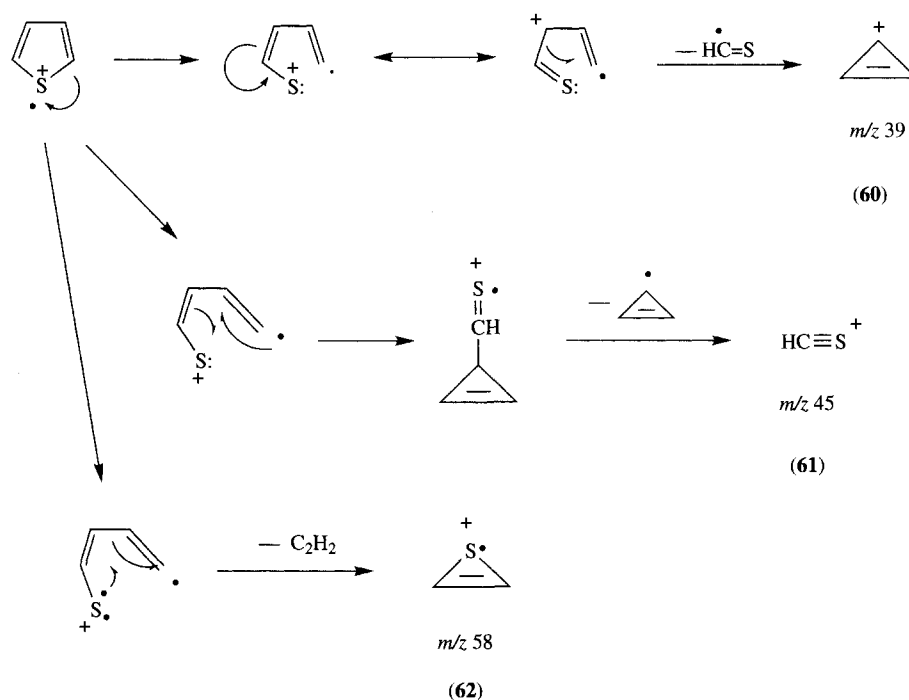


Figure 2 Fragmentation pattern in thiophene.

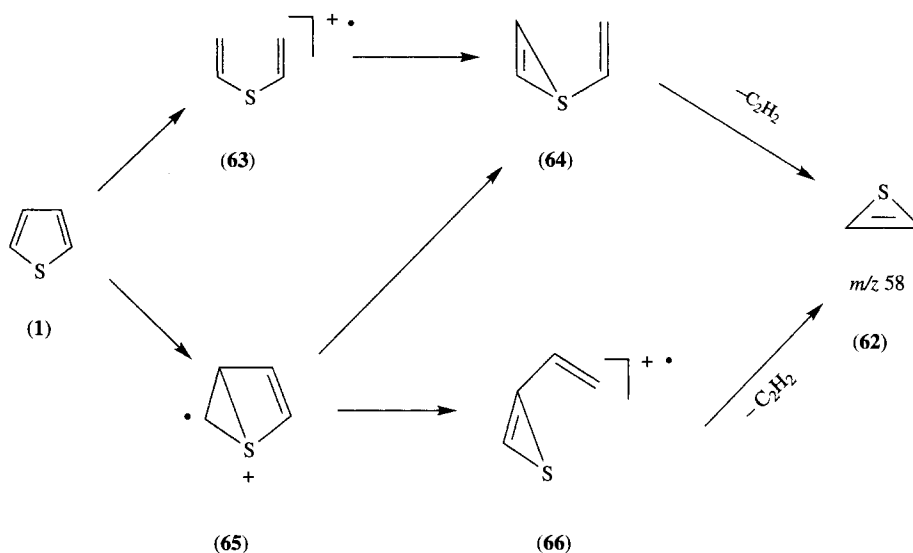


Figure 3 Fragmentation pattern based on bond orders.

position. If another such group is present at the 5-position, the molecular ion is found to be the base peak <B-67MI 209-01, 76JCED380>.

In the collision-activated dissociation MS of thiophenic ions, the ions formed due to the loss of the $\text{SH}^\bullet$ radical are prominent in polythiophenes but not in the case of alkylthiophenes <82ANC574>. The ions are generated under positive chemical ionization conditions.

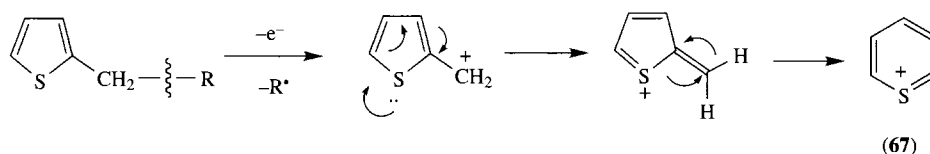
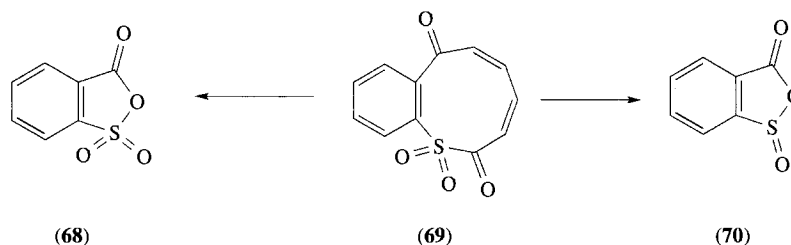


Figure 4 Fragmentation pattern for 2-alkylthiophenes.

Dibenzothiophene undergoes surface-catalyzed oxidation when subjected to negative chemical ionization (NCI) with oxygen <93MI 209-02>. In the mass spectrum, the m/z ion at 184 initially attributed to $M^{-\bullet}$, is now shown to be the anion of 2-sulfobenzoic acid cyclic anhydride (**68**). Formation of (**68**) is presumed to arise by initial oxidation of dibenzothiophene to the sulfone (m/z 216) followed by oxidation of the angular carbon atoms to form the bicyclic intermediate (**69**) (m/z 248). Further attack by oxygen cleaves the ring to form (**68**) or (**70**) (Scheme 4). The mechanism has been proposed on the basis of further studies on the NCI(O_2) MS of dibenzothiophene-5-oxide, -5,5-dioxide, and also of various alkyl dibenzothiophenes where similar patterns were observed.



Scheme 4

2.09.3.2.5 Ultraviolet spectroscopy

The ultraviolet spectrum of thiophene and its derivatives has been extensively investigated <72JCS(F2)2009, 78JCP(69)5077, 81BCJ1511, 82JCS(P2)761, 86HC(44/3)1, 91HC(44/4)1>. In the gas phase, the UV spectrum consist of three bands originating at 240, 207, and 188 nm, respectively. Initial reports <41PRS201> claimed only one transition between 200–240 nm while Milazzo described three transitions <48G835>.

High resolution and temperature dependance measurements were recorded for the lowest energy UV transition (230–250 nm) of thiophene. Based on these results, the first system of the UV spectrum is assigned to the $\psi_4 \leftarrow \psi_3$ (B_2) transition <82JCS(P2)761>. The results were confirmed by comparisons of the UPS and UV spectral properties of the Group 16 five-membered heterocycles. A vibrational progression in the 965 cm^{-1} frequency mode is found to dominate. The absorption spectra between 225 nm and 246 nm has also been studied at elevated temperatures <85SA(A)1413>. At 296 K, 13 features are observed between 227.6 nm and 242.7 nm. At 573 K there is almost no vibrational structure in the system, while near 773 K the vibration structure is lost.

Based on the correlation of electron transitions and ionization potentials, the transition at 207 nm was assigned as the $A_1 \leftarrow A_1$ ($\pi_4^* \leftarrow \pi_2$) valence transition <86JST(140)253>. The spectra of halothiophenes has been recorded between 200–225 nm. The band system observed in this region in the vapor phase, is absent in solution spectra and are assigned to Rydberg transitions.

The electronic absorption spectra of 2,2'-bithienyl and its derivatives have also been investigated <85BCJ2126> and the transitions in the spectra of 5- and 5,5'-substituted 2,2'-bithienyl compounds assigned to intramolecular charge transfer, locally excited transitions, and $n \rightarrow \sigma^*$ transitions. The MOs of 2,2'-bithienyl are built up of two thiophene molecules and excited states arise from mutual interaction of these excited states. This interaction is maximum when the molecule is planar. Distortion causes the spectrum of bithienyls to approach that of thiophene. In thiophene, a singlet $\pi \rightarrow \pi^*$ transition is observed at 235 nm as well as a shoulder at about 268 nm. By comparison, the spectrum of 2,2'-bithienyl shows three well defined regions, none of which corresponds to thiophene, indicating the molecule to be coplanar, rigid, or semirigid. The three transitions are all $\pi \rightarrow \pi^*$ and the broadness of the lowest energy band indicates that, in solution, a number of almost planar

conformations exist, all of which are of comparable energy. The coplanar configuration could be either *cis* or *trans*, with the latter having a smaller dipole moment. The spectrum displays a very small shift with solvent polarity, implying that the molecule possesses low polarity and thus the *trans* conformation. Solid state studies show the molecule to favor the planar-*trans* orientation $\langle 68\text{AX(B)467} \rangle$ while gas phase and solution studies indicate it to be twisted $34\text{--}85^\circ$ from the *cis* form $\langle 85\text{BCJ2126} \rangle$.

2,2'-Bithienyl with alkyl or chloro substituents shows similar patterns. The presence of formyl or acetyl groups at the 5- and 5,5'-positions causes a significant shift for the lowest energy transition due to the perturbation caused by the carbonyl group which is shifted in polar solvents. Theoretical calculations indicate that 5-formyl-2,2'-bithienyl is roughly planar and interaction between thiophenecarbaldehyde and thiophene is predominant.

2.09.3.2.6 Infrared spectroscopy

The IR spectra of five-membered heterocycles has been extensively surveyed. For thiophenes, this was first done by Gronowitz in the early 1960s $\langle 63\text{AHC(1)1} \rangle$ and has been reviewed again $\langle 91\text{HC(44)41} \rangle$. The literature is also rich with theoretical calculations on the vibrational frequencies of thiophene and on its oligomers (see 2.09.2.8).

The 21 fundamental vibrations of thiophene are composed of eight vibrations of A_1 symmetry, seven of B_1 symmetry, and three each of A_2 and B_2 . The assignments of all 21 vibrations have been documented by Rico *et al.* as early as 1965 $\langle 65\text{SA689} \rangle$ and are listed in Table 8. Based on these frequencies, harmonic force constants, and mean amplitudes of vibration have been developed $\langle 69\text{ACS3139, 69ACS3407} \rangle$.

The complete vapor phase assignment including IR vapor, Raman vapor, and liquid spectra, have been determined to obtain a complete set of vibrational frequencies in the vapor and liquid states of thiophene $\langle 94\text{SA(A)765} \rangle$. The results confirm the assignments made earlier by Rico *et al.* $\langle 65\text{SA689} \rangle$ and, for several of the fundamental modes, the vapor frequency has now been measured or improved.

Due to the presence of the larger sulfur atom and correspondingly longer C—S distance, the *a* and *b* axial moments-of-inertia are not similar. Thus, in the IR vapor spectrum, the A_1 -type bands now exhibit a clearer and more intense central Q-branch. Nine of the twelve possible combinations of the A_1 transition are identified by Q-branches in the Raman vapor spectrum. Nineteen new transitions have been observed here which were not previously reported by Rico *et al.*

For the B_1 species, the C—H stretching mode at 3125 cm^{-1} is unobserved here. It is possible that it is obscured by ν_1 for observation in either IR or Raman spectra. In the liquid Raman spectrum, the fundamental at 1085 cm^{-1} is also not observed, being completely hidden by the A_1 band ν_5 . The A_2 and B_2 fundamentals exhibit $3\text{--}5\text{ cm}^{-1}$ positive shifts from the vapor to liquid states for the C—H wag groups and $\approx 1\text{ cm}^{-1}$ for the nonplanar ring modes.

The far IR absorption spectrum of thiophene in the liquid and plastic phase has been studied over the region $10\text{--}200\text{ cm}^{-1}$ $\langle 81\text{SA(A)977} \rangle$. The frequencies indicate the presence of librational motions of the molecule in potential wells and are highly hindered.

The temperature dependence on the IR absorption and Raman scattering bandwidths of some fundamental modes of A_1 , B_1 , and B_2 symmetries of thiophene has been investigated in the region $400\text{--}1600\text{ cm}^{-1}$ $\langle 81\text{CPH251} \rangle$. The spectroscopic study was carried out in the liquid phase which requires simultaneous investigation of the bands. Results show that the IR and anisotropic Raman bandwidths increase with increasing temperature and more so in the latter case. The rotational diffusion coefficients obtained from the bandwidths indicate that the rotational and vibrational relaxation phenomena occur simultaneously. The IR and isotropic and anisotropic Raman band profiles of the A_1 symmetry mode have also been studied in the liquid phase $\langle 81\text{CPH265} \rangle$.

The near-IR-visible vibrational spectra of thiophene compounds is dominated by two distinct anharmonic aryl C—H stretching overtones $\langle 91\text{JPC7659} \rangle$. At particular levels of C—H stretch excitation, the overtone pattern is disturbed leading to a broadening and a loss of vibrational structure at $\Delta\nu_{\text{CH}} = 4$, while being retained at $\Delta\nu_{\text{CH}} = 3$ and 5 $\langle 93\text{JPC809} \rangle$. The sensitivity of the spectral patterns to small changes in the C—H stretching frequency arises due to isolated nonlinear resonances present in these regions.

The spectrum of the out-of-plane band of thiophene centered at 712 cm^{-1} has been measured in Doppler-limited resolution with a diode-laser spectrometer and with a high-resolution FT spectrometer $\langle 93\text{ZN(A)1193} \rangle$. The band at 712 cm^{-1} which is the ν_{19} band $\langle 65\text{SA689} \rangle$, is of symmetry B_1 .

Here, the heavy atoms of the ring displace vertically to the molecular plane. This moment involves a variation of the dipole moment in this direction and thus displays a *c*-type transition. The planarity defects in the centrifugal distortion are very small. In the excited state, the inertial defects in the ν_{19} band are found to be large and negative.

For 2- and 3-substituted thiophenes, the three C—H ring stretching bands are observed in the region 1350–1550 cm^{-1} , the intensities of which depend on the type of substitution $\langle 70\text{SA(A)}1651 \rangle$. In addition to this, up to two bands corresponding to in-plane C—H deformations are observed in the region 1030–1085 cm^{-1} . Furthermore, the absorption pattern in the region 750–900 cm^{-1} is different for 2- and 3-substituted derivatives making it easy to identify the substitution pattern. Similarly, 2,5-dialkylthiophenes display a strong absorption at ca. 800 cm^{-1} $\langle 64\text{CB3263} \rangle$.

The IR and Raman spectra of thiophene, 2,2'-bithienyl and of the cycloalkane end-capped oligothiophenes has been studied and the results compared with theoretical calculations at the *ab initio* SCF level $\langle 93\text{JST}(298)65 \rangle$. The scaled theoretical and experimental vibrational frequencies and theoretical IR and Raman intensities as well as their assignments for the above compounds have been discussed.

2.09.3.2.7 Photoelectron spectroscopy

The photoelectron spectra (PES) of thiophenes has been well studied $\langle 69\text{IJM}471, 70\text{T4505}, 71\text{IJM}(6)177, 76\text{JES179}, 79\text{CPL}(61)355 \rangle$. The UV PES of thiophene (using He^{I} (21 eV) photon excitation) was first measured up to 18 eV by Eland $\langle 69\text{IJM}471 \rangle$, and later up to 25 eV by Derrick *et al.* using He^{II} (40 eV) photon excitation $\langle 71\text{IJM}(6)177 \rangle$. Theoretical studies have been carried out to assist in assigning the ionization energies to the appropriate occupied molecular orbitals and this has been discussed in Section 2.09.2.6.

Of the three lowest ionization potentials, the band at 8.87 eV has been assigned to the $1a_2(\pi_3)$ orbital, while that at 9.49 eV is due to the $2b_1(\pi_2)$ orbital $\langle 71\text{IJM}(6)177 \rangle$. Both these orbitals are found to be weakly C—C bonding. The intensive band at 12.1 eV assigned earlier to be the π_2 band was later shown to arise from σ orbitals and is now assigned as the $6a_1$ orbital. The Penning spectrum $\langle 80\text{JES235} \rangle$ correctly assigns the band at 12.5 eV to be the $1b_1(\pi)$ orbital.

The He^{I} and He^{II} PES of thiophene was later rerecorded up to 15 eV $\langle 82\text{JCS(P2)}539 \rangle$. Based on their observations and from previous theoretical calculations (see Section 2.09.2.6), the seven band systems (with their ionization energies (eV) in parenthesis) are assigned as: $1a_2(\pi)$ (8.85), $2b_1(\pi)$ (9.49), $6a_1(\pi)$ (12.00), $1b_1(\pi)$ (12.46), $4b_2$ (13.11), $5a_1$ (13.80), $3b_2$ (14.23) which is similar to the *ab initio* results of von Niessen *et al.* $\langle 76\text{JES179} \rangle$.

Ionization energies of thiophene derivatives with electron releasing substituents at the 2-position cause an increase in the separation between the π_2 and π_3 orbitals. This arises due to a greater destabilizing effect on the energy of the π_3 orbital than on the π_2 orbital. Electron withdrawing substituents at the 2-position increase the ionization energies of both orbitals $\langle 76\text{JCS(P2)}276 \rangle$.

The He^{I} line PES has also been determined for a variety of thiophene-fused troponones $\langle 84\text{BCJ856} \rangle$. The PES of the thiophenotroponones are quite similar to the corresponding benzo-troponone analogues. This resemblance for the first band system is due to the fact that the initial ionizations are related to the molecular orbitals that are mainly localized in the troponone ring rather than the benzene or heterocyclic ring. Since the electronegativities of sulfur and carbon are approximately the same, replacement of a $\text{CH}=\text{CH}$ bond by sulfur does not alter the IP to a noticeable extent.

Photoelectron spectroscopy is an efficient tool for the gas phase characterization of various elusive compounds $\langle 89\text{CSR317} \rangle$. It has also been used to investigate the products formed on flash vacuum pyrolysis of alkylthio derivatives of Meldrum's acid $\langle 91\text{JOC3445} \rangle$. The PES of the thiophen-3(2*H*)-ones formed were similar to authentic samples from which it is apparent that no keto–enol tautomerism occurred in the gas phase and that only the keto tautomers are formed in the gas phase.

The ionization potentials of benzo[*b*]thiophene $\langle 69\text{IJM}471 \rangle$ and dibenzothiophene $\langle 73\text{JCS(F2)}1155 \rangle$ have also been determined and the assignments compared with various theoretical calculations $\langle 83\text{JST}(105)375 \rangle$.

The x-ray photoelectron spectrum (XPS) of thiophene has been measured in the vapor phase using $\text{Mg-}K_{\alpha}$ x-ray excitation $\langle 71\text{MI}209-01 \rangle$. The x-ray spectra complement PES since x-ray transmissions are controlled by selection rules and thus enables the valence MO structure to be studied. Initial studies of x-ray emission from vapor phase thiophene have been limited to the $\text{S-L}_{\text{II,III}}$ $\langle 76\text{JCP}(64)3021 \rangle$ and to the $\text{S-}K_{\beta}$ measurement $\langle 74\text{MI}209-01 \rangle$. The high resolution sulfur K_{β} emission

and S-K absorption spectra of vapor phase thiophene have been measured <86JCP(84)4228>. The data has been compared with MNDO MO calculated intensities and the sulfur 1s binding energy determined to be 2477.6 eV.

2.09.4 THERMODYNAMICS

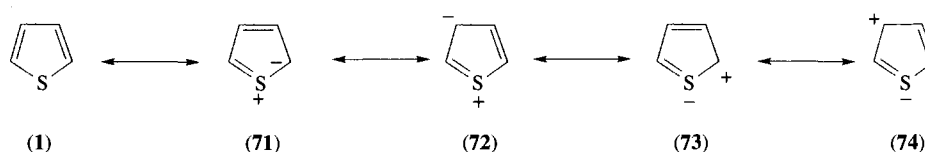
2.09.4.1 Aromaticity

The fact that aromatic compounds <B-71MI 209-02, B-89MI 209-02> differ from unsaturated aliphatic compounds was recognized as early as the nineteenth century. However, chemists are still unable to agree on a suitable definition of aromatic character <68MI 209-01>. Qualitatively, the concept of aromaticity is quite clear <74AHC(17)255, B-85MI 209-02>. A compound is considered aromatic if it:

- (i) is a cyclic compound with a large resonance energy, i.e., its stability is increased when compared to its classical localized structure;
- (ii) possesses an aromatic sextet which arises due to the presence of a closed loop of electrons;
- (iii) follows the Hückel rule which states that a ring system with $(4n + 2)\pi$ electrons is aromatic while one with $(4n)\pi$ electrons is not;
- (iv) is capable of sustaining an induced ring current;
- (v) has a lack of marked bond length alternation; and
- (vi) displays enhanced thermo stability.

Most aromatic compounds possess the aromatic sextet and are generally six-membered rings. However, these sextets are also found in five-membered rings, when the ring is made up of a pair of double bonds and the fifth atom possesses an unshared pair of electrons. The five *p*-orbitals overlap to form three bonding and two antibonding orbitals. Pyrrole, furan, and thiophene are some of the heterocycles that fall under this category.

Although it is easy to establish that naphthalene is less aromatic than two benzene rings or that benzo[*c*]thiophene is less aromatic than benzo[*b*]thiophene, it is more difficult in other cases. Thus a quantitative scale of aromaticity is needed. Attempts have been made in a few reviews to introduce methods and concepts where by it is possible to justify an aromaticity scale to determine and thus view aromaticity as a quantitative concept <74AHC(17)255, 80IZV3, 85CHE717, 91H(32)127>. Important criteria for heterocycles include its hybridization, electronegativity, and stereochemistry. For thiophene, the matter is further complicated by the fact whether the 3*d*-orbital of sulfur also participates in bond formation. If so, then the two resonance structures (73) and (74) in Scheme 5 should also be considered in determining the degree of aromaticity. However, as was previously determined (see Section 2.09.2.3), calculations have shown that for the ground state, the energy of thiophene is not affected by the inclusion of the 3*d*-orbitals.



Scheme 5

In order to determine a quantitative measurement of aromaticity, one must be capable of obtaining:

- (i) experimental methods that measure the energy or any particular parameter of the compound and compare it with a classical compound that does not possess the cyclic conjugation;
- (ii) experimental methods that have investigated geometries and magnetic properties of aromatic compounds and compare these with nonaromatic compounds; and
- (iii) MO calculations dealing with the above.

Although there is a large amount of data available, it is still difficult to construct an accurate aromaticity scale for the following reasons:

- (i) experimental results for any specific parameter are generally not available for a full series of compounds;
- (ii) errors in experimental measurements obtained from various sources;
- (iii) the precise definition of the nonaromatic or classical model that should be used to compare the aromatic compound with; and

(iv) doubt as to choice of proper parameters to be used for theoretical MO and semiempirical methods and especially for heteroatoms possessing *d*-orbitals such as sulfur.

Early methods for the determination of aromaticity involved resonance energies which were determined from heats of hydrogenation and combustion <B-75MI 209-02>. Although the results by these two methods are quite similar for benzene, their application to heterocycles are more difficult. Combustion of heterocycles leads to oxyacids which can then attack the materials used for combustion or due to poisoning of the catalyst in the hydrogenation method. Variations arise from inconsistencies in the thermochemical data for the localized models, as well as selection of the model itself. For example, the stabilization energy of benzene is calculated to be 36 kcal mol⁻¹ when the localized model is assumed to be cyclohexane and 48.7 kcal mol⁻¹ from ethylene <B-75MI 209-02>. The large variations in the resonance energies of thiophene obtained from various studies is depicted in Table 17. Although the values vary, the order of aromaticity generally observed is benzene > thiophene > pyrrole > furan.

Table 17 Resonance energies (kcal mol⁻¹) calculated from heats of combustion.

Ref.	Benzene	Thiophene	Ref.	Benzene	Thiophene
33JCP(1)606	37.0	31.0	B-55MI 209-02	36.0	28.7
49CB358	35.9	29.1	B-55MI 209-02	36.4	27.7
50JA4278	36.0	31.0	B-62MI 209-01		17.1 ^a
51CB916	36.0	24.1	B-75MI 209-02	36.0 ^a	17.1 ^a
63T1175	22.0	16.0			

^aFrom heats of hydrogenation.

Molecular orbital calculations have also been employed for the comparison of aromatic resonance energies. Although the Hückel rule is generally used as a distinction for aromaticity, difficulties arise in its application for large annulenes and so is not usually used as an index for the classification of aromaticity <B-69MI 209-01>. The Dewar resonance energy (DRE) assumes the localized structure to be a conjugated polyene and is determined as $DRE = \Delta H_f - \Delta H_{f(\text{loc})}$ <67MI 209-02>. For heterocyclic compounds, a modified version of the resonance energy calculation (DRE') is used where the resonance energy is calculated from the AM1 heats of formation <70JA1453>.

The Hess-Schaad resonance energy (HSRE) is another MO-based method used to determine aromaticity and is based on the difference between the energy of the HMO π -system and the energy of the empirically isolated π -bond <71JA305, 73JA3907, 75T295>. In order to obtain the energies of the localized sulfur precursor, the π -bond energies are evaluated from conjugated acyclic polyenes containing a sulfur atom. This is done in an additive fashion from the empirical bond energies of the respective C—C bonds and carbon-heteroatom bonds. In this manner, the resonance energies of a variety of cyclic and bicyclic thiophenes, thiaannulenes, thienothiophenes, and other fused thiophene derivatives have been determined <73JA3907>.

The resonance energy for thiophene as calculated by Dewar is found to be 6.5 kcal mol⁻¹ and shows a decrease in aromaticity in the order benzene > thiophene > pyrrole > furan. This is in agreement with other criteria such as chemical reactivity, etc. The HSRE is found to be 4.5 kcal mol⁻¹ and the order is benzene > pyrrole > thiophene > furan. Furthermore, the resonance energies obtained by this method are generally much lower than those by Dewar. Obviously, the parameters are defined much more clearly in the Dewar system.

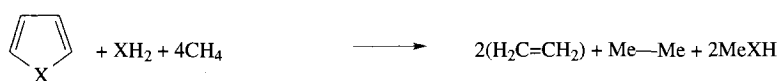
Heats of formation calculated by the AM1 method can be used to determine aromatic energies of heterocycles <89H(28)1135>. The aromatic energy of an aromatic molecule is defined as minus the difference between its energy and that of its analogue in which one of the bonds in the ring is replaced by a localized bond. A positive value implies stabilization. This is quite easily done if the ring contains an even number of atoms, but not necessarily so for odd-numbered rings.

The heteroaromatic analogues are more complicated due to the effects of ring strain and the problems in finding suitable nonaromatic precursors. Since AM1 calculations are generally not very accurate for these compounds the aromatic energies are calculated using experimental values for the heats of formation. Thiophene can be considered to be formed from divinyl sulfide. However, since the heats of formation of divinyl sulfide is not available, it is estimated from the heats of formation of diethyl sulfide <B-86MI 209-03>. Assuming the heat of hydrogenation of divinyl sulfide is similar to divinyl ether, the heat of formation for classical (strain-free) thiophene is calculated to be 43.9 kcal mol⁻¹. With the observed ΔH_f for thiophene being measured at 27.5 kcal mol⁻¹, this gives the estimated value of the aromatic energy of thiophene to be 16.4 kcal mol⁻¹. By comparison,

the aromatic energies of pyrrole and furan are estimated to be 22.5 and 12.1 kcal mol⁻¹, respectively, indicating pyrrole to be more aromatic than thiophene.

Another quantitative index of aromaticity is the measure of the delocalization stabilization. Here the stability of the parent molecule is compared to the isolated classical bonds obtained by the bond separation reaction or the superhomodesmotic reaction <88JA4204>. In the latter case, the prototypes contain larger fragments of the parent heterocycle. The energy differences (ΔE) and the zero-point enthalpy differences (ΔH^0_0) for these reactions have been calculated using the 3-21G* Hartree-Fock energies. The bond separation reaction and the superhomodesmotic reaction for thiophene and for the Group 16 five-membered heterocycles in general is depicted in Figure 5.

Bond separation reaction



Superhomodesmotic reaction

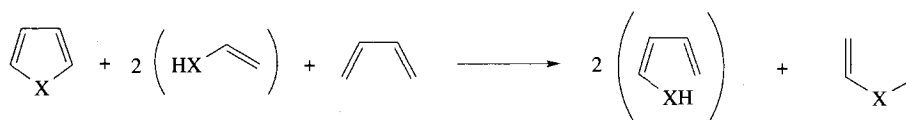


Figure 5 Aromaticity as determined by bond separation and superhomodesmotic reactions.

For thiophene, the energy differences by the bond separation and the superhomodesmotic reactions are 30.21 and 10.07 kcal mol⁻¹, respectively, while the zero-point enthalpy differences are 32.54 and 9.80 kcal mol⁻¹, respectively. In general, the values for the energy differences by the superhomodesmotic reaction are much lower than those obtained by the bond separation method. This is due to the fact that in the former case, there is a much smaller change in the molecular and electronic structures since the predecessor is made up of much larger fragments of the parent molecule. For the Group 16 five-membered rings, results from the superhomodesmotic reaction predict thiophene and selenophene to be more stabilized than furan.

Ab initio SCF-MO studies on the aromaticity of some five-membered heterocycles have mostly been performed at the STO-3G level, while for sulfur, the STO-3G* level is also included <88JST(163)173>. Three approaches were studied; the first uses a quantitative perturbation molecular orbital (PMO) approach and is known as the PMO method, the second is based on the computation of the π -orbital energies and is called the orbital energy method, while the total energy method, which as its name suggests, deals with the total energy approach. Optimized geometries are calculated for the compounds of interest at the levels indicated above and the values used to determine the π delocalization energies.

The PMO method tends to fail when the perturbation becomes large as in cases where bonding π interactions are studied. Hence PMO estimates of the π delocalization energy tend to be inaccurate to a certain degree. It however affords an estimation of the energy effect associated with every π -orbital interaction. On the other hand, the orbital energy method does not highlight specific energy effects associated with the various orbital interactions but does provide accurate values of the overall π delocalization energy. The two methods thus complement each other quite well and hence are generally used together.

In the total energy method, the π delocalization energies are calculated as the difference between the total energy of the composite system (E_T) and the total energy of the system in absence of all π interactions (E^0_T). The PMO method and the total energy method both show thiophene to be more stabilizing than their acyclic counterparts. The same holds true for the other five-membered rings pyrrole and furan. For cyclopentadiene, the values indicate it to be destabilizing and more so than

its acyclic analogue. For thiophene, the results obtained using the STO-3G* values parallel that of the experimental resonance energies viz. benzene > thiophene > pyrrole > furan.

The total energy method, however, does not afford satisfactory results for thiophene. STO-3G values show thiophene and pyrrole to be destabilizing by this method. Here, the presence of a C—X bond indicates the importance of the inductive effect, and hence the π delocalization energy is small. If one considers the difference $\Delta(E_T - E^0_T)$ between the cyclic and the acyclic species, the energy effects associated with the heterocycles become stabilizing and the trend follows that observed from experimental values. Thus, while the π delocalization energy from the total energy method ($E_T - E^0_T$) affords a value of 2.62 kcal mol⁻¹, $\Delta(E_T - E^0_T)$ gives a value of -8.36 kcal mol⁻¹.

For heterocyclic systems, the interaction between the butadiene fragment and the heteroatom moiety deals with a four-electron destabilization interaction ($n_x - \phi_1$) involving the occupied orbital ϕ_1 of butadiene and the n_x orbital (which corresponds to a lone pair p -orbital) of the heteroatom, and a two electron stabilizing interaction ($n_x - \phi_3$) where ϕ_3 is the empty orbital of the butadiene fragment (Figure 6). In all cases, the stabilizing effect is larger than the destabilizing interaction and hence the overall result is stabilization. The destabilization interaction becomes much less in thiophene due to the longer C—S bond which results in a reduction in the overlap and a larger delocalization energy than in pyrrole or furan. The experimental trend is followed when the STO-3G* basis set is employed since the energy of the sulfur lone-pair orbital is lowered in going from the STO-3G to the STO-3G* basis set. The d -orbitals as such do not directly affect the π delocalization energies but reduce the energy gap between the ϕ_3 orbital of the butadiene fragment and the n_x orbital of sulfur, making the $n_x - \phi_3$ interaction for sulfur more stabilizing.

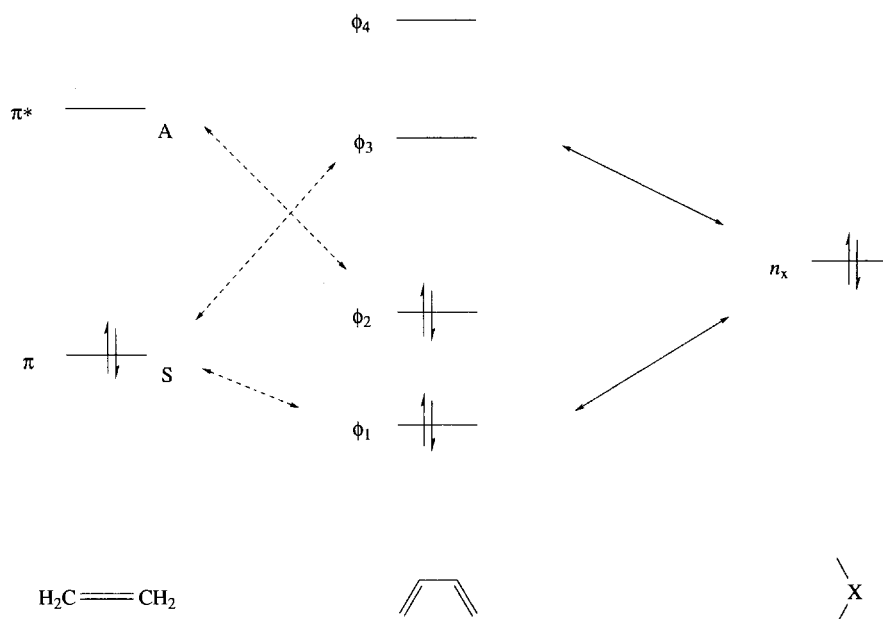


Figure 6 Interaction between the butadiene fragment and heteroatom moiety or ethylene fragment.

Benzene possesses a much larger π delocalization energy than the three heterocycles discussed here, due to the additional stabilizing interaction involving the ϕ_2 orbital of the butadiene fragment and the π^* orbital of the ethylene fragment, which is not possible in heterocycles. Since the energy difference between the two orbitals is quite small, this interaction is strongly stabilizing. The other two interactions are similar to those found for the heterocycles and hence this additional $\phi_2 - \pi^*$ interaction leads to a larger aromaticity of benzene.

A quantitative aspect of aromaticity can also be determined by nonenergetic methods. One criterion is the mesomeric dipole moment (μ_m) which is calculated as the difference between the dipole moments of the heterocycle and its fully saturated analogue (B-71MI 209-02). However, since the direction of the dipole moment in pyrrole is opposite to that of some of the other five-membered heterocycles, an unusually large value of μ_m for pyrrole causes this measure to be less reliable than some of the other quantitative concepts.

Ring geometry is another yardstick used to measure aromaticity. Here the ratio of the C(2)—C(3) to C(3)—C(4) bond length affords a quick estimate of ring aromaticity. This bond alternation

method is not too accurate since it does not account for the different sizes of the heteroatom, nor their electronegativity. A more reliable index is $\Sigma\Delta N$ which is the sum of the differences in bond orders of the three nonequivalent bonds $\langle 74JCS(P2)332 \rangle$. The bond order (N) of a bond in a molecule can be readily calculated from its bond length (R) using the relationship $N = aR^{-2} + b$ where a and b are constants obtained from the dependence of the bond order on bond length for selected standard compounds $\langle 47JCP(15)305 \rangle$. Although one can now compare aromaticities, it does not allow for comparison between different ring sizes. This problem was circumvented by Pozharskii $\langle 85CHE717 \rangle$ who developed a new index ΔN which is the average of the fluctuations of all the ring bonds including the single bonds.

The Julg aromaticity index (A_1) $\langle 67TCA249 \rangle$ is based on taking the mean square deviations of the C—C bond lengths as a measure of aromaticity, and is calculated by the formula shown in Equation (2).

$$A_1 = 1 - \frac{225}{n} \sum_{(rs)} \left(1 - \frac{d_{rs}}{d'} \right)^2$$

where n = number of π -electrons

d_{rs} = bond length

$d' = \frac{1}{n} \sum_{(rs)} d_{rs}$ i.e., the average bond length.

(2)

Since all bonds in benzene are equal in length, the bond alternation ratio and the Julg aromaticity index for benzene is one. The Julg index has been modified to account for resistance to circulation of the π -electrons which is a major factor in electronegative heteroatoms $\langle B-71MI 209-03 \rangle$. This correction factor A_2 is given by Equation (3).

$$A_2 = \prod_{(ij)} \left[1 - \left(\frac{\Delta q_{ij}}{d_{ij}} \right)^2 \right]$$
(3)

In Equation (3) Δq_{ij} is the difference in the π -charge on neighboring atoms, and d_{ij} the interatomic distance.

Based on this, the new Julg index $A = A_1 A_2$. This index however, has a few setbacks, the main one being the difficulty in evaluating the π -charges for molecules with heavy heteroatoms. The aromaticity indices of thiophene and benzene as determined by geometric criteria are shown in Table 18.

Table 18 Aromaticity indices from geometric criteria.

Criteria	Benzene	Thiophene	Ref.
μ_m	0	1.38	72JA8854
Bond alternation	1	0.963	61JSP(7)58
$\Sigma\Delta N$	0	0.90	74JCS(P2)332
ΔN	0	0.27	85CHE717
A_1	1	0.93	74JCS(P2)332
A	1	0.67	B-71MI 209-03
I_x	100	66	85T1409/86T89
I_A	100	81.5	92T335
RE	45.8	43	92T335

The character of a chemical bond is associated to its vibrational frequency. Bonds with low force constants possess high vibrational frequencies and small amplitudes of vibration and are rigid, while those with large mean amplitudes are the most reactive. Ionic and polar bonds fall into this latter category. The fundamental frequencies of a molecule can be obtained from vibrational spectra. From this, it is possible to calculate the force field which in turn, is used to obtain the mean amplitudes of vibration for the individual bonds in a molecule. This process has been studied in the Group 16 five-membered heterocycles $\langle 85MI 209-03 \rangle$.

The symmetric and asymmetric C—X stretching frequency decreases as one goes down the column from oxygen to tellurium while the interatomic distances increase. This implies a decrease in the force constants and a corresponding increase of the mean amplitudes of vibration. The C—O bond is thus more rigid than the C—Te bond. The force constants for the C=C is found to increase in

the order thiophene < selenophene < tellurophene < furan, a trend also observed in the symmetric stretching frequency of the double bonds implying greater localization of the double bond and thus a decrease in aromaticity. Thus thiophene is shown to be more aromatic than furan. Similar comparisons with the C—C bond in benzene show these heterocycles to be less aromatic than benzene.

If ring substitution should occur via a loose vibration, i.e., a C—H bond with a low force, it might be possible to predict the order of reactivity. However, for these heterocycles, the C—H force constants and mean amplitudes of vibration are approximately equal, making it difficult to compare reactivity. On the other hand, the potential energy distribution shows a vibrational coupling between the C_α —H and C_β —H, the degree of coupling being furan < selenophene ~ tellurophene < thiophene, which is the opposite of the reactivity order in electrophilic substitution <73JCS(P2)2097>. Thus, it is possible to determine the order of aromaticity for a variety of heterocycles from the calculated force constants and mean amplitudes of vibration.

An alternate approach to aromaticity involves a relationship between bond order and ring current applicable to monocyclic, polycyclic, nonplanar, and nonbenzenoid rings <83JOC1344>. The semiempirical MO method SINDO1 is used to perform the calculations. Aromatic compounds tend to reduce the alternation in bond length between a single and double bond as would be otherwise found in a classical, nonresonating structure. This is complicated when a heteroatom is introduced in the molecule since it is not easy to determine how much bond alternation has been reduced. By utilizing only bond orders, one can overcome this problem. Since the magnitude of the ring current is determined by the weakest link in the ring, the bond with the lowest bond order is the weakest link. Since these are not π -electron bond orders, this method is not limited to planar systems. The advantage here is that the results do not depend on parametrization, nor is there a need for comparison with a fictitious model.

The concept of bond orders for aromaticity has also been exploited by Bird <85T1409, 86T89, 87T4725> and is based on a statistical evaluation of the deviations in peripheral bond orders, which are readily obtainable from bond lengths. Thus aromaticity indices for a variety of five-membered (I_5), six-membered (I_6) and benzo-fused five-membered ($I_{6,5}$), and six-membered ($I_{6,6}$) systems have been determined using the relationship (Equation (4)).

$$\text{Aromaticity index } (I_v) = 100 \left(1 - \frac{V}{V_K} \right)$$

$$\text{where coefficient of variation } V = \frac{100}{\bar{N}} \sqrt{\frac{\sum (N - \bar{N})^2}{n}} \quad (4)$$

$\bar{N}$ = arithmetic mean of the various bond orders (N), and
 n = number of bonds.

The nondelocalized Kekulé form V_K depends on the ring system. Thus $V_K = 35$ for five-membered and for fused five- and six-membered rings, and 33.3 for six-membered rings. Since benzene is a fully delocalized system, V has the value zero and I_6 for benzene is 100. Similarly, the aromaticity index (I_5) for thiophene is 66 while those for pyrrole and furan are 59 and 43, respectively. Quantitatively, these values agree with that predicted from their chemical behavior.

Although the indices are capable of comparing the degree of aromaticities, they are sensitive to ring type. Thus it is not possible to compare the aromaticity of a five-membered vs a six-membered or a benzo-fused heterocycle. Subsequent work by Katritzky *et al.* <89JA7> established good correlation between the aromaticity indices and resonance energies. This relationship is used by Bird to define a unified aromaticity index which compares the Hückel molecular orbital delocalization energies of the parent five-, six-membered and benzo-fused carbocycles <92T335>. Benzene is still the standard and has an I_6 value of 100. The other ring sizes follow the relationship $I_A = I_6 = 1.235 \times I_5 = 1.34 \times I_{6,6} = 2.085 \times I_{5,6}$ (see Table 18). There is an excellent relationship between this index and the aromaticity parameter which is determined by the dilution shift method <74JCS(P2)332>.

It is also possible to determine the relationship between I_A and resonance energy. Resonance energies have been calculated by employing a Laidler bond energy scheme <B-70MI 209-02>. Bond energies used for C—C were $-4.62 \text{ kcal mol}^{-1}$, for C=C, $37.90 \text{ kcal mol}^{-1}$, and for C—S, $5.29 \text{ kcal mol}^{-1}$. The resonance energy of thiophene is then 43.0 kcal (Table 18), while that for benzo[*b*]thiophene is calculated to be 69.8 kcal <92T335>. By comparison, the values for benzene and naphthalene are 45.8 and 80.3 kcal , respectively. The order of aromaticity is found to be benzene > thiophene > pyrrole > furan which is in agreement with many of the other systems. The

resonance energies calculated by this method are related to the unified aromaticity index by the equation $RE = 0.531 \times I_A$ making it possible to rank the aromaticity of any heterocycle as long as the ring bond lengths are available for estimation of their resonance energies.

π -Electron contributions to the diamagnetic susceptibility (χ^1) have also been used as an index to measure aromaticity <87CHE454>. These are calculated relative to benzene and depend on the measure of the induced ring current. Thus it is possible to construct a scale of aromatic character for five-, six-membered, and fused heterocycles. Based on this method, the diamagnetic susceptibility of thiophene (relative to benzene) is found to be 0.664.

The aromaticity of thiophene has also been measured by NMR ring currents. However, various results have been obtained by different workers. Thus the aromaticity determined by Elvidge <65CC160> is calculated to be 75%, while that by DeJongh and Wynberg <65T515> was estimated to be 77%. On the other hand, calculations by Abraham *et al.* <65CC43> gave a value of 100%; a similar value was also obtained for furan. Obviously this is not an accurate concept for aromaticity. The main problem arises in obtaining the appropriate model compounds and making the right corrections. Another spectroscopic based aromaticity concept is the difference in the chemical shifts of the α - and β -protons <65SA85>. The smaller the difference, the greater the aromaticity. According to this criteria, the order for aromaticity is benzene > thiophene > pyrrole > furan.

The observation that the shifts of nuclear protons due to the introduction of a methyl group depends on the aromaticity of a ring has led to the development of another aromaticity index (B) <74JCS(P2)332>. A quantitative relationship of this index is shown in Equation (5). Interestingly, while some aromaticity parameters determine pyrrole to be slightly more aromatic than thiophene, this method shows pyrrole to be even less aromatic than furan.

$$B = 1 / \sum_{ij} |(\Delta\delta_2)_i - (\Delta\delta_2)_j| \quad (5)$$

In Equation (5) $\Delta\delta_2$ is the difference in the ring proton chemical shifts of the 2-methyl derivative and of the parent heterocycle, and i and j refer to the nonequivalent protons (3-*H*, 4-*H*, and 5-*H*).

The dilution shift parameter ($\Delta\delta_1 V_m$) is another spectroscopic parameter used to determine aromaticity, where $\Delta\delta_1$ is the difference between the chemical shifts of aromatic protons in a pure liquid and in an infinitely dilute solution in a nonpolar solvent, and V_m the molar volume of the compound <74JCS(P2)332>. Based on this concept, the aromaticity of a compound is defined as $A = \Delta\delta_1 V_m^{2/3}$ <72BSF1022>. The values correlate well with the Pauling resonance energies <33JCP(1)606> and enables estimates of resonance energies of heterocycles which could not have otherwise been obtained.

The solvent shift parameter (S) is the difference in the chemical shift of the protons of a mixture of acetonitrile and cyclohexane in cyclohexane as the solvent and in the aromatic compound as the solvent <71JA556>. The premise behind this concept is that the positive end of the acetonitrile dipole locates on top of the aromatic ring where the π -electron density and diamagnetic shielding is high. The chemical shifts thus depend on the degree of aromaticity; the greater the aromaticity, the greater the anisotropy effect and the larger the S value. A scale relative to benzene is thus created. While most normal alkenes are zero, benzene has a value of 1.0 and thiophene is calculated to be 0.745.

The diamagnetic molar susceptibility (χ_m) also depends on ring current and can be measured relatively easily. The difference between this value and that obtained for a classical localized model affords the diamagnetic susceptibility exaltation (Λ) <B-71M1 209-04>. A comparison of the aromaticity indices of thiophene and benzene by magnetic criteria are displayed in Table 19. In all cases, the order of aromaticity as determined by these magnetic criteria follow the sequence benzene > thiophene > pyrrole > furan.

Carbon-13 NMR studies have also been used as a quantitative measure of electron deficiency and excessiveness <82OMR(19)192>. The difference in the chemical shifts observed between a given unsubstituted carbon atom and its substituted counterpart, varies significantly for different aromatic compounds. The presence of an electron-withdrawing substituent X will cause a larger chemical shift at the substituted carbon if the ring is electron rich. If the ring is electron deficient, the extent of deshielding will be less since there is a counter effect here. Based on this, the ratio $^{13}\rho$ is defined by Equation (6).

$$^{13}\rho = \left[\frac{\delta(\text{ppm})(\text{het} - X) - \delta(\text{ppm})(\text{het} - H)}{\delta(\text{ppm})(\text{benzene} - X) - \delta(\text{ppm})(\text{benzene} - H)} \right] \quad (6)$$

For π -deficient systems, this ratio is < 1, while for π -excessive, it is > 1. As a result it is possible

Table 19 Aromaticity indices of benzene and thiophene based on magnetic criteria.

Method	Benzene	Thiophene	Ref.
χ^1	1	0.664	87CHE454
$\delta_a - \delta_b$	0	0.19	65SA85
Dilution shifts	13.93	11.56	74JCS(P2)332
Solvent shifts	1	0.745	71JA556
B	5.0	3.85	74JCS(P2)332
Λ^a	13.7	13.0	B-71MI 209-04

^aIn units of $-4\pi 10^{-12} \text{ m}^3 \text{ mol}^{-1}$.

to obtain a quantitative measure of the π -excessiveness or deficiency and thus the degree of aromaticity. Based on this, the aromaticity indices for 2- and 3-substituted thiophenes were calculated to be 1.35 as compared to 1.14 and 1.31 for the corresponding furan derivatives, indicating thiophene to be more aromatic than furan.

The absolute hardness of a chemical species has been shown to be a good measure of aromaticity <88TL4843>. As a first approximation, it can be assumed to be equal to half the difference between the ionization potential and its electron affinity <83JA7512>. Alternatively, if the Hartree-Fock or the Hückel model is used, the absolute hardness can also be represented as half the HOMO-LUMO energy gap <86PNA(83)8440>.

Zhou and Parr have introduced the concept of relative hardness (η_r) and topological resonance energy per π electron (TREPE) <89JA7371>. Within the Hückel theory, η_r is the difference between the absolute hardness of the molecule and the absolute hardness of its hypothetical acyclic analog. Consequently, the absolute and the relative hardness have been obtained for a variety of benzenoid and heteroconjugated hydrocarbons and compared with either the resonance energy, the resonance energy per π electron (REPE), or the TREPE. In almost all cases, there is good correlation between the hardness and energies. According to the hardness concept, thiophene is shown to be aromatic and follows the trend observed by TREPE and REPE.

Katritzky *et al.* <89JA7> have developed a principal component analysis (PCA) system to determine aromaticity. Various characteristics consisting of geometrical, energetic, and magnetic data for a series of compounds are assembled and subjected to PCA. Values for the characteristics of individual compounds recalculated from the scores were found to exhibit good agreement with those used in the treatment. Scores can then be estimated to predict values of characteristics for other compounds where there is limited data available.

The PCA method transforms a given set of principal components that are uncorrelated to each other. The principal component model is described by Equation (7).

$$x_{ik} = \bar{x}_{ik} + \sum_{(a=1)}^A t_{ia} p_{ak} + e_{ik} \quad (7)$$

where

 $\bar{x}_{ik}$ = mean scaled value of the experimental quantities t_{ia} = scores i = chemical compound under study p_{ak} = loadings k = variable (experimental measure) e_{ik} = residuals a = principal component

The geometrical properties selected are the Bird's aromaticity indices which are calculated from experimental bond lengths (I_x) <85T1409, 86T89> and also from AM1 calculated ring geometries (I'_x) <90JPR(332)853>, the Julg index which is based on the concept of ring current <83JOC1344> and the Pozharskii index (ΔN) <85CHE717>. Energetical properties considered are the DRE <70JA1453>, HSRE <73JA3907, 75T295>, the difference in the experimental heats of aromatization (ΔH_A) <69JA6321>, the experimental heats of formation (ΔH_f) <83JCC84>, and the calculated values ($\Delta H_{f(\text{MNDO})}$) <83MI 209-01> or ($\Delta H_{f(\text{AM1})}$) <90JPR(332)853>. Magnetic criteria include the diamagnetic susceptibility exaltation (Λ) <74AHC(17)255>, and an aromaticity index I_1 based on the anisotropic polarizability of the molecule <84JGU1427>. For heterocycles containing a nitrogen atom, the ^{15}N chemical shifts were also included.

From the PCA model, a SIMCA modeling and classification method is applied <B-86MI 209-04>. The partial least squares method (PLS) is then used to predict the values of each of the variables

used in the PC analysis and thus assess the self-consistency of the model. To confirm the predictive power or reliability of a characteristic, a data point x_{ik} is successively omitted and a PC analysis performed with the remaining set of data. The new value for the data omitted is recalculated using the new scores and new values of the loadings. The closer the recalculated value to the input value, the greater the reliability and the predictive power of the model. Principal component scores t_1 , t_2 , and t_3 are calculated for each compound as are the residual standard deviations. By plotting a three-dimensional graph of the scores t_1 , t_2 , and t_3 it is possible to separate the compounds into groups. From the first principal component scores t_1 , it is possible to distinguish five-membered rings from six-membered and also oxygen containing heterocycles from those containing sulfur and nitrogen. The second principal component scores t_2 , depend mainly on the number of heteroatoms in the molecule irrespective of whether they are five- or six-membered. The t_3 scores depend on the heteroatom present in five-membered rings.

For benzene, the t_1 , t_2 , and t_3 scores are all positive. Heterocycles with two or more nitrogens are found in the region with t_1 positive and t_2 negative. Pyrrole, furan, and thiophene all show t_1 as negative and t_2 as positive. For those containing no oxygen and more than one heteroatom, t_3 is negative. Oxygen containing heterocycles, e.g., furan, oxazole and isooxazole all show a positive score for t_3 .

For thiophene, the principal component scores t_1 , t_2 , and t_3 as obtained from the principal component analysis model and with nine characteristics are -1.643 , 0.731 , and -2.433 , respectively <89JA7>. With 11 characteristics, the values change to -0.918 , 2.444 , and -1.737 , respectively <90JPR(332)853>. Although the values differ, the scores of the compound obtained in both papers show a good correlation implying a reliable quantitative concept. The multiple correlation coefficient is a good measure as to the reliability of the parameters. The closer the recalculated values are to the input values, the more reliable the model and the characteristic employed. Most of the characteristics displayed high correlation coefficients (>0.8). However, for the five-membered heterocycles, the $\Delta H_{f(AM1)}$ are less well predicted by this treatment since the heats of formation are overestimated by the AM1 method.

The principal component analysis model has also been employed for the comparison of aromaticity for benzo-fused five- and six-membered heterocycles <90JPR(332)870>. The loadings for the bicyclic compounds show a close correlation to those of the corresponding monocyclic heterocycles. The data for the characteristics and the scores for thiophene and for benzo[b]thiophene (along with benzene and naphthalene) are listed in Table 20.

Table 20 Characteristic data and scores for thiophene,^a benzene,^a benzo[b]thiophene,^b and naphthalene.^b

Property	Thiophene	Benzene	Benzo[b]thiophene	Naphthalene
Bird's aromaticity index (I_a) ^c	66.0	100.0	57.0	77.3
Bird's aromaticity index (I'_a) ^d	67.7	100.0	76.1	76.9
Jug's aromaticity index (RC)	1.53	1.77	1.52	1.65
Pozharskii's index (ΔN)	0.25	0	0.18	0.18
Dewar resonance energy (DRE) ^e	1.08	3.77	2.48	3.36
Dewar resonance energy (DRE) ^f	0.96	3.84	1.18	3.35
Hess-Schaad resonance energy (HSRE) ^g	0.032	0.065	0.044	0.055
Heat of formation (ΔH_f) ^h	27.6	19.8	40.0	36.1
Heat of formation ($\Delta H_{f(AM1)}$) ⁱ	40.1	22.0	53.9	40.8
Molar diamagnetic susceptibility (χ_m) ^j	57.4	54.8	93.3	91.9
Diamagnetic susceptibility exaltation (Λ) ^j	17.8	17.9	34.9	36.2
Score t_1	-0.92	3.20	1.30	3.57
Score t_2	2.44	3.20	-2.04	-1.12
Score t_3	-1.74	1.18	0.39	8.80

^a<90JPR(332)853>. ^b<90JPR(332)870>. ^cFrom experimental bond lengths. ^dFrom AM1 ring geometries. ^eFrom experimental heats of formation (kcal mol⁻¹). ^fFrom AM1 heats of formation (kcal mol⁻¹). ^gIn units of β . ^hkcal mol⁻¹. ⁱAM1 heats of formation. ^j($\times 10^{-6}$).

From Table 20, we see a change in sign for the t_2 score of naphthalene as compared to benzene. This arises due to a change in sign of the loading value p_2 which is allowed in a principal component analysis system where the relevant information is associated with the magnitude rather than the absolute value. Replacement of a CH=CH in naphthalene by sulfur reduces t_1 by a reasonable amount and even more so when it is replaced by nitrogen or oxygen. The magnitude of t_1 increases when a benzo ring is attached to thiophene but t_2 generally becomes smaller. Although this latter

effect is small in going from thiophene to benzothiophene, it is much more pronounced in others such as benzene, pyrazole, and triazole.

Most of the characteristics are predicted quite well. In general, the multiple correlation coefficient of the values recalculated by the PLS method for five- and six-membered heterocycles are much higher than for the benzo-fused analogues. This indicates smaller deviations in the recalculated values for the former cases. For thiophene, the largest deviations (using the PLS method) were observed in the recalculated values of HSRE, and the diamagnetic susceptibility exaltation. For benzo[*b*]thiophene, there is negligible deviation for the recalculated value of I'_x , but a large deviation for the I_x recalculated value. The other geometric characteristics are predicted quite well. Amongst the energy characteristics, the recalculated DRE and HSRE values show a good correlation coefficient but a much larger deviation for the AM1 calculated energy (DRE'). The recalculated values of χ_m for benzothiophene and for other sulfur containing bicyclics are lower, while oxygen containing compounds have higher recalculated values.

Although the data for the two series of compounds $\langle 90JPR(332)853, 90JPR(332)870 \rangle$ are completely different, there is a significant relationship between the loadings and the scores for both series which indicates good reliability for this aromaticity concept.

Energy-based criteria for aromaticity have, in general, not correlated well with experimentally obtained data and that from chemical reactivity. This lack of correlation is especially pronounced in heteroaromatic compounds. Kollmar $\langle 79JA4832 \rangle$ employed a "reference" molecule, localized the π -orbitals and calculated the energy of the optimized molecule with localized double bonds. Although satisfactory results were obtained for benzene and naphthalene, the method is not so reliable for heterocycles $\langle 90IJQ843 \rangle$. This is circumvented by localizing only the lowest π -orbital rather than the entire π -bonding system $\langle 86ZN(A)49 \rangle$. The aromatic criteria of five-membered heterocycles obtained by this modification correlates well with chemical reactivity, thermodynamic data and the Bird's aromaticity index. This has been further modified by localizing only the lowest π -orbital and subtracting the eigenvalues calculated for the lowest delocalized π molecular orbital $\langle 90IJQ843 \rangle$. $\Delta\epsilon_{\pi L}$ (NLMO) is thus defined as: $\Delta\epsilon_{\pi L}$ (NLMO) = ϵ_{π} (MO) - ϵ_{π} (NLMO) where ϵ_{π} (MO) = eigenvalues of the lowest π delocalized MO, and ϵ_{π} (NLMO) = eigenvalues of the lowest π natural localized MO.

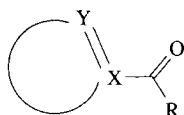
The localization procedure developed by Boys $\langle 60MJ 209-01 \rangle$ was also used and $\Delta\epsilon_{\pi L}$ (Boys) was calculated as above. Values calculated by both methods correlate well with experimental aromaticity criteria although the Boys method provides better correlation. The order of aromaticity as shown by these calculations is thiophene > pyrrole > furan which is not always the case by some of the other methods.

2.09.4.2 Conformational Analysis

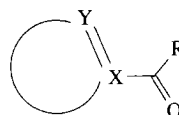
Organic carbonyl compounds in which the carbonyl group is not part of a cyclic structure possess interesting conformational properties depending on the system. For example, the carbonyl group $-\text{C}(\text{O})\text{R}$ attached to an atom X in a ring, gives rise to two scenarios:

(i) if X is in an sp^2 -hybridization state as in an aromatic or heterocyclic system (75), it can exist as either the Y,O-*cis* (75a) or Y,O-*trans* (75b) conformer in the ground state. The transition state would be where the C=O bond is perpendicular to the plane of the ring;

(ii) if X is an sp^3 -hybridized atom, a threefold barrier exists which depends on the ring conformation.

Y,O-*cis*

(75a)

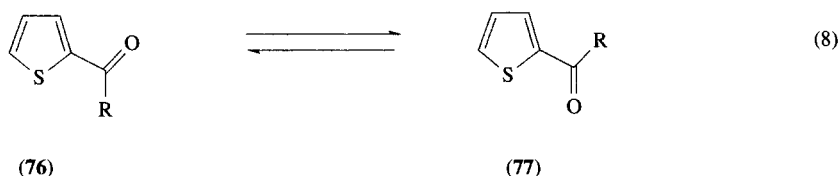
Y,O-*trans*

(75b)

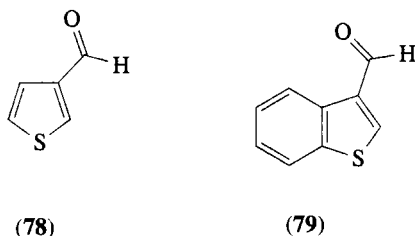
The rotational isomerism in carbonyl-containing compounds of five-membered heterocycles has been well reviewed $\langle 77JCS(P2)1601, 81RCR336, 84KGS579, 87AHC(41)75 \rangle$. The energy of rotation around two sp^3 -hybridized C—C bonds is about 2.9 kcal mol⁻¹, while that for two sp^2 -hybridized C=C

bonds (*cis-trans* isomerism) is about 5–7 kcal mol⁻¹. In carbonyl containing heterocyclic compounds, the extent of π -conjugation between the carbonyl group and the ring is the main factor in determining the energy of rotation. For thiophene derivatives, this could be of the order of 10.7–12 kcal mol⁻¹.

2-Formylthiophene or thiophene-2-carbaldehyde can exist either as the S,O-*trans* (76) or S,O-*cis* (77) conformer (Equation (8)). Initial results from microwave spectroscopy indicated the compound to exist as the *trans* isomer in the gas phase <65ZN(A)1323>. This was later found to be wrong and the *cis* conformer was shown to be the major isomer <76CA(84)58451>. The predominance of this conformation has been supported by experimental and calculated dipole moments <74JGU1291, 75JCS(P2)744>, from Kerr constants <75JCS(P2)744> and by other methods <87AHC(41)75> including theoretical calculations <71JGU374, 73T2545, 75JOU1968, 79JA311>. The energy barrier for the *trans-cis* isomerization was determined to be 10 kcal mol⁻¹ as determined by ultrasonic pulse techniques <69JCS(A)713>. It has been shown that the molecule is planar and that >90% of the molecule exists in the S,O-*cis* orientation <85JCS(P2)1839>. The same holds true for the corresponding benzo[b]thiophene derivative <83JCS(P2)911>. Compound S,O-*cis*-thiophene-2-carbaldehyde (77) is also more polar than the *trans* form <79JA311> and is thus even more preferred in polar solvents. With the exception of furan-2-carbaldehyde, the conformations are quite rigid for most of the other five-membered heterocycles.



CNDO/2 <74JOU848> and *ab initio* calculations <79JA311> show the S,O-*trans* form of 3-formyl derivatives (78) to be slightly more stable. NMR studies show a large preference for the S,O-*trans* form of benzo[b]thiophene-3-carbaldehyde (79).



Steric interactions exist between the formyl group and the adjacent hydrogen atom. For thiophene-3-carbaldehyde (78), there are two adjacent hydrogen atoms while for the 2-formyl derivatives there is only one. Thus in the ground state, the latter displays greater stabilization than the former <84JCS(P2)819>.

The presence of a substituent at the adjacent position tends to alter the conformation due to hydrogen bonding, steric and electrostatic effects. Thus a C(3) substituent in thiophene-2-carbaldehyde (77) increases the stability of the S,O-*cis* form. For the 3-formyl derivative, a —TeMe substituent at the C(2) or C(4) position leads to a predominance of the *cis* conformer <80JST(67)251>.

For 2,5-diformyl derivatives, the 2-*cis*,5-*cis* form predominates although 20–30% of the *cis,trans* form is also detected <72JCS(P2)755>. There is however less than 1% of the *trans,trans* conformer. For thiophene-2,3-dicarbaldehyde, the *cis,cis* form is preferred over the *trans,trans* by a factor of 2:1 <71TL145>.

Theoretical calculations <79JA311> show the *cis* form of thiophene-2-carbaldehyde (77) to be more stable by 0.71 kcal mol⁻¹, while for substituents other than carbonyl groups, e.g., 2-hydroxy- and 2-methyl-thiophene, the S,H-*trans* orientation is more stable by 2.2 and 0.88 kcal mol⁻¹, respectively. The opposite holds true for the 3-substituted derivatives where calculations show the *cis* form to be more stable. The *cis* conformer is preferred in both 2- and 3-vinylthiophene.

In ketones, the coplanarity between the ketone group —C(O)R and the heterocycle depends strongly on steric interactions. For 2-acetylthiophene, the S,O-*cis* form (77; R = Me) is preferred and the molecule is practically planar <81RCR336>. Low temperature ¹³C NMR confirms the predominance (>95%) of the S,O-*cis* form <85JCS(P2)1839>, while at room temperature, lanthanide-

induced shift (LIS) NMR shows it to be about 85% in the case of the methyl derivative, and 75% for the isopropyl case (**77**; R = Prⁱ). A similar result is also observed by LIS NMR studies for 2-acetylbenzo[*b*]thiophene <83JCS(P2)911>.

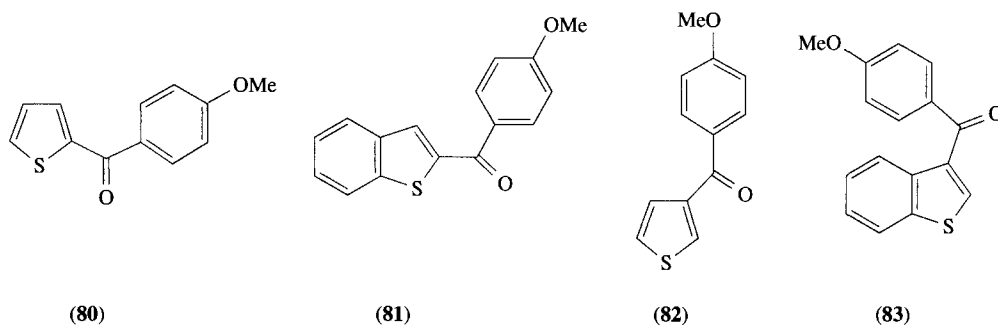
As in the 3-formyl derivatives, both conformers are observed in 3-acetylthiophene, the ratio of S,O-*trans* to S,O-*cis* being roughly 3 : 2 <76OMR(8)525>. Furthermore, the presence of an alkyl group reduces the energy barrier for *cis-trans* isomerization for 2- and 3-keto substituents as compared to their formyl analogues <85JCS(P2)1839>.

For aroyl derivatives, the phenyl ring is too large to be planar with the heterocycle. Since the carbonyl group conjugates better with the heterocycle rather than with a phenyl group, it is the latter which is twisted out of the plane <78JCS(P2)1232>. However, it has been difficult to accurately verify the actual conformation by NMR, UV, and dipole moments. It is possible that both the carbonyl group as well as the phenyl ring are twisted to a certain degree, although the angle in the first case is practically zero. For thiophene, the S,O-*cis* orientation is preferred and the phenyl ring is twisted by 55–67°, which increases to about 90° when *ortho* substituents are present on the phenyl ring <81JMC865, 81RCR336>. In 3-benzoylthiophene, equilibrium mixtures of the S,O-*cis* and S,O-*trans* forms exist. As before, the phenyl ring is twisted to a large degree.

Theoretical calculations of the total molecular energy show that both rings in 2-benzoylthiophene are twisted relative to the carbonyl plane. They also confirm experimental data which shows that the phenyl ring is twisted to a larger extent and that the S,O-*cis* form is more stable. *Ab initio* calculated charge densities on sulfur (+0.273) and on oxygen (−0.223) indicated the possibility of an interaction between the two atoms and thus a preference for the *cis* conformer. For 3-benzoylthiophene, the two rings are rotated almost equally.

Conformational studies were carried out on the 2- and 3-*p*-anisoyl derivatives of thiophene and benzo[*b*]thiophene by x-ray and LIS NMR analysis <87JCS(P2)1851>. The 4-methoxy substituent was selected since a substituent at the 4-position is not expected to significantly alter the conformational preference of the two rings but it helps provide simple spectra.

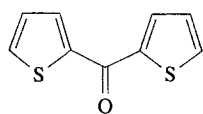
X-ray studies show the 2-aroyl derivatives (**80**) and (**81**) to favor the S,O-*cis* orientation and the 3-aroyl compounds (**82**) and (**83**) to adopt the S,O-*trans* form. In (**82**) and (**83**), the C(2)—C(3)—C(O) angle is approximately equal to the C(4)—C(3)—C(O) angle while for the 2-aroyl case (**80**) and (**81**) the S—C(2)—C(O) angle is much smaller than the C(3)—C(2)—C(O) angle. In general, the heterocyclic moiety has more coplanar character for the 2-substituted compounds than the 3-substituted compounds. For the 2-aroyl case, the S···O distance is smaller than the sum of the van der Waals radii, probably due to interactions between the two atoms. Packing energy calculations show that for (**80**)–(**82**), the nonbonded energy is increased by rotating the heterocycle and reaches a maximum after 80° and 260° rotations and a minimum at 180°. For (**83**) the values are high over the region 10–350°.



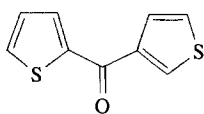
NMR studies indicate a low field shift (ca. 0.6 ppm) on 3-*H* for (**80**) and (**81**) and on 2-*H* for (**82**) and (**83**) which indicated preferred S,O-*cis* and S,O-*trans* conformations, respectively for the two classes. The ¹H NMR spectrum of (**80**) and (**81**) in CDCl₃ shows the presence of both conformers with the S,O-*cis* form being preferred. For (**82**) the S,O-*trans* form is slightly preferred but in (**83**), only the S,O-*trans* form is observed.

In bithienyl ketone the two heterocycles are twisted in a conrotatory fashion from each other by about 40° with respect to the carbonyl group <72CR(C)1112> with the S,O-*cis* conformer prevailing for both rings. X-ray analysis of 2,2'-(**84**), 2,3'-(**85**), and 3,3'-(**86**) bithienyl ketone show (**85**) and (**86**) to be disordered. In all cases, the preferred conformation between the carbonyl group and the thiophene moiety is the same as that observed for the formyl and acetyl derivatives <89JCS(P2)1741>. *Ab initio* calculations at the 3-21G* level show that for (**84**), the S,O-*cis*,S,O-*cis* orientation is

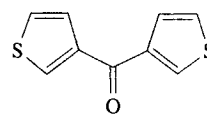
found in greater than 70%; in good agreement with that observed from LIS NMR measurements. Furthermore, the twist of the rings obtained from both methods is also in close agreement.



(84)



(85)



(86)

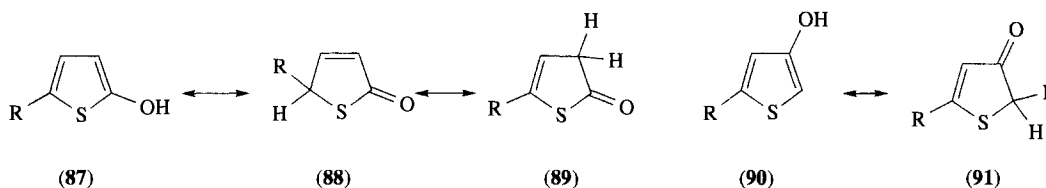
Theoretical calculations and LIS NMR measurements on (85) indicate the S,O-*cis* orientation for the 2-thienyl fragment while the 3-thienyl ring adopts both forms. In the solid state, the 3-S,O-*trans* form is observed with both rings being twisted from the carbonyl plane. In (86), the *trans* conformer is predicted to be more stable, in agreement with LIS NMR results.

2.09.4.3 Tautomerism

The presence of a hydroxy-, thio-, or amino-substituent in five-membered heterocycles, leads to various tautomeric forms. In most cases, one of the tautomeric forms is not aromatic and the degree to which the tautomeric form exists depends on the loss in resonance energy. Thus, compounds which are less aromatic would be more prone to tautomerize than the more aromatic ones.

2.09.4.3.1 Compounds with a hydroxy group

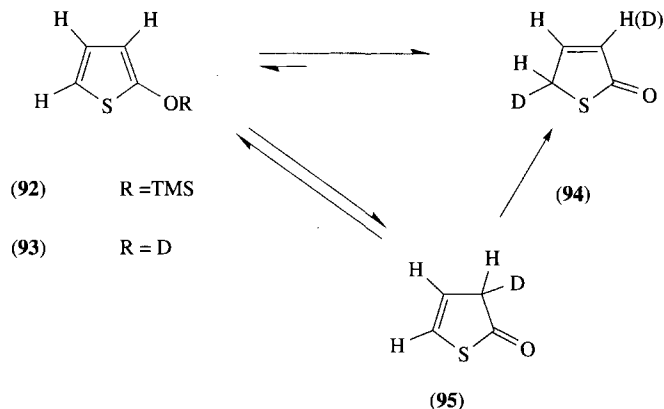
Hydroxythiophenes are very unstable, air- and acid-sensitive compounds (B-76MI 209-01). 2-Hydroxythiophenes are generally found in the tautomeric forms (87)–(89), while the 3-hydroxy isomer can exist in either the hydroxy form (90) or the keto form (91). Depending on the nature of the substituents and the solvent system, either of the tautomeric forms (90) or (91) can be the predominant one. Although some of these systems could exist as mixtures or exclusively in the keto form, we shall generically refer to them as hydroxythiophenes for simplicity.



(i) 2-Hydroxythiophenes

For the 2-hydroxythiophenes, the equilibrium is almost completely in the keto form. Furthermore, the conjugated isomer 2(5*H*)-thiophenone (88) is generally more stable and formed to a larger extent than (89). The greater thermodynamic stability of (88) is due to the conjugation between the keto group and the double bond, while in (89), the stability exists only by hyperconjugation between the double bond and the substituent (if any) in the 5-position. In the parent molecule where there is no substituent, only (88) is detected.

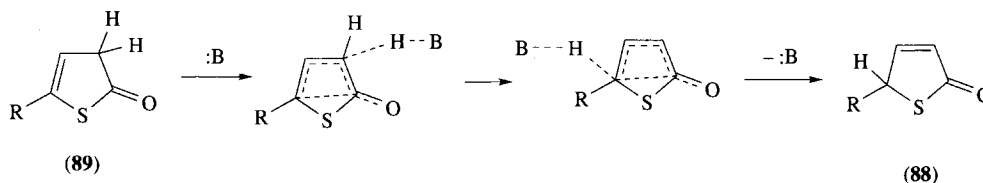
Hydrolysis of 2-[(trimethylsilyl)oxy]thiophene (**92**) in $\text{DMSO-}d_6/\text{D}_2\text{O}$ to (**93**) occurs readily at 32°C $\langle 89\text{JA}5346 \rangle$. However, it is unstable and isomerizes rapidly (ca. 10 min) to a 2:1 mixture of the 5-deuterio (**94**) and the 3-deuterio (**95**) keto derivatives (Scheme 6). The latter is unstable and converts to 5-deuterio-3-thiolen-2-one (**94**) within an hour.



Scheme 6

In general, the 2(*5H*)-thiophenones (**88**) have higher dipole moments than the 2(*3H*)-thiophenones (**89**). Thus the equilibrium shifts to the former with increasing polarity of the solvent $\langle 68\text{AK}461 \rangle$. With a phenyl group at the 5-position, only 5-phenyl-2(*5H*)-thiophenone (**88**; R = Ph) is formed as evidenced by the NMR spectrum in CCl_4 $\langle 64\text{AK}211 \rangle$. However, in methanol, the hydroxy compound (**87**; R = Ph) has also been detected in amounts of 25–30%. For substituents at the 3- and 4-position, the 2(*5H*)-thiophenone form predominates since, in addition to the keto group, the substituent can also conjugate with the double bond $\langle 62\text{ACS}789 \rangle$.

The base-catalyzed rearrangement of 5-alkyl-2(*3H*)-thiophenone (**89**) to the corresponding 2(*5H*)-form (**88**) is first order in substrate and also in base. The tautomerism proceeds through a two-stage mechanism depicted in Scheme 7. For R = Me, tautomer (**88**) is more stable than (**89**) by 2.8 kcal mol^{-1} with an activation energy barrier of 21.5 kcal mol^{-1} $\langle 68\text{MI} 209-02, 86\text{HC}(44/3)1 \rangle$. Evidence for the two-step mechanism has been verified by hydrogen–deuterium exchange studies at the 3-position $\langle 68\text{AK}247 \rangle$.



Scheme 7

In most cases, the keto form (**88**) and/or (**89**) predominates. Spectroscopic evidence of the hydroxy form has been detected when there is a carbonyl substituent in the adjacent position. This conformation is stabilized by intramolecular chelation $\langle 67\text{T}871 \rangle$.

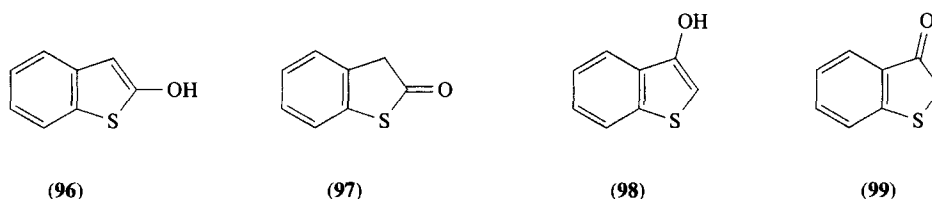
In the ^1H NMR spectrum of (**88**), the coupling constant between the hydrogen atoms at the 3- and 4-position are larger (ca. 6.1 Hz) than those found in thiophene $\langle 64\text{AK}211 \rangle$. Carbonyl substituents at the 3-position which generally cause the equilibrium to shift exclusively to the hydroxy form, show unusually large (> 6.0 Hz) $J_{4,5}$ coupling constants $\langle 67\text{T}871 \rangle$. Chemical shifts and coupling constants of a variety of hydroxythiophenes have been tabulated elsewhere $\langle 82\text{S}1056, 86\text{HC}(44/3)1 \rangle$ and will not be discussed here.

The carbonyl absorption in 2(*5H*)-thiophenones (**88**) is found in the region $1670\text{--}1695\text{ cm}^{-1}$, while that in the 2(*3H*)-thiophenones is found between $1730\text{--}1750\text{ cm}^{-1}$ $\langle 63\text{AK}239 \rangle$. The former derivatives also display a strong band between 805 cm^{-1} and 970 cm^{-1} while the nonconjugated keto forms (**89**) have a strong band between 970 cm^{-1} and 985 cm^{-1} . The appearance of a band in only one region is usually a good indication of the exclusiveness of that tautomeric form $\langle 64\text{AK}211 \rangle$.

The UV spectra of hydroxythiophenes has been summarized recently by Gronowitz and Hörnfeldt <86HC(44/31)>. In general, the 2(5*H*)-thiophenones display a band around 220 nm in the UV spectra which shifts to higher wavelength when substituents at the 3- and 4-position (including hydroxy groups) are introduced. The second band at 265 nm is generally unaffected by the introduction of substituents <63AK(21)239>.

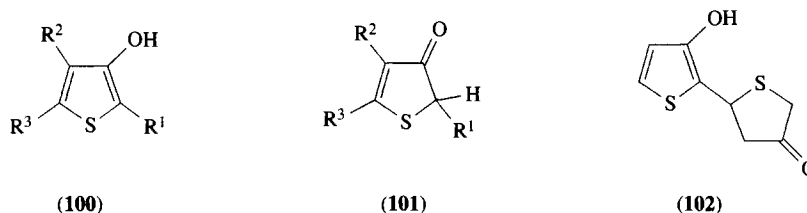
(ii) 2-Hydroxybenzo[*b*]thiophenes

In the solid form and in solution, only the keto form (**97**) exists <70AHC(11)296>. The hydroxy form (**96**) was detected by ¹H NMR spectroscopy when its trimethylsilyl precursor was hydrolyzed (CD₃COCD₃/D₂O/DCI). At -25°C, the signal at 6.45 ppm which is assigned to the alkenic proton at the 3-position disappears within 15 min and is replaced by a broad singlet at 4.2 ppm, indicating rapid conversion to the keto form (**97**) <89JA5346>.



(iii) 3-Hydroxythiophenes

Spectral evidence for 3-hydroxythiophenes show them to exist as mixtures of both forms (**100**) and (**101**) <56JCS4985, 57JCS4620>. Recent synthesis of 3-hydroxythiophene shows the keto form (**101**) to predominate by a factor of 2.9 <86TL3275, 89JA5346>. It is, however, unstable and tends to dimerize to the bithienyl (**102**) <90CC375>. 3-Hydroxythiophene was prepared from the trimethylsilyl derivative and was sufficiently stable to obtain its NMR in a variety of solvents <89JA5346>. In most cases, 100% of the enol form was detected. In CCl₄, both tautomers were observed, contrary to an earlier report where only the keto form was observed <86TL5155>. Although ketonization of 3-hydroxyfuran in water or DMSO affords over 99% of the keto form at equilibrium, substantial amounts of 3-hydroxythiophene can be detected at equilibrium <87PAC1577>.



The 2-methyl derivative is reasonably stable and exists mainly in the hydroxy form (**100**; R¹ = Me), while there is nearly a 1 : 1 ratio for the 2-*t*-butyl derivative <65ACS1249>. For 2,5-dialkyl substituted compounds the keto form (**101**) is generally favored over the hydroxy form by a ratio of 2 : 1 <76CS126>. In solution, the keto-enol equilibrium is established very rapidly, the equilibrium ratio varying from 1.04 to 8, depending on the solvent used. The high percentage of the hydroxy form in thiophene and benzothiophene in contrast to the predominance of the keto form in pyrrole and furan systems is attributed to the higher aromaticity of thiophene <74AHC(17)255>, and thus a larger energy barrier required to break the aromatic sextet <76CS126>.

Initial reports of carboxy substituents on 3-hydroxythiophenes indicated that the compounds all existed in the hydroxy form <65T3331, 67T871, 68CJC2255, 71T3839>, irrespective of the position of the substituent. However, reports have indicated the existence of tautomeric mixtures of both forms <70JCS2409>.

NMR data of substituted 3-hydroxythiophenes indicate that in cases where the hydroxy group is nonchelated (e.g., with alkyl substituents), the hydroxylic proton resonates between 5.6 and 6.7 ppm

<65ACS1249> while intramolecularly chelated hydroxylic protons are observed at about 9.5 ppm <65T3331>. The NMR spectra of 2-cyano- (**100**; $R^1 = \text{CN}$) and 2-phenyl-3-hydroxythiophene (**100**; $R^1 = \text{Ph}$) show the hydroxylic protons at 8.8 and 5.5 ppm, respectively, indicating the former is intramolecularly chelated <71T3853>. The strong absorption between 3130 cm^{-1} and 3180 cm^{-1} in the IR spectrum, which does not change with concentration, further indicates the presence of chelation in the cyano derivative.

In the IR spectra of 3-hydroxy derivatives, in addition to the carbonyl band between $1655\text{--}1680\text{ cm}^{-1}$, an O—H stretching band (not observed in the case of the 2-hydroxy derivatives) is also observed between 3300 cm^{-1} and 3600 cm^{-1} <76CS126>.

(iv) 3-Hydroxybenzo[b]thiophenes

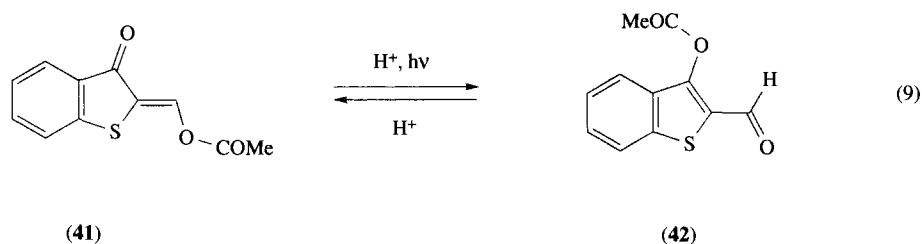
As in the case of 3-hydroxythiophenes, the benzo analogue is also unstable. There have been conflicting reports as to whether the hydroxy tautomer (**98**) or the keto form (**99**) is more stable. IR evidence indicates that in the solid state, it exists in the keto form <58JCS1217>. However, Buu-Hoi *et al.* <65BSF2658> report the ^1H NMR spectrum of the compound (in $\text{CDCl}_3/\text{CF}_3\text{CO}_2\text{H}$) which displays five aromatic protons and no aliphatic signals for the protons at the 2-position of the keto form. They thus assumed presence of only the enol form (**98**). Spectra run later in CCl_4 and CDCl_3 showed >95% of the keto form <74CS184>. This conflicting observation is attributed to the slow equilibrium of the two tautomers <69T2807, 84CHEC-1(4)34>. The half-life for the conversion of the enol form (**98**) to the keto form (**99**) is found to be 3 min at pH 2.3 and at 20°C .

Synthesis has indicated isolation of the keto form in the solid state <74CS(6)184, 87PAC1577, 89JA5346>. A strong carbonyl absorption is observed at 1690 cm^{-1} and the absence of a band between 3000 cm^{-1} and 4000 cm^{-1} indicates the absence of a hydroxy group. The ^1H NMR spectrum run in $\text{DMSO}-d_6$ also implies the exclusive existence of the keto form, but it is gradually converted to the enol form (**98**). The rate of conversion depends on the amount of water present in the solvent. In CDCl_3 , only the keto form is observed which remains unchanged over time. Hydrolysis of the trimethylsilyloxy derivative of 3-hydroxybenzo[b]thiophene generates the hydroxy compound (**98**) exclusively <86TL3275>. However, this is not stable and slowly tautomerizes to a mixture containing 40% of the keto form (**99**) <89JA5346>.

The effect of solvent on the equilibrium constants has been studied for the 3-hydroxy derivatives of five-membered heterocyclics and their benzo-fused derivatives <86TL3275, 89JA5346>. The enols are much more stable in hydrogen bonding solvents than in aprotic solvents. Thus while (**98**) is barely detected in C_6D_6 , CCl_4 , or CDCl_3 , it is predominant in $\text{C}_5\text{D}_5\text{N}$ and $\text{DMSO}-d_6$. All enols (including those of furan and pyrrole) show spontaneous or water-catalyzed ketonization. For acid-catalyzed ketonization, the 3-hydroxy monocyclic compounds tautomerize 10 to 10^3 times faster than their benzo analogs, presumably via a mesomeric effect due to the lone pair on the heteroatom. This is greatest in the case of the nitrogen heterocycles. During base-catalysis, ketonization of 3-hydroxybenzo[b]thiophene (**98**) and 3-hydroxybenzo[b]furan occur 100 times faster than in 3-hydroxyindole.

The rate of ketonization of the hydroxythiophenes and benzothiophenes in acetonitrile–water (9:1) is as follows: 2-hydroxybenzo[b]thiophene > 2,5-dihydroxythiophene > 2-hydroxythiophene > 3-hydroxybenzo[b]thiophene > 3-hydroxythiophene. 3-Hydroxythiophene does not ketonize readily in the above solvent system, but in 1:1 acetonitrile–water, it ketonizes 6.5 times slower than 2-hydroxythiophene <87PAC1577>.

The acetyl derivative of 3-hydroxybenzo[b]thiophene-2-carbaldehyde exists in the keto form 2-acetoxymethylenebenzo[b]thiophen-3(2*H*)-one (**41**) which is stable in hydrocarbon solvents up to 100°C <85JOU784>. In the presence of acids it undergoes rearrangement to the carbaldehyde (**42**) (Equation (9)). Equilibrium is reached within 2 h at 25°C . The IR spectrum of (**41**) displays two strong bands at 1685 and 1770 cm^{-1} due to the ring carbonyl and the ester group, respectively. At equilibrium, bands at 1660 cm^{-1} and 1765 cm^{-1} are detected which are assigned to the aldehyde and hydroxyacetyl groups in (**42**). In the ^1H NMR spectrum, the intensity of the methine proton at 8.85 ppm is reduced and a new signal at 9.95 ppm appears, which corresponds to the aldehyde proton in (**42**). Compound (**41**) exists in the *Z* form as shown by the ^1H NMR spectrum where the signal for the methine proton is downfield at 8.85 ppm which can only arise when the methine proton is in the deshielding zone of the carbonyl group.

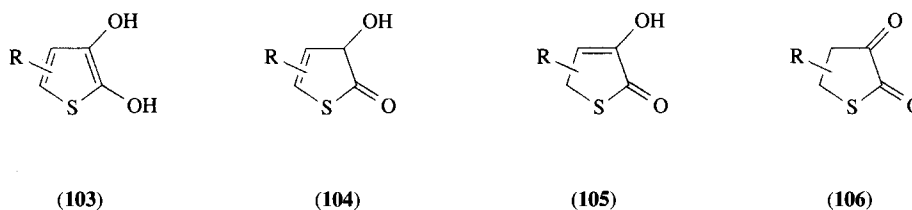


The structures of the two compounds were also confirmed by x-ray spectroscopy. For (41), a weak intramolecular hydrogen bond is observed between the methine proton and the oxygen atom of the acetyl group ($O \cdots H = 2.35 \text{ \AA}$) indicating the latter to be in the *S-trans* position relative to the exocyclic $C=C$ bond. The molecule is almost planar with the largest deviation being $1.324 \text{ \AA}$. In the rearranged molecule (42), the acetyl group is turned from the plane by an angle of 87.7° since this is the least sterically hindered conformation. The $O-COMe$ bond is stronger in (42) than in (41) as shown by the smaller bond length in (42). Since (42) exists in the *Z* conformation, it is not aligned correctly for the acylotropic rearrangement. It is possible however, that in acid medium, the first step is formation of an *H* complex or an ion pair which permits isomerization from the *Z* conformer to the *E* conformer which then undergoes acylotropic rearrangement. A similar type of behavior has also been reported for 2-*N*-acylaminomethylene derivatives <85JOU784>.

2.09.4.3.2 Compounds with more than one hydroxy group

(i) 2,3-Dihydroxythiophenes

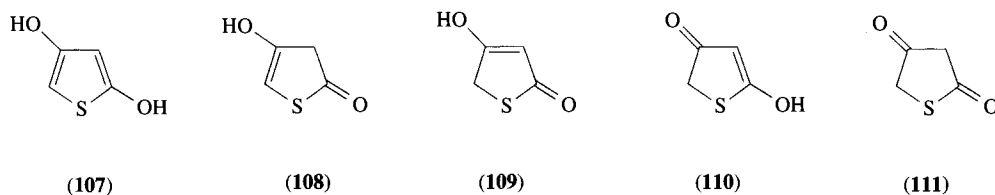
In general, 2,3-dihydroxythiophenes can possess four tautomeric forms (103)–(106). Spectroscopic evidence shows the hydroxyketo form (105) to predominate <71T3839>. The presence of a doublet at 3.87 ppm and integrating for two protons indicates that none of the other forms exist. The hydroxylic proton is observed at 6.40 ppm indicating no intramolecular chelation with the oxo group. In the IR spectra, peaks at 1680 cm^{-1} and 1645 cm^{-1} correspond to the $C=O$ and $C=C$ bonds, while the broad band at 3320 cm^{-1} implies a hydroxy stretching of an enol group.



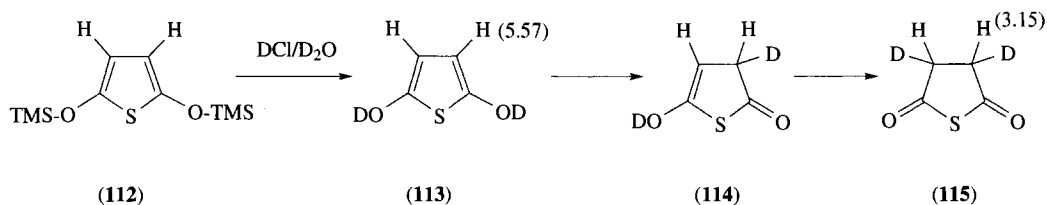
Initial reports of 2,3-dihydroxy-5-ethoxycarbonylthiophene (103; $R = 5-CO_2Et$) indicated it to exist in the tautomeric form (104) <65T3331>. Comparison of the IR spectrum with those of related compounds assigns the band at 1470 cm^{-1} to an unconjugated ester function implying that the correct structure is (105; $R = 5-CO_2Et$) <71T3839>. In most cases the 3-hydroxy-2-keto form observed parallels that in the furan series, the only exception being the 4-bromo compound which strangely enough exists as the diketo tautomer (106; $R = 4-Br$).

(ii) 2,4-Dihydroxythiophenes

2,4-Dihydroxythiophenes can exist in any of the five tautomeric forms (107)–(111). In most cases, the 4-hydroxy-2-keto form (109) predominates. The NMR spectra of these compounds usually display a doublet at 4.0 ppm and a triplet at 5.4 ppm with a long range coupling of $J = 0.9 \text{ Hz}$ <71T3839>.

*(iii) 2,5-Dihydroxythiophenes*

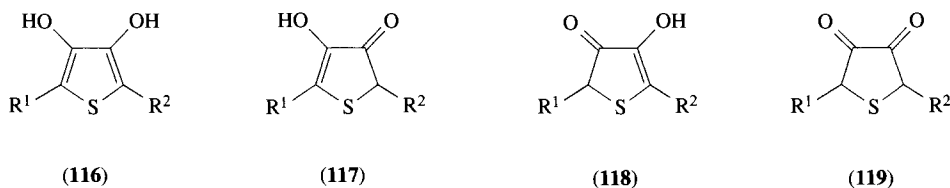
The 2,5-dihydroxythiophenes are only found in the dioxo form <63T1867, 68ACS1056, 71T3839>. By careful hydrolysis of 2,5-bis[(trimethylsilyl)oxy]thiophene (**112**), it is possible to generate the *O*-deuteriated-dihydroxy compound (**113**) as confirmed by the singlet at 5.57 ppm in the ^1H NMR spectrum of the compound <89JOC1211>. Disappearance of this signal with concomitant appearance of a signal at 3.15 ppm indicates formation of the diketo tautomer (**115**). This conversion is complete within an hour. During a similar hydrolysis <87PAC1577>, the mono- and dihydroxy intermediates were detected but not (**114**) indicating that the tautomerism from (**114**) to the diketo form (**115**) occurs rapidly (Scheme 8).



Scheme 8

(iv) 3,4-Dihydroxythiophenes

Mono- and disubstituted-3,4-dihydroxythiophenes can potentially exist in four tautomeric forms (**116**)–(**119**). For unsubstituted and for most disubstituted derivatives, the hydroxyketo form (**117**) or (**118**) is observed. In monosubstituted products ($\text{R}^1 \neq \text{H}$, $\text{R}^2 = \text{H}$), (**117**) is the only one observed <71T3839>. These compounds also follow a trend similar to that found in the furan series with the exception of the parent compound 3,4-dihydroxyfuran which exists in the dioxo form <60JA3219>. The only thiophene derivative not to follow the trend is the 2,5-diethoxycarbonyl substituted compound which exists in the dihydroxy form (**116**; $\text{R}^1, \text{R}^2 = \text{CO}_2\text{Et}$).

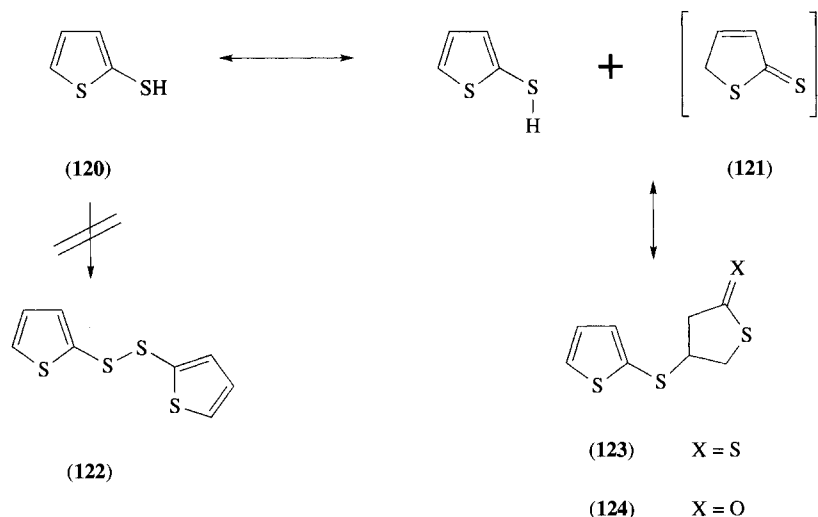


The ^1H NMR spectra of these compounds generally show the hydroxyl proton at about 5.9 ppm. The presence of a carboethoxy group in the molecule, shifts the hydroxyl proton downfield to about 9.0 ppm due to intramolecular hydrogen bonding. The IR spectra are similar to those of the 2,3-dihydroxythiophenes. The UV spectra of 3,4-dihydroxythiophene displays a shoulder at 335 nm <71T3839>. The introduction of a methyl substituent at the 2- and/or 5-position gives rise to two bands at 255 nm and 343 nm.

2.09.4.3.3 Compounds with a thiol group

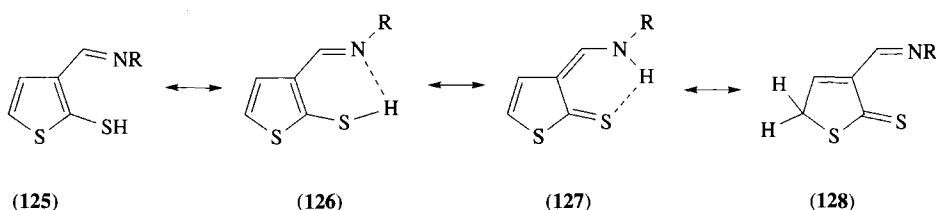
Unlike the hydroxy derivatives, no tautomerism tends to exist in the simple 2- and 3-thiophenethiols <77ACS(B)198>. Similarly, 3-thiolbenzo[*b*]thiophene exists in the 3-thiol form

<70JCS(C)2431>. 2-Thiophenethiol (**120**) is not stable and slowly solidifies on standing. Although one would expect the disulfide (**122**) to form due to air oxidation, it is the dimer (**123**) which is actually formed. It is possible that there is a slow tautomerism to the mercapto form (**121**) which is then susceptible to Michael addition by (**120**) to yield the dimer (**123**) which can be converted back to (**120**) <89JOC3223>. The possibility of (**121**) forming was corroborated by the fact that Michael addition of (**120**) with 2-hydroxythiophene (which exists exclusively in the keto form (**88**)), afforded the dimer (**124**) (Scheme 9).



Scheme 9

The presence of a proton acceptor site external to the ring such as an aldimine group at the 3-position, leads to the possibility of four tautomeric forms (**125**)–(**128**) (Scheme 10). The ^1H NMR and IR spectra of these compounds indicate that in the solid state, as well as in solution, the compound exists as (**127**). At higher temperatures, the equilibrium shifts to the left forming (**126**). The nature of the hydrogen bonding depends on the substituent at nitrogen. The chemical shift for the hydrogen atom is in the region 12–14 ppm <70CHE1232>. A similar behavior is also observed in the corresponding benzo[*b*]thiophene analogs <74JPR(316)970>.



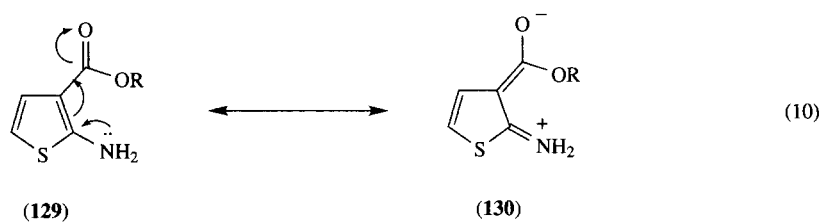
Scheme 10

2.09.4.3.4 Compounds with an amino group

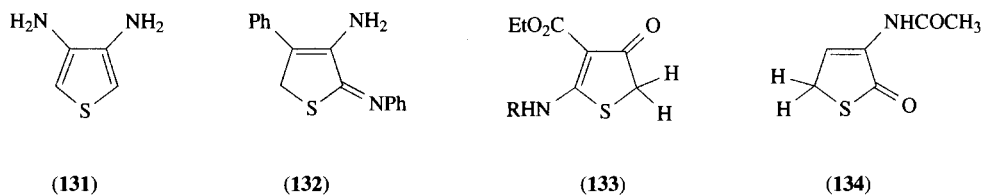
Tautomerism of amino-substituted systems in five-membered rings has previously been reviewed <63AHC(2)1, B-76MI 209-01>. However, literature since 1980 has been sparse with respect to tautomeric interest in these compounds. This is not surprising since as in the case for the thiol derivatives, 2- and 3-amino derivatives of thiophene and benzo[*b*]thiophene generally exist in the amino form <65JOC4074, 71T5873, 73JHC1067>. MO calculations on simple aminothiophenes show them to exist in the aromatic amino form rather than in the nonaromatic imino tautomeric form <81MI 209-01>. The 2- and 3-aminothiophenes are generally unstable in air. They are much more stable as salts or as their *N*-acyl derivatives and are also more stable in solution than in the neat liquid state.

Proton NMR studies on 2- and 3-thiophenamine <61AK515, 77S255, 82RTC205> as well as their substituted derivatives all indicate the exclusive existence of the imino form. The only case where

an imino form does exist is in the presence of an electron-withdrawing substituent in the *ortho* position (**129**). Thus, due to the push-pull effect, the imino form (**130**) can be generated (Equation (10)).



Amongst the diamines, the electron-rich 3,4-diaminothiophene (**131**) exists in the amino form <83BSF153>, while the 2,3-diamino derivative exists in the amino-imino form (**132**) <75ZC100>. In the hydroxyamino compounds, it is usually the hydroxy system that tautomerizes to the keto form rather than the amino function to the imino one (see compounds (**133**) and (**134**)) <78JOC3539, 81JCS(P1)1078>.



2.10

Thiophenes and their Benzo Derivatives: Reactivity

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2.10.1 INTRODUCTION

The reactivity of thiophenes and benzothiophenes had been dealt with at great length in the first edition of *Comprehensive Heterocyclic Chemistry* (CHEC-I) (84CHEC-I(4)741). Since then, a defini-

tive, five-part volume on the chemistry of thiophenes has been published, with Prof. Gronowitz as the editor, in the series *Chemistry of Heterocyclic Compounds*. This has naturally given a more detailed account of the various aspects of the reactivity of thiophenes and their derivatives. The present review therefore is concerned mainly with the progress in this field since the publication of the above two volumes, with appropriate cross-references to the earlier work. The organization of the present chapter follows the same general pattern as in CHEC-I, except for the deletion of those sections where no significant results have been reported in the interim period. Further, new sections have been introduced to deal with the progress achieved in the currently popular field of transition metal-mediated reactions.

2.10.2 REACTIVITY OF FULLY CONJUGATED RINGS

2.10.2.1 General Survey of Reactivity

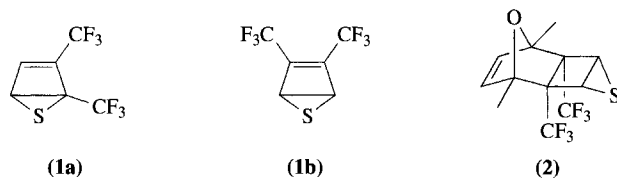
The dominant theme since the mid-1980s in the chemistry of thiophenes has undoubtedly been the application of transition metal catalysis. There are two distinct aspects of this work: the first is the use of Pd or Ni as catalysts for bond formation. The other is the study of transition metal-mediated ring opening of thiophene by cleavage of S—C bonds. The former is discussed in Section 2.10.4.7. In addition, some mechanistically interesting results have also been reported during this period. These include the preparation of the first fully characterized, stable pentacoordinated sulfur compound with four C—S bonds (Section 2.10.3.1) and the first clear evidence for an organosulfur ate complex (Section 2.10.2.6.4).

2.10.2.2 Unimolecular Thermal and Photochemical Reactions

The organization of this section is the same as that in CHEC-I, except for the addition of 3,4-dimethylenethiophene.

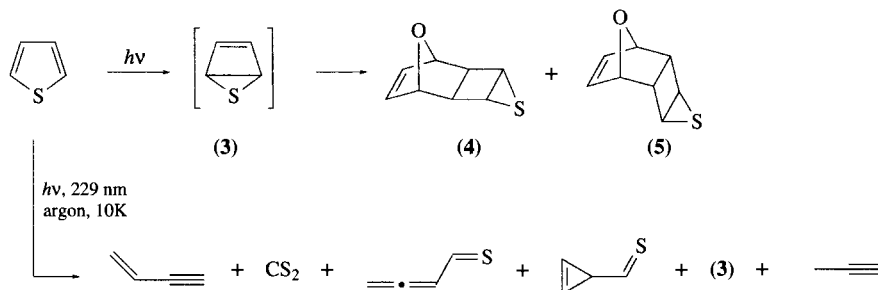
2.10.2.2.1 Phototranspositions

A review has covered the literature on Dewar heterocycles, including Dewar thiophenes up to 1981 (82AHC(31)169). The synthesis and properties of the stable Dewar thiophene with four trifluoromethyl groups had been described earlier. Subsequently, the photolytic transformation of bis(trifluoromethyl)thiophenes in the gas phase was investigated (84TL1917). The 2,3-disubstituted derivative, on photolysis, gives an equilibrium mixture of the two Dewar thiophenes (**1a**) and (**1b**) (8 : 1), along with the rearranged 2,5-, 2,4-, and 3,4-bis(trifluoromethyl)thiophene. Interestingly, the Dewar isomer (**1b**) seems to be more reactive than (**1a**) towards dienes; thus, on reaction with 2,5-dimethylfuran, the mixture of (**1a**) and (**1b**) gives exclusively the adduct (**2**) derived from (**1b**). Irradiation of 2,5-bis(trifluoromethyl)thiophene gives no Dewar isomers; only the rearranged 2,4-disubstituted isomer is formed, probably via a cyclopropenylthio ketone or a tricyclic isomer. Photolysis of 3,4-bis(trifluoromethyl)thiophene also does not lead to any Dewar thiophene; only a trace of the 2,4-disubstituted isomer could be detected.



The parent Dewar thiophene (**3**) has been generated and trapped (85JA723). When a solution of thiophene in furan (mole ratio, 1 : 10) was irradiated at 229 nm at 25°C, the two 1 : 1 adducts (**4**) and (**5**) were formed in a combined yield of 1.2% of thiophene consumed. The structure and stereochemistry of these two adducts have been elucidated by detailed NMR analysis. In order to prove that it was indeed a photolysis product that was trapped by furan and not a short-lived excited state of thiophene, the photolysis of thiophene alone was carried out at –170°C in a glassy

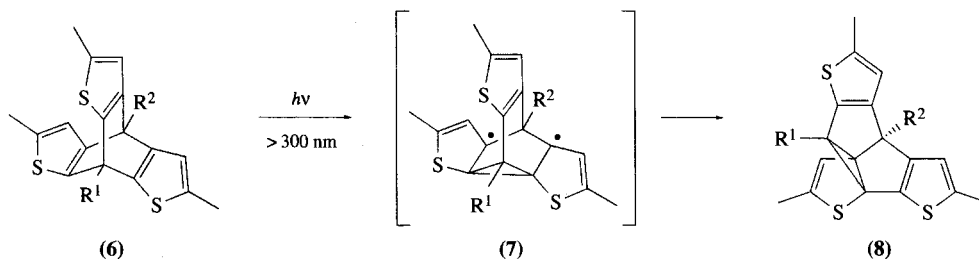
matrix and the product then reacted with furan in the dark to give the same two adducts. The photolysis (229 nm) has also been repeated in an argon matrix at 10 K. The products have been identified on the basis of their IR bands (Scheme 1) <86JA1691>. The Dewar structure (3) has been assigned to the photoproduct responsible for the strong absorption at 740 cm^{-1} . Irradiation of this at 254 nm or even at longer wavelengths ($> 320\text{ nm}$) appeared to result in its reversion to thiophene.



Scheme 1

Tetrakis(trifluoromethyl) Dewar thiophene has been converted to the Dewar furan in several steps <82JA847>. The first step in the sequence is the cycloaddition to pyrrole, followed by Ph_3P -induced desulfurization.

A theoretical study of the photochemical isomerization of thiophene as well as of its 2-cyano and 2-methyl derivatives has been published <91JOC129>. Photolysis of the thiophenetriptycenes (6) proceeds by the expected di- π -methane rearrangement to give (8) via the biradical (7) (Scheme 2). The cyclopropane ring in (8) then opens up to give products depending on the substituents R^1 and R^2 <93CL681>.

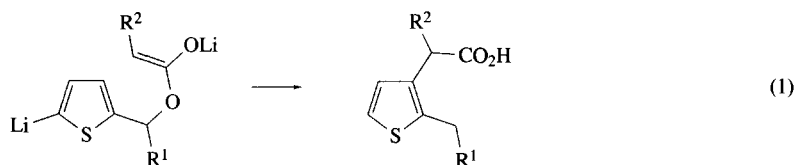


Scheme 2

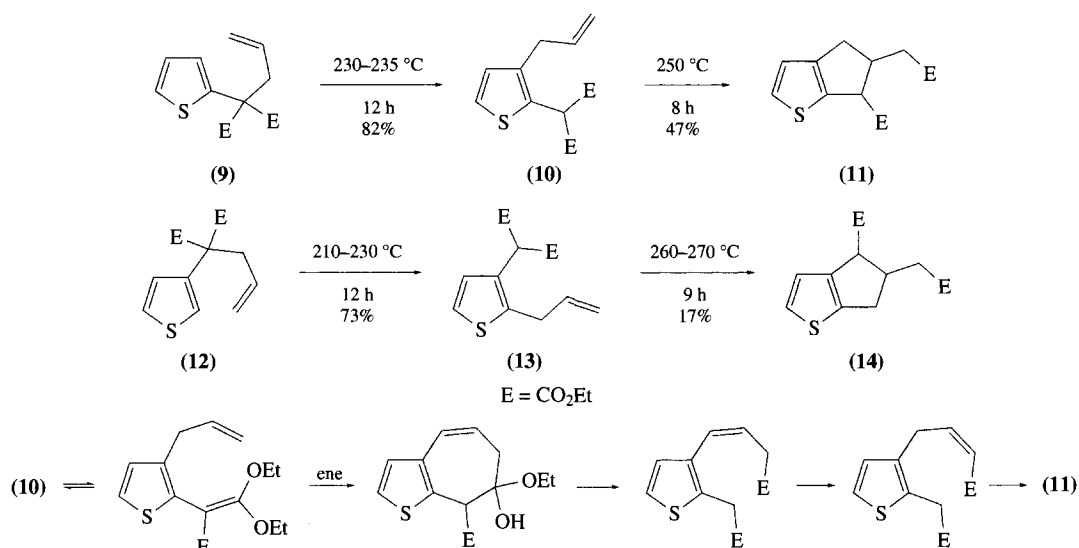
2.10.2.2.2 Sigmatropic rearrangements

Thermal rearrangement of diethyl α -allyl-2-thienylmalonate (9) and the corresponding 3-thienyl isomer (12) has been investigated <86JOC183>. In both cases, the normal [3,3]sigmatropic rearrangement products (10) and (13) are formed; these then, on further heating, produce the cyclopenta[*b*]thiophene derivatives (11) and (14). The cyclization to the cyclopentane derivative might proceed through a sequence of ene reaction, ring opening and ring closure (Scheme 3). Similar results have been obtained in the benzo[*b*]thiophene series <93JOC4360>.

The lithium enolates of a series of 1-(2-thienyl)alkyl alkanoates, generated by treatment with 2.2 equivalents of lithium *N*-isopropylcyclohexylamide undergo the Claisen rearrangement in refluxing THF to yield 2-(3-thienyl)alkyl alkanoic acids in 30–80% yield (Equation (1)) <83BCJ1665>.



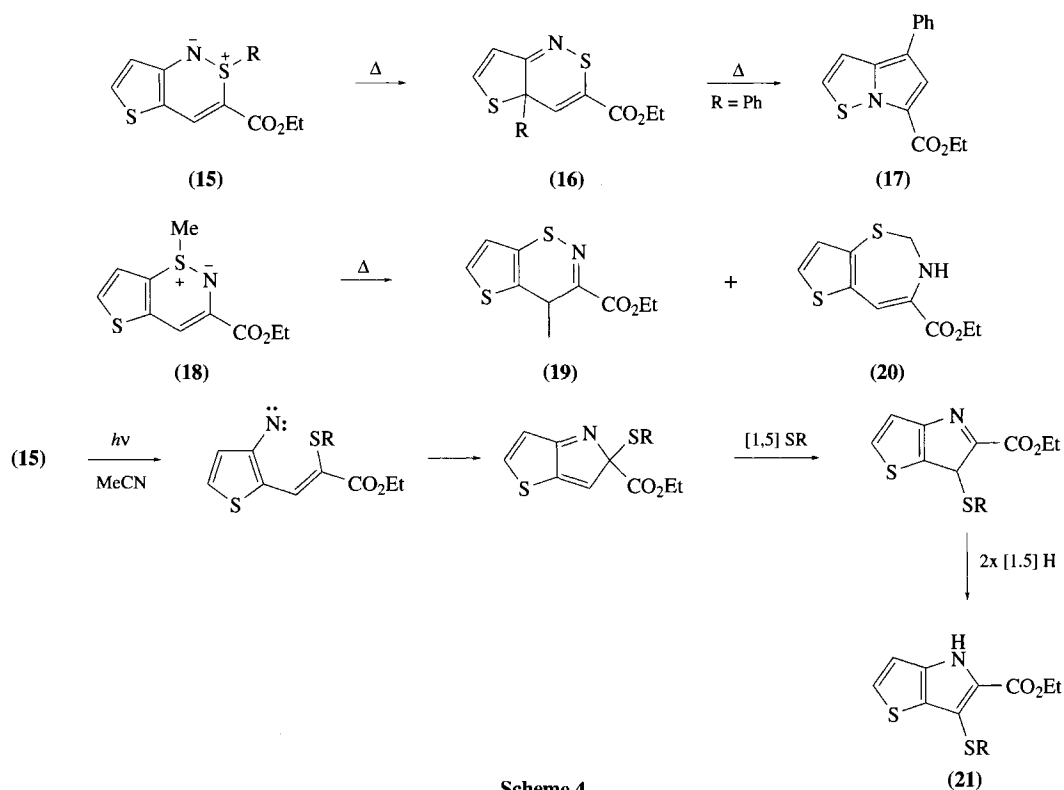
The thermal and photochemical rearrangement of the thieno[3,2-*c*][1,4,2]thiazines (15) has been



Scheme 3

investigated (82CC884, 86JCS(P1)491, 86JCS(P1)497, 86JCS(P1)501). On thermolysis, (15) undergoes a [1,4] migration of the sulfur substituent to produce the nonaromatic compound (16) (Scheme 4). The alternative [1,2]-shifted product was not observed. Further thermolysis of (16) ($R = \text{Ph}$) leads to the pyrrolo[1,2-*b*] isothiazole (17) with extrusion of sulfur. The isomeric thiazine (18) is thermally less stable. In boiling toluene, it gave two products, (19) and (20), the former arising by a [1,4] shift and the latter probably by a Stevens-type rearrangement.

Irradiation of (15) (MeCN, 300 nm or 350 nm) gave the isomeric thieno[3,2-*b*]pyrrole (21). It is

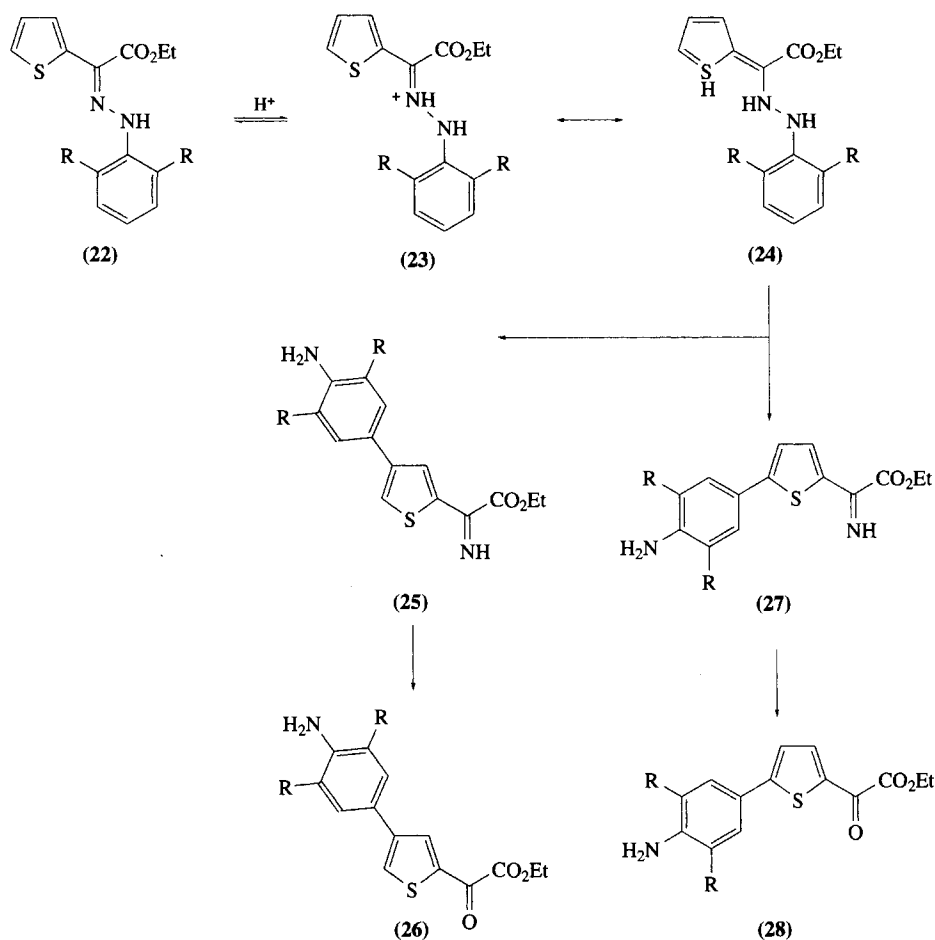


Scheme 4

likely that this proceeds through the initial formation of the nitrene, followed by ring closure and migration of the RS group and hydrogen (Scheme 4).

Two examples of the relatively rare [5,5] sigmatropic rearrangements have been observed when arylhydrazones of ethyl 2-thienylglyoxylate are treated with polyphosphoric acid (Scheme 5) <82JOC1691>. Thus, the 2,6-dimethylphenylhydrazone (**22**; R = Me) gives a 40% yield of (**26**; R = Me) while the unsubstituted phenylhydrazone (**22**; R = H) gives a 35% yield of a 7:3 mixture of (**26**; R = H) and (**28**; R = H). It is postulated that the first step in this transformation is a protonation to form (**23**); in the mesomeric structure (**24**), two distinct [5,5] sigmatropic systems can be discerned, leading respectively to (**25**) and (**27**) which on hydrolytic cleavage lead to the observed products.

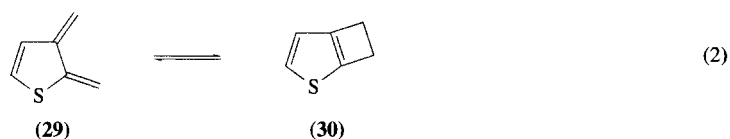
For the sake of convenience, the rearrangements of thiophene S,C-ylides and S,N-ylides are discussed in Section 2.10.3.3.



Scheme 5

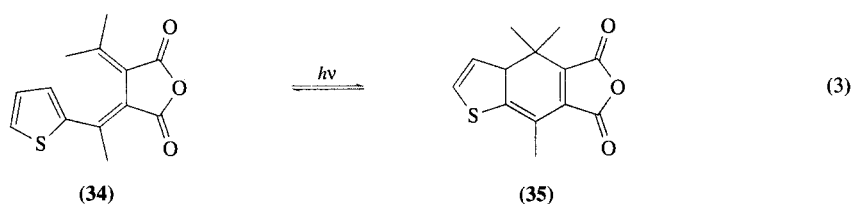
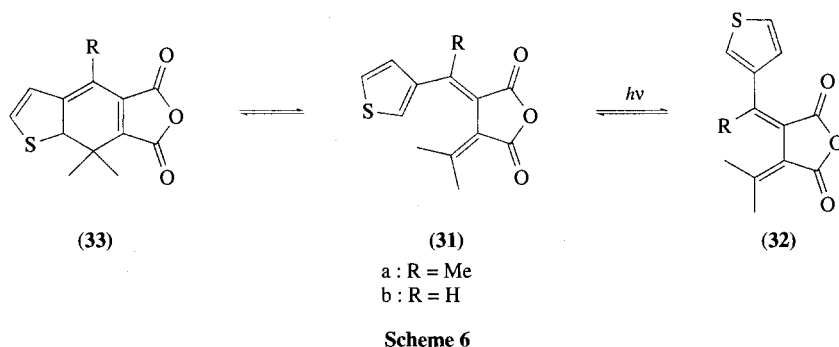
2.10.2.2.3 Electrocyclizations

The thermal equilibrium between 2,3-dihydro-2,3-dimethylenethiophene (**29**) and 1,2-dihydrocyclobuta[*b*]thiophene (**30**) has been investigated (Equation (2)) <88CB791>. The former was prepared by 1,4-elimination of HCl from 2-(chloromethyl)-3-methylthiophene in the vapor phase at 600°C. The results of photoelectron spectroscopic analysis in the gas phase as well as UV/vis-absorption spectroscopy in an argon matrix indicate no evidence for the ring-closed form (**30**). However, interconversion of (**29**) and (**30**) was achieved by irradiation in an argon matrix with light of the appropriate wavelength.



The light-induced reversible isomerization between two forms having different absorption spectra is referred to as photochromism and the compounds capable of this reaction are called "photochromic molecules." Obviously such systems may find application as optical data storage media, provided they satisfy certain essential criteria: (i) almost complete conversion in either direction; (ii) thermal stability of both isomers; and (iii) fatigue resistance of the compounds, i.e. no significant side reactions. Towards this objective, several hexatriene-cyclohexadiene systems, with the thiophene ring contributing one double bond, have been investigated. The thiophene rings seem to contribute to the thermal stability of both isomers.

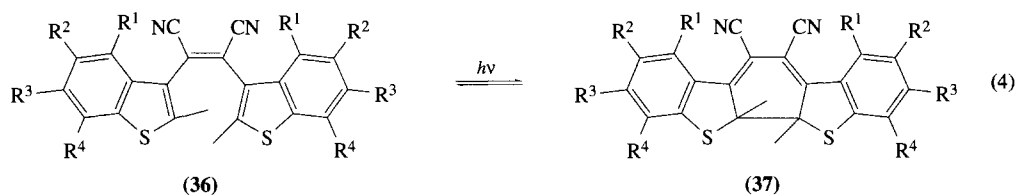
The "fulgides" are derivatives of bismethylenesuccinic anhydride; Stobbe showed a long time ago that if one of the substituents on the methylene groups was phenyl, the compound was photochromic. As a part of a study on heterocyclic fulgides, Heller and co-workers have studied the thermal and photochemical reactions of (*E*)-isopropylidene- α -[2- and (3-thienyl)ethylidene]succinic anhydrides <85JCS(P1)957>. Irradiation of the (*E*)-fulgide (**31a**) at 366 nm results in the formation of (**33a**) which has a deep red color. The reverse ring opening can be brought about by irradiating with white light. After 70 h irradiation of (**31a**), the product contained (**31a**), (**32a**) and (**33a**) in the ratio 6:3:1. Surprisingly, the fulgide (**31b**) does not undergo photocyclization; it merely isomerizes to the *Z*-form (**32b**). The electrocyclization of (**31a**) to (**33a**) could also be brought about thermally. The thermal disrotatory opening of (**33**) to (**31**) could be prevented by introducing a methyl group at the 2-position of thiophene; the steric hindrance between the two *cis*-oriented methyl groups apparently is responsible for this stability (Scheme 6). The 2-thienylfulgide (**34**) is similarly photochromic, leading to (**35**) on irradiation at 366 nm (Equation (3)).



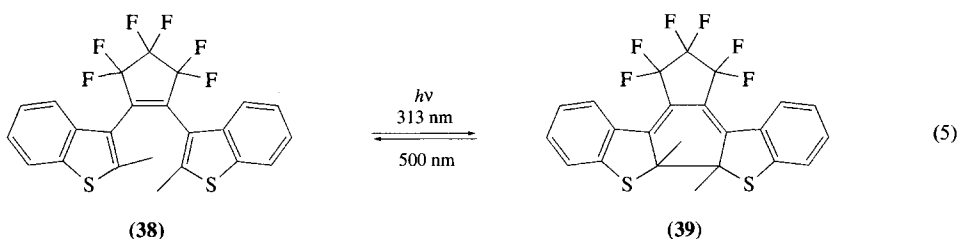
In an interesting application of this result, a photoswitchable unit has been incorporated by covalent linkage in a protein molecule <92JA3150>. This has resulted in the ability to photoregulate the binding properties of the protein by effecting minor perturbations in its conformations. The protein chosen was concanavalin A. This protein was covalently modified by reacting it with a thiophenefulgide derivative which resulted in amide bond formation with the ϵ -NH₂ groups of lysine residues. The resulting modified protein exhibited photochromic properties. Irradiation with light of wavelength between 300 nm and 400 nm resulted in conrotatory cyclization. The reverse conversion could be achieved by irradiation with light of wavelength >475 nm.

1,2-Diarylethenes containing heterocyclic rings have the advantage that they can undergo reversible photocyclization, but at the same time are thermally stable. The closed-ring molecules absorb light at significantly longer wavelengths. However, a complicating factor could be photo-induced

cis-trans isomerization; this could result in "fatigue." In a series of publications, Irie and co-workers have made tremendous progress and introduced several new concepts in the design of suitable diarylethene photochromic systems incorporating thiophene or benzo[*b*]thiophene rings (91BCJ202, 92CL2257). 1,2-Dicyano-1,2-bis(2-methylbenzo[*b*]thiophen-3-yl)ethene (36) and its ring-substituted derivatives (cyano or halogeno) are very interesting in this context (90BCJ1311, 92BCJ430). The compounds were synthesized by the coupling of two molecules of the cyanomethylbenzo[*b*]thiophene in strong aqueous NaOH. The *cis*-isomers were separated and used for photochromism studies. Irradiation of (36) ($\lambda_{\text{max}} < 400$ nm) at an appropriate wavelength led to the ring-closed forms (37) (λ_{max} 500 nm). The products appeared to be thermally stable (Equation (4)).

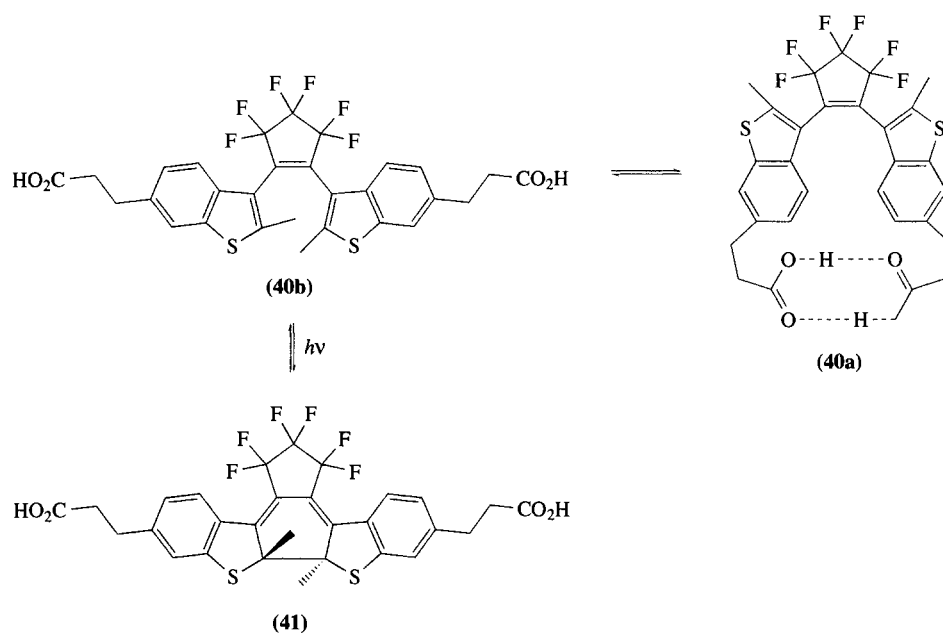


In a significant improvement, the central double bond of the diarylethene has been incorporated in a perfluorocyclopentene ring. The diarylethene is thus locked in the *cis*-configuration, thereby preventing fatigue due to *cis-trans* isomerization. The fluorine atoms give added thermal stability to the products (92CC206). The compound (38) was prepared by adding 3-lithio-2-methylbenzo[*b*]thiophene to perfluorocyclopentene, followed by elimination. In hexane solution (38) has λ_{max} at 258 nm. On irradiation with light at 313 nm, cyclization takes place to produce (39) with λ_{max} at 517 nm (Equation (5)). At photostationary conditions, the ratio (38):(39) is 55:45. The reverse reaction could be brought about by irradiating with light of wavelength ≥ 500 nm. The coloration (cyclization)–decoloration (ring-opening) cycle could be repeated 14000 times without significant loss of performance (90%).



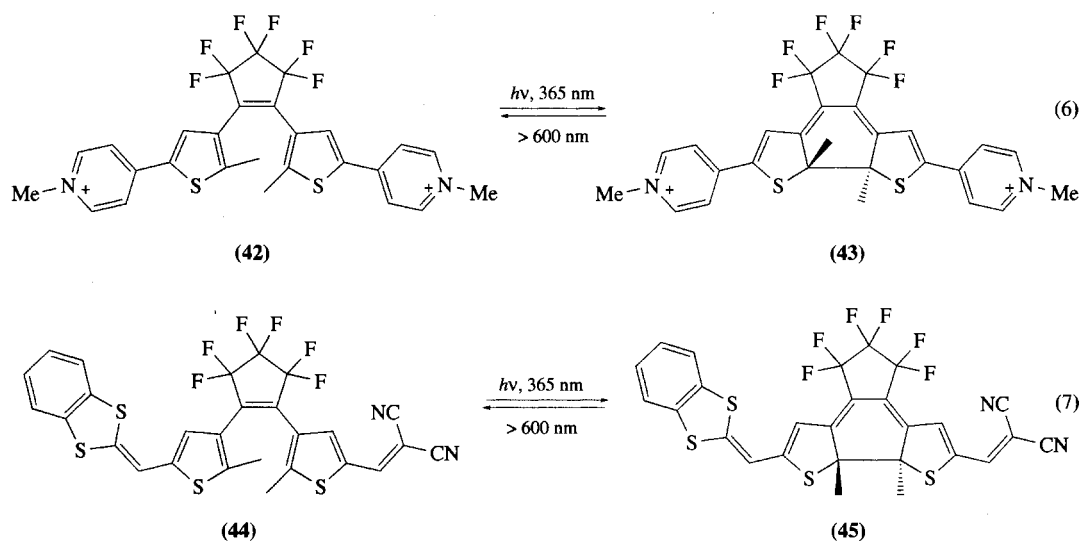
A radical advance was achieved by introducing propionic acid residues at the 6 and 6' positions of (38). This compound (40) provides a beautiful illustration of the extremely interesting concept of designing photochromic molecules possessing a "clasp" which can be released by another "switch" molecule (92JA8715). Compound (40) exists in the intramolecular H-bonded conformation (40a) in cyclohexane. In this conformation, it is unable to undergo photo-induced conrotatory electrocyclicization. However, on addition of ethanol, the intramolecular H bonds are broken and the molecule is free to switch over to the conformation (40b). Irradiation with light of wavelength 313 nm then results in cyclization to form (41) which exhibits an absorption band at about 525 nm (Scheme 7).

Lehn and co-workers have extended Irie's ideas in an approach to the design of light-triggered electrical and optical switching devices (93CC1439). The basic concept was to utilize the photochromic property of dithienylethenes as a switch; the crucial advance here was the appropriate choice of substituents on the thiophene rings. Three types of molecule have been synthesized. In the first (42), the central dithienylperfluorocyclopentene unit has two pyridinium rings attached in such a way that in the cyclized form (43) they are at the termini of an extended conjugated system (Equation (6)). Irradiation of (42) with light of wavelength 365 nm resulted in almost complete conversion to (43). The difference in λ_{max} of (42) and (43) was of the order of 300 nm. The ring opening of (43) to form (42) could be carried out by irradiation with light of wavelength > 600 nm. The second type of molecule investigated by Lehn is the push–pull system represented by (45). This is the cyclized form of (44). The conversion of (44) to (45) ($h\nu$, λ 365 nm) and the reverse transformations of (45) to (44) ($h\nu$, $\lambda > 600$ nm) again proceed with very high conversion (> 98 – 99%) (Equation (7)). The difference in λ_{max} between (44) and (45) is 464 nm. The nonlinear optical behavior of (45) is being



Scheme 7

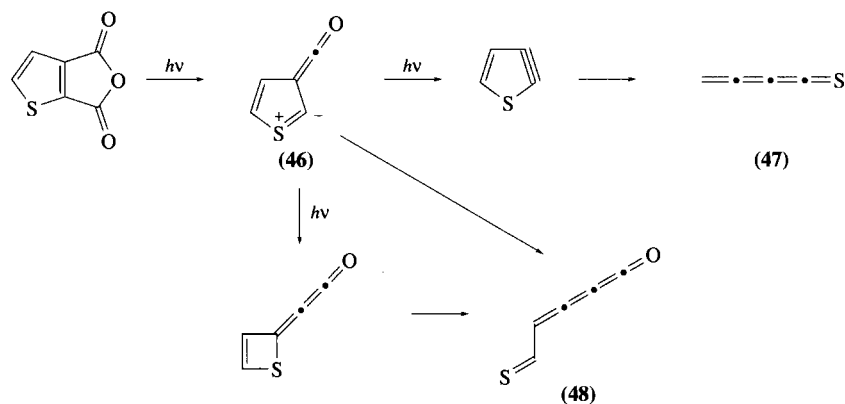
investigated. The third type incorporates a dual-mode switching device based on photochromic and electrochromic properties. In this case, the thiophene rings carry *p*-hydroxyphenyl substituents and can be oxidized to the quinonoid form <94CC1011>.



2.10.2.2.4 Thiophyne and 3,4-dimethylenethiophene

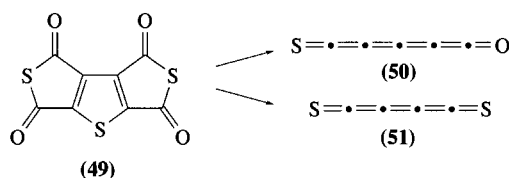
An excellent review on heterynes has critically examined the evidence for the formation of thiophyne <82T427>. The conclusion is that so far, the flash-vacuum pyrolysis (FVP) experiments on the dicarboxylic acid anhydrides provide reasonably satisfactory evidence for the generation and trapping of thiophyne and benzothiophyne. The FVP of 2,3-thiophenedicarboxylic acid anhydride has been reinvestigated recently by using the technique of matrix isolation combined with FT-IR spectroscopy <92CB423>. Earlier, Reinecke carried out the pyrolysis of this compound under a stream of a suitable trapping agent and the adducts of (presumably) thiophyne with the diene isolated <84CHEC-1(4)750>. In the present study, no conclusive proof for the existence of 2,3-didehydrothiophene (thiophyne) could be found. The main products (isolated in an argon matrix at 12

K) were CO_2 , CO , CS_2 , C_2H_2 , OCS , thioketene and CS . Irradiation of the anhydride with light of 313 nm in an argon matrix at 12 K gave (46); further photolysis led to (47) and (48) (Scheme 8). Whether thiophyne is an intermediate in the conversion of (46) to (47) is not clear.



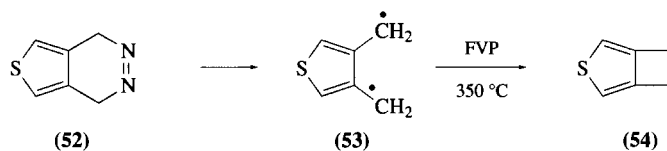
Scheme 8

Thiophene tetracarboxylic bithioanhydride (49), either on flash pyrolysis or on matrix photolysis gives (50); (51) has also been obtained on matrix photolysis (Scheme 9) <91CB2613, 91CB2617>.

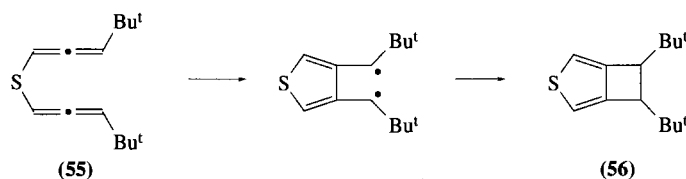


Scheme 9

The π -conjugated non-Kekule molecule, 3,4-dimethylenethiophene (53) has been generated from the diazene (52) by either thermolysis (RT; 4 h) or photolysis (310–380 nm, -78°C). The purple-colored biradical (53) undergoes thermal dimerization at this temperature (Scheme 10). It is stable up to 160 K as frozen glassy solutions in Carbowax 400 or 2-methyltetrahydrofuran. It has λ_{max} 568–572 nm ($\epsilon > 10^4$). ESR studies have shown no evidence of triplet states; the molecule has therefore been assigned a singlet structure <86JA8088, 89JA3659, 89JA3671, 91JA2318>. The cycloaddition reactions of (53) are discussed in Section 2.10.2.9.4. Bond formation between the two radicaloid carbon atoms takes place under FVP conditions to form the four-membered fused-ring compound (54). A similar molecule (56) with two *t*-butyl substituents had earlier been prepared from the bis(allyl)sulfide (55) by warming to 50°C (Scheme 11) <78TL691>.



Scheme 10



Scheme 11

2.10.2.3 Electrophilic Attack on Carbon: General Considerations

Electrophilic substitution of thiophene has been expertly reviewed by Taylor <86HC(44/2)1>. In addition, a whole monograph has been devoted to the compilation and analysis of quantitative data on electrophilic substitution of heterocycles <90AHC(47)87>. The highlights from the conclusions reached in the section on thiophene are presented below.

(i) Aromaticity and relative reactivity: The ground-state stability and aromaticity decrease in the order benzene > thiophene > pyrrole > selenophene > tellurophene > furan. Hence, the above heterocycles will be more reactive than benzene. However, among these heterocycles, the order of reactivity will also be governed by the polarizability of the heteroatom. Thus, sulfur is more polarizable than oxygen, but thiophene has greater ground-state stability than furan. A single σ^+ parameter will therefore not accurately describe the quantitative reactivities of these heterocycles under all conditions. (For a definition of σ^+ <84CHEC-I(4)52>.)

(ii) Relative rates: The order of reactivities at position 2 in the above heterocycles appears to be pyrrole > furan > tellurophene > selenophene > thiophene. Where data are available for both 2- and 3-positions, the following order is seen: 2-furan > 2-thiophene > 3-furan > 3-thiophene. Reactivity parameters ($-\sigma^+$) for 2- and 3-positions of thiophene for reactions of varying ρ -values (ranging from -0.66 to -12.0) have been given.

(iii) Sensitivity to substituent effects: Conjugative effects are strongly transmitted in the above five-membered heterocycles. Obviously, the four-carbon chain is important in this transmission. The greater the degree of bond fixation in the heterocycle, the more difficult will it be to place a double bond across C-3 and C-4. The ease of transmission will therefore parallel the aromaticity, viz. benzene > thiophene > pyrrole > selenophene > tellurophene > furan. Thiophene is less effective at transmitting substituent effects than benzene (except in protiodesilylation). Hammett ρ factors for benzene and thiophene have been compared. The greater ability of substituents on thiophene to attain coplanarity with the heteroaromatic ring, when compared with the situation with benzene, may result in a magnification of the resonance effect ($+M$ or $-M$). For the same reason, for reactions involving side-chain carbocations, the heterocycle may appear to be a better transmitter than benzene.

Electrophilic reactions of benzothiophenes have also been analyzed <90AHC(47)181>. There are no quantitative reactivity data for benzo[*c*]thiophenes because of stability problems. In benzo[*b*]thiophene, position 3 is usually more reactive than position 2; this can be easily understood in terms of the number of canonical structures having an aromatic benzene ring for the transition state for substitution. Thus, when conjugative effects are relatively unimportant, the reactivity order is $3 > 2$. But for reactions with transition states nearer to the Wheland intermediate, position 2 might become as reactive as position 3. In the benzene part, the positional reactivity is $6 > 5 > 4 > 7$. Turning to the quantitative aspect, the σ^+ values are not constant; they generally increase with increasing demand for resonance stabilization of the transition state, such as in acetylation and molecular halogenation.

2.10.2.4 Electrophilic Attack on Carbon: Specific Reactions

2.10.2.4.1 Proton acids

Acid-catalyzed hydrogen exchange in thiophenes has been exhaustively reviewed <86HC(44/2)1, 90AHC(47)87>. One of the conceptually new developments in this area is the recognition that thiophene (as well as furan and pyrrole) can participate in hydrogen-bond formation in certain acidic solvents, thereby reducing the reactivity. This effect, although small, may still be significant. Evidence for this came initially from the determination of rate coefficients for detritiation of 2-tritio- and 3-tritiothiophene in 100% TFA and TFA-HOAc (35:65). The relative rate for thiophene in these two systems is 2420, compared with 5230 for mesitylene. The lower value for thiophene is ascribed to hydrogen bonding of the solvent to sulfur, thereby reducing the availability of the lone-pair for resonance with the π -electrons of the ring. The extent of hydrogen bonding decreases with increasing proportion of HOAc in the mixture.

The kinetics of protonation and hydronium-ion catalyzed hydrolysis of alkoxythiophenes, alkoxybenzo[*b*]thiophenes, and (alkylthio)benzo[*b*]thiophenes have been determined by $^1\text{H-NMR}$ spectroscopy <87T69, 92T7823>. Thus, when 3-methoxythiophene is dissolved in $\text{CD}_3\text{CN}:\text{D}_2\text{O}$ which is 0.1 M in DCl, the H-2 signal disappears completely after 3 min at 32°C , but no hydrolysis takes

place. With 2-methoxythiophene, exchange at position 5 is complete before hydrolysis takes place, but exchange at position 3 is only 30 times faster than hydrolysis. In the case of 3-methoxybenzo[*b*]thiophene, some hydrolysis was detected before exchange was complete. With both 2-(methylthio)- and 3-(methylthio)benzo[*b*]thiophene the exchange rate was much faster than the rate of hydrolysis. The conclusion has been reached that in all these cases, C-protonation is rapid and reversible; the slow step is attack by water. Based on these results, the reversibility of protonation of such enolethers has been used as a measure of aromaticity of the system. The aromaticity index is defined as:

$$\text{Aromaticity index} = \log k_{\text{ex}}/k_{\text{hydr}}$$

2.10.2.4.2 Nitration

Nitration of thiophenes has been exhaustively reviewed <86HC(44/2)523, 90AHC(47)87>. Very few new results have been reported in this area since the mid-1980s.

2-Acetyl-5-nitrothiophene can be prepared by regiospecific nitration of the oxime ether of 2-acetylthiophene (yield 75%), followed by hydrolysis <92S849>.

2.10.2.4.3 Reaction with electrophilic sulfur

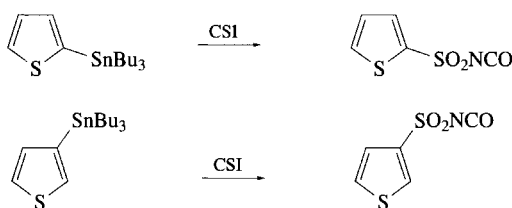
Methods available for the synthesis of thiophene derivatives in which the ring carbon atom is linked to a sulfur have been reviewed <86HC(44/3)135>. The compounds covered include thio-phenethiols, sulfides, sulfoxides, sulfones, sulfonic acid derivatives, thiocyanatothiophenes, etc. A brief account of the reactions of thiophenes with electrophilic sulfur had also been presented in CHEC-I <84CHEC-I(4)741>. Therefore only those few reports which have appeared subsequent to the above will be discussed here.

3-Aroylthiophenes have been sulfonated with conc. H_2SO_4 /acetic anhydride to give 4-aro-ylthiophene-2-sulfonic acids in good yield <89JHC1793>. Reaction of the above substrates with chlorosulfonic acid led to intractable materials.

Several thiophenesulfonyl chlorides have been prepared by using the $\text{DMF-SO}_2\text{Cl}_2$ complex for chlorosulfonation <85BCJ1063>. This reaction failed with 2-acetylthiophene, which gave only unidentified materials. In the case of 2-methoxythiophene, only formylation at position 5 resulted.

Sulfonation of 2-acetylthiophene at position 5 has been achieved in 93% yield by first converting 2-acetylthiophene to its oxime ether. Sulfonation was carried out with fuming sulfuric acid <92S849>.

One of the most interesting new developments in this area is that of “*ipso*-specific” introduction of the sulfonyl isocyanato group <93JOC7022>. Normally CSI reacts with thiophene on the electrophilic isocyanato group to give, after hydrolysis, the corresponding carboxamide. However, 2-thienyl- and 3-thienyl(tributyl)stannanes react with CSI at 20°C to give the corresponding sulfonyl isocyanates by *ipso*-substitution (Scheme 12). The yields are >90%.



Scheme 12

2.10.2.4.4 Halogenation

The halogenation of thiophene and substituted thiophenes has been dealt with adequately in CHEC-I <84CHEC-I(4)741>. Subsequently, an exhaustive account of all aspects of halogenation of thiophenes was published <86HC(44/2)159>. A more recent review of the halogenation of five-membered heterocycles has made an extensive survey of the literature on halogenation of thiophenes

<93AHC(57)291>. It is therefore not proposed to reproduce all the available data in this chapter. Instead, only the major advances will be highlighted, together with newer information.

The most significant advance in the synthesis of β -halogenothiophenes has been the zeolite-catalyzed isomerization of the α -isomers <87AG(E)468>. This is dealt with in Section 2.10.4.6.5.

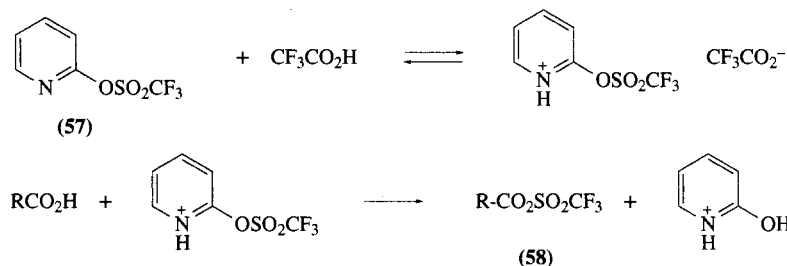
Regiospecific chlorination and bromination at the 5-position of 2-acetylthiophene has been achieved by converting it initially to the oximinoether and then halogenating it (molecular chlorine or bromine in the presence of Fe). Final hydrolysis of the oximinoether leads to the product <92S849>.

Benzyltrimethylammonium (BTMA) polyhalides have been shown to be useful for halogenation of thiophenes <91BCJ2566>. The reaction is carried out in acetic acid. The tetrachloroiodate ($\text{PhCH}_2^+\text{NMe}_3 \cdot \text{ICl}_4^-$) leads to chlorination; for bromination and iodination, the tribromide ($\text{PhCH}_2^+\text{NMe}_3 \cdot \text{Br}_3^-$) and dichloroiodate ($\text{PhCH}_2^+\text{NMe}_3 \cdot \text{ICl}_2^-$) are used in conjunction with zinc chloride. The reagents are stable solids. However, it has not been possible to stop the chlorination of thiophene at the monochloro level using this reagent. Otherwise, very good yields of the products have been obtained.

Benzeneseleninyl chloride in combination with AlCl_3 , has been used for α -chlorination of thiophenes; bromination takes place if AlBr_3 is used <93AHC(57)291>.

2.10.2.4.5 Acylation

The acylation of thiophenes has been reviewed <86HC(44/3)309>. The best catalyst for the Friedel–Crafts acylation of thiophene, using free carboxylic acids, appears to be 2-(trifluoromethylsulfonyloxy)pyridine (**57**) in conjunction with TFA. The 2-acylthiophene is formed in quantitative yield, without any evidence of the thiophene dimerizing. The reaction might be mediated by the mixed anhydride (**58**) (Scheme 13) <88BCJ455> (cf. <84CHEC-1(4)741>).



Scheme 13

Friedel–Crafts acetylation of 2-ferrocenylthiophene leads to the acetyl group entering position 5 of the thiophene ring. Vilsmeier formylation similarly gives the 5-formyl derivative. Yields are around 20–25% <94T5765>.

The introduction of a thiocarboxamide function at the 2-position of thiophene by reaction with alkyl and aryl isothiocyanates is best carried out in MeNO_2 solution with AlCl_3 as the catalyst. Yields are excellent. 2,5-Dimethylthiophene is thioacylated at position 3 under these conditions, but the yields are lower <86T3683>.

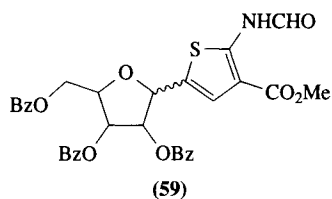
The AlCl_3 -catalyzed Fries rearrangement of 3-alkanoyloxythiophenes leads to 3-hydroxy-2-alkanoylthiophenes <86JCS(P1)507>. The rearrangement may occur by an intermolecular mechanism as shown by crossover studies. This parallels the earlier results on 2-acetoxythiophenes.

2.10.2.4.6 Alkylation

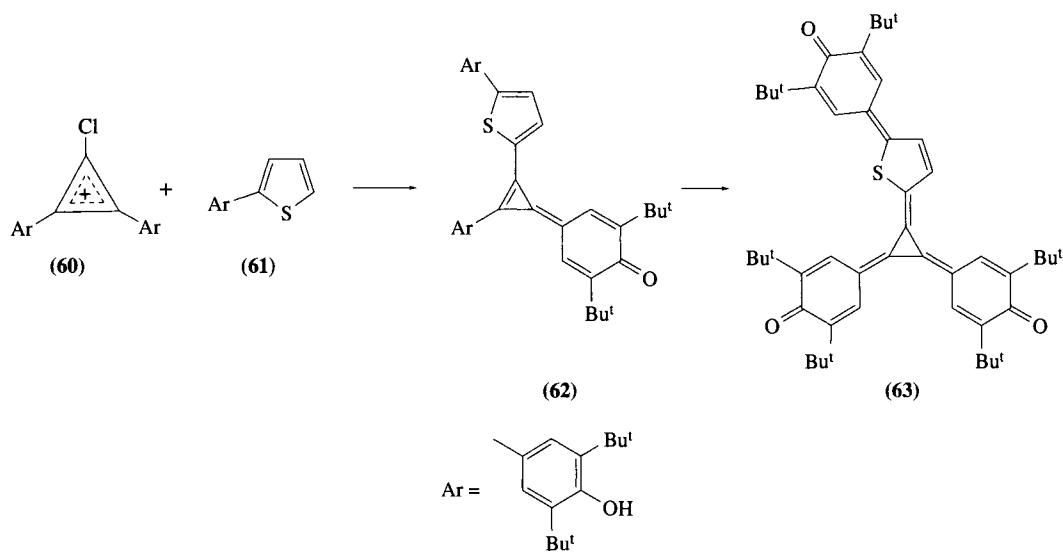
Silica gel has been found to be an effective catalyst for the *t*-butylation of thiophene and benzo[*b*]thiophene using *t*-butyl bromide. 2,5-Di-*t*-butylthiophene and 3-*t*-butylbenzo[*b*]thiophene can be prepared easily by this procedure <84JOC4161>. Alkylation of thiophene with *t*-butyl chloride, isopropyl chloride or ethyl chloride at -70°C in the presence of AlCl_3 produces σ -complexes under kinetic control. On thermal equilibration, migration of alkyl from position 3 to position 2, as well as disproportionation to dialkyl- and trialkyl thiophenes can occur <86T759>. 3,4-Di-*t*-butyl-

thiophene has been prepared by ring synthesis and its reaction with electrophiles studied. With AlCl_3 at room temperature, it rearranges quantitatively to 2,4-di-*t*-butylthiophene <88TL1161>.

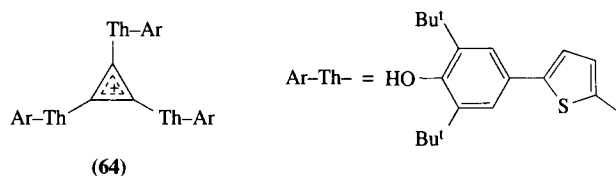
C-Glycosylation of a thiophene has been reported <88TL3537>. SnCl_4 -catalyzed reaction of *N*-formyl-4-aminothiophene-3-carboxylic ester with 1-*O*-acetyl-tri-*O*-benzoyl- β -D-ribofuranose gave the product (59) in 43% overall yield, with a β/α ratio of 2.4. Fusion of a pyrimidine ring on the thiophene in (59) gave a C-nucleoside inosine analogue.



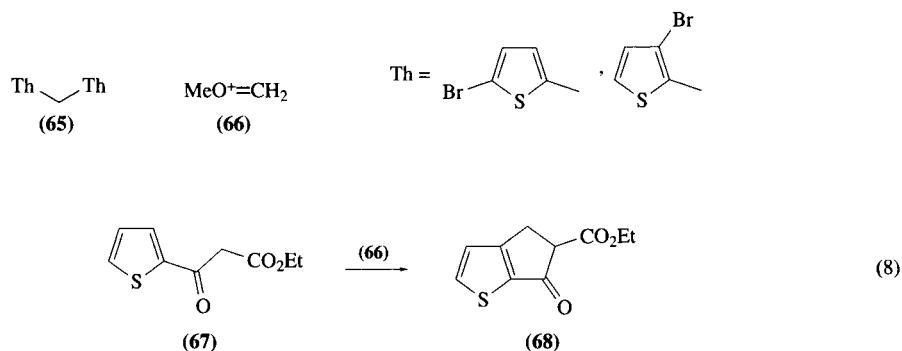
The synthesis of trithienylcyclopropenium salt by the reaction of trichlorocyclopropenium ion with excess thiophene has been mentioned in CHEC-I <84CHEC-I(4)741>. This reaction has been used to synthesize radialenes attached to quinones. Treatment of 2,6-di-*t*-butylphenol with the trichlorocyclopropenium ion gave 1,2-bis(hydroxyphenyl)chlorocyclopropenium ion (60). This, on reaction with the thienylphenol (61) in chloroform at room temperature, followed by reflux, gave a 20% yield of (62) (Scheme 14). Oxidation of this with potassium ferricyanide gave (63) <91TL3507>. Similarly, reaction of C_3Cl_3^+ with 3 equiv. of the thienylphenol (61) in MeCN gave a 37% yield of (64). Treatment of this cation with excess KOH powder in CH_2Cl_2 in a vacuum-sealed tube under sonication gave the corresponding dianion with extended conjugation <93CL911>.



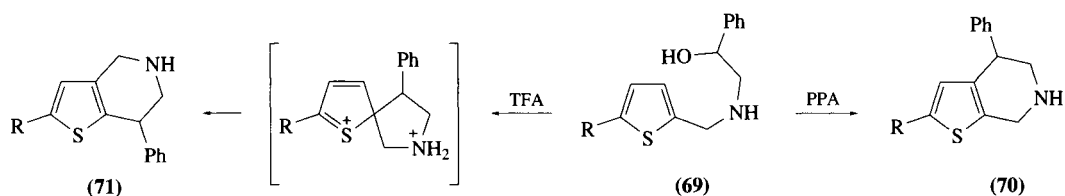
Scheme 14



The methoxyacetyl chloride- AlCl_3 reagent reacts with 2- and 3-bromothiophene to give dithienylmethanes (65) in 45–50% yields. The reaction might proceed through the cation (66) <83TL1933>. This complex has also been found to be a good reagent for methylenation and cyclization of the thiophene (67) to form (68) (Equation (8)) <92TL4771>.

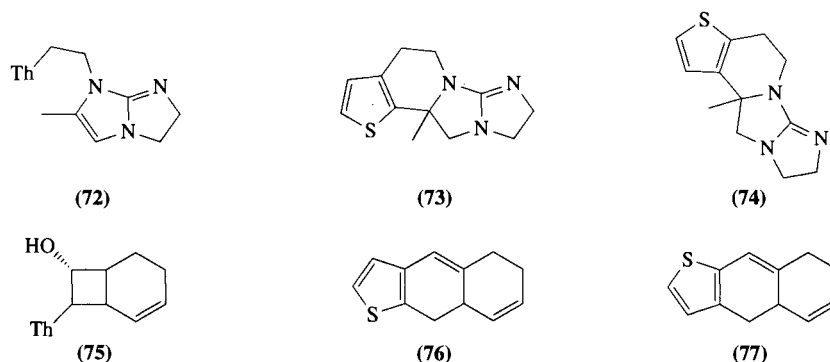


Several other cyclizations have been achieved by intramolecular electrophilic attack on thiophene. Thus, acid-catalyzed cyclization of suitable substrates has been used to synthesize several thienomorphans <91H(32)107>. Interestingly, the product of cyclization of (69) depends on the acid catalyst employed. While polyphosphoric acid gives the expected (70), refluxing TFA leads to the rearranged product (71) <82CC793>.



Scheme 15

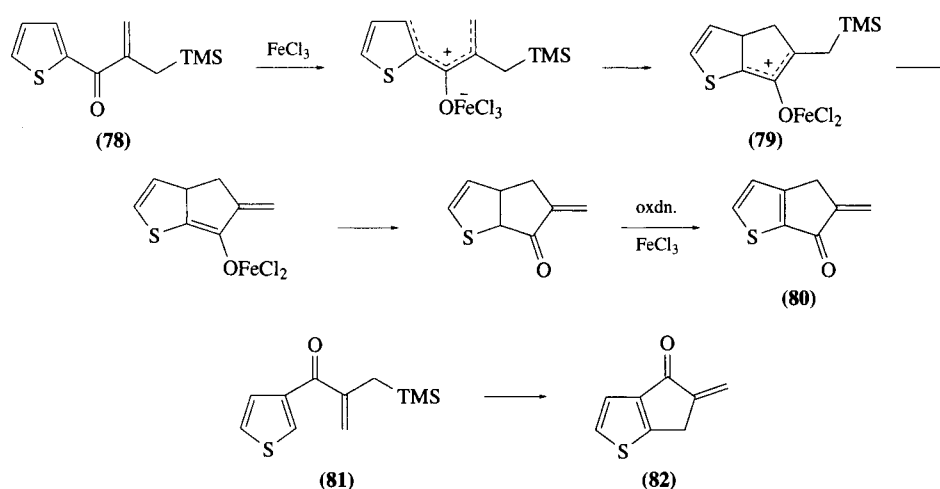
Acid-catalyzed generation of amidinylium ions from (72) leads to cyclization. Depending on the starting material, cyclization has been achieved both at positions 2 and 3 of thiophene, leading to (73) and (74), respectively <90S72>. Similarly, acid-catalyzed rearrangement of the cyclobutanols (75) leads to the tricyclic compounds (76) or (77) depending on the original linkage of the thiophene unit <87CJC1663>.



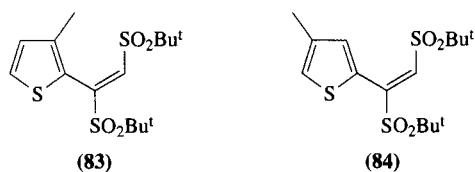
Cyclohepta[*a*]thieno[*c*]naphthalenylium ions have been synthesized by intramolecular cyclization of *o*-(thienyl)phenyltropylidenes, using trityl tetrafluoroborate to generate the carbocations. The yields are >70% <92CL1213>.

Reaction of the thienyl vinyl ketones (78) and (81) with FeCl_3 at 30°C gave the α -methyl-enecyclopentanones (80) and (82). The suggested mechanism is shown in Scheme 16 <92TL3495>; the surprising aspect is the loss of silicon rather than a proton from (79).

Alkenyl chains can be directly introduced into the thiophene ring by appropriate choice of electrophile. Bis(*t*-butylsulfonyl)acetylene adds to 3-methylthiophene to give an 82:18 mixture of the 2-alkenyl (83) and 5-alkenyl (84) derivatives <90TL2173>. This Michael-type addition of thiophene does not seem to have a precedent.



Scheme 16



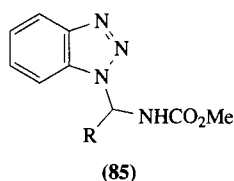
Highly conjugated donor–acceptor systems, in which the acceptor is a tricyanoethylene attached to a thiophene have been synthesized $\langle 93\text{CC1118} \rangle$. An example is shown in Equation (9).



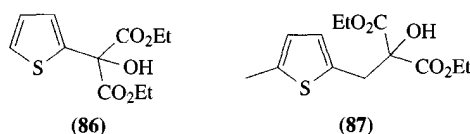
2.10.2.4.7 Reactions with aldehydes and ketones

The use of methoxyacetyl chloride as a formaldehyde equivalent has been referred to in Section 2.10.2.4.6. At least two other aldehyde equivalents have been used to alkylate thiophenes. Dimethoxymethane reacts with 2-bromothiophene to give the corresponding dithienylmethane in excellent yield $\langle 94\text{TL3493} \rangle$. This is the starting point for the synthesis of a tetrathiaannulene. Another aldehyde equivalent is *N*-(α -benzotriazolylalkyl)carbamate (**85**), itself prepared from the condensation of benzotriazole, the concerned aldehyde, and alkyl carbamate. The advantage of using this reagent is that both symmetrical dithienylmethanes and unsymmetrical diheteroarylmethanes can be synthesized $\langle 93\text{JOC4376} \rangle$. The reactions are carried out at room temperature in CH_2Cl_2 in the presence of ZnCl_2 .

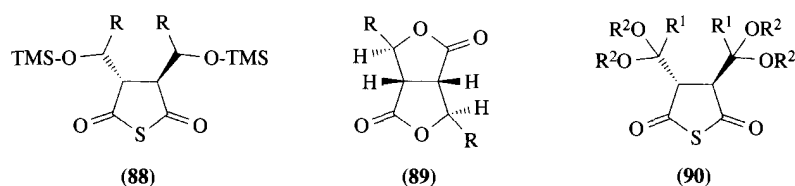
Ketones activated by adjacent carboxylic ester groups react with thiophenes under high-pressure



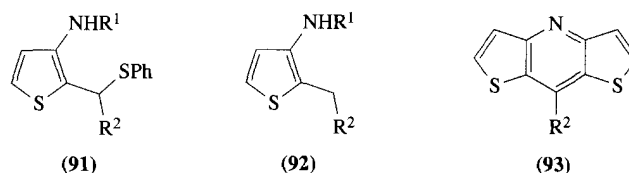
conditions <84TL5747>. Thiophene itself reacts with diethyl mesoxalate at position 2 to give **(86)** in 30% yield, but 2,5-dimethylthiophene reacts on the methyl group to give **(87)** in 85% yield.



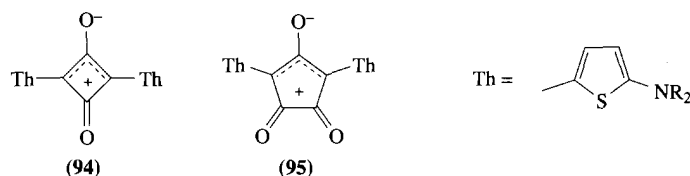
Electrophilic attack by aldehydes can also take place at positions 3 and 4 in suitably substituted thiophenes. Thus, 2,5-bis(trimethylsilyloxy)thiophene reacts with aromatic aldehydes in the presence of the Lewis acid trimethylsilyl trifluoromethanesulfonate (0.1–1.0%) to form 3,4-disubstituted thiosuccinic anhydrides **(88)** in quantitative yield. With aliphatic aldehydes, the yields are lower, the catalyst being ZnBr_2 ; the products so obtained have been hydrolyzed in the presence of lead acetate to form the lactones **(89)** in 30–40% yields. 2,5-Bis(trimethylsilyloxy)thiophene also reacts with orthoesters in the presence of trimethylsilyl triflate to form **(90)** <87C152, 92LA615>.



N-Substituted 3-aminothiophenes react with aldehydes in the presence of catalytic *p*-toluenesulfonic acid (pTSA) to form bis(3-amino-2-thienyl)methane derivatives in very good yields. If the reaction medium contains thiophenol, then the phenylthiomethylthiophene **(91)** is formed. Interestingly, if the reaction of the 3-aminothiophene with aldehyde is carried out in the presence of diphenyldiselenide and an acid catalyst, the 2-alkyl-3-aminothiophenes **(92)** are formed in good yields. It is likely that the selenophenol generated *in situ*, forms the phenylselenide analog of **(91)**, which is reduced by attack of a second molecule of selenophenol <93TL5715>. The *N*-unsubstituted 3-aminothiophene gets converted to the tricyclic heterocycle **(93)** on treatment with aliphatic or aromatic aldehydes and TFA in CH_2Cl_2 . This transformation involves an oxidation step as well. Treatment of di(3-thienyl)amine with the aldehyde and catalytic pTSA at 0°C gives the dihydro derivative of **(93)**. A longer reaction time leads to a dimer containing four thiophene rings <93TL5719>.



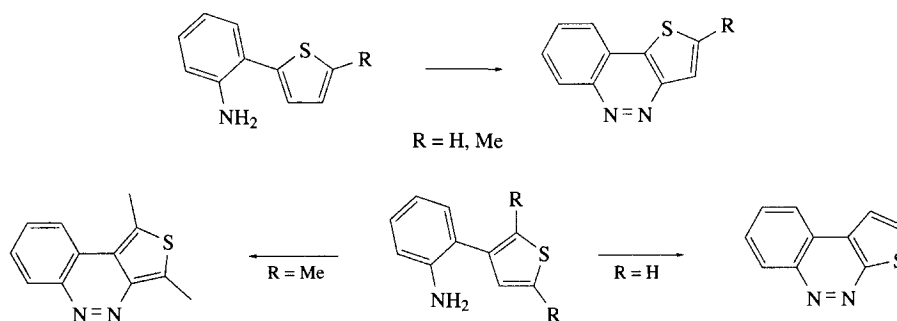
2-Dialkylaminothiophenes react at position 5 with squaric acid and croconic acid to form the squaraines **(94)** and croconines **(95)** <92CRV1227, 93LA935>. Other enaminic reactions of aminothiophenes are discussed in Section 2.10.4.3.2.



2.10.2.4.8 Diazo coupling

Thiophene and its monomethyl derivatives do not undergo coupling with diazonium salts in intermolecular reactions <84CHEC-I(4)763>. Surprisingly, however, it has been found that intra-

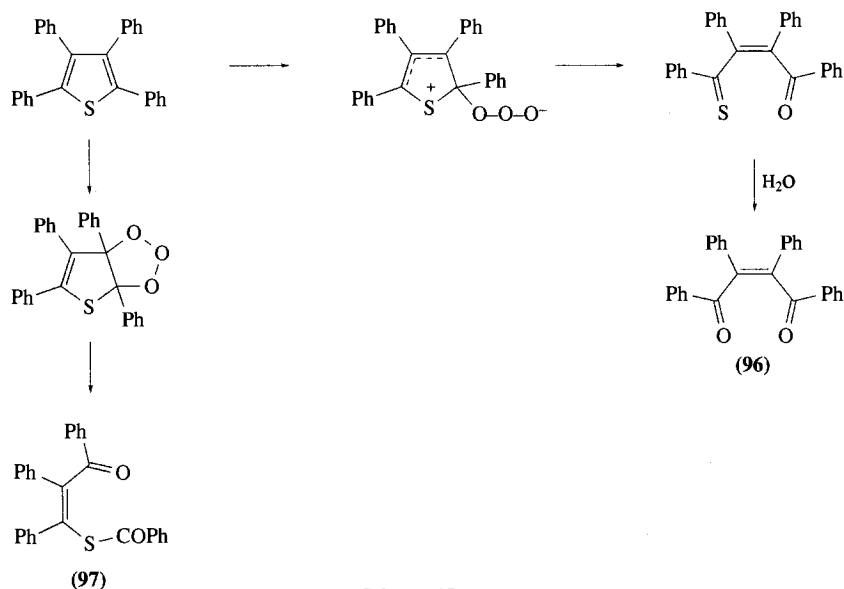
molecular diazo coupling can be achieved on thiophene <85JCS(P1)131>. Starting from suitable (*o*-aminophenyl)thiophenes, this leads to various isomeric thieno[*c*]cinnolines (Scheme 17). Methyl substitution increases the yield of the diazo-coupled product. Naturally, coupling at position 2 is more facile than at position 3; if both the α -positions are blocked in a 3-(*o*-aminophenyl)thiophene, then coupling can take place at position 4.



Scheme 17

2.10.2.4.9 Electrophilic oxidation

The reaction of 2,5-diphenyl- and 2,3,4,5-tetraphenylthiophene with ozone has been studied <85JOC1778>. At 0°C, the latter gave, after ethanolic workup, the butenedione (**96**) (61%) and thioester (**97**) (20%). No sulfone corresponding to the starting material could be detected. The butenedione obviously arises by an electrophilic attack at position 2 by ozone (Scheme 18). Similarly, 2,5-diphenylthiophene gave an 18% yield of $\text{PhCO}-\text{CH}=\text{CH}-\text{COPh}$.



Scheme 18

2.10.2.5 Electrophilic Attack on Sulfur

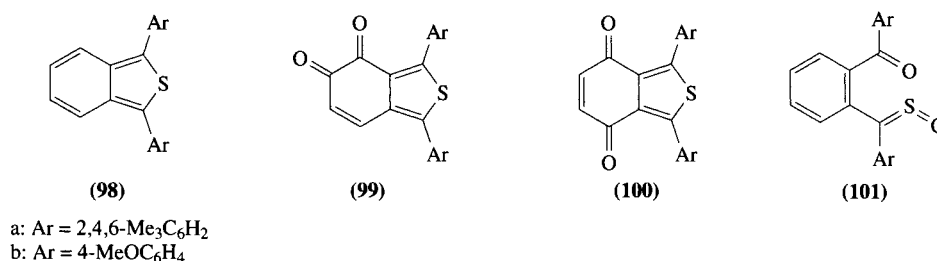
2.10.2.5.1 Formation of thiophenium ions

The alkylation of thiophene and benzo[*b*]thiophene to form 1-alkylthiophenium and 1-alkylbenzo[*b*]thiophenium salts was discussed in CHEC-I <84CHEC-1(4)765>. 1-Methylbenzo[*b*]thiophenium fluoborate has been obtained also by methyl transfer from *O*-methyl dibenzofuranium

fluoroborate to benzo[*b*]thiophene (92T4005). *S*-Arylation of benzo[*b*]thiophene has been achieved using diaryliodonium salts (93CL1703). Thus, heating benzo[*b*]thiophene with diphenyliodonium triflate in the presence of a catalytic amount of copper(II) acetate at 140 °C for 30 min, gave 1-phenylbenzo[*b*]thiophenium triflate in 87% yield. An x-ray crystallographic study shows that the bonds around sulfur are pyramidal, suggesting that the sulfur is in an sp^3 hybridized state. The method has been extended to the preparation of several analogues.

2.10.2.5.2 Oxidation to sulfoxides and sulfones

The oxidation of thiophenes and benzo[*b*]thiophenes to the corresponding 1,1-dioxides by MCPBA has been discussed in CHEC-I (84CHEC-I(4)741). It has been found that oxidation of 2,5-disilylated thiophenes with MCPBA gives the 2,5-disilylated thiophene-1,1-dioxides which are stable towards dimerization (92H(34)1085). Benzo[*c*]thiophene 2-oxide or 2,2-dioxide could not be isolated from a similar MCPBA oxidation of 1,3-diarylbenzo[*c*]thiophene. Instead, the quinones (99) and (100) were formed from the hindered (98a) whereas (98b) gave the hetero-ring-opened product (101) in 54% yield (90CB2065).



Dimethyldioxirane has been found to be an excellent reagent for oxidizing thiophenes to thiophene-1,1-dioxides (90TL5955). The oxidation proceeds at room temperature under neutral conditions. The yields with 2,5-disubstituted thiophenes are excellent; even sterically hindered thiophenophanes lead to the dioxides in very good yields. However, if only one of the thiophene α -positions is substituted, the resultant dioxide could not be isolated, since it underwent rapid Diels–Alder dimerization. The reagent was superior to MCPBA even for thiophenes deactivated by the presence of electron-withdrawing groups. Thus 2-acetyl-5-methylthiophene gave a 53% yield of the dioxide with dimethyldioxirane whereas MCPBA was reported to be ineffective in this case.

(Hexamethylphosphoramide)oxodiperoxomolybdenum(VI), MoO₅·HMPA, is also capable of oxidizing thiophenes and benzo[*b*]thiophene to their 1,1-dioxides (85CPB5071). 2,5-Diphenylthiophene gives a 37% yield of the 1,1-dioxide, while benzo[*b*]thiophene gives the dioxide in 86% yield.

2.10.2.6 Nucleophilic Attack on Ring Atoms

Attack by nucleophiles on the carbon atoms as well as on sulfur of the thiophene will be dealt with in this section.

2.10.2.6.1 Ring-opening reactions

The desulfurative opening of thiophene and benzo[*b*]thiophene rings by Grignard reagents in the presence of bis(triphenylphosphine)nickel dichloride to form butadienes has been mentioned in CHEC-I (84CHEC-I(4)741). This reaction has been further elaborated (84CC617). Methyl-, *n*-butyl- and phenylmagnesium bromides have been used in this study. The substrates were thiophene, 2-methylthiophene and 2,5-dimethylthiophene. *Z,Z*-Dienes were formed exclusively with methylmagnesium bromide and *n*-butylmagnesium bromide whereas phenylmagnesium bromide tended to give *E,E*-dienes.

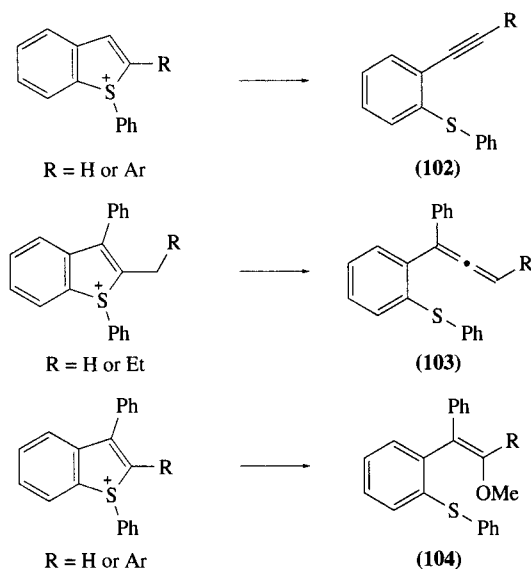
2.10.2.6.2 Addition of nucleophiles across the 2,3-double bond

The addition of amines to the 2,3-double bond of benzo[*b*]thiophene has been referred to in CHEC-I <84CHEC-I(4)741>. A curious case of an intramolecular addition of an aromatic ring (*ortho* to a phenol) in the presence of acid, has also been reported <83JCS(P1)909>.

In the presence of AlCl_3 , aromatic compounds (e.g. toluene) and phenolic ethers (e.g., anisole) add to the 2,3-double bond of benzo[*b*]thiophene <82JCS(P1)815>. In the absence of an external nucleophile, dimerization takes place. Mixtures of 2- and 3-aryl-2,3-dihydrobenzo[*b*]thiophenes result in all these reactions. However, 4-, 5- and 6-hydroxybenzo[*b*]thiophenes gave only the 2-aryl-2,3-dihydrobenzo[*b*]thiophenes in this reaction.

2.10.2.6.3 Reaction of nucleophiles with cationic species

S-Alkylbenzo[*b*]thiophenium ions react with nucleophilic substrates by alkylating them <89AHC(45)151>. However, *S*-arylbenzo[*b*]thiophenium ions react quite differently. Although there is no reaction with CN^- or SCN^- , these compounds undergo ring opening by cleavage of the $\text{S}-\text{C}_2$ bond when treated with NaOMe in MeOH <92CL1357>. The actual product formed depends on the substitution pattern on the benzo[*b*]thiophene. With 3-unsubstituted compounds, the primary process is abstraction of the proton attached to C-3; subsequent cleavage of the $\text{S}-\text{C}_2$ bond results in the formation of acetylenes (**102**) in quantitative yield (Scheme 19). If position 3 is blocked by a phenyl group, the reaction takes a different course depending on the substituent at position 2. With a 2-alkyl substituent (Me or Pr^n), side-chain deprotonation and ring opening lead to the allenenes (**103**) in excellent yields. On the other hand, if position 2 is unsubstituted, or if an aryl group is located there (no acidic proton on the α -carbon of the side chain) reaction with methoxide leads to 1-methoxy-2-phenyl-2 [*o*-(phenylthio)phenyl]ethenes (**104**).

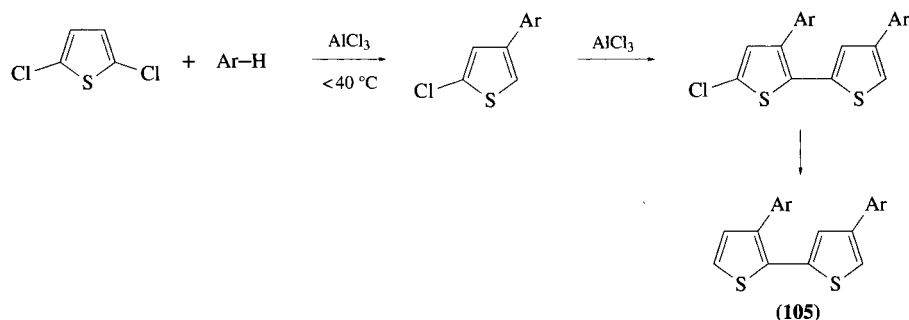


Scheme 19

Ionic hydrogenation has been referred to in CHEC-I <84CHEC-I(4)741>. Logically, this has to find a place in this section, since it represents reaction of a thiophenium ion with a hydride ion derived from triethylsilane. This has been reviewed <85HC(44/1)457>. Reduction of 5-hydroxybenzo[*b*]thiophene with triethylsilane in TFA has given the 2,3-dihydroderivative <91SC959>.

In the presence of AlCl_3 , 2-chlorothiophene reacts with active aromatic compounds to form 2-arylthiophenes <86BCJ83>. The reaction is carried out between 0°C and 40°C . The probable mechanism involves protonation of the thiophene followed by nucleophilic attack by the active aromatic species. Surprisingly, under the same conditions, 2,5-dichlorothiophene reacts with various aromatic compounds regioselectively to form 4-aryl-2-chlorothiophenes in good yields <88BCJ3779>. On treat-

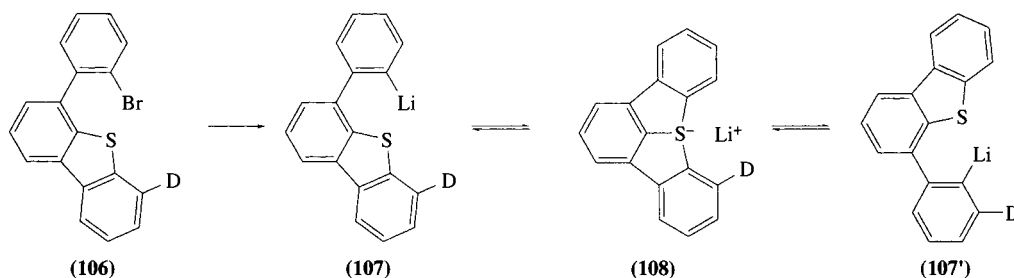
ment with an equimolar quantity of AlCl_3 , these 4-aryl-2-chlorothiophenes undergo a self-Friedel-Crafts arylation to form the bithienyl derivative which can be dehalogenated to **(105)** (Scheme 20) <91BCJ864>.



Scheme 20

2.10.2.6.4 Nucleophilic attack on sulfur

Convincing evidence has been presented for the first time for the intermediacy of an organosulfur ate complex in which the sulfur is linked only to three carbon atoms <92JA7937>. The strategy adopted was to synthesize a rigid molecule with ideal geometry favoring formation of the ate complex. The deuterium-labeled bromo compound **(106)** was converted to the lithio derivative **(107)**. This resulted in equilibration of the isotope label in less than 1 min at -78°C in THF. This means that **(107)** must have been converted to **(107')** via the ate complex **(108)** (Scheme 21). This Li/S exchange at 0°C was found to be 10^9 times faster than the intermolecular Li/S exchange of Ph_2S with PhLi .



Scheme 21

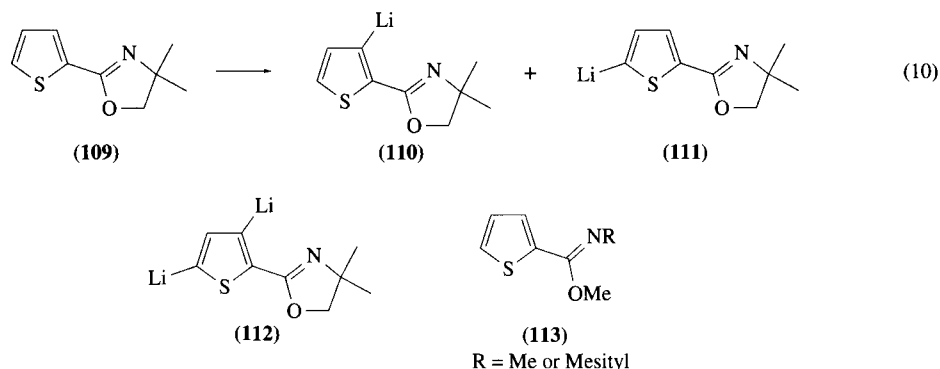
2.10.2.7 Nucleophilic Attack on Hydrogen Attached to Carbon

Deprotonation of neutral species has been dealt with in detail in CHEC-I. In the last few years, the major effort in this area has been towards identification of substituents which would specifically direct metalation to the neighboring CH on the thiophene nucleus. An exhaustive review on directed *ortho*-metalation <90CRV879> has also referred to the use of this strategy in thiophene chemistry.

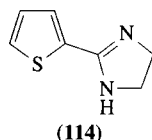
Several different substituents located at position 2 of thiophene have shown the ability, fully or partially, to direct metalation to position 3: oxazoline, imidazoline, secondary carboxamide, carboxylate, sulfonamide and α -aminoalkoxide. The ultimate objective is to secure a high-yielding, regioselective route for the synthesis of 2,3-disubstituted thiophenes.

A detailed paper has been published on the directed lithiation of 2-oxazolinythiophene <85JCS(P1)173>. The highlights are as follows: the highest selectivity for β -lithiation of **(109)** (formation of **(110)**) is attained with $n\text{-BuLi}$ in hexane at -78°C . Regioselectivity is decreased on addition of TMEDA. Highest selectivity for 5-lithiation (formation of **(111)**) is obtained with LDA in THF (Equation (10)). The advantage of using the oxazoline group is that it can be easily transformed into a carboxyl under mild conditions (NaOCl , H_2O , $\text{Bu}_4\text{N}^+\text{HSO}_4^-$, 12 h, 20°C ; KOH ,

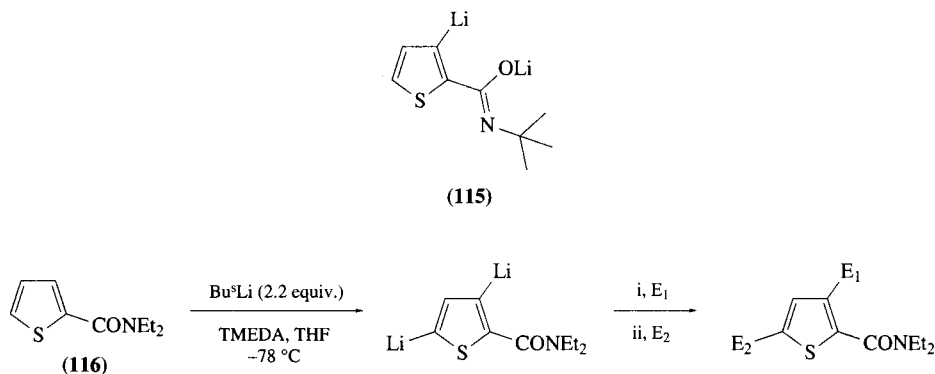
H₂O, 5 h, 20°C). Use of 3.3 equiv. of *sec*-butyllithium has been shown to give the dilithiated compound (**112**), thus making it possible to synthesize 3,5-disubstituted thiophene-2-carboxylic acids <85TL5335>. Surprisingly, the acyclic imidates (**113**) do not show any C-3 directing effect on lithiation <94T4149>. A wide variety of conditions has been tried, (*n*-BuLi, *sec*-BuLi, LDA, THF, DME, hexane, Et₂O, TMEDA), but in all cases, virtually exclusive C-5 lithiation has been shown to occur. This is in contrast to the behavior of the cyclic imide (**109**).



As a natural extension of the oxazoline study, Chadwick and Ennis have also investigated the behavior of the analogous imidazoline (**114**) <91T9901>. Treatment of this with 2.5 equiv. of *n*-BuLi in THF at -78°C for 2 h gave a regiospecific (98%) lithiation at position 3; this result was not affected by the addition of TMEDA. In contrast to this, treatment of (**114**) with LDA (2.2 equiv.) in THF at -78°C led only to the 5-lithio derivative (78%).



Carboxamides as 2-substituents on the thiophene ring have been investigated for their β -directing effect on metalation. The tertiary carboxamide (CONEt₂) was totally disappointing in this regard. However, the secondary carboxamide (CONHCMe₃) has turned out to be an excellent 2-substituent for directing metalation towards position 3. Using 2.2 equiv. of *n*-BuLi in DME at -10°C or in THF at -78°C , the dianion (**115**) could be generated almost quantitatively. This reacted with a variety of electrophiles to give 3-substituted-2-thiophenecarboxamides <85JOC4362>. The only drawback in this scheme appears to be the difficulty in the hydrolysis of the secondary amide. The tertiary amide (**116**) can be used for generating the 3,5-dilithio species which can then be reacted with electrophiles at both locations; the 3-lithio is the first one to react (Scheme 22) <85TL1149>.

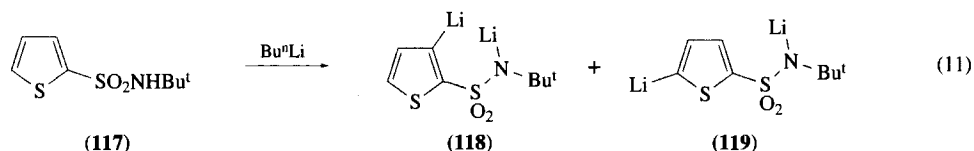


Scheme 22

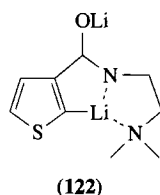
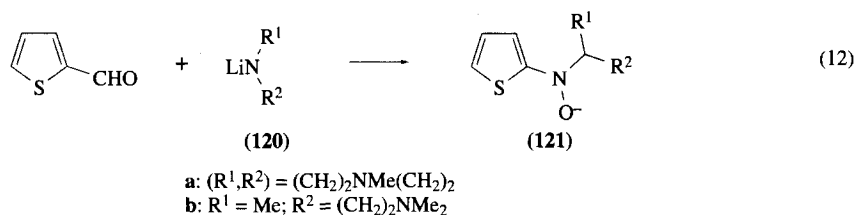
The carboxylate anion itself can function as a β -directing group towards metalation if it is attached to position 2 of the thiophene ring <85TL1777>. However, this effect is dependent on the reagent employed for lithiation. Treatment of thiophene-2-carboxylic acid with 2.2 equiv. of *n*-BuLi

in THF at -78°C , followed by quenching with CD_3OD leads to 3-deuteriothiophene-2-carboxylic acid (93%), with only 5% of the 5-deuterio isomer. However, the use of LDA in THF at -78°C gives only the 5-lithiothiophene-2-carboxylate.

The secondary sulfonamido group ($-\text{SO}_2\text{NHR}$) appears to have a partial *ortho*-directing effect on lithiation. Thus treatment of *N*-*tert*-butylthiophene-2-sulfonamide (**117**) with *n*-BuLi gives a mixture of the two dianions (**118**) and (**119**) (Equation (11)). Under kinetically controlled conditions, the ratio (**118**):(**119**) is 2.7 : 1, but on equilibration, (**119**) precipitates out from the mixture, thereby driving the equilibrium towards the 5-lithio isomer <91JOC4260>. The directing effect of the anionic sulfonamide does not appear to be as pronounced as that of the anionic carboxamide.



α -Aminoalkoxides formed *in situ* by the addition of lithium dialkylamides to thiophenecarbaldehydes have been studied as *ortho*-directing groups in lithiation <87JOC104>. The effect is strongly dependent on the amine. Thus the adduct (**121a**) of thiophene-2-carbaldehyde and lithium *N*-methylpiperazide (**120a**) gives only 5-methyl thiophene-2-carbaldehyde on lithiation and quenching with methyl iodide. But the amide (**120b**) of *N,N,N'*-trimethylethylenediamine leads to the 5-methyl- and 3-methyl thiophene-2-carbaldehydes in the ratio 1 : 2 (Equation (12)). A similar difference is observed when the substrate is thiophene-3-carbaldehyde. Compound (**120a**) leads mainly to 5-methylation, while (**120b**) gives exclusively 2-methylthiophene-3-carbaldehyde. This can be explained as being due to the formation of the intermediate (**122**).

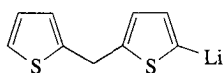


3-Aminothiophene and 3-aminobenzo[*b*]thiophene, in which the amino group is protected by either the trifluoroacetyl group or by the *t*-butoxycarbonyl group, have been shown to give the *N,C*-2 dianions on treatment with 3.2 equiv. of *n*-BuLi at -20°C <92JOC2184>.

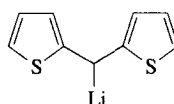
A few other thiophene lithiations of interest have been reported. 2-Iodo-5-lithiothiophene has been prepared in good yield and reacted with several electrophiles <90SL755>. The key to success was the use of LDA in THF at -40°C . 2,5-Dilithiation of thiophene with *n*-BuLi and TMEDA in hexane <77JCS(P1)887> followed by reaction with DMF gives thiophene-2,5-dicarbaldehyde <88S316>.

The regiochemistry of lithiation of di(2-thienyl)methane has been clarified <93TL8143>. Under kinetically controlled conditions (*n*-BuLi, Et_2O or THF, $<0^{\circ}\text{C}$), C-5 metalation takes place (**123**). However, the methylene protons are more acidic than the C-5 protons, with DMSO ($\text{p}K_{\text{a}}-35$) being intermediate in acidity. Hence dimsyl anion effects metalation at the central connecting carbon. Thus treatment of di(2-thienyl)methane with NaH or *n*-BuLi in DMSO-THF (1 : 1) cleanly generates the anion (**124**). LDA in THF leads to the same result, apparently because the liberated diisopropylamine can mediate the equilibrium between (**123**) and (**124**), in favor of the thermodynamically more stable (**124**).

The lithiation of several methylthiophene carboxylic acids has been referred to in CHEC-I. As a further example in this series, the lithiation of 2-methylthiophene-3-carboxylic acid by means of

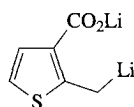


(123)

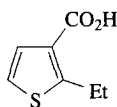


(124)

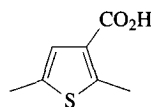
LDA (THF, -30°C) has been shown to give the dilithium salt (**125**) regioselectively $\langle 89\text{BCJ}2725 \rangle$. However, the regioselectivity decreased when *n*-BuLi was used for lithiation; a 1 : 1 mixture of (**126**) and (**127**) is obtained on methylation.



(125)



(126)



(127)

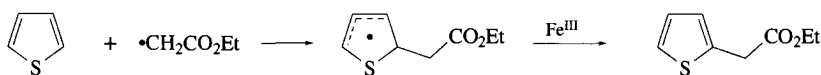
2.10.2.8 Reactions with Radicals and Electron-deficient Species

2.10.2.8.1 Homolytic substitution

The homolytic substitution of thiophene has been reviewed exhaustively by Porter $\langle 85\text{HC}(44/1)651 \rangle$. The subject has also been extensively surveyed in CHEC-I $\langle 84\text{CHEC-I}(4)741 \rangle$.

Kinetic EPR spectroscopy has been used for determining absolute rate constants for addition of diethoxyphosphonyl, phenyl and triethylsilyl radicals to thiophene $\langle 83\text{JOC}3704 \rangle$. The data show that the reactivity of thiophene and furan towards these radicals is intermediate between those of simple alkenes and benzene.

The homolytic substitution of thiophene by electrophilic carbon radicals has been developed into a good general method for the synthesis of (2-thienyl)acetic and (2-thienyl)propionic acids. The electrophilic radical, $\cdot\text{CH}_2\text{CO}_2\text{Et}$, generated from $\text{ICH}_2\text{CO}_2\text{Et}$, H_2O_2 and catalytic Fe^{2+} in DMSO, reacts with thiophene to form (2-thienyl)acetic ester in 62% yield $\langle 92\text{JOC}6817 \rangle$. The suggested mechanism is shown in Scheme 23. The success of the reaction is ascribed to the fact that Fe^{3+} is a weak oxidant and can oxidize the σ -radical to the aromatic product. Another interesting way of generating the required electrophilic carbon radical is by making use of the known autoxidation of triethylborane, leading to the formation of ethyl radical *in situ*; reaction of this with α -iodoacetic ester or α -bromopropionic ester gives the required electrophilic radical. Thus, when BEt_3 in hexane is added at room temperature to thiophene, ferric sulfate and the halo compound in DMSO, with free access to atmospheric oxygen, the 2-substituted thiophene is produced in about 50% yield $\langle 93\text{TL}5015 \rangle$. The role of the Fe^{3+} is again to oxidize the initially formed σ -radical.

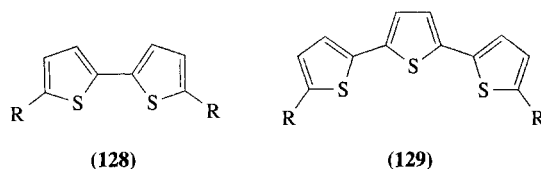


Scheme 23

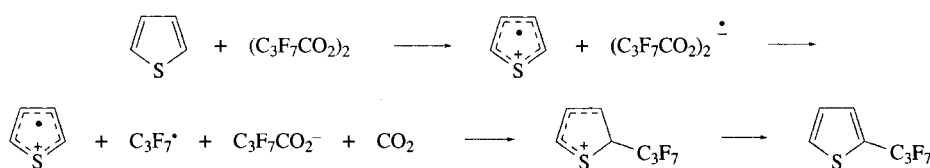
Oxidatively generated electrophilic malonyl radicals react regioselectively with thiophene to form (2-thienyl)malonic ester $\langle 88\text{H}(27)2627 \rangle$. Treatment of thiophene with cerium(IV) sulfate and an excess of dimethyl malonate in methanol at room temperature yields 2-thienylmalonic ester in 85% yield. In this case, the initially formed σ -radical is oxidized by the cerium(IV) sulfate. 2-Methylthiophene similarly gives the 5-malonyl-substituted product. Benzo[*b*]thiophene leads to the 2-malonyl-benzo[*b*]thiophene in 41% yield. Monosubstituted malonates could also be used in this reaction. Thiophene and 2-acetylthiophene react with triethyl methanetricarboxylate in the presence of Mn(III) to form the corresponding thienylmethane tricarboxylic esters. Yields are about 55% $\langle 91\text{S}567 \rangle$.

The reaction of thiophene with benzyl bromide in the presence of $\text{RuCl}_3 \cdot x\text{H}_2\text{O}$ (140°C , 24 h) gives a 10 : 1 mixture of 2-benzyl and 3-benzylthiophene (ca. 40% yield), along with dibenzyl (7%) $\langle 88\text{ICA}(145)179 \rangle$. Similarly, benzo[*b*]thiophene is benzylated to a mixture of 2-benzyl and

3-benzylbenzo[*b*]thiophene (42%). Substituted benzyl halides can also be used in this reaction. The formation of dibenzyl as a by-product suggests that this is a homolytic substitution on thiophene by benzyl radicals. Earlier, the reaction of thiophene with several alcohols (ROH) at 140°C in the presence of $\text{RuCl}_3 \cdot x\text{CH}_2\text{O}$ had been studied <86CC1255>. The products included the dimer (128) and the trimer (129).



Bis(heptafluorobutyryl)peroxide has been used for the introduction of the heptafluoropropyl group into thiophenes <86BCJ215, 89BCJ2416>. The reaction (Freon-113, 40°C, 3 h) was found to be regioselective, giving 2-(heptafluoropropyl)thiophene in 98% yield. The reaction appears to proceed by one-electron transfer from thiophene to the reagent, as shown in Scheme 24. The orienting effects of various thiophene substituents on the heptafluoropropylation have been investigated <88BCJ3549>. 2-Methyl- and 2-bromothiophene lead to 5-heptafluoropropyl derivatives, while 3-bromothiophene gives a mixture of 2- and 5-heptafluoropropylated products. These results are reminiscent of electrophilic substitutions. But there are also some significant deviations. Thus with 2-formylthiophene, a mixture of 3- and 5-heptafluoropropyl derivatives (1 : 1) is formed, but no substitution at position 4 is observed. In the case of benzo[*b*]thiophene, the fluoroalkyl group enters positions 2 and 7 but not at 3. These observations appear to confirm the electron-transfer mechanism proposed. Bis(chlorodifluoroacetyl) peroxide reacts similarly with thiophene to give 2-(chlorodifluoromethyl)thiophene exclusively in 68% yield <92CL227>.

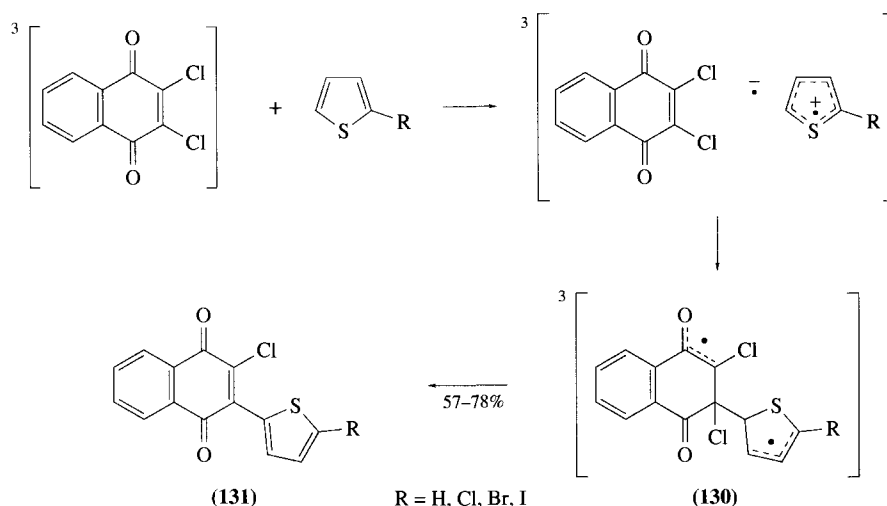


Scheme 24

Another interesting method for perfluoroalkylation of thiophenes is through the use of a perfluoroalkanesulfonyl chloride in conjunction with a ruthenium(II) phosphine complex <94JCS(P1)1339>. Thus the reaction of tridecafluorohexanesulfonyl chloride with thiophene at 120°C in the presence of $\text{RuCl}_2(\text{PPh}_3)_3$ gives 2-(tridecafluorohexyl)thiophene in 77% yield. The reaction apparently proceeds by initial one-electron transfer from the Ru^{II} to the sulfonyl chloride; the anion radical thus formed cleaves homolytically to give perfluoroalkanesulfonyl radical which loses SO_2 to generate the perfluoroalkyl radical. Addition of this to thiophene is followed by hydrogen-atom abstraction by $\text{Ru}^{\text{III}}\text{Cl}$ species to yield the product with regeneration of the Ru^{II} catalyst. Several substituted thiophenes have also been subjected to this reaction. When the substituent (Me, Me_3Si , Br, Cl, CHO) is at position 2, the perfluoroalkylation takes place at position 5. When the substituent (Me, Br) is at position 3, the perfluoroalkylation takes place at positions 2 and 5.

Several photoinduced substitution reactions of thiophene have been disclosed recently. A few early examples of this type of reaction had been mentioned in CHEC-I <84CHEC-I(4)741>. All these reactions appear to be initiated by single-electron transfer. The photoreaction of 2,3-dichloro-1,4-naphthoquinone with thiophenes gives 2-chloro-3-(2-thienyl)-1,4-naphthoquinones (131). The success of the reaction appears to depend on the oxidation potential of the thiophene <87BCJ1847>. The reaction may be initiated by one-electron transfer from ground-state thiophene to the triplet excited state of the quinone to give an ion-radical pair, which could combine to form (130) (Scheme 25).

A similar charge-transfer complex between ground-state thiophene and an excited 3-bromocoumarin has been postulated to be an intermediate in the photocoupling of the two <90S719>.

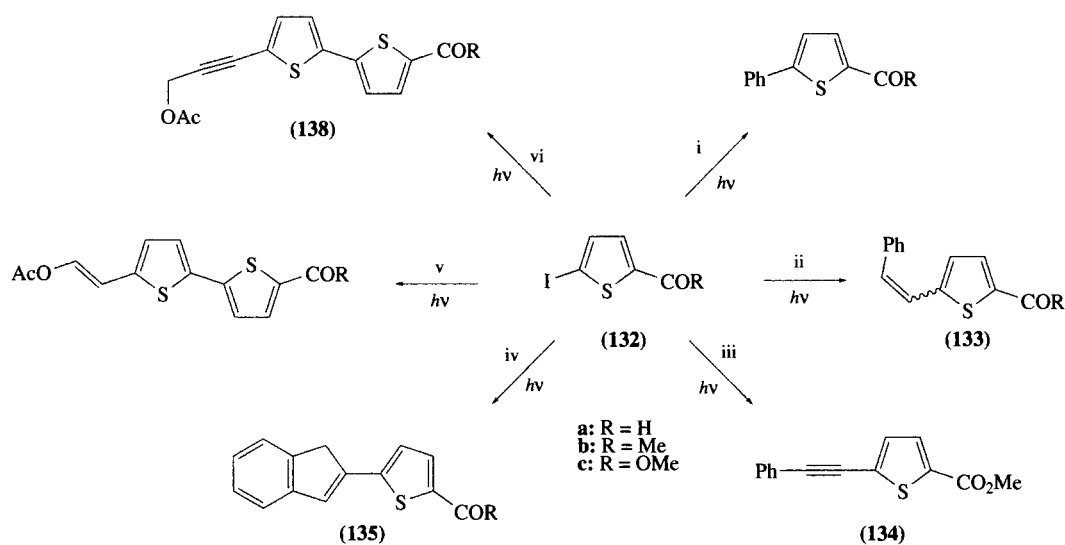


Scheme 25

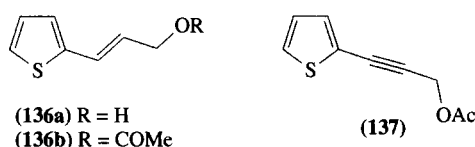
(2-Thienyl)pyridines and (2-thienyl)quinolines have been synthesized by high-pressure mercury lamp irradiation of thiophene with the respective iodothyridine or iodoquinoline $\langle 90\text{H}(30)957 \rangle$. The yields are moderate. Several photosubstitution reactions initiated by single-electron transfer, in which the thiophene functions as an *acceptor*, have recently been reported. Irradiation of 5-iodothiophene-2-carbaldehyde (**132a**), 2-acetyl-5-iodothiophene (**132b**) or methyl 5-iodothiophene-2-carboxylate (**132c**) in benzene leads to phenylation at position 5 in excellent yields $\langle 89\text{G}419 \rangle$. Other aromatic heterocycles, e.g. thiophenes or thiazoles, could be used in place of benzene. The arylation always seems to take place at the position conjugated with the carbonyl group in (**132**). 3,5-Diiodothiophene-2-carbaldehyde gives 3,5-diphenylthiophene-2-carbaldehyde in 91% yield. The reaction appears to proceed via a triplet exciplex formed by single-electron transfer from the donor (arene) to the acceptor (**132**). This is thus completely different from the synthesis of phenylthiophene by photolysis of iodothiophene in benzene, which proceeds via a thienyl radical (CHEC-I) $\langle 84\text{CHEC-I}(4)741 \rangle$. The photosubstitution has been extended to the reaction of (**132**) with arylalkenes and arylalkynes (Scheme 26) $\langle 90\text{JOC}4019 \rangle$. Thus (**132a**) and (**132b**) react photolytically with styrene to produce *cis* and *trans* mixtures of (**133a**) and (**133b**). Similar reactions take place with 2-vinylthiophene, 2-vinylfuran and benzo[*b*]furan. The yields are very good. In contrast, irradiation of 2-iodothiophene in the presence of styrene gives only polymeric material. Since 2-iodothiophene is known to generate the thienyl radical under these conditions, this suggests that in the photosubstitution of (**132**) the mechanism is different. Evidence has been presented for a single-electron transfer as the first step. The solvent separated ion pair can undergo radical coupling with subsequent elimination of HI. The ester (**132c**) on irradiation with phenylacetylene gives (**134**). Similarly, photosubstitution of (**132a**) or (**132b**) with indene leads to (**135**) in 75% yield $\langle 89\text{H}(29)1331 \rangle$. The scope of such photosubstitution reactions has been delineated $\langle 94\text{JCS}(P1)1245 \rangle$. The thiophenes (**132**) do not couple with nitrobenzene, benzonitrile, methyl benzoate, the allylic alcohol (**136a**) or thienylmethanol. However, the acetate (**136b**) did couple, but with altered regioselectivity; the substitution took place on the hetero-ring and not on the alkene (compare styrene or 2-vinylthiophene). Similar coupling of (**132**) with 2-(3-acetoxyprop-1-ynyl)thiophene (**137**) gave (**138**). The acetylene coupling has been used for the synthesis of a few naturally occurring thienylacetylenes $\langle 92\text{T}9315 \rangle$. Photolysis of (**132**) in the presence of benzimidazole leads to coupling at position 4 of the latter $\langle 91\text{H}(32)1059 \rangle$.

2.10.2.8.2 Reaction with carbenes

In the first edition (CHEC-I) $\langle 84\text{CHEC-I}(4)741 \rangle$ it was mentioned that three types of products were identified in the reaction of thiophenes with carbenes generated from diazo compounds, cyclopropanes, *S,C*-ylides, and 2-substituted thiophenes, corresponding to carbene attack at the 2,3-double bond, ring sulfur or at C-2, respectively. However, it now appears very likely that the

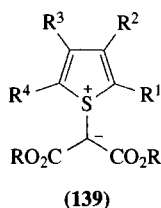


Scheme 26



initial attack by singlet carbene or carbenoid on thiophene *always* takes place on the ring sulfur to produce thiophene S,C-ylides; these can undergo a variety of transformations to produce at least six types of products. An authoritative review on this has been written by Porter <89AHC(45)151>.

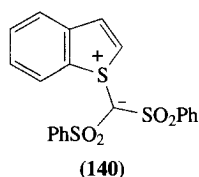
(i) Stable ylides: Thiophenium bis(alkoxycarbonyl)methylides (139) are obtained in high yield by rhodium(II) carboxylate-catalyzed reaction of diazomalonate esters with thiophene derivatives <88JCS(P1)1023>. But other α -diazocarbonyl compounds do not, in general, give rise to stable ylides. An exception is the reaction of 2,5-dichlorothiophenes with α -diazocarbonyl compounds such as those from dimedone, Meldrum's acid, etc. in the presence of rhodium(II) catalyst <89JOC4822>; this leads to stable thiophenium S,C-ylides.



- a: $\text{R}^1 = \text{R}^2 = \text{R}^3 = \text{R}^4 = \text{Cl}$
 b: $\text{R}^1 = \text{R}^4 = \text{Cl}$; $\text{R}^2 = \text{R}^3 = \text{H}$
 c: $\text{R}^1 = \text{R}^2 = \text{R}^3 = \text{R}^4 = \text{H}$
 d: $\text{R}^1 = \text{R}^4 = \text{Me}$; $\text{R}^2 = \text{R}^3 = \text{H}$

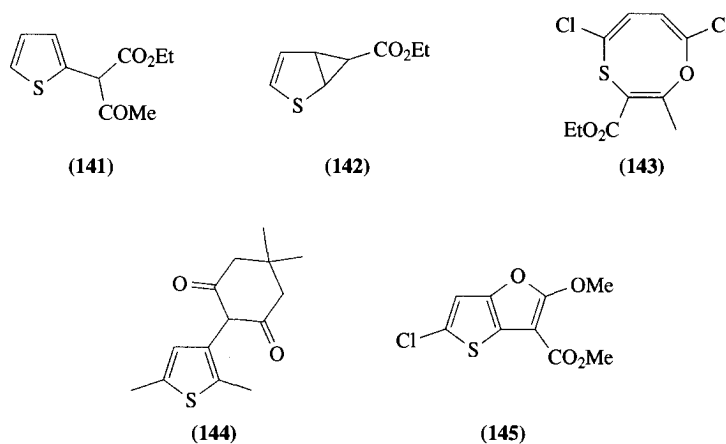
Likewise, several stable S,C-ylides derived from benzo[b]thiophene and some 2,3-disubstituted benzo[b]thiophenes have been reported recently <91T8611> by rhodium(II) acetate-catalyzed reaction with β -dicarbonyl diazo compounds. Ylides (e.g. (140)) from benzo[b]thiophene and dibenzo-thiophene have also been reported by *trans*-ylidation using phenyliodonium bis(phenylsulfonyl)-methylide <88JHC1599>.

Detailed NMR studies have been carried out to determine the rotational barrier around the S—C ylide bond in the thiophene and benzo[b]thiophene series <89AHC(45)151, 91T8611>. The x-ray crystal structure reveals that the sulfur is pyramidal.



The fascinating rearrangements occurring on thermal decomposition of these ylides are discussed in section 2.10.3.3.

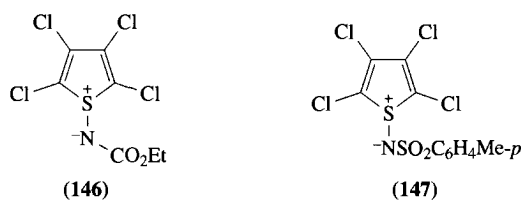
(ii) Unstable S,C-ylides: Structure of isolable products. As mentioned earlier, carbene attack seems always to take place on the ring sulfur of thiophenes. In those cases where the resultant ylides are not stable enough to be isolated, a variety of other products have been identified. The entire reaction manifold has been neatly pictured in Porter's review <89AHC(45)151>. Representative examples are (141) to (145). The detailed pathways to these products will be discussed in Section 2.10.3.3.



2.10.2.8.3 Reaction with nitrenes

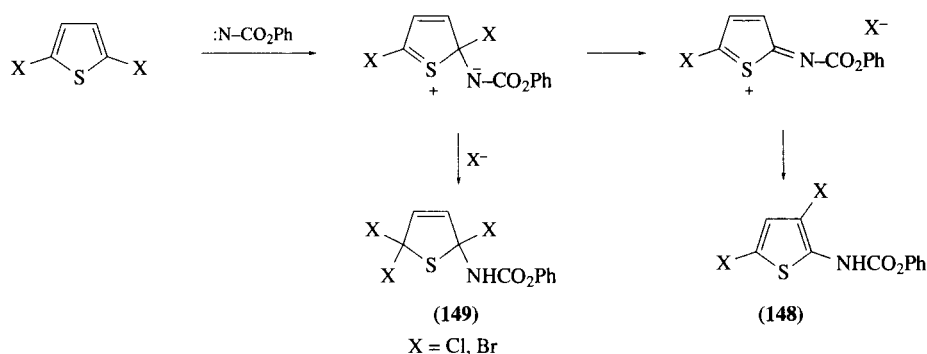
In CHEC-I the statement was made that "nitrene attacks only the carbon and never the sulfur atom of thiophene" <84CHEC-I(4)741>. The only exception to this was the presumed formation of an S,N-ylide leading to thermal fragmentation of the thiophene ring. However, since 1984, Meth-Cohn has prepared and characterized several S,N-ylides formed by the attack of a nitrene on the ring sulfur atom of thiophene. This has been covered in a recent review <89AHC(45)151>.

It had been shown earlier that ethoxycarbonylnitrene attacks thiophene and its simple alkyl derivatives at the α -position; subsequent ring opening and reclosure with loss of sulfur leads to pyrroles. However, with polyhalogenothiophenes, the reaction takes a different course. Thermal generation of the nitrene by decomposition in tetrachlorothiophene solution at 130°C leads to the S,N-ylide (146) in 44% yield <84CC190>. The ^{13}C NMR spectrum of this product shows only two thiophenic carbon signals at 131.1 ppm and 131.4 ppm. Structure (146) is confirmed by the fact that on hydrogenolysis (Raney Ni), tetrachlorothiophene and ethyl carbamate are produced. The S—N bond can also be cleaved by photolysis, leading to the nitrene; thus photolysis in cyclohexane leads to the isolation of ethyl cyclohexylcarbamate and tetrachlorothiophene in greater than 80% yield. The ylide (147) could be made similarly, although in lower yield (24%) from tetrachlorothiophene and *p*-tolylsulfonyl azide.



It has subsequently been shown $\langle 86\text{TL}1105 \rangle$ that **(146)** and **(147)** are unique only in regard to their stability and not with respect to attack of nitrene at the sulfur atom. In a reinvestigation of the reaction of ethyl azidoformate with several thiophenes it has been shown that S,N-ylides are indeed formed as transient products in every case; these could be trapped by cycloaddition with acenaphthylene (see Section 2.10.3.3) leading to fluoranthene derivatives.

However, the instability of the S,N-ylides except in the case of tetrachlorothiophene may lead to the formation of other observed products. Thus, when 2,5-dichloro or dibromothiophene was heated at 145°C with phenyl azidoformate, two products, **(148)** and **(149)**, were obtained in about 10–15% yield; the postulated mechanism for the formation of these is shown in Scheme 27 $\langle 86\text{JCS(P1)233} \rangle$. The intriguing suggestion has been made that for the reaction of nitrenes with thiophenes, S-attack is the rule rather than an exception and that all the observed products could be the result of further transformations $\langle 86\text{TL}1105 \rangle$. This is discussed in Section 2.10.3.3.



Scheme 27

An S,N-ylide from dibenzothiophene has been prepared by the modified chloramine-T method and its conformation in the solid state as well as in solution investigated $\langle 91\text{JOC}3259 \rangle$.

2.10.2.8.4 Electrochemical reactions

This section will discuss both electrochemical reductions and electrochemical oxidations of the thiophene and benzo[*b*]thiophene systems.

(i) Electrochemical reductions

Electrochemical reduction can offer an attractive alternative to Birch reduction. The advantages could be: reduction in aqueous or mixed organic–aqueous media, possibility of isolating products not expected to be stable to metal ammonia conditions, ability to deal with base-sensitive substrates or products, etc. Reduction of benzo[*b*]thiophene in an electrolyte containing tetrabutylammonium hydroxide in water, using a mercury cathode and a platinum anode gave 2,3-dihydrobenzo[*b*]thiophene and the ring-cleaved 2-ethylbenzenethiol $\langle 86\text{JOC}3468 \rangle$. The electrochemical reduction of thiophene-2-carboxylic acid has already been referred to in CHEC-I.

(ii) Electrochemical oxidations

The electrochemical oxidation of thiophene and its derivatives forms part of a recent exhaustive review of such oxidations of several heteroaromatic compounds $\langle 86\text{CJC}76 \rangle$. The mechanism has been explained. The initially formed radical cation is sufficiently stable to dimerize, but not so stable as to diffuse away from the electrode surface. Chain propagation takes place by coupling of the monomer radical cation with the dimer radical cation and so on. Cyclic voltammetric data for thiophene and its derivatives have been provided.

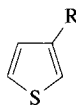
In CHEC-I, the capture of the cation radical generated from 2,5-dimethylthiophene by a nucleophile such as cyanide or solvent methanol, was discussed. Similarly, it has been found that the electrochemical oxidation of benzo[*b*]thiophene generates a cation radical. This can be captured by

nucleophiles such as cyanide ion; this results in substitution of CN at a nucleophilic center of the starting material <92JCS(P1)333>.

Electrochemical oxidation is mainly useful for the preparation of thiophene polymers <92CRV711>. The oxidative coupling polymerization of thiophenes can be carried out by using either chemical (e.g. FeCl_3) or electrochemical methods. The products are mainly α,α -coupled thiophenes <91MM678> (see Section 2.10.4.7.3). The electrogeneration of conducting polythiophene derivatives offers several advantages: direct grafting on to electrode surface, preparation of electrodes of large surface-to-volume ratio, etc.

Polythiophenes obtained via electrochemical polymerization of thiophene, 2,2'-bithienyl and 2,2':5',2''-terthienyl have differing electrical properties; this may be due to the presence of varying amounts of α,β -coupling in the polymers. It is expected that the proportion of α,α -coupling would increase in the polymers from thiophene, 2,2'-bithienyl and 2,2':5',2''-terthienyl. 1,4-Dithienyl and 1,3-dithienylbenzene have also been electropolymerized under low current density <89TL3461>. Electrochemical oxidation of symmetrical dimethylbithiophenes with free α -positions has given electroactive polymers. α,α -Dimethylbithiophene could not be polymerized under these conditions, indicating the importance of free α -positions in this process; however, some α,β -coupling in thiophene polymerization cannot be ruled out <87CC1476>. It has been found that a small amount of 2,2'-bithiophene greatly facilitates the electrochemical polymerization of thiophene monomers. The reason for this is that the 2,2'-bithiophene, by virtue of its lower oxidation potential, is oxidized first to form a radical cation. The neutral thiophene monomer is subjected to an electrophilic attack by this radical cation. The bithiophene thus acts as a polymerization initiator <93CC1160>. In 2,5''-dimethylterthiophene both the terminal α -positions are blocked. The reversible one-electron electrochemical oxidation of this has been studied <92JA2728>. From the spectral data and ESR measurements, it has been hypothesized that the initially formed cation radical forms a π -dimer.

The properties of polythiophenes can be fine-tuned by introducing appropriate substituents in the monomers. Thus electropolymerization of the monomer (**150**) has led to a polythiophene possessing polyether side chains <89CC679>.



- (**150**) $\text{R} = -(\text{CH}_2)_2-\text{O}-(\text{CH}_2)_2-\text{OMe}$
 (**151**) $\text{R} = -\text{C}_6\text{H}_4-(\text{CH}_2)_7-\text{Me}$
 (**152**) $\text{R} = -(\text{CH}_2)_3-\text{O}-\text{CH}_2\text{CH}(\text{Ph})-\text{Et}$
 (**153**) $\text{R} = \text{CH}_2-\text{CH}(\text{Me})-\text{Et}$

The attachment of long flexible side chains to the thiophene rings in polythiophenes renders them soluble in common organic solvents. But the side-chain interaction may also lead to the phenomenon of "thermal undoping" of the doped polymer. In order to get over this, a benzene ring has been interposed between the thiophene and the linear alkyl chain as in (**151**). Polymerization of this was carried out with FeCl_3 as the one-electron oxidant <92MM4297>.

Polythiophenes possessing chiral functional groups have also been synthesized by electro-oxidation. The S and R enantiomers of the monomer (**152**) were first synthesized by conventional methods and then subjected to electropolymerization in a one-compartment, three-electrode cell containing $\text{Bu}_4\text{N}^+\text{PF}_6^-$ in nitrobenzene at 5°C under argon in galvanostatic conditions <88CC658>. Indian workers have similarly electropolymerized the chiral monomer (**153**) (>95% *ee*). The oxidation was performed in dry MeCN in the presence of $\text{Et}_4\text{N}^+\text{BF}_4^-$ under nitrogen <88CC917>.

There is one interesting example where electrochemical oxidation has failed, but chemical oxidation has succeeded in producing the desired polymer. The sodium salt of 3-(3-thienyl)-propanesulfonic acid has been polymerized at room temperature in an aqueous medium, using FeCl_3 as the oxidant. The polymeric material (ferrous salt) is then treated with NaOH to get the sodium salt of the polymer; this can be exchanged for other cations by treatment with ion-exchange resins <90CC1694>.

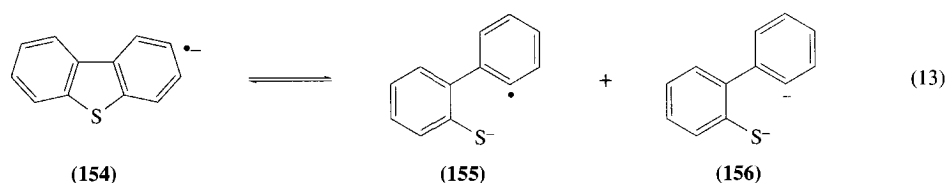
Several benzo[c]thiophenes with substituents on the benzene ring have been electropolymerized recently <94CC825>.

2.10.2.8.5 Reactions with free electrons

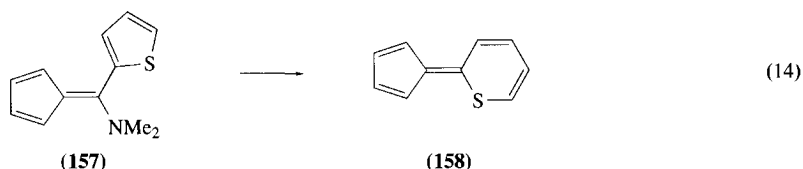
Benzo[b]thiophene dianion has been prepared by reduction of benzo[b]thiophene with sodium metal at -78°C in $[\text{}^2\text{H}_8]\text{THF}$. The ^1H and ^{13}C NMR spectra of the purple solution obtained prove

that it is the dianion and not a radical anion. This is the first example of a sulfur-containing $4n$ polycyclic dianion. The dianion undergoes oxidation to benzo[*b*]thiophene with oxygen $\langle 85CC1033 \rangle$. If lithium metal is used instead of sodium for the reduction, only 2-lithiobenzo[*b*]thiophene is obtained.

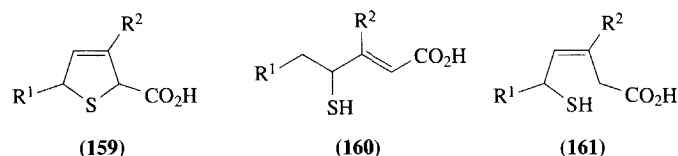
Treatment of dibenzothiophene with potassium metal generates the radical anion (**154**). This dissociates thermally via S—C bond cleavage to the radical anion (**155**). The latter reacts with another molecule of (**154**) to form the dianion (**156**) and dibenzothiophene. Flash photolysis of (**156**) in the presence of excess dibenzothiophene leads to (**154**) (Equation (13)) $\langle 82JCS(P2)1113 \rangle$.



Treatment of 6-dimethylamino-6-(2-thienyl)fulvene (**157**) with lithium naphthalenide (2.2 equiv.) in THF at -78°C , followed by quenching with water gives (**158**) in 15–20% yield (Equation (14)) $\langle 91TL3499 \rangle$. This novel conversion involves a reductive ring opening of the dianion to an allene and recyclization with elimination of dimethylamine.



The reduction of thiophenes with alkali metals in liquid ammonia can lead to 2,5-dihydrothiophenes, ring-opened products or desulfurized products $\langle 85HC(44/1)457 \rangle$. 2-Acylthiophenes lead to 2,5-dihydrothiophene derivatives on treatment with sodium and liquid ammonia in the presence of ethanol $\langle 81CL1341 \rangle$. Full details of the lithium/ammonia reduction of thiophene-2-carboxylic acids have been published, including a complete mechanistic picture $\langle 83JOC3206 \rangle$. Reduction of the lithium salts of the acids, in the absence of a proton donor, leads to 2,5-dihydrothiophene-2-carboxylic acids (**159**) after quenching with NH_4Cl . Reduction of the free carboxylic acid with 3 equiv. of lithium gave a mixture in which (**159**) was the major component; one of the side products in this case was the mercapto acid (**160**). With excess lithium (10 equiv.), a greater proportion of desulfurized products was obtained. On the other hand, reduction with lithium (5 equiv.) and ammonia in the presence of methanol, gives the ring-opened (*Z*)-5-mercapto-3-alkenoic acids (**161**).



The Birch reduction of 2-thienylacetic acid and 2-(3-thienyl) alkanoic acids has been shown to involve regioselective ring opening by cleavage of the S—C₅ bond $\langle 83BCJ1665 \rangle$ leading to β,γ -unsaturated carboxylic acids. Surprisingly, it was found that 2-*n*-propylthiophene also underwent regioselective ring opening in the Birch reduction giving, after benzylation, (*Z*)-4-benzylthio-3-heptene in 72% yield (see also $\langle 85HC(44/1)457 \rangle$).

2.10.2.8.6 Catalytic hydrogenation

An exhaustive review on all aspects of thiophene-ring reduction, including catalytic hydrogenation reduction by dissolving metals and ionic hydrogenation has appeared $\langle 85HC(44/1)457 \rangle$. CHEC-I has also dealt with these subjects in sufficient detail $\langle 84CHEC-I(4)741 \rangle$. The ongoing intense activity in the area of transition metal-catalyzed hydrodesulfurization is covered in Sections 2.10.2.8.8 and 2.10.6.

2.10.2.8.7 Reactions at surfaces

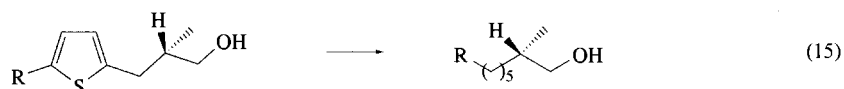
The adsorption of thiophene on ZSM5 zeolites and further surface reactions have been studied by means of IR spectroscopy, mass spectrometry, and gravimetry. Thiophene is weakly hydrogen bonded to the SiOH groups on the zeolite surface. With cation-exchanged zeolites, a strong coordinative type of bonding takes place between the thiophene and Na^+ or K^+ ; the hetero ring appears to be oriented in a plane parallel to the surface of the zeolite. The strength of the interaction is proportionate to the Lewis acid strength of the metal cation. With the strong Bronsted acid sites in HZSM5, the thiophene initially hydrogen bonds to the SiOHAl groups; this is then followed by ring opening and oligomerization. Simultaneously, other acid-catalyzed reactions also take place, leading to coke deposition $\langle 92\text{JPC}2669 \rangle$. A zeolite prepared by exchange of NaY with an ammonia solution containing FeCl_2 and subsequently activated at 500–600°C, was found to be highly active in the hydrodesulfurization of dibenzothiophene $\langle 90\text{BCJ}2745 \rangle$. Apparently, dibenzothiophene is first adsorbed on Fe^{2+} through its sulfur atom, with consequent weakening of the C—S bonds. This results in attack by a proton from the zeolite catalyst on the carbon, with removal of the sulfur.

One-electron oxidation has been observed in di-, tri- and quaterthiophenes loaded on NaZSM5 zeolites. The resulting cation radicals appear to be quite stable within the zeolite channels. On standing at room temperature, or with mild heating, they undergo oligomerization $\langle 92\text{C}359 \rangle$.

2.10.2.8.8 Desulfurization

The subject of Raney nickel desulfurization has been exhaustively dealt with in CHEC-I. A few further interesting applications of this procedure will be presented in this section, along with the use of some nickel(0) complexes and nickel boride for this purpose. The major goal of understanding the mechanism of hydrodesulfurization by studying metal complexes as models is briefly dealt with in Section 2.10.6.

Crown ethers containing thiophene have been smoothly desulfurized by Raney nickel $\langle 89\text{BCJ}838 \rangle$. Chiral nonracemic 2-methyl-1-alkanols have been generated by Raney nickel desulfurization of the thiophene derivatives, which are themselves obtainable by bakers' yeast reduction of the corresponding aldehydes (Equation (15)) $\langle 92\text{JOC}2052 \rangle$. There was a small but significant amount of racemization when the desulfurization was carried out with substrates having a free OH group, but when this was protected by acylation, there was no racemization in the desulfurization step.



Several nickel complexes have been studied as homogeneous catalysts for the desulfurization of thiophene, benzo[b]thiophene and dibenzothiophene. Thus, treatment of dibenzothiophene with two equivalents of (2,2'-bipyridyl)(1,5-cyclooctadiene)nickel(0) in THF results in the cleavage of the S—C bond and formation of nickel-substituted biphenyl derivatives; hydrolytic workup gives biphenyl $\langle 83\text{JOC}2963 \rangle$. Several other nickel complexes have similarly been found useful for desulfurization $\langle 89\text{JOC}4848 \rangle$. An electron-transfer mechanism may be operative. On the other hand, a free-radical mechanism has been suggested for the Raney nickel desulfurization of dibenzothiophene, leading to biphenyl $\langle 89\text{BCJ}557 \rangle$.

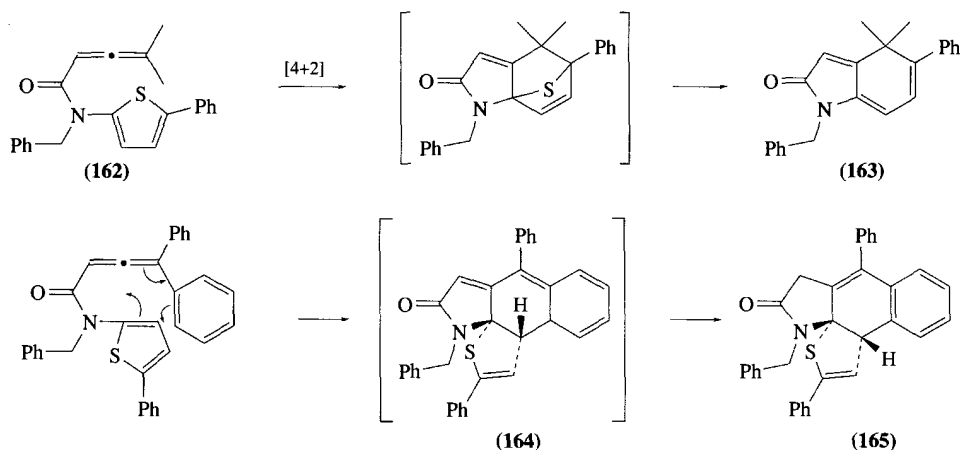
Nickel boride (prepared by adding NaBH_4 to nickel chloride in methanol–THF) has been found to be an efficient, nonpyrophoric reagent for the desulfurization of benzo[b]thiophene and dibenzothiophene $\langle 90\text{CC}819, 92\text{JOC}1986 \rangle$. The reaction proceeds under very mild conditions and is probably mediated by nickel hydride.

2.10.2.9 Reactions involving a Cyclic Transition State with a Second Molecule

In CHEC-I, cycloaddition reactions involving thiophene were further classified according to the number of π electrons of the thiophene ring taking part in the process. Since there are few reactions in which different modes of cycloaddition have been observed with the same reactants, such a classification has been dispensed with in the present review. Instead, an extra section has now been included, which deals with the cycloaddition reactions of 3,4-dimethylenethiophene. An exhaustive review of the cycloaddition reactions of thiophenes appeared in 1985 $\langle 85\text{HC}(44/1)671 \rangle$.

2.10.2.9.1 Thermal cycloaddition reactions

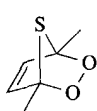
A remarkable set of reactions has been described in which the thiophene can function either as a diene or as a dienophile in an intramolecular Diels–Alder reaction $\langle 90CC405 \rangle$. The *N*-(2-thienyl)allene carboxamide (**162**) on heating at 130°C leads to (**163**) by a [4 + 2] cycloaddition in which the thiophene functions as a 4π component, followed by the usual extrusion of sulfur. However, if the two methyl groups in (**162**) are replaced by phenyl groups, the product obtained is (**165**). This could have arisen by a [2 + 4] cycloaddition to form (**164**), with the thiophene being the 2π component. Subsequent aromatization would then give (**165**) (Scheme 28). An alternative explanation for the formation of (**164**) is a [3,3]sigmatropic rearrangement of a [4 + 2] cycloadduct.



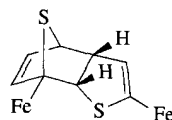
Scheme 28

The importance of choosing the proper benzyne precursor for a successful cycloaddition with thiophene was mentioned in CHEC-I. The most suitable precursor was found to be diphenyliodonium-2-carboxylate. This reaction has been critically reexamined $\langle 88JOC5799, 89JOC2142 \rangle$. Several mono- and di-substituted thiophenes have been used as substrates in this study, and minor products identified. The major pathway appears to be the [4 + 2] cycloaddition, leading to naphthalene after sulfur elimination. The [2 + 2] pathway is also operative, especially with 3-halothiophenes. A [3 + 2] cycloaddition, followed by elimination of acetylene, leads to the observed small amounts of benzo[*b*]thiophene.

The oxidative ring opening of 2,5-dimethylthiophene with singlet oxygen was discussed in CHEC-I $\langle 84CHEC-I(4)741 \rangle$. The initial cycloaddition to form the thiazonide (**166**) $\langle 78AG811 \rangle$ has subsequently been confirmed $\langle 84TL4921 \rangle$. Secondary transformations result in the observed products reported earlier $\langle 87TL4981 \rangle$. 2-Ferrocenylthiophene on attempted Friedel–Crafts acylation in the presence of $AlCl_3$, gave a dimer in 24% yield. The structure of this dimer has been elucidated from MS and NMR studies. It appears to be the [4 + 2] *endo*-cyclodimerized product (**167**) $\langle 94T5765 \rangle$. This is the first example of a thiophene dimerization under such mild conditions (0°C, CH_2Cl_2 , $AlCl_3$).



(166)



(167) Fe = Ferrocenyl

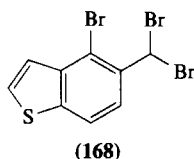
The [4 + 2]cycloaddition reactions of benzo[*c*]thiophenes have been further exploited. 4,5,6,7-Tetrafluorobenzo[*c*]thiophene undergoes cycloaddition to DMAD and tetrafluorobenzyne; this is followed by sulfur extrusion, yielding a naphthalene and an anthracene, respectively $\langle 90JCS(P1)1919 \rangle$. The Diels–Alder reaction of 1,3-bis(4-methoxyphenyl)benzo[*c*]thiophene, with *N*-phenylmaleimide proceeds normally. However, with a sterically hindered substrate like 1,3-bismesitylbenzo[*c*]-

thiophene normal [4 + 2] cycloaddition does not take place; instead, a very low yield of the adduct of the dienophile across the 4,7-positions in the benzene ring was obtained <90CB2065>.

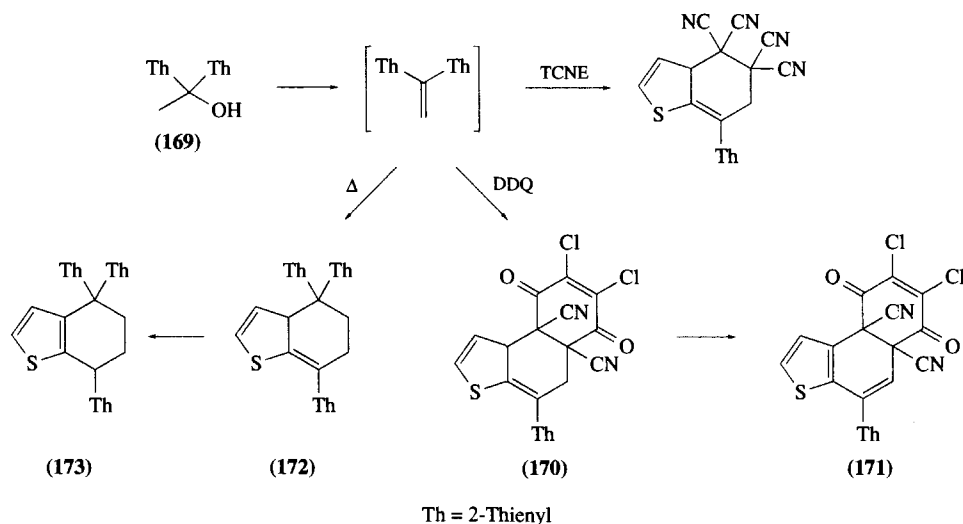
Intramolecular nitrile oxide addition to the 2,3-double bond of thiophene has been used to synthesize several spiro-annulated heterocycles <88S342>.

2.10.2.9.2 Cycloadditions involving side-chain π -systems in addition to the thiophene double bond

Several [4 + 2] cycloadditions of 2-vinyl and 3-vinylthiophene with dienophiles have been reported <85T2435, 87T269, 87T991>. Earlier, similar reactions of the silyl enol ether of 2-acetylthiophene with several dienophiles had been recorded <84JCR(S)218>. 2-Vinylthiophene also reacts with tetrabromocyclopropene by [4 + 2] cycloaddition; subsequent loss of HBr with concomitant opening of the cyclopropane ring leads to the benzo[*b*]thiophene derivative (**168**) in 32% yield <90TL4581>.



The cycloaddition of 1,1-bis(2-thienyl)ethylene (prepared *in situ* from the ethanol (**169**) with strong electron acceptors such as TCNE or ddq has been shown to proceed via a radical ion pair formed by electron transfer (Scheme 29) <90H(31)1873>. The reaction with TCNE is rapid and quantitative, taking place at room temperature in 15 min. With ddq, the initial product (**170**) is further oxidized to (**171**). When the ethanol (**169**) is heated alone in the dark at 150°C, it generates the aromatized cycloadduct (**173**) in 70% yield. Other minor products possibly result from a radical process <91CB1203>. On the other hand, irradiation of the alcohol (**169**) generates 1,1-bis(2-thienyl)ethylene cleanly, which is subsequently transformed to the cycloadduct (**172**). A radical cation may be implicated in this photochemical [4 + 2] cycloaddition also.

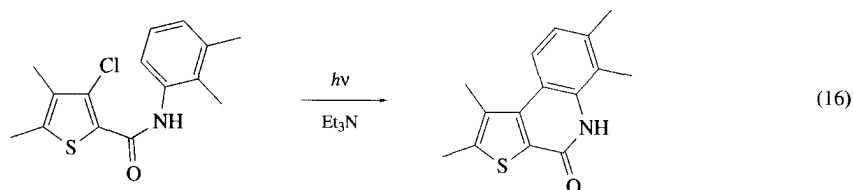


Scheme 29

2.10.2.9.3 Photocycloadditions

The sensitized photochemical [2 + 2] cycloaddition of halogenated alkenes to benzo[*b*]thiophene has been explored further. With 2,3-dichlorobenzo[*b*]thiophene as the substrate, addition of vinyl bromide gives a 1 : 1 mixture of two stereoisomers. It has been concluded that the reaction proceeds

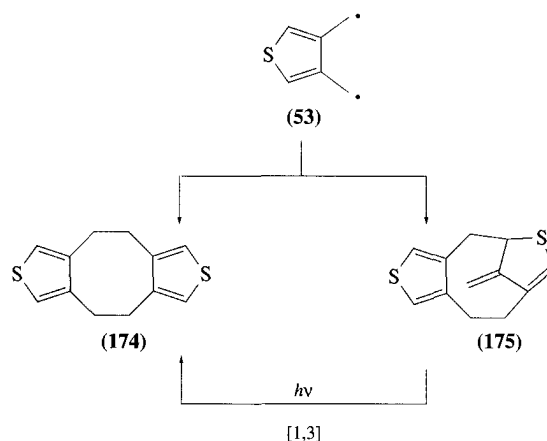
via a biradical intermediate <83JOC1725>. Several photoinduced cyclizations of the type shown in Equation (16) have been carried out by Castle and co-workers <91JHC1309>.



2.10.2.9.4 Cycloadditions of 3,4-dimethylenethiophene

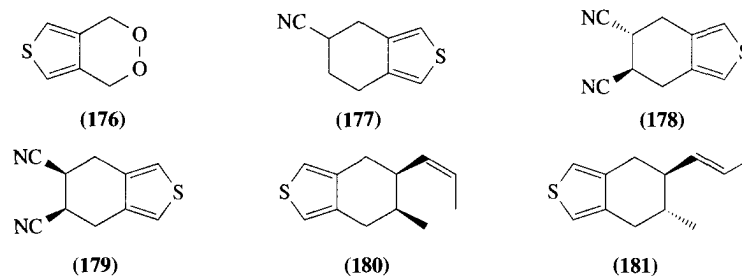
The generation of the singlet diradical, 3,4-dimethylenethiophene, is discussed in Section 2.10.2.2.4. The cycloaddition reactions of the species are discussed in the present section.

The biradical (**53**) forms a symmetrical (**174**) and an unsymmetrical (**175**) dimer. The latter undergoes a photolytic [1,3]sigmatropic rearrangement to the symmetrical dimer (**174**) (Scheme 30) <87TL4263, 89JA3659>.



Scheme 30

The diradical (**53**) reacts with oxygen to form the peroxide (**176**). This could arise either by conversion to the triplet state and then concerted cycloaddition with $^3\text{O}_2$, or by a two-step addition of the singlet biradical (**53**) with $^3\text{O}_2$ to form (one bond at a time) a triplet adduct, intersystem crossing and then ring closure. The biradical (**53**) can also be trapped by electron-poor alkenes; the yields of cycloadducts are almost quantitative. Thus, acrylonitrile gives (**177**). The cycloaddition is highly stereospecific: fumaronitrile gives the *trans*-adduct (**178**), while maleonitrile gives the *cis*-adduct (**179**). Similarly, the *cis,cis*-hexa-2,4-diene gives (**180**) while the *trans,trans*-diene gives (**181**). The stereospecificity of these cycloadditions suggests that (**53**) is a singlet and that the cycloaddition is concerted. The analogy to Diels–Alder reaction is striking <89JA8732>.



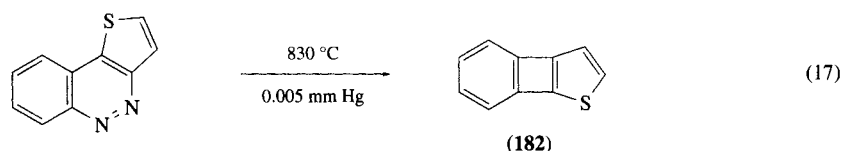
Berson and co-workers have further studied the kinetics of the cycloaddition of 2,5-dimethyl-3,4-dimethylenethiophene with *para*-substituted styrenes and benzaldehydes in order to understand

better the mechanism of such reactions <91TL5305>. For the reaction with substituted benzaldehydes, the Hammett plot is linear. But with substituted styrenes, positive σ (electron-withdrawing) substituents cause a linear enhancement of the rate, while negative σ substituents only result in the rate flattening out, becoming unresponsive even to the strongest electron-releasing substituents.

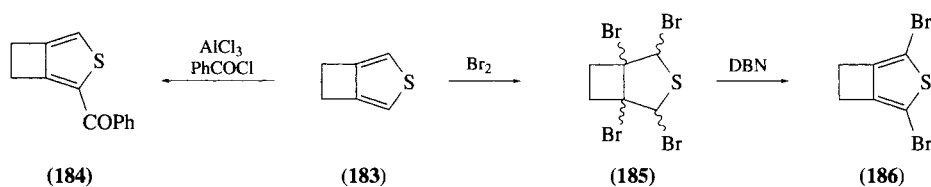
2.10.2.10 Reactions of Strained Thiophenes

The reactivity profile of the thiophene ring undergoes a significant change when it is fused to a three- or four-membered ring, due to the severe angle strain. Such compounds exhibit a considerable amount of alkenic and dienic character.

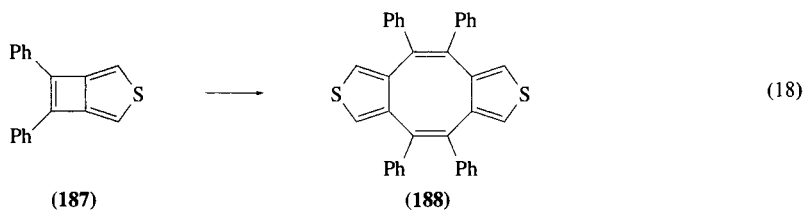
The thiophene analogue (**182**) of biphenylene has been generated by FVP of the cinnoline (Equation (17)) in 14% yield <79TL3571>. The compound is thermally unstable, decomposing within a few hours at room temperature. At 0°C, under nitrogen for 1 week, it gives a dimer. The benzothiophene analogue of (**182**) has also been prepared by a similar FVP of a cinnoline <84TL4283>. This compound appears to be more stable than (**182**).



Garratt *et al.* have employed the Wittig reaction to synthesize a series of cyclobutathiophenes. The parent, 3-thiabicyclo[3.2.0]hepta-1,4-diene (**183**) is thermally stable upto 235°C <72CC1014, 74JOC2222>. It undergoes Friedel–Crafts acylation to give the benzoyl derivative (**184**) in 19% yield (Scheme 31). However, with bromine, it gives the tetrabromide (**185**) in 90% yield. This could be converted to the dibromothiophene (**186**) by base. Both (**183**) and (**186**) could be oxidized to the respective *S,S*-dioxides by MCPBA. 6,7-Diphenyl-3-thiabicyclo[3.2.0]heptatriene (**187**) has been prepared using the same Wittig approach <72JA1022>. This compound is transformed to the dimer (**188**) either on thermolysis or photolysis (Equation (18)).

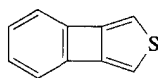


Scheme 31



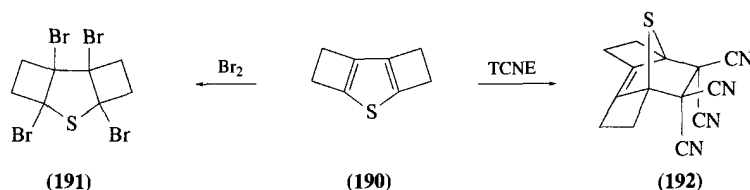
The benzene-fused analogue (**189**) has also been prepared in 14% yield by a Wittig reaction <72JA7087>. This could be oxidized to the sulfoxide and sulfone by H₂O₂ in acetic acid. Neither the sulfoxide nor the sulfone showed any tendency to dimerize by a Diels–Alder mode. This is in contrast to simple thiophene *S*-oxides, and is probably because any such dimerization would result in the formation of a benzocyclobutadiene structure. Photolysis of the sulfone leads to a head-to-head dimer. The thiophene ring in (**189**) is very reactive. Bromination with pyridinium bromide

perbromide gives two stereoisomeric tetrabromides. These could be converted by base to the corresponding dibromothiophene.



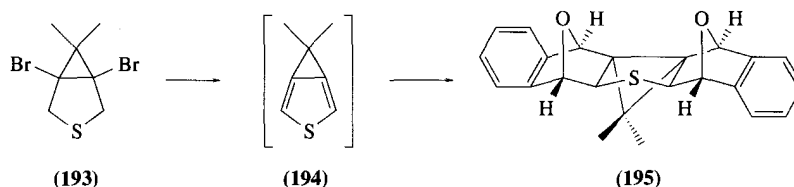
(189)

The most detailed studies in this series have been carried out on the thiophene (190) with two cyclobutane rings fused at the 2,3- and 4,5-positions (93JA4612). It is a reasonably stable, crystalline solid. From the $J_{13\text{C-H}}$ coupling constants, the s character of the methylene C—H bonds is estimated to be about 29%. Bromine rapidly adds to (190) at room temperature in CH_2Cl_2 to give the tetrabromide (191) as a mixture of two stereoisomers (Scheme 32). Under the same conditions, tetramethylthiophene gives only side-chain brominated products. The alkenic properties of (190) are also shown by the enhanced reactivity towards dienophiles. It reacts with TCNE at room temperature to give the adduct (192) in 83% yield. Similarly, maleic anhydride gives a mixture of *endo*- and *exo*-adducts in 87% yield. Tetramethylthiophene does not react with either TCNE or maleic anhydride. The enhanced reactivity of (190) is ascribed to an increase in HOMO energy by ring strain, thereby decreasing the energy difference between HOMO diene and LUMO dienophile. FVP of (190) leads to benzo[*b*]thiophene in 16% yield. The mechanism is not known.



Scheme 32

Fusion of a three-membered ring to thiophene makes it even more unstable. Evidence has been presented for the formation of cyclopropa[*c*]thiophene (194) (90TL1315). Thus dehydrobromination (KOBU^1) of (193) in the presence of isobenzofuran gave the adduct (195) in 57% yield (Scheme 33).



Scheme 33

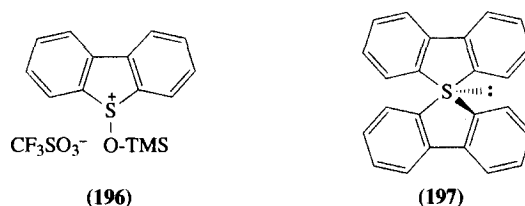
2.10.3 REACTIVITY OF NONCONJUGATED RINGS

2.10.3.1 Thiophene-1-oxides

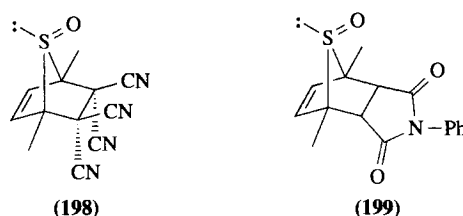
The scanty literature reports available up to 1985 have been covered in three reviews (84CHEC-1(4)741, 85HC(44/1)571, 85HC(44/1)671). However, a few publications of considerable theoretical interest have appeared since then.

Although σ -sulfuranes had been postulated as reactive intermediates earlier, they had not been isolated (84CHEC-1(4)741). The first stable pentacoordinated sulfur compound with four C—S bonds has now been synthesized and fully characterized (92CC1141). The *O*-trimethylsilyl derivative (196) of dibenzothiophene *S*-oxide was treated at -78°C with 2,2'-dilithiobiphenyl. This led to (197) in 96% yield. The structure of (197) was fully secured by ^1H and ^{13}C NMR spectroscopy as well as by x-ray diffraction. In the solid state, it has a slightly distorted pseudotrigonal bipyramidal geometry with two apical S—C bonds, two equatorial S—C bonds, and the lone-pair electrons occupying the

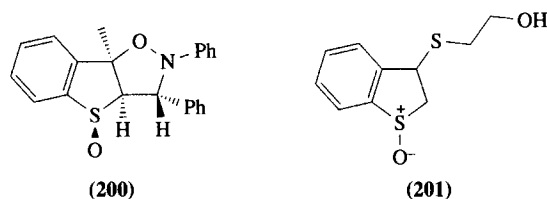
third equatorial position. This is the first fully characterized tetraorganosulfurane having two different types of S—C bonds.



The π -facial selectivity in [4 + 2] cycloadditions of thiophene-1-oxide has been investigated <89TL5077>. 2,5-Dimethylthiophene-1-oxide was generated *in situ* by MCPBA oxidation of 2,5-dimethylthiophene. This was then cycloadded to naphthaquinone, TCNE, *N*-phenylmaleimide, and 2-chloroacrylonitrile. In each case, the structure of the product was established by x-ray analysis. It was found that in every case, addition had occurred exclusively in a contrasteric manner, *syn* to the sulfur–oxygen bond as in (198) and (199). This preference for approach of the dienophile *anti* to the sulfur lone pair has been explained as due to stabilization of the transition state by favorable interaction with the σ^* orbital of the incipient bond.



The [2 + 4] cycloaddition of 3-methylbenzo[*b*]thiophene-1-oxide with *N*, α -diphenyl nitron gives only (200) with the dipole approaching in an *endo*-fashion, *syn* to the S—O bond <84JCS(P2)1>. However, as reported in CHEC-I, <84CHEC-I(4)741>, the [2 + 4] cycloaddition with mesitonitrile oxide gives a 1 : 1 mixture of both stereoisomers <82JOC2461>. The reaction is, however, regiospecific. During a study of the oxidative metabolism of thiophene derivatives, it was found that thiophene-1-oxides were active electrophilic metabolites which bind covalently to hepatic proteins. As a model compound, benzo[*b*]thiophene-1-oxide was prepared and shown to react with mercaptoethanol in MeOH forming the adduct (201) <91JA7825>.



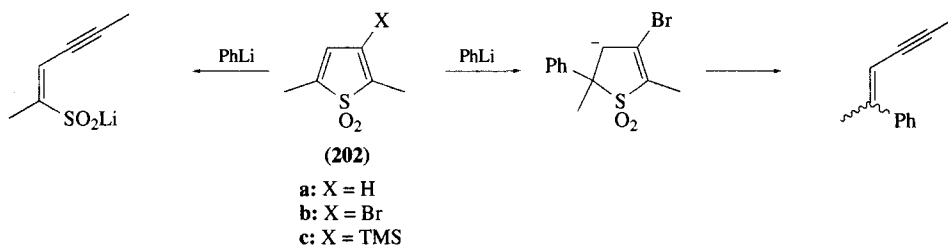
2.10.3.2 Thiophene-1,1-dioxides

The chemistry of thiophene-1,1-dioxides has been reviewed earlier <85HC(44/1)571, 85HC(44/1)671>. In addition, CHEC-I has provided a fairly exhaustive coverage of this subject <84CHEC-I(4)741>. Some interesting new reactions which have come to light since then are discussed below.

2.10.3.2.1 Reaction with nucleophiles

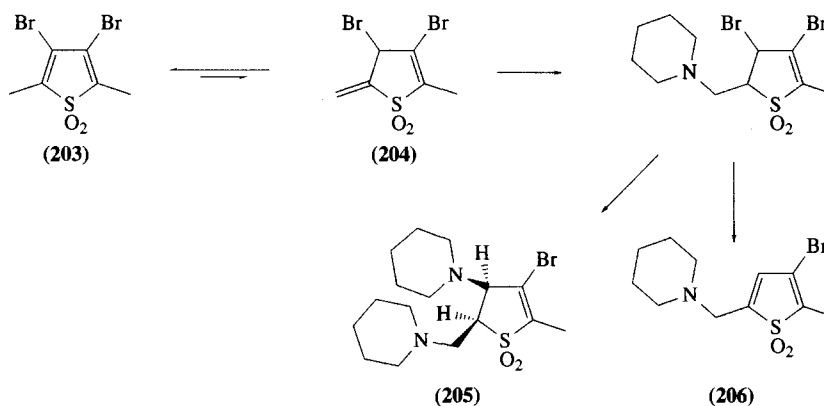
An authoritative account has recently appeared, covering the work of the Swedish school in the last few years <93PS(74)113>. This is especially devoted to the ring-opening reactions observed on treatment of thiophene-1,1-dioxides with nucleophiles. Treatment of 2,5-dialkyl-3-bromothiophene-1,1-dioxides such as (202b) with organolithium reagents leads to ring opening by two different

pathways (Scheme 34). The first is halogen-metal exchange, followed by ring opening to an enyne-sulfinate which can be trapped by electrophiles such as benzyl bromide. The second pathway is an initial addition of the organolithium reagent to the double bond, followed by nonstereospecific ring opening with loss of SO_2 and LiBr , to form *E/Z* mixtures of enynes. In contrast, Grignard reagents lead to more complex products. The primary attack seems to take place at C-4, followed by nucleophilic attack on a second molecule of (202b) with loss of one SO_2 and a Br^- . A cage molecule results. The bromine in (202b) can be displaced by alkyl or aryl groups by treating with organocopper reagents.



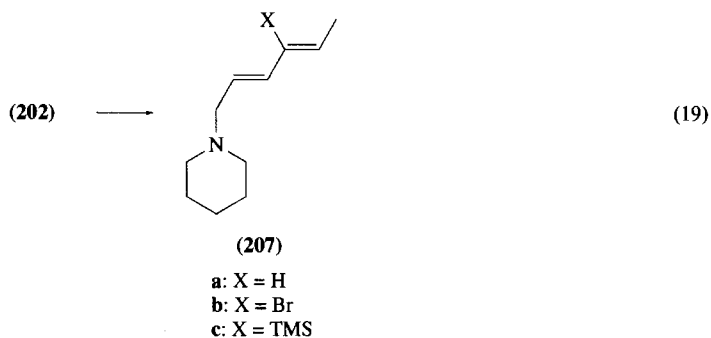
Scheme 34

3,4-Dibromo-2,5-dimethylthiophene-1,1-dioxide (203) reacts with piperidine in benzene to form 3-bromo-2-methyl-4-piperidino-5-piperidinomethyl-*cis*-4,5-dihydrothiophene-1,1-dioxide (205) (Scheme 35). The *exo*-methylene tautomer (204) of (203) is probably involved in this transformation. If the reaction is carried out at 100°C , 3-bromo-2-methyl-5-piperidinomethyl thiophene-1,1-dioxide (206) is formed in 84% yield.

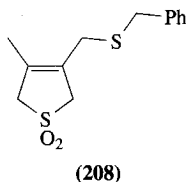


Scheme 35

2,5-Dimethylthiophene-1,1-dioxide and its 3-substituted derivatives (202a,b,c) undergo stereoselective ring opening on treatment with piperidine in refluxing toluene to form 6-piperidino-2,4-hexadienes (207a,b,c) (Equation (19)). This may also involve addition to the *exo*-methylene tautomer, followed by shifting of the double bond and cheletropic extrusion of SO_2 [94S40]. Other cyclic secondary amines behave in the same manner. On the other hand, with aqueous piperidine and (202a) Michael addition to the ring double bond takes place, leading to 2,5-dimethyl-3-piperidino-*trans*-2,3-dihydrothiophene-1,1-dioxide in quantitative yield.

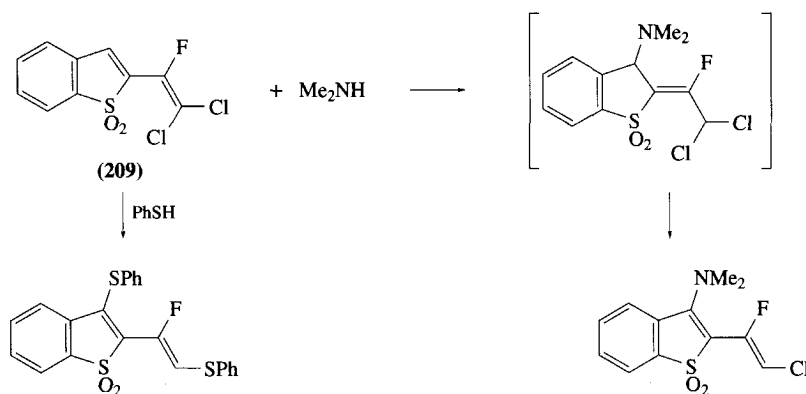


Reaction of 3,4-dimethylthiophene-1,1-dioxide with benzylthiolate at room temperature gives 3-benzylthiomethyl-4-methyl-2,5-dihydrothiophene-1,1-dioxide (**208**) in 86% yield. Again an *exo*-methylene tautomer is implicated. A similar reaction takes place with *sec*-amines. With the 2,5-dimethyl isomer (**202a**), reaction with benzylthiolate is analogous to that with aqueous piperidine.



Reaction of the bromo compound (**202b**) with sodium ethoxide in ethanol leads to *trans* and *cis* (ratio 4 : 1) 2-ethoxymethyl-3-ethoxy-5-methyl-2,3-dihydrothiophene-1,1-dioxide.

Several 2-(haloethenyl)benzo[*b*]thiophene-1,1-dioxides have been prepared and their reaction with nucleophiles studied <89JOC3311>. Thus the dichlorofluoro compound (**209**) reacts with amines by initial addition at position 3, followed by 1,4-elimination of HCl (Scheme 36). Alkoxide acts in a similar manner. Thiophenol leads to the bis (phenylthio) product.

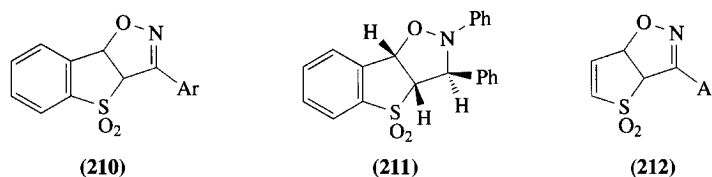


Scheme 36

2.10.3.2.2 Cycloaddition reactions

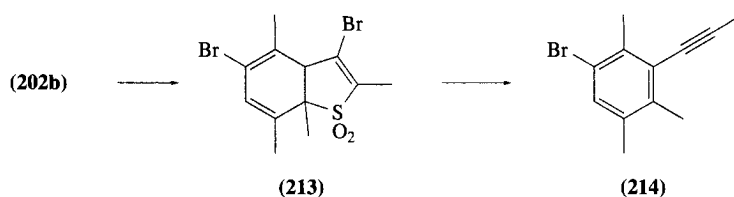
A detailed discussion of the earlier work on this can be found in CHEC-I. The cycloaddition of tetrachlorothiophene-1,1-dioxide with azulene takes place in the [4 + 2] mode rather than a [4 + 6] mode. A frontier orbital-based explanation has been provided for this <85TL2607>. 2-Menthyl-3,4,5-trihalothiophene-1,1-dioxides have been found to be asymmetric diene equivalents <88T3127>. Dienophiles such as styrene, allyl alcohol and acrylamide lead to regio- and stereoisomerically pure adducts, which then eliminate SO₂ to form dihydrobenzene derivatives.

Mesitonitrile oxide and *N*, α -diphenyl nitron undergo regioselective cycloaddition to benzo[*b*]thiophene-1,1-dioxide, giving (**210**) and (**211**). The cycloaddition of mesitonitrile oxide with thiophene-1,1-dioxide is also regiospecific, leading to (**212**) <82JOC2461, 82T3629, 84JCS(P2)1>.



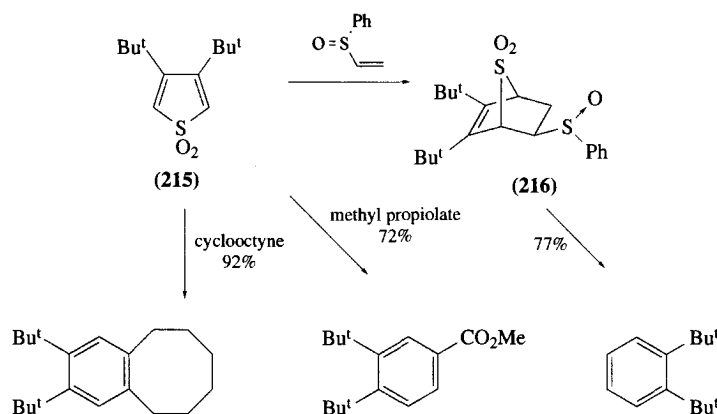
The bromo compound (**202b**) undergoes [4 + 2] cycloaddition with itself; subsequent extrusion of SO₂ gives a 3*a*, 7*a*-dihydrobenzo[*b*]thiophene derivative (**213**). Further heating results in ring opening

to form an acetylene (**214**) (Scheme 37) <93PS(74)113>. This reaction has been extended to several other substituted 3-bromothiophene-1,1-dioxides.



Scheme 37

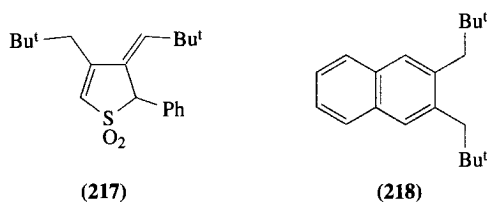
In cycloadditions involving thiophene-1,1-dioxides, one of the most interesting new developments is the use of 3,4-di-*tert*-butylthiophene-1,1-dioxide (**215**) for the synthesis of other *ortho*-di-*tert*-butyl-substituted aromatic and heteroaromatic compounds. Compound (**215**) can be prepared easily in large quantities from pinacolone. Heating (**215**) with phenyl vinyl sulfoxide in refluxing *o*-chlorotoluene gave *o*-di-*tert*-butylbenzene in 77% yield; obviously the initial adduct (**216**) had eliminated a molecule each of SO₂ and benzenesulfenic acid in this process. Substituted acetylenes similarly add to (**215**) and eliminate SO₂ to provide *o*-di-*tert*-butylbenzenes with other substituents attached. Two such examples are given in Scheme 38 <88JA6598>.



Scheme 38

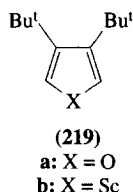
Successive cycloaddition of (**215**) with two molecules of *N*-phenyl-1,2,4-triazolin-3,5-dione, followed by alkaline hydrolysis leads to 4,5-di-*tert*-butylpyridazine by spontaneous air oxidation and extrusion of nitrogen <89H(29)1241, 90JA7648>.

The same concept has been extended to the synthesis of aromatic and heteroaromatic compounds bearing two 1-adamantyl substituents <90JA5654> and two neopentyl substituents <94TL2709> at *ortho*-positions. Interestingly, the reaction of 3,4-dineopentylthiophene-1,1-dioxide with benzyne (from 2-carboxybenzenediazonium chloride) gives (**217**) as the major product (68%) via an ene reaction; the cycloadduct-SO₂ extrusion product (**218**) is formed in 20% yield. The latter undergoes further cycloaddition with another molecule of benzyne to give a dibenzo barrelene derivative (7.5%). The formation of such barrelene derivatives had been observed earlier with a few other thiophene-1,1-dioxides as well <86H(24)1233>.



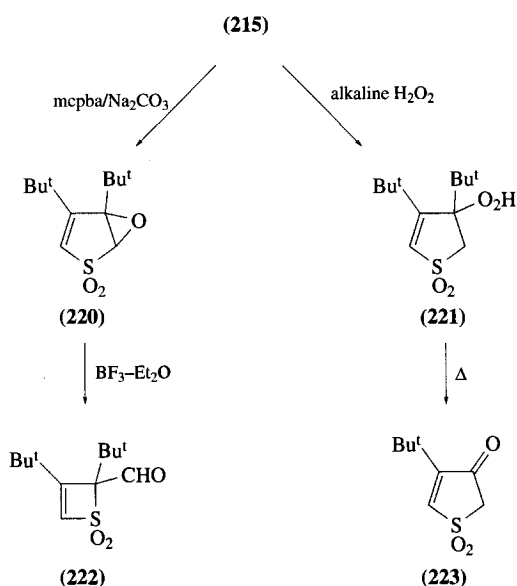
2.10.3.2.3 Other reactions

FVP of 3,4-di-*tert*-butylthiophene-1,1-dioxide (**215**) gave a 12% yield of the furan (**219a**) <90TL4473>. Reaction of (**215**) with elemental selenium gave (**219b**) in 21% yield.



Thiophene-1,1-dioxides can be stabilized by the introduction of a silyl group at position 2. This can be subsequently removed by treatment with alumina. Thus, 4-bromo-2-methylthiophene-1,1-dioxide is best prepared by oxidizing 4-bromo-2-methyl-5-trimethylsilylthiophene, and then removing the TMS group by Al_2O_3 <91ACS632>. 2,5-Disilylated thiophene-1,1-dioxides have also been prepared and found to be stable <92H(34)1085>. These can be converted to 2,5-dibromo or 2,5-diiodothiophene-1,1-dioxides by treatment with bromine or iodine, respectively, in the presence of AgBF_4 .

The oxidation of 3,4-di-*tert*-butylthiophene-1,1-dioxide (**215**) by various reagents has been examined (Scheme 39) <91JOC4001>. Oxidation by peracids under basic conditions (MCPBA/ Na_2CO_3 or $\text{CF}_3\text{CO}_3\text{H}/\text{Na}_2\text{HPO}_4$) gives a good yield of the epoxysulfone (**220**), whereas oxidation with alkaline H_2O_2 gives the hydroperoxide (**221**). The former undergoes Lewis acid-catalyzed ring contraction to the thiete-1,1-dioxide (**222**) in 40% yield. The hydroperoxide (**221**), on heating above its melting point, gives the ketone (**223**) quantitatively. Several other congested thiophene-1,1-dioxides have been subjected to MCPBA oxidation to produce either the epoxysulfones or the thiete-1,1-dioxides <92TL7539>.



Scheme 39

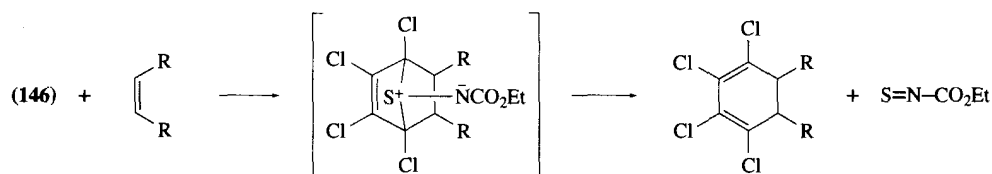
2.10.3.3 Thiophene S,N-ylides and S,C-ylides

The reactions of thiophene S,N- and S,C-ylides have been adequately reviewed <89AHC(45)151>.

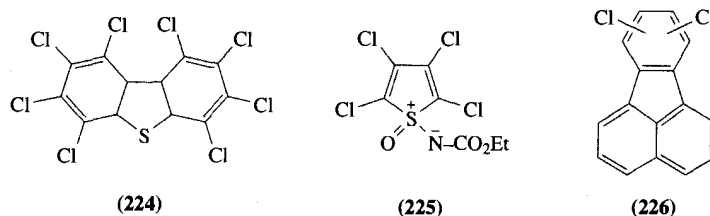
Thiophene S,N-ylides are comparable with the 1-mono- and 1,1-dioxides in that they exhibit diene rather than aromatic properties <86JCS(P1)233>. The S,C-ylides appear to be more sluggish in cycloaddition reactions and are therefore considered more "aromatic." However, MO calculations show that the sulfur atom is distinctly pyramidal in both species.

2.10.3.3.1 Cycloaddition reactions of *S,N*-ylides and *S,C*-ylides(i) The ylides as 4 π -components

The *S,N*-ylide (**146**) obtained from tetrachlorothiophene and ethoxycarbonylnitrene undergoes [4 + 2]cycloaddition with a range of alkenes. The adduct then undergoes a cheletropic extrusion of the acylthionitroso compound $\text{EtO}_2\text{C}-\text{N}=\text{S}$, to form a tetrachlorodihydrobenzene unit (Scheme 40). Excellent yields of the final product are obtained with alkenes which are sterically undemanding. These include acenaphthylene, allyl alcohol, etc. Surprisingly, even thiophene behaves as a bis-dienophile, leading to (**224**) in 17% yield.



Scheme 40



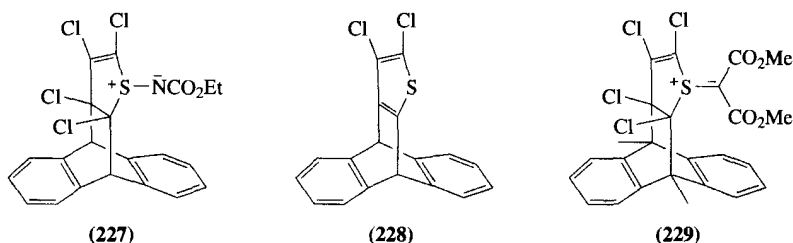
Oxidation of the *S,N*-ylide (**146**) with MCPBA gave the product (**225**) in 84% yield. This sulfoxilimine corresponds to the thiophene-1,1-dioxide. This compound also undergoes the expected [4 + 2]cycloaddition with acenaphthylene followed by cheletropic extrusion, leading to the same product as with the parent *S,N*-ylide <86JCS(P1)233>.

In contrast, the *S,C*-ylide (**139a**) prepared from tetrachlorothiophene and diazomalonate undergoes cycloaddition with acenaphthylene much more slowly; however, after heating at 80°C for 7 h, the reaction leads to the aromatized fluoranthene (**226**) in 97% yield. The formation of (**226**) involves a proton shift and loss of chlorine; the exact mechanism and the location of the chlorine atoms are not clear <86JCS(P1)233>.

The above sequence has been utilized to generate and study the reactivity of thionitroso compounds <84CC1144, 86JCS(P1)245>.

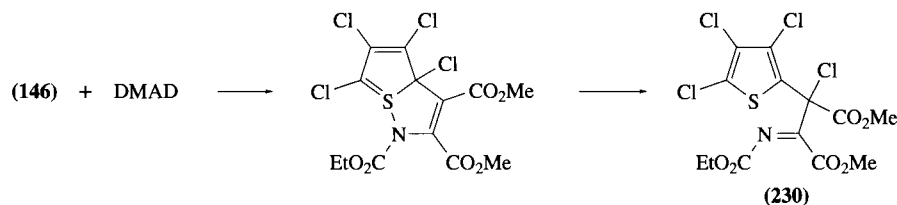
(ii) The ylides as 2 π -components

The *S,N*-ylide (**146**) can act as a dienophile in [4 + 2] cycloadditions with dienes. Thus, anthracene gives (**227**) in 65% yield. This adduct can be converted to the triptycene analogue (**228**) in quantitative yield by treatment with zinc dust <86JCS(P1)233>. The net result of this transformation is equivalent to the cycloaddition of thiophyne. The *S,C*-ylide (**139a**) similarly reacted with 9,10-dimethylantracene to give (**229**) in 56% yield.



(iii) The ylide as a 6π -component

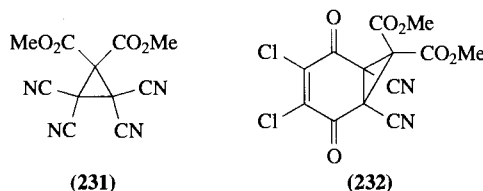
Dimethyl acetylenedicarboxylate reacted with the S,N-ylide (**146**) slowly to give a 10% yield of the product (**230**). The initial reaction corresponds to a $[6 + 2]$ cycloaddition which need not be concerted (Scheme 41). In addition to this mode, the normal $[4 + 2]$ cycloaddition also took place, leading to dimethyl tetrachlorophthalate in 30% yield $\langle 87\text{JCS(P1)2659} \rangle$. The S,C-ylide (**139a**) does not undergo any such cycloaddition with dimethyl acetylenedicarboxylate.



Scheme 41

2.10.3.3.2 Cyclopropanation reactions of S,C-ylides

The use of the S,C-ylide (**139b**) as a source of bismethoxycarbonylcarbene in the presence of transition metals has been referred to in CHEC-I $\langle 84\text{CHEC-I(4)784} \rangle$. This had been used for cyclopropanation of alkenes $\langle 78\text{CC641} \rangle$. It was assumed that the transition metal helped in the cleavage of the S—C bond, and the resulting metal–carbene complex was the active species in the cyclopropanation. Recently, however, some uncatalyzed cyclopropanations of electron-poor Michael acceptors by means of the S,C-ylide (**139c**) have been reported $\langle 88\text{ACS(B)166} \rangle$. Thus, TCNE leads to (**231**) and ddq to (**232**) in 90% and 80% yield, respectively. The latter is a regiospecific reaction. Interestingly, the ylide (**139b**) reacts much more slowly with TCNE, while the tetrachlorothiophene ylide (**139a**) did not react at all.

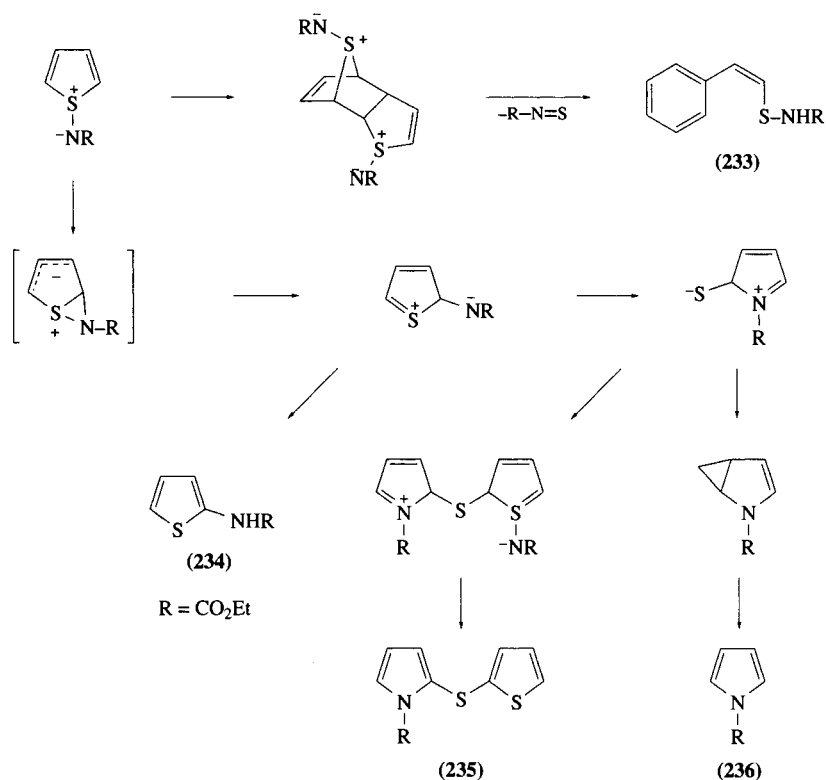


2.10.3.3.3 Thermal transformations of thiophene S,N- and S,C-ylides

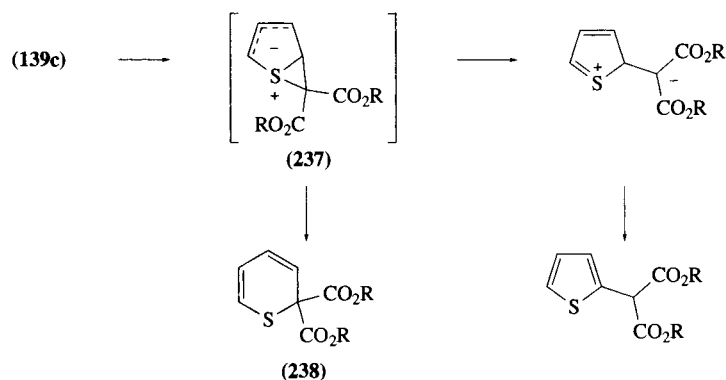
When ethyl azidoformate is heated at 130°C in thiophene, a series of products (**233**) to (**236**) are formed. These have been carefully isolated and identified $\langle 86\text{TL1105} \rangle$. All the products could conceivably arise from an initial S,N-ylide (Scheme 42)

It was mentioned earlier that the S,C-ylide (**139b**) generates a carbenoid species and dichlorothiophene in the presence of transition metal catalysts. Thermolysis of (**139b**) in the absence of such metal catalysts leads to (**145**) through an intramolecular rearrangement $\langle 84\text{CHEC-I(4)741, 89AHC(45)151} \rangle$.

When the two chlorine substituents at positions 2 and 5 are not present, the thermal reaction of the S,C-ylide takes a different course. A tremendous amount of painstaking experimental work has been put in by two schools of research; Porter and co-workers concentrated on ylides of the type (**139e**) $\langle 85\text{CC1590, 88JCS(P1)803, 88JCS(P1)809} \rangle$ while Skramstad and co-workers devoted their attention towards intramolecular carbene attack $\langle 84\text{CC208, 86ACS(B)178, 86ACS(B)303, 93ACS1214} \rangle$. The entire area has been summarized by Porter $\langle 89\text{AHC(45)151} \rangle$. In essence, both groups have isolated 1,2-rearranged products as well as thiapyrans (e.g. (**238**) and (**240**)) on thermolysis of such thiophene S,C-ylides. It is likely that both types of products arise from a common intermediate (or transition state) such as (**237**) as shown in Scheme 43. Cyclopropanes (e.g. (**239**)) can arise as shown in Scheme 44. The cyclopropane derivatives can also rearrange to give 3-substituted thiophenes.



Scheme 42

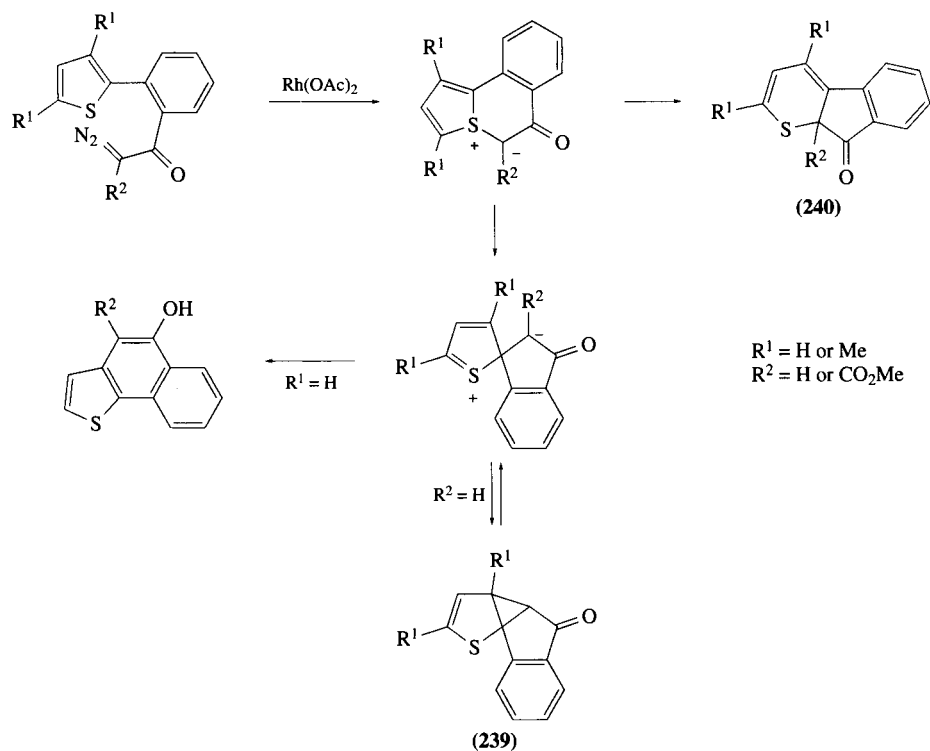


Scheme 43

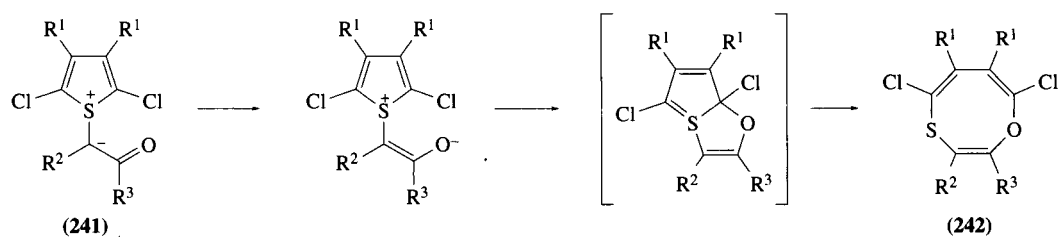
The ylides (**241**) obtained from 2,5-dichlorothiophene and diazoketones readily undergo a totally different type of thermal rearrangement to give oxathiocines (**242**) (Scheme 45) <88CC138, 89JOC4822>. The rearrangement occurs even with ylides which are not stable enough to be isolated.

2.10.3.4 Oxothiophenes Tautomeric with Hydroxythiophenes

In this section, the chemistry of thiophene-3(2*H*)-ones (**243**) and thiophene-2(5*H*)-ones (**244**) is discussed (Scheme 46). When R¹ = H, these are tautomeric with 3-hydroxythiophenes and 2-hydroxythiophenes, respectively. For the sake of convenience, even the chemistry of nonenolizable *x,x*-dialkylthiophenones (R¹, R² ≠ H) is included in this section. Earlier, these subjects had been dealt with in CHEC-I under the section on the reactivity of *O*-linked substituents <84CHEC-I(4)741>.

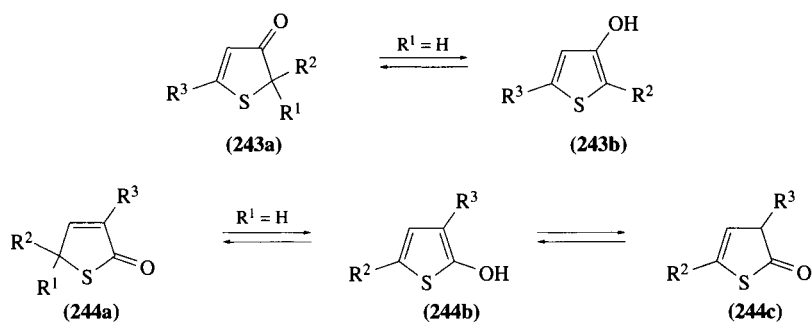


Scheme 44



Scheme 45

Subsequently, an exhaustive review of such compounds has been included in the monograph edited by Gronowitz and Hornfeldt (86HC(44/3)1). Attention will therefore be focused on the more recent developments in this area, while at the same time highlighting the earlier results. It is to be noted that while formally (243) is at the ketone level of oxidation, (244) is at the carboxylic acid level of oxidation.



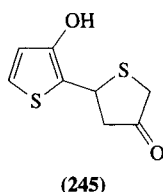
Scheme 46

2.10.3.4.1 Tautomerism

It has been mentioned <86HC(44/3)1> that (**243**; $R^1 = R^3 = H$, $R^2 = Me$) exists at equilibrium as a mixture of the keto form (**243a**) (20%) and enol form (**243b**) (80%). In the case of the *t*-butyl derivative (**243**; $R^1 = R^3 = H$, $R^2 = Bu^t$) it was 45% of (**243a**) and 55% of (**243b**). For the 2,5-dimethyl derivative (**243**; $R^1 = H$, $R^2 = R^3 = Me$), the keto–enol equilibrium constant in cyclohexane was 2.61, while in methanol it was 1.21.

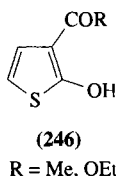
In the case of 2-thiophenones, a third tautomer (**243c**) is also possible when $R^1 = H$. 2(3*H*)-Thiophenones (**244c**) are thermodynamically less stable than 2(5*H*)-thiophenones (**244a**). Typically, with a 5-alkyl substituent (**244**; $R^1 = R^3 = H$), the equilibrium consists of 80–85% (**244a**) and 15–20% (**244c**).

Several 2-alkyl, 2-aryl and 2-functionalized derivatives of (**243**) have now been prepared in high yield by gas-phase pyrolysis (FVP; 625°C; 10^{-3} Torr) of appropriate open-chain precursors <90CC375>. This includes the parent compound (**243**; $R^1 = R^2 = R^3 = H$), which dimerizes to (**245**) in a few days. The parent 3(2*H*)-thiophenone exists in chloroform as a 2.9 : 1 mixture of (**243a**) and (**243b**).



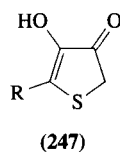
A new method has also been developed for the generation of the enol form of 2- and 3-hydroxythiophenes as well as of 2- and 3-hydroxybenzo[*b*]thiophenes. The method consists of hydrolyzing the corresponding trimethylsilyl ethers under mild conditions <86TL3275, 89JA5346>. Based on this, the keto–enol tautomerism of all four systems has been studied; this includes kinetics of ketonization of the enols as well as determination of the equilibrium constants. In order to facilitate the NMR study, the hydrolysis has been performed in deuterated solvents, typically, 90% CD_3COCD_3 , 10% D_2O , 5×10^{-4} M DCl at 32°C. The results are as follows. 3-Hydroxybenzo[*b*]thiophene has a half-life of ca. 1 day; at equilibrium (in the above mixture), approximately 40% keto form is present. The equilibrium is solvent-dependent. Thus, in DMSO it exists as enol (87%) and keto (13%), whereas in $CDCl_3$, only the keto form is seen. 3-Hydroxythiophene exists as the enol (100%) in DMSO- d_6 , CD_3COCD_3 and CH_3OH ; in CCl_4 , the amount of enol present is 33%. The enol form of 2-hydroxybenzo[*b*]thiophene has been generated by the above procedure at $-34^\circ C$; after 15 min at $-25^\circ C$, it completely changed over to the keto form. The hydrolysis of the silyl ether of 2-hydroxythiophene was followed by ^1H NMR spectroscopy. After 10 min, the 2-deuteroxythiophene was converted into a mixture of the 3-thiolen-2-one (**244a**; $R^1 = R^3 = H$, $R^2 = D$) (65%) and the 4-thiolen-2-one (**244c**; $R^2 = H$, $R^3 = D$) (35%). The latter was unstable and was converted to the 3-thiolen-2-one which was consequently partially deuterated at positions 3 and 5, but not at 4. The equilibrium constants, K_{enol} (E/K) for the four systems in water have been estimated to be: 0.085 (3-hydroxy-benzo[*b*]thiophene); 2.96 (3-hydroxythiophene); 3.42×10^{-6} (2-hydroxybenzo[*b*]thiophene); and $< 10^{-2}$ (2-hydroxythiophene).

Compounds such as (**246**) having a carbonyl substituent at position 3 and a hydroxyl at 2 exist exclusively in the enol form. On the other hand, if the ester is located at position 5, then about 15% of the keto form is seen at equilibrium along with 85% of the enol. 3-Hydroxythiophene-4-carboxylic acid esters seem to exist as a mixture of both tautomers <86HC(44/3)1>.



2,5-Dihydroxythiophene probably exists completely in the dioxo form (thioanhydride), but 2,3-dihydroxythiophene exists exclusively as 3-hydroxythiophene-2(5*H*)one (**244a**; $R^1 = R^2 = H$, $R^3 = OH$). 3,4-Dihydroxythiophene with an alkyl substituent at position 2 (or 5) exists as (**247**).

3,4-Dihydroxy-2-thiophenecarboxylic acid ethyl ester is best prepared by acid hydrolysis and



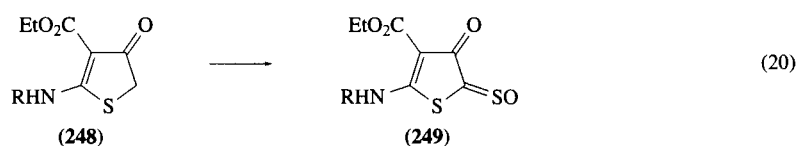
decarboxylation of the 2,5-dicarboxylic ester <93TL8229>. An earlier report <71T3839> had suggested that this compound existed in the keto form. However, detailed NMR spectroscopy has now shown clearly that it exists in the dihydroxy form. The existence of an equilibrium with the less stable keto form is inferred from the fact that the C-5 hydrogen is rapidly exchanged for deuterium when the compound is dissolved in NaOD/D₂O.

2.10.3.4.2 Alkylation and acylation

Alkylation of the ambident anion derived from hydroxythiophenes has been discussed exhaustively <86HC(44/3)1>. In general, soft electrophiles like methyl iodide lead to C-methylation, while the hard electrophile, dimethyl sulfate, gives predominantly the O-methylated product. Similarly acyl chlorides and acid anhydrides give only the O-acylated products. In conformity with the above, it has recently been reported that treatment of (243; R¹ = R² = R³ = H) with acetyl chloride and triethylamine leads to regiospecific O-acetylation. Methylation with methyl toluene-*p*-sulfonate/NaH gives only the O-methyl derivative <90CC375>.

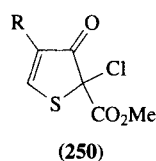
2.10.3.4.3 Condensation reactions at the methylene group

Condensation reactions of the active methylene of the keto form of hydroxythiophenes with carbonyl compounds have been referred to earlier <84CHEC-I(4)741>. Some further interesting examples have come to light. Acid-catalyzed self-condensation of 3(2*H*)-benzo[*b*]thiophenone has led to some novel polycyclic compounds, including a symmetrical trimer <86T763>. The methylene group of thiophen-3(2*H*)-one is active; thus (248) on treatment with thionyl chloride gives the sulfine (249) (Equation (20)). The C=S of this product undergoes cycloaddition with several butadienes <84CC502>. Benzo[*b*]thiophen-3-one also behaves similarly.



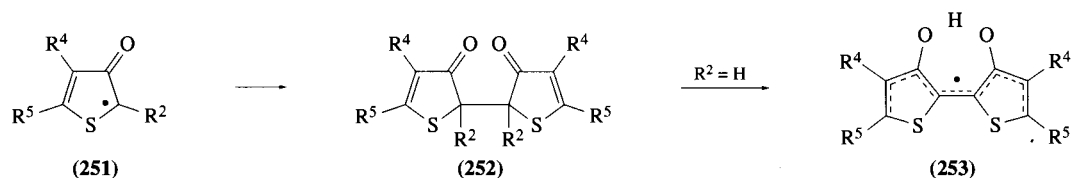
2.10.3.4.4 Halogenation

Chlorination of methyl 3-hydroxythiophene-2-carboxylate by sulfur chloride gives the 2-chloro-thiophen-3-one (250). This reacts with a series of nucleophiles at position 5 by an addition-elimination process, giving 5-substituted-3-hydroxythiophene-2-carboxylates <86JCR(S)168, 87JHC1301, 90JHC315>.

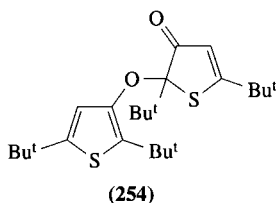


2.10.3.4.5 Oxidative coupling

Oxidative coupling has been referred to earlier <84CHEC-I(4)741> and discussed in detail in a monograph <86HC(44/3)1>. Recently, the consequences of hydrogen abstraction from thiophen-3(2*H*)-ones by photolytically generated *tert*-butoxyl radicals have been investigated <92JCS(P2)935>. The initially formed radical (**251**) undergoes dimerization to (**252**). If R^2 is a hydrogen, then this can further generate the dimer radical (**253**) (Scheme 47). These results agree with the earlier oxidation studies <72ACS31> which had led to the isolation of (**252**; $R^2 = \text{Me}$, $R^4 = R^5 = \text{H}$). When position 2 is substituted by the bulky *tert*-butyl group, coupling takes place via oxygen to form (**254**).



Scheme 47



2.10.3.4.6 Cycloaddition

Thiophen-2(5*H*)-one is capable of functioning as a dienophile. Lewis acids catalyze the cycloaddition. Thus cyclopentadiene reacts with thiophen-2(5*H*)-one in the presence of $\text{BF}_3 \cdot \text{Et}_2\text{O}$ at room temperature to give the adduct as a mixture of *exo* and *endo* isomers <82JOC4777>.

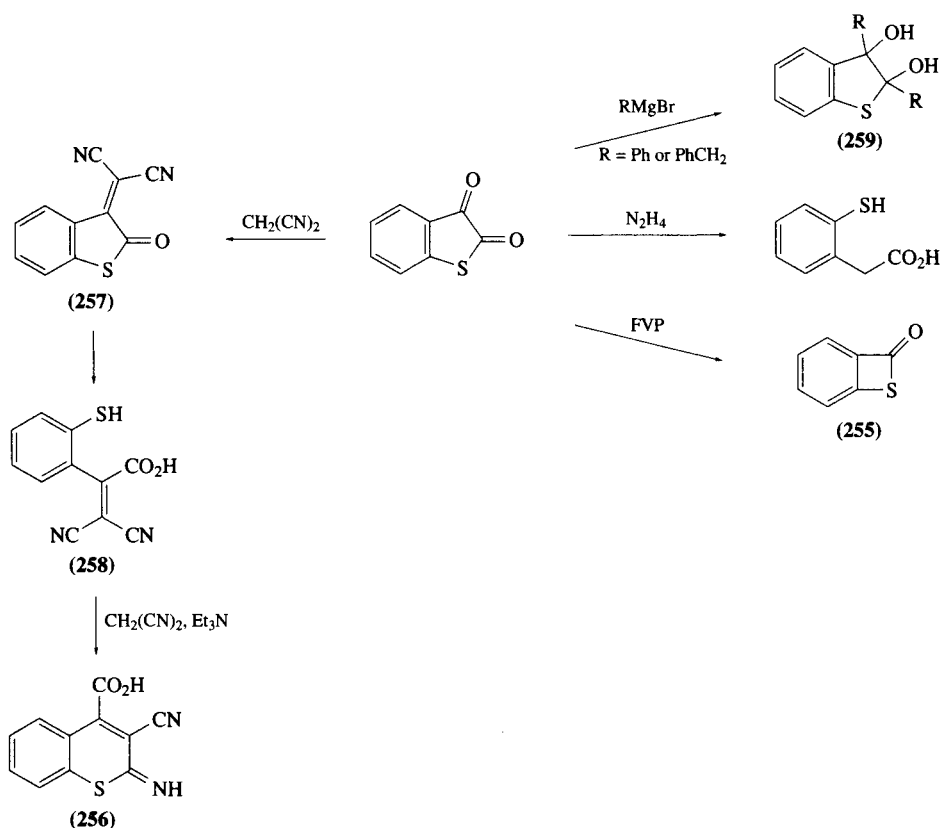
2.10.3.4.7 2,3-Diones

A review on the chemistry of benzo[*b*]thiophene-2,3-dione has covered the literature up to 1987 <88H(27)1489>. FVP of the dione gives (**255**) (Scheme 48). Several new ring-opening reactions of the dione have been reported. Thus, reaction with malononitrile in the presence of triethylamine gives (**256**), while the same reaction without the base gives (**257**). The former obviously arises via (**258**) (Scheme 48). Reaction of the dione under Huang–Minlon conditions with hydrazine hydrate leads to 2-mercaptophenylacetic acid. Phenylmagnesium bromide or benzylmagnesium bromide reacts at both carbonyls of the dione to form diols (**259**).

The 4-benzoyl-2,3-thiophenedione (**260**) undergoes thermal or photochemical cycloaddition with diphenylketene to give the spirothiete (**261**) and the thiophen-2-one (**262**) <91T3045>. The probable mechanism is shown in Scheme 49. With ketenimines, however, the reaction takes a different course, the initial step being a [4 + 2] cycloaddition (Scheme 49) <87H(26)625>. The product is either (**263**) or (**264**) depending on the substituent *R*.

2.10.3.4.8 Sulfoxides and sulfones

The stereochemistry of alkylation of the carbanion generated from 2-methylbenzo[*b*]thiophen-3(2*H*)-one-1-oxide has been investigated <84CPB898>. The *cis* and *trans* isomers (**265a**) and (**265b**) were separately subjected to base treatment, followed by alkylation with RX (Scheme 50). The *trans*:*cis* ratio in the product mixture was determined by ^1H NMR spectra. It was found that in both cases, the alkylation occurred stereoselectively *cis* to the S—O bond, the (**266**) *trans*:*cis* ratio



Scheme 48

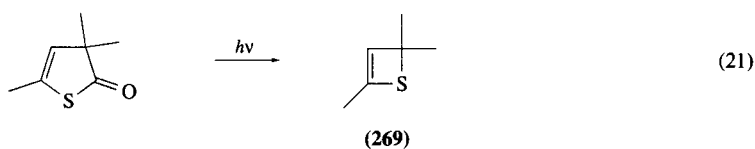
being ca. 1:9. An explanation for this has been offered: the carbanion has the pyramidal configuration; of the two possibilities (267) and (268) for this, the latter would be destabilized because of the repulsive interaction between the anion lobe and the lone pair on the sulfur.

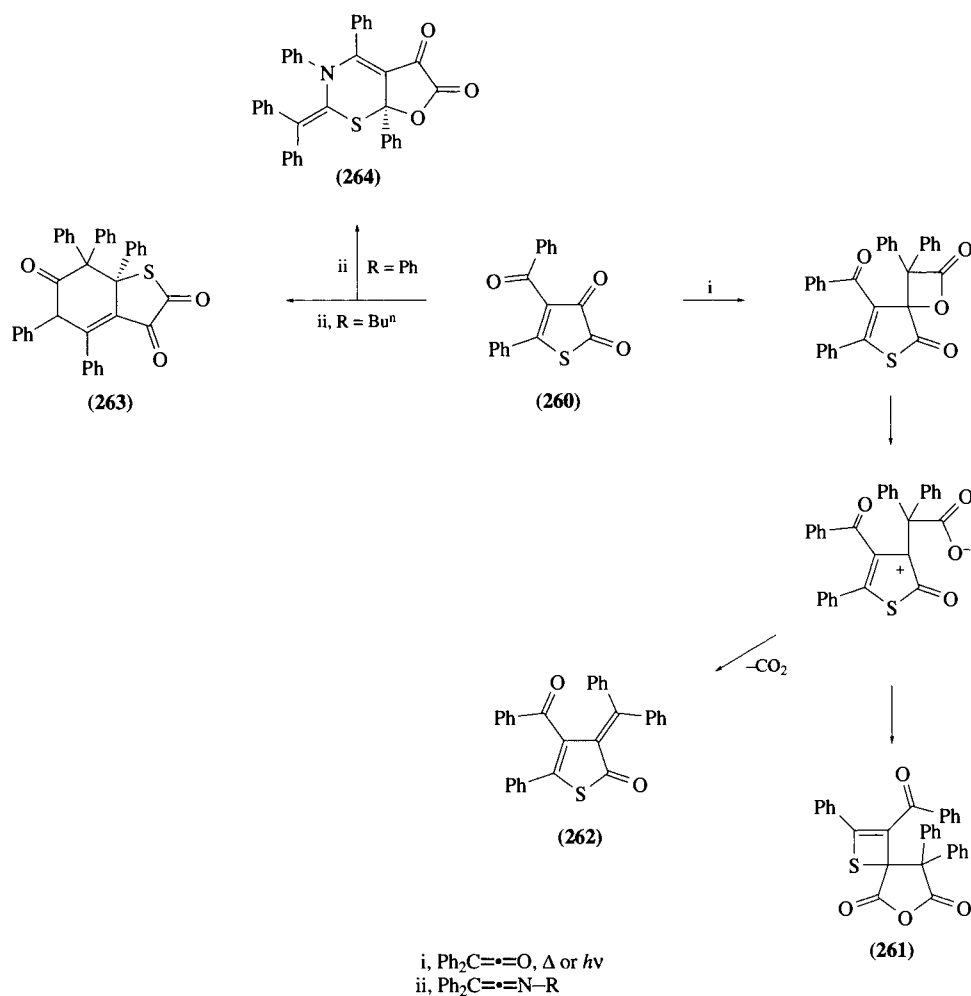


2.10.3.4.9 Photochemistry

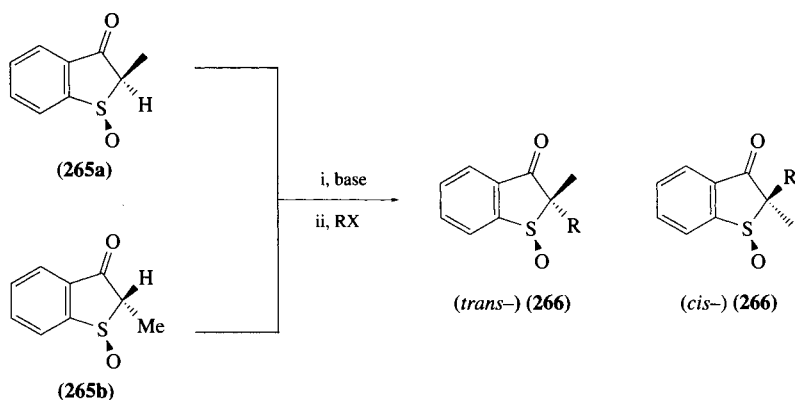
The photolysis of all three species, thiophen-2(3*H*)-one, thiophen-2(5*H*)-one and thiophen-3(2*H*)-one, has been investigated by Margaretha and co-workers.

Irradiation of 3,3,5-trimethyl-2(3*H*)-thiophenone leads to the thiete (269) by decarbonylation (Equation (21)) <89AG(E)1499>. It was realized later, however, that the photolytic decarbonylation of such 2-(3*H*)-thiophenones is more complex than initially assumed. Products depend on the substitution pattern as well as on the actual substituents. A detailed mechanistic route has been suggested for the formation of thietes, thiophenes, thiapyrans, etc. from the excited state of the substrate <92CB2311>.



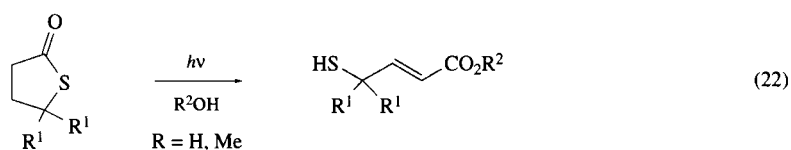


Scheme 49

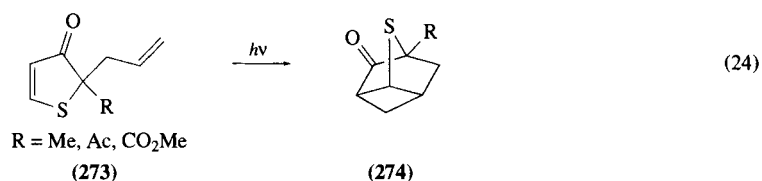
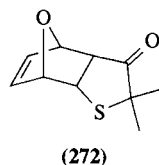
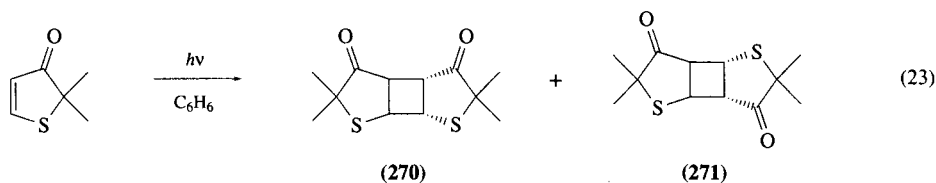


Scheme 50

Photolysis of thiophen-2(5*H*)-one in alcohols with 300 nm light leads to the formation of 4-mercaptocrotonates (Equation (22)). It has been suggested that this is formed from the excited singlet state of the substrate. The 5,5-dimethyl analogue undergoes a similar photolytic ring cleavage, but the 3,4-dimethyl derivative appears to be stable under these conditions. A detailed mechanism for the above observations has been suggested (84HCA2198, 85HCA2350, 89HCA83).



On irradiation in benzene at 366 nm, 2,2-dimethylthiophen-3(2*H*)-one undergoes regio- and stereospecific dimerization (Equation (23)) <84HCA1402>. The head-to-head (**270**) and the head-to-tail (**271**) dimers are formed in the ratio 10:1. Irradiation of the same thiophenone in furan leads to a 1:1 mixture of the cycloadduct (**272**) and the tricyclic dimer (**270**) <92HCA1925>. 2-Propenylthiophen-3(2*H*)-ones (**273**), on being irradiated at a wavelength > 340 nm, are converted in a regioselective and quantitative manner to the tricyclooctanones (**274**) (Equation (24)) <85HCA1129, 92HCA1633>.



The photochemistry of spiro[benzo[*b*]thiophen-2(3*H*)1'-cyclopropan]-3-one (**275**) and the corresponding dioxide (**276**) has been investigated (Scheme 51) <84BCJ2325>. Photolysis of the former in benzene results in cleavage of the cyclopropane ring. The *S,S*-dioxide (**276**) behaves differently. In hydrogen-donating solvents (toluene or isopropanol) it abstracts a hydrogen from the solvent and dimerizes to (**277**). On the other hand, in benzene as solvent, the C—S bond is cleaved homolytically, followed by SO₂ elimination, and substitution on benzene. The final product is a diphenyl derivative (**278**).

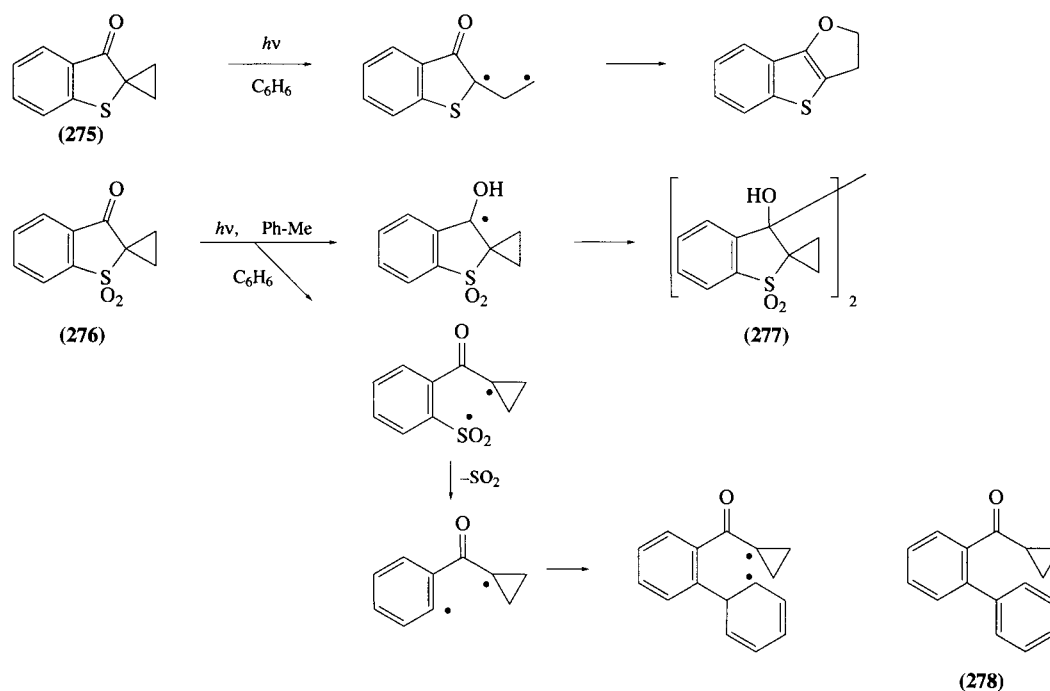
2.10.3.4.10 Macrocyclic polyethers

Crown ethers incorporating two thioindigo units and a disulfide bridge in the side chain have been synthesized. These have been shown to undergo reversible photoinduced *trans-cis* isomerization <94CC917>.

2.10.3.5 Dihydrothiophenes

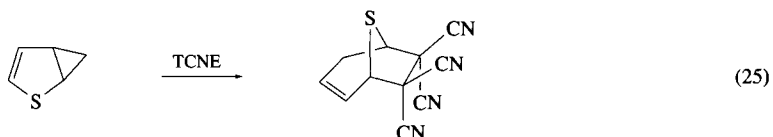
2.10.3.5.1 2,3-Dihydrothiophenes

This section covers the following topics: (i) newer reactions of 2,3-dihydrothiophenes; (ii) generation and reactivity of 2,3-dihydro-2,3-bismethylenethiophenes; and (iii) reactions of 2,3-dihydrothiophene 1,1-dioxides.

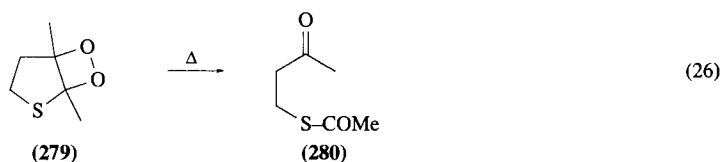


Scheme 51

(i) 2,3-Dihydrothiophene undergoes regioselective [2 + 2] cycloaddition with ketenes to give cyclobutanones. These products have been further transformed into other polycyclic compounds <85TL1791, 86JHC1551>. The cycloaddition of nitrile oxides to thiophene was discussed in CHEC-I <84CHEC-I(4)741>. The notable feature there was the formation of the bis-adducts. In order to investigate the regiochemistry of the second cycloaddition, the reaction of nitrile oxides with 2,3-dihydrothiophene has been studied. Surprisingly, it has been found that the regioselectivity is rather poor in this reaction, with both regioisomers being formed in the ratio 88:12 <84T441>. An explanation based on frontier orbital interactions has been provided. Homothiophene undergoes a $[(\sigma^2 + \pi^2) + \pi^2]$ cycloaddition with TCNE at 20°C in a quantitative reaction (Equation (25)) <85AG(E)594>.

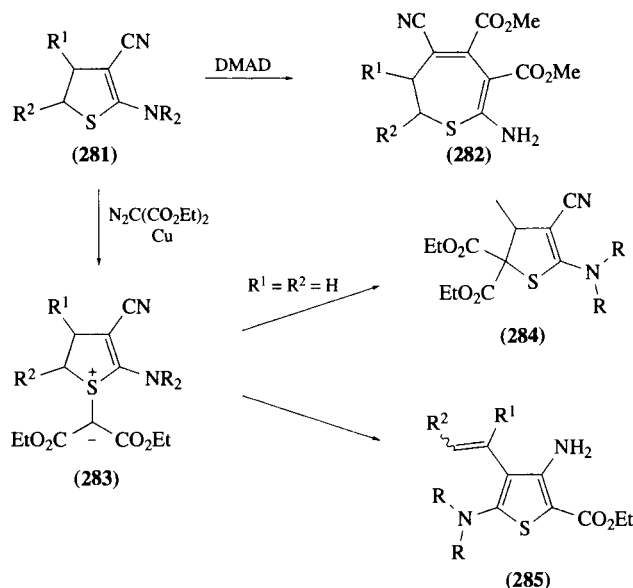


Sensitized photooxygenation of 4,5-dimethyl-2,3-dihydrothiophene has been carried out at -78°C . Catalytic amounts of 2,4-di-*tert*-butylphenol were added as radical inhibitors in order to prevent any free-radical type autoxidation. The major product (82–90%) was the dioxetane (279) along with small quantities of hydroperoxides. Thermolysis of (279) gave the ketoester (280) (Equation (26)). Singlet oxygen appears to attack the double bond of 2,3-dihydrothiophene to form the dioxetane directly, without going through an open zwitterionic intermediate <88JOC1492>. Similarly, dioxetanes from other 4,5-dialkyl-2,3-dihydrothiophenes have been isolated and characterized <91JOC4027>.



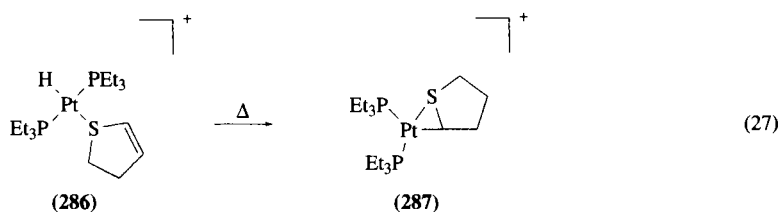
2-Amino-3-cyano-4,5-dihydrothiophenes (281; R = H) react with DMAD in HMPT or DMSO to give the thiopin derivative (282; R = H) by cycloaddition and ring expansion (Scheme 52)

<86CPB396>. Treatment of the enamionitriles (**281**) with diethyl diazomalonate in the presence of copper powder gave the dihydrothiophenium 1-bis(ethoxycarbonyl)methylides (**283**) in good yields. These were thermally stable. Treatment with base led to two types of products, (**284**) and/or (**285**), depending on the nature of R^1 and R^2 . Possible mechanisms for the formation of these products have been discussed. Initial β -elimination may be followed by episulfide formation; subsequent rearrangement can occur in two different modes leading to (**284**) or (**285**) <93LA1263>.

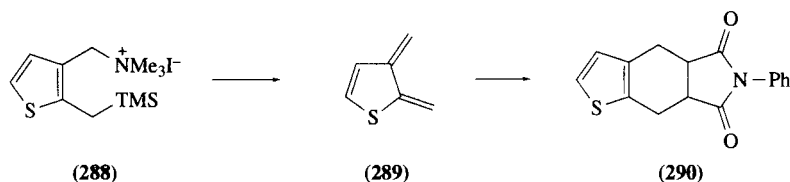


Scheme 52

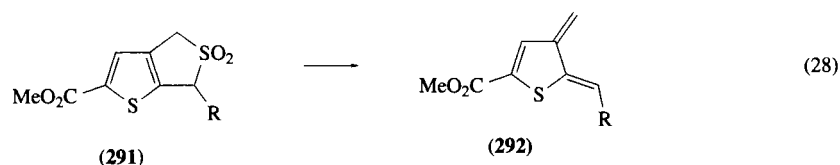
2,3-Dihydrothiophene forms a sulfur-coordinated platinum complex (**286**) on reaction with $HPt(acetone)(PEt_3)_2$. This then slowly rearranges to (**287**), hydrogenation of which gives butane (Equation (27)) <89OM1856>.



(ii) Since 1987, several methods have been reported for the generation of 2,3-dimethylene-2,3-dihydrothiophene (**289**). Treatment of 2,3-bis(bromomethyl)thiophene with sodium iodide in DMF at $80^\circ C$ in the presence of *N*-phenyl maleimide gave the adduct (**290**), indicating that the dimethylene derivative (**289**) had been generated *in situ* (Scheme 53) <87TL6085>. 2,3-Dimethylene-2,3-dihydrobenzo[*b*]thiophene has been generated and trapped in a similar manner <88CB1203>. The fluoride ion-induced fragmentation of (**288**) is a mild and efficient way of generating (**289**) <88TL2689>. In the absence of a dienophile, the bismethylenedihydrothiophene (**289**) appears to dimerize, forming a spiro compound (see below). Such spiro compounds are stable in solution, but polymerize in the neat state. Thermal cheletropic extrusion of SO_2 from the sulfone (**291**; $R = H$) is another route to the generation of 2,3-bismethylenethiophene derivatives such as (**292**) which can be trapped with dienophiles (Equation (28)) <90TL1491>. The advantage of this method is that the sulfone can be regioselectively alkylated to give a variety of 6-alkyl derivatives.

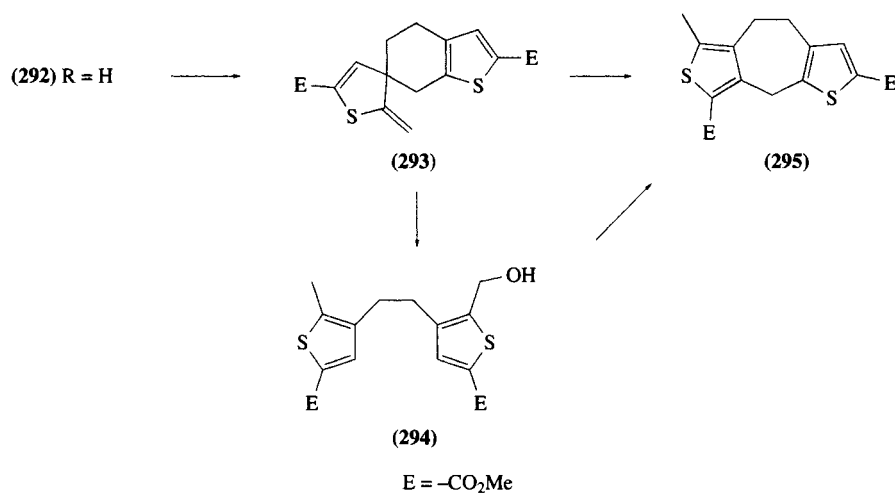


Scheme 53



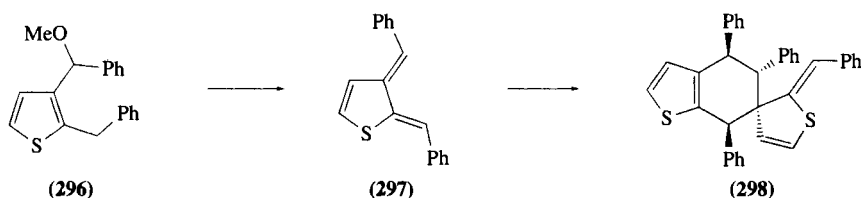
Flash pyrolysis of 2- or 3-chloromethyl-3(or 2)-methylthiophene produces 2,3-bismethylene-2,3-dihydrothiophene (**289**). However, in contrast to the same material produced in solution by the other methods, this could not be trapped by dienophiles. Its presence in the pyrolysate was inferred from the reaction of the pyrolysate with thiophenol, when a mixture of adducts was formed <88TL117>.

Mention has been made earlier of the dimerization of (**289**) to an unstable spiro compound. The stability could be increased by the introduction of an ester group. Dimerization of (**292**) ($R = H$) (prepared from 2,3-bis-(chloromethyl)thiophene-5-carboxylic ester by treatment with NaI in DMF) gave the spiro compound (**293**) (54%) (Scheme 54). The spiro ring could be opened by treatment with acid to form the alcohol (**294**). Reaction of either (**293**) or (**294**) with conc. H_2SO_4 gave the cycloheptene (**295**), thus proving the structure of the spiro compound <91JOC6948>.



Scheme 54

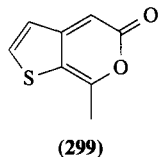
Efforts have been made to stabilize the 2,3-bismethylene-2,3-dihydrothiophene system by introducing suitable substituents. Thus the compound (**297**) with phenyl groups at the exocyclic double bonds was generated by base-catalyzed elimination of methanol from (**296**) <91ACS919>. However, (**297**) also dimerized easily and the only isolable product (30%) was the spiro compound (**298**) (Scheme 55). This corresponds to the *exo*-addition of the diene system to the 3-methylene bond of another molecule. The cycloaddition appears to be orbital symmetry controlled. Only one isomer was obtained. Similarly, (**297**) could be trapped by means of norbornene; cycloaddition led to the *exo*-adduct in 40% yield.



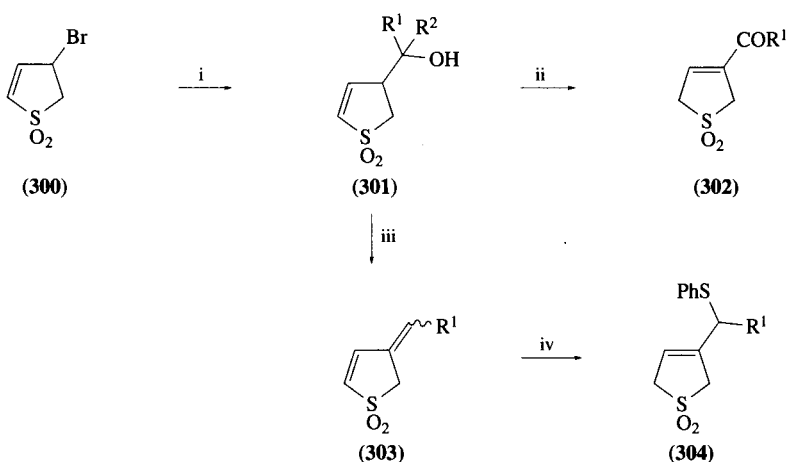
Scheme 55

If the diene system present in (**289**) is made part of a pyrone ring, then the molecule becomes stable. Thus, the molecule (**299**) could be easily prepared from thiophene-3-acetic acid by reaction

with acetic anhydride. This reacts with DMAD to form a [4 + 2]cycloadduct which extrudes CO₂ to form a benzo[*b*]thiophene <88TL5817>. The benzo[*b*]thiophene analogue behaves in a similar manner, leading to a dibenzothiophene. Intramolecular Diels–Alder cycloadditions have also been carried out on suitably substituted derivatives <90JCS(P1)2909>.

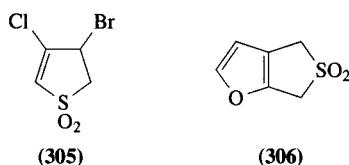


(iii) 2,3-Dihydrothiophene-1,1-dioxides have been studied as precursors to functionalized 2,5-dihydrothiophene-1,1-dioxides; the latter, of course, are latent butadienes. The allylic bromide (300) could be converted to the homoallylic alcohol (301) by reaction with the appropriate aldehyde or ketone in the presence of Zn–Ag (Scheme 56). Excellent yields under mild conditions could be realized by ultrasonic irradiation. The allylzincation reaction leads exclusively to the γ -substituted product. In no case was an α -substituted product detected <87CC1552>. Oxidation of (301; R² = H) with pyridinium chlorochromate gave the rearranged ketone (302). The alcohols (301) could be dehydrated to the butadienes (303) via mesylation. Soft nucleophiles such as PhS[–] added to (303) in the 1,4-mode to give (304). Similar allylzincation of (305) has been used to prepare (306) the precursor to the furan analogue of (289) <92H(34)663>.



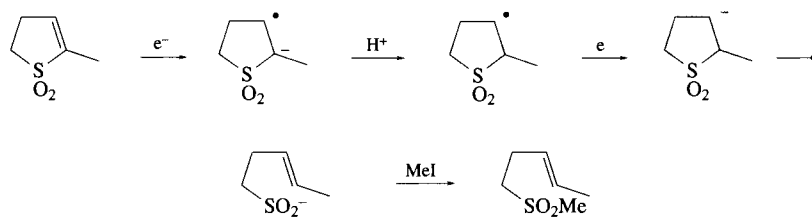
- i, Zn–Ag; R¹COR²
 ii, R² = H; pyridinium chlorochromate
 iii, R² = H, MsCl; LiOH
 iv, PhS[–]
 v, Raney Ni or Bu₃SnH

Scheme 56



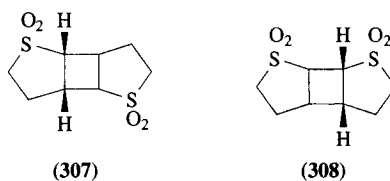
The Diels–Alder cycloaddition of 2,3-dihydrothiophene-1,1-dioxide with open and cyclic 1,3-dienes proceeds with moderate yields <94JCS(P1)927>. FVP of the resultant adducts has been studied.

2,3-Dihydrothiophene-1,1-dioxides undergo reductive C–S bond cleavage and ring opening to furnish acyclic sulfones (after a final alkylation) <92JOC5015>. The reaction is brought about by ultrasonically dispersed potassium in the presence of a proton source, and involves two one-electron reduction steps. The final alkylation converts the sulfinate to a sulfone (Scheme 57).

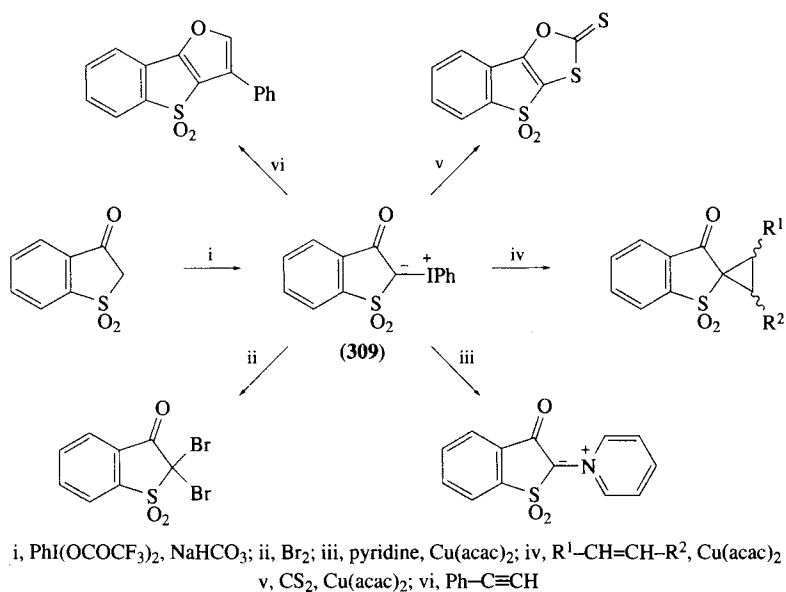


Scheme 57

Irradiation of 2,3-dihydrothiophene-1,1-dioxide (>280 nm) in the presence of acetone as the photosensitizer gives the head-to-tail dimer (**307**) (16%) and the head-to-head dimer (**308**) (8.5%) (<89T1667>).

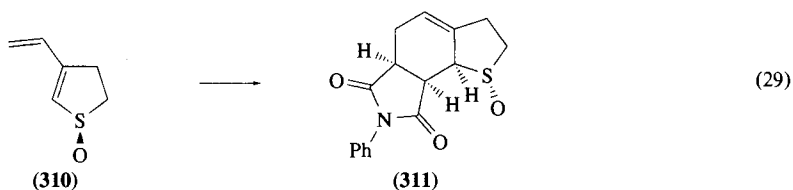


The iodonium ylide (**309**) has been prepared from 3-oxobenzo[*b*]thiophene-1,1-dioxide by treatment with phenyl iodosyl bis(trifluoroacetate). This reacts with nucleophiles to produce various novel molecules (Scheme 58) (<89TL6673>).



Scheme 58

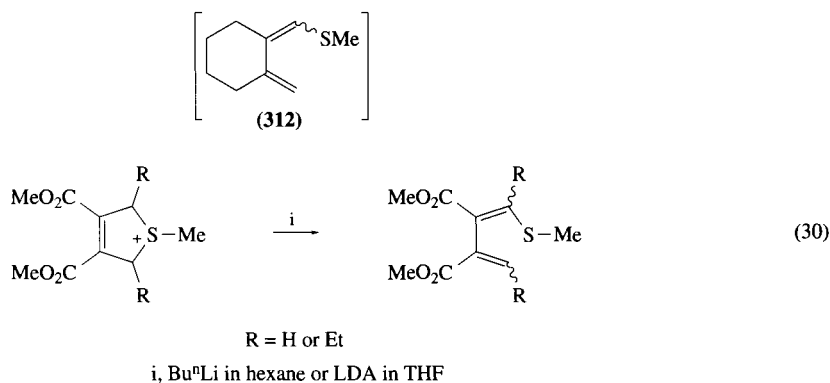
The face selectivity in the Diels–Alder addition of the sulfoxide (**310**) to *N*-phenylmaleimide has been investigated (<88JA4625>). A single cycloadduct (**311**) was formed in good yield (Equation (29)). This was proved to be the *endo* adduct resulting from addition *anti* to the sulfoxide oxygen.



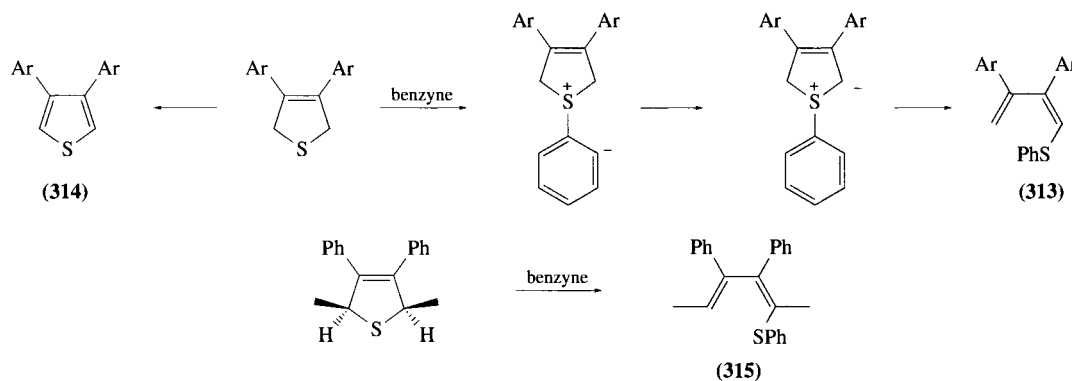
2.10.3.5.2 2,5-Dihydrothiophenes

This section consists of two parts: (i) reactivity of 2,5-dihydrothiophenes and (ii) reactivity and applications of 2,5-dihydrothiophene-1,1-dioxides.

(i) The base-induced ring opening of *S*-methyl-2,5-dihydrothiophenium salts was discussed in CHEC-I <84CHEC-I(4)741>. Some more examples of this kind have been reported <85JCS(P1)1161>. 1,3,4-Trimethyl-2,5-dihydrothiophenium iodide gave 1-methylthio-2,3-dimethyl-1,3-butadiene in 19% yield on treatment with NaH in DMF. 2-Methyl-1,3,4,5,6,7-hexahydrobenzo[*c*]thiophenium salts behaved similarly, giving the butadiene (312) which could be trapped with maleic anhydride. Good yields in the cycloreversion were obtained when ester groups were present at positions 3 and 4 of 2,5-dihydrothiophene (Equation (30)).



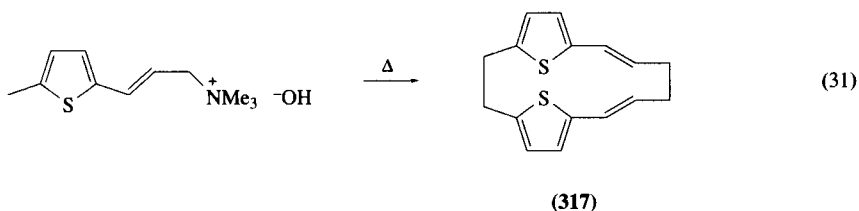
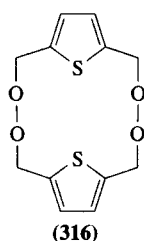
A novel method for preparing 1-phenylthio-2,3-disubstituted butadienes (313) involves reaction of 3,4-diaryl-2,5-dihydrothiophenes with benzyne. The mechanism probably involves initial betaine formation, followed by conversion to the ylide. Disrotatory ring opening would then give the product. The major product has the *Z* configuration (Scheme 59). A minor product is the thiophene (314); this may be formed by a symmetry allowed hydrogen transfer <89TL847>. *cis*-2,5-Dimethyl-3,4-diphenyl-2,5-dihydrothiophene on reaction with benzyne gives the configurationally pure 1-phenylthio-1,3-butadiene (315) in a stereospecific manner; as usual, a small amount of 2,5-dimethyl-3,4-diphenylthiophene (6%) is formed in this instance also.



Scheme 59

The desulfurization of 2,5-dihydrothiophene on Mo(110) has been studied <91JA820>. The reactivity of this appears to be qualitatively different from that of tetrahydrothiophene or thiophene itself; this has been attributed to the fact that the intramolecular elimination process is kinetically favorable for 2,5-dihydrothiophene.

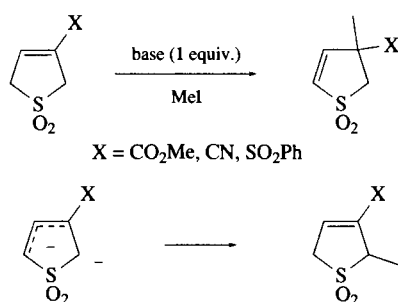
The generation of 2,5-bismethylene-2,5-dihydrothiophene has been discussed in CHEC-I <84CHEC-I(4)741>. ¹H and ¹³C NMR spectra of this compound prepared by FVP of 5-methylthienyl benzoate have been recorded at -75°C <94TL4175>. At room temperature, it is converted to the cyclic dimer (5%) and trimer (30%). In the presence of oxygen (triplet) it forms the cyclic bisperoxide (316). An interesting extension has given the thiophenophane (317) with two double bonds (Equation (31)) <92TL2633>.



(ii) CHEC-I dealt at length with 2,5-dihydrothiophene-1,1-dioxides (3-sulfolenes); thermal disrotatory cheletropic extrusion of sulfur dioxide leads to substituted butadienes with defined configuration. Treatment with excess LAH is a milder alternative to thermolysis. The chemistry of 3-sulfolenes has burgeoned in the last few years because of the variety of substituted butadienes that become accessible by this route. A recent exhaustive review deals with the synthesis, reactivity and applications of 3-sulfolenes <89OPP259>. This has covered the literature up to 1988. Hence only a few significant reactions covered in the review will be reported here, followed by more recent developments in this area. In the following account, reactions for which no specific journal source has been indicated, refer to the above review article.

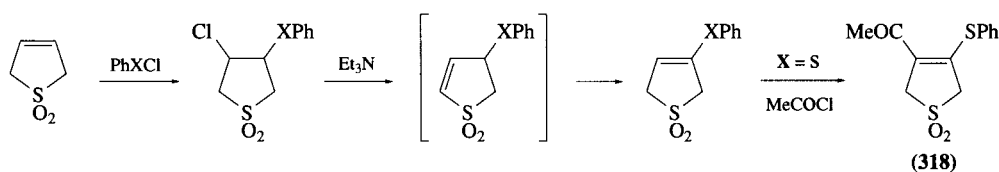
Early attempts at α -substitution in 3-sulfolene by generation of the anion and reaction with electrophiles were not very successful; the reason for this was the occurrence of anionic cycloreversion of 3-sulfolene, leading to butadienyl sulfonates. However, this problem has been tackled successfully by carrying out the deprotonation at -105°C . Another strategy is to use LHMDs as the base in HMPA–THF. Using such procedures, 2-trimethylsilyl as well as 2-tributylstannyl 3-sulfolenes have been prepared <93S943>. The latter undergoes Pd^0 -catalyzed cross-coupling with vinyl iodides to form 2-vinyl-3-sulfolenes. If the sulfolene carries an alkyl or halogen substituent at position 3, deprotonation/alkylation takes place at position 2. On the other hand, with a 3-aryl or 3-trimethylsilyl substituent, the new group enters position 5 of the ring. Sequential 2,5-dialkylation of 3-sulfolene has also been reported; here also, a 3-trimethylsilyl substituent causes a change in the pattern, leading to the 5,5-disubstituted-3-trimethylsilyl-3-sulfolene.

If an electron-withdrawing group (CO_2Me , CN or SO_2Ph) is present at position 3 of 3-sulfolene, only γ -substitution takes place, leading to 2-sulfolenes <90JOC5410> (Scheme 60). However, if the dianion is generated at -78°C and reacted with MeI , then the 2-methyl derivative is produced. The strategy of monoalkylating a dianion has been successfully extended to the synthesis of 2-allyl-3-methoxycarbonyl-3-sulfolene <92SL272>. Similarly, the 2,5-dianion can be generated from 3-trimethylsilyl-3-sulfolene; as expected, the first alkylation on this takes place at position 2 <89OPP259>. The regioselectivity of alkylation of 5-substituted benzo[*c*]thiophene-2,2-dioxides has been investigated; the 5-alkoxy derivative leads to substitution at position 3 <94H(38)41>.



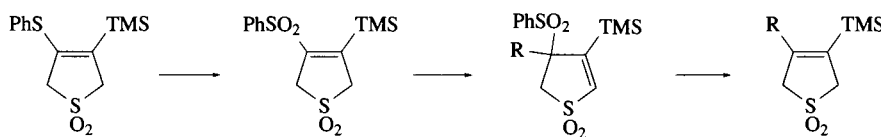
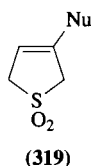
Scheme 60

Introduction of a substituent at position 3 in 3-sulfolene can be achieved in a variety of ways. The most useful method is the addition-elimination reaction (Scheme 61). The phenylthio compound can be acetylated to give (318). Bromine adds to the double bond in 3-sulfolene to form the dibromo derivative, which reacts with nucleophiles to form (319). 3-Sulfolenes undergo Pd-catalyzed coupling with iodoarenes to form 3-aryl-3-sulfolenes. 3-Sulfolene can be nitrated by N_2O_4 to give 3-nitro-3-sulfolene. Several methods have been evolved for introducing heteroatom substituents at positions 3 and 4 of 3-sulfolene (88JOC3027). These essentially depend on a sequence of addition, elimination and isomerization. 3-Alkyl-4-(trimethylsilyl)-3-sulfolenes have been prepared from 3-(phenylthio)-4-(trimethylsilyl)-3-sulfolenes as shown (Scheme 62) (93JOC967). MCPBA oxidation gave the phenylsulfone which was deprotonated with $Bu^{\text{n}}Li$ in THF at -78°C in the presence of HMPA, and then quenched with alkyl halide. Final treatment of the product with Na/Hg and phosphoric acid in THF resulted in desulfonylation. A similar strategy has been used for synthesizing 3,4-dialkyl-3-sulfolenes (90TL1035).



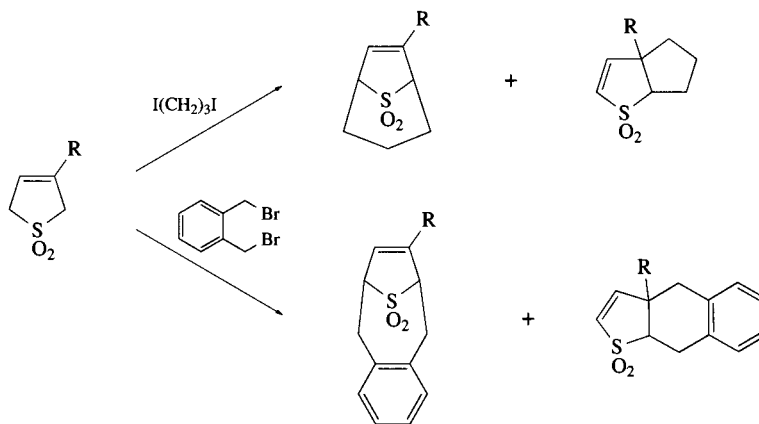
X = S or Se

Scheme 61



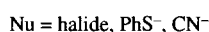
Scheme 62

The reaction of 3-sulfolenes with 1,3-diiodopropane and 1,2-bis(bromomethyl) benzene in the presence of two equivalents of base has been studied in detail (91JOC4560). Both bridged and linearly fused bicyclic systems are formed (Scheme 63). The proportion of the former can be increased by introducing a bulky substituent at position 3.



Scheme 63

Side-chain functionalization of substituted 3-sulfolenes has given a variety of important intermediates (89OPP259). Some examples are given in Scheme 64.



Scheme 64

(320) $\xrightarrow[100-130\text{ }^{\circ}\text{C}]{\Delta}$ (321)

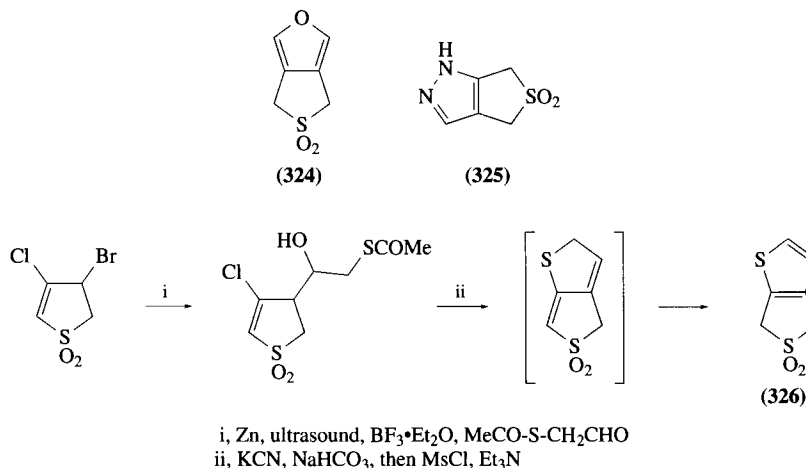
Ar =

(322) R = H or Me (323)

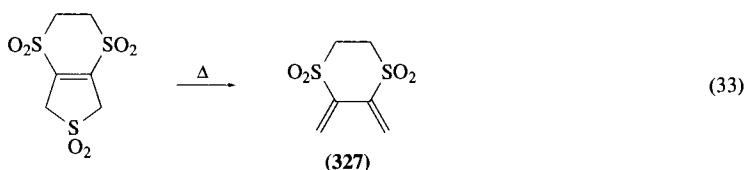
Scheme 65

2.10.3.6 Tetrahydrothiophenes

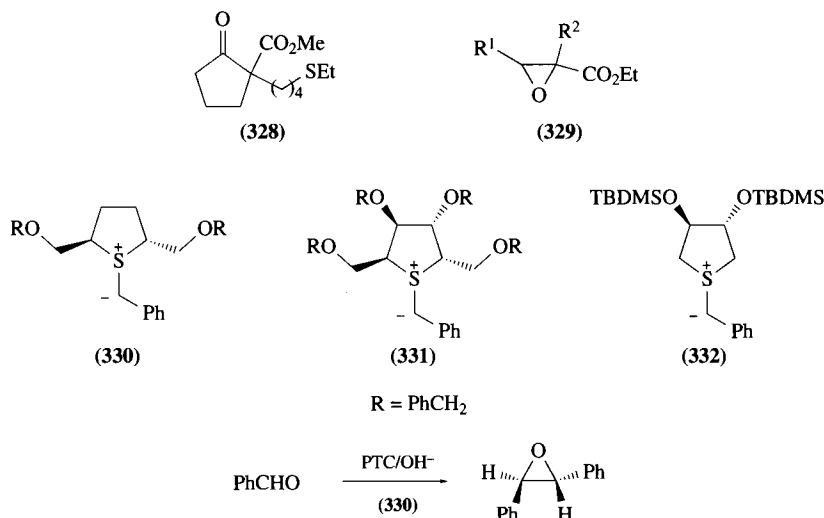
The reactivity of tetrahydrothiophene (thiolane), tetrahydrothiophene-1-oxide and tetrahydrothiophene-1,1-dioxide (sulfolane) have been discussed in detail in CHEC-I <84CHEC-I(4)741>. The present review will therefore deal only with the more recent results in this area.



Scheme 66

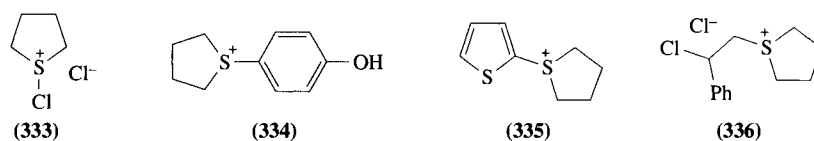


The kinetics of the sulfur inversion process in an *S*-phenylthiolanium compound has been investigated by ^1H NMR spectroscopy <88BCJ707>. *S*-Ethylthiolanium fluoborate reacts with enolates derived from β -ketoesters to give ring-opened products (e.g. (328)) <83JOC8>. Thiolanium ylides can be used for the synthesis of α,β -epoxyesters from aldehydes <87SCI593>. The only product isolated (e.g. (329)) has the *E* configuration at the epoxide. *S*-Benzyl ylides derived from C_2 symmetric thiolanes have been used for preparing chiral epoxides <90TL35>. Benzaldehydes give *trans*-stilbene oxides with up to 83% ee (Scheme 67). The ylides that have been used for this reaction are (330), (331), and (332). Surprisingly, ylides (330) and (332), both afford the (*R,R*) epoxide.

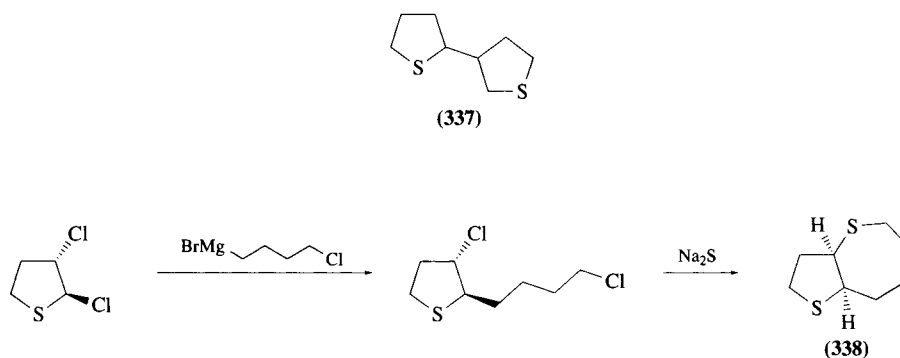


Scheme 67

The chlorination of tetrahydrothiophene by NCS has been investigated in detail <85T3845>. The product can be either 2-chlorotetrahydrothiophene or 2,3-dichlorotetrahydrothiophene depending on the polarity of the solvent. Nonpolar solvents (benzene) favor the former, while the dichloro compound is formed in more polar solvents (CH_2Cl_2). The reactivity of the chlorine complex (333) of tetrahydrothiophene has been studied in detail <85JOC2840>. Electrophilic substitution on phenol gives the sulfonium salt (334), the substitution taking place at the *para* position; if the *para* position is blocked, substitution takes place at the *ortho* position. With thiophene, it forms (335), the best solvent being liquid SO_2 . The *S*-chloro compound also adds to double bonds, styrene giving (336) in 85% yield. As discussed in CHEC-I the decomposition of (333) seems to give initially 2-chlorotetrahydrothiophene and 2,3-dihydrothiophene. The latter can react with another molecule of (333) at the double bond.

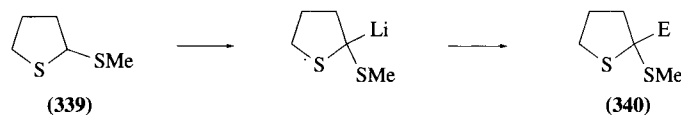


Refluxing tetrahydrothiophene with TiCl_4 for several hours produces a small quantity of (337). The yield of this could be improved by deliberately introducing some 2,3-dihydrothiophene into the above reaction mixture <86JCS(P1)1861>. A radical mechanism has been proposed for the formation of (337). *trans*-2,3-Dichlorotetrahydrothiophene has been used as the starting material for several fused bicyclic systems <91JCS(P1)285>. Initial nucleophilic substitution takes place at position 2. Thus reaction with a Grignard reagent in benzene-ether as a solvent, followed by treatment with Na_2S in DMSO gives (338) (Scheme 68). The first displacement probably proceeds by an $\text{S}_{\text{N}}1$ mechanism.



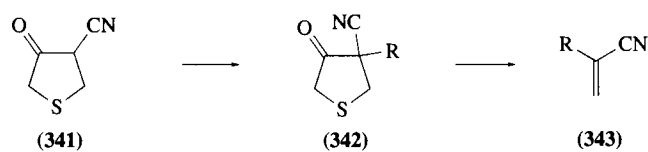
Scheme 68

The direct replacement of chlorine in 2-chlorotetrahydrothiophene by means of MeSH leads to low yields of (339). However, reaction with diphenylacetic acid and Et_3N gives the diphenylacetic ester which can be converted to (339) in excellent yield by MeSH . Alternatively, 2-mercaptotetrahydrothiophene can be prepared by the thiourea method (CHEC-I) and methylated to give (339) <92CB1641>. Deprotonation of (339) with Bu^nLi at -30°C gives the 2-lithio derivative; this reacts with a variety of electrophiles (alkyl halides, carbonyl compounds) to form (340) (Scheme 69).

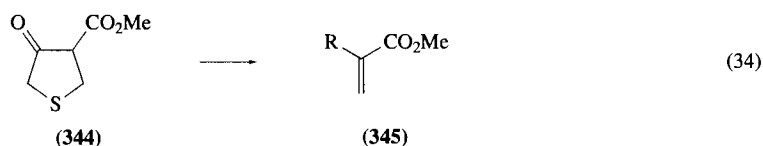


Scheme 69

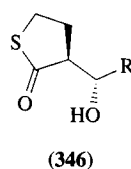
The β -ketonitrile (341) can be *C*-alkylated to (342) (with only small amounts of the *O*-alkylated product), which can be converted to α -substituted acrylonitriles (343) by alkali treatment (Scheme 70) <85S969>. The acrylic esters (345) can be prepared by a similar sequence from the β -ketoester (344) (Equation (34)) <82CC1265>.



Scheme 70

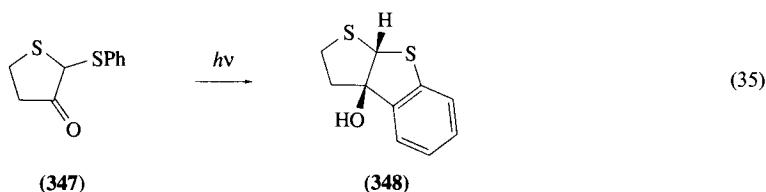


The lithium enolate derived from γ -thiobutyrolactone reacts with various aliphatic aldehydes to form the aldols (**346**) with high *anti* selectivity, but benzaldehyde gives a mixture of *syn* and *anti* aldols in a 1 : 1 ratio <90TL2453>. The silyl ketene acetal from γ -thiobutyrolactone led to *syn*-aldols in a fluoride ion-catalyzed reaction.



Pyrolysis of either 2-benzoyloxytetrahydrothiophene or 2-acetoxytetrahydrothiophene gives 2,3-dihydrothiophene in good yields <86JCS(P1)1861, 86JOC113>.

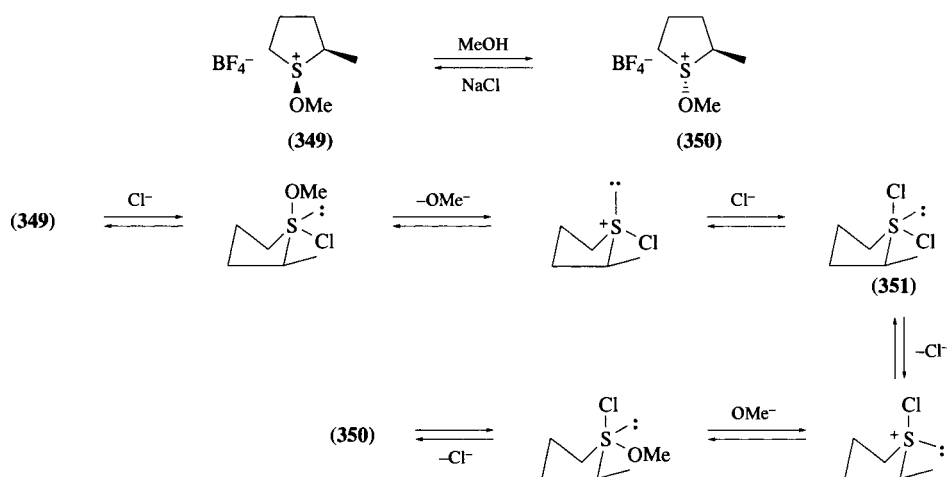
Irradiation of 2-phenylthio-3-oxotetrahydrothiophene (**347**) in MeCN gives the tricyclic compound (**348**) in 43% yield (Equation (35)) <82T75>. The mechanism probably involves a conrotatory photocyclization of the enol form of (**347**) to produce a thiocarbonylylide, followed by intramolecular protonation.



$\text{H}_2\text{O}_2/\text{V}_2\text{O}_5/t\text{-BuOH}$ is a good reagent for oxidizing tetrahydrothiophene derivatives to the corresponding 1-oxides <87T249>. The stability, stereochemical preference and reactivity of diastereomeric 1-methoxy-2-methylthiolanium fluoroborates (**349**) and (**350**) have been investigated in detail <93T8407>. Authentic samples of *cis*-(**349**) and *trans*-(**350**) isomers were prepared by methylation of the corresponding sulfoxides by $(\text{Me}_3\text{O})^+\text{BF}_4^-$. In methanol solution (or CD_3OD), in the presence of NaCl, an equilibrium is established between the two diastereomers. At equilibrium, the *cis* : *trans* ratio is about 70 : 30. The same mixture is also obtained when 2-methyltetrahydrothiophene is treated with *t*-BuOCl at -78°C in methanol and the products isolated as fluoroborates. The equilibration probably takes place via a common dichlorosulfurane intermediate (**351**) (Scheme 71). The relative thermodynamic stability of the *cis*-isomer has been attributed to a favorable $\sigma(\text{C}-\text{H})-\sigma^*(\text{S}-\text{O})$ stereoelectronic effect.

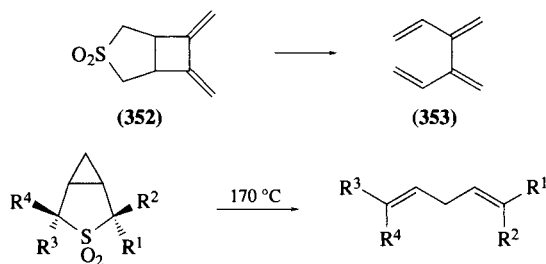
Tetrahydrothiophene-1-oxide has been sequentially alkylated at positions 2 and 5 <92CPB1961>. The base used for deprotonation in the first step was NaH/THF, and in the second, Bu^nLi /THF.

The pyrolysis of sulfolanes has been discussed in CHEC-I. This has been utilized for the generation of [4]dendralene (**353**) having two overlapping cross-conjugated diene systems <85CC1785>. The photoadduct of 3-sulfolene and maleic anhydride was converted by a standard series of reactions to (**352**), FVP of which (550°C , 10^{-3} mmHg) gave (**353**). The diene (**352**) can also be reacted with dienophiles, and the resulting cycloadducts pyrolyzed to form hexatrienes; these would undergo electrocyclization to yield cyclohexadienes. Thermolysis of 3-thiabicyclo [3.1.0]hexane 3,3-dioxides



Scheme 71

leads to extrusion of SO_2 and formation of 1,4-dienes. This fragmentation (Scheme 72) has now been shown to occur by a concerted disrotatory mode $\langle 94\text{H}(38)35 \rangle$.



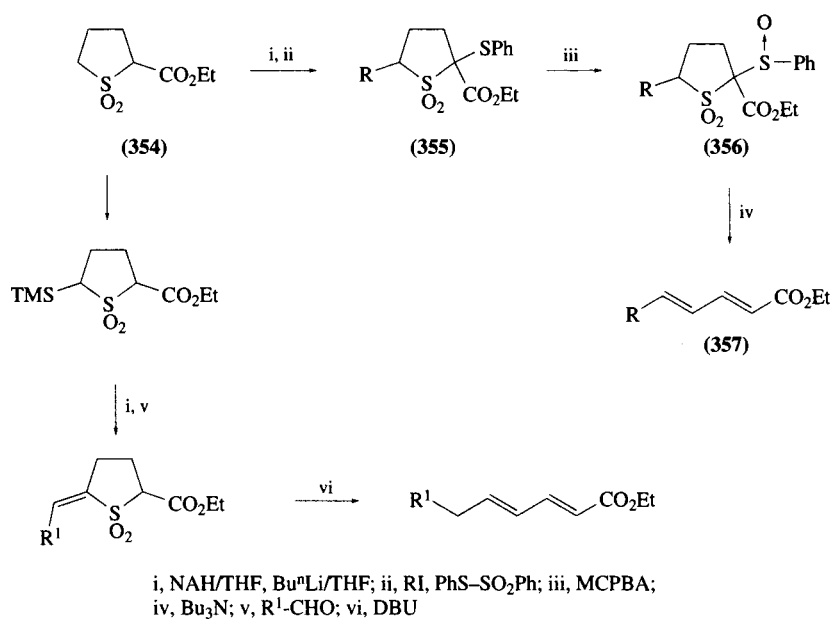
Scheme 72

Several strategies have been evolved for the generation of substituted 3-sulfolenes from sulfolanes. The ultimate goal is of course to subject the 3-sulfolenes to thermal cheletropic extrusion of SO_2 , leaving substituted butadienes. In one such method, 2-carbethoxysulfolane (**354**) is converted to its dianion by first refluxing with NaH in THF for 5 min and then treating with Bu^nLi at 0°C . Two alternative routes have been developed from this dianion $\langle 89\text{CL}1203 \rangle$. In the first method, the dianion is successively treated with alkyl iodide and then with *S*-phenyl benzenethiosulfonate to form (**355**). This is then carefully oxidized with MCPBA to the sulfoxide (**356**). Final heating with tributylamine results in elimination of sulfenic acid to form the 2-sulfolene, isomerization to the 3-sulfolene and cheletropic ring opening to give (**357**) (Scheme 73). The second route involves initial trimethylsilylation at position 5, and then a Peterson reaction; subsequent base-induced *endo*-migration of the double bond completes the sequence (Scheme 73).

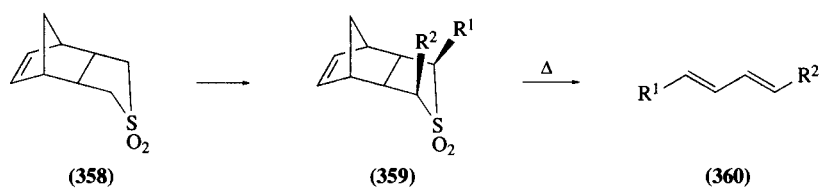
Yet another ingenious method of stereospecifically synthesizing *cis*-2,5-dialkylated 3-sulfolenes is to start with the sulfolane (**358**). Sequential alkylation ($\text{Bu}^n\text{Li}/\text{RX}$) leads stereospecifically to *cis*-(**359**). A retro Diels–Alder reaction followed by cheletropic extrusion of SO_2 , results in (**360**) (Scheme 74) $\langle 83\text{TL}1247 \rangle$.

The β -ketonitrile (**341**) has been transformed into 3-cyano-3-sulfolene $\langle 88\text{T}6451 \rangle$. This is a stable precursor of 2-cyanobuta-1,3-diene.

Appropriately substituted tetrahydrothiophenes have been used as starting materials for 3-sulfolenes which are fused to other heterocycles. As mentioned in the previous section, these are convenient precursors for *ortho*-bismethylene heterocycles. Thus the masked β -ketoaldehyde (**361**) with hydrazine hydrate followed by acid treatment, gave the bicyclic compound (**362**), which could be oxidized by MCPBA to the 3-sulfolene (**363**) (Scheme 75) $\langle 93\text{JOC}493 \rangle$. Similarly, treatment of the ketoaldehyde derived from (**361**) with hydroxylamine gave the isoxazole (**364**) which could be

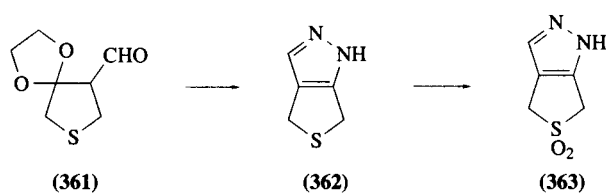


Scheme 73

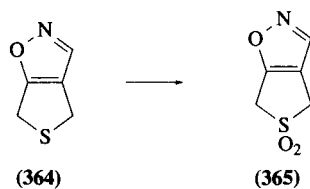


Scheme 74

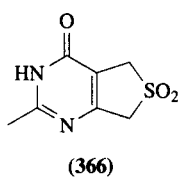
oxidized to the sulfolene (365) (Equation (36)) <92TL8121>. The pyrimidine-fused sulfolene (366) has been prepared from the β -ketoester (344) <93SL347>. Careful treatment of (367) with a molar equivalent of PCl₃ at room temperature leads to the oxazole fused 3-sulfolene (368) (Equation (37)) <94JOC2241>.

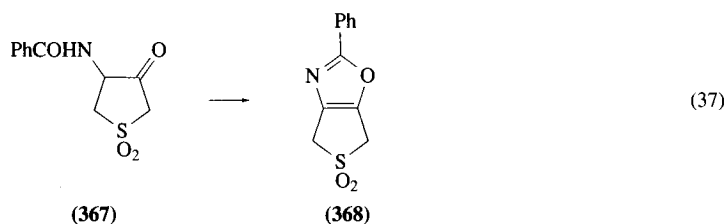


Scheme 75



(36)





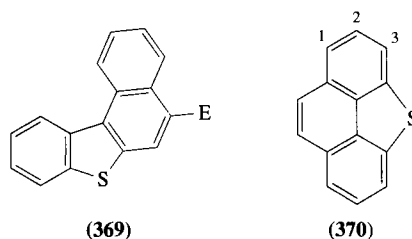
2.10.4 REACTIVITY OF SUBSTITUENTS ATTACHED TO THE RING CARBON ATOMS

2.10.4.1 Fused Benzene Rings

2.10.4.1.1 Electrophilic attack

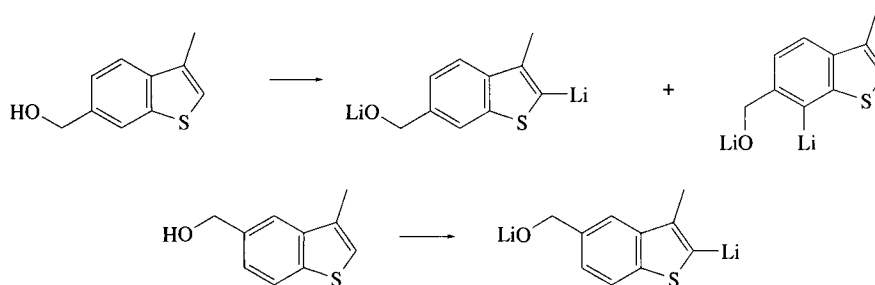
Several standard electrophilic reactions have been carried out on dibenzothiophene <85JHC215>. Nitration with conc. HNO_3 and glacial acetic acid leads to a mixture (40% each) of 2-nitrodibenzothiophene and dibenzothiophene-5-oxide. Nitration of the latter (fuming nitric acid and conc. sulfuric acid) gives 3-nitrodibenzothiophene-5-oxide, which could be deoxygenated to 3-nitrodibenzothiophene by 48% hydrobromic acid and glacial acetic acid.

Friedel–Crafts acylation, nitration, sulfonation, and bromination of benzo[*b*]naphtho[1,2-*d*]thiophene take place at the position shown in (369) <84PJC1243>. Nitration as well as Friedel–Crafts acetylation of phenanthro[4,5-*bcd*]thiophene (370) give mixtures of 1- and 3-monosubstituted derivatives <90JHC1969>.



2.10.4.1.2 Nucleophilic attack

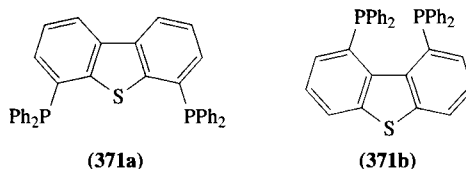
Lithiation of 3-methylbenzo[*b*]thiophene with $\text{Bu}^\text{t}\text{Li}$ takes place exclusively at position 2. However, 6-hydroxymethyl-3-methylbenzo[*b*]thiophene, on treatment with 2.5 equiv. of $\text{Bu}^\text{t}\text{Li}$, gives a mixture of the 2-lithio and 7-lithio derivatives in the ratio 2 : 1. This unprecedented direct lithiation on the benzene ring of benzo[*b*]thiophene has been attributed to the cooperative effect of the alkoxide anion and the ring sulfur <85H(23)2391>. In confirmation of this, the 5-hydroxymethyl analogue lithiates exclusively at position 2 (Scheme 76). Surprisingly, if the 3-methyl group is not present, then the major product of lithiation of the 6-(hydroxymethyl)benzo[*b*]thiophene is the 2-lithio derivative (67%), with only a 3.5% yield of the 7-lithio derivative. This has been attributed to the inductive effect of the 3-methyl group which would decrease the acidity of the 2-proton.



Scheme 76

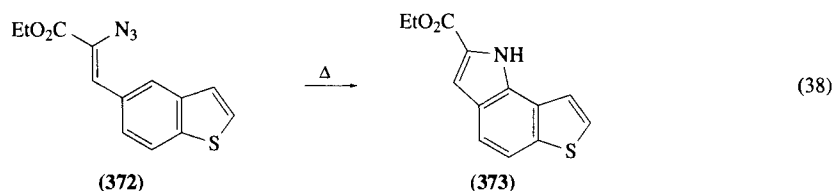
Katritzky and Perumal have made a systematic study of the lithiation of dibenzothiophene and its derivatives <90JHC1737>. BuⁿLi lithiates unsubstituted dibenzothiophene at position 4. With excess BuⁿLi, over a prolonged period, about 15–20% of 4,6-dilithiodibenzothiophene is formed. 4-Methyldibenzothiophene lithiates on the methyl group, but surprisingly, the 4-ethyl derivative lithiates at position 6. The 4-lithio derivative has been converted to 4-bromodibenzothiophene by means of CNBr and to 4-chlorodibenzothiophene by treatment with *p*-toluenesulfonyl chloride. Dibenzothiophene thiols have been prepared by treating the lithio derivatives with tetra-isopropylthiuram disulfide, followed by hydrolysis <87JHC749>. Similarly, thioethers have been synthesized by reacting the lithio derivatives with disulfides.

In a surprising development, it has been found that treating dibenzothiophene with 2 equiv. each of BuⁿLi and TMEDA in *n*-heptane at 0°C gives the 4-lithiated product, but when the temperature is raised to the reflux temperature, 4,6-dilithio and 1,9-dilithiodibenzothiophene are formed in the ratio 3 : 1. The latter probably arises by intermolecular transmetalation of the initially formed 4,9-dilithio isomer. Reaction of the dilithio derivatives with Ph₂PCl has given 4,6-bis(diphenylphosphino) dibenzothiophene (**371a**) and 1,9-bis(diphenylphosphino)dibenzothiophene (**371b**) <93TL2107>. In the mass spectrum, (**371b**) shows a fragment ion by the loss of a phenyl group; this probably takes place with the concomitant formation of a P—P bond (see also Section 2.10.4.1.4).



2.10.4.1.3 Reactions with radicals and electron-deficient species

Thermolysis of the azidocinnamate (**372**) gave ethyl thieno[2,3-*g*] indole-2-carboxylate (**373**) in 78% yield (Equation (38)) <89JCS(P1)603>.



Cathodic reduction of dibenzothiophene in the presence of a weak acid such as phenol, leads to the formation of the 1,4-dihydro derivative in 75% yield <89BSF699>.

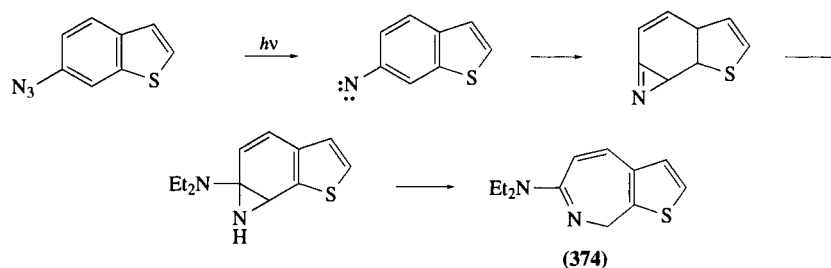
2.10.4.1.4 Reactions of substituents on benzene rings

The behavior of a nitrene attached to positions 4,5,6 or 7 of benzo[*b*]thiophene has been discussed in CHEC-I <84CHEC-I(4)741>. The 6-azide was unique in that, on irradiation in the presence of Et₂NH, it gave rise to the ring-expanded product (**374**) via the nitrene, azirine and aziridine (Scheme 77). The corresponding 2,3-dihydrobenzo[*b*]thiophene derivative provided a surprise <83TL5147, 88JCS(P1)1933>. Photolysis of the azide in a mixture of Et₂NH and THF (which stabilizes singlet nitrenes) gave a 49% yield of the azepine derivative (**376**), presumably via the azirine (**375**). The expected products from the isomeric azirine (**377**) were not detected (Scheme 78).

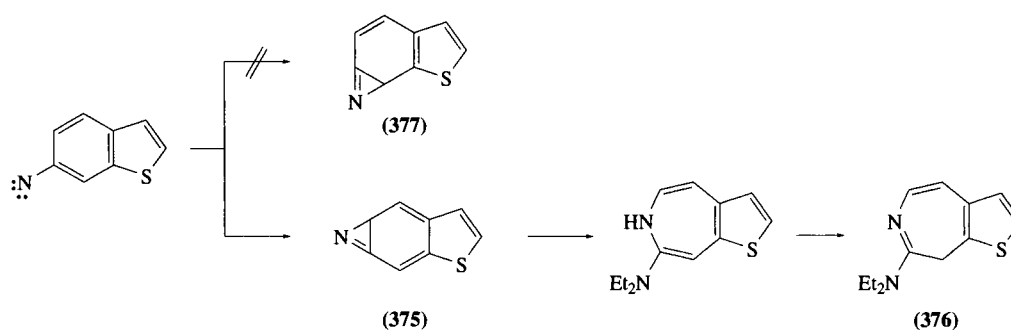
Quinones related to benzo[*b*]thiophene have been included in a review of heterocyclic quinones <89AHC(45)37>. Anodic oxidation of 4-methoxybenzo[*b*]thiophene to form the quinone bisketal has been discussed in CHEC-I <84CHEC-I(4)741>. The method has been extended to give (**378**) in 85% yield <83ACR74, 83JOC4312>. CAN oxidation of 4,7-dimethoxy-1,3-diphenylbenzo[*c*]thiophene gives the quinone (**379**) in 80% yield <89BSF708>.

1-, 2-, 3-, or 4-Bromodibenzothiophene could be converted to the corresponding hydroxy derivative by treatment with copper powder and barium hydroxide or sodium hydroxide <85JHC215>.

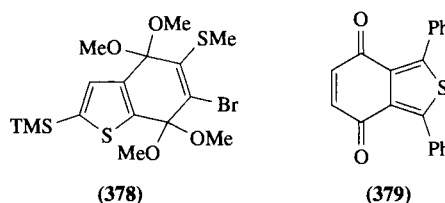
The fluorine atom at position 5 of 4,5,6,7-tetrafluorobenzo[*c*]thiophene can be displaced by



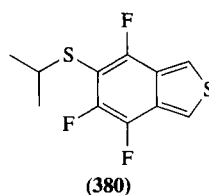
Scheme 77



Scheme 78



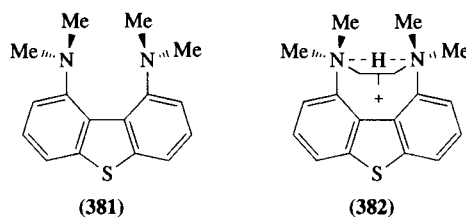
lithium propane-2-thiolate to form (380) <90JCS(P1)1919>. The tetrafluorobenzo[*c*]thiophene itself is reasonably stable.



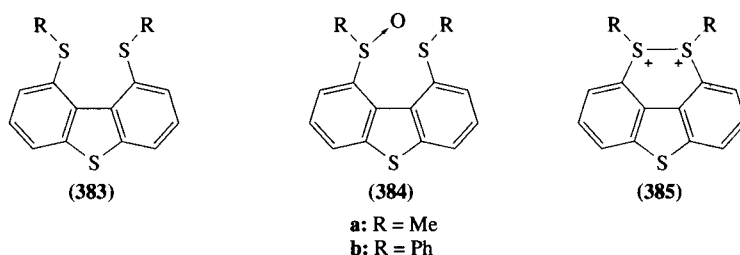
In a competitive halogen–Li exchange reaction, it has been found that 4-bromo-3-methylbenzo[*b*]thiophene exchanges much faster than 6-bromo-3-methylbenzo[*b*]thiophene; this has been attributed to the relief of steric strain in the former <85H(23)2391>. In 2,4- or 2,6-dibromobenzo[*b*]thiophene, selective removal of the 2-bromo substituent is possible. Treatment with one equivalent of BuⁿLi followed by hydrolysis gives 4- or 6-bromobenzo[*b*]thiophene <85H(23)2391>.

Extremely interesting results have been obtained on the behavior and properties of sterically congested 1,9-disubstituted dibenzothiophenes. 1,9-Bis(dimethylamino)dibenzothiophene (381) has been synthesized as a member of the family of “proton sponges” <88TL1905>. As expected, even with an excess of tetrafluoroboric acid, it forms only the monoprotonated species (382). The basicity of (381) is quite high ($\text{p}K_{\text{a}} = 11.9 \pm 0.1$); this falls in between the corresponding fluorene and phenanthrene analogues. X-ray crystal structures of (381) and (382) reveal that there is a strong steric strain in (381) due to the repulsive interaction between the dimethylamino groups. On the other

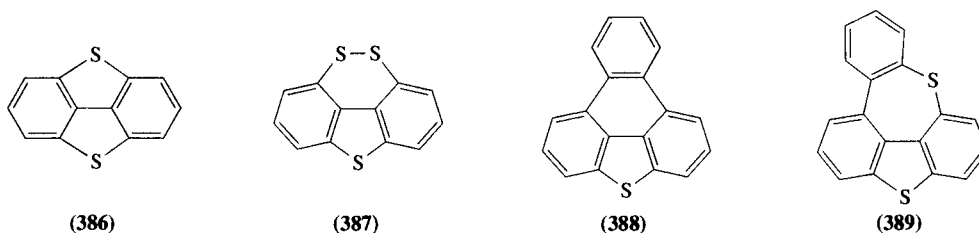
hand, there is a release of steric strain in (382) due to the introduction of a very strong $\text{N} \cdots \text{H} \cdots \text{N}$ hydrogen bond consequent on protonation.



Sterically congested 1,9-bis(methylthio)dibenzothiophene (383a) and 1,9-bis(phenylthio)dibenzothiophene (383b) have been prepared, and oxidized by MCPBA to the respective monosulfoxides (384a) and (384b). In the ^1H NMR spectrum in CDCl_3 (384a) shows two singlets at 2.79 ppm and 2.33 ppm for the two methyl groups. However, on dissolution in conc. D_2SO_4 , the two peaks are shifted downfield and merge to exhibit only one singlet at 3.04 ppm. Quenching this solution with ice-water regenerated the sulfoxide (384a). This suggests the formation of the dication (385a) in conc. D_2SO_4 . This has been confirmed by deuteration experiments. Interestingly, (383a) also, on dissolving in D_2SO_4 , is oxidized to (385a). Cyclic voltammograms confirm these findings. In a similar manner, dissolution of (383b) or the sulfoxide (384b) in conc. D_2SO_4 generates the dication (385b) <92TL1489, 92H(32)101>.



Thermolysis of (383a) gives (386) (<1%) and (387) (61%), whereas photolysis in benzene leads to (386) (11%) and (387) (16%). In contrast, photolysis of (383b) gives (388) (72%) and (389) (26%) <92TL6355, 93H(35)53>. Photolysis of 1-(phenylthio)dibenzothiophene did not proceed under similar conditions, suggesting that a strong S—S interaction is a necessary precondition for this type of cyclization.

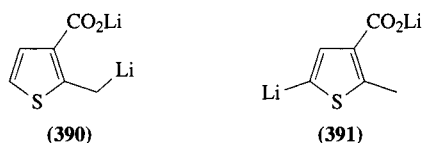


2.10.4.2 Other C-Linked Substituents

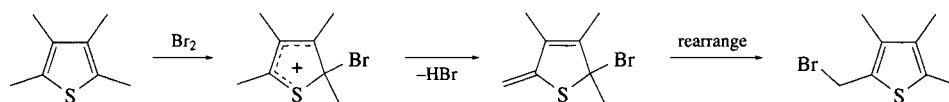
2.10.4.2.1 Alkyl, alkenyl and alkynyl groups

Regioselective side-chain lithiation could be carried out on 2-methylthiophene-3-carboxylic acid. This is in contrast to the reported behavior of the isomeric 3-methylthiophene-2-carboxylic acid which gives a 35:65 mixture of side-chain and nuclear (at position 5) lithiation <80JOC4528>. Dilithiation of 2-methylthiophene-3-carboxylic acid with 2 equiv. LDA in THF at -30°C gave only the dilithium derivative (390) (80%), which could be reacted with electrophiles at the methylene

carbon <89BCJ2725>. Surprisingly, if Bu^nLi were used for the lithiation instead of LDA, there was no regioselectivity; (391) was formed along with (390) in 1 : 1 ratio.



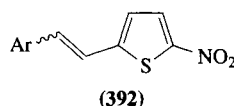
The side-chain halogenation of tetraalkyl substituted thiophenes has been studied in detail <93TL5725>. Nuncatalyzed bromination takes place regioselectively at the α -position. Thus tetramethylthiophene reacts with bromine (1 equiv., CCl_4 , -18°C) to give 2-bromomethyl-3,4,5-trimethylthiophene in 76% yield. If 2 equiv. bromine are used, then 2,5-bis(bromomethyl)-3,4-dimethylthiophene is formed almost quantitatively. Similarly, side-chain monochlorination can be brought about by 1 equiv. NCS or SO_2Cl_2 at -18°C . Dichlorination can be achieved by using 2 equiv. SO_2Cl_2 . With excess SO_2Cl_2 , even tetrachlorination is possible. These reactions probably proceed by an ionic mechanism as shown in Scheme 79. If one of the ring carbons is unsubstituted, then ring halogenation can occur predominantly.



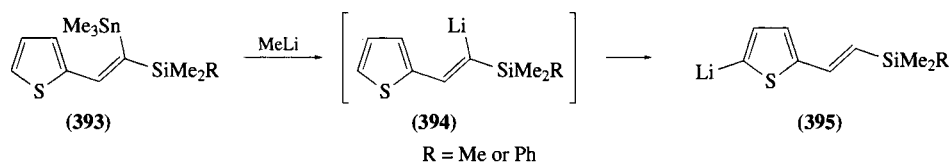
Scheme 79

2-Methyl-5-nitrothiophene condenses with DMF acetal to give the enamine which is converted to 5-nitro-2-thienylacetonitrile on reaction with excess hydroxylamine-*O*-sulfonic acid in water <88CCC114>.

Activated aromatic compounds can be alkenylated with (*Z*)-2-bromo-1-(5-nitro-2-thienyl)ethylene in the presence of aluminum chloride to form (392) <86CCC1127>.



Stereodefined 2-(2-thienyl)-1-silyl-1-stannylethenes have been made use of in synthesizing several 2-alkenyl-5-substituted thiophenes <90G457, 91SC1875>. The basis for this is the rearrangement of the ethenyllithium (394) (derived from (393)) to (395) (Scheme 80). The latter can be subjected to reaction with a variety of electrophiles.

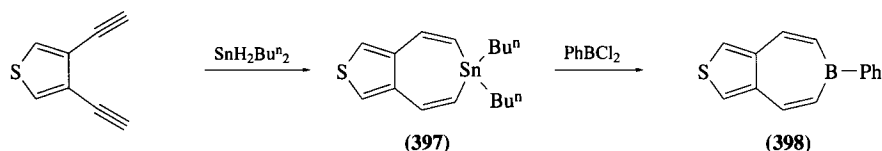
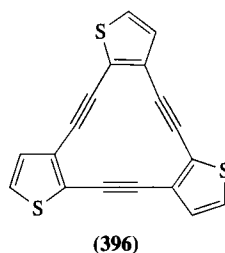


Scheme 80

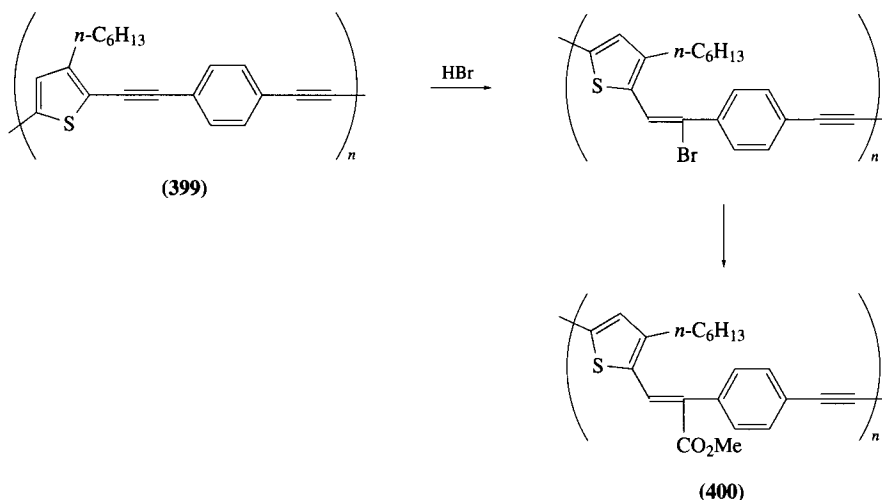
3-Ethynyl-2-iodothiophene has been converted to the copper acetylide and thence to the cyclo-triynes (396) by refluxing in pyridine under anaerobic conditions <90SL427>. This type of reaction has also been referred to in Section 2.10.4.7.3.

3,4-Bisethynylthiophene has been converted to the tin derivative (397) by treatment with dibutyltin hydride, KOH and 18-crown-6 in benzene (85% yield). Reaction of this with PhBCl_2 for 10 min gave 1-phenylthieno [3,4-*d*]borepine (398) in 40% yield (Scheme 81) <92JA1479>.

The triple bond in soluble polymers of structure (399) can be functionalized by initial addition of HBr, followed by Ni(0)-catalyzed carbonylation of the vinyl bromide. Final methanolysis gives the ester (400) (Scheme 82) <93CL1959>.



Scheme 81



Scheme 82

2.10.4.2.2 Substituted alkyl groups

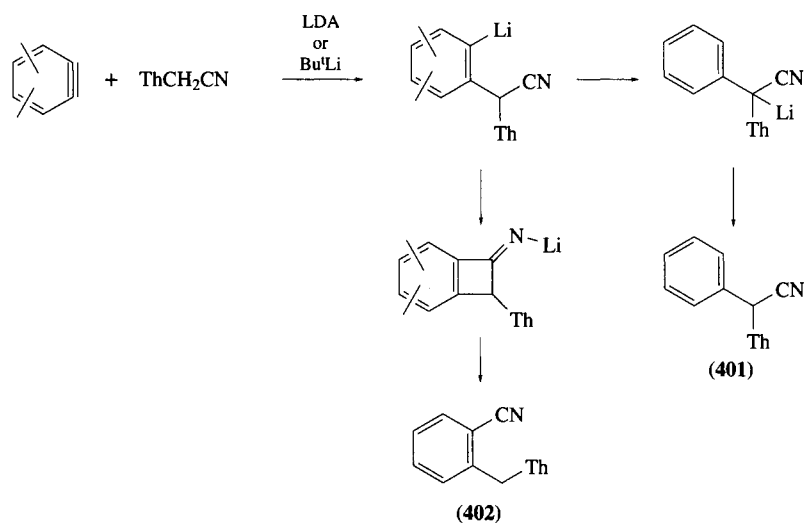
This section deals with the generation and reactivity of carbanions, carbenium ions and carbon radicals adjacent to the thiophene ring. A few miscellaneous substituted alkyl groups will also be covered.

(i) Carbanions

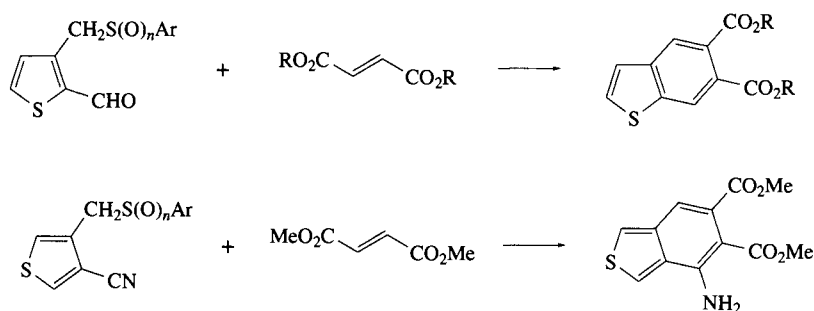
The addition of the anion generated from 2-thienylacetonitrile and 3-thienylacetonitrile to various substituted benzyne has been investigated <92H(34)1239>. Depending on the substitution pattern, either the arylation product (**401**) or the rearranged compound (**402**) could predominate in the reaction product (Scheme 83).

An extremely interesting synthesis of benzo[*b*]thiophenes and benzo[*c*]thiophenes is based on the concept of building a benzene ring on a preexisting, suitably substituted thiophene substrate <86JOC230>. The thiophene ring carries an electrophilic substituent (CHO, CO₂Et, CN) *ortho* to a potential carbon nucleophile (CH₂SOAr or CH₂SO₂Ar). The reaction is initiated by generation of the carbanion by means of a base, Michael addition to an acceptor, cyclization on the electrophilic group and final elimination of arenesulfinic or arenesulfenic acid (Scheme 84). Yields are between 50 and 60%.

The central methine proton in 1,1-bis(5-acetyl-2-thienyl)ethane is acidic; the corresponding anion can be generated by NaH–THF. Alkylation can be carried out with several alkyl halides, but



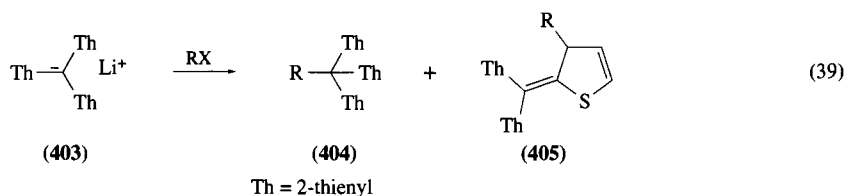
Scheme 83



Scheme 84

Michael addition gives poor yields of the adducts. There appears to be severe steric inhibition to reactions at the bridge carbon <94JCR(S)16>.

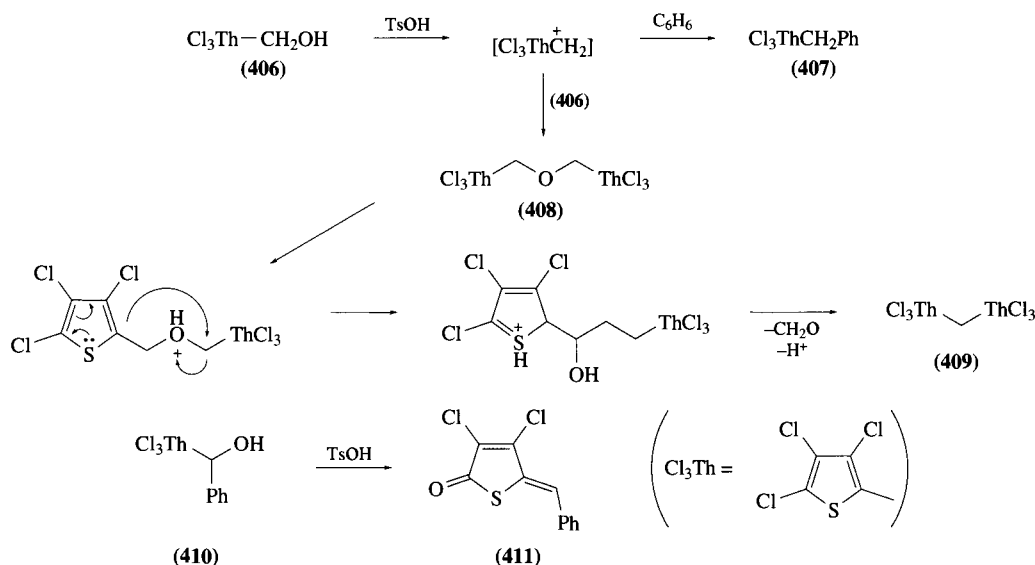
Treatment of tri-(2-thienyl)methane with Bu^nLi (1.3 equiv.) in the presence of TMEDA (1.5 equiv.) in THF at -78°C gives exclusively the tri-(2-thienyl)methylithium (**403**) (96%) without any nuclear lithiation <92CL703>. This lithiation is faster than that of triphenylmethane. Treatment of (**403**) with primary alkyl halides leads to alkylation at the carbanion center, forming (**404**). However, secondary alkyl halides give mixtures of (**404**) and (**405**) (Equation (39)). With tertiary alkyl bromides, elimination is the predominant reaction, leading to recovery of tri-(2-thienyl)methane.



(ii) Carbenium ions

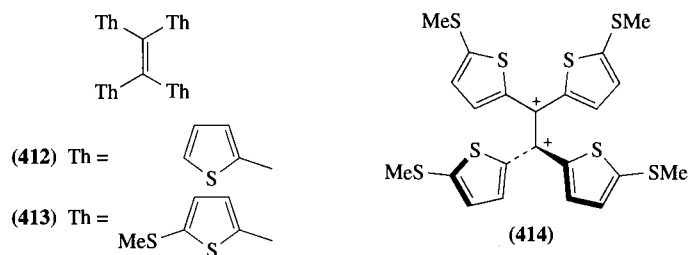
3,4,5-Trichloro-2-thienyllithium prepared from tetrachlorothiophene by lithium-chlorine exchange (Bu^nLi) reacted with aldehydes and ketones to generate carbinols. Treatment of the (2-hydroxymethyl) trichlorothiophene (**406**) with catalytic TsOH in refluxing benzene gave the electrophilic substitution product (**407**). Replacement of benzene with nitrobenzene as solvent led to

the formation of the ether (**408**) and the dithienylmethane (**409**). Pure (**408**) could be converted to (**409**) under the same conditions, possibly by the mechanism shown in Scheme 85. Under the same conditions, the carbinol (**410**) gave the thialactone (**411**) <93ACS131>.

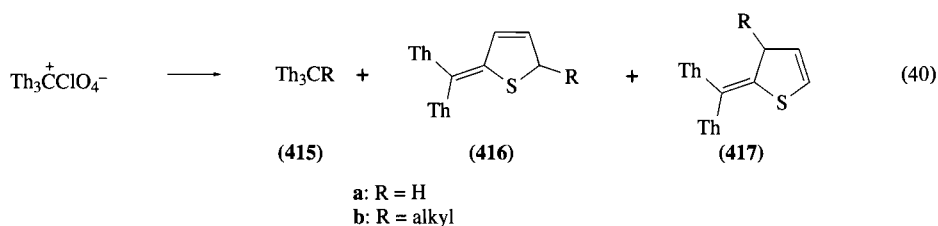


Scheme 85

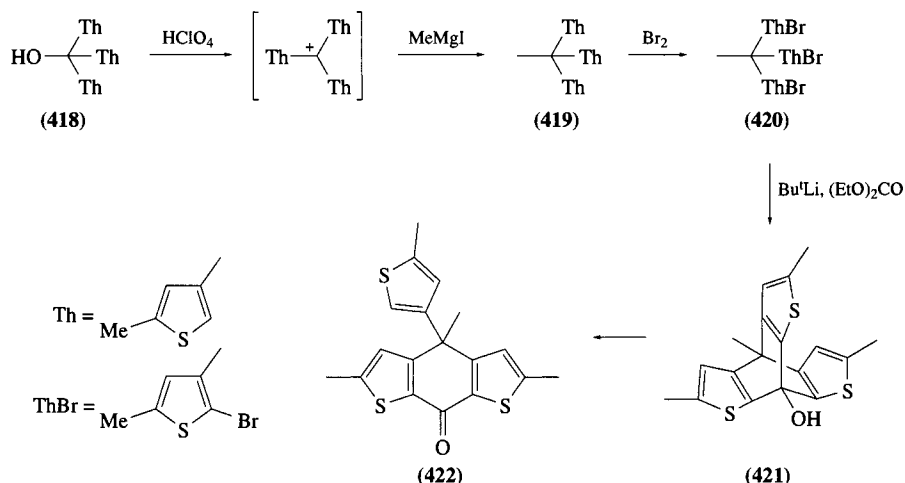
The unsubstituted tetra(2-thienyl)ethylene (**412**) was synthesized by reductive coupling of bis(2-thienyl)ketone <92AG(E)455>. Lithiation (Bu^nLi), followed by treatment with dimethyl disulfide gave (**413**). Electrochemical oxidation of (**413**) generates the blue dication (**414**), isolated as a stable perchlorate salt. The x-ray structures of (**413**) and (**414**) reveal an interesting change in molecular geometry on oxidation. Neutral (**413**) possesses a planar *trans*-dithienylethylene structure with the other two thiophene rings orthogonal to the molecular plane. In contrast, in the salt (**414**), the two halves of the molecule are twisted around the central C—C bond. Each of the two dithienylcarbenium units is nearly planar. The cations appear to be stabilized over a conjugated polymethine framework with 12 electrons distributed over 11 atoms.



The reactions of tri-(2-thienyl)methyl carbenium ion have been studied <90TL2623>. Hydride (LAH) attacks both the carbenium ion center as well as position 5 of thiophene to give (**415a**) (55%) and (**416a**) (32%), respectively. Several Grignard and organolithium reagents react similarly giving mixtures of (**415b**) and (**416b**); in addition, they also attack position 3 of thiophene to form (**417**) (Equation (40)). The reactions may proceed by a single electron transfer mechanism. Both (**416**) and (**417**) aromatize on treatment with KOBu^t .



The synthesis of thiophene triptycene (**421**) makes successive use of halogen-metal exchange, carbinol formation, and generation of carbenium ions <91CC751>. Lithiation of 4-bromo-2-methylthiophene, followed by reaction with diethyl carbonate gave the carbinol (**418**). The carbenium ion derived from this by perchloric acid, was methylated by MeMgI to generate the quaternary carbon in (**419**) (Scheme 86). Bromination to the tribromo derivative (**420**), lithium exchange by means of Bu^tLi and a second reaction with diethyl carbonate gave the triptycene (**421**). When this carbinol (**421**) was treated with acid, or with BuⁿLi or even refluxed in *o*-dichlorobenzene (ODCB), it was quantitatively converted to the ketone (**422**).



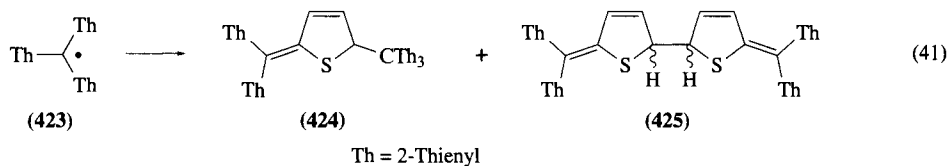
Scheme 86

Detailed ¹H and ¹³C NMR spectroscopic studies have been carried out on the tris(thienyl)methyl carbenium ions <86TL5657, 91JOC3224>. Both the 2-thienyl and 3-thienyl series have been investigated. The former is more effective in delocalizing the positive charge; the sulfur atom localizes more positive charge when the thiophene ring is attached to the carbenium centre at C-3.

Tris (2-benzo[*b*]thienyl)(methoxy)methane has been prepared by methanolysis of the carbinol via the carbenium ion <89JCR(S)190>.

(iii) Carbon radicals

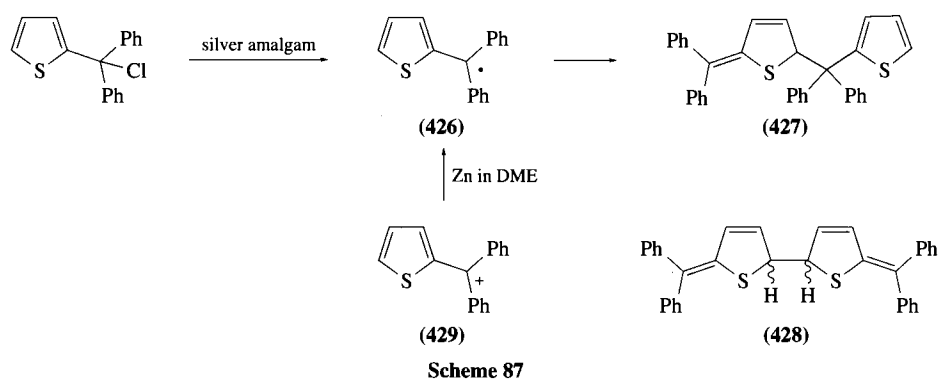
Reduction of tri-(2-thienyl)methyl carbenium tetrafluoroborate by zinc in DME at 65°C for 1 h produces, via the radical (**423**), two dimers (**424**) and (**425**) (Equation (41)). The former is the kinetically favored product, which changes to the thermodynamically preferred (**425**) on thermal equilibration. This obviously takes place by dissociation into the radical (**423**) <90TL2627>.



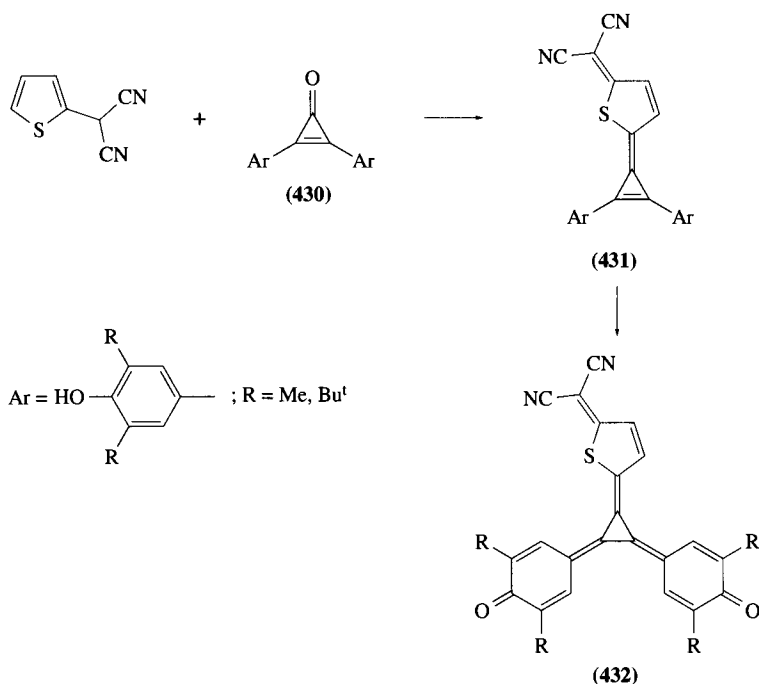
Treatment of diphenyl(2-thienyl)methyl chloride with silver amalgam in benzene at room temperature generated the radical (**426**) which dimerized to form (**427**) in 75% yield <90H(31)1915>. Reduction of the chloride with zinc in benzene gave (**427**) and a small amount (20 : 1 ratio) of (**428**) (Scheme 87). The same result was obtained when the carbenium ion (**429**) was reduced with zinc in DME.

(iv) Miscellaneous

2-Thienylmalononitrile has been synthesized and condensed with 1 equiv. cyclopropenone (**430**) in acetic anhydride containing β-alanine to give (**431**) in 80% yield (Scheme 88) <94CC519>. These

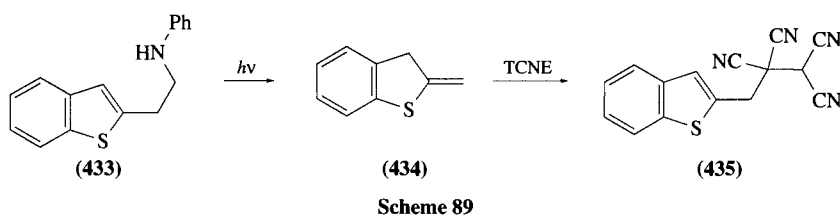


were then oxidized (PbO_2) to form the [3]radialenes (**432**). Cyclic voltammetry of (**432**) shows the occurrence of reversible two-stage one-electron reductions. These compounds are therefore powerful acceptors, forming molecular complexes with electron donors such as tetrathiafulvalene.

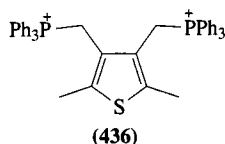


Photolysis of 2-(anilinoethyl)benzo[*b*]thiophene (**433**) in benzene gave 2-methylene-2,3-dihydrobenzo[*b*]thiophene (**434**). This may be caused by a photoinduced electron transfer from the nitrogen to the benzo[*b*]thiophene. Compound (**434**) reacts with TCNE in an ene reaction to give (**435**) (Scheme 89). On treatment with acid, (**434**) aromatizes ^{<88JCS(P1)2579>}.

The reaction of the bisphosphonium salt (**436**) with several quinones has been investigated



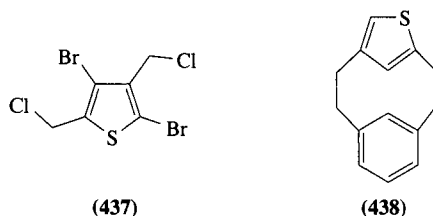
<86JCS(P1)415>. The simple double Wittig reaction was observed only in one instance. In most cases, an initial mono Wittig was followed by Michael addition of a second ylide molecule, with subsequent elimination of triphenylphosphine.



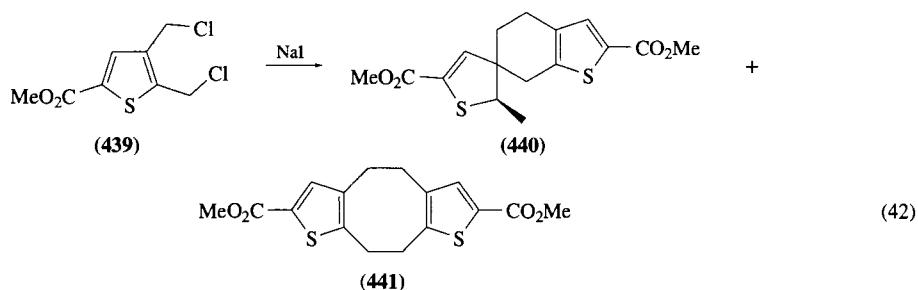
2.10.4.2.3 Halomethyl groups

Thenyl derivatives including halomethyl, hydroxymethyl, and aminomethyl thiophenes have been exhaustively reviewed <86HC(44/3)975>.

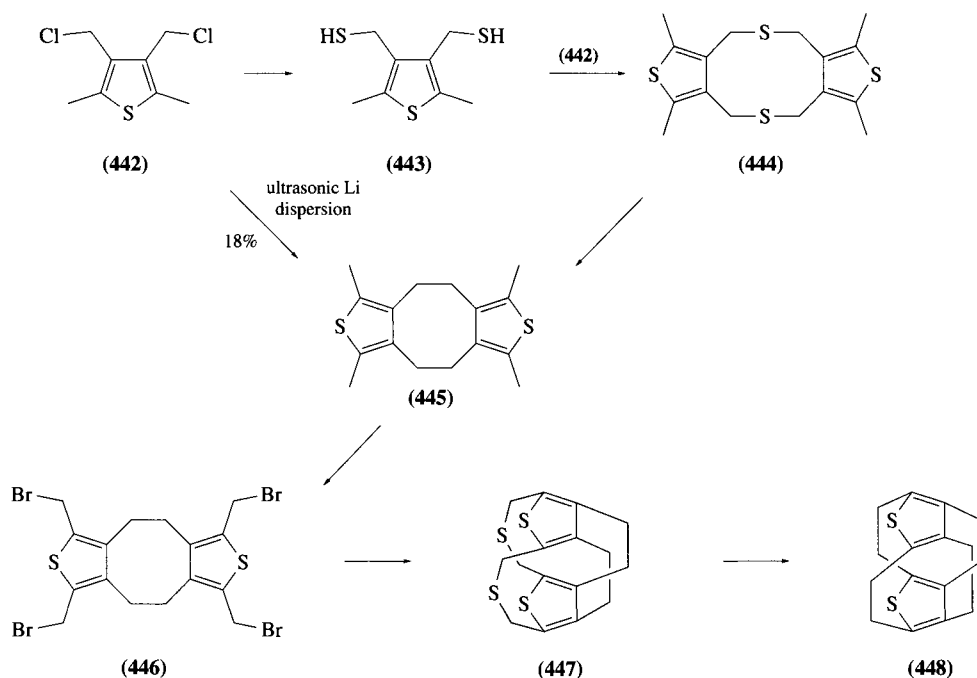
The *meta*-cyclo(2,4)thiophenophane (**438**) has been synthesized from 3,5-bis(chloromethyl)-2,4-dibromothiophene (**437**) <92JOC746>. The bromine atoms serve as “positional protective” groups and are removed at a later stage. The chloromethyl groups were converted to mercaptomethyl and coupled with 1,3-bis(bromomethyl)benzene. Debromination (BuLi, H₃O⁺), oxidation to sulfone (MCPBA) and pyrolysis gave the thiophenophane (**438**).



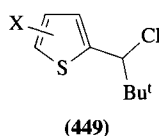
The culmination of this approach has been the first synthesis of “superthiophenophane”—[2₄](2,3,4,5)thiophenophane (**448**) <92JOC4654>. This makes extensive use of chloromethylthiophenes. In initial experiments, it was found that dimerization of methyl 2,3-bis(chloromethyl)thiophene-5-carboxylate (**439**) by treatment with NaI gave mainly the spiro compound (**440**) (54%) and only a poor yield of the required [2.2](2,3)thiophenophane (Equation (42)) (**441**). Attempts to synthesize a tetrathia-bridged compound from tetrakis(chloromethyl)thiophene by treatment with Na₂S or thio-urea were also unsuccessful. The final successful route (Scheme 90) starts from 3,4-bis(chloromethyl)-2,5-dimethylthiophene (**442**). Coupling of (**442**) and the derived bis(mercaptomethyl) compound (**443**) gave the tricyclic dithia compound (**444**). Oxidation to the disulfone, followed by pyrolysis gave (**445**). A better route to (**445**) was coupling of (**442**) by treatment with lithium dispersion under ultrasound irradiation. Bromination of (**445**) (NBS in CH₂Cl₂) gave the tetrabromo derivative (**446**), which led to (**447**) by intramolecular cyclization with Na₂S. MCPBA oxidation to the disulfone and pyrolytic extrusion of SO₂ gave the superthiophenophane (**448**).



The reaction of the neopentyl chlorides (**449**) with various nucleophiles has been studied <87AJC49>. The mechanism of nucleophilic displacement depends on the substituents on the thiophene ring. Chlorocarbenes have been generated from 2-(dichloromethyl)- and 3-(dichloromethyl)thiophenes by treatment with KOBu^t at 0 °C <89JCR(S)376, 90JCR(S)134>. These can be trapped by alkenes to form cyclopropanes.



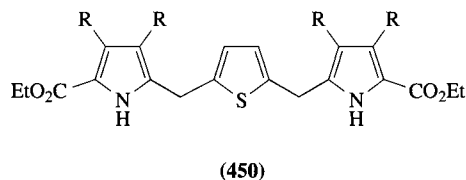
Scheme 90



2.10.4.2.4 Hydroxymethyl and thiomethyl groups

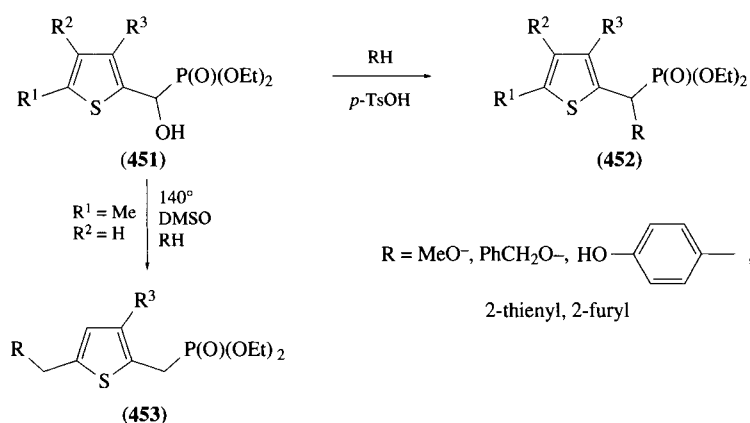
The formation and reactions of carbenium ions has been discussed in Section 2.10.4.2.2.

Acid-catalyzed condensation of 2,5-di(acetoxymethyl)thiophene with 3,4-dialkylpyrrole-2-carboxylic ester gives (450) <92T9661>. The acid-catalyzed reaction of 2-thienylphosphonyl carbinols (451) with nucleophiles such as alcohols, phenols, thiophenes and furans has been studied <92SC699>. In MeCN as solvent, at room temperature, in the presence of equimolar amounts of *p*-TsOH, good yields of products (452) were obtained if the thiophene (451) had at least one methyl substituent. When phenol was the nucleophile, alkylation took place at the *para*-position. When the temperature of the reaction was increased (140°C, DMSO), the thiophene (451; R¹ = Me, R² = H) yielded the isomeric product (453) (Scheme 91). The 3-thienyl isomer of (451) did not undergo a clean reaction.



The conversion of 2-hydroxymethylthiophenes to the corresponding 2-aminomethyl derivatives always poses problems, since, if the alcohol is activated in the first step, it tends to ionize to the stabilized cation which can then attack another thiophene molecule in an electrophilic reaction. A successful solution to this problem is to treat the alcohol with NaN₃ in presence of Ph₃P and CCl₄ resulting in the formation of the azidomethyl compound <91JOC7179>. Here, isolation of the active intermediate is avoided. The resulting azide can be reduced to the amine by Ph₃P in pyridine.

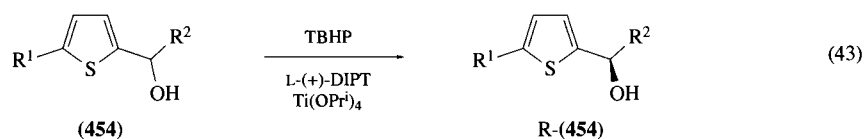
Carbinols formed by reaction of Grignard reagents with thiophene-3-carbaldehyde can be hydrogenolyzed in acid medium under palladium catalysis <93JMOC201>. The best catalyst was PdCl₂



Scheme 91

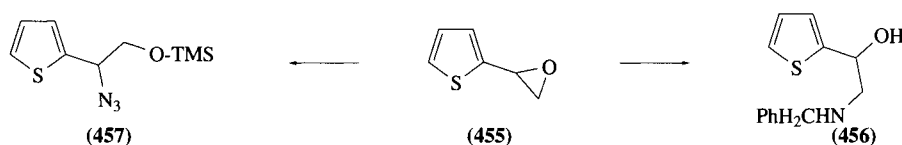
(5%/substrate); the HCl liberated on hydrogenation of this seemed to be necessary for the successful hydrogenolysis. Thus $\text{Pd}(\text{OAc})_2$ was ineffective in bringing about this transformation. The best results were obtained in acetic acid as solvent at 65°C using PdCl_2 as catalyst. This is certainly a surprising result in view of the known propensity of such catalysts to be poisoned by thiophenes.

Kinetic resolution of 2-thienylcarbinols is possible using the Sharpless reagent $\langle 89\text{JOC}994 \rangle$. These substrates meet the essential requirement of the Sharpless procedure: presence of a hydroxyl group at the chiral center and a nearby oxygen-accepting system (here, the sulfur atom of the thiophene). Treatment of racemic (454) with 3 equiv. TBHP in the presence of L-(+)-diisopropyl tartrate and titanium isopropoxide led to selective oxidative polymerization of one of the enantiomers (Equation (43)). The remaining unoxidized material was found to have the (*R*)-configuration, with *ee* > 95%.



The reaction of trialkylaluminum with 2-thiophenemethanol yields $\{[(\text{R}_2\text{Al})_3(\mu\text{-OCH}_2\text{Th})_6]\text{Al}\}$ ($\text{R} = \text{Me, Et; Th} = 2\text{-thienyl}$) $\langle 94\text{OM}2079 \rangle$.

2-Thienylepoxyde (455) can be regioselectively opened by nitrogen nucleophiles, in a noncatalyzed reaction. The regiochemistry differs, depending on the nucleophile used. Benzylamine attacks the less substituted carbon giving the aminoalcohol (456), while trimethylsilylazide yields (457) by attack on the methine carbon (Scheme 92) $\langle 92\text{TL}7413 \rangle$.

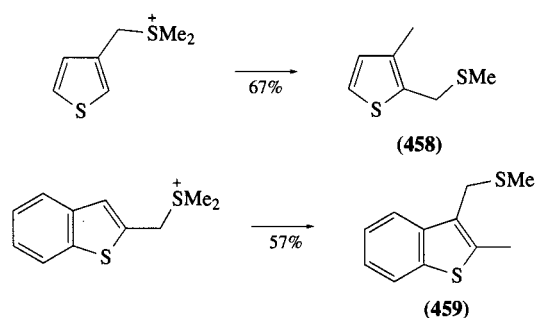


Scheme 92

[2,3]Sigmatropic rearrangement of the sulfonium ylides from 3-(bromomethyl)thiophene and 2-(bromomethyl)benzo[*b*]thiophene has been reported $\langle 89\text{BCJ}958 \rangle$. Treatment of the bromomethyl compounds with dimethylsulfide gave the sulfonium salts. These were deprotonated (NaOEt or NaH) to generate the ylides. Sommelet–Hauser rearrangement of the ylides gave the products (458) and (459) (Scheme 93).

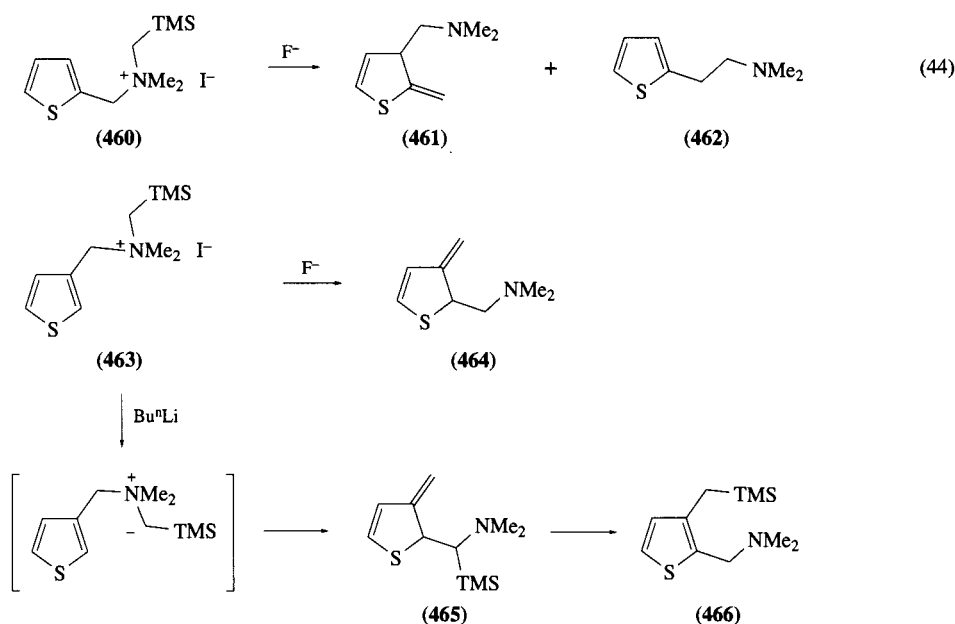
2.10.4.2.5 Aminomethyl groups

The synthesis of thiophenophanes from 2-(dimethylaminomethyl)-5-methylthiophene has been discussed in CHEC-I. This has been extended to the vinylogous compound (317) as well (see Section 2.10.3.5.2).



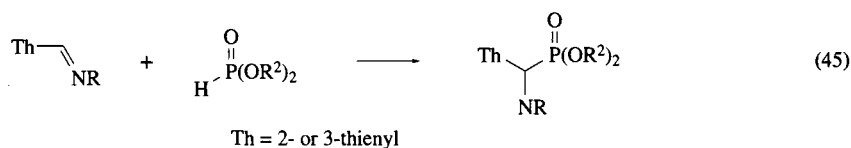
Scheme 93

The Sommelet–Hauser rearrangement of (thienylmethyl)ammonium *N*-methylides has been studied using the fluoride-induced desilylation reaction, especially in relation to competing Stevens rearrangement. Treatment of (460) with CsF in HMPA at room temperature gave (461) and (462) in the ratio 92:8 (total yield 85%) (Equation (44)). The 3-thienyl analogue (463) similarly gave (464) as the major product (Scheme 94). Reaction of (461) and (464) with BuⁿLi resulted in aromatization. Deprotonation of (463) by BuⁿLi gave (466), possibly via the intermediate (465) <92JOC5419>.



Scheme 94

α -Aminophosphonic acids in which the α -carbon bears a 2-thienyl or 3-thienyl substituent, have been prepared by reacting imines (from thiophene carbaldehydes) with diethyl or dibenzyl phosphonate containing a labile P—H bond (Equation (45)). The reaction is best carried out under ultrasonic activation <94S51>.

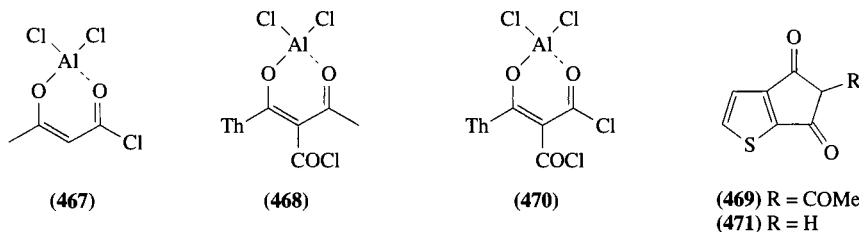


2.10.4.2.6 Carboxylic acids and nitriles

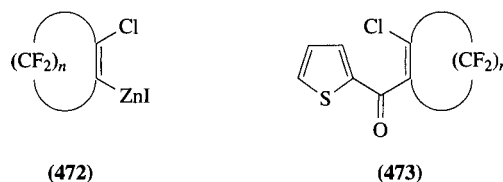
Thiophene carboxylic acids have been extensively reviewed <84CHEC-I(4)741, 86HC(44/3)565>. pK_a measurements have been carried out on 4-*X*-thiophene-2-carboxylic acids and 2-*X*-thiophene-4-

carboxylic acids <93JCR(S)300>. In both series, the substituent X is in a *meta*-like position. It has been found that the transmission of substituent effects in the above *meta*-like relationships is quite similar to that observed in *meta*-substituted benzoic acids. The thiophenic σ values are very similar to the benzenic σ values. The conclusion has been reached that in both *meta*- and *para*-like disubstituted thiophenes, the hetero ring is able to transmit the electronic effects of the substituents as in the benzene ring, i.e., through the heterocycle as a whole and not via the heteroatom.

The aldol-like self-condensation of acetyl chloride in the presence of AlCl_3 gives the Al-chelate of acetoacetyl chloride (**467**). Reaction of this with thiophene-2-carboxylic acid chloride gives (**469**) in 72% yield via (**468**) <92JCS(P1)2985>. Use of malonyl chloride instead of acetyl chloride gives (**471**) (78%) via (**470**).



The 2-chloro(perfluoro)cycloalkenylzinc reagent (**472**) reacts with thiophene carboxylic acid chlorides in the presence of CuBr to form the ketones (**473**) in excellent yields. The reaction is probably mediated by the corresponding 2-chloro(perfluoro)cycloalkenyl copper <91JCS(P1)1601>.



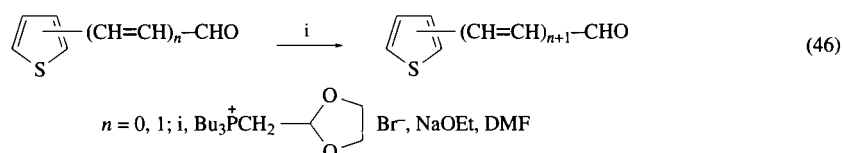
Methyl 3-aminothiophene-2-carboxylate can be easily transformed into the *N,N*-dimethyl or *N,N*-diethyl (but not *N,N*-diisopropyl) amide by treatment with 2 equiv. of the corresponding lithium amide. The reaction conditions are mild, and the yields are good <92TL6453>. Trifluoroacetamides of aminothiophene and aminobenzo[*b*]thiophene have been prepared by Curtius rearrangement of the corresponding acylazides (toluene or benzene) and treatment of the resultant isocyanates with $\text{CF}_3\text{CO}_2\text{H}$ at room temperature <92H(34)149>.

Liquid crystalline polyesters have been synthesized from thiophene-2,5-dicarboxylic acid and several substituted hydroquinones <92MM563>.

2.10.4.2.7 Aldehydes and ketones

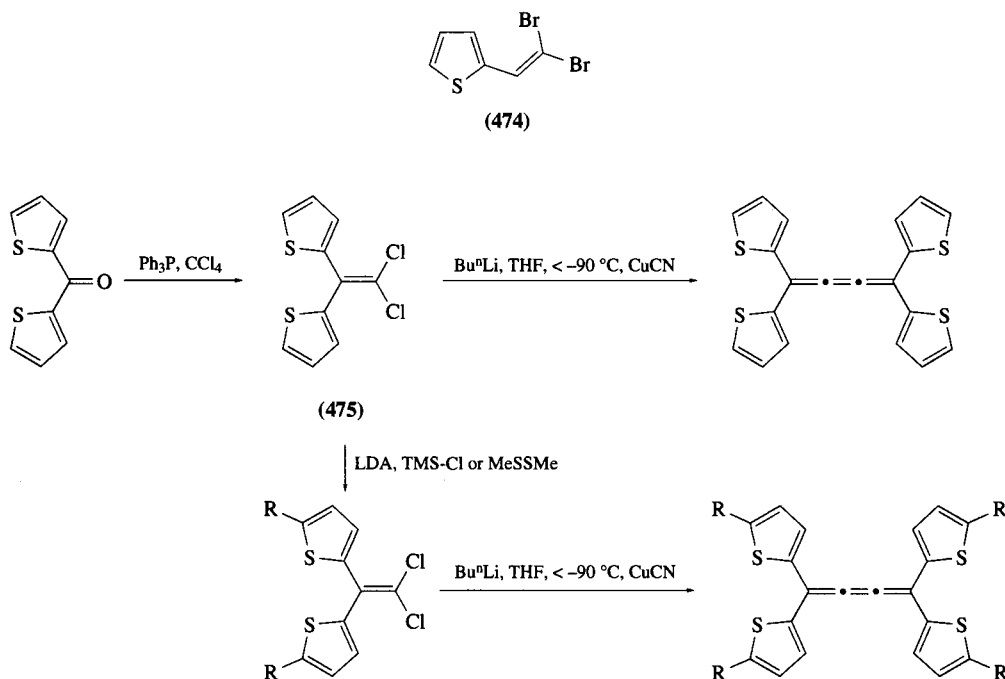
The common reactions of aldehydes and ketones attached to thiophene rings have already been dealt with exhaustively in earlier reviews <84CHEC-I(4)741, 86HC(44/3)324>. Only new and interesting applications will therefore be discussed in this section.

The Wittig reaction along with its modification (Horner–Emmons–Wadsworth) has been employed to generate a variety of interesting structures. For example, conjugated polyenals have been synthesized as shown in Equation (46) <88SC51>.



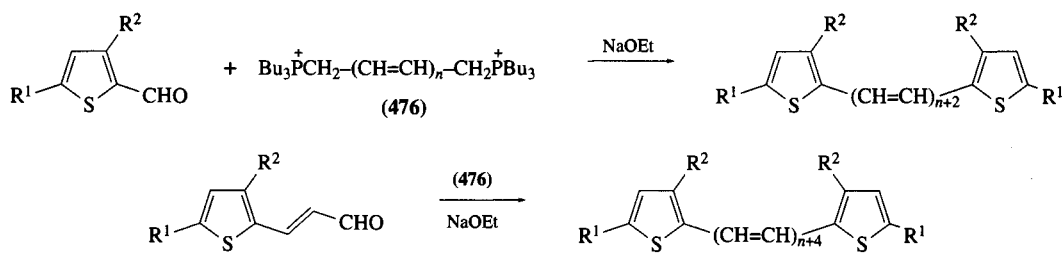
Conversion of thiophene 2-carbaldehyde to the dibromovinyl derivative (**474**) can be accomplished by CBr_4 and Ph_3P . On metalation with Bu^nLi at -78°C , followed by quenching with water, this is converted to 2-thienylacetylene <82JOC2201, 86H(24)365>. A side reaction in this is the metalation at position 5 of the thiophene. Tetra(2-thienyl)butatriene and some of its derivatives

have been synthesized from di(2-thienyl) ketone by a simple three-step procedure (Scheme 95) <92CC778>. The first step is dichloromethylenation by $\text{Ph}_3\text{P}-\text{CCl}_4$ to give (475). This could be regioselectively lithiated either at position 5 of the thiophene units, or at the dichloroethene unit by appropriate choice of reagent. LDA directly lithiated the thiophene ring, leading to 5-substituted derivatives on quenching with electrophiles. On the other hand, treatment with *n*-butyllithium below -90°C and subsequent reaction of the lithio derivative with copper(I) cyanide gave the tetrathienylbutatriene.



Scheme 95

α,ω -Dithienyl polyenes have been synthesized from thiophene 2- and 3-carbaldehydes by making use of the bisphosphoranes (476) (Scheme 96) <91JCS(P1)799, 92JCS(P2)1959>. Some novel push-pull systems containing thiophenes have been constructed by using the Wittig reaction (Scheme 97). These molecules exhibit interesting nonlinear optical properties <93CC90>.

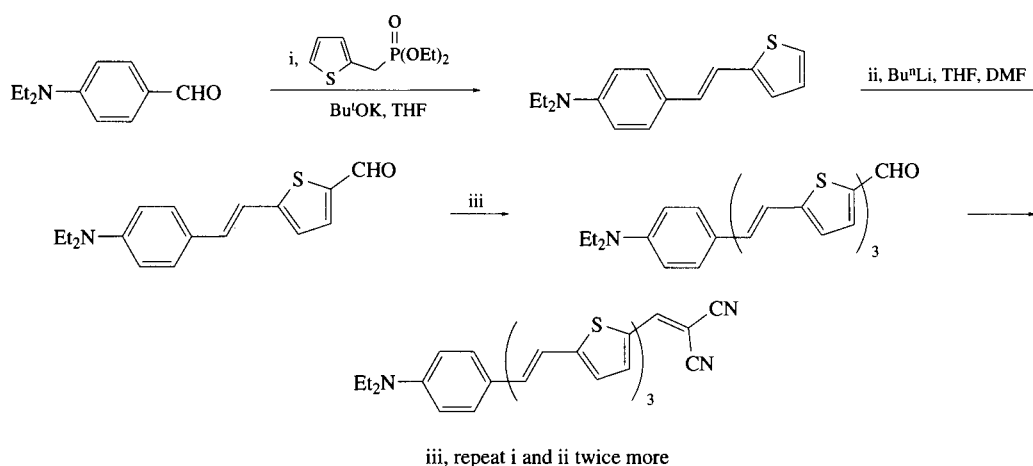


Scheme 96

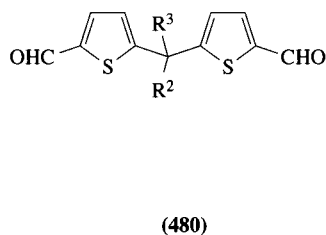
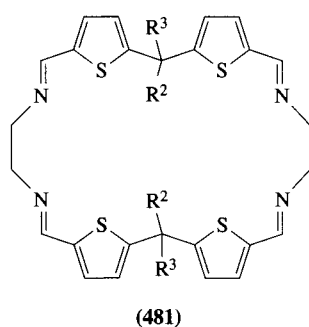
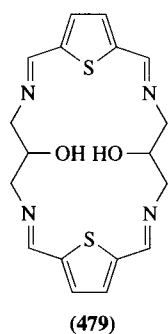
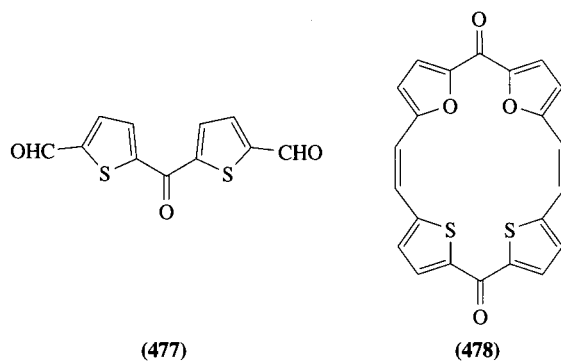
A double Wittig reaction on the dialdehyde (477) under appropriate dilution conditions has been employed to generate the [22]annulenoquinone (478) <93AG(E)1333>. The molecule (478) is not planar. One furan ring is twisted out of the plane of the rest of the molecule.

Thiophene aldehydes have been used earlier for the construction of macrocyclic Schiff bases <81CC628>. An example is the synthesis of the macrocyclic tetraimine Schiff base (479) from thiophene 2,5-dicarbaldehyde and 1,3-diamino-2-hydroxypropane <87JCS(D)219>. The synthesis does not involve use of a metal template. An interesting extension is the synthesis of the macrocyclic Schiff base (481) containing four thiophene units from the dialdehyde (480) <89BCJ1346>. The condensation is achieved at room temperature in CHCl_3 .

Thiophene 2,5-dicarbaldehyde is the starting point for the synthesis of a series of extended donors,



Scheme 97

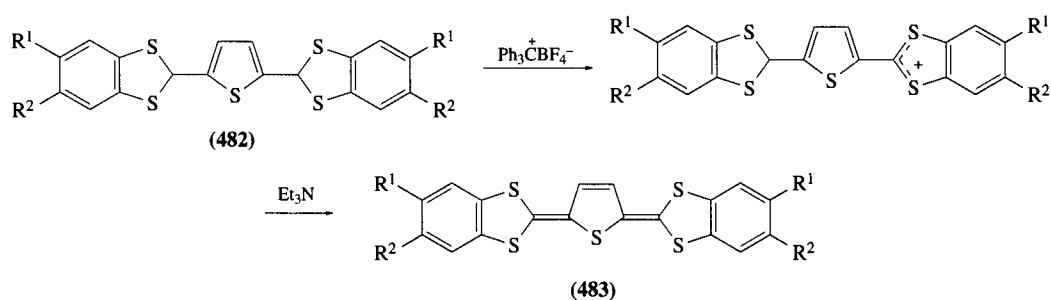


2,2'-(2,5-dihydrothiophene-2,5-diylidene) bis (1,3-benzodithiole)s (**483**) (Scheme 98) <89TL2091>. Condensation with benzene-1,2-dithiol to give (**482**) is followed by removal of a hydride and subsequently of a proton.

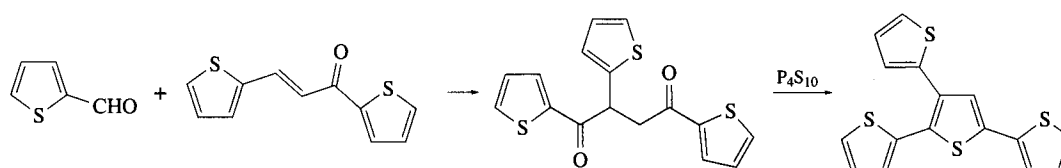
The titanium-induced reductive coupling of 3,4-dibutoxythiophene-2,5-dicarbaldehyde (TiCl_4 , Zn) has given poly (3,4-dibutoxy-2,5-thienylene-vinylene), with good solubility in organic solvents <93CL1085>.

The addition of thiophene carbaldehyde to α,β -unsaturated ketones has been utilized to synthesize several branched quaterthienyls <91JOC5095>. An example is given in Scheme 99.

The benzoin condensation using thiophene 2-carbaldehyde and 3-carbaldehyde has been reinvestigated <92JHC149>. Three different catalysts have been tried: KCN, a cyanide resin and thiazolium salt. With the 2-aldehyde, thenoin yields were low (maximum 20%); this was accompanied by significant amounts (55%) of the oxidation product, thenil. It has been suggested that thenil might be formed by a radical mediated reaction. The 3-aldehyde, on the other hand, gives a 64% yield of the thenoin without any thenil being formed.

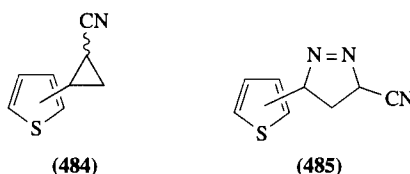


Scheme 98

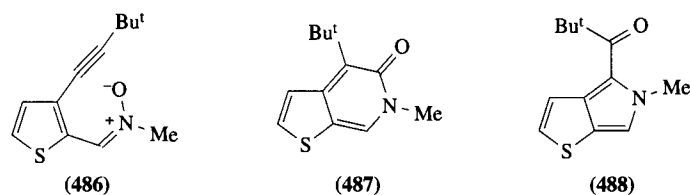


Scheme 99

The generation of carbenes from tosylhydrazones of thiophene aldehydes, and their reactivity have been discussed in CHEC-I, <84CHEC-I(4)741>. A few further reactions of such carbenes have been reported. In presence of acrylonitrile, the product could be either the cyclopropane (**484**) (from the carbene) or the pyrazoline (**485**) (by cycloaddition of the diazo intermediate) <88H(27)1141>. The carbene can similarly be trapped by *cis* and *trans* stilbene, dimethyl fumarate or maleate to form the respective cyclopropanes <87BCJ4317>.

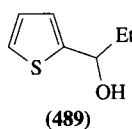


Thermolysis of the nitrone (**486**) obtained from the corresponding aldehyde leads to the thieno [2,3-*c*]pyridone (**487**) (25%) and the thieno [2,3-*c*]pyrrole (**488**) (11%). The suggested mechanism involves the intermediacy of a carbene <92TL61>.

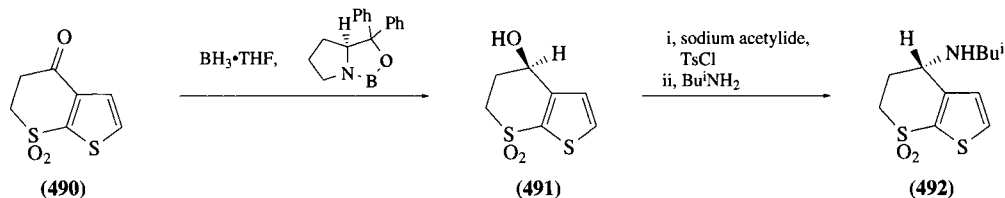


N,N-Dimethylhydrazone of thiophene-2-carbaldehyde gets converted to 2-cyanothiophene on treatment with DMAD. A six-membered transition state is likely to be involved in the elimination of dimethylaminomaleic ester from the initial adduct <88H(27)1227>.

Catalytic enantioselective alkylation of thiophene 2-carbaldehyde using dialkylzinc reagents has led to the alcohols in good chemical yield and high optical purity <91JCS(P1)25>. Thus, reaction of the aldehyde with diethylzinc in the presence of (*R*)-(-)-1-*i*-butyl-2-piperidinoethanol (2 mol %) gave the product (*R*)-1-(2-thienyl) propan-1-ol (**489**) in 90% yield and 95% *ee*.

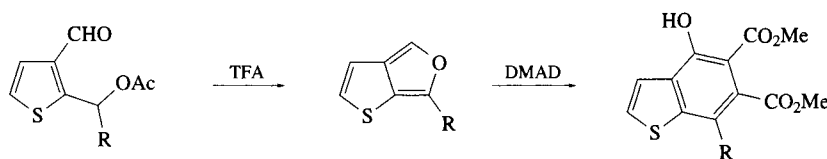
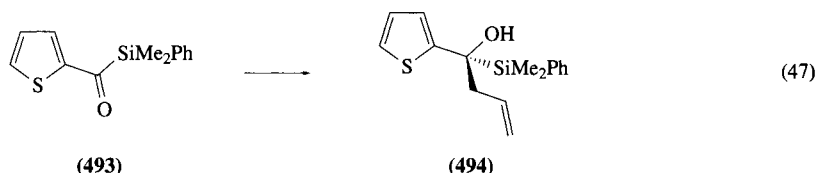


Asymmetric reduction of the ketosulfone (490) has been tried under three different protocols: (i) (–)-B-chlorodiisopinocampheylborane, (ii) yeast reduction, and (iii) borane reduction catalyzed by chiral 1,3,2-oxazaborolidine. Of these, the third method gave the best results for large-scale process development <92JHC627>. The product (491) could be converted to (492) by tosylation and subsequent displacement of the —OTs group by isobutylamine (Scheme 100). This was then sulfonated (oleum) to yield a 90 : 10 mixture of the 2- and 3-sulfonic acids.



Scheme 100

Asymmetric allylboration of the acylsilane (493) using B-allyldiisopinocampheylborane has given *tert*-carbinol (494) in low *ee* (17%) (Equation (47)) <94TL985>. The capture of a neighboring carbenium ion by the oxygen of the aldehyde has been used to synthesize thieno[2,3-*c*]furans <91CC1635>. These undergo cycloaddition with DMAD (Scheme 101).



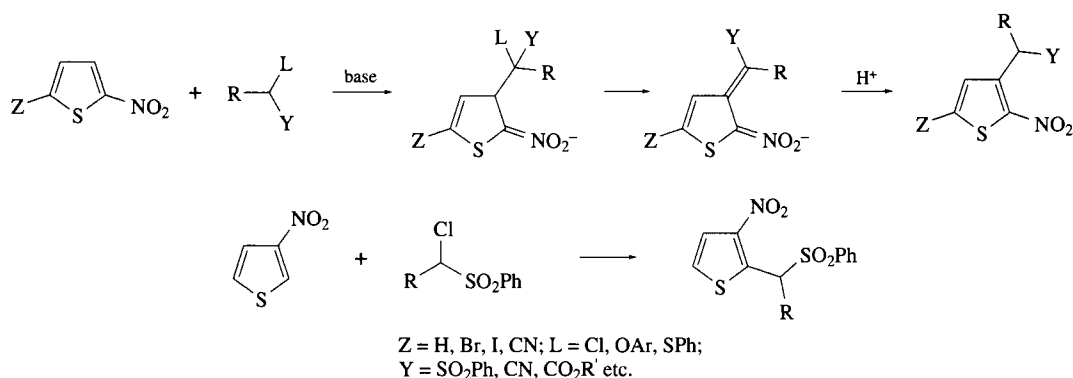
Scheme 101

2.10.4.3 N-Linked Substituents

2.10.4.3.1 Nitro compounds

Nucleophilic substitution of nitrothiophenes has been reviewed recently <92HC(44/4)295>. A reaction of great synthetic potential is the vicarious nucleophilic substitution of hydrogen in nitrothiophenes <87ACR282, 91S103>. The underlying concept is as follows: carbanions bearing leaving groups (L) at the carbanion center can initially add to nitrothiophenes forming σ^H adducts; subsequent elimination of HL, followed by protonation would lead to products which can formally be regarded as having been obtained by substitution of Nu for H. The carbanions are usually generated *in situ* by the action of base on the corresponding CH acids (Scheme 102). With 2-nitrothiophenes, usually H-3 of the thiophene is replaced, if the carbanion is CHLY, but with tertiary carbanions, some 5-substitution can also occur. 3-Nitrothiophene gives only the 2-substituted product (Scheme 102). The base employed is usually KOBu^t, KOH, NaOH or NaH; the preferred solvents are DMSO, liquid ammonia or DMF. Even CHCl₃ and CHBr₃ react under these conditions forming 3-dihalomethyl-2-nitrothiophene, which can be hydrolyzed to the corresponding aldehyde. An extension of this to other nucleophiles has resulted in the synthesis of 3-hydroxy-2-nitrothiophene from 2-nitrothiophene by treatment with cumene hydroperoxide. Similarly, 4-amino-1,2,4-triazole is the nucleophile for achieving nuclear amination, but the yield is low <88JOC3978>.

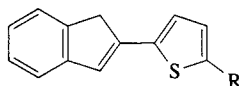
An interesting feature of this process is that vicarious nucleophilic substitution of hydrogen usually proceeds faster than conventional nucleophilic substitution of halogen activated by the nitro group.



Scheme 102

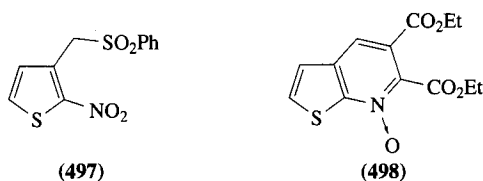
Meisenheimer complexes of nitrothiophenes continue to be investigated $\langle 84\text{CHEC-I}(4)741 \rangle$. A ^{13}C NMR spectral study defines the ^{13}C chemical shift changes accompanying the formation of such complexes $\langle 91\text{JCS}(P2)1631 \rangle$. The effect of substituents on the rate and equilibrium constants for the formation of Meisenheimer adducts has been determined $\langle 89\text{JCS}(P2)1609 \rangle$.

Photosubstitution of the nitro group in 2-nitrothiophene has been reported $\langle 94\text{TL}633 \rangle$. Thus, photolysis of 2-nitrothiophene in the presence of indene in acetonitrile gave **(495)** in excellent yield. Similarly, photolysis of 2-iodo-5-nitrothiophene in the presence of indene gave a 3 : 1 mixture (total yield quantitative) of **(496)** and **(495)**.



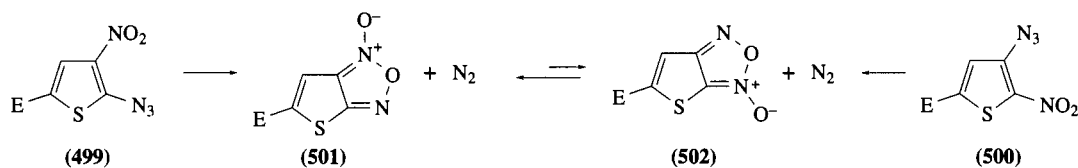
(495) R = H
(496) R = I

Substituted nitrothiophenes serve as precursors for a variety of condensed thiophenes. The synthesis of thienopyrroles has been reviewed $\langle 85\text{S}143 \rangle$. Treatment of (2-nitro-3-thienyl)methyl phenyl sulfone **(497)** with diethyl maleate in the presence of K_2CO_3 and 18-crown-6 gave the thienopyridine *N*-oxide **(498)** in 38% yield $\langle 92\text{ACS}689 \rangle$. The reaction apparently proceeds by Michael addition, followed by elimination of phenylsulfinate and cyclization. The starting material can be prepared by vicarious nucleophilic substitution discussed earlier.

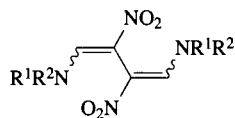


3-Azido-2-nitrothiophene undergoes thermal cyclization, with loss of N_2 , to an equilibrium mixture of thieno[3,2-*c*]- and thieno[2,3-*c*]furan oxides $\langle 74\text{JOC}2956 \rangle$. Kinetic and thermodynamic data have now been generated on the related cyclization of 2-azido-5-methoxycarbonyl-3-nitrothiophene **(499)** and of 3-azido-5-methoxycarbonyl-2-nitrothiophene **(500)** to the corresponding thieno[3,2-*c*]- and thieno[2,3-*c*]furan oxides **(501)** and **(502)** (Scheme 103). The equilibrium between **(501)** and **(502)** has been studied by dynamic ^1H NMR spectroscopy $\langle 89\text{JCS}(P2)127 \rangle$. The isomerization rates are very large compared with the cyclization rates; hence the same mixture results from both **(499)** and **(500)**. The equilibrium between **(501)** and **(502)** is fast on the NMR time scale.

The ring opening of 3,4-dinitrothiophene by primary and secondary amines has been fully explored $\langle 90\text{TL}4933, 92\text{T}4407 \rangle$. The optimum reaction conditions appear to be to treat a suspension of the dinitro compound in EtOH with 4 molar equivalents of amine at 0°C . Good yields of the bis(nitroenamine)**(503)** have been obtained.



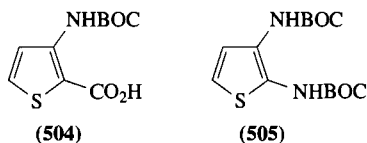
Scheme 103



$\text{R}^1 = \text{alkyl}; \text{R}^2 = \text{H or alkyl}$
 $\text{R}^1 = \text{aryl}; \text{R}^2 = \text{H}$

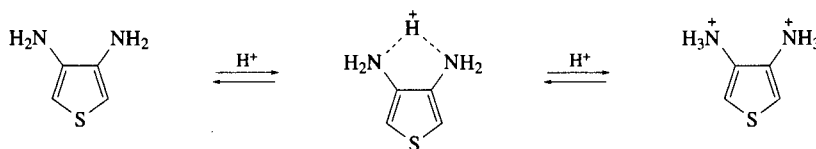
2.10.4.3.2 Amines and derivatives

2,3-Diaminothiophene has been prepared and isolated as the hydrobromide. The free base is not stable <85JCR(S)296>. The *t*-butoxycarbonyl derivative of 3-aminothiophene was carboxylated at position 2 by lithiation (Bu^nLi) followed by reaction with CO_2 . Reaction of this carboxylic acid (504) with diphenyl phosphorazidate in *t*-butanol gave the bis-carbamate (505). Removal of the protecting groups by HBr gave the hydrobromide salt of 2,3-diaminothiophene. 2,3-Diaminobenzo[b]thiophene was made by a similar procedure.



Reduction of 2,5-dibromo-3,4-dinitrothiophene has given 3,4-diaminothiophene as a stable crystalline solid <83BSF(2)153, 86BSF259, 86BSF267>. This has been converted to diamides and dicarbamates. Nitration of the diacetamide gave either the mono or dinitro derivative. 3,4-Diaminothiophene underwent thiocyanation at the 2 and 5 positions. The dipivalamide could be lithiated to the mono or dilithio derivative and then reacted with CO_2 . 3-Amino- and 3,4-diaminothiophene have been condensed with formaldehyde and then reduced (NaBH_4) to give 3-methylamino- and 3,4-bis(methylamino)thiophene, respectively. Other secondary amines have been prepared by reduction (LAH) of amides or carbamates; tertiary amines have been synthesized by prior *N*-alkylation of the amides, followed by reduction (LAH).

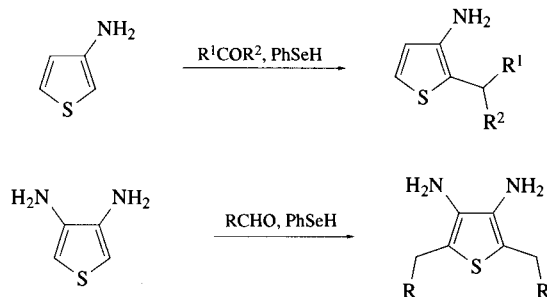
Both 3-amino- and 3,4-diaminothiophene exhibit enaminic character. On acid-catalyzed deuteration, deuterium enters the thiophene α -positions. On this basis, it was concluded that 3,4-diaminothiophene is C-protonated in acid. However, a detailed potentiometric as well as ^1H NMR study of the protonation of 3,4-diaminothiophene has revealed that both monoprotonation as well as diprotonation occurs on the nitrogen atoms (Scheme 104); there was no evidence for protonation at the α -carbon atoms <93JOC4696>. The pK_a values in 50% DMSO–water mixture are 3.96 and 0.98.



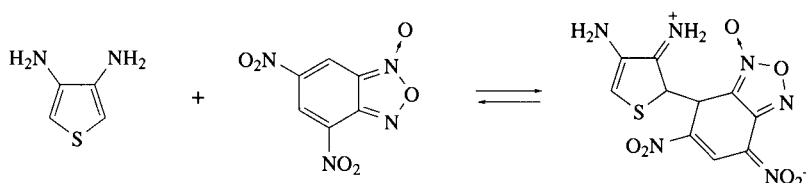
Scheme 104

However, it is undoubtedly true that several other reactions take place at the α -carbon, apart from deuteration. Thus, bromination and phenylselenenylation occur at the thiophene α -positions <86BSF267>. C-Alkylation of 3-aminothiophene and 3,4-diaminothiophene can be carried out by

reaction with aldehyde or ketone and 2 equiv. of selenophenol (Scheme 105). The powerful electrophile, 4,6-dinitrobenzofuroxan also attacks only the α -carbon of 3,4-diaminothiophene; there is no tendency for the latter to react as a nitrogen nucleophile. Excess reagent leads to the bis-C-alkylated product (Scheme 106) <93JOC4696>.

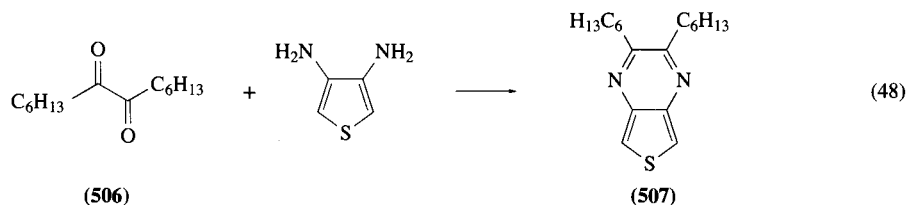


Scheme 105



Scheme 106

3,4-Diaminothiophene could be used for the construction of condensed hetero bi- and tricycles, employing standard reactions like condensation with diketones, Skraup reaction, etc. <83BSF(2)159>. Condensation with the α -diketone (**506**) leads to 2,3-dihexylthieno[3,4-*b*]pyrazine (**507**) (Equation (48)). Oxidative polymerization of this (FeCl₃) has resulted in a new low band-gap polyheterocycle <92CC1672>.

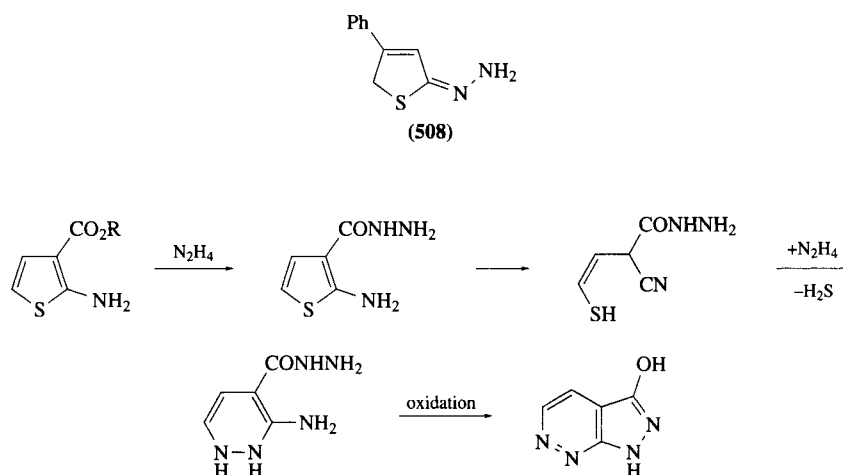


Substituent effects on the protonation (aq. HClO₄) of 5-*X*-3-nitro-2-aminothiophenes and of 3-*X*-5-nitro-2-aminothiophenes have been determined. Good linear free-energy correlations have been found with thiophene σ -values <83JCS(P2)481, 89JCS(P2)791>.

3-Aminothiophene-2-carboxylic acid has been diazotized with isoamyl nitrite to form the diazonium salt which, on thermolysis, loses CO₂. The resultant diazonium salt has been coupled with 2-naphthol. Similarly, 3-aminobenzo[*b*]thiophene-2-carboxylic acid has been converted to the corresponding diazonium salt. This preferential loss of CO₂ from *ortho*-diazonium carboxylates is unprecedented <89TL2345>.

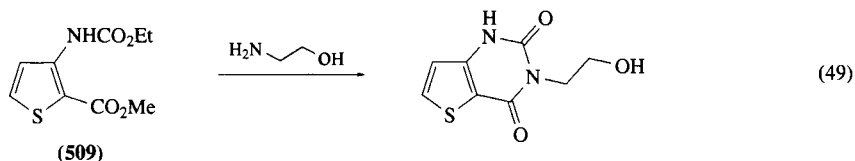
It has been reported that treatment of 2-amino-4-phenylthiophene with hydrazine results in displacement of NH₂ <88CB573>. This might happen by ring opening, addition of hydrazine, and ring closure with elimination of ammonia. Surprisingly, the product seems to exist in the hydrazone form (**508**). Reaction of 3-aminothiophene-2-carboxylic ester with hydrazine gives the expected hydrazide. But a similar reaction with 2-aminothiophene-3-carboxylic ester gives pyrazolo[3,4-*c*]pyridazines. This obviously involves ring opening and elimination of sulfur. One possible route is shown in Scheme 107 <92JOC1018>.

Aminothiophene derivatives possessing an *ortho*-carbonyl or nitrile substituent are good precursors for several heterocondensed thiophene systems. It is impossible to list all the different structures that have been synthesized by this procedure. Only a few examples are given below. Thieno[2,3-*d*]pyrimidines have been made from 2-aminothiophene-3-carbonitrile and guanidine



Scheme 107

<90HCA797>. Thieno[3,2-*d*]pyrimidinedione derivatives have been made by reacting the carbamate (509) with ethanolamine (Equation (49)) <89CPB1197>. Other thienopyrimidinedione derivatives have been made from *ortho*-ureidothiophene carboxylic acids <88JMC1786>. 3-Aminothiophene-4-carboxylic acid ester has been converted to a thieno[3,4-*c*]-1,2,6-thiadiazine derivative <89S280>. Thieno[3,2-*d*]-1,2,3-triazin-4-(3*H*)-ones have been prepared by diazotizing 3-aminothiophene derivatives having a carboxamide, hydrazide or oxime at position 2 <93JCR(S)72>.

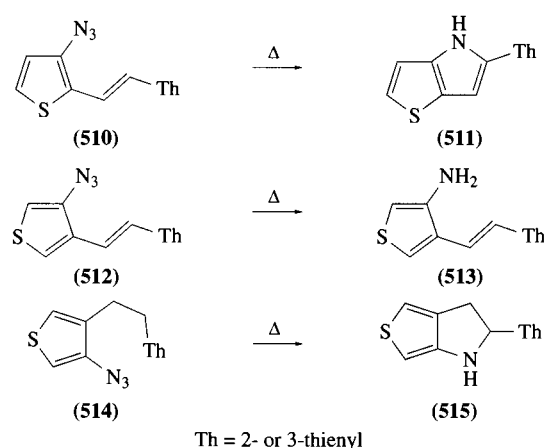


2.10.4.3.3 Azides and nitrenes

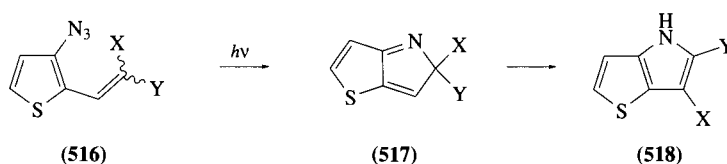
The intramolecular capture of a nitrene by a suitably located alkenic carbon was referred to in CHEC-I <84CHEC-I(4)741>. In that case, the nitrene had been generated by the action of triethyl phosphite on a nitro group. The same type of reaction has been carried out by thermolysis of an azide. Thus, thermolysis of the 3-azido-2-vinylthiophene derivative (510) gave the substituted thienopyrrole (511) (Scheme 108). However, under similar conditions, the 3-azido-4-vinylthiophene derivative (512) gave only the amine (513). But the dithienylethane derivative (514) gave the cyclized product (515) <86JHC513>. Photolysis of 3-azido-2-vinylthiophenes (516) in which the terminal alkene carbon atom carries two substituents, leads to the thienopyrrole (517). One of the substituents on the quaternary carbon then migrates (1,5-sigmatropic shift), ultimately resulting in (518) (Scheme 109). Using a series of substrates with different X and Y groups, the order of migratory aptitude has been determined to be $\text{RSO} > \text{RS} \approx \text{H} > \text{RSO}_2 > \text{RCO} > \text{EtO}_2\text{C}$ <85CC1818>.

The nitrene generated by thermolysis of 3-azidothiophene can also be captured by a suitably positioned divalent sulfur to form an S,N-ylide <86JCS(P1)483>. 2-Azathiabenzene (519) fused to the thiophene ring have been prepared in this manner (Scheme 110). The 3,4-disubstituted thiophene (520) gave an unstable cyclic sulfimide (521). Cyclic sulfimides in which the positions of the sulfur and nitrogen atoms are reversed with respect to the thiophene ring could also be made by this procedure.

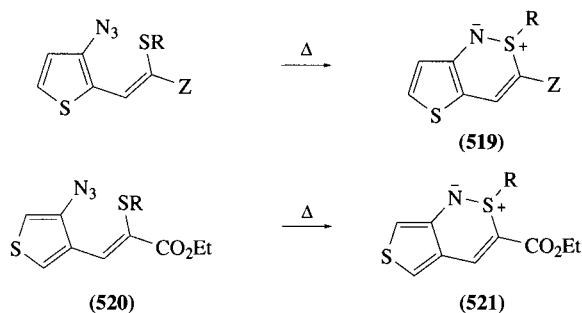
Thermolysis or photolysis of aromatic azides can lead to trappable azirines in equilibrium with singlet nitrenes. 3-Azidobenzo[*b*]thiophene was heated in Et_2NH or Me_2NH in a sealed tube at 90 °C for 5 h. This gave (524) in 75% yield, along with about 10% of the amine (525). This has been explained as follows. Thermolysis of the azide gives the azirine (522); this can either react with R_2NH to give (524), or rearrange to singlet nitrene (523a). Intersystem crossing gives the triplet (523b), which can then yield (525) (Scheme 111) <89G411>. Interestingly, 2-azidobenzo[*b*]thiophene



Scheme 108



Scheme 109

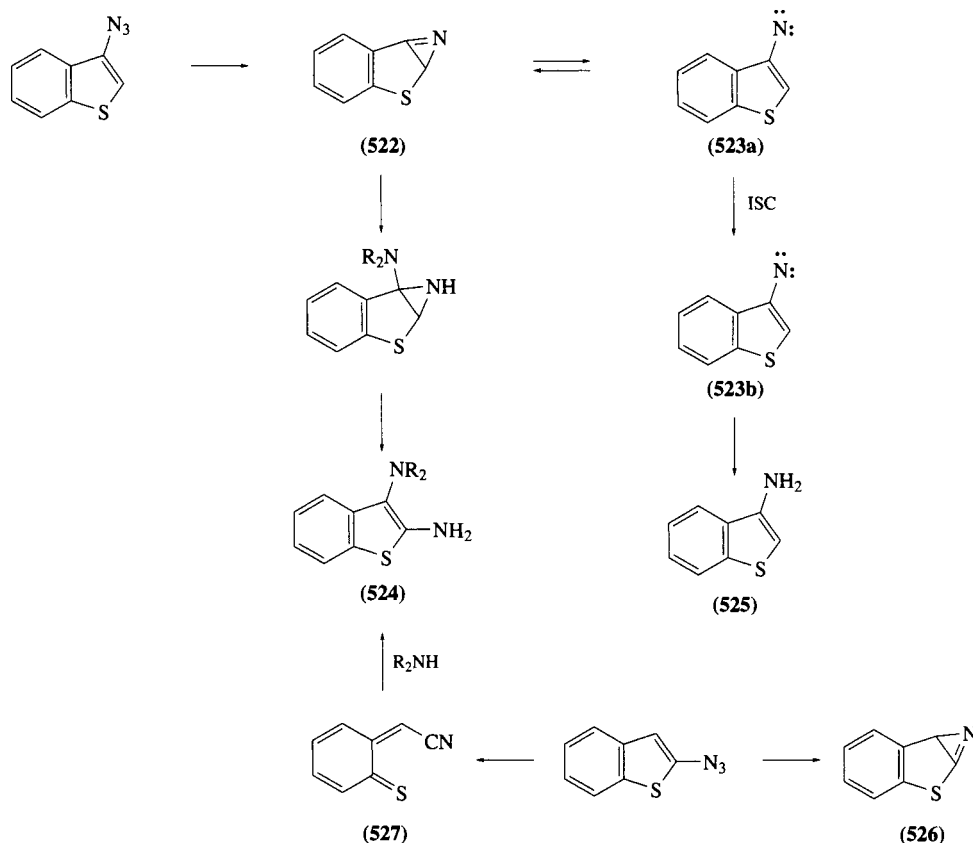


Z = CO₂Et, CHO or CN
R = alkyl or Ph

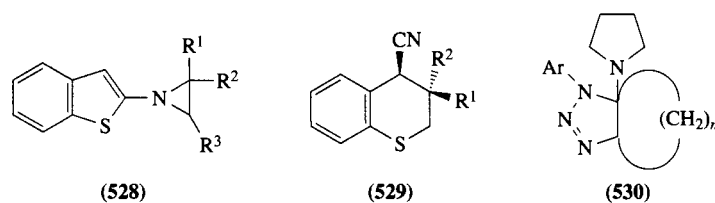
Scheme 110

also gives (524) under the same conditions. Since this cannot be explained as arising from the azirine (526), a ring opening to (527) has been postulated (see Scheme 111).

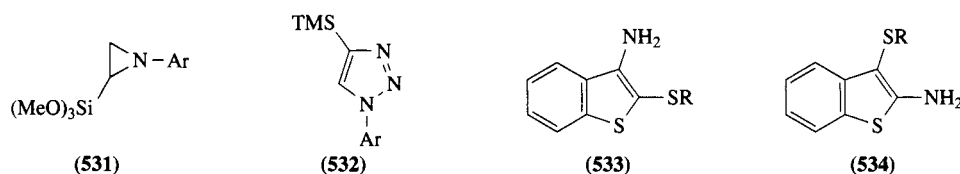
Such a ring opening of a 2-nitrenothiophene was also postulated earlier to account for the formation of a pyrrole derivative. Definitive evidence for this type of ring opening has now been provided in the benzo[*b*]thiophene series (88JCS(P1)3375). Thermal decomposition of 2-azido-benzo[*b*]thiophene in the presence of a number of electron-rich and electron-poor alkenes has given the aziridines (528) and/or 4-cyanothiochromans (529) in good yields. The latter can obviously arise by cycloaddition of the alkene to (527). The formation of aziridines (528) is favored by electron-poor alkenes (methyl acrylate, acrylonitrile) and by a decrease in the reaction temperature. Since no triazolines could be detected in the product mixture, it was suggested at this point that a singlet nitrene was the common precursor to both (527) and (528). However, further studies suggest that aziridines may indeed be formed via triazolines, and the enethione (527) by concerted ring opening and nitrogen extrusion from the azide (90JCS(P1)2971). These conclusions are based on the following two critical observations: the 2-azide reacts with cyclic enamines to form the triazolines (530) quantitatively. With other, stereodefined alkenes, it was found that the formation of the aziridines (528) was nonstereospecific—a finding inconsistent with addition of singlet nitrene to a C=C double bond.



Scheme 111

Ar = 2-benzo[b]thienyl; $n = 3$ or 4

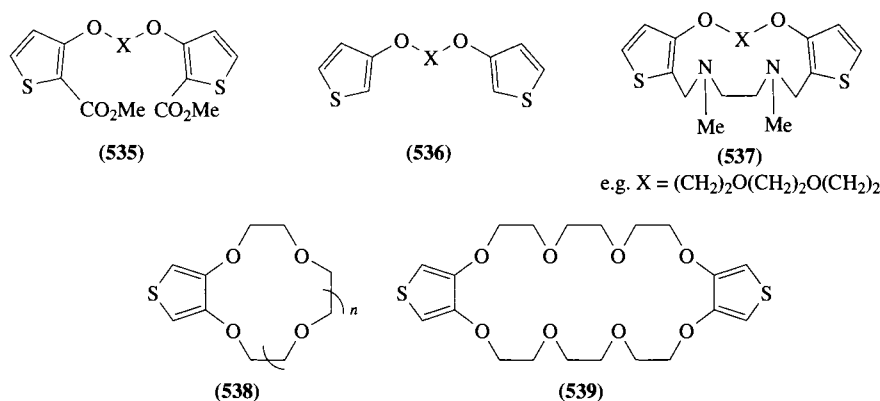
2-Azido- and 3-azidobenzo[b]thiophene have been reacted with trimethoxy(vinyl)silane to give the aziridines (**531**) in quantitative yield. No evidence for the cycloadduct, triazoline, could be obtained. The azides, however, cycloadd to (trimethylsilyl)acetylene at room temperature to give the triazoles (**532**) in quantitative yield <91JCS(P1)2789>. A perplexing observation is that 3-azido-benzo[b]thiophene reacts with alkanethiols at room temperature to give the 2-alkylthio-3-amino-benzo[b]thiophene (**533**), while the 2-azide gives the isomeric 3-alkylthio-2-aminobenzo[b]thiophene (**534**) <89G411>.



Ar = 2-benzo[b]thienyl or 3-benzo[b]thienyl

2.10.4.4 O-Linked Substituents

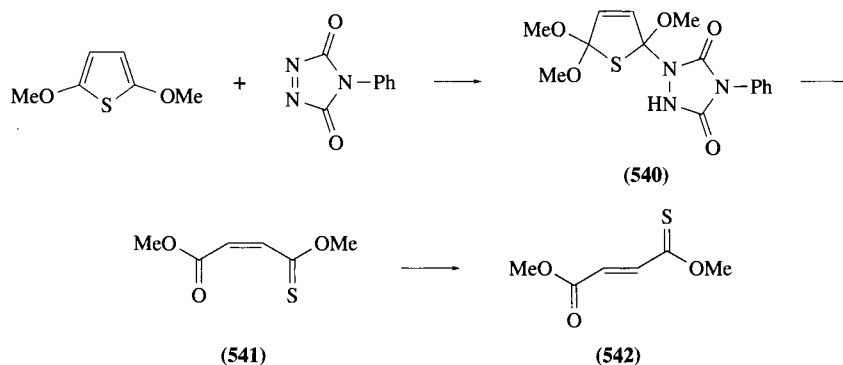
Several thiophene-based macrocyclic ethers have been synthesized. Since hydroxythiophenes themselves are not stable enough for this purpose, the strategy has been to employ the corresponding *ortho*-hydroxythiophene carboxylic esters as the starting materials and to remove the carboxylic function at a later stage. Thus, 3-hydroxythiophene-2-carboxylic ester has been converted to the ether (**535**), hydrolyzed and decarboxylated to (**536**). A Mannich reaction on this leads to the desired macrocycle (**537**) <93CC1733>. Similarly, crown ethers of the types (**538**) and (**539**) have been synthesized starting from 3,4-dihydroxythiophene-2,5-dicarboxylic ester <89BCJ838>.



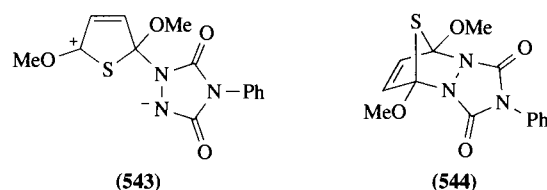
Base catalysis in the piperidino-demethoxylation of 2-methoxy-3-nitrothiophene was reported earlier <82JCS(P2)721, 84CHEC-I(4)741>. A similar study has now been carried out on 3-methoxy-5-methyl-2-nitrothiophene in methanol <93ACS157>. The reaction is catalyzed by methoxide. The mechanistic pattern is not significantly different from the previous one.

The hydrolysis of 2,5-dimethoxythiophene leads to methyl 2-methoxythiocarbonylpropionate. The corresponding thiomaleate (**541**) has now been prepared <82CC1033>. Reaction of 4-phenyl-1,2,4-triazolin-3,5-dione with 2,5-dimethoxythiophene in wet methanol gives (**541**) in 87% yield (Scheme 112); this easily isomerizes to the thiofumarate (**542**). If the reaction is run in anhydrous methanol at -15°C , the unstable adduct (**540**) can be isolated. This rapidly hydrolyzes to (**541**). The formation of (**540**) could have taken place via either the zwitter ion (**543**) or the Diels–Alder adduct (**544**).

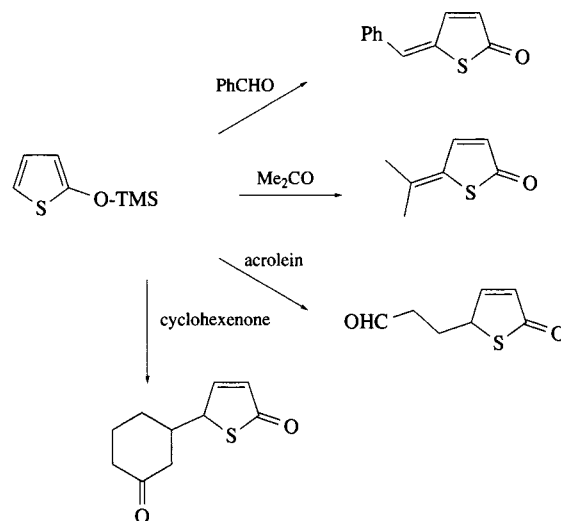
2-(Trimethylsilyloxy)thiophene has been synthesized and its reactivity studied <84JOC551>. Treat-



Scheme 112

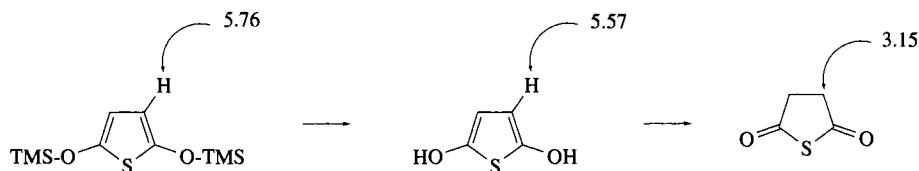


ment of 2,5-dihydrothiophene-2-one with 2 equiv. of trimethylchlorosilane and 1.2 equiv. of (diethylamino)trimethylsilane gave 2-(trimethylsilyloxy)thiophene in 65% yield. In the presence of Lewis acid catalysts (SnCl_4 or AlCl_3), this reacts with carbon electrophiles to give 5-substituted 2,5-dihydrothiophene-2-ones (Scheme 113). In contrast, 3-(trimethylsilyloxy)thiophene did not lead to the expected products with carbon electrophile $\langle 86\text{TL}5155 \rangle$.

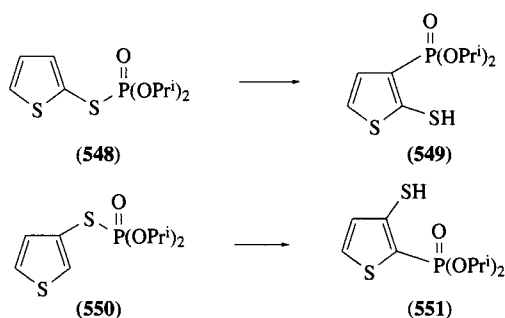


Scheme 113

The generation of unstable enol tautomers in solution has been discussed in Section 2.10.3.4. The method depends on careful hydrolysis of the *O*-trimethylsilyl derivatives. This has been extended to 2,5-dihydroxythiophene as well $\langle 89\text{JOC}1211 \rangle$. 2,5-Bis[(trimethylsilyl)oxy]thiophene in DMSO-d_6 was treated with 1% of $7 \times 10^{-4} \text{ M}$ DCl in D_2O ; the ^1H NMR signals of the starting material disappeared almost immediately, to be replaced by those of 2,5-dihydroxythiophene. Over the course of 1 h, this species changed over to succinic thioanhydride (Scheme 114a).



Scheme 114a

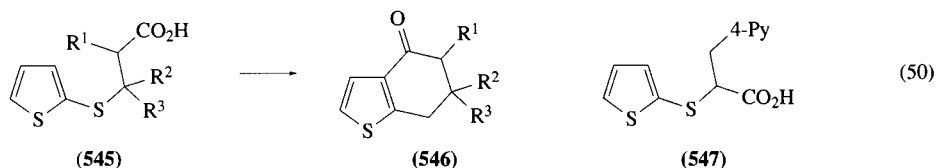


Scheme 114b

2.10.4.5 S-Linked Substituents

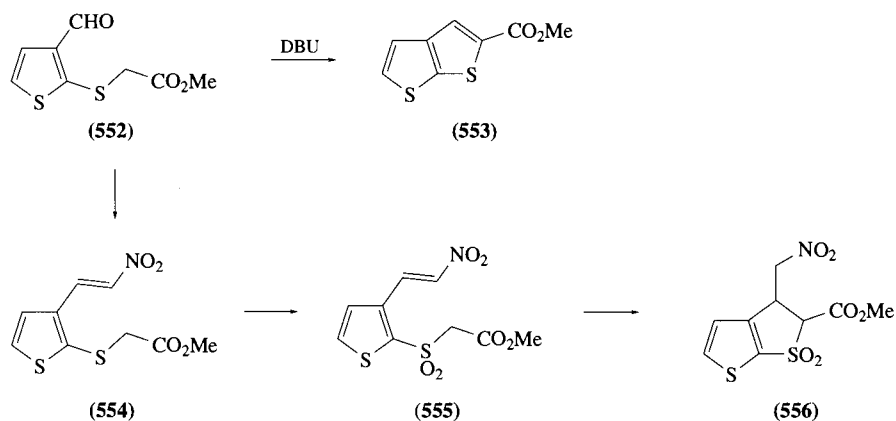
2-Mercaptothiophene has been added to various acrylic acids; the Michael adducts (**545**) have been cyclized to (**546**) (Equation (50)) $\langle 88\text{JOC}9 \rangle$. (4-Pyridyl)acrylic acid gave an anomalous product—the

anti-Michael adduct (**547**); presumably the addition took place on the protonated pyridinium species. The TBDMS ester of (4-pyridyl)acrylic acid gives the normal Michael adduct.



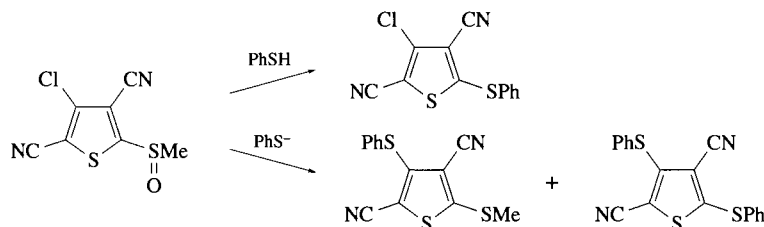
Sulfur-to-carbon migration of a phosphorus substituent has been reported (94TL3083). *O,O*-Diisopropyl-*S*-(2-thienyl)thiophosphate (**548**) obtained by phosphorylation of thiophene-2-thiolate was treated with LDA in THF. This induced the rearrangement, leading to diisopropyl(2-mercapto-3-thienyl)phosphonate (**549**) in 21% yield (Scheme 114b). *O,O*-Diisopropyl-*S*-(3-thienyl)thiophosphate (**550**) was prepared from lithium thiophene-3-thiolate and diisopropyl chlorophosphate. Similar rearrangement of this gave diisopropyl(3-mercapto-2-thienyl)phosphonate (**551**) in 47% yield.

S-(3-Formyl-2-thienyl)mercaptoacetic ester (**552**) has been cyclized by base to thieno[2,3-*b*]thiophene (**553**) (Scheme 115). Similarly, the isomeric *S*-(2-formyl-3-thienyl)mercaptoacetic ester gives thieno[3,2-*b*]thiophene (91JMC1805). The thieno[2,3-*b*]thiophene system has also been built up from (**552**) (91JHC13). The strategy was to use an intramolecular Michael addition to construct the second thiophene ring. The nitroalkene (**554**), however, could not be induced to undergo ring closure. Oxidation to the sulfone (**555**) and treatment with base led to ring closure, forming the product (**556**). The sulfone could ultimately be deoxygenated by reduction with dibal-H.



Scheme 115

Alkylsulfinyl groups at position 2 of an activated thiophene undergo facile nucleophilic displacement by alkyl or aryl mercaptans (Scheme 116) (88T5921). However, if thiophenolate anion is used in this reaction instead of neutral thiophenol, the products are completely different.



Scheme 116

2.10.4.6 Halo Groups

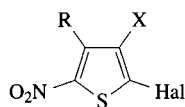
An exhaustive review on the reactivity of halothiophenes is available (86HC(44/2)159). This deals with the reactions of halothiophenes with electrophiles, nucleophiles, and radicals, as well as the reductive dehalogenation of halothiophenes.

2.10.4.6.1 Nucleophilic displacement

Nucleophilic displacement reactions of halogenated thiophenes fall into two categories: (i) those pertaining to substrates in which the halogen is activated by a suitably located electron-withdrawing substituent; and (ii) those dealing with unactivated halothiophenes. Both classes were discussed in detail in CHEC-I <84CHEC-I(4)741>. In addition, a detailed account of such nucleophilic displacements has been provided in recent reference books <86HC(44/2)159, 92HC(44/4)295>.

(i) Activated halothiophenes

Spinelli and co-workers have shown that linear free-energy *ortho*-correlations can be successfully established for the nucleophilic displacement reactions on activated halogenothiophenes. This has been validated on the benzenethiolate debromination of 2-bromo-3-*X*-5-nitrothiophenes (**557**) <91JCR(S)270>, the chlorine-isotopic exchange between $^{36}\text{ClLi}$, and some 2-chloro-3-*X*-5-nitrothiophenes (**558**) <88JCR(S)198>, and the piperidino-debromination of some 2-bromo-3-*X*-4-methyl-5-nitrothiophenes (**559**) <85JCS(P2)519>.



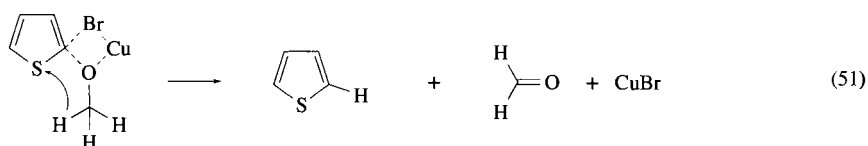
(**557**) Hal = Br; R = H
 (**558**) Hal = Cl; R = H
 (**559**) Hal = Br; R = Me

X = Me, H, Br, Cl, CONH₂, CO₂Me, NO₂, SO₂Me, Ac, CN

Base catalysis of piperidino-demethoxylation of 2-methoxy-3-nitrothiophene was discussed in CHEC-I <84CHEC-I(4)741>. The study has been extended to the reaction of 2-methoxy-5-methyl-3-nitrothiophene with pyrrolidine and piperidine <93JCR(S)440>. The push-pull nature inherent in the 2-methoxy-3-nitrothiophene system apparently makes the methoxy group a sluggish nucleofuge; hence the need for catalysis. The displacement of bromine in 2-bromo-3,5-dinitrothiophene by various *meta*- and *para*-substituted anilines also appears to be base-catalyzed <90JCS(P2)2153>.

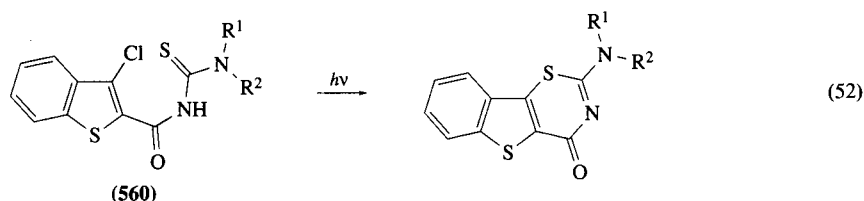
(ii) Unactivated halothiophenes

The best conditions for the nucleophilic displacement of bromine from 2- and 3-bromothiophene by alkoxide have been worked out <90SC213, 92T3633>. Thus, 2-methoxythiophene could be prepared in 83% yield by refluxing 2-bromothiophene in methanol containing excess sodium methoxide, along with copper(I) bromide as catalyst. For the analogous preparation of 3-methoxythiophene, addition of a polar cosolvent (e.g. 1-methyl-2-pyrrolidone) was found to be beneficial. It was necessary in all cases to use a high concentration of NaOMe in MeOH (10 mol L⁻¹). It is believed that a copper(I) alkoxide is formed first, which is the actual catalyst. An interesting observation which came to light in the course of the above investigations was the fact that 2-bromothiophene was prone to reductive debromination especially if a polar cosolvent was present. A copper-mediated mechanism has been proposed to explain this; the mechanism involves a hydride shift as shown in Equation (51). Dibromothiophenes are even more sensitive to the reduction process than 2-bromothiophene. Thus, in the reaction of 2,3-dibromothiophene with methoxide, the product mixture consisted of 2,3-dimethoxythiophene (30%) and 3-methoxythiophene (60%).



The Grignard cross-coupling between 3-bromothiophene and chlorotrimethylsilane to yield (3-thienyl)trimethylsilane is accelerated several fold by ultrasonic irradiation <93SC1235>.

Photolysis of the thioureas (**560**) leads to cyclization by displacement of the chlorine (Equation (52)) <86CCC2839>.



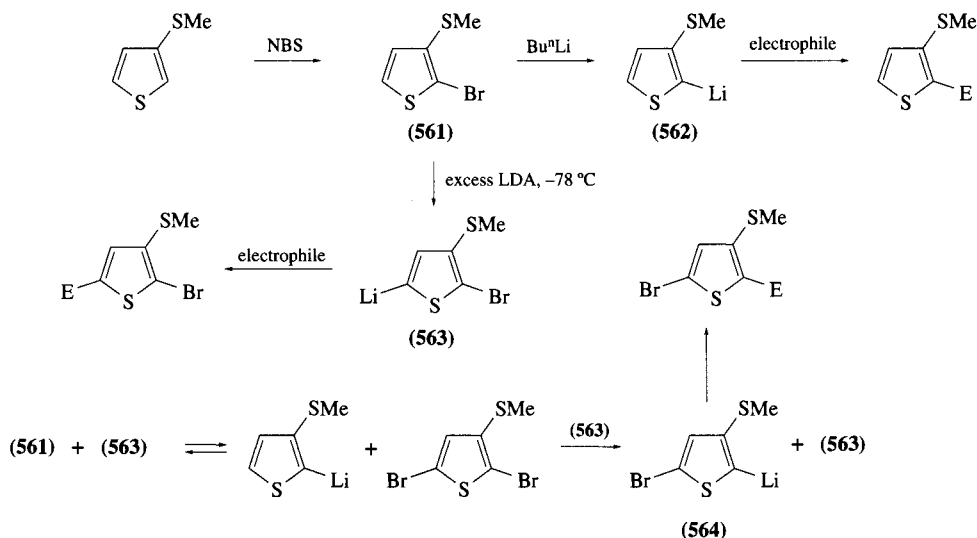
2.10.4.6.2 Halogen-metal exchange

It has been found that 3-lithiothiophene is stable even at room temperature in hexane <94TL3673>. This makes it easy for the synthesis of 3-functionalized thiophenes, since even less reactive electrophiles can be used. A typical procedure involves reaction of 3-bromothiophene with Bu^nLi at -40°C in hexane to which about 10% THF has been added. The solution is then warmed to room temperature and treated with electrophiles to give good yields of the products. C—C, C—S, C—hal and C—Si bonds have been created at position 3 by this method.

The stepwise lithium exchange of 2,3-dibromothiophene referred to in CHEC-I <84CHEC-I(4)741> has been used to generate a 1-alkenylthieno [2,3-*c*]furan <88TL1137>. The 2-lithio-3-bromothiophene is first reacted with DMF to get the 2-carbaldehyde; then the 3-lithio compound is reacted with a ω -unsaturated aldehyde. The product is converted to the required thieno [2,3-*c*]furan by standard reactions.

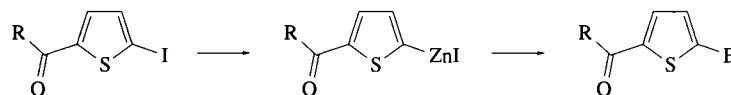
The relative merits of direct deprotonation and halogen-metal exchange for the regiospecific generation of thienyllithium derivatives, and the use of these two as complementary methods have been discussed <85JOC1002>. Deprotonation of 3-(methylthio) thiophene with Bu^nLi leads to a 3 : 1 mixture of 2-lithio and 5-lithio derivatives. On the other hand, NBS bromination of 3-(methylthio)thiophene, followed by treatment of the bromo compound (**561**) with Bu^nLi , gives exclusively the 2-lithio derivative (**562**). Deprotonation of the bromo compound (**561**) by slow addition to an excess of LDA at -78°C leads to the 5-lithio derivative (**563**) (since LDA is a strong base, but a poor nucleophile). Reaction of these lithio derivatives (**562**) and (**564**) with the appropriate electrophilic reagent leads to the required products, which can be debrominated if necessary. In the deprotonation of (**561**), if only one equivalent of LDA is used instead of excess, and if the addition is so rapid that there is in fact an excess of (**561**) then a rearrangement can take place to give the isomeric thienyllithium (**564**) (Scheme 117).

2-Thienylzinc derivatives have been prepared from the corresponding 2-iodothiophenes under



Scheme 117

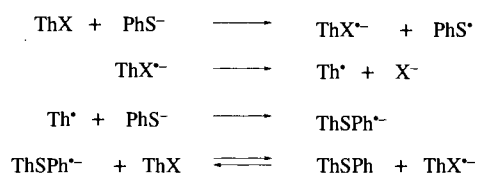
mild conditions in >80% yield by treatment with a Zn(Ag) couple deposited on graphite <94TL1047>. The zinc insertion is facilitated by the presence of an electron-withdrawing group on the thiophene ring (Scheme 118). The thienylzinc derivative can be reacted with electrophiles under Pd⁰ catalysis or after transmetalation with copper by means of CuCN·2LiCl.



Scheme 118

2.10.4.6.3 Generation of thienyl radicals

A few further examples of nucleophilic displacement of halogen from halogenothiophenes by a radical chain S_RN₁ mechanism have been reported. The photostimulated reaction of 2-halo or 3-halothiophene with excess of tetrabutylammonium benzenethiolate in MeCN takes place by an S_RN₁ mechanism <87JOC5382>. The 2- or 3-(phenylthio)thiophene is formed in modest yields. The initiation and propagation steps are shown in Scheme 119. The first step may be the photostimulated electron transfer from thiolate anion to the halothiophene (ThX). The reaction does not take place in the dark.

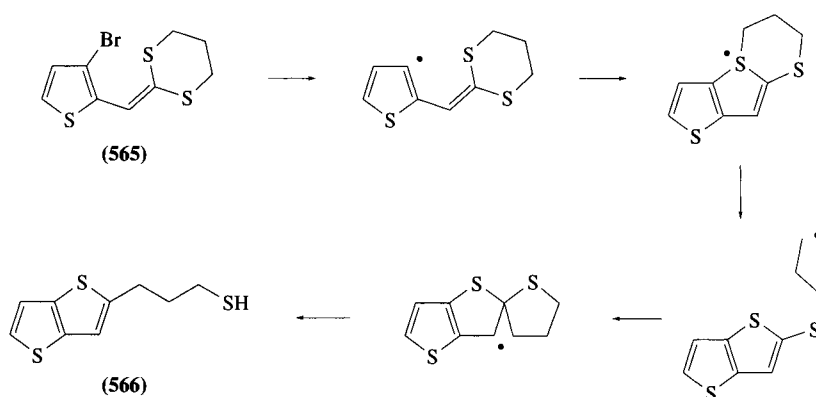


Scheme 119

Potassium enolates of cyclohexanone, acetone, and acetophenone react with 3-iodobenzo[*b*]thiophene in DMSO in the dark to give the α-heteryl ketones in low yield <92H(34)1039>. The initiation step in the radical chain process is probably an electron transfer from the enolate to the 3-iodobenzo[*b*]thiophene. The low yields of the products are partly due to competing ionic reactions.

An extremely interesting example of “cascade” radical reactions has been reported recently <93TL5653>. The initial radical generation from the 3-bromothiophene (**565**) is brought about by tributyltin hydride. This is then followed by cyclization, fragmentation, recyclization by the 5-*exo*-trig mode and a final fragmentation. After all this, the product (**566**) is obtained in 74% yield (Scheme 120).

The photolysis of iodothiophene to generate thienylradical and subsequent reaction with benzene



Scheme 120

to form phenylthiophene has been mentioned in CHEC-I. This has been extended to the synthesis of α -terthiophene (89G419). The photoreaction of 2,5-diiodothiophene with thiophene in MeCN solution gave α -terthiophene in 91% yield.

2.10.4.6.4 Reductive dehalogenation

3-Bromothiophene can be prepared by selective debromination of 2,3,5-tribromothiophene by zinc and acetic acid (73OSC(5)149). This selective debromination strategy has been extended to the 2,2'-bithiophene series as well (91H(32)1805). 3,3',5,5'-Tetrabromination of 2,2'-bithiophene (bromine, glacial acetic acid) was followed by selective debromination by means of zinc/acetic acid/*n*-propanol/water to give the 3,3'-dibromo compound in 88% yield. Subsequent (NiCl₂ · dppp)-catalyzed Grignard cross-coupling gave 3,3'-dialkyl-2,2'-bithiophenes.

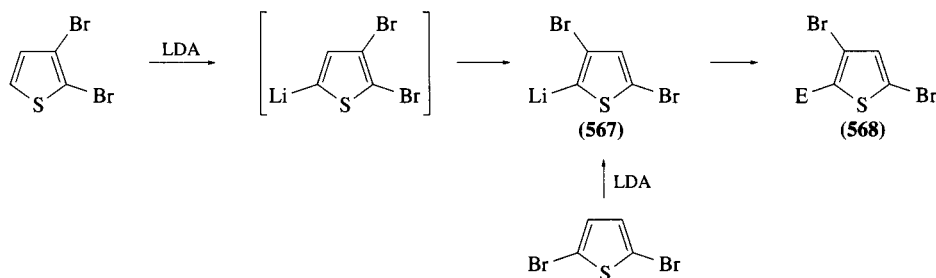
Reductive debromination of bromothiophene-2-carboxylic acids with Raney Cu–Al alloy in alkaline D₂O gives the corresponding deuteriated thiophene-2-carboxylic acids in high isotopic purity (88JCR(S)136).

A simple method has been described for the preparation of 3-bromo- and 3,4-dibromothiophenes from polybrominated thiophenes by selective debromination brought about by electrochemical reduction (90S403). The yields are very good. It is possible to control the reaction conditions so as to achieve either mono-debromination (position 2) or di-debromination (positions 2 and 5).

2.10.4.6.5 Rearrangement reactions

α -Halothiophenes undergo rearrangement reactions in strongly basic media, resulting in the formation of thermodynamically more stable products. This 'halogen dance' of thienyl halides was discussed extensively in CHEC-I (84CHEC-I(4)741). Based on this, methods have been developed for the large-scale synthesis of 3-bromo- and 3,4-dibromothiophene. Treatment of 2-bromothiophene with excess sodamide in liquid ammonia, and subsequent quenching with solid NH₄Cl, gives 3-bromothiophene in 73% yield (90SC1697). The synthesis of 3,4-dibromothiophene from 2-bromothiophene depends on an efficient disproportionation. This is facilitated by treatment with a substoichiometric amount of a mixture of equimolar amounts of sodamide and potassium *tert*-butoxide in liquid ammonia. A mixture of products is obtained, the main component (58–68%) of which is 3,4-dibromothiophene (90SC2119).

Deprotonation of 2,3-dibromothiophene by LDA in THF at –80°C, followed by reaction with methyl iodide gave 3,5-dibromo-2-methylthiophene instead of the expected 2,3-dibromo-5-methylthiophene. Obviously, the initially generated 2,3-dibromo-5-thienyllithium has rearranged to 3,5-dibromo-2-thienyllithium (567). Quenching the lithiated species (567) with dry methanol gave 2,4-dibromothiophene (89S771). Lithiation of 2,5-dibromothiophene with LDA under similar conditions also gave (567) by rearrangement (Scheme 121) (83H(20)2035).



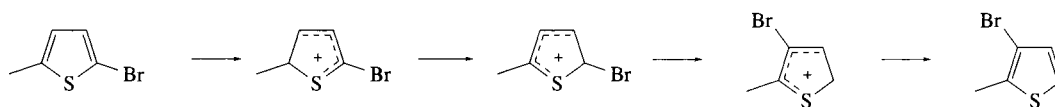
Scheme 121

It had been reported earlier (72TL3507, 84JOC5250) that deprotonation (LDA) of 2,5-dibromothiophene, followed by quenching with trimethylsilyl chloride, gave 2,5-dibromo-3-[trimethylsilyl]thiophene. This is obviously not correct in the light of the rearrangements discussed above. The actual product formed in this reaction has been proved to be 3,5-dibromo-2-(trimethylsilyl)thiophene (568; E = SiMe₃) (90JOC2993). The same product is also obtained by treating 2,3-dibromothiophene with LDA in THF and quenching with trimethylsilyl chloride (Scheme 121). The structure

of (568) ($E = \text{SiMe}_3$) was established by α -debrominating it and comparing the product with authentic 3-bromo-2-(trimethylsilyl)thiophene.

The rearrangements discussed above have all been brought about by strong base. The most surprising development in this area, however, is the discovery that β -halothiophenes can be obtained in good yields by isomerization of α -halothiophenes on silicon-rich zeolites of the pentasil type <87AG(E)468>. The zeolite used is the strongly acidic H-ZSM-5. Thus, 2-chlorothiophene is converted to 3-chlorothiophene with high selectivity on a H-ZSM-5 fixed bed catalyst at 300–450°C and atmospheric pressure. The advantage of this procedure is that secondary processes such as oligomerization or sulfur elimination do not seem to occur. Similarly, 2-bromo- and 2-iodothiophenes are also isomerized in the liquid phase at 150°C and 100°C, respectively. Interestingly, 2-bromo-5-chlorothiophene is isomerized at 180–190°C to 4-bromo-2-chlorothiophene. The mechanism of these isomerizations has not been discussed in detail.

The only other comparable rearrangement takes place on protonation of 2-bromo-5-methylthiophene by HSO_3F <86BCJ307>. Initially, at -70°C , protonation takes place at position 5; at -50°C , this changed almost completely to 2*H*-2-bromo-5-methylthiophenium ion; but from -30°C to $+10^\circ\text{C}$, this underwent another change to form 2*H*-4-bromo-5-methylthiophenium ion. Quenching with water and extraction gave 3-bromo-2-methylthiophene (Scheme 122).



Scheme 122

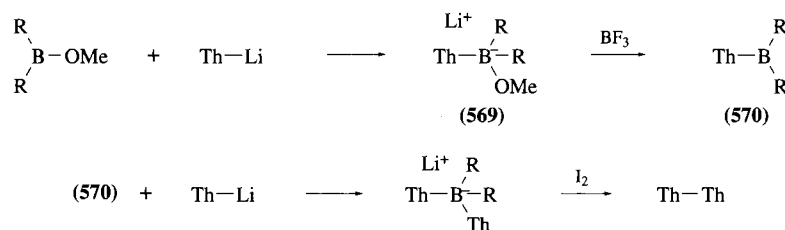
2.10.4.7 Metallo Groups

2.10.4.7.1 Metal-metal exchange

Thienyllithium compounds undergo a wide variety of transmetallation reactions. Some of these (B, Si, Se, Cu) were referred to in CHEC-I. A compilation of data published during 1949–1985 is now available <92HC(44/5)257>. Hence only the more recent and interesting developments in this area are discussed below.

(i) C—B bond

As mentioned in CHEC-I, thienylboronic acids can be prepared from thienyllithium derivatives. A series of pyridylthiopheneboronic acid esters have been prepared and converted to the respective hydroxythiophenes <92ACS654>. The lithium–boron exchange constitutes the first step in the stepwise synthesis of α -thiophene oligomers (Scheme 123) <83TL4043>. The resulting ate-complex (569) was neutralized with $\text{BF}_3 \cdot \text{Et}_2\text{O}$ and the product (570) again reacted with a second equivalent of 2-thienyllithium. Final oxidative coupling with iodine led to 2,2'-bithienyl. The sequence could be repeated further to give oligomers.

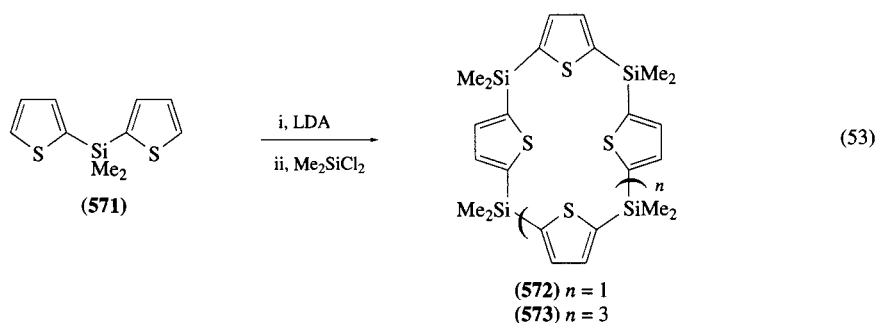
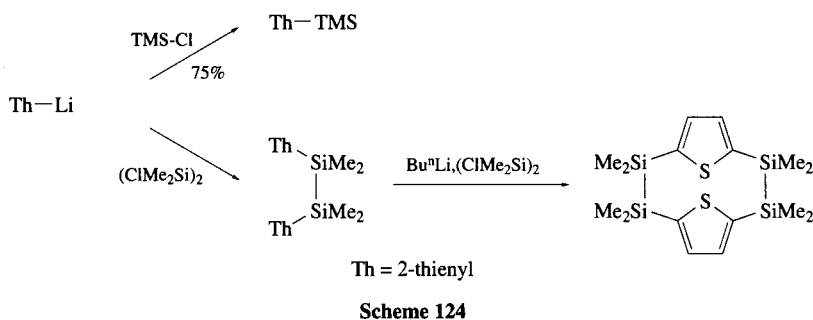


Th = 2-thienyl

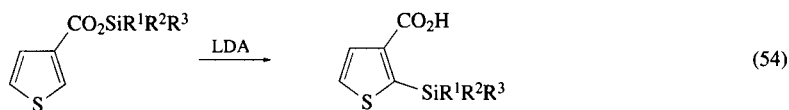
Scheme 123

(ii) C—Si bond

The synthesis of 2-(trialkylsilyl) thiophenes from 2-thienyllithium and the corresponding chlorosilanes is extremely facile and useful. Disilanes have also been used in this reaction, leading to macrocycles containing silicon (Scheme 124) <88T281>. 2,5-Disilylthiophenes have been prepared similarly. Treatment of 2-thienyllithium with 0.5 equiv. of dichlorodimethylsilane yields (**571**) in 74% yield <92H(34)1085>. This could be converted to the macrocycles (**572**) and (**573**) by lithiation followed by treatment with dichlorodimethylsilane (Equation (53)).



An extremely interesting development is the intramolecular silyl group migration from a silyl ester of thiophene-3-carboxylic acid to position 2 of the ring (Equation (54)) <91SL33>. The yields are moderate. 3-Thienyllithium has been converted to 3-(tri-*n*-butylsilyl)thiophene by reaction with *n*-Bu₃SiCl <94TL3673>.

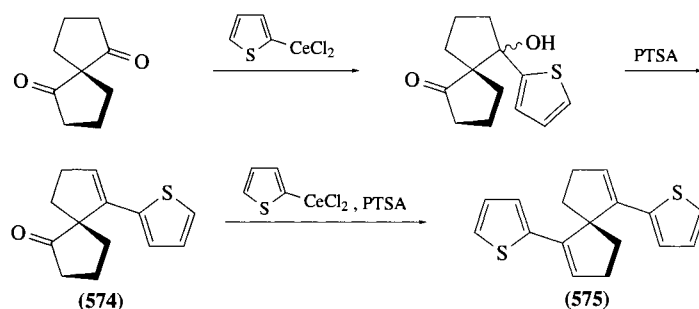


(iii) C—Ge and C—Sn bonds

Trialkylsilicon, germanium or tin derivatives have been introduced at position 5 of thiophene-2-carbaldehyde via lithiation <92S954>. The first step is the protection of the aldehyde as the amino-alkoxide by reaction with lithium *N*-methylpiperazide (LNMP). Subsequent treatment with BuLi followed by R³MCl and hydrolysis by cold brine gave the desired products.

(iv) C—Ce bond

2-Thienylcerium chloride has been prepared from 2-thienyllithium. This has been used to prepare spiro compounds such as (**574**) and (**575**) (Scheme 125) <91CC1614>.

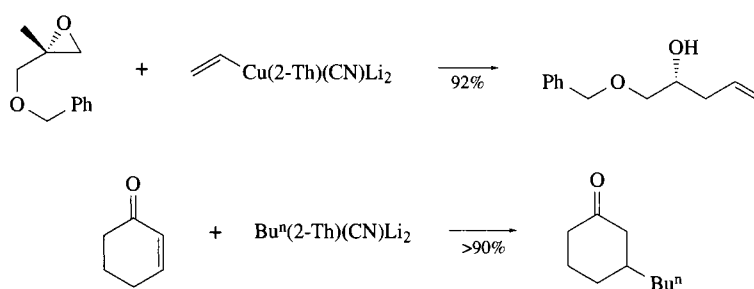


Scheme 125

(v) Cyanocuprates

2-Lithiothiophene has found application as a “dummy ligand” in higher-order cyanocuprates $\langle 87S325 \rangle$. Addition of CuCN to 2-thienyllithium leads to the formation of the lower-order cuprate $(2\text{-Th})\text{Cu}(\text{CN})\text{Li}$ [$(2\text{-Th}) = 2\text{-thienyl}$]. This has a good shelf-life and can be stored, making it a convenient reagent for making the higher-order cuprate $\langle 87TL945 \rangle$. Subsequent introduction of a molecule of another alkyl or alkenyllithium leads to the higher-order cuprate $\text{R}(2\text{-Th})\text{Cu}(\text{CN})\text{Li}_2$. In reaction with various electrophiles, such as epoxides, alkyl halides, α,β -unsaturated ketones, etc. it is the R group which is transferred and not the 2-thienyl group (Scheme 126). This concept has been extended to dilithium (trimethylstannyl)(2-thienyl)(cyano)cuprate, $\text{Me}_3\text{SnCu}(2\text{-Th})(\text{CN})\text{Li}_2$ $\langle 88JOC5366 \rangle$. This reagent effects the conjugate transfer of the Me_3Sn group to a variety of α,β -unsaturated carbonyl compounds and α,β -acetylenic esters.

A highly reactive zero-valent copper species has been prepared by direct reduction of lithium 2-thienylcyanocuprate in THF with lithium naphthalenide $\langle 89SC1833 \rangle$.



Scheme 126

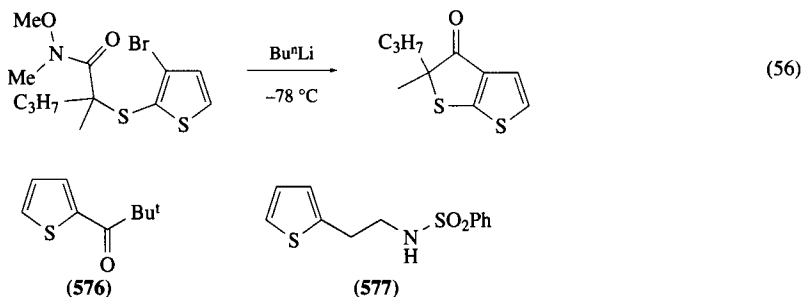
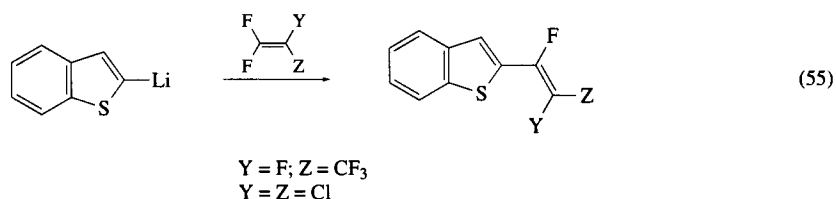
2.10.4.7.2 Formation of C—C bonds

The formation of C—C bonds by treating thienyllithiums with appropriate electrophiles was discussed in detail in CHEC-I $\langle 84\text{CHEC-I}(4)741 \rangle$. Hence only the more recent and novel applications of this technique will be reported in this section.

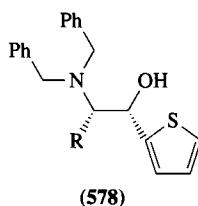
An interesting new addition to the methods available for constructing arylthiophenes is the addition of thienyllithium to arynes generated from aryl triflates and LDA $\langle 93JOC4722 \rangle$. Both *ipso*- and *cine*-substitution are possible. *o*-Tolyl triflate gave the *ortho*- and *meta*-substituted products in the ratio 43 : 57, but 2-methoxyphenyl triflate gave exclusively the *meta*-substituted product.

The haloethenyl substitution method, referred to in CHEC-I, has been extended to benzo[*b*]-thiophenes (Equation (55)) $\langle 89JOC3311 \rangle$. *N*-Methoxy-*N*-methylamides have turned out to be useful acyl transfer reagents by reaction with thienyllithiums $\langle 90TL6269 \rangle$. A side reaction is the formation of thienylmethanol by reaction with the formaldehyde liberated from the reagent. For intramolecular acylation on a 3-lithiothiophene derivative, the *N,O*-dimethylhydroxamate has been found to be the best electrophile (Equation (56)) $\langle 91TL721 \rangle$. The reaction of 2-thienyllithium with *N*-acylated aziridines has been investigated $\langle 90LA113 \rangle$. While 1-pivaloylaziridine leads to the ketone (576) (attack at the carbonyl), reaction with benzenesulfonylaziridine leads to opening of the aziridine

ring to form (**577**). This could be converted to 2-aminoethylthiophene by reductive cleavage using sodium bis(2-methoxyethoxy)aluminum hydride.

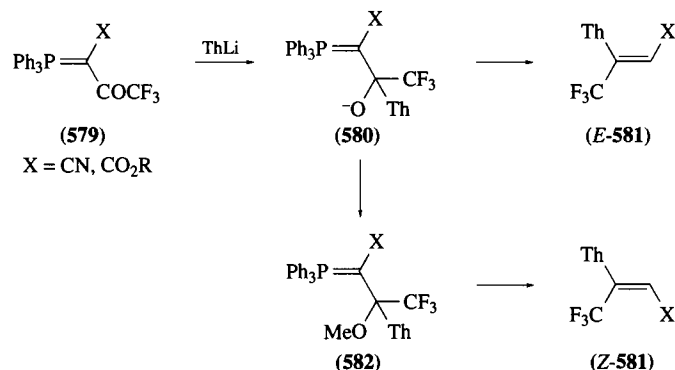


Chiral *N,N*-dibenzylated α -aminoaldehydes react with 2-thienyllithium to form aminoalcohols (**578**) containing two chiral centers. The diastereomeric excess (*de*) ranges from 52 to 90%. A nonchelation controlled addition has been postulated <89H(28)707>.



C—C bond formation via lithiothiophenes has been used for the construction of a heterotriptycene <91CC751> (see Section 2.10.4.2.2). Other complex molecules, such as xestospongins, have been synthesized using thienyllithiums for C—C bond formation <94JA2617>.

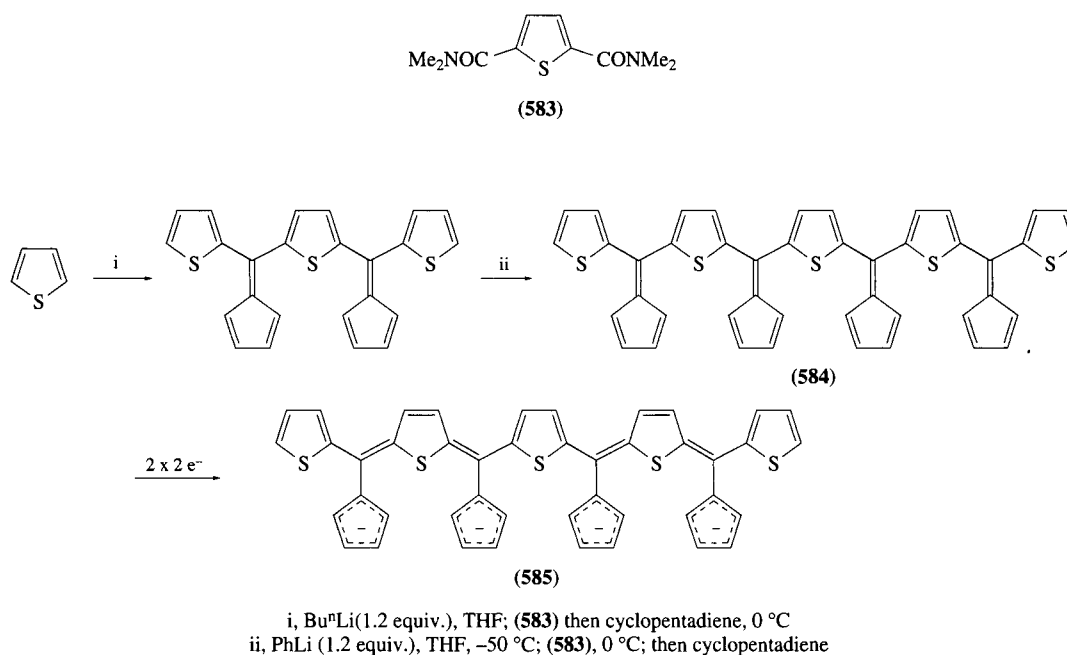
Reaction of 2-thienyllithium with the fluorinated phosphoranes (**579**) gave the ylide anion (**580**) which after protonation and elimination of Ph_3PO afforded the product (**581**) in which the *E*-isomer was predominant. However, methylation of (**580**) with methyl iodide gave the *O*-methyl derivative (**582**); this on hydrolysis gave mainly *Z*-(**581**) (Scheme 127) <94JCS(P1)1473>.



Scheme 127

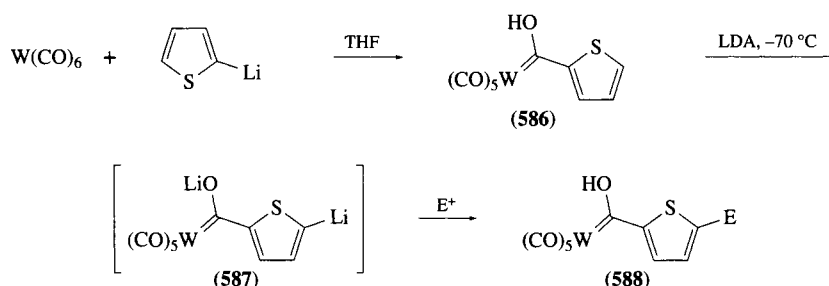
Oligo-6-(2-thienyl)pentafulvenes have been synthesized by converting 2-thienyllithium into a diketone by treatment with the diamide (**583**), and, in a one-pot reaction, condensing the latter with

cyclopentadiene (Scheme 128) <93TL3449>. The tetramer (**584**) is easily reduced electrochemically, probably to a polyanion (**585**) with an oligoacetylene spine.



Scheme 128

Fischer carbene complexes have been generated from 2-thienyllithium. Interestingly, the complex (**586**) can be converted to the dianion (**587**) and reacted with electrophiles to form (**588**) (Scheme 129). No directed lithiation at position 3 could be observed <92JA2985>.

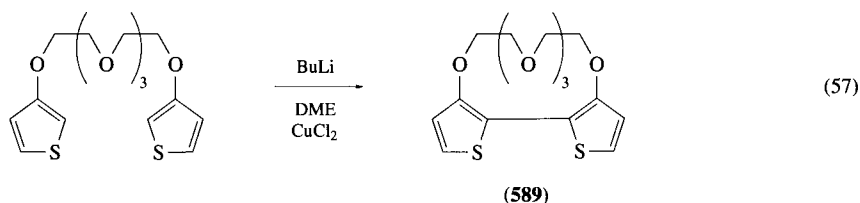


Scheme 129

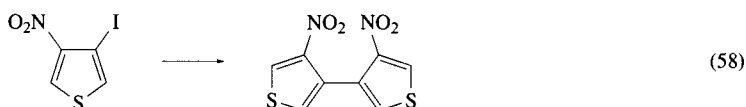
2-Thienylzinc iodides can be prepared from iodothiophenes by treatment with activated zinc on graphite. These can be transmetalated with CuCN·2LiCl and reacted subsequently with an allylic halide, or acylated with acid chlorides <94TL1047>.

The preparation of 2-thienylcerium reagent has been referred to in Section 2.10.4.7.1. This reagent reacts with spirodiketones to form the corresponding alcohols <91CC1614>. Apparently 2-thienyllithium fails in this reaction, but excess 2-thienylmagnesium bromide can be used instead of the cerium reagent.

Oxidative dimerization by treating thienyllithium with copper(II) chloride was reported in CHEC-I. This method has found application in the synthesis of polythiophenes attached to crown ethers; these compounds exhibit selective response to alkali-metal ions and can therefore function as sensors. An example of the synthesis is given in Equation (57) <93JA12214>. The monomer (**589**) was converted to the 5,5'-dilithiated derivative and then polymerized either by direct reaction with Fe(acac)₃ or by conversion to the tin derivative followed by Pd-catalyzed cross-coupling (see Section 2.10.4.7.3).



Ullmann coupling reactions of iodothiophenes were referred to in CHEC-I <84CHEC-I(4)741>. This procedure has been made use of in the synthesis of cyclobuta [1,2-*c*:3,4-*c'*]dithiophene <85CC880>. The first step in the synthesis is the Cornforth modification of the Ullmann procedure (MeCN, Me₂CO, aq. NH₃, Cu, CuSO₄; 25°C) (Equation (58)). The Ullmann reaction has also been used for the synthesis of several substituted phenylthiophene derivatives <85JHC1667>.

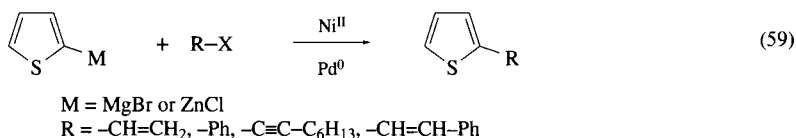


2.10.4.7.3 Nickel- and palladium-catalyzed cross-coupling reactions

An excellent review of Ni- and Pd-catalyzed cross-coupling reactions in heterocycles has appeared recently <92S413>. This has covered literature up to 1990 and has exhaustively described all the interesting transformations in the thiophene series. In the following account, those reactions which do not mention any specific journal source refer to this review article.

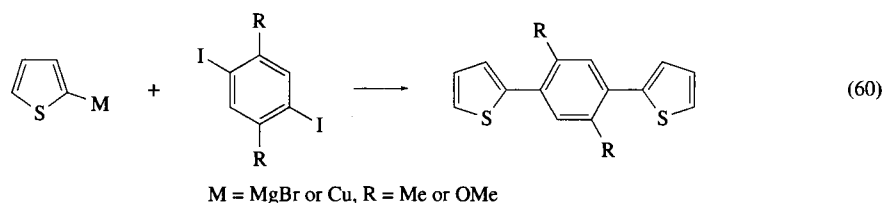
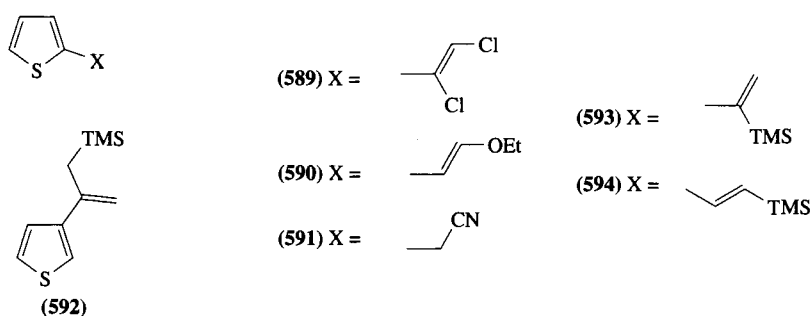
The reaction of thienyllithium with organic halides to form C—C bonds has been discussed in Section 2.10.4.7.2. This cannot, however, be extended to aryl, alkenyl or heteroaryl halides in which the halogen is attached to an *sp*² carbon. Such cross-coupling can be successfully achieved by nickel or palladium-catalyzed reaction of the unsaturated organohalide with a suitable thienyl metal derivative. The metal is usually zinc, magnesium, boron or tin; occasionally lithium, mercury, copper, and silicon derivatives of thiophene have also found application in such reactions. In addition to this type, the Pd-catalyzed reaction of halogenated thiophenes with suitable alkenes and alkynes, usually referred to as the Heck reaction, is also discussed in this section. Besides these, a few other miscellaneous Pd-catalyzed substitution reactions on thiophene will also be referred to.

2-Thienylmagnesium or zinc derivatives can be coupled with vinyl halides, bromo- or iodobenzene, or ethynyl bromide under Ni or Pd catalysis (Equation (59)). The reaction can be extended to the synthesis of β -styrylthiophene; in this case the double-bond configuration is retained in the product. Dichloro- and trichloroethylenes have also been used in this type of reaction, as well as β -bromo-vinylethers. Functionalized side chains can thus be introduced on the thiophene nucleus. Two examples prepared thus are (589) to (592). The Pd-catalyzed coupling of 2-thienylzinc bromide with (1-bromoethenyl)trimethylsilane has been investigated in detail; the rearranged product (594) is formed in addition to the expected (593) <90T2623>.

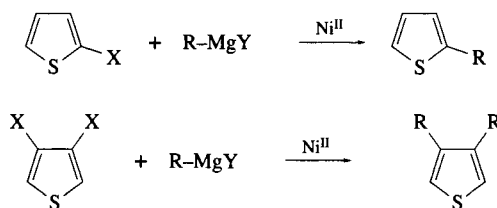


Novel quinones with interesting amphoteric redox properties have been synthesized by making use of the cross-coupling reaction between thienylzinc chlorides and iodobenzenes <91JA4576>.

Several tricyclic monomers required for oxidative (electrochemical or chemical) polymerization have been prepared by coupling reactions. Thus, 1,4-diiodo-2,5-dimethylbenzene was coupled with 2-thienylmagnesium bromide in the presence of nickel(II) bis(diphenylphosphino)propane dibromide (Ni(dppp)Br₂). However when methoxy groups were present in the benzene ring *ortho* to the site of coupling, the decreased electrophilicity necessitated the use of a more nucleophilic thiophene derivative, 2-thienylcopper (Equation (60)).

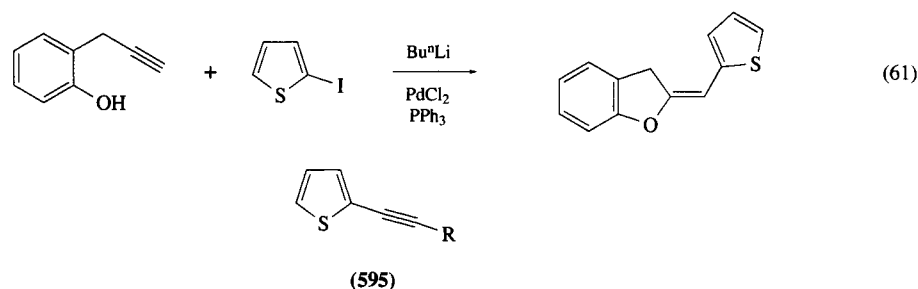


In a reversal of the functionalities in the reactants, thienyl halides can be cross-coupled with Grignard reagents under Ni^{II} catalysis (Scheme 130). This method has provided access to 3-(ω -haloalkyl)thiophenes which can be used as building blocks for functionalized polythiophenes <90AG(E)419>.

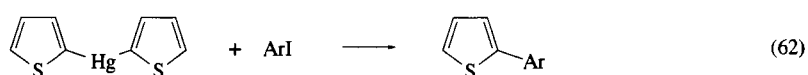


Scheme 130

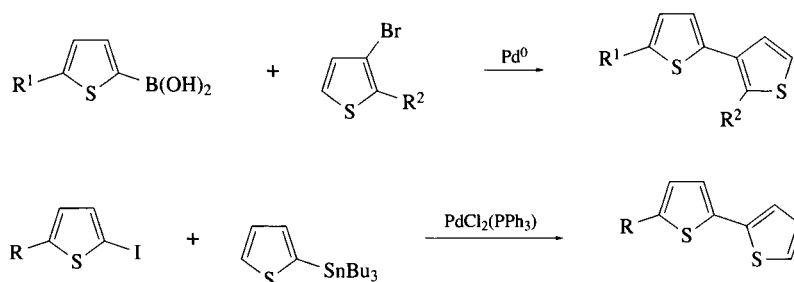
The palladium-catalyzed cyclization and cross-coupling of acetylenic alcohols with 2-iodothiophene to generate enol ethers should probably be included here (Equation (61)) <92JOC2213>. Similarly, (trimethylsilyl)ethynylmagnesium bromide can be coupled with 2-iodothiophene under Pd catalysis to produce (595; R = SiMe_3). Alkynylzinc chlorides also react with 2-bromo or 2-iodothiophene to produce (595). Phenylethynylcopper reacts with 2-iodothiophene to form (595; R = Ph).



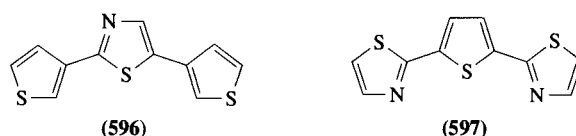
The mercuric derivative can be used instead of the magnesium derivative in the cross-coupling reaction (Equation (62)). 1-(*tert*-Butyldimethylsilyl)-3-indolylzinc chloride has been coupled with 2- and 3-bromothiophenes in the presence of a Pd-catalyst to yield the corresponding 3-thienylindoles <94TL793>.



Di- and polythienyls can be prepared by cross-coupling of 2- and 3-thienylboronic acids in the presence of Pd^0 catalyst. Another similar method is the cross-coupling of iodothiophenes with stannylthiophenes (Scheme 131). Stannylthiophenes have been used to prepare 3-bromo-2-(2-,3- or 4-pyridyl)thiophenes by coupling with the corresponding bromopyridines under Pd^0 catalysis <92ACS654>. Gronowitz and co-workers have synthesized several other ring systems by appropriate use of either boronic acid or tin derivatives in such coupling reactions <89S130, 90JHC1127>. A critical comparison of three different organometallic species for the preparation of terthienyls and other oligoheterocycles by the cross-coupling method has been made by Gronowitz and Peters <90H(30)645>. The Grignard cross-coupling reaction is limited to those substrates whose functional groups are compatible with the organomagnesium reagent. The thiopheneboronic acids have the limitation that in some instances extensive deboronation occurs even under weakly alkaline conditions; an excess of the boronic acid is therefore necessary. The oligoheterocycles (**596**) and (**597**) with a central thiazole or a thiophene ring are best prepared by coupling the respective trimethylstannyl derivative with the dibromoheterocycle. A molecule, in which two terthienyl units are attached to a naphthalene ring at positions 1 and 8, has been synthesized by the Grignard cross-coupling method <92TL7553, 93T3735>.

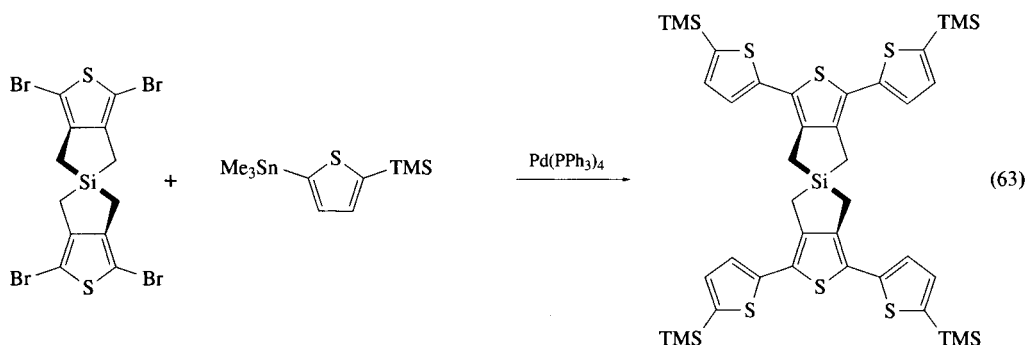


Scheme 131



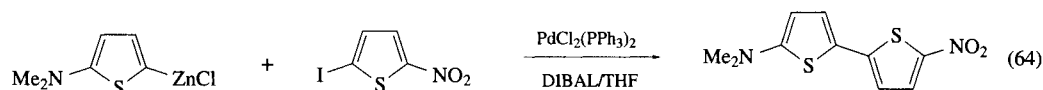
The direct one-pot synthesis of 2,5-di(4-pyridyl)thiophene has been achieved by the palladium catalyzed cross-coupling between 4-(trimethylstannyl)pyridine and 2,5-dibromothiophene <92BCJ1855, 92CC620>. These are useful precursors for viologens with potential application as molecular electronic devices.

Other interesting applications of the Pd^0 -catalyzed coupling of a bromothiophene with a stannylthiophene are in the formation of polymeric molecules in which one insulating polymeric chain is fixed at a 90° angle (achieved through a central spiro framework) to another conducting polymer, via a nonconjugating σ -bonded network. The basic reaction is shown in (Equation (63)) <90JA5662>.

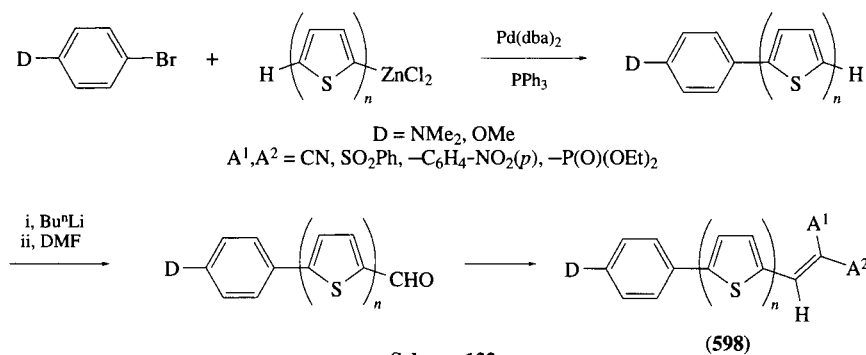


Oligothiophenes containing donor and acceptor substituents are of interest for the investigation of the electronic interaction between the two end groups, and their use as nonlinear optical materials.

5-Dimethylamino-5'-nitro-2,2'-bithiophene has been synthesized as shown in Equation (64) <93AG(E)719>.

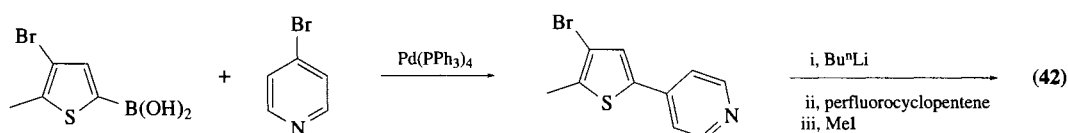


Earlier, several 2-phenylthiophene derivatives in which the benzene ring contained an acceptor group at the *para*-position had been synthesized by the Pd-catalyzed coupling between 2-thienylmagnesium bromide or 2-thienylzinc chloride and the corresponding iodobenzene <90JOM(390)389>. A similar synthetic scheme has been employed for the synthesis of compounds (**598**) with further extension in the conjugation (Scheme 132) <90TL4743>.



Scheme 132

Another area of extraordinary current interest is that of designing “switchable molecular wires displaying light-triggered reversible changes in electrical and optical properties” <93CC1439>. Cross-coupling with the help of 2-thienylboronic acid has been used effectively in the synthesis of some prototypes (Scheme 133). The light-induced reversible electrocyclization of (**42**) has been referred to in Section 2.10.2.2.3.



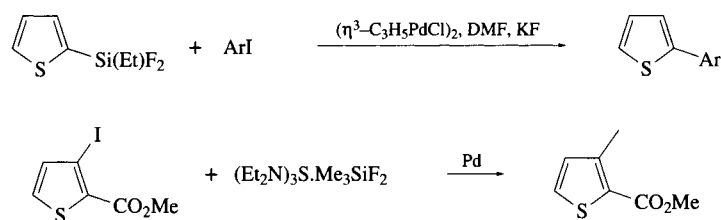
Scheme 133

The Pd⁰-catalyzed coupling reaction of β -5-iodo-2'-deoxy-3',5'-di-*O*-acetyluridine with 2-trimethylstannylthiophene and 2-phenyl-5-trimethylstannylthiophene has given the corresponding thiophene substituted nucleosides <91JHC529>. The unprotected 5-iodo-2'-deoxyuridine has also been coupled with 2-(trimethylstannyl)- or 2-(tributylstannyl)thiophene <91JMC2383>; a Pd^{II} catalyst was used in a coordinating solvent (THF or dioxane), whereas in a noncoordinating solvent (toluene), a Pd⁰ catalyst was found to be essential.

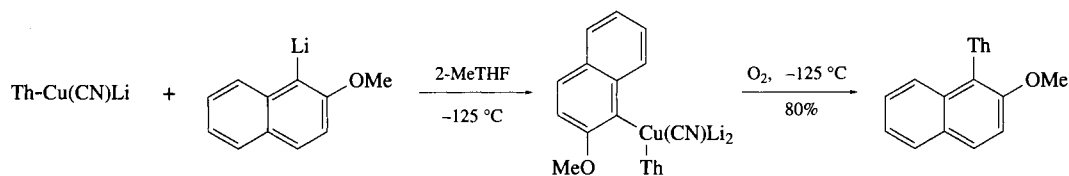
An interesting addition to the variety of organometallic species which can take part in the coupling reaction is the recent report on the fluoride ion and Pd catalyst-promoted cross-coupling of ethyl (2-thienyl)difluorosilane with aryl iodides (Scheme 134) <90H(30)303>. The fluorine atom on silicon appears to accelerate the rate of the cross-coupling reaction. The method can also be used for introducing a methyl group on the thiophene. The strategy is to use tris(diethylamino)sulfonium difluorotrimethylsilicate to couple with iodothiophene in the presence of (η^3 -C₃H₅PdCl)₂ in DMF.

One of the most recent developments in the area of biaryl coupling is the use of higher order mixed diarylcyanocuprates; these kinetically prepared higher-order cuprates function well at very low temperatures to yield unsymmetrical coupling products (Scheme 135) <94TL815>. Two dissimilarly substituted thiophenes have also been coupled by this procedure.

The Heck reaction has been used to synthesize several alkenylthiophenes. Under Pd catalysis, thienyl halides react with suitable alkenes to form the products (**599**). In the case of allylic alcohols, the carbonyl compound (**600**) is the main product (Scheme 136). The method has been extended to the synthesis of styrylthiophenes <88TL4409>. The product appears to have the *E*-configuration about the double bond, whereas the expected simple *cis*-addition of the thienylpalladium species followed

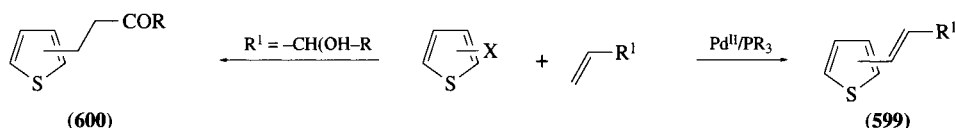


Scheme 134



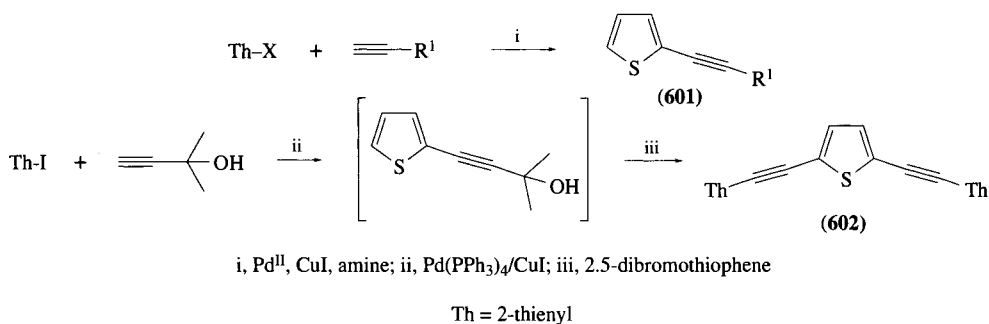
Scheme 135

by *syn*-elimination of [Pd]-H should have given the product with a *Z*-configuration. The result therefore has been explained as due to the occurrence of an additional inversion step.

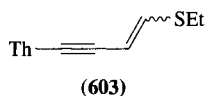


Scheme 136

The Sonogashira alkynylation has been successfully extended to the thiophene series to synthesize the alkynylthiophenes (**601**). This method has been used to prepare 2,5-di(2-thienylethynyl)-thiophene (**602**) (Scheme 137). Conjugated enynes (**603**) have also been prepared by this method <92TL4229>.

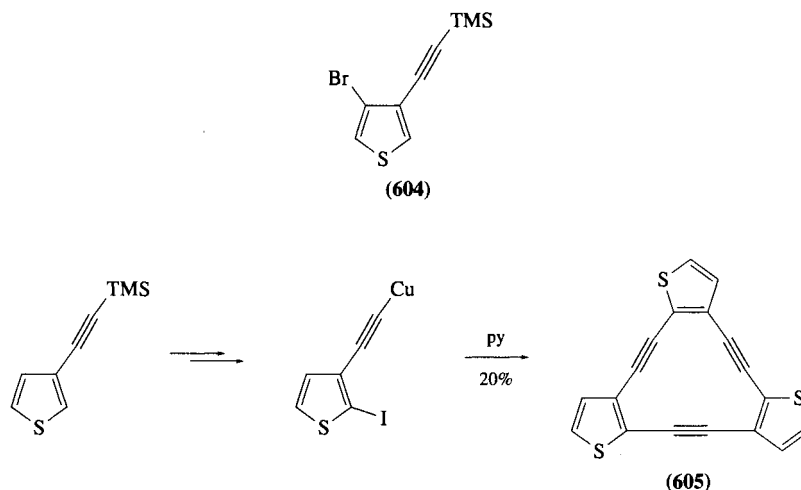


Scheme 137



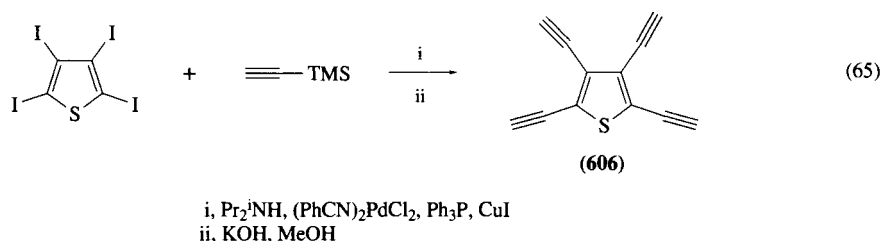
Reaction of 3,4-dibromothiophene with trimethylsilylacetylene in refluxing diethylamine in the presence of catalytic amounts of $\text{PdCl}_2 \cdot (\text{PPh}_3)_2$ and copper(I) iodide afforded mainly the mono-substitution product (**604**) <90SC2275>. The bis(trimethylsilylethynyl)-substituted thiophene could, however, be obtained in 85% yield by refluxing the 3,4-dibromothiophene with the acetylene (2.4 equiv.), $\text{PdCl}_2(\text{PPh}_3)_2$ (0.04 equiv.) and CuI (0.04 equiv.) in piperidine <92JA1479>. This could subsequently be converted to 1-phenylthieno[3,4-*d*]borepine. Similarly, starting from 2,3-dibromo-

thiophene, 2,3-bis(trimethylsilylethynyl)thiophene could be prepared in 28% yield <93CL933>. The acetylene coupling route has been extended to the synthesis of a trithienocyclotriyne (**605**) (Scheme 138) <90SL427, 94OM451>. Similarly 8,11-thio-leukotriene B₃ has also been synthesized by making use of the acetylene coupling reaction <90SL755, 90JCS(P1)194>.



Scheme 138

One of the most interesting applications of such acetylene coupling is the preparation of the per(acetylenated)thiophene (**606**) (Equation (65)) <88JOC2489>. Coupling of 3-iodobenzo[*b*]thiophene with several terminal acetylenes in the presence of Pd(PPh₃)₂Cl₂, CuI and Et₃N has been shown to give the 3-ethynyl products in good yield <88CPB2248>.



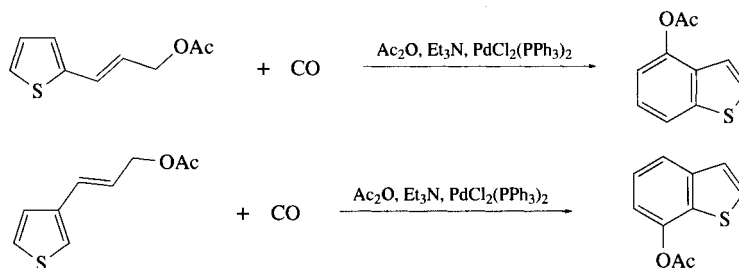
A reaction formally resembling the Heck alkenylation and Sonogashira alkynylation is the coupling of aryl halides with thiophene and benzo[*b*]thiophene in the presence of tetrakis(triphenylphosphine)palladium <90H(31)1951, 92H(33)257>. The yields are moderate to good. A different type of coupling is exemplified by the preparation of 4-(2-thienyl)pyridine <90BCJ636>. In this method, 4-iodopyridine was reacted with 2-iodothiophene in the presence of hydrazine hydrate and palladium amalgam to give the product in 30% yield. As expected, 4,4'-bipyridyl is a by-product in this reaction.

Palladium-mediated oxidative arylation of thiophene has also been reported <85JOC5272>. Thus, treatment of 2-formylthiophene with palladium acetate in a mixture of acetic acid and benzene gave 2-formyl-4-phenylthiophene (30%), 2-formyl-5-phenylthiophene (5%) and 5,5'-diformyl-2,2'-bithienyl (16%). It has been suggested that preliminary palladation of benzene would lead to 4-phenylation of the thiophene, while palladation of the thiophene would produce the 5-phenylated product.

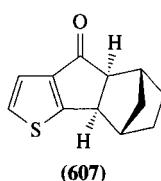
(i) Other Pd-catalyzed reactions

2-Bromothiophene and 3-bromothiophene have been reacted with alkali cyanide in the presence of *trans*-chloro(2-naphthyl)-bis(triphenylphosphine)nickel to generate the corresponding cyano compounds <90SC3153>. Cyclocarbonylation of 3-(thienyl)allyl acetates has led to acetoxycyanothiophenes <91JOC1922>. The reaction is carried out in the presence of Ac₂O, Et₃N and a catalytic amount of PdCl₂(PPh₃)₂ at 130–170°C under 50–70 atm. pressure of CO (Scheme 139). 3-

Bromothiophene has been converted to the corresponding acid fluoride by Pd-catalyzed carbonylation in the presence of KF in DMF <92BCJ1741>. 2-Iodothiophene reacts with CO and norbornene in the presence of Pd⁰ and TIOAc to form the cycloadduct (**607**) in very good yield <94TL3197>.



Scheme 139



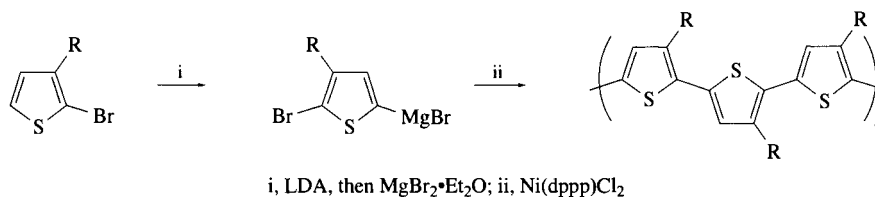
A phosphorus substituent has been introduced into the thiophene nucleus by a similar Pd⁰-catalyzed reaction between 2-thienyl bromide and monobutyl alkyl(aryl) phosphonite.

Palladium-catalyzed germylation of 2,5-dibromothiophene using (ClMe₂Ge)₂ has been reported. The monomer on Wurtz type reaction with sodium leads to the polymer <92CL647>.

(ii) *Preparation of thiophene oligomers and polymers by using the cross-coupling reactions*

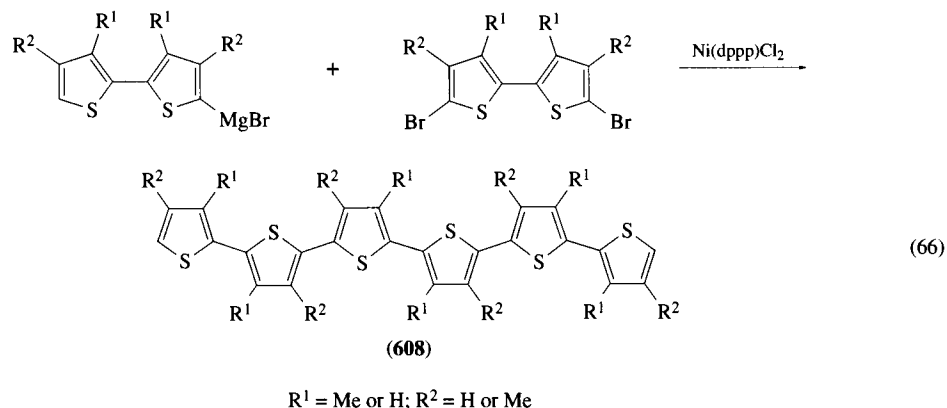
A tremendous amount of work has recently been directed towards the synthesis of thiophene oligomers and polymers. This is due to the current interest in research on conducting polymers and molecular electronics. A review has appeared recently on polythiophenes <92CRV711>. Two main approaches have been used for making such polymers: (i) electrochemical oxidation of monomeric thiophenes (see Section 2.10.2.8.4) and (ii) transition metal-catalyzed cross-coupling reactions. The cross-coupling reaction between heterocyclic halides and Grignard reagents, catalyzed by nickel-phosphine complexes was extensively investigated by Kumada and co-workers in 1982 <82T3347>. The reaction soon found application in the synthesis of oligomeric thiophenes <87CC1021>. Pelter *et al.* extended the concept by using the cross-coupling reaction between regiospecifically bismetallated dithienylbenzenes and dibromobenzenes <87TL5213, 89TL3461>. The starting material was prepared by coupling 2-thienylzinc chloride with 1,4-dibromobenzene in the presence of a palladium catalyst. Several substituents could be introduced in the benzene ring at will. Several 2,5-dibromo-3-alkoxythiophenes have been polymerized in the presence of zero-valent nickel complexes <93CL415>.

Earlier synthetic methods employed to generate mono-functionalized polythiophenes had led to couplings at the 2,5-positions with random regiospecificity. Thus, a mixture of polymeric structures had resulted from such procedures. A regioselective synthesis has now been developed, giving almost exclusively head-to-tail couplings (Scheme 140) <92CC70, 93JA4910>. The corresponding zinc compound can also be used <92JA10087>.

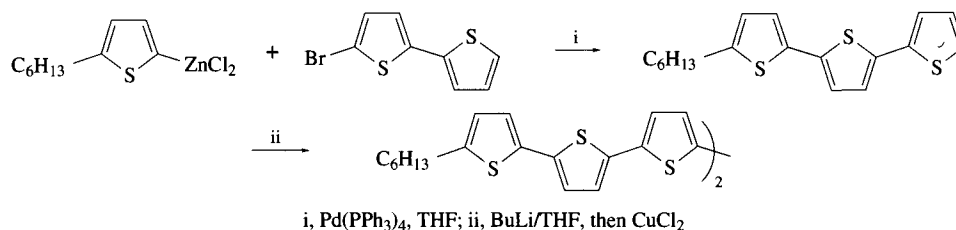


Scheme 140

The hexamethylsexithiophenes (**608**) with predetermined head-to-head or tail-to-tail linkage of the monomers have been synthesized by nickel–phosphine-catalyzed Grignard coupling (Equation (66)) <92T6701>.



Thin-film devices based on oligothiophenes have been reported to act as semiconducting layers. The electronic properties of such organic semiconductors can be fine-tuned by introducing suitable substituents or by varying the conjugation length. Recently, it has been demonstrated that increasing the van der Waals attraction between the constituting molecules increases the self-recognition properties, thereby leading to highly ordered films. This has been brought about in a conjugated sexithiophene oligomer by incorporating *n*-hexyl residues at the two free α -positions of the terminal thiophene nuclei <93JA8716>. The synthesis is shown in Scheme 141 and is based on the Pd-catalyzed coupling of two thiophene residues followed by an oxidative dimerization brought about by Cu(II). Poly(1,4-phenylene-2,5-thiophenes) with different alkoxy chains attached to the phenyl ring have been prepared, using the Stille reaction, from 2,5-distannylthiophene and the requisite dialkoxy diiodobenzene <93AG(E)1345>.



Scheme 141

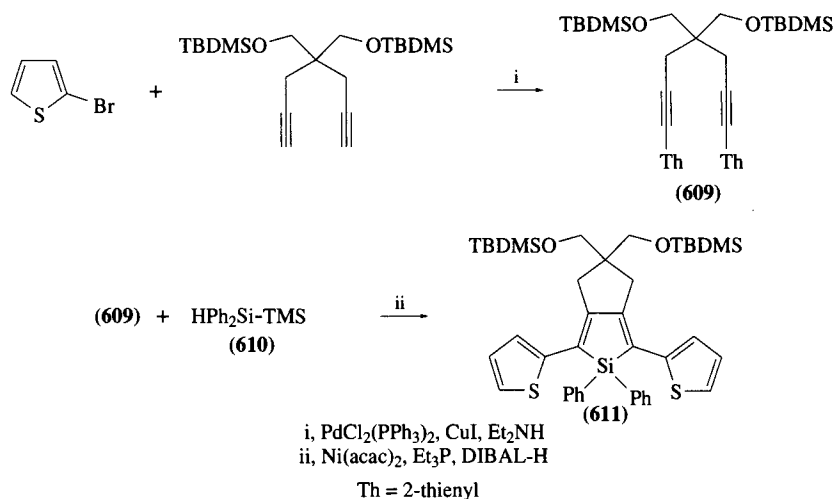
Copolymers possessing electron-rich thiophene rings alternating with electron-deficient units may lead to interesting properties resulting from intramolecular charge transfer. The random copolymerization of thiophene and pyridine has been achieved <92CL1003>. The catalyst used was a mixture of bis(1,5-cyclooctadiene)nickel and 2,2'-bipyridine. A more rational approach is the Ni(0) complex-catalyzed polymerization of thienylpyridine monomers <91CC1210>.

The electron-deficient silole ring has also been incorporated in the polythiophene chain (Scheme 142) <92JA5867>. Sonogashira alkynylation of 2-bromothiophene followed by nickel-catalyzed intramolecular cyclization of the resulting (**609**) with hydrodisilane (**610**) gave the monomer (**611**). Mono- and di-bromination of (**611**) at the thiophene α -position, followed by coupling with suitable 2-thienyl organometallics, has given a wide variety of oligomers.

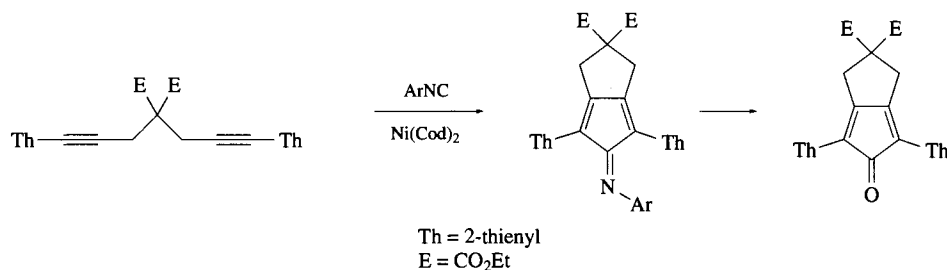
Thiophene–cyclopentadienone cooligomers have been similarly prepared from an acetylenic precursor (Scheme 143) <94CC229>. The crucial step is the intramolecular cyclization of a diyne with 2,6-xylylisocyanide.

2.10.4.7.4 Formation of C—O bonds

In an excellent review covering all aspects of the chemistry of hydroxythiophenes, the synthesis of these compounds from thienyllithium and thienylmagnesium derivatives has been discussed



Scheme 142



Scheme 143

<86HC(44/3)1>. The two most important methods are: (i) conversion of thienyllithium to a thienylboronic acid by reaction with alkyl borate, followed by oxidation with H_2O_2 , and (ii) reaction of thienylmagnesium derivatives with *t*-butylperbenzoate, followed by dealkylation of the *t*-butyl ethers with acid. The boronic acid method has been used for the synthesis of several pyridine-substituted hydroxythiophenes <92ACS654>.

Oxysilylation of thiophene has been accomplished by reacting 3-thienyllithium with bis(trimethylsilyl)peroxide. The product, 3-trimethylsilyloxythiophene, was obtained in 60% yield <86TL5155>.

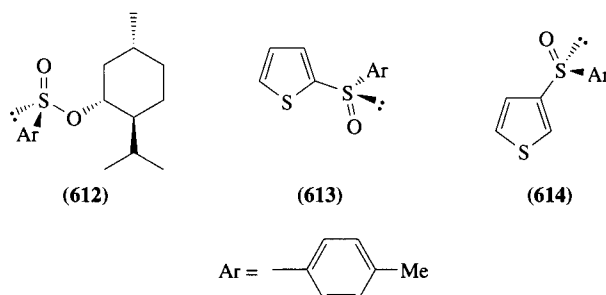
2.10.4.7.5 Formation of C—S bonds

Thienyllithium or thienylmagnesium derivatives can be reacted with sulfur to produce thiols <86HC(44/3)135>. The reaction with disulfides to generate *S*-alkyl thiophenethiols is quite useful. Tetraisopropylthiuramdisulfide with 2-thienyllithium gives *S*-(2-thienyl)-*N,N*-diisopropylthiocarbamate in 96% yield. The reaction of 3-thienyllithium with several symmetrical diaryldisulfides and dialkyldisulfides has provided the respective 3-thienylsulfides <90JHC127, 94TL3673>.

Thienyl sulfoxides can be prepared by reacting thienyllithium or thienylmagnesium bromide with sulfinates. This method has been used to generate enantiomerically pure thienyl *p*-tolyl sulfoxides <92TA857>.

Addition of 2- or 3-thienyllithium to optically pure (–)-menthyl-(*S*)-*p*-toluenesulfinate (612) in THF at -78°C and quenching with aqueous NH_4Cl gave the products (613) and (614), with, however, low *ee*. The optical purity of the products was improved dramatically by using the magnesium derivative. Thus, addition of freshly prepared MgBr_2 to the thienyllithium before addition of (612) gave (613) and (614) with 100% *ee*. It has been suggested that with the magnesium

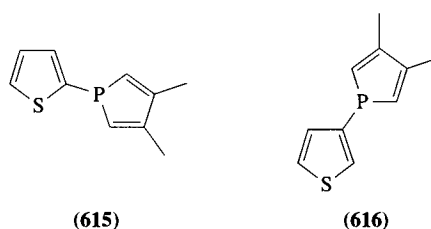
reagent, clean S_N2 reaction occurs, whereas with thienyllithium, a rather ionic S_N1 mechanism might operate.



Several benzo[*b*]thiophene-2-sulfonamides have been synthesized in a regiospecific way by first reacting the 2-lithio derivative with sulfonyl chloride or SO_2 followed by further transformations <89JMC2548>.

2.10.4.7.6 Formation of C—N and C—P bonds

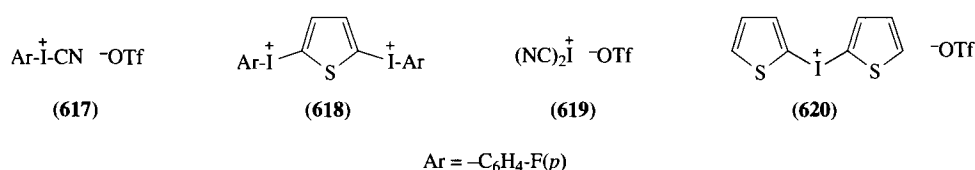
There seem to be very few methods available for forming C—N bonds using metallothiophenes. C—P bonds have been created by reacting 2- and 3-thienyllithium with 1-cyano-3,4-dimethylphosphole. The corresponding 1-(thienyl)phospholes (615) and (616) are obtained. 2,5-Dilithio- and 3,4-dilithiothiophene lead to the respective *bis*(1-phospholyl)thiophenes <92BSF1>.



2.10.4.7.7 Formation of C—halogen bonds

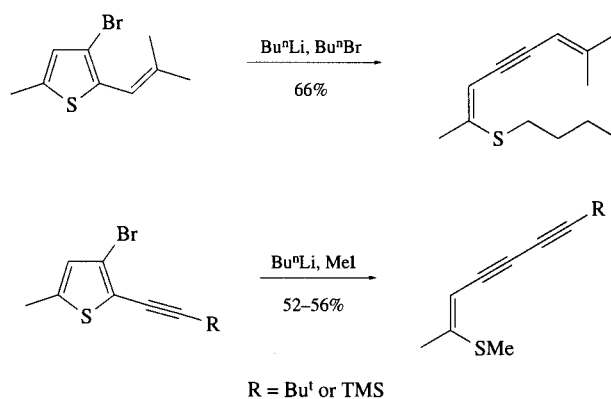
Hexachloroethane has proved to be a convenient source of chlorine for the conversion of thienyllithium to chlorothiophene. Quite unexpectedly, trichloroacetonitrile also acts as a chlorinating agent <86HC(44/2)159>. Similarly for bromination, CBr_4 is a good source of bromine. Iodination is usually carried out with molecular iodine on thienyllithium. Another useful method is cleavage of thallium derivatives with KI. The stable 3-thienyllithium in hexane:THF (10:1) has been reacted with 1,2-diiodoethane to produce 3-iodothiophene in 63% yield <94TL3673>.

Aryl(cyano)iodonium triflates react with tributyltin derivatives of aromatic and heteroaromatic compounds forming the corresponding (aryl)iodonium salts in high yield under mild conditions ($-40^\circ C$ to room temperature). The reagents are prepared from aryl iodides by oxidation with peroxytrifluoroacetic acid and subsequent reaction with trimethylsilyl triflate and trimethylsilyl cyanide. Thus the 4-fluorophenyl(cyano)iodonium triflate (617) reacts with 2,5-bis(tributylstannyl)thiophene to form (618) in 25% yield <93TL6853>. Similarly, (dicyanoiodonium)triflate (619), prepared by reaction of iodosyl triflate with trimethylsilyl cyanide, reacts with 2-(tri-*n*-butylstannyl)thiophene at $-40^\circ C$ in CH_2Cl_2 forming the iodonium salt (620) in high yield <92TL1419>. The preparation of this using chlorovinyl iodosodichloride was referred to in CHEC-I <84CHEC-I(4)741>.



2.10.4.7.8 Ring-opening reactions

The ring-opening reactions of 3-thienyllithium mentioned in CHEC-I <84CHEC-I(4)741> have been exploited for the construction of highly unsaturated thioenynic structures in a regio- and stereospecific manner (Scheme 144) <84JOC2018>.

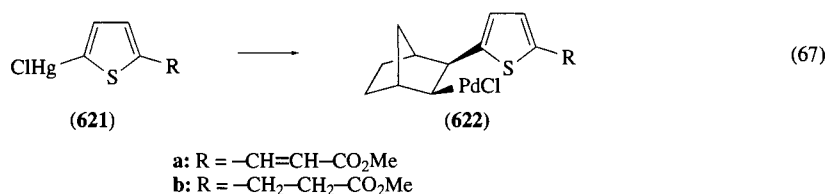


Scheme 144

2.10.4.7.9 Reactions of mercury and thallium derivatives

Regioselective carboxylation of thiophene by Pd-mediated reaction with CO in the presence of mercuric chloride, mercuric acetate or thallium(III) acetate has been reported <90JMOC23>. It is preferable to carry out the reaction in two steps—initial mercuriation, followed by reacting the thienylmercury derivative with CO in the presence of palladium acetate.

Thienylmercury derivatives (**621**) react with norbornene and LiPdCl₄ in acetonitrile to form the adducts (**622**) (Equation (67)) <86JOC5221>. The mercury derivative (**621b**) also reacts with (norbornadiene)palladium dichloride to give a nortricyclyl adduct.

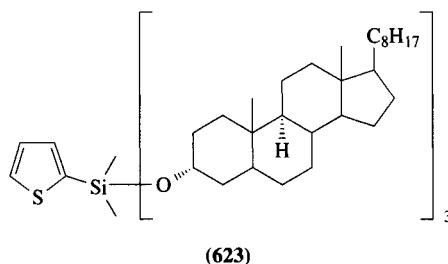


2.10.5 REACTIVITY OF SUBSTITUENTS ATTACHED TO THE THIOPHENE SULFUR ATOM

The reactions of thiophene S,N-ylides and S,C-ylides have already been discussed in Section 2.10.3.3.

The sulfur atom of thiophene can function as a template for radical-relay chlorinations. An excellent application of this concept is in the regioselective chlorination of a steroid molecule <82JA2045>. Reaction of (2-thienyl)trichlorosilane with 3 α -cholestanol gave the tris(cholestanyl)-silylether (**623**). When this was irradiated in CH₂Cl₂ solution with 2 equiv. of sulfuryl chloride and a catalytic amount of AIBN, the 9(11)-olefin was produced in 45% yield after alkaline hydrolysis and elimination of HCl. Apparently, a chlorine atom becomes attached to the sulfur of the thiophene;

this is then regiospecifically relayed to the hydrogen attached to C-9 of the steroid. This generates a C-9 radical which is chlorinated by SO_2Cl_2 . Obviously, the yield of 45% indicates that more than one steroid nucleus is attacked by "template control."



2.10.6 REACTIVITY OF METAL π -COMPLEXES OF THIOPHENE

The current interest in the interaction of metals and thiophenes stems from the efforts to elucidate the mechanism by which metals facilitate desulfurization of thiophenes and benzo[*b*]thiophenes in fossil fuels. The industrial process of hydrodesulfurization (HDS) involves extrusion of sulfur from organosulfur compounds in petroleum-based feedstocks by catalytic hydrogenolysis at 400°C. The usual catalyst is a cobalt- or nickel-promoted molybdenum sulfide or tungsten sulfide, supported over alumina. Of all the sulfur compounds present, thiophene, benzo[*b*]thiophene, and especially dibenzothiophene are the most difficult ones to desulfurize. The efforts to understand the mechanism of this HDS process are based on preparing and characterizing transition metal complexes of thiophenes and investigating their reactivity, especially their ring-opening reactions. The earlier theories were reviewed in 1988 <88ACR387>.

Thiophenes can bind to metals via four bonding modes: (i) η^5 -thiophene complexes; (ii) η^4 -thiophene complexes; (iii) η^2 -thiophene complexes; and (iv) η^1 -S-bonded complexes. In addition to these, the metal might insert into the C—S bond of thiophene to form a six-membered ring containing the metal and sulfur. All these are closely interconnected and have been fully characterized..

ACKNOWLEDGEMENT

We are deeply indebted to Surekha Deshpande and Pabba Chittari for their help in preparing this manuscript. S. Rajappa thanks CSIR for a grant under the Emeritus Scientist Scheme.

2.11

Thiophenes and their Benzo Derivatives: Synthesis

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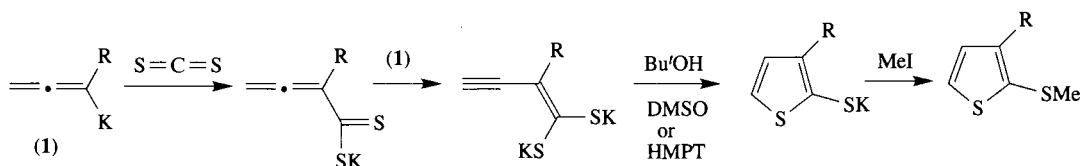
2.11.1 INTRODUCTION

This chapter deals with the advances in the synthetic methods of thiophenes and their benzo derivatives. The references cover the literature of the period 1983–1993 and also 30 papers in 1994. Stress has been laid on the practical or newly developed syntheses and syntheses of structurally interesting compounds, although efforts have been made to cover the literature published in the above period as extensively as possible. A large number of new synthetic methods have been developed since the early 1980s. This is partially due to the increasing need for thiophene derivatives as the starting materials for constructing a variety of molecular devices in addition to the need in the field of pharmaceuticals and agrochemicals. For synthetic studies on thiophenes in the period before 1983, readers are recommended to consult the excellent chapter by Campaigne in the first edition of *Comprehensive Heterocyclic Chemistry* (84CHEC-I(4)863).

Sulfur exists in many forms in organic compounds: thiols (thiolates), sulfides, disulfides, sulfonium salts, sulfoxides, sulfones, and sulfonic acids and their esters are representative compounds containing sulfur. There also exist sulfur-containing transient intermediates such as thiyl radicals and sulfenium ions in addition to rather labile species such as sulfonium ylides and sulfuranes. All of these compounds serve as starting materials for the construction of the thiophene ring. In addition, there are many inorganic compounds that serve as the sulfur source for constructing a thiophene nucleus. Elemental sulfur, hydrogen sulfide, metal sulfides, phosphorus pentasulfide, Lawesson's reagent, sulfur mono- and dichlorides, and thionyl chloride are the representative sulfur sources of thiophene synthesis. This makes possible a plethora of combinations that can lead to the formation of the thiophene ring. Therefore, the synthetic methods of thiophenes are much more complex and abundant in numbers than those of other five-membered heteroarenes such as furans and pyrroles.

As in the case of Campaigne's review, the synthetic methods are described in order of bond formation, beginning with the formation of one bond, between sulfur and C-2 of thiophene, and proceeding around the ring, then two bonds, etc. However, the classification of the synthetic methods is often difficult because of the intricate plot of the synthesis. In this chapter, the synthetic methods are classed, in many cases, according to the final reaction leading to the thiophene nucleus but not the starting materials since this permits a more clear-cut classification. Thus, the thiophene synthesis (82JOM(238)C17) given in Scheme 1 is treated as the synthetic method of one-bond formation between sulfur and C-2 of thiophene, even though it may also be classed as the method of two-bond formation between C—C—C and C—S units. Thiophene syntheses using other heterocyclic compounds as the starting material are treated in independent sections. Also treated in independent sections are the benzo[b]- and benzo[c]-thiophene syntheses by annelation of thiophenes.

In order to aid the quick understanding of the readers, most synthetic methods are shown by chemical equations, and the yields and reaction conditions are also given in as much detail as possible.

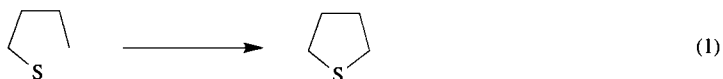


Scheme 1

2.11.2 RING SYNTHESIS BY FORMATION OF ONE BOND

2.11.2.1 Formation of a Bond Adjacent to the Sulfur Atom

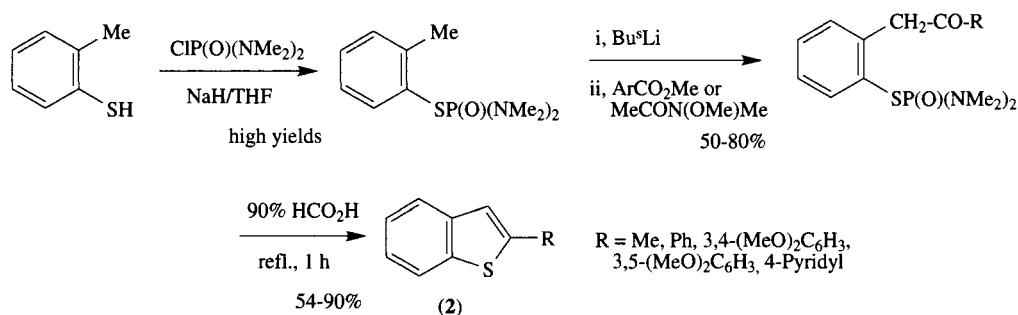
According to the classification mentioned above, the thiophene ring synthesis by forming one carbon—sulfur bond adjacent to sulfur is described first (Equation (1)).



This approach to the thiophene ring seems most direct and involves (i) intramolecular nucleophilic addition of thiol, thiolate, and dithiocarboxylate sulfurs and, in a rare case, sulfide sulfur to sp^2 and sp carbons; (ii) electrophilic attack of sulfenium and sulfonium ions and their equivalents on unsaturated carbon—carbon bonds; (iii) addition of thiyl radicals to unsaturated carbon—carbon bonds; (iv) addition of vinyl and aryl radicals to the sulfur atom of sulfides; and (v) electrophilic attack of carbocation on the sulfur atom of sulfides.

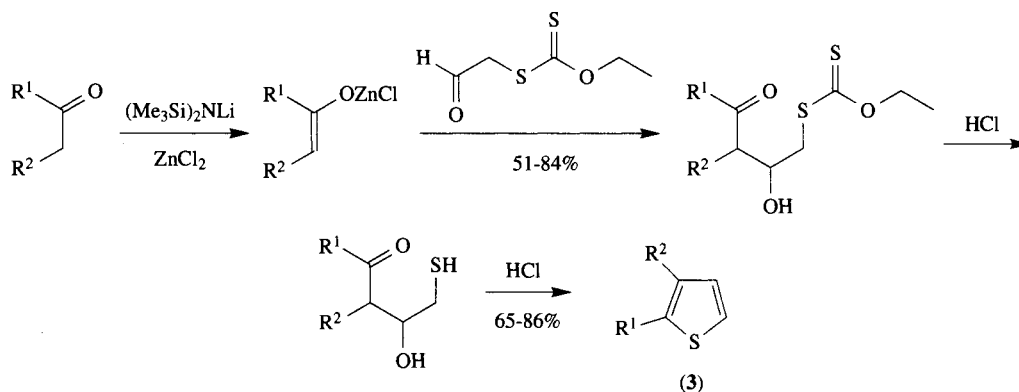
2.11.2.1.1 Processes involving nucleophilic addition of thiol, thiolate, and dithiocarboxylate to sp^2 and sp carbons

2-Methyl- and 2-aryl-substituted benzo[*b*]thiophenes (2) can be synthesized starting from *o*-toluenethiol in three steps, which involves the intramolecular addition of thiols, produced by acid hydrolysis, to the carbonyl group followed by dehydration as the final step (Scheme 2) <91JHC173>.

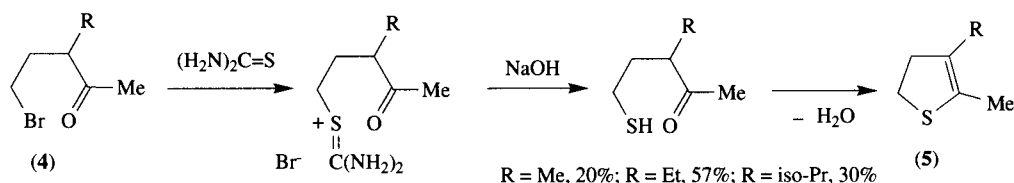


Scheme 2

2-Substituted, 2,3-disubstituted, and 2,3-annelated thiophenes (3) can be prepared by processes involving reaction of ketone enolates with carbonodithioic acid *O*-ethyl *S*-(2-oxoethyl)ester. Hydrolysis of the resulting aldols, intramolecular addition of thiol group to the carbonyl group, and elimination of two molecules of water lead to the thiophenes (3) (Scheme 3) <92HCA907>. In a similar way, 2,3-dialkyl dihydrothiophenes (5) can be prepared from bromoketones (4) by intramolecular addition of the thiolate to the carbonyl group (Scheme 4) <91JOC4027>.

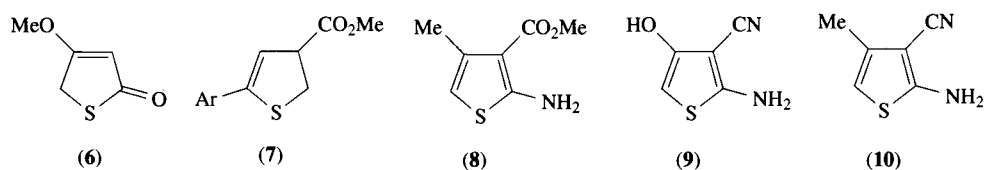


Scheme 3

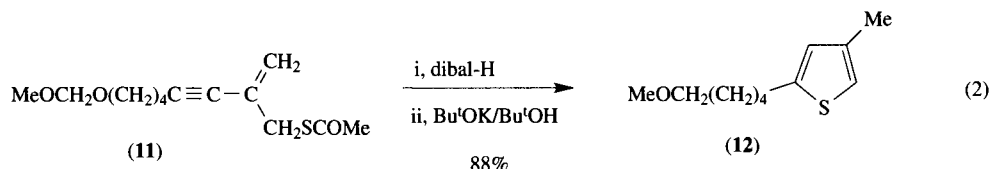


Scheme 4

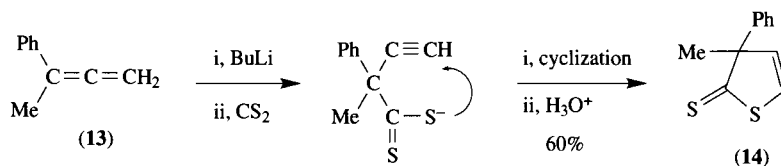
Many patents describe the preparation of thiophenes such as (6)–(10) by processes involving intramolecular addition of thiol (thiolate) groups to nitrile or carbonyl groups <84JAP(K)59204186, 85JAP(K)6001180, 85JAP(K)60208976, 88EUP261668, 88GEP(O)3630070, 90JAP(K)02193990>.



Intramolecular addition of thiolate, dithiocarboxylate, and enedithiolate sulfurs to unsaturated carbon-carbon bonds also provides a convenient way to thiophenes. Thus, reductive cleavage of the thioester group of the compound (11) followed by intramolecular addition of the thiolate sulfur to the triple bond affords the thiophene (12) directly (Equation (2)) <93SL209>. In this way, a number of alkyl-substituted and six-membered ring annelated thiophenes were synthesized in good overall yields.

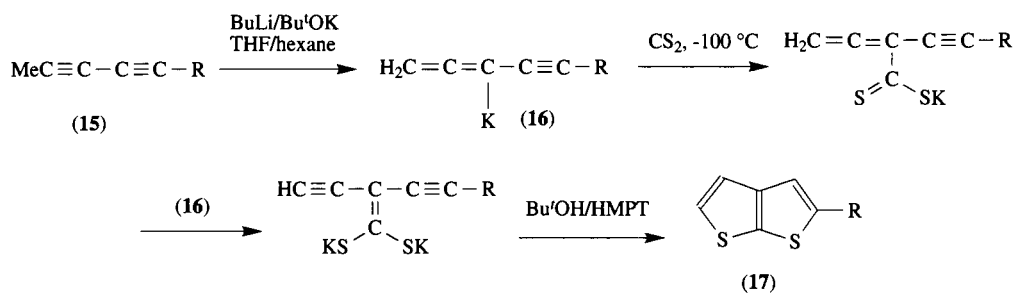


Addition of carbon disulfide to the carbanion, generated from the allene (13), and intramolecular addition of the dithiocarboxylate sulfur to the triple bond gives the compound (14) (Scheme 5) <85TL5131>. Metallation of the diynes (15), followed by the successive addition of carbon disulfide, Bu^tOH, and HMPT, affords thienothiophenes (17) in reasonable yields (Scheme 6), and the use of the sulfides (18) as the starting material produces the thienodithiins (19) (Equation (3)); when R is TMS in the starting compounds, the unsubstituted parent compounds are obtainable in these cases since desilylation probably takes place at some intermediate stage <83CC1056>.

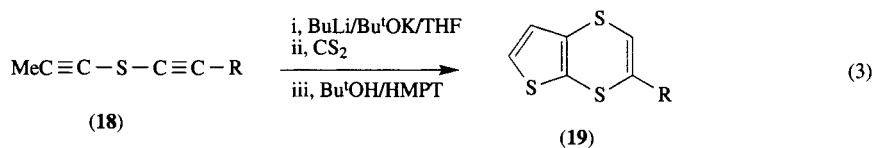


Scheme 5

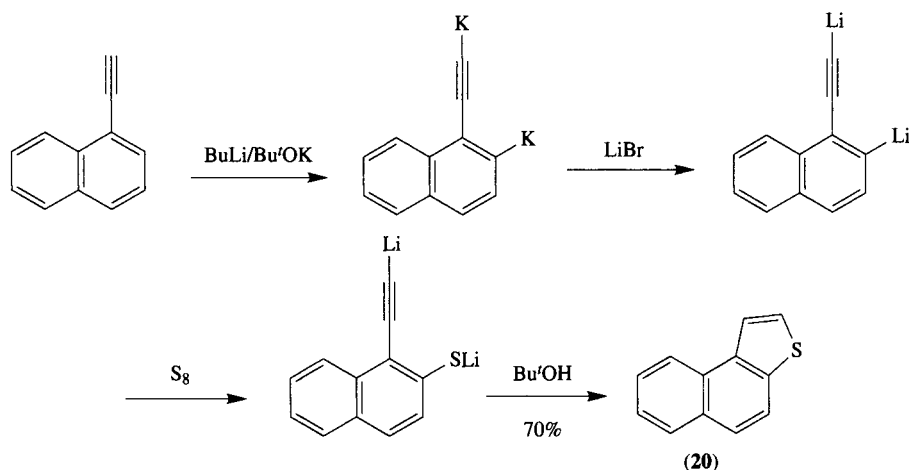
Dimetallation of alkynes also provides a practical synthesis of thiophenes. Dimetallation of (1-naphthyl)ethyne, reaction with elemental sulfur, and quenching of the reaction with *t*-butanol affords the thiophene (20) in one pot (Scheme 7) <89SC2691>. In another example, the alkynes (21; R¹ = MeS, MeO, Bu^tO, Me₂N, Et₂N) were dimetallated, treated with non-enolizable thiocarbonyl compounds (R² = Bu^t, Ph, 2-thienyl, MeO, Bu^tO, Et₂N), and quenched with *t*-butanol to give (22), which eliminate methanethiol spontaneously to afford a series of 3,4-disubstituted thiophenes (23) in good yields (Scheme 8) <90SC3427>. Meanwhile, metallation of a 4 : 1 mixture of the ynamine and



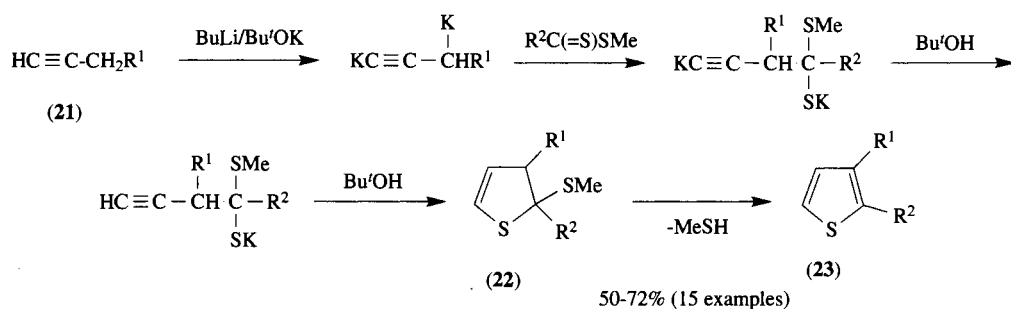
Scheme 6



the allenic amine (**24**; $\text{R}^1 = \text{Me, Et}$), followed by reaction with thiocarbonyl compounds ($\text{R}^2 = \text{Me}_2\text{N}$, Et_2N , MeS , Bu^t , Bu^tO , Ph , 2-thienyl) and then addition of water gives 2,5-disubstituted thiophenes (**25**) in good yields (Scheme 9) <86JOM(316)C21>.



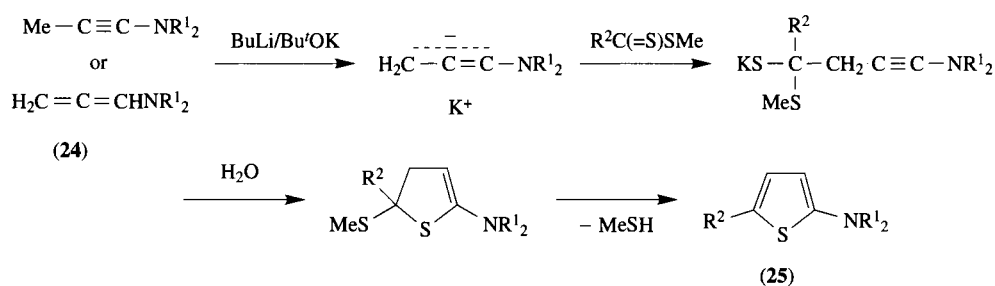
Scheme 7



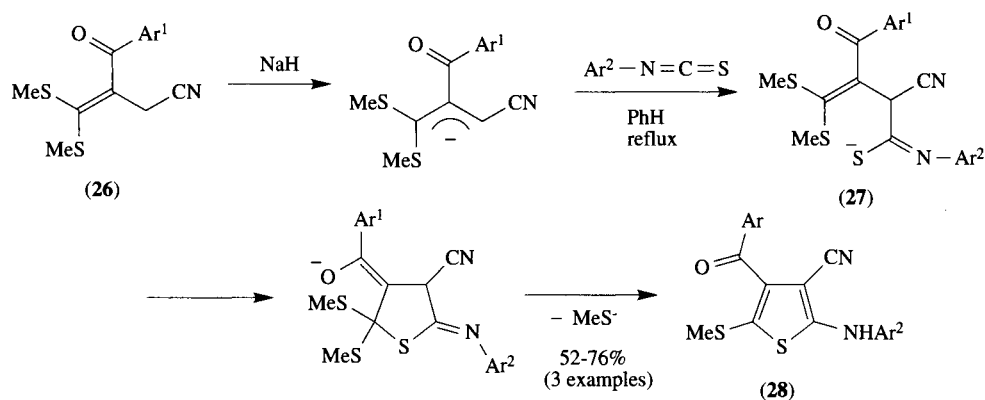
Scheme 8

Addition of aryl isothiocyanates to the carbanions, generated from ketene dithioacetals (**26**), produces the thiolate anions (**27**), which undergo an intramolecular Michael addition to give the thiophenes (**28**) (Scheme 10) <88JCS(P1)2365>.

The following example involves the interaction between dithiocarboxylate sulfur and cyclopropane ring. Thus, treatment of the ester enolate, generated from (**29**), with carbon disulfide and

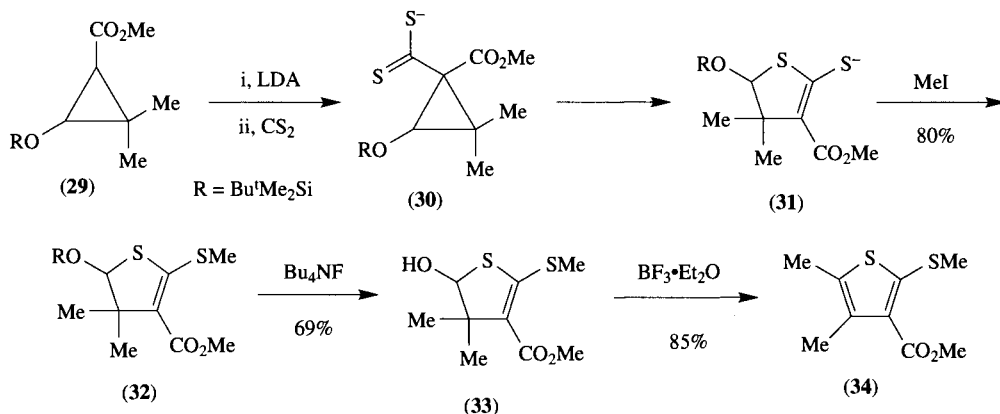


Scheme 9



Scheme 10

then with methyl iodide affords the dihydrothiophene (32). Rearrangement of the intermediate (30) to (31) takes place even at -78°C . Desilylation of (32) and the acid-catalyzed methyl migration of the resulting alcohol (33) gives the thiophene (34) (Scheme 11) <88LA465>.

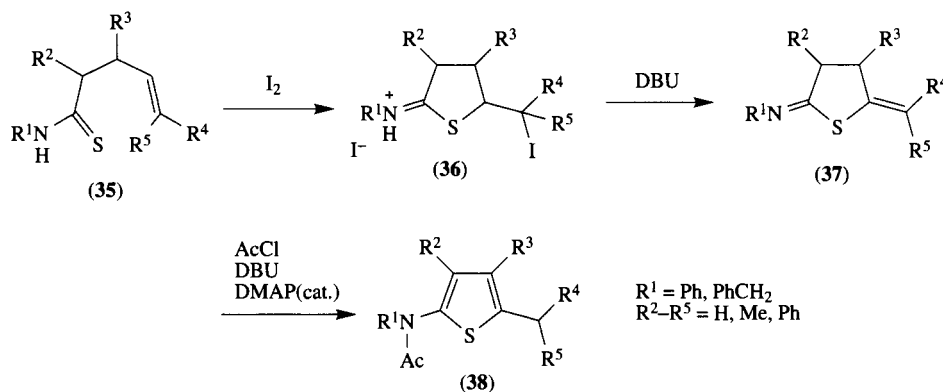


Scheme 11

2.11.2.1.2 Processes involving electrophilic attack of sulphenium and sulfonium ions and equivalents on unsaturated carbon-carbon bonds

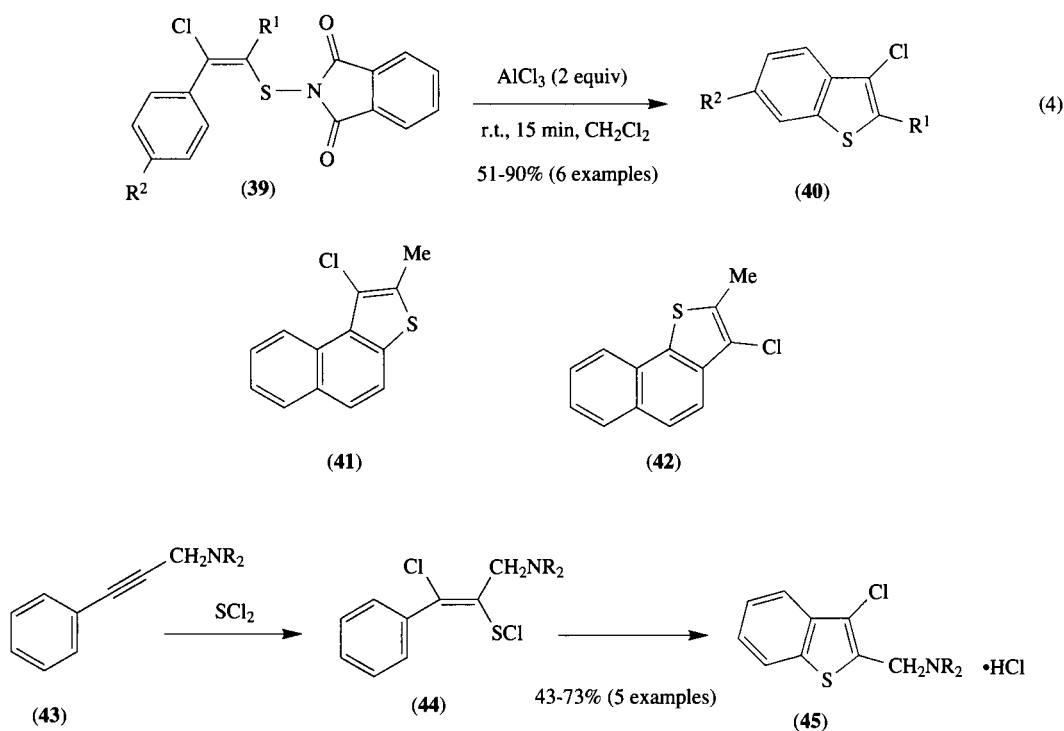
In all of the examples given above, the sulfur atom acted as a nucleophile. In contrast, there are a number of cases where electron-deficient sulfur species such as sulphenyl ion and its equivalent (e.g. disulfide/Lewis acid complex, sulfenic acid, sulphenyl halide), sulfonium ion, sulfine, etc., serve as an electrophile.

2-Aminothiophenes (**38**) can be prepared via a novel iodoiminolactonization of unsaturated secondary thioamides (**35**). Thus, the successive treatment of (**35**) with iodine, DBU, and acetyl chloride gives the thiophenes (**38**) in 25–57% overall yields (Scheme 12). Isolation of the intermediates (**36**) and (**37**), which are useful for further elaboration, is possible <86CC1671, 88T4777>.



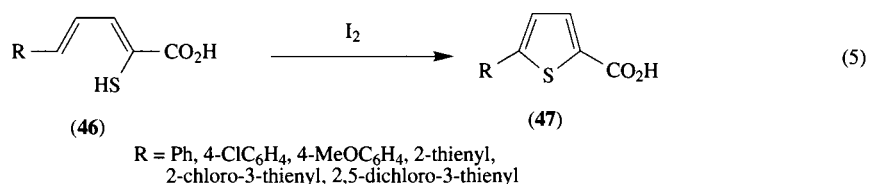
Scheme 12

(*E*)- β -Chlorovinylsulfenamides (**39**), obtainable by addition of phthalimidesulfonyl chloride to diaryl- or alkyl(aryl)ethynes, undergo $AlCl_3$ and other Lewis acid-catalyzed intramolecular electrophilic substitution to give benzo[*b*]thiophenes (**40**) (Equation (4)) <94S521>. Naphthothiophenes (**41**) and (**42**) are also prepared in this way. Reaction of alkynes (**43**) with sulfur dichloride proceeds in a similar way to give benzothiophenes (**45**) via sulfonyl chloride intermediates (**44**) (Scheme 13) <90KGS1331>.

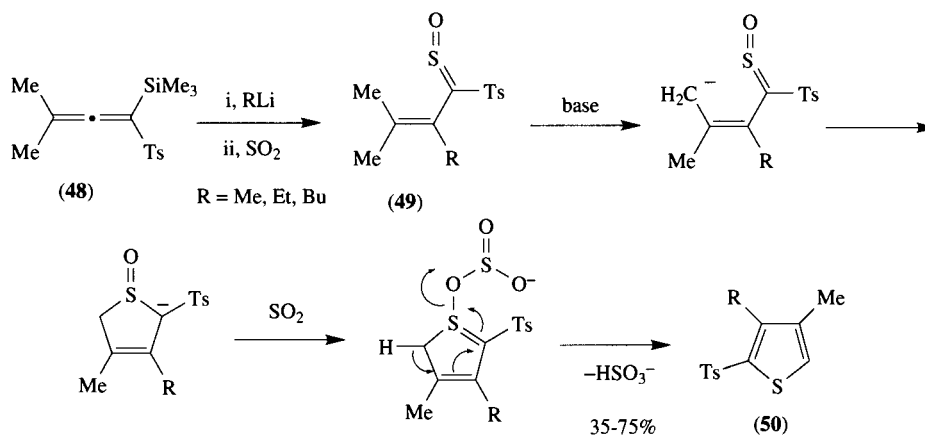


Scheme 13

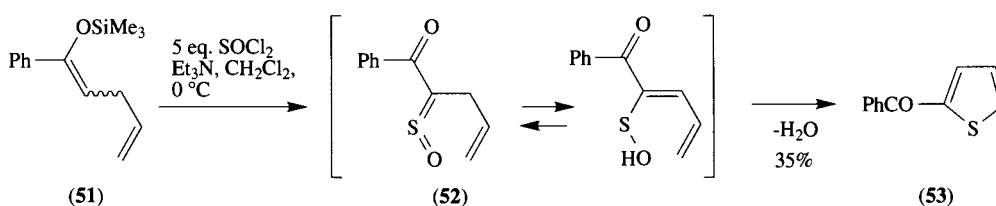
Oxidative ring closure of enethiols (α -thioketocarboxylic acid) (**46**), which proceeds via disulfides, produces thiophenes (**47**) in good yields (Equation (5)) <86EUP158380, 88JHC367>.



Conjugated vinyl sulfines (**49**), produced by treatment of the allene (**48**) with alkyllithium and then with sulfur dioxide, undergo rapid and unprecedented cycloaromatization to give thiophenes (**50**) in one pot (Scheme 14) <91TL3867>. The mechanism proposed involves generation of the allylic carbanions from (**49**) with trimethylsilanoate acting as the base, intramolecular addition of the carbanion carbon to the sulfine sulfur, and a novel Pummerer-type rearrangement induced by sulfur dioxide under basic conditions. On the other hand, the α -oxo sulfine (**52**), prepared from (**51**), is capable of enethiolization to give a vinyl sulfenic acid, which undergoes an intramolecular cyclization leading to the thiophene (**53**) (Scheme 15) <93PS(74)371>. The sulfoxide (**55**), obtainable in a high yield by thermolysis of (**54**), undergoes a unique Pummerer reaction, accompanied by methyl migration, to give thiophene (**56**) in a high yield (Scheme 16) <89KGS1629>.



Scheme 14

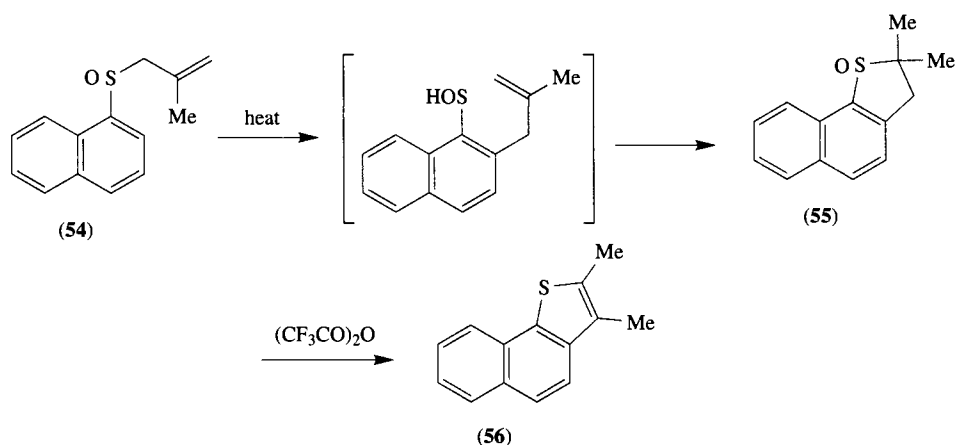


Scheme 15

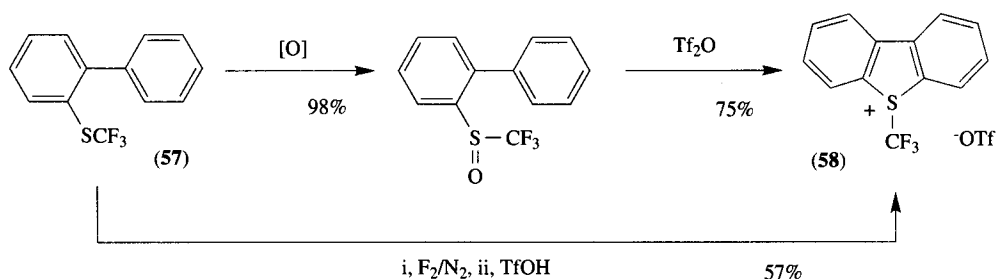
Thiophenium triflate (**58**), a useful reagent for trifluoromethylation, can be prepared either by oxidation of the sulfide (**57**) with MCPBA and then treatment of the resulting sulfoxide with an equivalent of triflic anhydride at room temperature or by treatment of (**57**) with fluorine diluted with nitrogen and then with an equivalent of triflic acid at -78°C (Scheme 17) <90TL3579>.

2.11.2.1.3 Processes involving addition of thiyl radicals to unsaturated carbon-carbon bonds

There are also cases where addition of thiyl radicals to unsaturated carbon-carbon bonds is the crucial step for the thiophene ring formation. Thus, *o*-substituted thiophenoxyl radicals (**60**), generated by flash vacuum pyrolysis (FVP) of readily available aryl salicylates (**59**), undergo a novel rearrangement-extrusion-cyclization sequence to give dibenzothiophenes (**61**) (Scheme 18) <90CC395>. The method has the advantage of good yields of the products and ready accessibility of

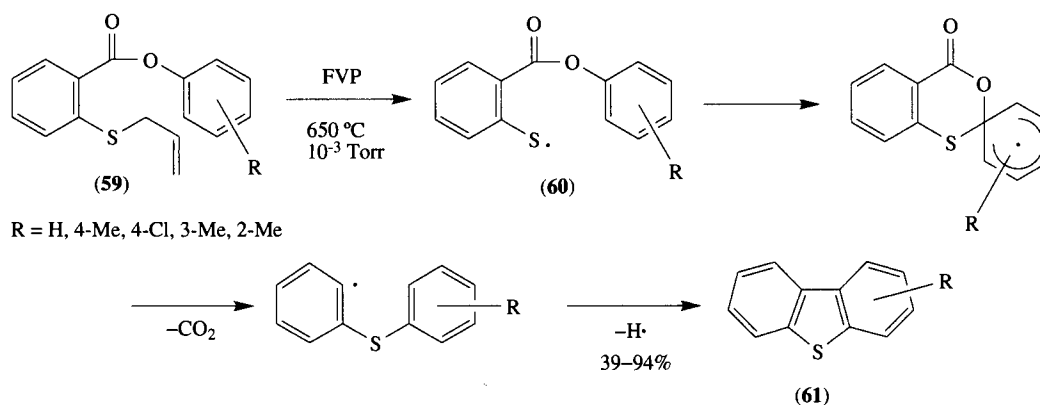


Scheme 16



Scheme 17

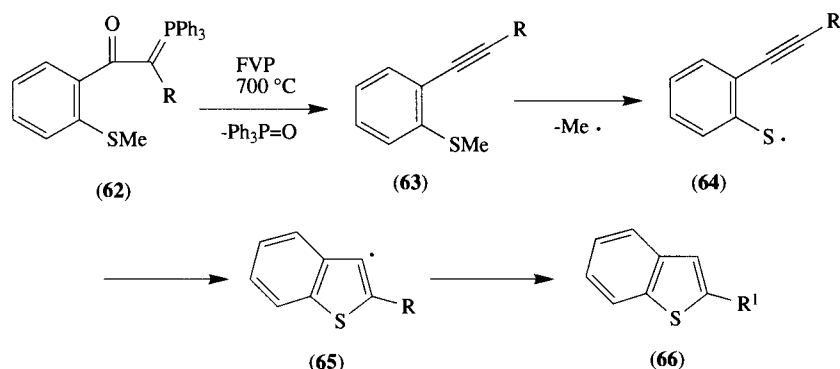
the starting materials (this synthesis, though treated here, could also be classed as ring synthesis by forming a bond γ to the sulfur).



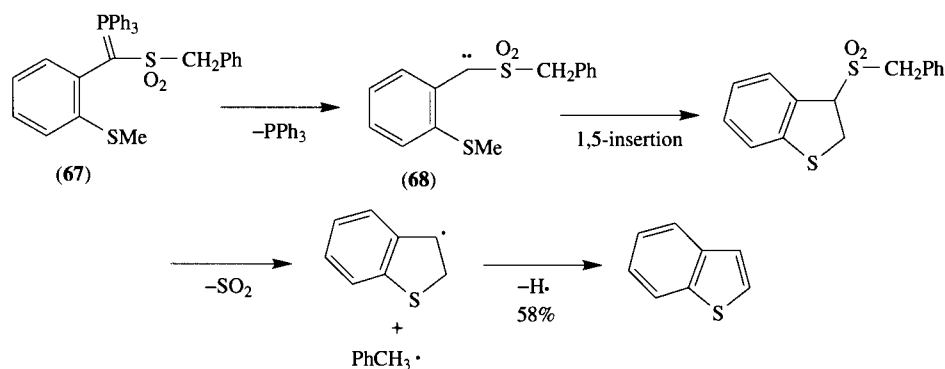
Scheme 18

FVP of the phosphorus ylides (62) affords initially the alkynes (63), which produce thiyl radicals (64) with loss of methyl radical. The radicals (64) cyclize to give thienyl radicals (65), which then undergo a novel fragmentation to give thiophenes (66) as the final products (Scheme 19); for example, when $\text{R} = \text{Et}$ in (62), (66; $\text{R}^1 = \text{vinyl}$) is obtained as the sole product in 49% yield, while when $\text{R} = \text{Pr}^n$, (66; $\text{R}^1 = \text{Me, Et, and vinyl}$) are obtained in 6, 45, and 9% yields, respectively

<91CC512>. Incidentally, FVP of the sulfonyl stabilized ylide (**67**) affords benzo[*b*]thiophene in 58% yield; the reaction involves the initial formation of the carbene (**68**) and its intramolecular C—H insertion (Scheme 20) <94JCS(P1)2455>.

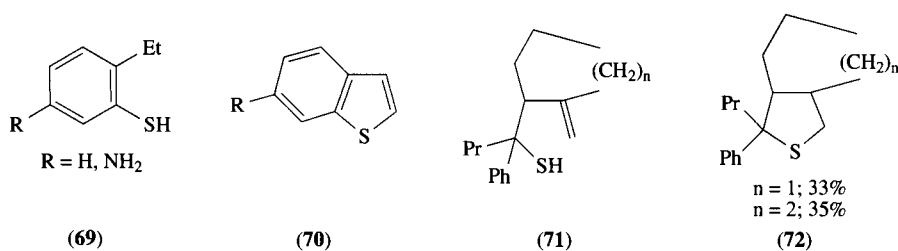


Scheme 19



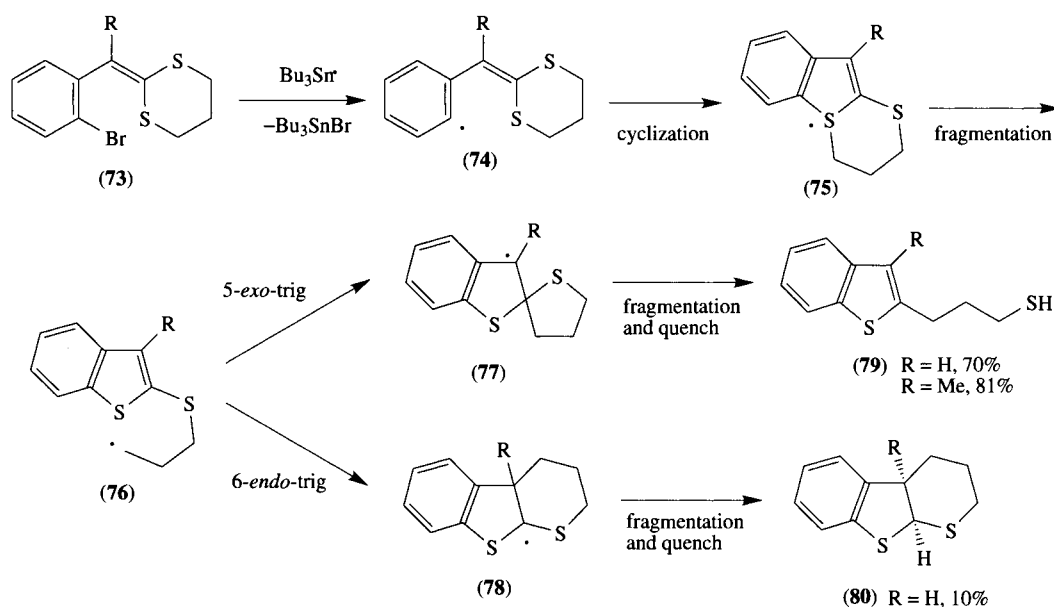
Scheme 20

Formation of benzothiophenes (**70**) by catalytic dehydrogenation of (**69**) probably involves thiyl radicals as an intermediate <83JCR(S)276>. Similarly, formation of tetrahydrothiophenes (**72**) by cyclization of (**71**) on treatment with $\text{Pr}_3\text{B}/\text{O}_2$ in methanol may also involve a thiyl radical intermediate <89IZV2115>.

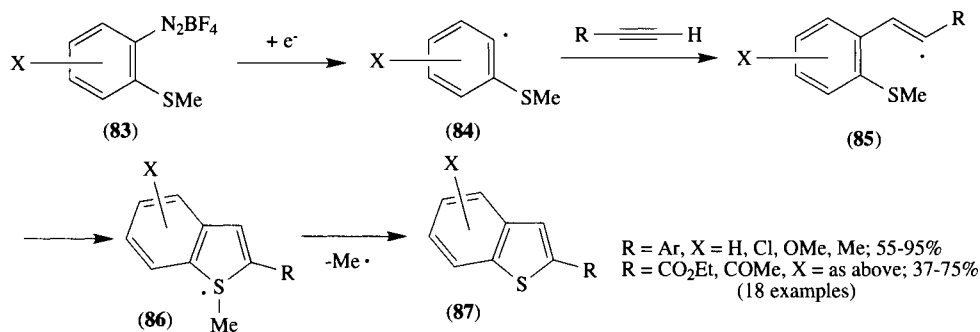
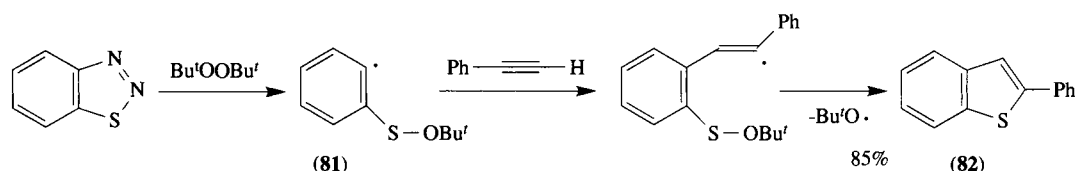


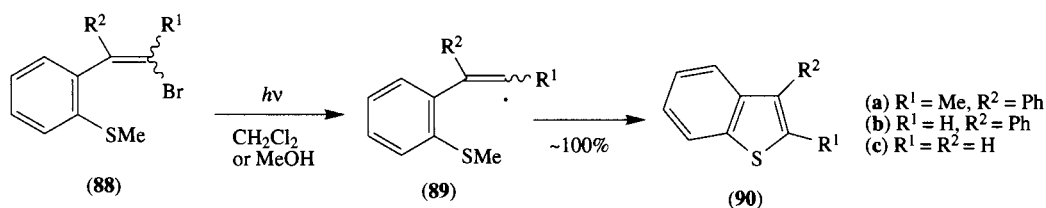
2.11.2.1.4 Processes involving attack of vinyl or aryl radicals on sulfur

Treatment of the ketenethioacetals (**73**) with Bu_3SnH under radical forming conditions (AIBN, PhH , reflux) directly affords thiophenes (**79**) and/or (**80**) (Scheme 21) <93TL5653>. The reaction involves a radical centered tandem cyclization–fragmentation sequence; for example, cyclization (**74**)→(**75**), fragmentation (**75**)→(**76**), cyclization (**76**)→(**77**), fragmentation (**77**)→(**79**). Application of this procedure to some other annelated thiophenes was also described.



Heating 1,2,3-benzothiadiazole with phenylethyne in di-*t*-butylperoxide at reflux affords 2-phenylbenzo[*b*]thiophene (**82**) (Scheme 22) <84JOC4482>. The mechanism involving a radical intermediate (**81**) was proposed. Treatment of diazonium salts (**83**) with alkynes in acetone in the presence of NaI or freshly prepared copper powder, or in DMSO containing FeSO₄, provides a more practical synthesis of benzothiophenes (**87**) (Scheme 23) <85CC1390>. Also in this case, the proposed mechanism involves addition of aryl radicals (**84**) to alkynes leading to vinyl radicals (**85**) which then cyclize, via an S_Hⁱ mechanism, to give sulfuranyl radicals (**86**). The latter then extrudes a methyl radical to give thiophenes (**87**). In a similar way, vinyl radicals (**89**), generated by photolysis of (**88**), undergo cyclization to give benzothiophenes (**90**) quantitatively (Scheme 24) <88CL1637>.

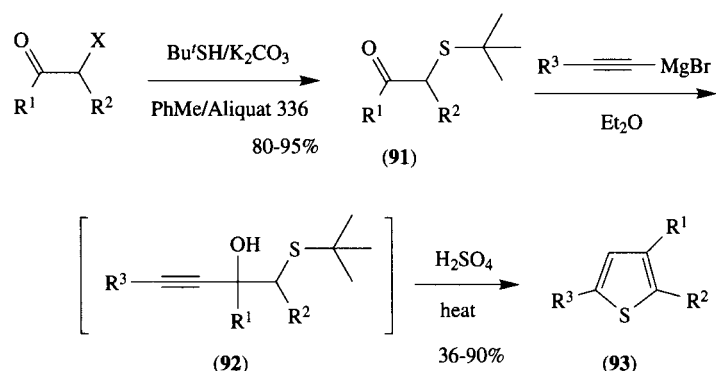




Scheme 24

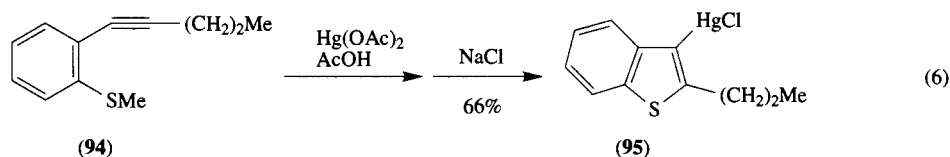
2.11.2.1.5 Processes involving attack of carbenium ions on sulfur

Treatment of crude alkynylalkanols (**92**), which are obtainable from (**91**) and 1-alkynylmagnesium halides, with hot aqueous acid provides a convenient synthesis of 3,5-di- and 2,3,5-trisubstituted (five-, six-, and seven-membered ring-fused) thiophenes (**93**) (Scheme 25) <89S210>. Starting materials (**91**) are easily synthesized from α -halo ketones in high yields. The reaction may involve electrophilic attack of vinyl cation, produced by protonation of the triple bond, on the sulfur.

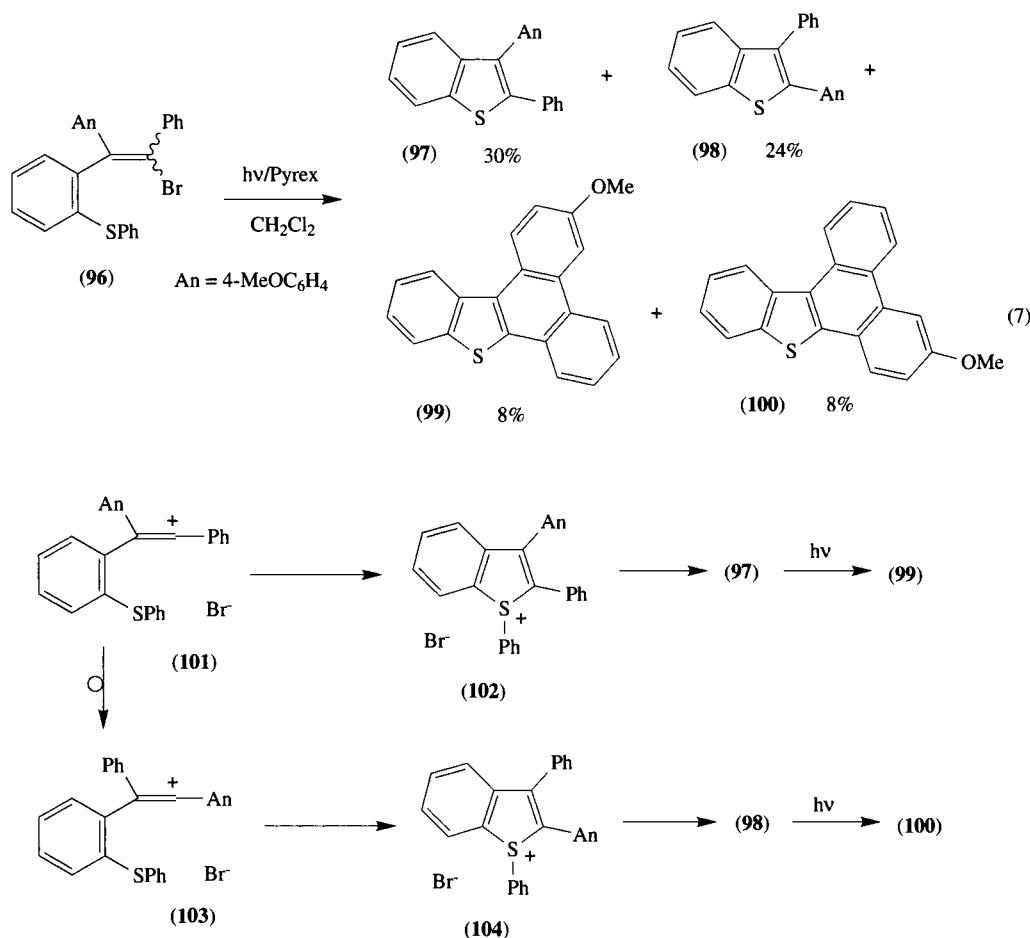


Scheme 25

An intramolecular solvomercuration of (**94**) in acetic acid affords the thiophene (**95**) upon aqueous sodium chloride workup (Equation (6)) <84JA4218>. The reaction is applicable to the preparation of a variety of five- and six-membered heterocycles.



In a marked contrast to the photolysis of (**88**), photolysis of (**96**) gives the rearranged product (**98**) in addition to the normal product (**97**) (Equation (7)) <86CL839>. Compounds (**99**) and (**100**) are due to further photolysis of (**97**) and (**98**), respectively. The formation of (**97**) and (**98**) was explained by assuming the rearrangement of the vinyl cation (**101**) to (**103**) and their cyclization to (**102**) and (**104**), respectively (Scheme 26). The vinyl cation intermediate (**101**) is generated by an electron transfer process from the initially formed radical pair.



Scheme 26

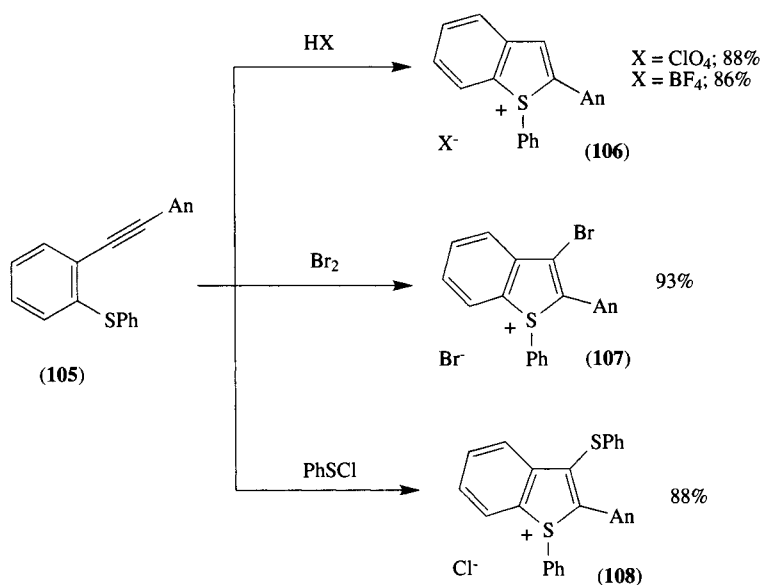
Although, in the photolysis of (96), benzothiophenium ions (102) and (104) were postulated as the precursor compounds leading to (97) and (98), respectively, they could never be isolated. However, action of electrophiles on *o*-(phenylthio)phenylalkynes allows the easy preparation of benzothiophenium salts (<86CL399, 89TL7445, 93T5055, 94JCS(P1)1907>). Thus, reactions of (105) with perchloric acid, tetrafluoroboric acid, bromine, and benzenesulfonyl chloride afford the corresponding thiophenium salts (106)–(108) in high yields (Scheme 27). In this way, a variety of thiophenium salts were prepared and the x-ray single crystal structure of 1,2,3-triphenylbenzo[*b*]thiophenium perchlorate was determined.

The ketene, generated from the acid chloride (109), undergoes [2 + 2] cycloaddition with the azomethine (110) to give the lactam (111), which with iodine, is converted to the dihydrothiophene derivative (112), a compound having a β -lactam core structure related to the penam antibiotics (Scheme 28) (<94JOC5858>). In a similar way, several compounds related to (112) were synthesized in good yields.

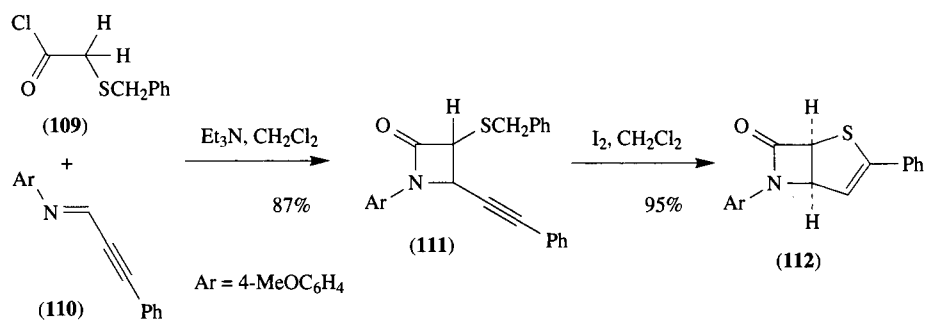
A high yield synthesis of 2-aminothiophenes (113) by the process involving acid-catalyzed electrophilic attack of the nitrile group on the sulfide sulfur was reported (Scheme 29) (<91SUL197>).

2.11.2.1.6 Thio-Claisen rearrangement

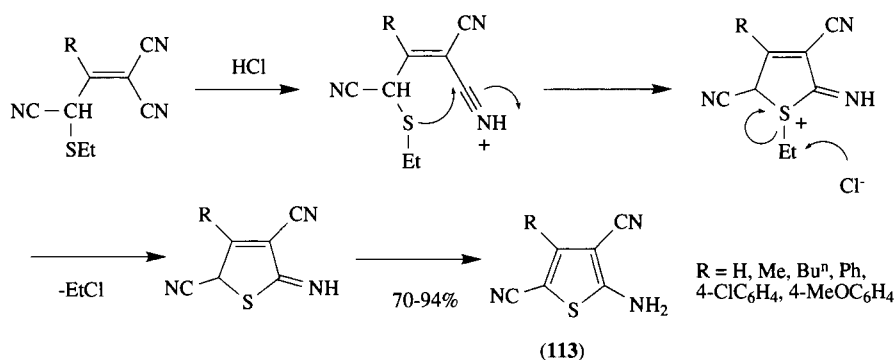
If we consider the intermediate of thio-Claisen rearrangement, it is reasonable that the ring synthesis via thio-Claisen rearrangement is treated in this section. Isomerization of the sulfide (114) to the thiophene (115) was examined under a variety of conditions (Scheme 30) (<85JCS(P1)2643>). The most successful result was attained when (114) was distilled *in vacuo* through a silica tube packed



Scheme 27

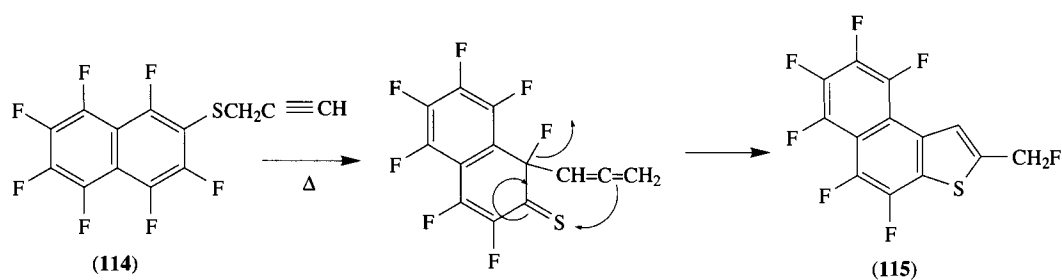


Scheme 28

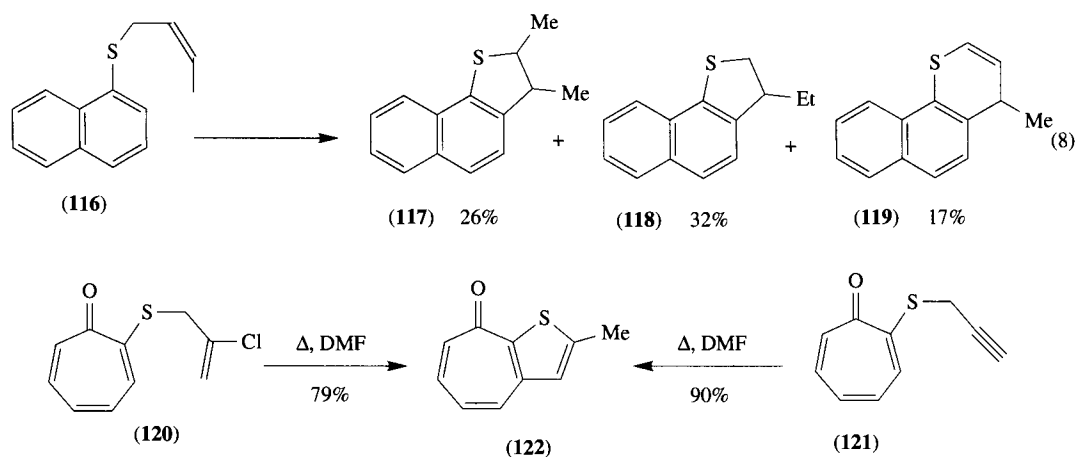


Scheme 29

with quartz wool at 360°C; (115) was obtained in 81% yield. Thermal isomerization of (116) (Equation (8)) gives compounds (117)–(119) <83MI 211-02>. Sulfides (120) and (121) undergo thio-Claisen rearrangement to give the thiophene (122) in good yields (Scheme 31) <83H(20)1709>. A range of thiophenes having substituents on the tropone nucleus was synthesized in this way.



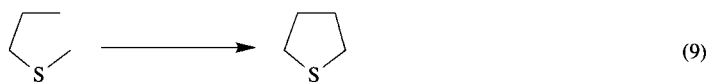
Scheme 30



Scheme 31

2.11.2.2 Formation of a Bond β to the Sulfur Atom

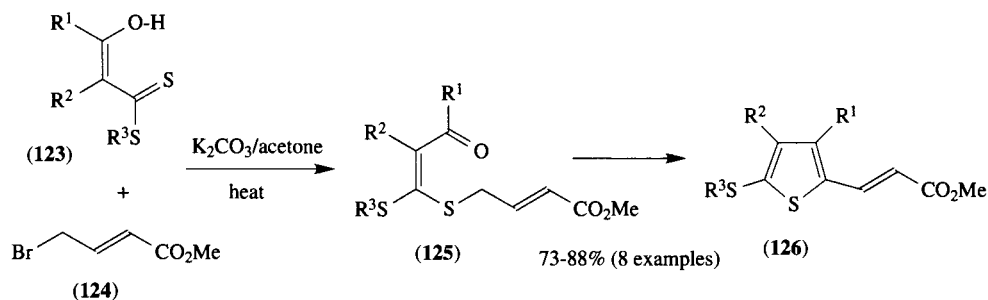
Several new syntheses which belong to this category have been developed. However, many syntheses reported since the early 1980s are applications or modifications of the traditional methods, though many of them are practical. The most frequently encountered method involves the intramolecular condensation between a carbonyl group with an α -carbon atom adjacent to the sulfur and activated by an electron-withdrawing group. Intramolecular addition of the α -carbanion adjacent to the sulfur to a cyano group is also used conveniently for the preparation of 3-aminothiophenes.



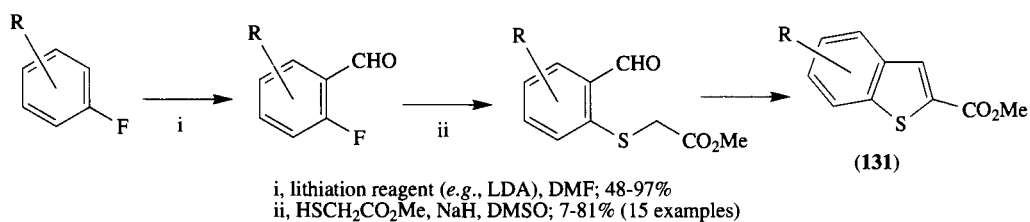
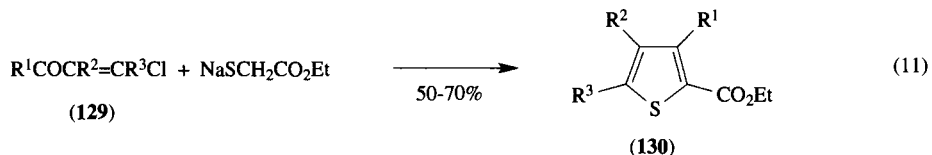
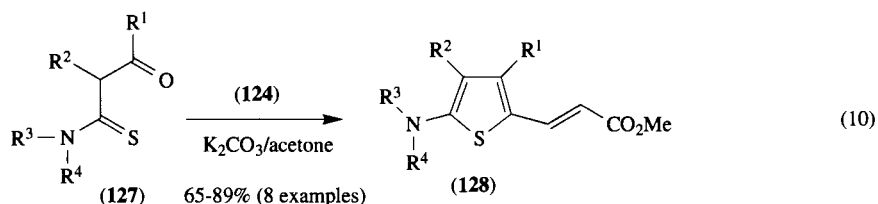
2.11.2.2.1 Intramolecular condensation of an active methylene with a carbonyl group and related groups

3-Oxodithioesters (123) reacts with methyl 4-bromocrotonate (124) to give the mixed dithioacetals (125), which immediately undergo an intramolecular aldol condensation to provide a practical synthesis of thiophenes (126) (Scheme 32) <88S556>. Similarly, reaction of 3-oxothioamides (127) with (124) gives thiophenes (128) (Equation (10)). Reaction of the chlorinated α,β -unsaturated ketones (129) with ethyl thioglycolate followed by aldol condensation also provides thiophenes (130) (Equation (11)) <90SC2537>. A simple two-step synthesis of substituted benzo[b]thiophenes (131) from aryl fluorides has been developed (Scheme 33) <92TL7499>. The route involves a selective lithiation *ortho* to fluorine, followed by formylation and subsequently, displacement of fluorine with thioglycolate and base-induced ring closure in a single operation. Benzothiophenes (133) <93SC743> and (135) <87H(26)2717> are also prepared from (132) (Equation (12)) and (134) (Equation (13)),

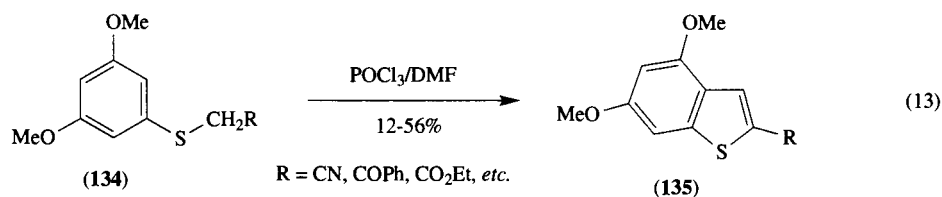
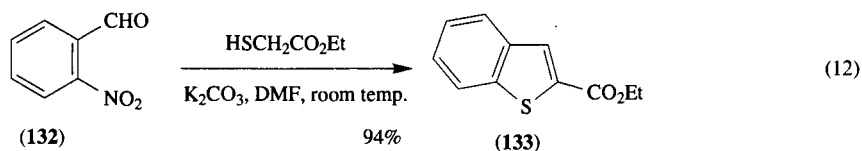
respectively, in a single operation. Treatment of the β -ketoenolates (**136**) with carbon disulfide in the presence of sodium hydride and subsequent alkylation yield the ketene *S,S*-acetals (**137**), which undergo cyclization to give a mixture of thiophenes (**138**) and (**139**) (Scheme 34) <92LA387>. Reaction of (**136**) with phenyl isothiocyanate followed by treatment with PhCOCH_2Cl affords 3-aryl-5-benzoyl-2-anilinothiophenes. Application of the procedure to the preparation of thieno[2,3-*b*]thiophenes was also reported.

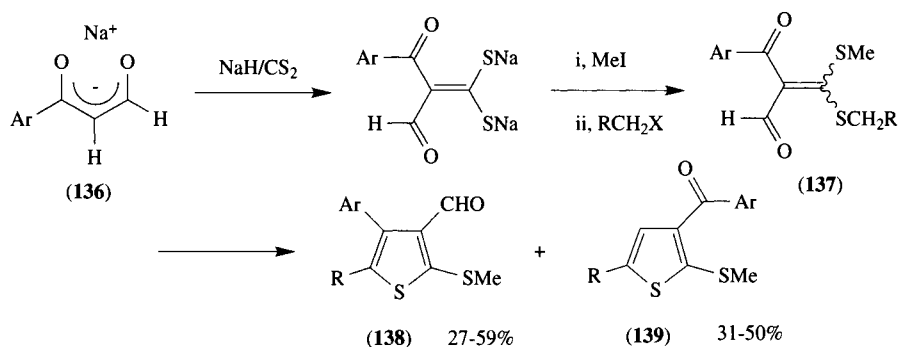


Scheme 32



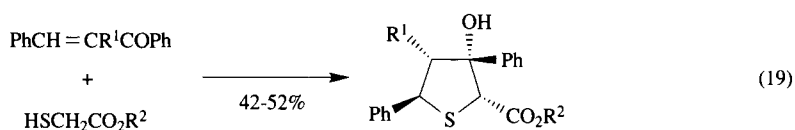
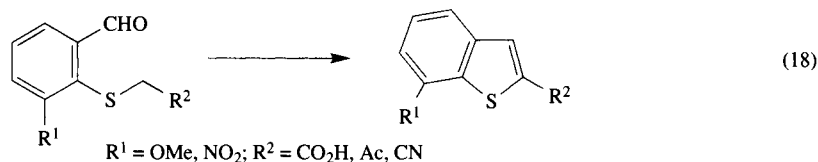
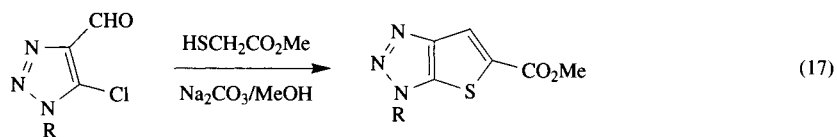
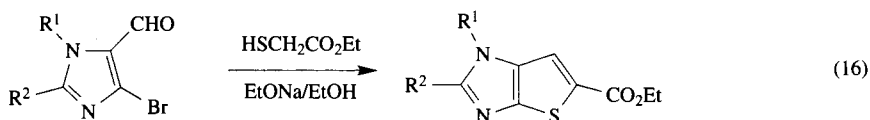
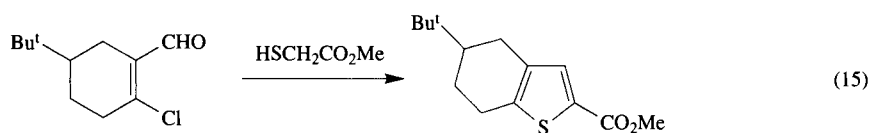
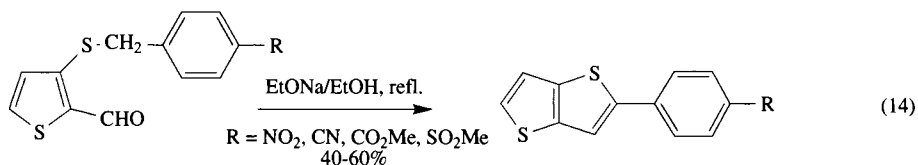
Scheme 33

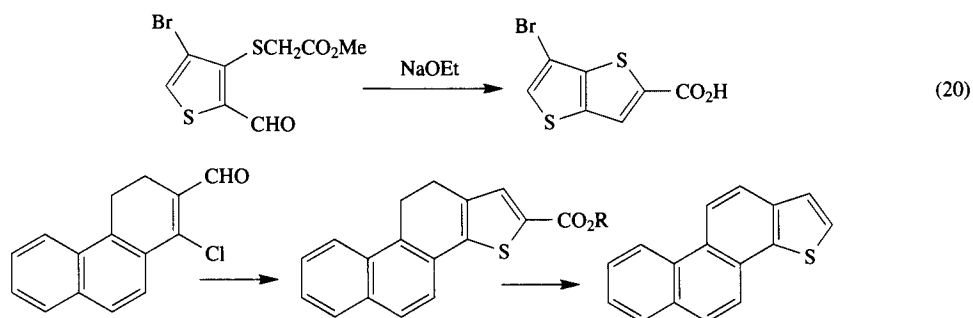




Scheme 34

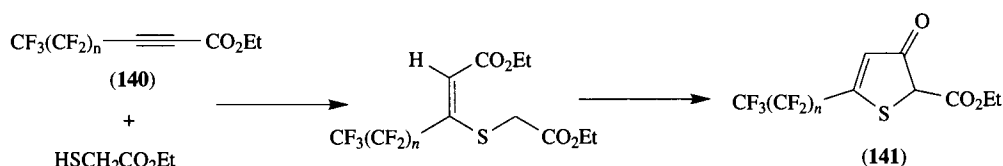
Other representative examples are given in Equations (14)–(20) and Scheme 35 <91JHC999>: (14) <94JCS(P1)2603>; (15) <89SC2731>; (16) <85CC1428>; (17) <86H2299>; (18) <83JCS(P1)2973, 84JCS(P1)385>; (19) <87SA(A)1397>; (20) <89TL3315>. A large number of patents claim the preparation of thiophenes and benzo derivatives by procedures similar to those given above <83EUP95340, 85MIP216237, 85GEP(O)3406997, 88EUP273602, 93EUP572712, 93JAP(K)05155881, 93JAP(K)05194484, 93JAP(K)05246981, 93USP5266705>.



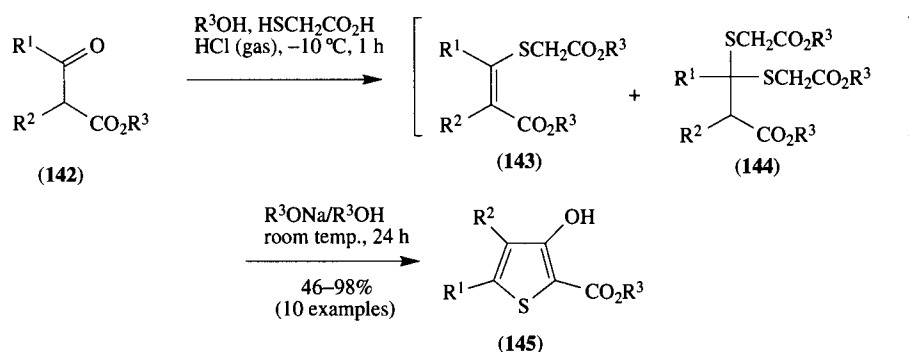


Scheme 35

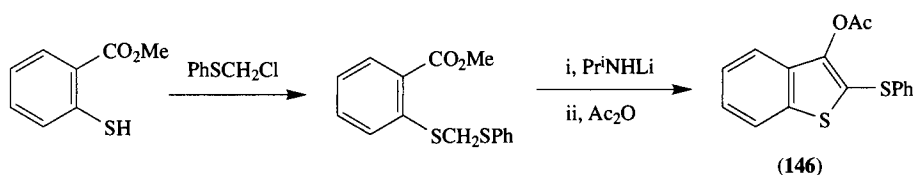
Syntheses given in Schemes 36–38 and Equation (21) involve the condensation of an activated methylene carbon with a carbonyl group of esters. Thus, Michael addition of ethyl thioglycolate to fluoroalkynes (**140**) followed by Dieckmann condensation provides a practical synthesis of thiophenones (3-hydroxythiophenes) (**141**) (Scheme 36) <86T663>. Preparation of thiophenes (**145**) from α -keto esters (**142**) is the modification of the Fiesselmann procedure (Scheme 37) <92S526>. A hydrogen chloride stream was bubbled into an alcoholic solution of (**142**) and thioglycolic acid. The isolated crude product, probably a mixture of compounds (**143**) and (**144**), was treated with sodium alkoxide to give the thiophenes (**145**). A variety of 2,3-annulated thiophenes were prepared in good yields by this modification. In the preparation of thiophenes (**146**) (Scheme 38) <86EUP177927> and (**147**) (Equation (21)) <84JAP(K)5942375>, strong bases were used to generate carbanions from weakly activated methylene carbons and to effect cyclization.



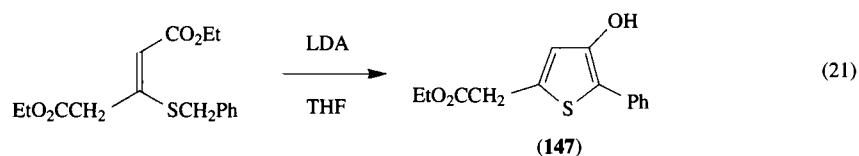
Scheme 36



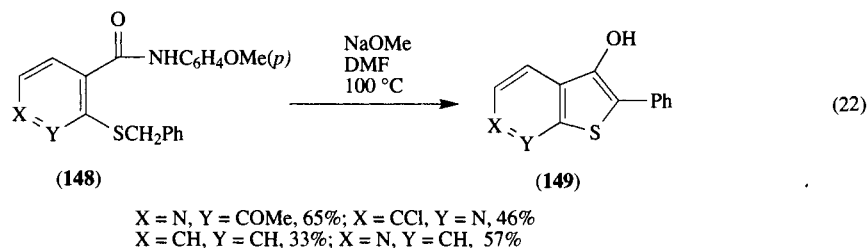
Scheme 37



Scheme 38

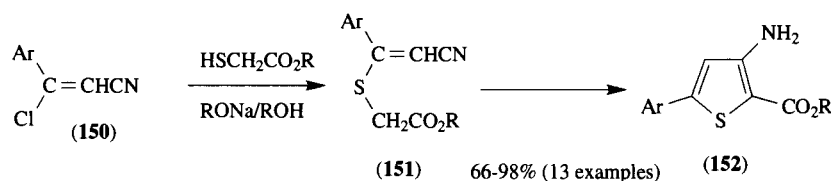


Preparation of thiophenes (149) from (148) (Equation (22)) provides an interesting example that the condensation of an active methylene with an amide carbonyl leads to thiophenes <93TL2875>.

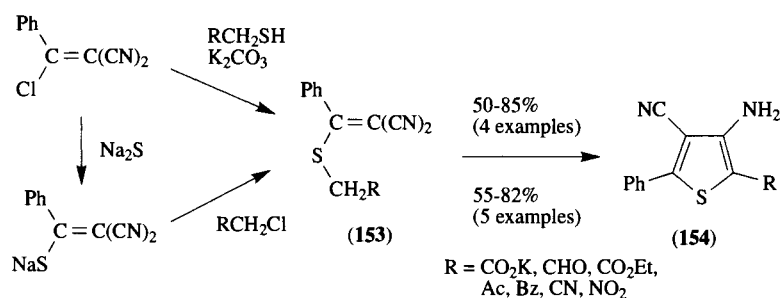


2.11.2.2.2 Intramolecular addition of an active methylene carbon atom to a nitrile group

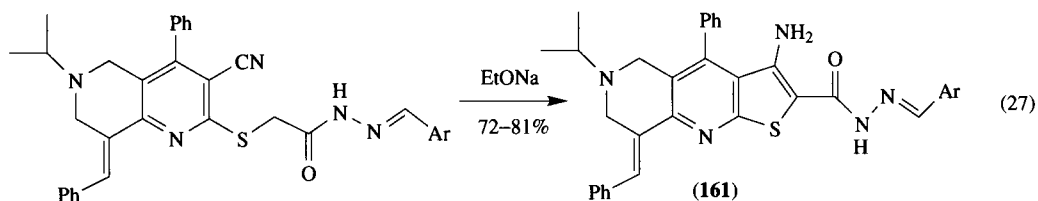
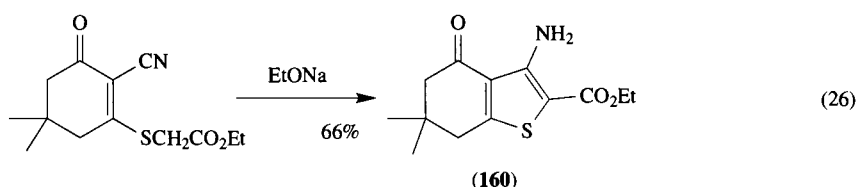
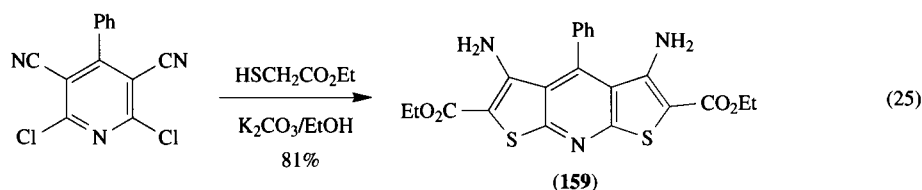
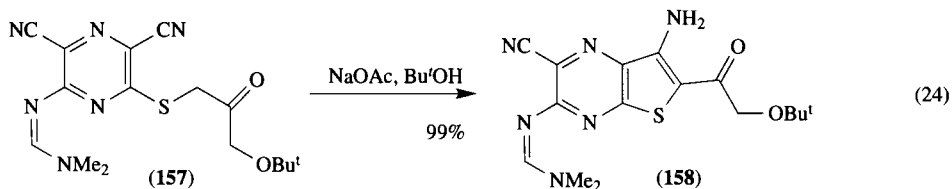
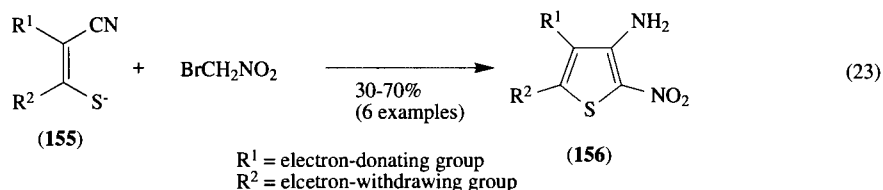
β -Chlorocinnamionitriles (150), which have become readily obtainable from aryl methyl ketones in one pot, react with thioglycolic acid esters in the presence of a base to give 3-aminothiophenes (152) (Scheme 39) <84S275>. The reaction involves a Dieckmann–Thorpe cyclization of the probable intermediates (151). In a similar way, compounds (153), produced from β -chloro- α -cyano-cinnamionitrile in two ways, undergo a base-catalyzed cyclization to give aminothiophenes (154) (Scheme 40) <92M455>. Bromonitromethane is a versatile electrophile and reacts with thiolates (155), which are obtainable either by successive treatment of nitriles with carbon disulfide and methyl iodide or by treatment of nitriles with isothiocyanates in the presence of a base, to provide a practical synthesis of 3-amino-2-nitrothiophenes (156) (Equation (23)) <83CC834, 86JCS(P1)1171>. An aminothiophene (158), a precursor compound in the total synthesis of urothione, was obtained by cyclization of (157) (Equation (24)) <89JA285>. Aminothiophenes (159) <94H(38)1299>, (160) <93MC160>, (161) <93BCJ3716>, and a number of related compounds were prepared in similar ways (Equations (25)–(27), respectively).



Scheme 39

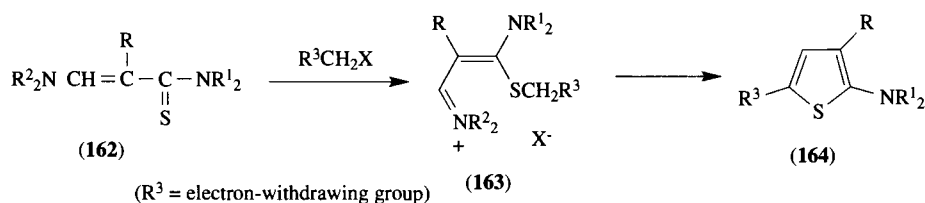


Scheme 40

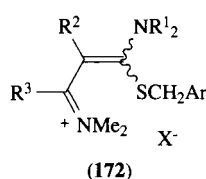
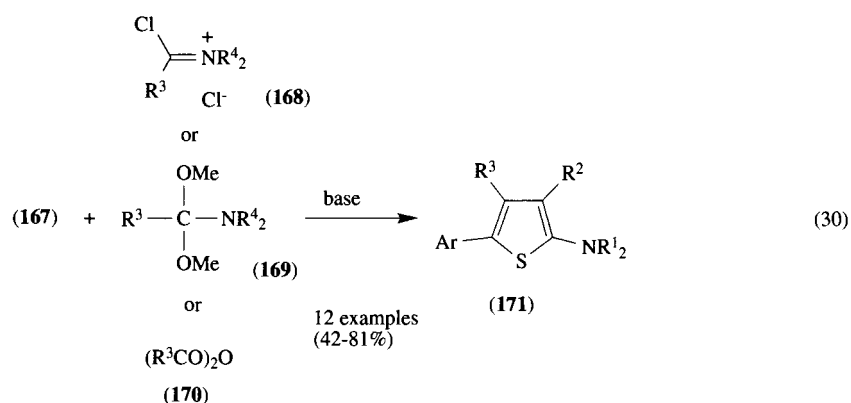
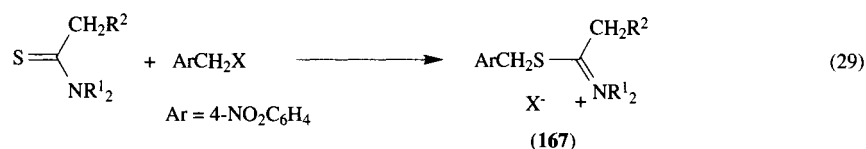
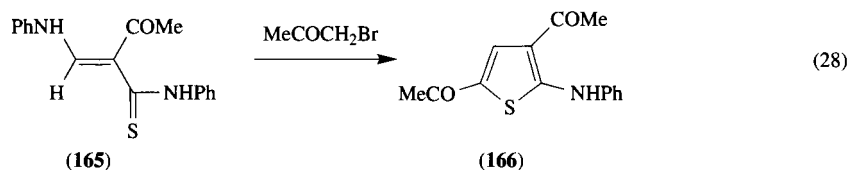


2.11.2.2.3 Miscellaneous cyclization

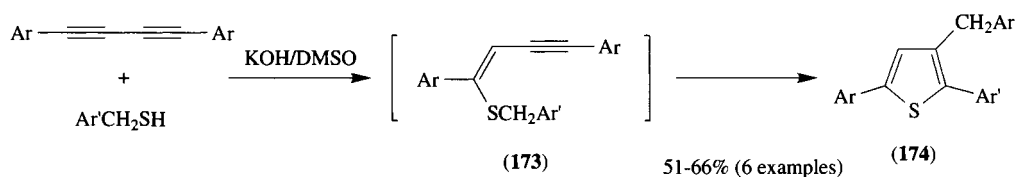
Reaction of halomethanes (carrying an electron-withdrawing substituent such as NO_2 , benzoyl, $p\text{-NO}_2\text{C}_6\text{H}_4$) with 3-aminothioacrylamides (**162**) in the presence of Et_3N affords 2-aminothiophenes (**164**) via intermediates (**163**) in good yields (Scheme 41) [83JPR168]. Similarly, reaction of (**165**) with bromoacetone produces the thiophene (**166**) (Equation (28)) [90UKZ971]. Reactions of the readily obtainable iminium salts (**167**) with amide chlorides (**168**), amide acetals (**169**), or acid anhydrides (**170**), give thiophenes (**171**) (Equation (30)) [85S412]. The reaction is carried out in the presence of triethylamine as the base. In the absence of the base, intermediates (**172**), which can be separately cyclized to the thiophenes, are isolable. Thus, the reaction involves the attack of acid derivatives (**168**)–(**170**) on the R^2 -substituted methylene group of (**167**) to give intermediates like (**172**), which undergo base-catalyzed cyclization to give (**171**).



Scheme 41



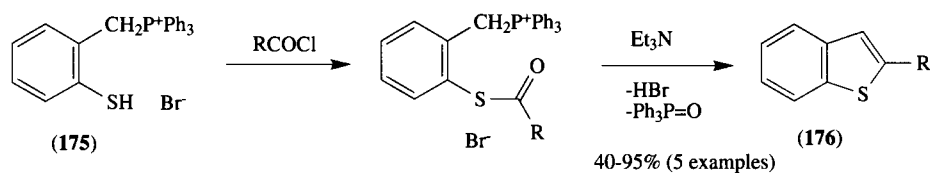
Reaction of 1,4-diarylbutadiynes with arenemethanethiols in DMSO in the presence of KOH affords the thiophenes (174) (Scheme 42) <93TL1753>. Carbanion formation at the benzyl carbon of the probable intermediates (173), followed by its nucleophilic attack on the *sp*-hybridized carbon leads to cyclization, and then aromatization to (174).



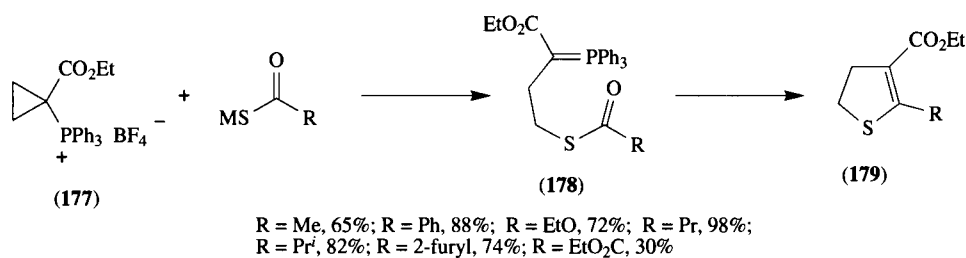
Scheme 42

Cyclization to thiophenes by an intramolecular Wittig reaction has been developed. Thus, the phosphonium bromide (175), obtainable from 2-thiolbenzenemethanol, is acylated and then cyclized with triethylamine as the base to give 2-substituted benzothiophenes (176) (Scheme 43) <86GEP(O)353308, 88S155>. The phosphonium salt (177) reacts with alkali-metal thiocarboxylates to give the intermediate phosphoranes (178), which cyclize to give 2,3-disubstituted 4,5-dihydrothiophenes (179) in one pot (Scheme 44) <94JCS(P1)2603>. Dehydrogenation of (179) with DDQ leads to the corresponding thiophenes.

FVP of the Meldrum acid derivatives (180) at 625°C (10⁻³ torr) proceeds cleanly and efficiently to give thiophenones (182) including the parent compound, which are often very unstable, air-sensitive, and acid-sensitive compounds (Scheme 45) <90CC375>; see also <88TL5919>. The mechanism involving ketenes (181) as intermediates was proposed for this conversion. Meanwhile, benzo-

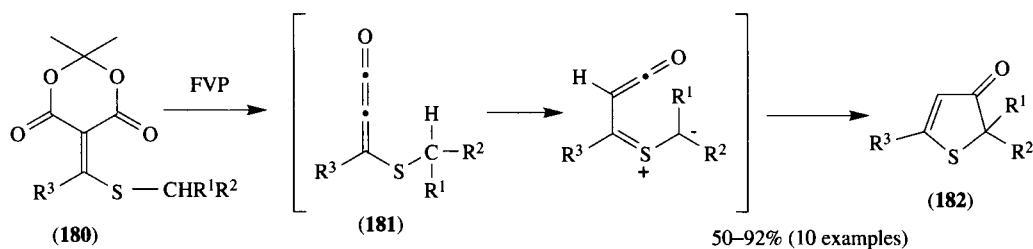


Scheme 43

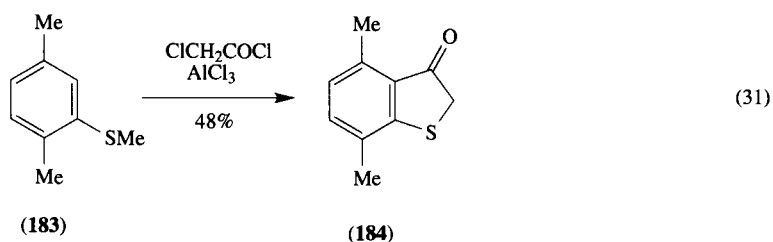


Scheme 44

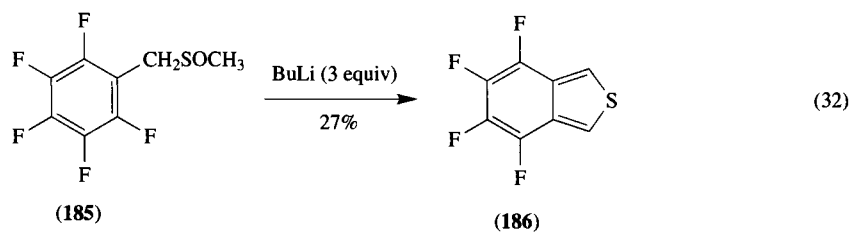
thiophenone (184) is obtainable by Friedel–Crafts reaction of the sulfide (183) with chloroacetyl chloride (Equation (31)) <85DOK(28)347>.



Scheme 45

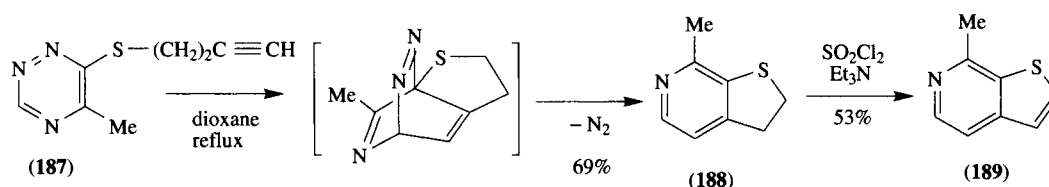


Treatment of the sulfoxide (185) with 3 equiv. of butyllithium affords the benzo[*c*]thiophene (186) in one pot as a volatile solid that was isolated with ease from the reaction mixture (Equation (32)) <90JCS(P1)1919>.



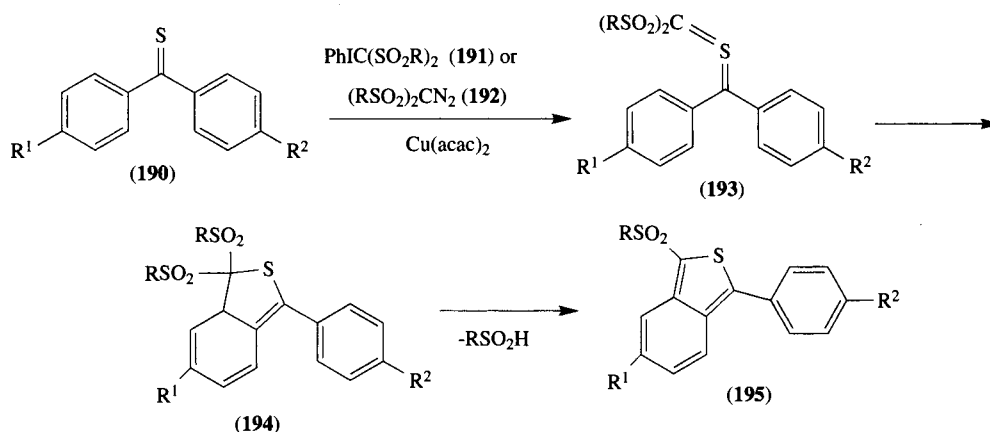
Intramolecular Diels–Alder reaction of the triazine (187) affords the dihydrothiophene (188), which is converted to the thiophene (189) via treatment with sulfur chloride (Scheme 46)

<89JOC4984>. Oxidation of (187) to the corresponding sulfoxide, intramolecular Diels–Alder reaction, and Pummerer dehydration lead to the same thiophene.

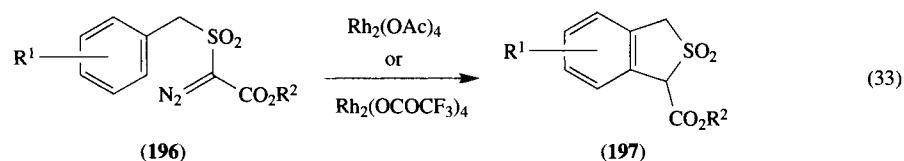


Scheme 46

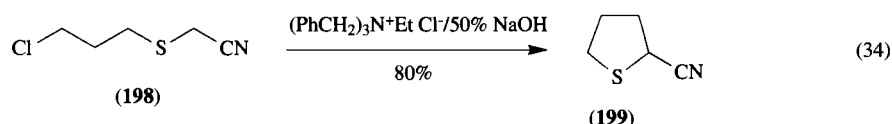
Reaction of thiobenzophenones (190) with the carbenoids generated from phenyliodonium bis(phenylsulfonyl)methylide (191; R = Ph) and bis(arylsulfonyl)diazomethanes (192; R = Ar) in the presence of a copper acetylacetonate catalyst affords a novel, interesting synthesis of benzo-[c]thiophenes (195) in good yields (Scheme 47) <91JCS(P1)1405, 94JCR(S)2>. The reaction probably proceeds via the initial formation of thiocarbonyl methylides, their cyclization to (194), and aromatization by elimination of a sulfenic acid. A previous report described the reaction with (191) but incorrectly assigned the product structures <89JCS(P1)379>. Rhodium acetate- or rhodium trifluoroacetate-catalyzed decomposition of a series of α -diazo- β -arylmethanesulfonyl esters (196) affords dihydrobenzo[c]thiophene 2,2-dioxides (197) in reasonable yields (Equation (33)) <89CJC1071>.



Scheme 47



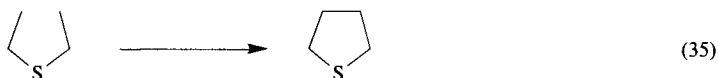
Intramolecular cyclization of compound (198) under phase-transfer catalysis conditions provides a convenient synthesis of 2-cyanotetrahydrothiophene (199) (Equation (34)) <87S452>.



2.11.2.3 Formation of a Bond γ to the Sulfur Atom

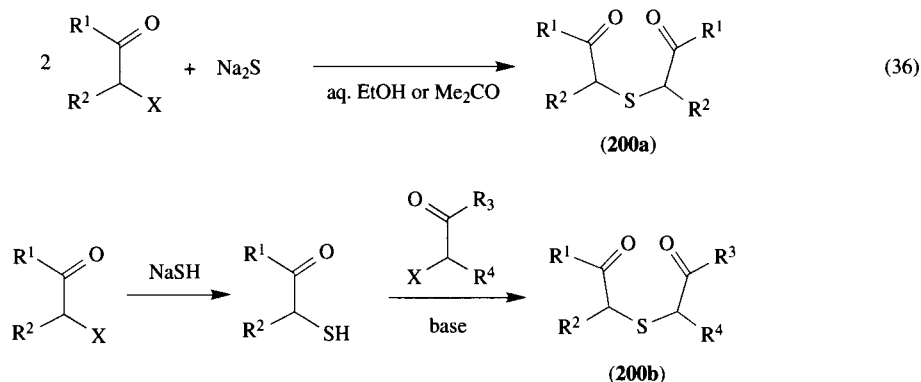
Several new thiophene syntheses that belong to this category have been developed. The new methods involve intramolecular reductive coupling of 3-thiopentane-1,5-diones, cycloaromatization

of unsaturated sulfides such as diallenyl and diethynyl sulfides and related sulfides, cyclization via vinyl radical intermediates, and intramolecular [4 + 2] cycloaddition, etc.

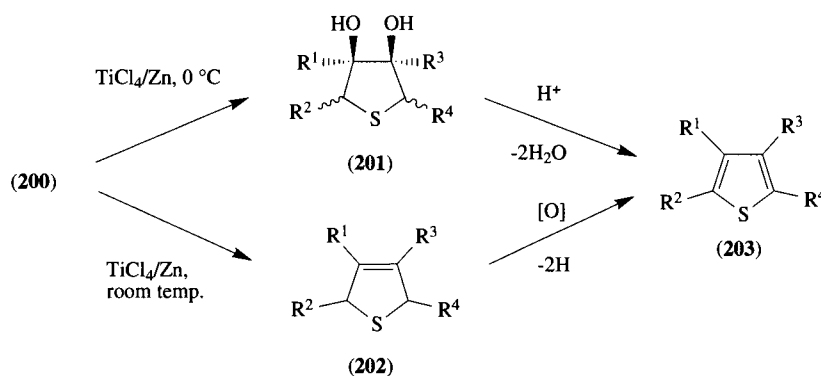


2.11.2.3.1 Intramolecular reductive coupling of diketo sulfides (3-thiapentane-1,5-diones)

A new practical thiophene synthesis, the Nakayama procedure, which involves an intramolecular pinacol coupling of diketo sulfides (3-thiapentane-1,5-diones) (**200**), was developed and reviewed <93PS(74)157, 94YGK308>. Symmetrically substituted diketo sulfides (**200a**) are obtainable nearly quantitatively by treating ethanol or acetone solutions of α -haloketones with aqueous sodium sulfide, while unsymmetrically substituted ones (**200b**) are prepared by reactions of α -haloketones with α -thiolketones that are obtainable by treatment of α -haloketones with sodium hydrogen sulfide (Equation (36) and Scheme 48). Treatment of (**200**) with a low-valent titanium reagent, prepared from TiCl_4 and Zn powder in THF, at 0°C or lower temperatures provides *cis*-thiolanediols (**201**) in 50–93% yield (12 examples). *p*-Toluenesulfonic acid-catalyzed dehydration of (**201**) works cleanly to give the corresponding thiophenes (**203**) in high yields (Scheme 49) <85TL1983, 87TL1799>. No product due to pinacol rearrangement formed. Meanwhile, the same reductive coupling at room temperature or higher temperatures produces the more reduced dihydrothiophenes (**202**) in 44–86% yields (14 examples) (Scheme 49) <85TL1981, 91BCJ3593>. Dehydrogenation of (**202**) with DDQ also provides the thiophenes (**203**) in excellent yields. The method is useful for the preparation of polysubstituted thiophenes, particularly for the preparation of thiophenes carrying substituents at 3- and 4-positions, to which introduction of substituents is not easy, once the thiophene ring is formed, since these positions are less reactive than 2- and 5-positions.



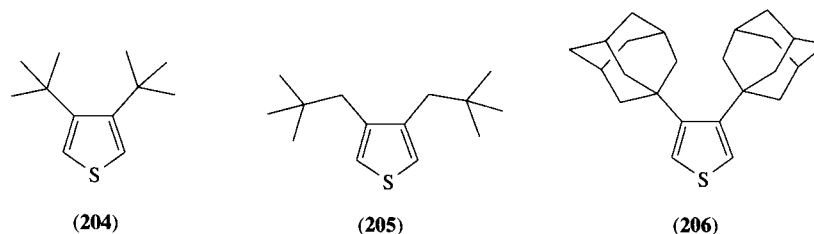
Scheme 48



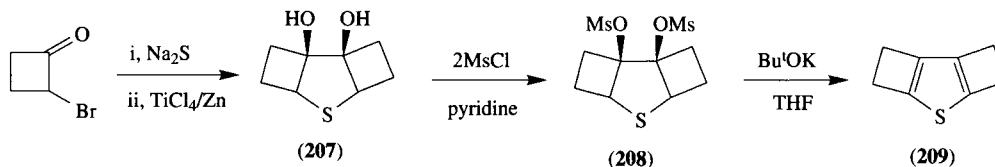
Scheme 49

The new method is also useful for the preparation of highly congested thiophenes. Thus, 3,4-di-*t*-butylthiophene (**204**) could be synthesized <88TL1161, 88JA6598, 90JA7648> by application of the

method in a better overall yield than previously reported. Preparation of the hitherto unknown 3,4-dineopentylthiophene (**205**) in a good overall yield has been reported <94TL2709>, and even more congested 3,4-di-1-adamantylthiophene (**206**) could be successfully synthesized starting from commercially available 1-chloroacetyladamantane <90JA5654>. The thiophene (**206**) is the first aromatic compound carrying two 1-adamantyl groups on the vicinal positions.

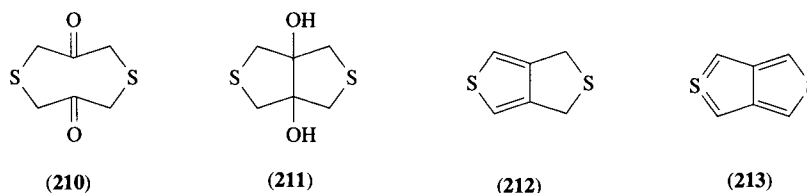


The new method enables us to prepare the highly angle-strained thiophene (**209**) with two fused four-membered rings starting from 2-bromocyclobutanone. Although in this case, attempted dehydration of the thiolanediol (**207**), directly leading to (**209**), was unsuccessful, it was overcome by converting (**207**) to the bis-mesylate (**208**) and then treating (**208**) with Bu^tOK, which gave (**209**) in a good overall yield (Scheme 50) <93JA4612>.

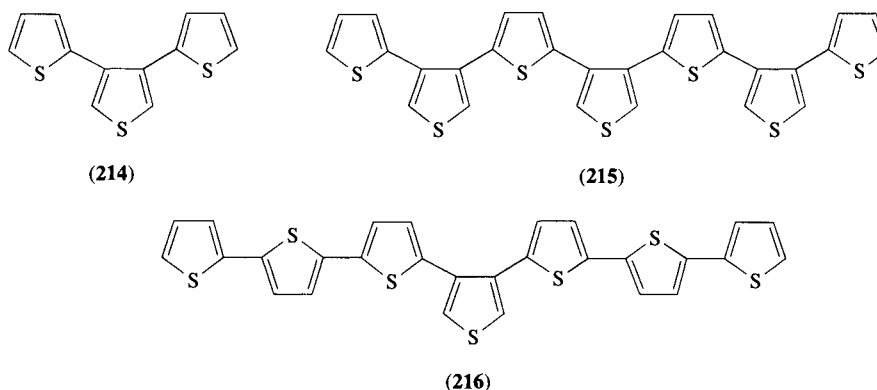


Scheme 50

Reductive coupling of the cyclic diketosulfide (**210**) produces the thiolanediol (**211**). Acid-catalyzed dehydration of (**211**) affords the thiophene (**212**), which serves as the precursor compound of the parent nonclassical thieno[3,4-*c*]thiophene (**213**) <88CC959>.



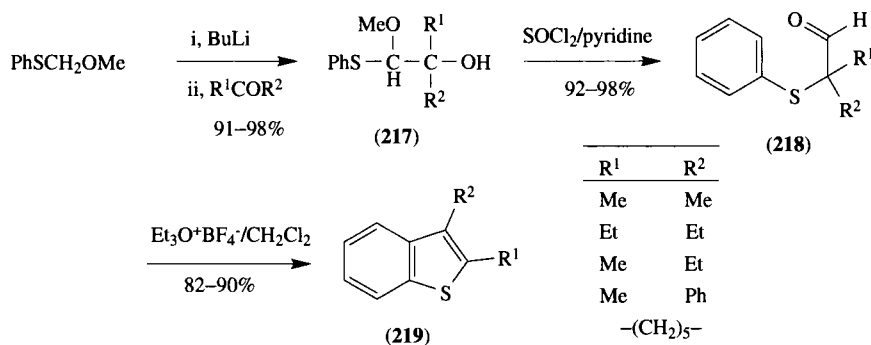
Furthermore, a number of structurally interesting polythiophenes such as (**214**)–(**216**) could be synthesized by application of the present method <87H(26)939, 87H(26)2599>.



The above thiophene synthesis is also applicable to the preparation of 2,5-dihydroselenophenes and selenophenes <87CC1072, 88TL1399>.

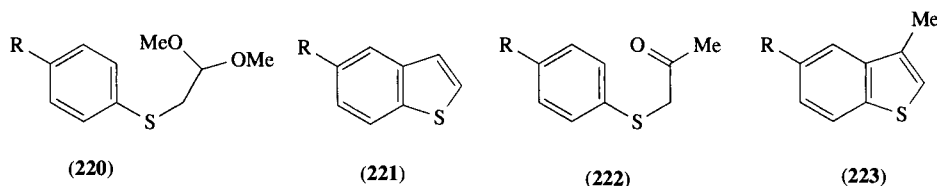
2.11.2.3.2 Intramolecular Friedel–Crafts or Wittig–Horner reaction

Treatment of 2-(phenylthio)alkanols (**217**), obtainable from methoxymethyl phenyl sulfide and ketones, with thionyl chloride affords the aldehydes (**218**) with rearrangement in high yields (Scheme 51). The cyclization of (**218**) to thiophenes (**219**) can be simply performed by stirring a solution of (**218**) with 2 equiv. of triethyloxonium tetrafluoroborate in dichloromethane. Although, in cases of $R^1 = \text{Me}$, $R^2 = \text{Ph}$ and $R^1 = \text{Me}$, $R^2 = \text{Ph}$, the formation of isomeric mixtures was expected, the reaction led to the predominant formation of the isomer (**219**) <85S434>.

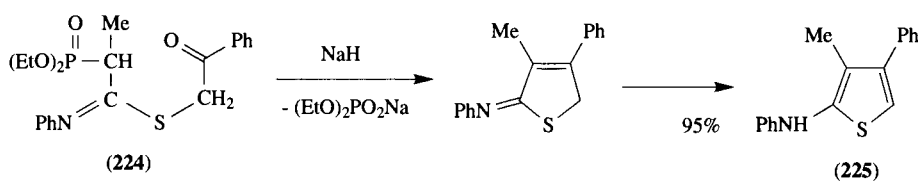


Scheme 51

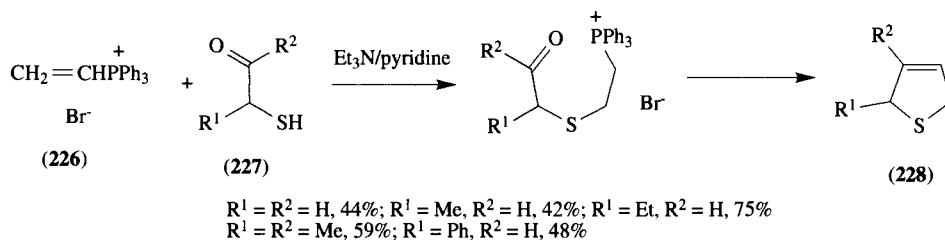
Several reports are concerned with the preparation of benzo[*b*]thiophenes by acid-catalyzed cyclization of 2-(phenylthio)ketones including the improved synthesis of (**221**) and (**223**) from (**220**) and (**222**), respectively <88JHC1271>; see also <92BRP2253847, 93EUP556680>.



Treatment of compound (**224**) with sodium hydride results in the Wittig–Horner reaction leading to thiophene (**225**) (Scheme 52) <85S172>. Addition of the thiols (**227**) to the double bond of the phosphonium bromide (**226**) followed by an intramolecular Wittig reaction affords the dihydrothiophenes (**228**) in reasonable yields (Scheme 53) <83CJC1872>.



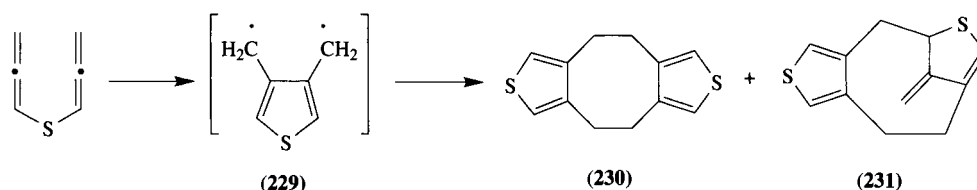
Scheme 52



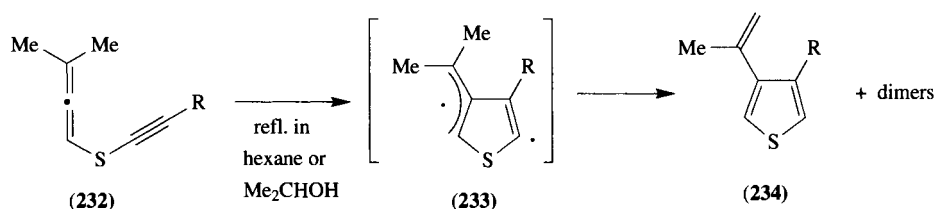
Scheme 53

2.11.2.3.3 Cycloaromatization of diallenyl, allenyl ethynyl, diethynyl, divinyl and related sulfides

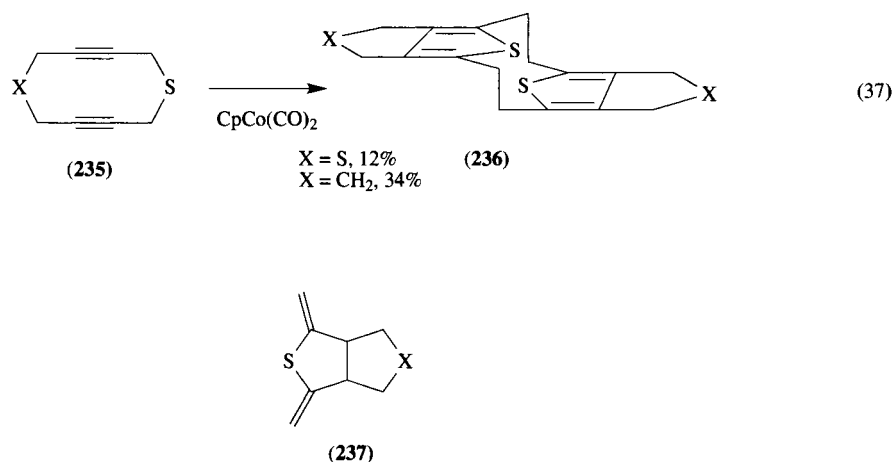
Thermal decomposition of diallenyl sulfide generates the diradical intermediate (229), which can be chemically trapped and affords the dimers (230) and (231) in the ratio 1.4:1 in the absence of trapping agents (Scheme 54) <87TL4263>. On the other hand, heating allenyl ethynyl sulfides (232) in refluxing hexane or isopropanol generates radical intermediates (233), which give thiophenes (234) along with some thiophenic dimers as the final products (Scheme 55) <85IJ108, 90JA5830>. A better yield of thiophenes are attained by thermolysis in isopropanol (R = H, 100%; R = Me, 75%). Treatment of cyclic diethynyl sulfides (235) with dicarbonyl(η^5 -cyclopentadiene)cobalt provides a one-pot synthesis of structurally interesting thiophenes (236), which might be formed via dimerization of intermediates (237) (Equation (37)) <91CB357>. Heating the sulfides (238) in refluxing acetone produces aminothiophenes (240) in one pot (Scheme 56) <83S410>. A mechanism involving thermal isomerization of (238) to (239) followed by ring closure was proposed. Application of the Pd⁰-catalyzed Heck reaction to sulfides (241) results in the formation of benzothiophenes (242) (Equation (38)) <93T11019>. The phenyl vinyl sulfone (243) undergoes photocyclization to give the tetracyclic compound (244) (Equation (39)); application to other similar systems was reported <93TL1855>. Dilithiation of diphenyl sulfone with butyllithium at -60°C and warming the mixture to 45°C affords dibenzothiophene (245) in 70% yield (Equation (40)) <83MI 211-01, 86MI 211-03>. Gas-phase thermolysis of divinyl sulfone at 400°C produces the parent thiophene in 60% yield <83ZOR1221>.

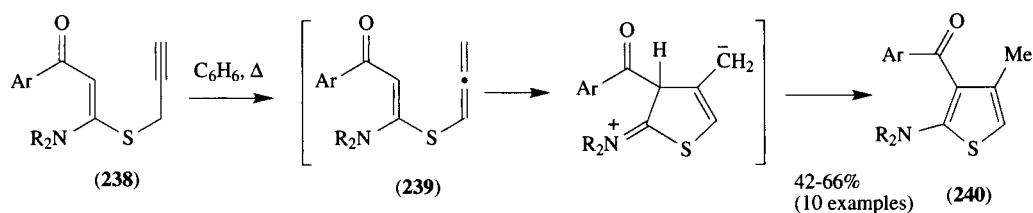


Scheme 54

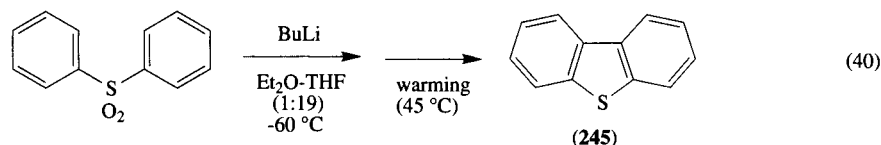
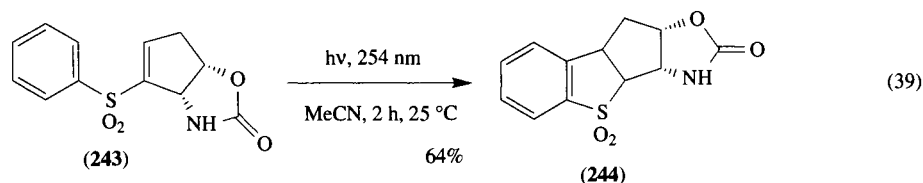
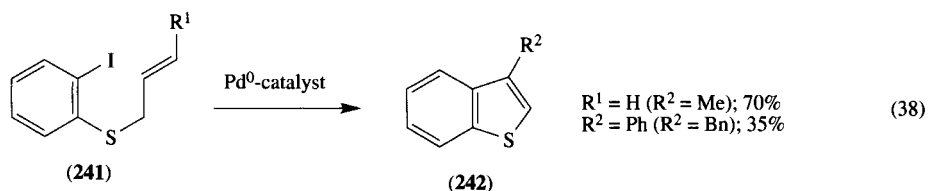


Scheme 55



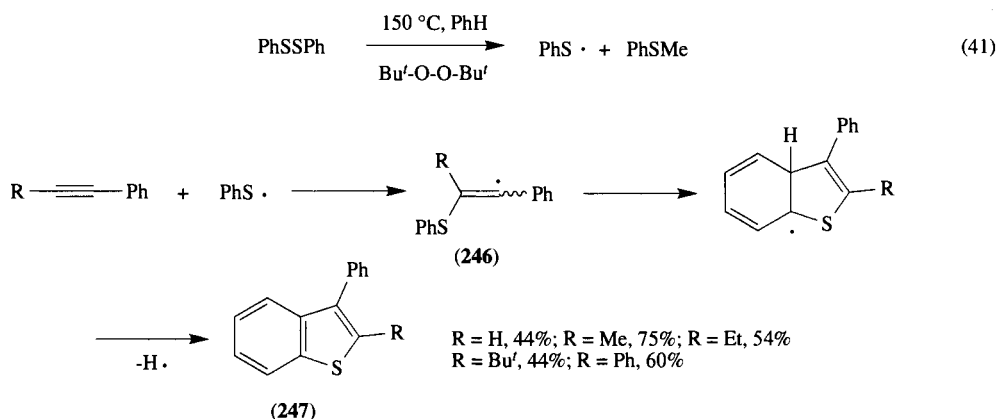


Scheme 56



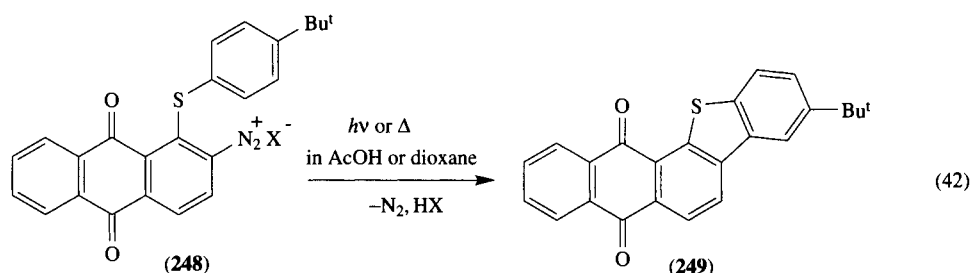
2.11.2.3.4 Cyclization of vinyl radical intermediates

Thermal reaction of diphenyl disulfide with phenylethyne promoted by di-*t*-butyl peroxide at 150 °C provides a useful synthetic route to benzothiophenes (247) (Equation (41) and Scheme 57). The reaction proceeds through intramolecular cyclization of vinyl radical intermediates (246) which result from addition of benzenethiyl radical to phenylethyne (92JCS(P1)1659). A number of benzo [b]thiophene syntheses similar to that described above, but in the gas phase at high temperatures, have been reported by Russian groups; in these cases, benzenethiols, diphenyl disulfide, and alkyl phenyl sulfides are employed as the source of benzenethiyl radicals (89KGS1565, 89ZOR220, 89ZOR1506, 91ZOR359, 92SUL137, 92ZOR751).



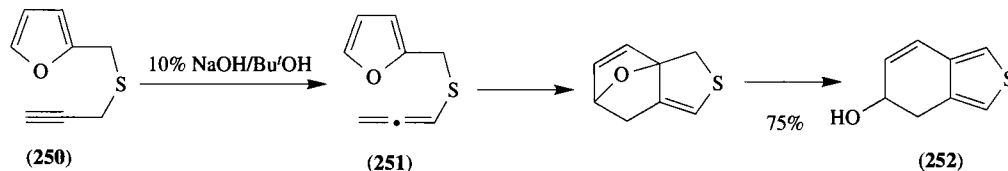
Scheme 57

Diazonium salts such as (**248**) undergo both photochemical and thermal cyclization to afford the thiophene (**249**) and derivatives in 80–90% yields (Equation (42)). The reaction may involve the cyclization of aryl radical intermediates <92IZV582>.

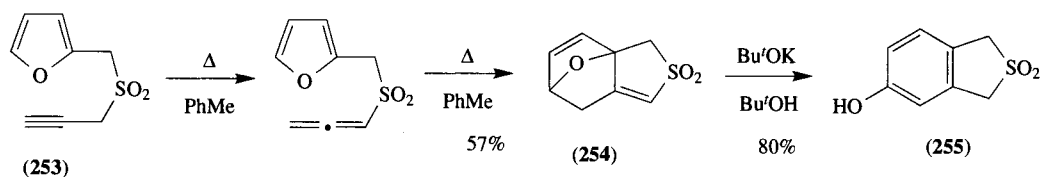


2.11.2.3.5 Intramolecular [4 + 2] cycloaddition

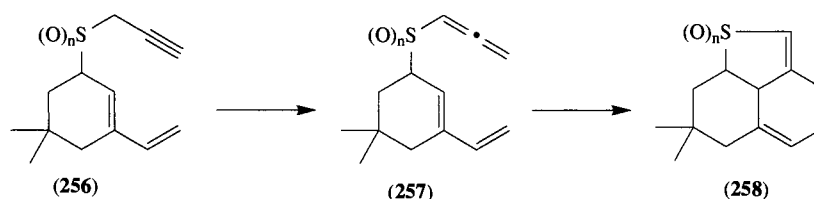
Heating the propargyl sulfide (**250**) with sodium hydroxide in *t*-butanol results in the isomerization to the allene (**251**) which undergoes an intramolecular Diels–Alder reaction to give the thiophene (**252**) via ring-opening of the adduct (Scheme 58) <88AP(321)403>. Meanwhile, heating the propargyl sulfone (**253**) in toluene allows the isolation of the initial adduct (**254**) (Scheme 59). Treatment of (**254**) with the base affords 5-hydroxybenzosulfolene (**255**) via fragmentation and subsequent aromatization <92CC735>. Similarly, the propargyl sulfide and sulfone (**256**) isomerize, on treatment with a base, to the corresponding allenyl compounds (**257**), which immediately undergo an intramolecular [4 + 2] addition to give the dihydrothiophene derivatives (**258**) (Scheme 60) <94JOC1621>. This strategy was successfully applied to the preparation of a large number of tricyclic dihydrothiophene derivatives.



Scheme 58



Scheme 59

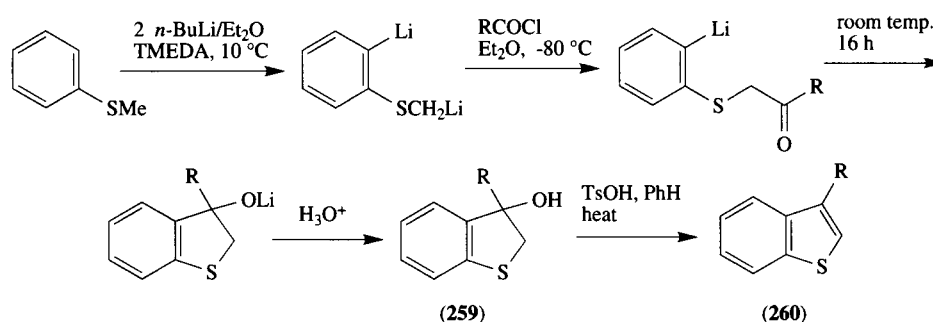


$n = 0$; aqueous NaOH, Bu^tOH, 83 °C, 1 h, 72% yield
 $n = 2$; alumina, PhH, 80 °C, 1 h, 47% yield

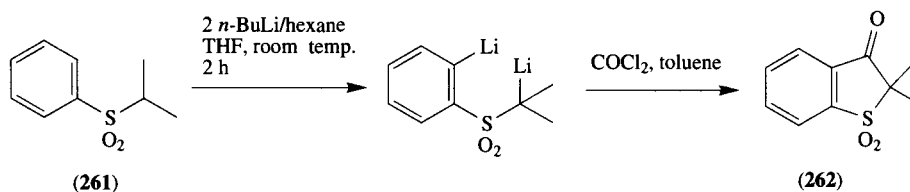
Scheme 60

2.11.2.3.6 Miscellaneous cyclization

Treatment of the dilithiated thioanisole with acyl halides at -80°C and then warming to room temperature affords the carbinols (**259**), which are dehydrated to give thiophenes (**260**) by treatment with *p*-toluenesulfonic acid (Scheme 61) <88S888>. Dilithiation of the sulfone (**261**) followed by reaction with phosgene produces the dihydrothiophene derivative (**262**) (Scheme 62) <93S41>. Preparation of aminothiophenes by heating *N*-arylcyanothioacetamides with Et_3N <84ZOR118> and by condensation of malononitrile with thiolketones <83JAP(K)58192879> were reported.



Scheme 61



Scheme 62

2.11.3 RING SYNTHESIS BY FORMATION OF TWO BONDS

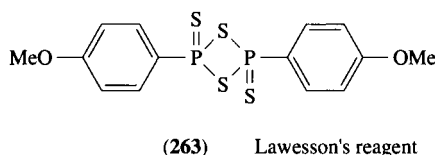
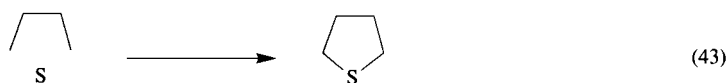
Thiophene rings might be constructed from the following sets of two components with formation of two new bonds. (a) $\text{C}-\text{C}-\text{C}-\text{C} + \text{S}$; (b) $\text{C}-\text{C}-\text{C}-\text{S} + \text{C}$; (c) $\text{C}-\text{C}-\text{S} + \text{C}-\text{C}$; (d) $\text{C}-\text{C}-\text{C} + \text{C}-\text{S}$; (e) $\text{C}-\text{S}-\text{C} + \text{C}-\text{C}$.

Among these combinations, the synthetic methods that belong to classes (a), (b), (c), (d), and (e) are available, but no examples of (d) are known according to the present classification.

2.11.3.1 Sulfuration of a Four-carbon Unit with a Sulfur Reagent

Reaction of a four-carbon unit with sulfur sources such as hydrogen sulfide, carbon disulfide, and elemental sulfur is one of the traditional thiophene syntheses that belongs to this category. A wide variety of hydrocarbons, e.g., alkanes, alkenes, dienes, alkynes, and diynes, serve as four-carbon units. Another practical method is the sulfuration of 1,4-dicarbonyl compounds (Paal synthesis). The method has become very popular with development of new sulfuration reagents such as Lawesson's reagent (**263**). The reaction of α,β -unsaturated nitriles with elemental sulfur in

basic media, Gewald synthesis, is also useful for the preparation of 2-aminothiophenes which are important compounds in dyestuff and pharmaceutical industries.

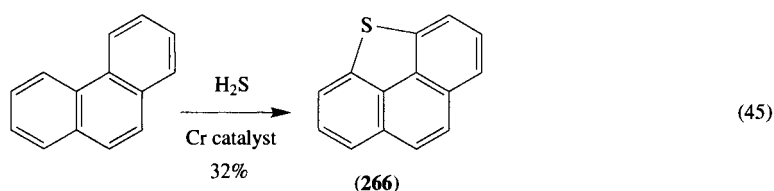
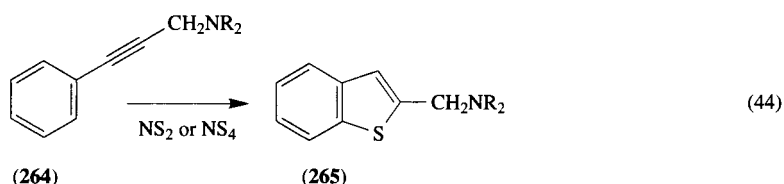


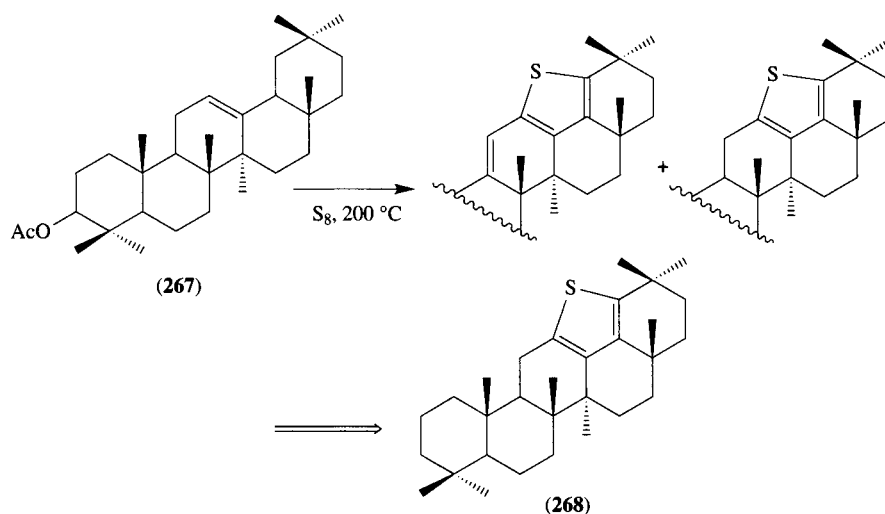
2.11.3.1.1 Sulfuration of alkanes, alkenes, and alkynes

The Socony Vacuum process, which uses butane and sulfur at elevated temperatures, and the Croda synthetic process, which employs functionalized hydrocarbons and carbon disulfide, provide important industrial routes to thiophenes. A review from 1994 covers the catalytic synthesis of thiophene and alkylthiophenes from ethyne, C_4 to C_6 alkanes, alkenes, alkadienes, C_7 , C_8 alkanes and hydrogen sulfide via heterocyclization $\langle 94RHA23 \rangle$. For example, the reaction of hydrogen sulfide with ethyne-containing electrocracking gas on $\gamma\text{-Al}_2\text{O}_3$ at 550°C may be of practical interest for obtaining thiophene. With a catalyst consisting of Cr_2O_3 , polirit, K_2O , and $\gamma\text{-Al}_2\text{O}_3$ in the ratio of 5 : 5 : 1 : 89, 2-methylthiophene is obtainable in 68% yield from 2-piperylene and hydrogen sulfide and 3-methylthiophene in 75% yield from isopentane and hydrogen sulfide.

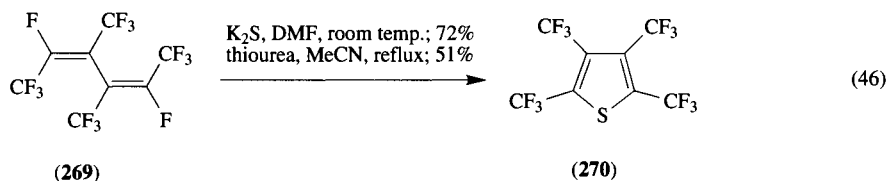
A large number of papers and patents describe the preparation of benzo[*b*]thiophene and its simple derivatives at elevated temperature reactions. They include reactions of PhEt , PhCH=CH_2 , $\text{PhCH}_2\text{CH}_2\text{OH}$, PhCHMe_2 , or $\text{PhCH}_2\text{CH}_2\text{Me}$ with carbon disulfide, reactions of PhEt , PhCH=CH_2 , or PhCH=CHCl with hydrogen sulfide, and reaction of phenylethyne with dimethyl sulfide $\langle 84ZOR1591, 86MI\ 211-02, 87ZOR2016, 89G627 \rangle$.

An interesting synthesis of benzothiophenes (**265**) by sulfuration of (**264**) with SN_2 or SN_4 has been reported (Equation (44)) $\langle 90KGS1331 \rangle$. The pre-1980 literature on the syntheses of various tetracyclic and pentacyclic thiophenes has been reviewed $\langle 82AHC(32)127 \rangle$. For the preparation of these compounds, direct insertion of a sulfur atom from the inorganic sources into an organic precursor compound is one of the important methods. The preparation of the thiophene (**266**) by reaction of phenanthrene with hydrogen sulfide in a vapor phase on a Cr catalyst (Equation (45)) has been described $\langle 87JHC357, 88JHC1111 \rangle$. Structure of the thiophene (**268**), derived from higher plant triterpenoids of the olenane series and detected in sediments deposited in evaporitic environments, was identified by chemical transformation (**267**) $\rightarrow$ (**268**) (Scheme 63) $\langle 91TL4179 \rangle$. A new synthesis of tetrakis(trifluoromethyl)thiophene (**270**) from the easily accessible diene (**269**) was developed (Equation (46)) $\langle 90CC1127, 94JCS(P1)3119 \rangle$.

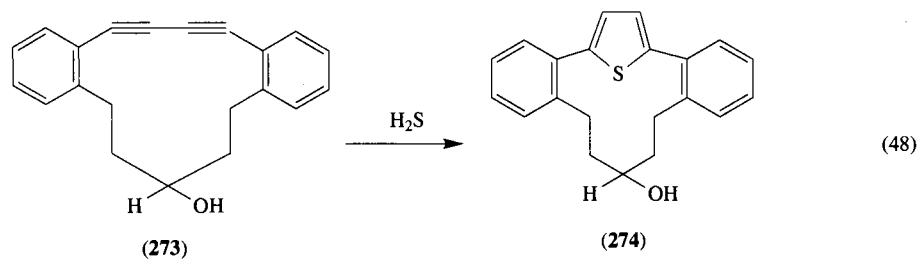
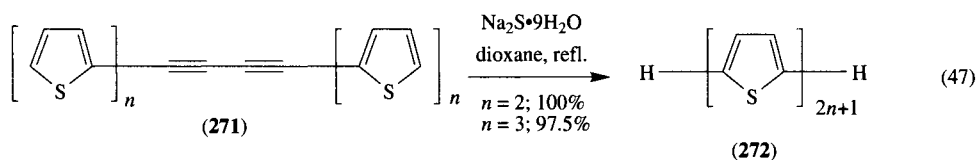




Scheme 63

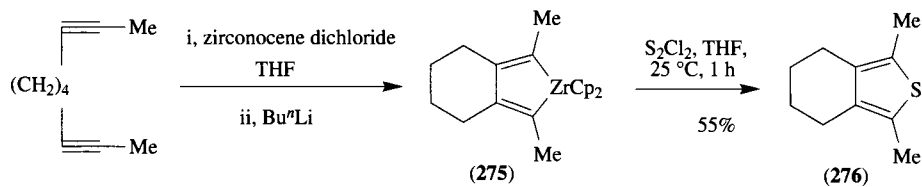


Addition of H_2S , $NaHS$, Na_2S , etc. to conjugated diynes provides a convenient route to a range of structurally interesting thiophenes and has experienced wide applicability [83H(20)1341, 83JOC4317, 86H(24)365, 86JCR(S)380, 87JCS(P1)2321, 93ZOR165]. Preparation of quater- and septithiophenes (272) by addition of Na_2S to diynes (271) (Equation (47)) [83JOC4317] and preparation of the cyclophane (274) by addition of H_2S to the cyclic diyne (273) (Equation (48)) [87JCS(P1)2321] are the representative examples.

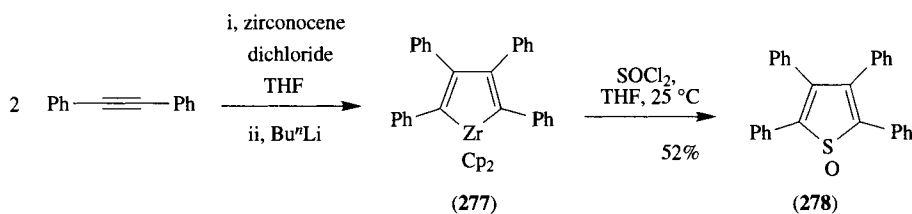


Zirconium metallacycles were found to serve as the precursor compound for the preparation of a variety of main group heterocycles including thiophenes and their *S*-oxides. The zirconium metallacycles (275) and (277) were prepared by treatment of 2,8-decadiyne and diphenylethyne with zirconocene dichloride in the presence of Bu^iLi and allowed to react with disulfur dichloride and thionyl chloride, respectively, to give the bicyclic thiophene (276) and the thiophene *S*-oxide (278)

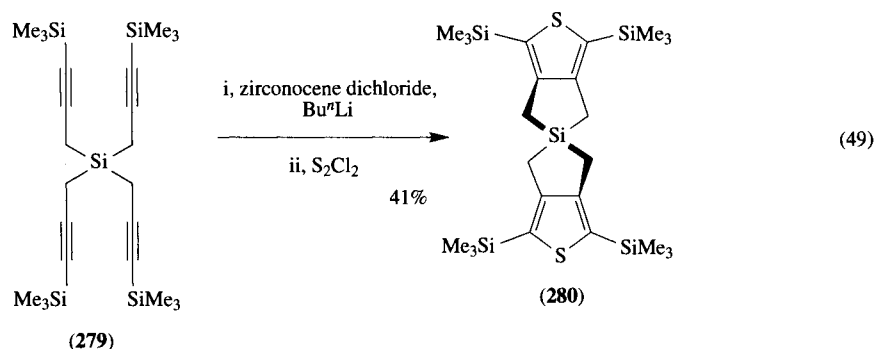
in one pot (Schemes 64 and 65) <88JA2310, 94JA1880>. A clever application of this procedure to **(279)** allows the preparation of the spiro compound **(280)**, which serves as the precursor compound of the thiophene-containing molecular devices (Equation (49)) <90JA5662>.



Scheme 64



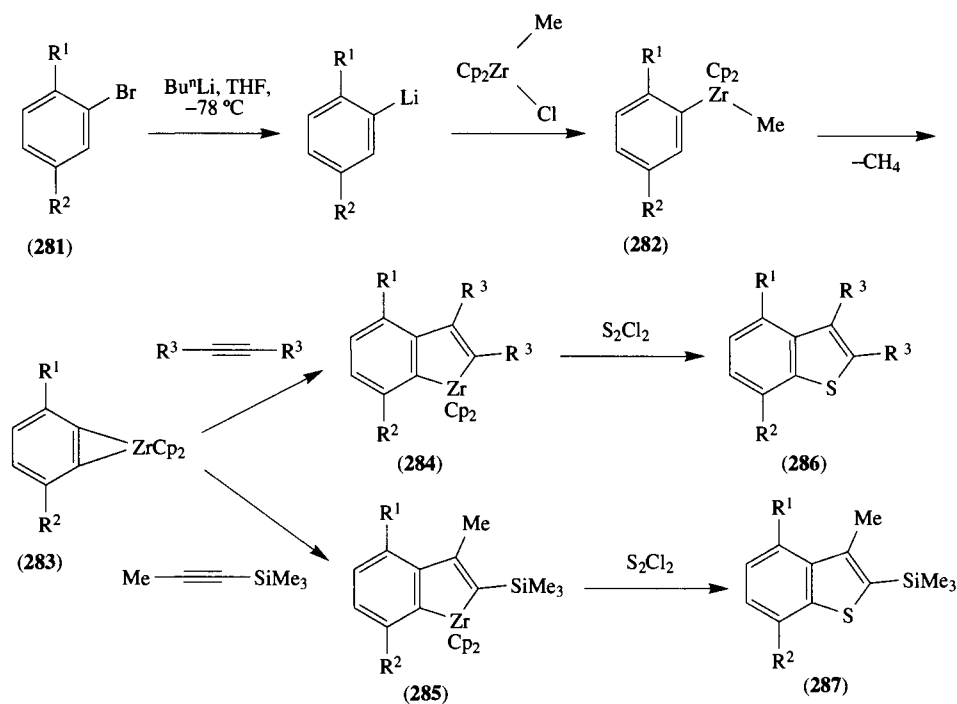
Scheme 65



Lithiation of commercially available bromoarenes **(281)** followed by treatment with zirconocene (methyl) chloride affords zirconocene complexes of substituted benzyne **(283)**, which react with symmetric alkynes and 1-(trimethylsilyl)propyne to give single regioisomeric zirconacycles, **(284)** and **(285)**, respectively (Scheme 66). Both **(284)** and **(285)** react with disulfur dichloride to produce benzothiophenes **(286)** and **(287)**, respectively, in 60–80% isolated yields in a one-pot procedure <89JOC2793>. Protodesilylation of **(287)** can be accomplished in >90% yield to give the corresponding 2-unsubstituted benzothiophenes by treatment with tetrabutylammonium fluoride in tetrahydrofuran. Application of the procedure to the preparation of 2,3-dihydrobenzo[*b*]thiophenes was also reported <91OM537>.

2.11.3.1.2 Gewald synthesis

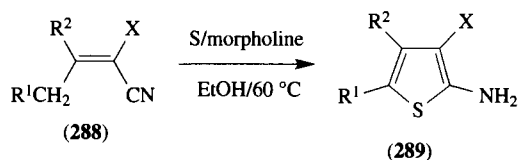
The reaction of α,β -unsaturated nitriles **(288)** with elemental sulfur in basic media, Gewald synthesis, provides a most convenient route to 2-aminothiophenes **(289)**, many of which are a useful class of compounds as intermediates in the preparation of dyestuffs and pharmaceuticals. Thus, a number of 2-aminothiophenes such as **(290)**–**(295)** is still a matter of extensive synthetic study by the Gewald method (see also <88KGS1045> and <89KFZ840>: **(290)** <83MI 211-02, 86MI 211-02>; **(291)** <83ZC179>; **(292)** <86KFZ39>; **(293)** <86JHC129>; **(294)** <91MI 211-02>; **(295)** <91JOC7179>).



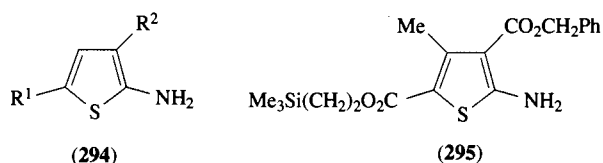
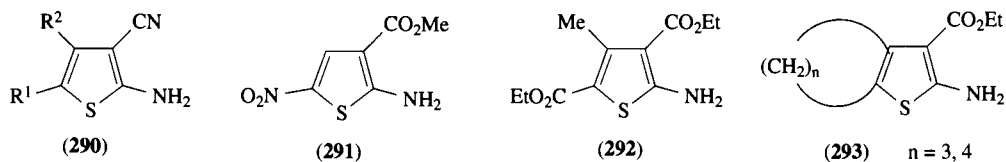
yields of benzothiophenes (286)
 $\text{R}^1 = \text{H}, \text{R}^2 = \text{Me}, \text{R}^3 = \text{Me}; 72\%$
 $\text{R}^1 = \text{H}, \text{R}^2 = \text{Me}, \text{R}^3 = \text{Et}; 71\%$
 $\text{R}^1 = \text{H}, \text{R}^2 = \text{OMe}, \text{R}^3 = \text{Me}; 71\%$
 $\text{R}^1 = \text{H}, \text{R}^2 = \text{OMe}, \text{R}^3 = \text{Et}; 80\%$
 $\text{R}^1 = \text{R}^2 = \text{Me}, \text{R}^3 = \text{Me}; 78\%$
 $\text{R}^1 = \text{R}^2 = \text{Me}, \text{R}^3 = \text{Et}; 67\%$
 $\text{R}^1 = \text{R}^2 = \text{OMe}, \text{R}^3 = \text{Me}; 60\%$
 $\text{R}^1 = \text{R}^2 = \text{OMe}, \text{R}^3 = \text{Et}; 74\%$

yields of benzothiophenes (287)
 $\text{R}^1 = \text{H}, \text{R}^2 = \text{Me}; 69\%$
 $\text{R}^1 = \text{H}, \text{R}^2 = \text{OMe}; 71\%$
 $\text{R}^1 = \text{R}^2 = \text{Me}; 75\%$
 $\text{R}^1 = \text{R}^2 = \text{OMe}; 69\%$

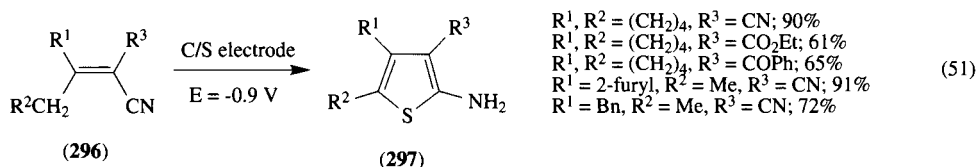
Scheme 66



(50)



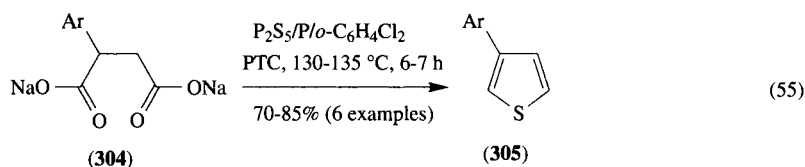
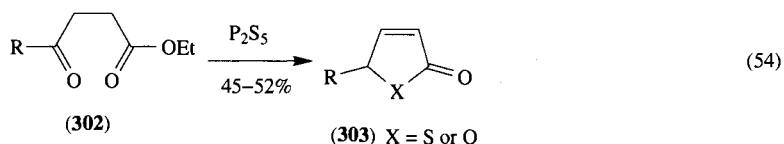
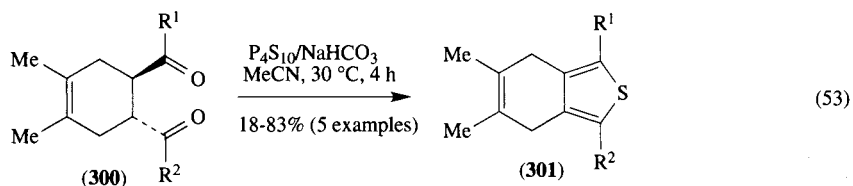
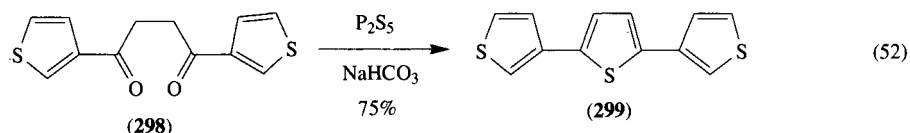
Electrochemical Gewald synthesis has been developed. Thus, when a mixture of powdered graphite and sulfur is used as the cathode at a working potential of about -0.9 V in DMF, the nucleophilic S_x^{2-} is generated and reacts with α,β -unsaturated nitriles (**296**) to give 2-aminothiophenes (**297**); yields of the products are sometimes better than those of the Gewald synthesis (Equation (51)) (<92SR405>).

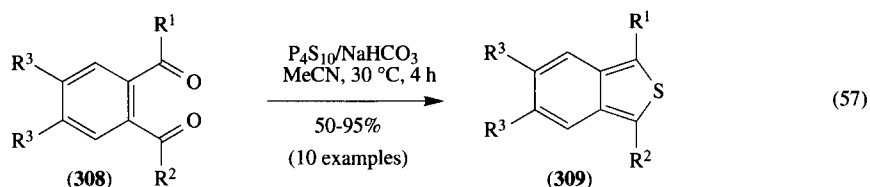
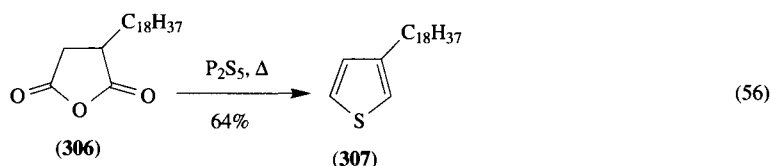


2.11.3.1.3 Sulfuration of 1,4-dicarbonyl and related compounds (Paal synthesis)

Sulfuration of 1,4-dicarbonyl and related compounds (Paal synthesis) provides one of the most widely used methods for the preparation of thiophenes. P_2S_5 (P_4S_{10}), combination of hydrogen sulfide and an acid catalyst, Lawesson's reagent (**263**), and bis(trialkyltin) sulfides are the sulfuration reagents used most commonly.

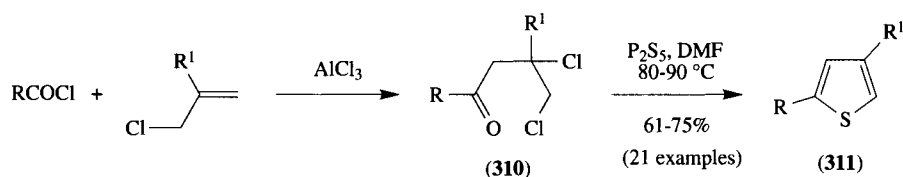
Transformations (**298**) $\rightarrow$ (**299**) (Equation (52)) (<85SC789> and (**300**) $\rightarrow$ (**301**) (Equation (53)) (<90S670>) illustrate the representative examples of the conversion of 1,4-diketones to thiophenes with P_2S_5 (P_4S_{10}) (see also <92AP(325)9>). When one carbonyl group is an ester carbonyl, the reaction leads to 2-thiophenones (contaminated with furanones) as exemplified in the conversion of (**302**) to (**303**) (Equation (54)) (<87KGS451>). Treatment of 1,4-dicarboxylic acid sodium salts (**304**) with P_2S_5 and phosphorus under rather forcing conditions satisfactorily provides thiophenes (**305**) (Equation (55)) (<87S1024, 89SC2809>). Even an acid anhydride (**306**) can be converted to the thiophene (**307**) (Equation (56)) (<92MIP14724>). Treatment of *o*-diaroyl- and *o*-bis(trimethylacetyl)-benzenes (**308**) with P_2S_5 in the presence of NaHCO_3 affords benzo[*c*]thiophenes (**309**) in good yields (Equation (57)), thus providing a very practical method for the preparation of benzo[*c*]thiophenes which are stabilized by aryl- or *t*-butyl groups on the 1- and 3-positions (<90S670, 90PS(54)209, see also 93CC172>).



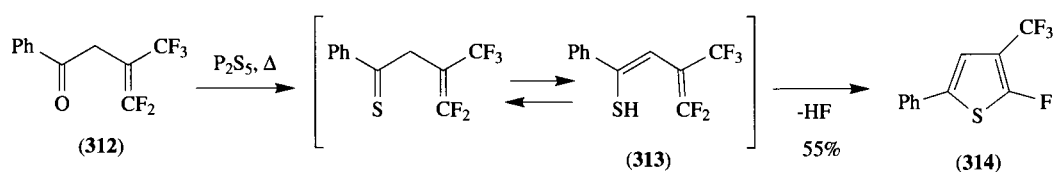


R^1, R^2 = mono-, di-, and tri-substituted phenyl, thienyl, or Bu^t; R^3 = H or Me

Compounds (310), obtainable from acyl chlorides and ally chlorides, were treated with P_2S_5 without purification to give thiophenes (311) (Scheme 67) <91SUL109>. In this case, compounds (310) are initially converted to the corresponding thioketones whose enethiolization, followed by intramolecular substitution and loss of HCl, leads to the thiophenes (311). In a similar way, the ketone (312) is converted to the thiophene (314) by treatment with P_2S_5 (Scheme 68) <94JFC(66)13>. The intermediate enethiol (313) undergoes an intramolecular addition to the double bond (these two examples may be classed as the ring synthesis by formation of a bond adjacent to the sulfur).

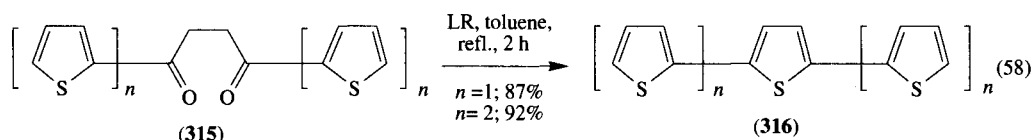


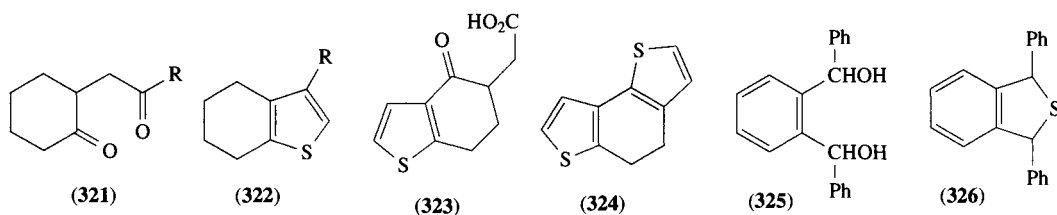
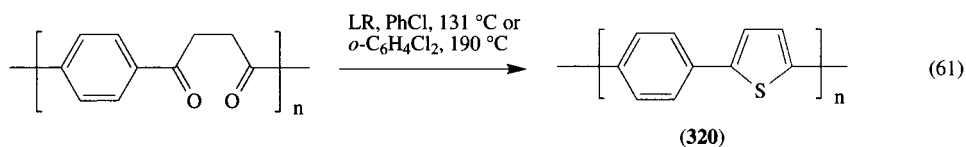
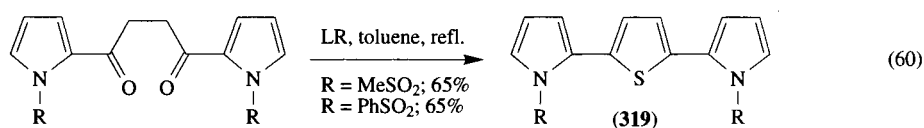
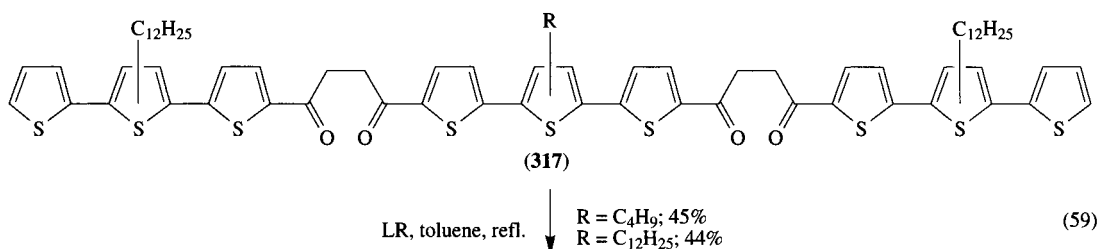
Scheme 67



Scheme 68

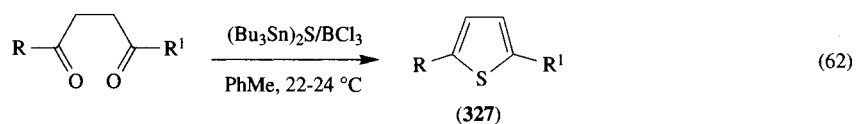
The use of Lawesson's reagent (263) as the sulfurating agent has become very popular in the last decade and a large number of interesting thiophene syntheses by this reagent have been developed since the first report in 1982 <82S1061, 83IJC(B)1187>. For example, treatment of diketones (315) with the reagent affords α -ter- and α -quinque-thiophenes (316) in high yields (Equation (58)) <84SCI, 91S462, 92MI 211-03> and that of (317) <91JA5887> produces undecathiophenes (318) which are the largest oligothiophenes of an α -type ever synthesized (Equation (59)). Application to the preparation of pyrrole-thiophene mixed oligomers such as (319) is also successful (Equation (60)) <90JOC2904, 92JOC4414>. Even preparation of polymers such as (320) could be attained (Equation (61)) <88CC1432>. Also reported are preparations of annelated thiophenes (322) from (321) <88JHC1437>, tricyclic thiophene (324) from the keto carboxylic acid (331) <86M621>, and dihydrothiophene (326) from (325) <93JCS(P)1113>.



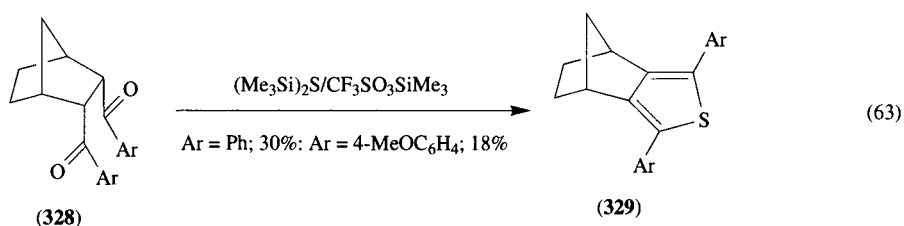


A few papers report the preparation of thiophenes by sulfuration of dicarbonyl compounds with a combination of hydrogen sulfide and an acid catalyst $\langle 83H(20)1941, 91MI 211-02, 92H(34)2263 \rangle$.

It was found that sulfuration of 1,4-dicarbonyl compounds with bis(trialkyltin) and bis(triphenyltin) sulfides in the presence of boron trichloride provides a new thiophene synthesis $\langle 92JOC1722 \rangle$. Particularly, sulfuration with bis(tributyltin) sulfide proceeds at room temperature to give the corresponding thiophenes (327) in high yields (Equation (62)). Trifluoromethyl trifluoromethanesulfonate-promoted bis(trimethylsilyl) sulfide sulfuration also converts 1,4-dicarbonyl compounds (328) to the thiophenes (329) in moderate yields (Equation (63)) $\langle 94JOC3695 \rangle$.

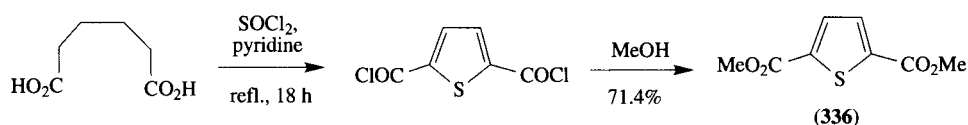
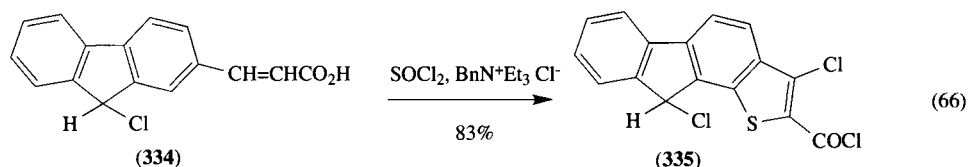
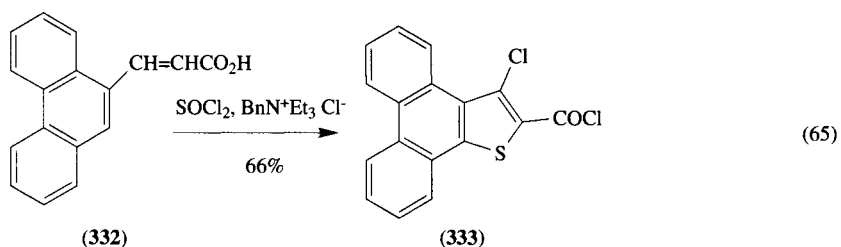
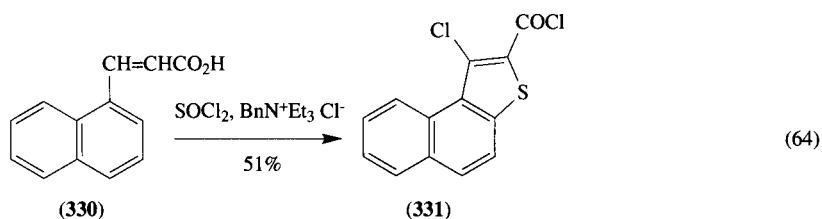


$R = R^1 = Ph$; 98%; $R = R^1 = 4-ClC_6H_4$; 92%; $R = Me, R^1 = Ph$; 95%;
 $R = Me, R^1 = 4-MeOC_6H_4$; 94%; $R = MeC\equiv C, R^1 = PhC\equiv C$; 15%;
 $R = PhC\equiv C, R^1 = PhC\equiv C$; 84%



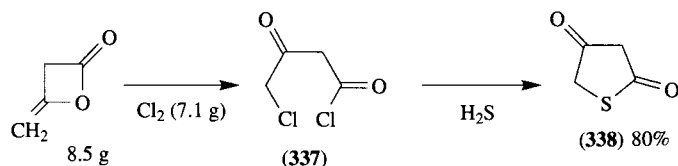
2.11.3.1.4 Miscellaneous cyclizations

Preparation of benzo[*b*]thiophene derivatives by treatment of cinnamic acids with thionyl chloride in the presence of pyridine is one of the well-documented procedures. Several papers describe the preparation of thiophenes (331), (333), and (335) from (330), (332), and (334), respectively, by application of this procedure (Equations (64)–(66)); in these cases, quaternary ammonium chloride was used instead of pyridine (83KGS192, 83KGS197, 88KGS33). Prolonged heating of adipic acid with thionyl chloride in pyridine followed by methanol workup provides the thiophene (336) directly (Scheme 69) (92MI 211-01).



Scheme 69

Reaction of hydrogen sulfide with the acyl chloride (337), obtainable by treatment of diketene with chlorine, affords thiotetronic acid (338) (Scheme 70) (85EUP189097).

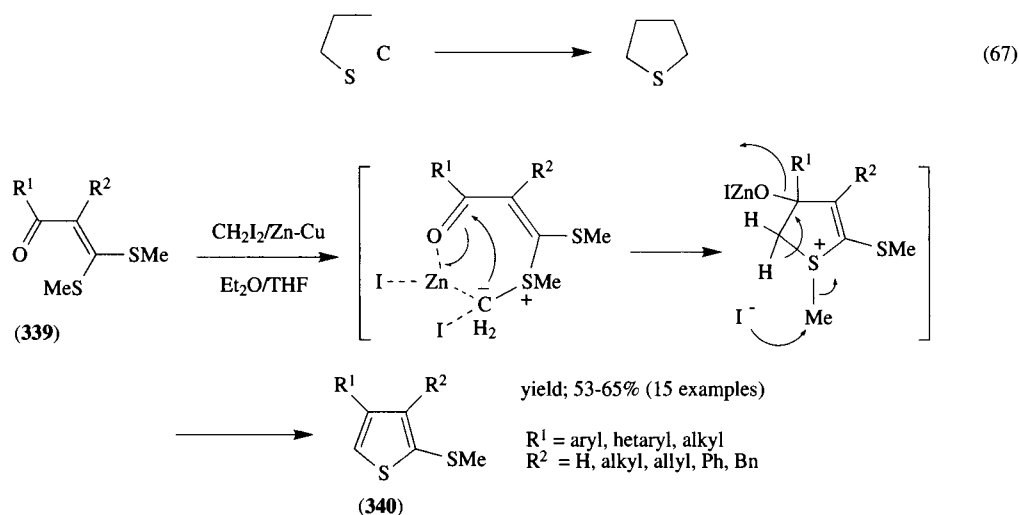


Scheme 70

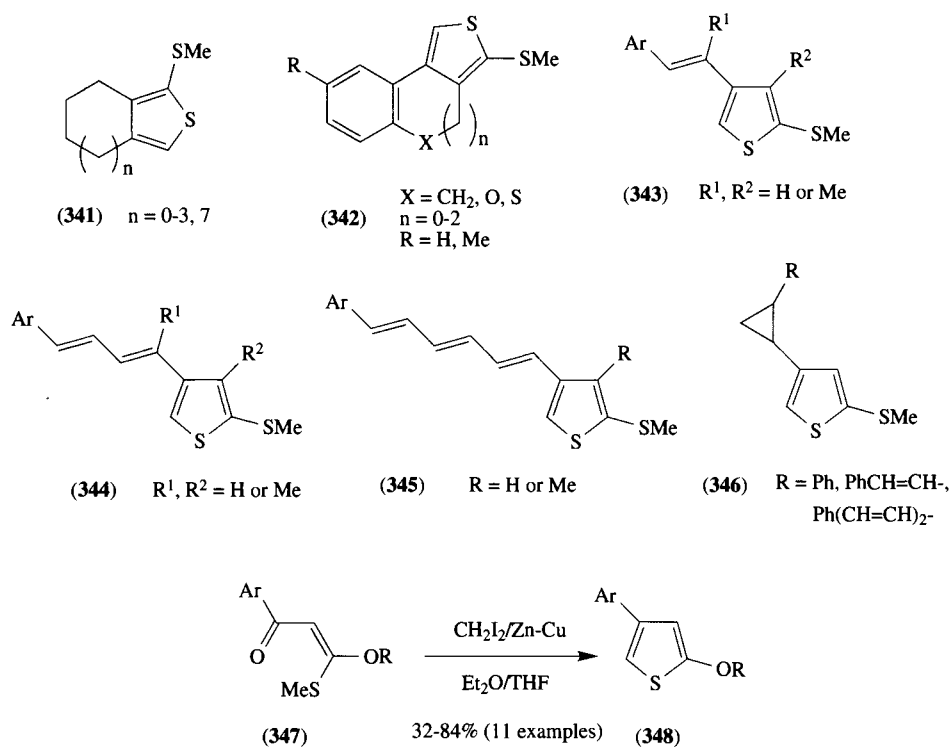
2.11.3.2 Combination of a C—C—C—S Unit with a One-carbon Unit

Only two examples that belong to this category are known. Incidentally, no report has appeared on the thiophene synthesis by combination of a C—C—S—C unit with a one-carbon unit. The

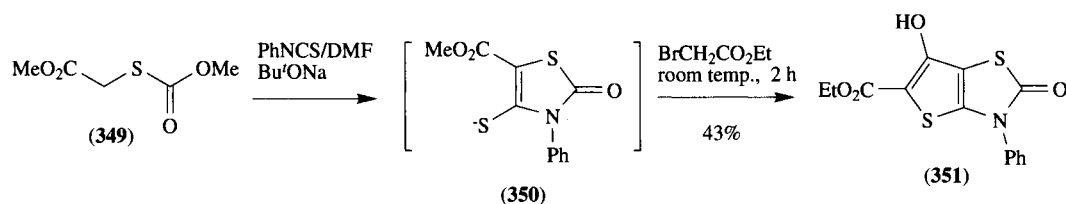
Simmons–Smith reaction on α -oxoketene dithioacetals provides a new general route to 3,4-disubstituted and annelated thiophenes. Thus, addition of an ether solution of α -oxoketene dithioacetals (**339**) to a Simmons–Smith reagent, prepared from a zinc–copper couple, methylene iodide, and a small amount of iodine in ether, and refluxing of the resulting mixture for 8–12 h affords the thiophenes (**340**) in good yields in one pot (Scheme 71) (89TL3093, 92T10377). The new method is versatile and thus satisfactorily applicable to the preparation of a wide variety of thiophenes such as (**341**)–(**346**) in good yields. Application to the *O,S*-acetals (**347**) is also satisfactory, thus providing a new route to 2-alkoxythiophenes (**348**) (Equation (68)) (93S959).



Scheme 71

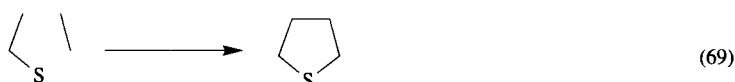


Phenyl isothiocyanate reacts with the thiocarbamate (**349**) in the presence of the strong base to give the adduct (**350**), which reacts with ethyl bromoacetate to yield the thiophene (**351**) in a one-pot procedure (Scheme 72) (90S621).

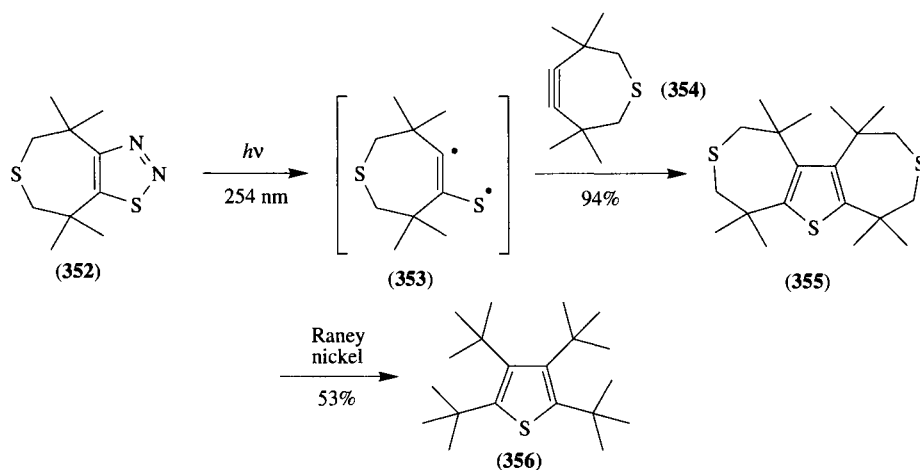


Scheme 72

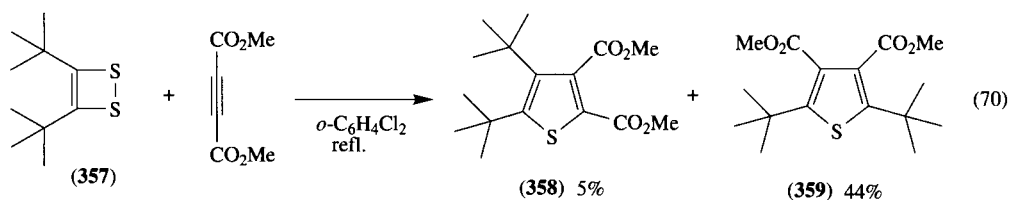
2.11.3.3 Combination of a C—C—S Unit with a C—C Unit

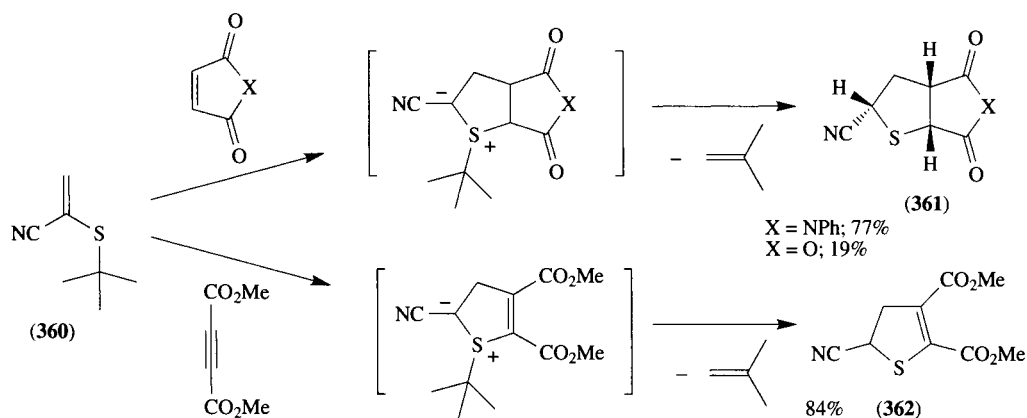


Ring synthesis by a [2 + 3] type cycloaddition belongs to this class. A diradical intermediate (353), generated by photolysis of the thiazole (352), is trapped by the strained alkyne (354) to give the thiophene (355) in a high yield. Selective desulfurization of (355) provides the highly congested tetra-*t*-butylthiophene (356), whose five-membered ring is no longer planar (Scheme 73) <92TL5947>. The reaction of the dithiete (357) with DMAD affords a mixture of thiophenes (358) and (359) (Equation (70)). Mechanism of the reaction is not clear, but respective C—C—S and C—S—C units of the thiophene ring of (358) and (359) apparently originate from (357) <90BCJ1026>. The first synthesis of 2,3-di-*t*-butylthiophene was attained by chemical transformation of (358). The *t*-butyl vinyl sulfide (360) undergoes a 1,3-dipolar cycloaddition with *N*-phenylmaleimide (maleic anhydride) and DMAD to give sulfur ylide intermediates, which collapse with elimination of isobutylene to give tetrahydrothiophenes (361) and dihydrothiophene (362) (Scheme 74) <83TL885>. The pyridinium ylide intermediate (364), generated by treatment of the salt (363) with triethylamine, reacts with arylmethylenecyanothioacetamides stereoselectively to give dihydrothiophenes (365) in one pot (Scheme 75) <91S277>. The photostimulated reaction of 2-naphthalenethiolate with *o*-diiodobenzene affords the thiophene (366) (Equation (71)) <93JOC2593>. The reaction with *o*-bromiodobenzene gives (366) in a decreased yield along with the substitution product on the sulfur with retention of bromine.

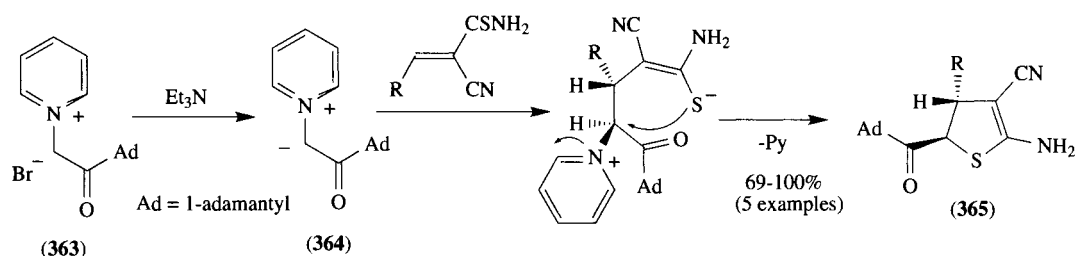


Scheme 73

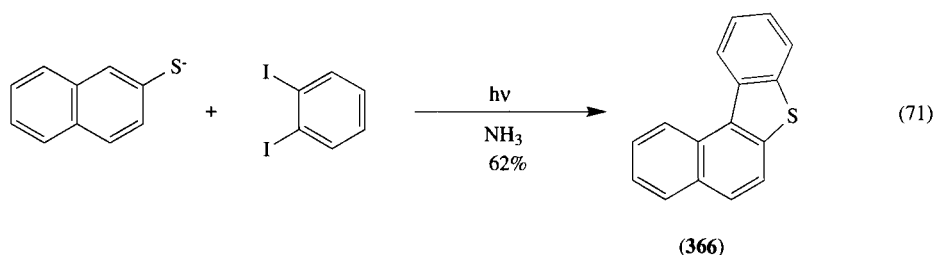




Scheme 74

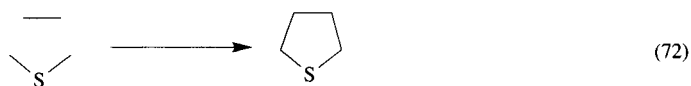


Scheme 75



2.11.3.4 Combination of a C—S—C Unit with a C—C Unit

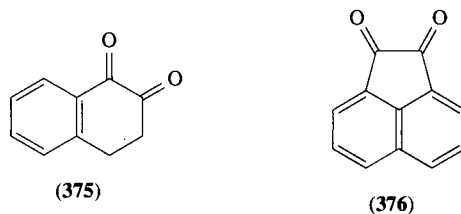
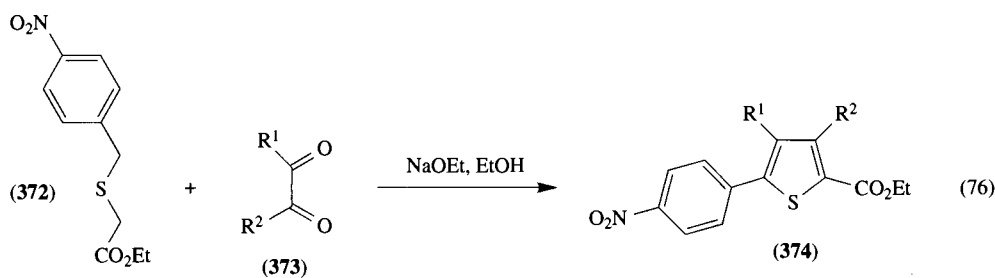
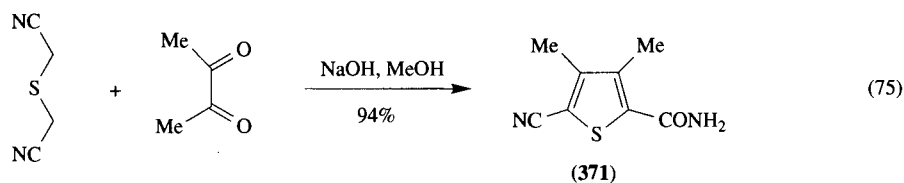
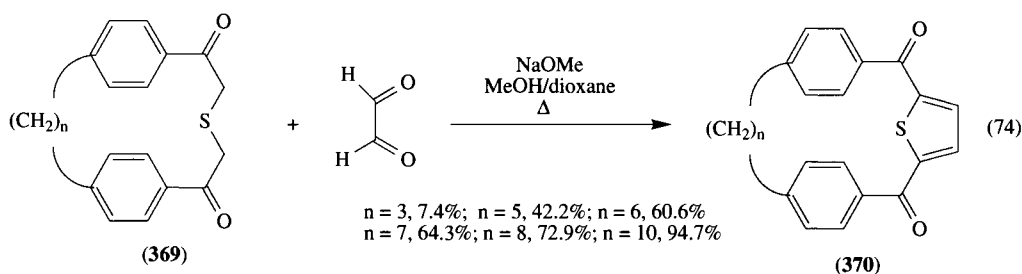
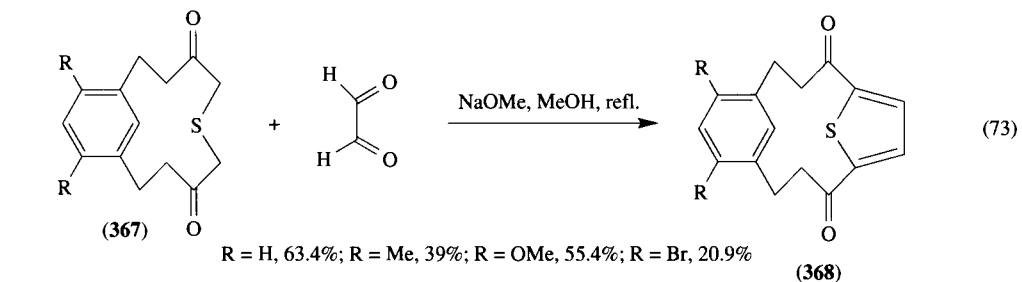
Hinsberg synthesis and 1,3-dipolar cycloaddition of thiocarbonyl ylides with dipolarophiles are typical thiophene syntheses of this class.



2.11.3.4.1 Hinsberg synthesis

Various α -dicarbonyl compounds condense with thioglycolic acid esters in the presence of sodium alkoxide to give thiophene-2,5-dicarboxylic acid derivatives (Hinsberg thiophene synthesis). A series of diketone sulfides (thia-1,5-pentanediones) also condense with glyoxal under mild conditions, thus providing a practical synthesis of 2,5-diacyl- and 2,5-diaroylthiophenes in excellent yields <79JHC1147>. Application of the reaction to the cyclic diones (367) allows the preparation of [3.3]metacyclo(2,5)thiophenophanes (368) in reasonable yields (Equation (73)) <84TL415>. Further application is found in the preparation of [n.1.1]*p*-cyclo(2,5)thiopheno-*p*-cyclophane derivatives (370) from cyclic diones (369) (Equation (74)) <84JOC1177>. The yield of (370) is generally good

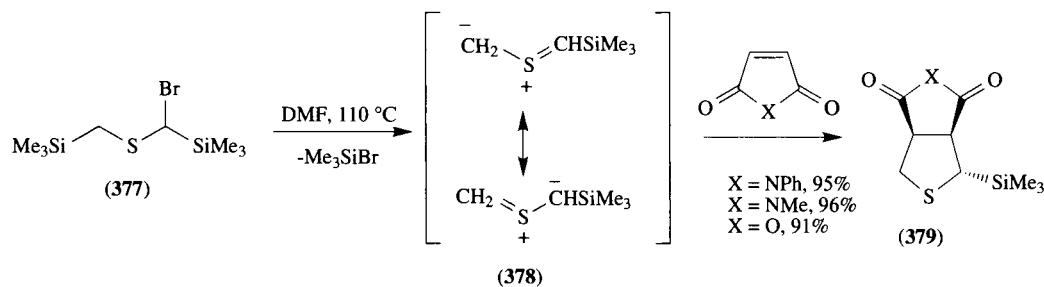
except that a sharp drop off occurs at $n = 3$, where the large strain and high rigidity of the conformation of the starting material and the product are expected. Bis(cyanomethyl) sulfide condenses with biacetyl to furnish the thiophene (371) (Equation (75)) <94JOC2223>. Compound (372), one of whose methylene groups is activated by *p*-nitrophenyl substituent, also condenses with α -dicarbonyl compounds (373) to give thiophenes (374) in good yields (Equation (76)) <91JHC1455>. Cyclic α -dicarbonyl compounds such as (375) and (376) also condense with (372) satisfactorily.



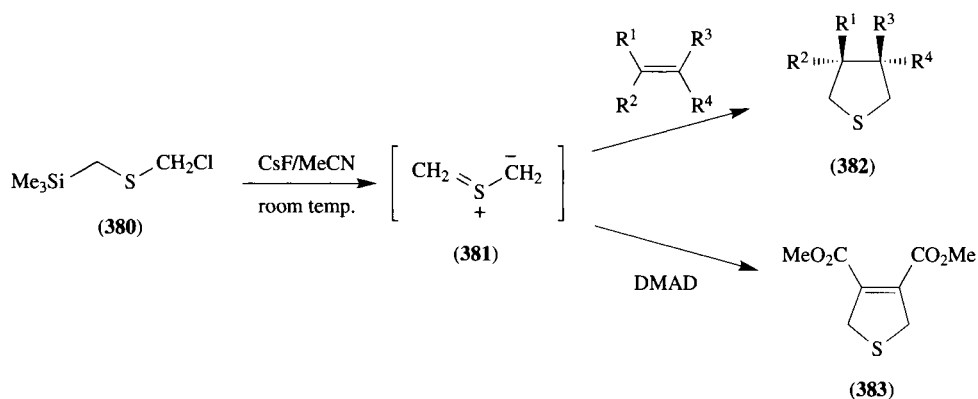
2.11.3.4.2 1,3-Dipolar cycloaddition

The thiocarbonyl ylide intermediate (378), generated by thermal extrusion of trimethylsilyl bromide from (377), undergoes 1,3-dipolar cycloaddition with *N*-phenyl- and *N*-methyl-maleimides

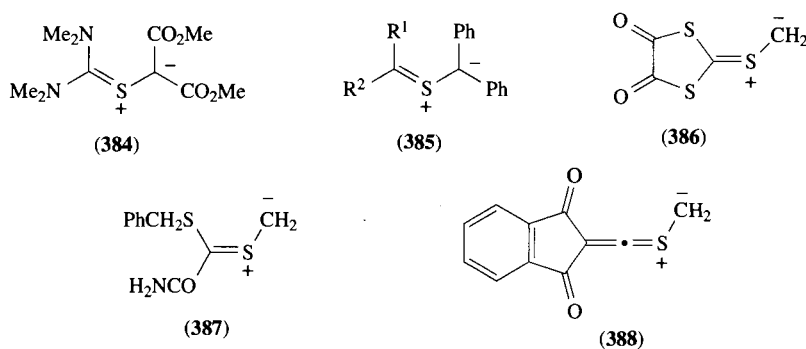
and maleic anhydride to give tetrahydrothiophene derivatives (**379**) stereoselectively (Scheme 76) <85TL3011>. The 1,3-dipolar cycloaddition of (**378**) with dimethyl fumarate and fumaronitrile gives a mixture of two possible stereoisomers and that with dimethyl maleate a mixture of four possible isomers. The parent thiocarbonyl ylide (**381**), generated by caesium fluoride-induced desilylation of (**380**), is trapped with alkenes bearing electron-withdrawing substituents and with DMAD to give tetrahydrothiophenes (**382**) (58–86%) and dihydrothiophene (**383**) (56%), respectively (Scheme 77) <86CC1073>. The reaction with alkenes is stereospecific and the original stereochemistry of the alkenes is retained. Thiocarbonyl ylides such as (**384**) <86SUL223>, (**385**) <89CC557>, (**386**) and (**387**) <89JOC3664>, and (**388**) <94TL3555> were generated and trapped by 1,3-dipolar cycloadditions to give the corresponding dihydro- and tetrahydrothiophene derivatives.



Scheme 76

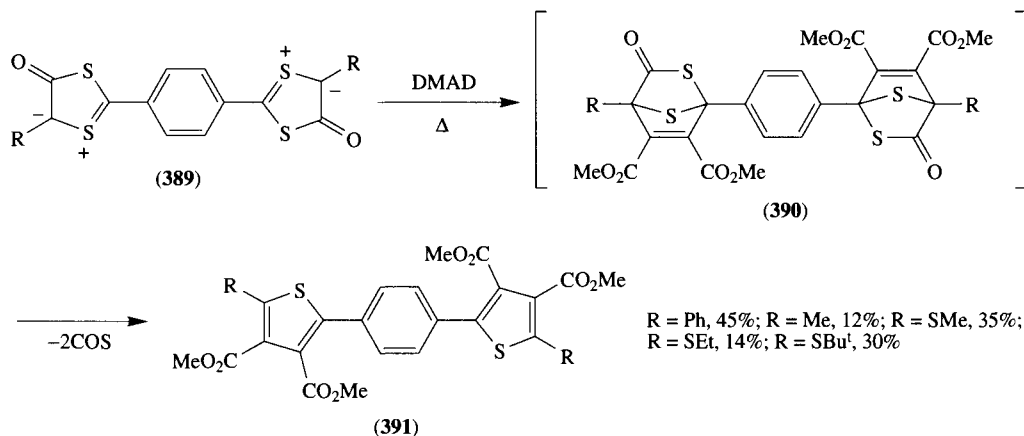


Scheme 77



Novel bis(1,3-dithiolylium-4-olates) (**389**) contain two masked thiocarbonyl ylide systems and thus undergo 1,3-dipolar cycloaddition with DMAD to give the initial adducts (**390**) which spon-

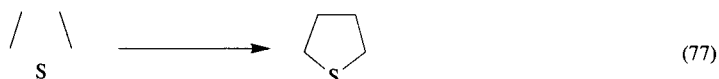
taneously extrude carbonyl sulfide to give the thiophenes (**391**) in moderate yields (Scheme 78) <88CB313>. Bis(1,3-dithiolium-4-olates) in which the 1,4-phenylene group of (**389**) is replaced by 4,4'-biphenylene, 2,6-naphthylene, and 1,4-dipiperazinediyl groups were also synthesized and allowed to react with DMAD to give the corresponding thiophene derivatives.



Scheme 78

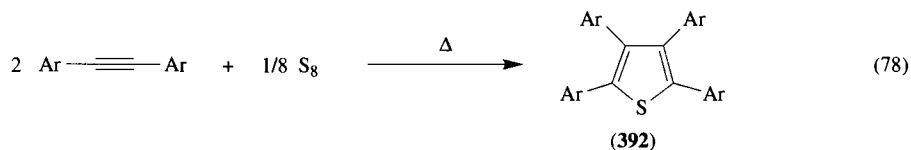
2.11.4 RING SYNTHESIS BY FORMATION OF THREE BONDS

It is theoretically possible to construct a thiophene ring by combining the following units with three bonds: (a) C—C—S + C + C, (b) C—S—C + C + C, (c) C—C—C + C + S, (d) C—C + C—C + S, and (e) C—C + C—S + C. Among these, only combination (d) (two two-carbon units + sulfur source) can provide practical synthetic methods for thiophenes.

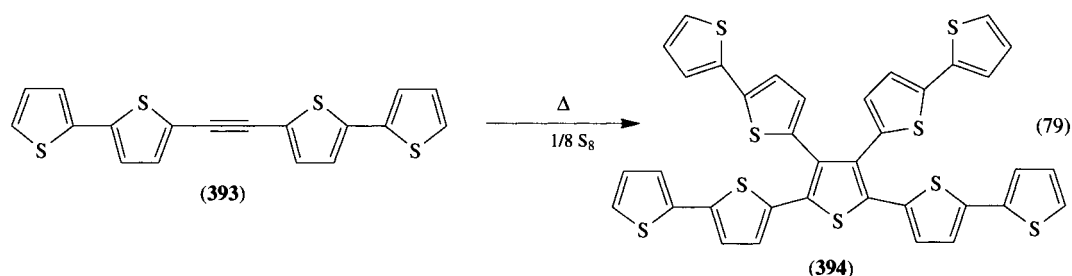


2.11.4.1 From Two Molecules of Alkynes and a Sulfur Source

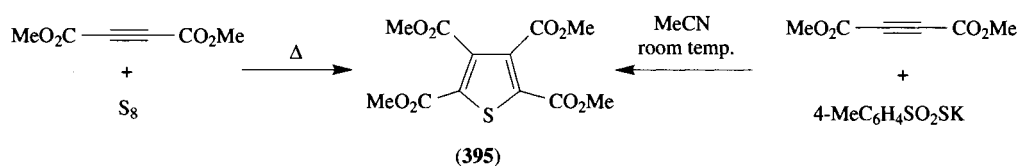
A series of diarylethyne react with elemental sulfur to give tetraarylthiophenes (**392**) in good yields (Equation (78)) <93SUL273>. Although the reaction requires rather forcing conditions, this provides a very practical method. For example, heating diphenylethyne (127 g) and elemental sulfur (11.6 g) neat at 220–240°C for 4 h affords practically pure tetraphenylthiophene (99 g) after simple workup. The method is also applicable to tetraarylselenophene synthesis. Application of the method to compound (**393**) allows the preparation of the structurally interesting oligothiophene (**394**) of a star-burst type (Equation (79)) <92H(34)1487>.



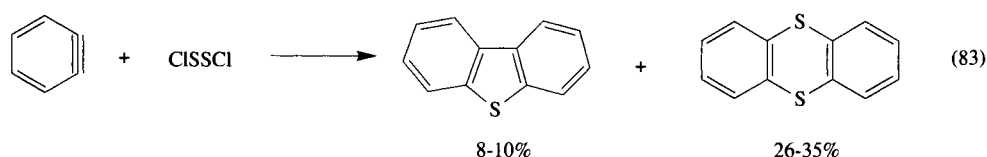
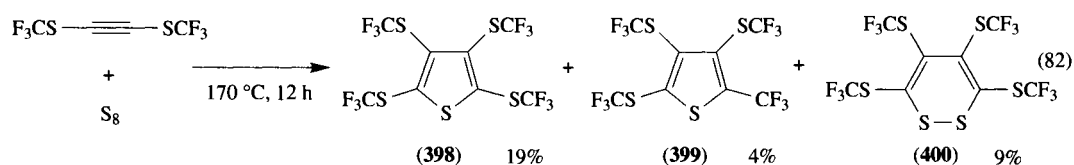
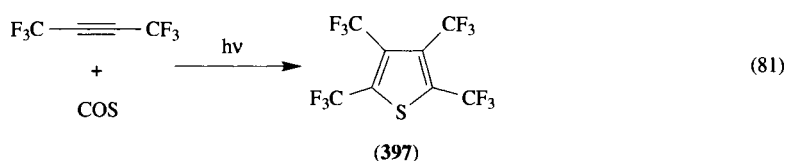
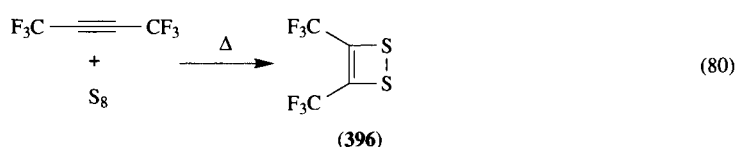
Ar = Ph, 79%; Ar = 4-ClC₆H₄, 88%; Ar = 4-MeC₆H₄, 63%; Ar = 4-C₆H₅C₆H₄, 82%;
 Ar = 4-MeOC₆H₄, 62%; Ar = 4-MeSC₆H₄, 81%; Ar = 1-naphthyl, 73%; Ar = 2-naphthyl, 59%;
 Ar = 2-thienyl, 57%; Ar = 2-pyridyl, 73%

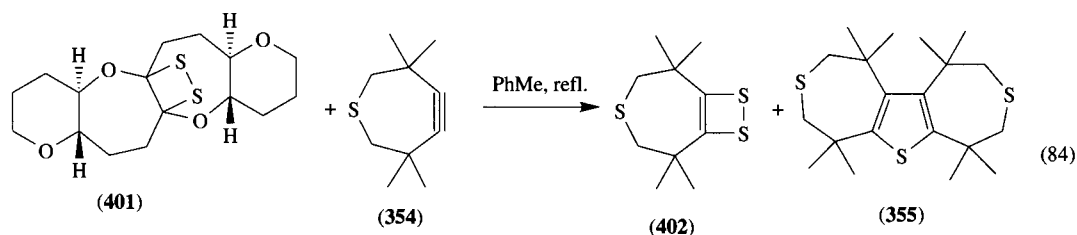


Tetramethyl thiophenetetracarboxylate (**395**) was prepared from DMAD in several ways. Heating DMAD and elemental sulfur in xylene at 160°C in a sealed tube affords (**395**) in 46% yield <71NKZ1193>, while, in the presence of a catalytic amount of selenium, the same reaction proceeds at 110°C to give (**395**) in 53% yield <87NKK1464> (Scheme 79). It has been found that DMAD reacts with potassium *p*-toluenethiosulfonate at room temperature to give (**395**) in 76% yield <92JOC5270>. Although the reaction of hexafluoro-2-butyne with elemental sulfur afforded the first isolable dithiete (**396**) (Equation (80)), the reaction with atomic sulfur, generated by photolysis of COS, gives the thiophene (**397**) (Equation (81)) at low conversion <83CJC2268>. Heating bis(trifluoromethyl)ethyne and elemental sulfur produces a mixture of thiophenes (**398**) and (**399**) along with 1,2-dithiin (**400**) (Equation (82)) <85JHC1631>. It is also known that benzyne, generated by thermal decomposition of 2-carboxybenzenediazonium chloride, reacts with disulfur dichloride to give a mixture of dibenzothiophene and thianthrene, though not practically important (Equation (83)) <89SUL83>. Dithiatopazine (**401**) serves as the sulfur source, thus affording the dithiete (**402**) and the thiophene (**355**) on reaction with the ethyne (**354**) (Equation (84)) <90JA3029>.

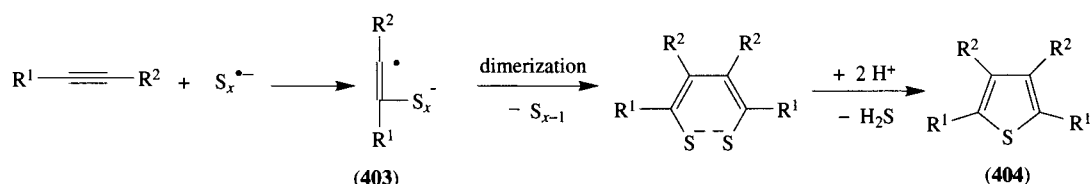


Scheme 79



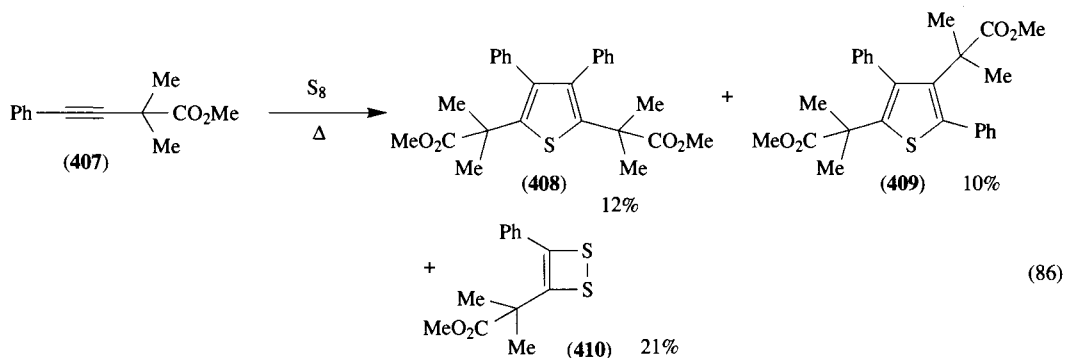
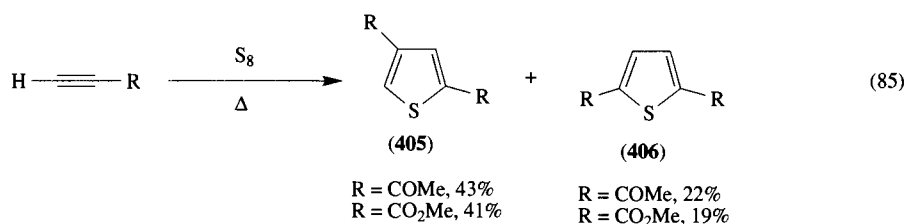


The electroreduction of alkynes, carrying at least one strongly electron-withdrawing substituent, at a compact sulfur-carbon electrode in aprotic media gives thiophenes (**404**) as the major product (Scheme 80) <86TL2261>. The reaction may involve addition of an electrochemically generated S_x radical anion to the alkynes and dimerization of the resulting radical anions (**403**) followed by protonation and loss of hydrogen sulfide. The addition step seems to proceed regioselectively to give the single thiophene isomer. On the other hand, thermal reaction of 3-butyne-2-one and methyl propiolate with elemental sulfur affords a mixture of thiophenes (**405**) and (**406**) (Equation (85)) <87H(26)2215, 87NKK1424>. Although the reaction of elemental sulfur with ethynes carrying two bulky substituents such as di-*t*-butyl- and di-1-adamantylethynes provides a convenient synthesis of 1,2-dithietes, the reaction with the ethyne (**407**) affords a mixture of thiophenes (**408**) and (**409**) in addition to the dithiete (**410**) (Equation (86)) <93BCJ623>.



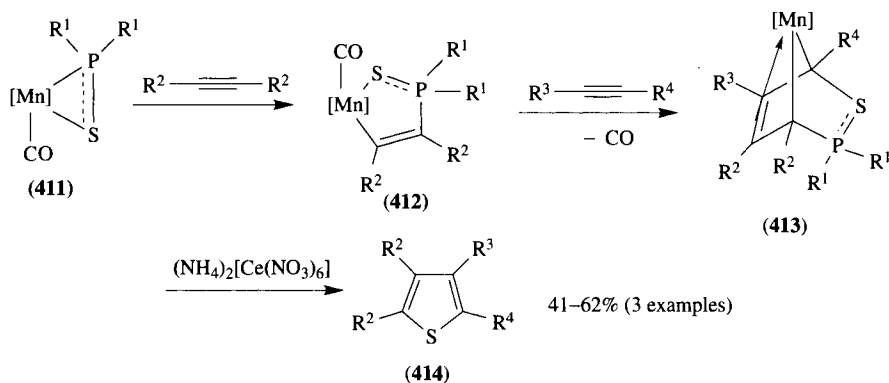
$R^1 = H, R^2 = Ph$, no reaction; $R^1 = H, R^2 = CO_2Et$, 10%; $R^1 = Ph, R^2 = CHO$, 82%; $R^1 = Ph, R^2 = CO_2Me$, 85%;
 $R^1 = Ph, R^2 = CN$, 80%; $R^1 = R^2 = CO_2Me$, 32%

Scheme 80



Thiophosphinitomanganese complexes (**411**) react with alkynes carrying an electron-withdrawing substituent to give five-membered heterocycles (**412**), which are transformed into thiaphos-

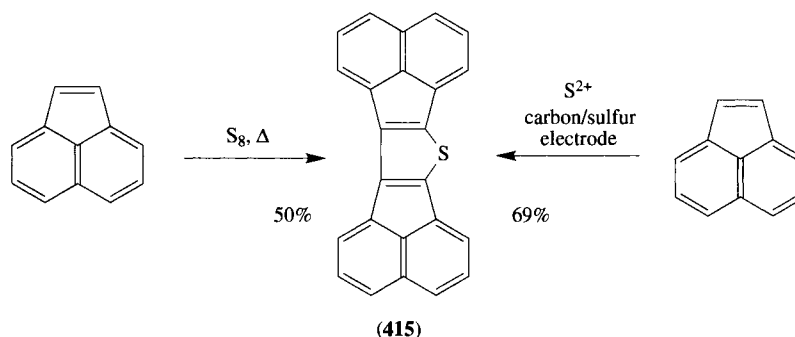
phamanganabicycloheptadienes (**413**) by further reaction with another alkynes. Oxidation of (**413**) affords thiophenes (**414**) (Scheme 81) <90CB783>.



Scheme 81

2.11.4.2 From Two Molecules of Alkenes and a Sulfur Source

Acenaphthylene reacts with elemental sulfur in DMF at 120°C to give thiophene (**415**); when the reaction was quenched at the early stage, a dihydro-1,4-dithiin, which is a probable intermediate leading to (**415**), is isolated <89SUL135>. Acenaphthylene also reacts with an S^{2+} species, generated by oxidation of a carbon–sulfur electrode, to give (**415**) in a better yield <94BSF789>.

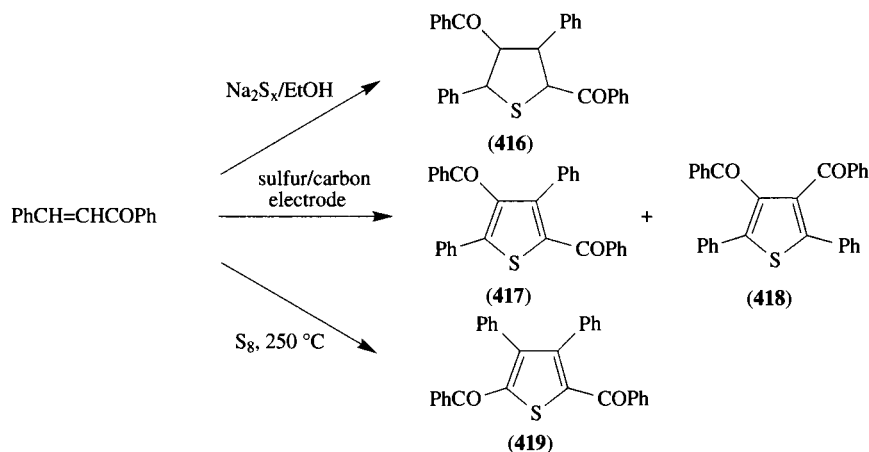


Scheme 82

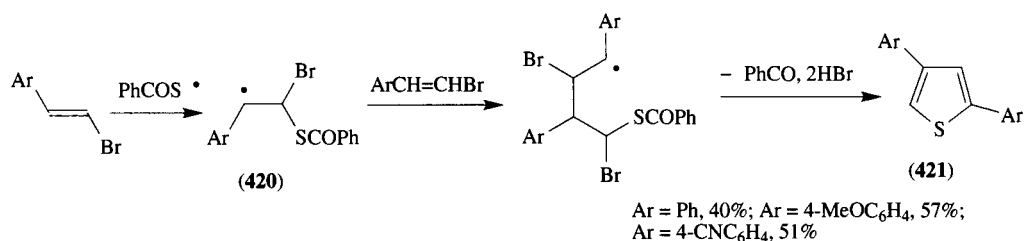
Chalcone (1,3-diphenylpropenone) reacts with sodium polysulfide (Na_2S_x), prepared from anhydrous Na_2S and sulfur in ethanol, to give a tetrahydrothiophene (**416**) in 74% yield (Scheme 83) <83S160>. On the other hand, the reaction of chalcone with a nucleophilic sulfur, generated by reduction of a carbon–sulfur electrode, affords a mixture of thiophenes (**417**) and (**418**) though in low yield <90BSF427>. It was also claimed that the reaction of chalcone with elemental sulfur produced the thiophene (**419**) <88IJC(B)179>. A variety of α,β -unsaturated ketones, like chalcone, react with sodium polysulfide to give tetrahydrothiophene derivatives in good yields <83S160, 83JOC4049, 85JOC85>.

Copper(I) salt promoted reaction of styryl bromides with dibenzoyl disulfide in a hot aprotic polar solvent produces 2,4-diarylthiophenes (**421**) in moderate yields (Scheme 84) <92CL1947>. The reaction may involve addition of PhCOS radical to the double bond of the substrate (activated by copper(I) salt), further addition of the resulting radical (**420**) to the substrate, and intramolecular radical substitution with extrusion of benzoyl radical followed by elimination of hydrogen bromide.

The reaction of the diazo compound (**422**) with molten sulfur affords the structurally interesting

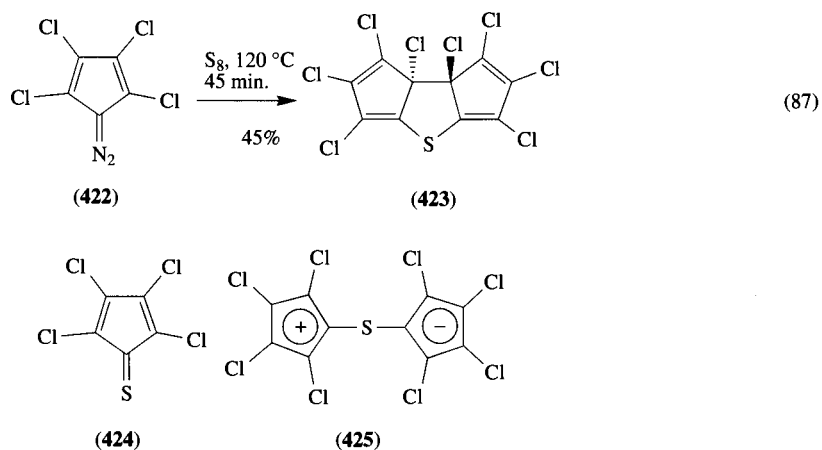


Scheme 83



Scheme 84

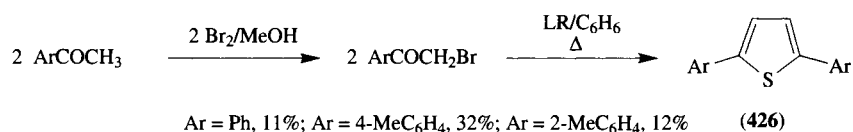
thiophene (Equation (87)) **(423)** <94SUL217>. The thioketone **(424)** and the ylide **(425)** are the proposed intermediates leading to **(423)**.



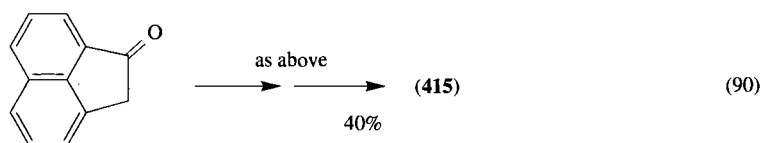
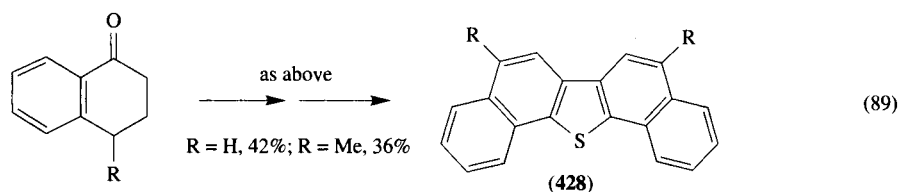
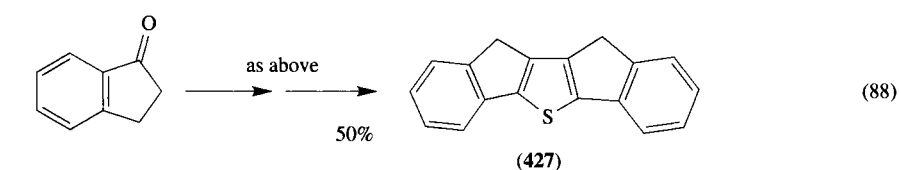
2.11.4.3 From Two Molecules of α -Bromo Ketones and a Sulfur Source

Acetophenones were brominated with bromine in methanol, the methanol was evaporated, and the resulting crude α -bromo ketones were heated with Lawesson's reagent in boiling benzene to give thiophenes **(426)** in low overall yields (Scheme 85) <88PS(35)127, 92MI 211-02>. In a similar way, 1-indanone, 1-tetralones, and acenaphthenone were converted to the thiophenes **(427)**, **(428)**, and **(415)**, respectively (Equations (88)–(90)), in reasonable overall yields. Although the yield of thiophenes is not necessarily excellent, the method is quite practical; in a larger-scale synthesis,

334 g (53%) of the thiophene (**428**; R = H) was obtained starting from 678 g of 1-tetralone by this simple operation.



Scheme 85



2.11.5 RING SYNTHESIS FROM OTHER HETEROCYCLIC COMPOUNDS

Many thiophenes are prepared by processes involving a ring transformation, that is, conversion of other heterocyclic compounds to thiophenes. Several syntheses of this type have been already described; however, there are still many syntheses which should be treated here independently.

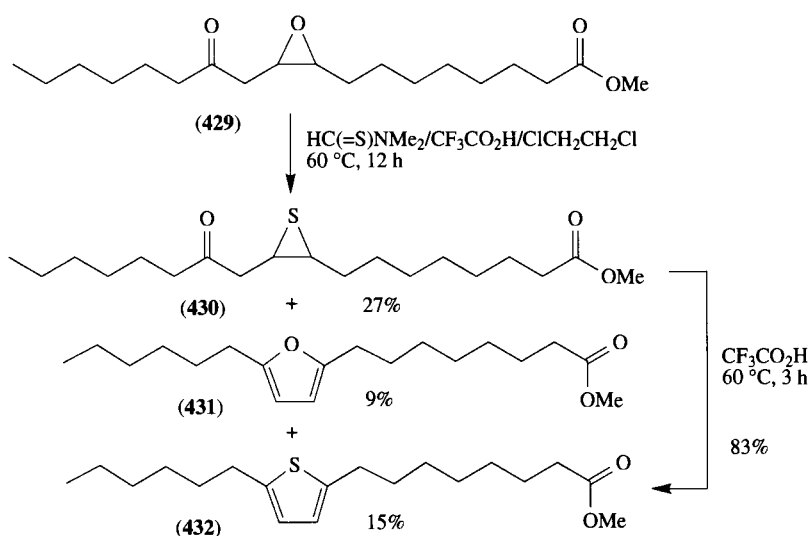
2.11.5.1 From Three- and Four-membered Heterocyclic Compounds

Treatment of the epoxide (**429**) with dimethylthioformamide in the presence of TFA produces a mixture of the thiirane (**430**), furan (**431**), and thiophene (**432**) (Scheme 86) <88S467>. The thiirane (**430**) is converted to (**432**) in good yield by treatment with TFA. Protonation of the carbonyl group of (**430**) followed by ring expansion and dehydration would explain the formation of (**432**) (Scheme 87).

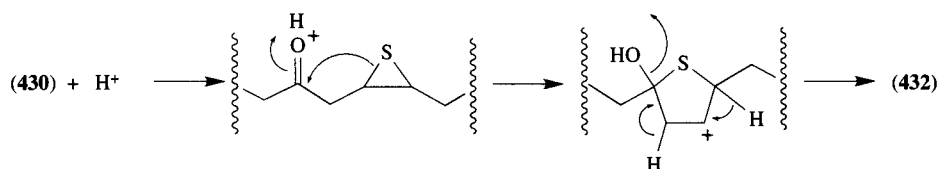
Butylthiomethylene ketones (**433**), on reaction with the lithium salt of a 2-methylthiooxazole derivative (**434**) and subsequent treatment of the unpurified reaction products with silica gel and hydrochloric acid, give the thiophenes (**437**) in 32–63% yields (Scheme 88) <83S575>. The proposed mechanism involves a thiomethylene transfer from the chelated lithio-oxazolines (**435**), leading to thiiranes (**436**), which produce (**437**) by ring expansion followed by loss of butanethiol.

The bicyclic compound (**438**), which is formed by [2 + 2] cycloaddition of 2H-thiete and TCNE in 55% yield (based on the precursor compound of the thiete), rearranges to give the thiophene (**439**) in high yield, when heated in the non-polar solvent benzene (Scheme 89) <85JOC799>.

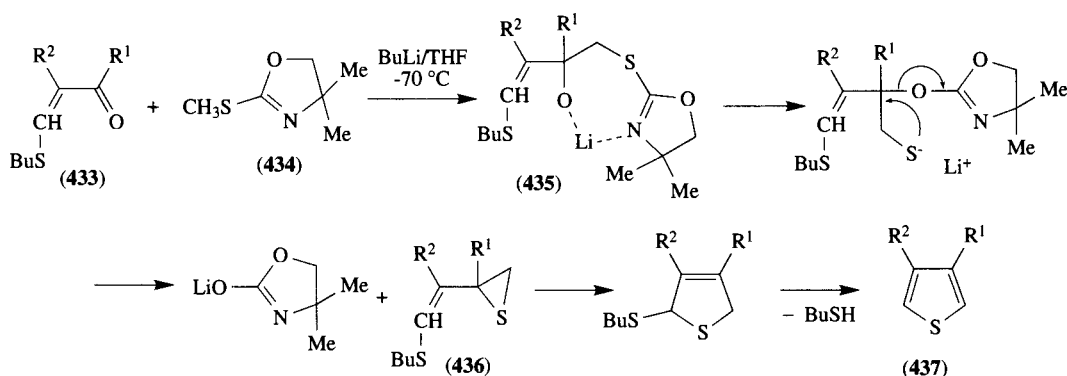
Treatment of the lactone (**440**) with hydrogen sulfide in the presence of Et₃N affords thiotetronic acid (**338**), which is treated with ketene to give the thiophene (**441**) <86EUP189096, 86EUP203495>.



Scheme 86



Scheme 87



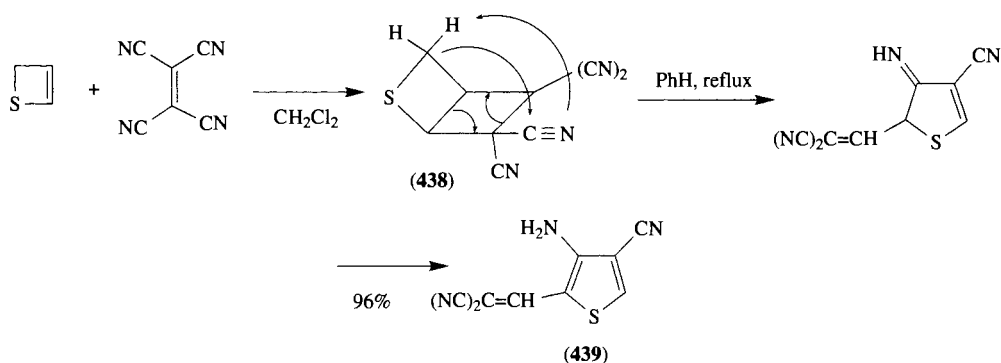
$\text{R}^1, \text{R}^2 = -\text{CMe}_2-(\text{CH}_2)_3-$, 63%; $\text{R}^1, \text{R}^2 = -\text{CHMe}-(\text{CH}_2)_2-$, 32%; $\text{R}^1, \text{R}^2 = -(\text{CH}_2)_4-$, 44%;
 $\text{R}^1 = n\text{-C}_5\text{H}_{11}$, $\text{R}^2 = n\text{-C}_4\text{H}_9$, 38%; $\text{R}^1 = \text{Et}$, $\text{R}^2 = \text{Me}$, 33%

Scheme 88

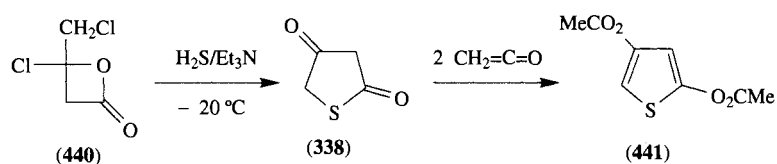
2.11.5.2 From Five-membered Heterocyclic Compounds

2.11.5.2.1 From furans

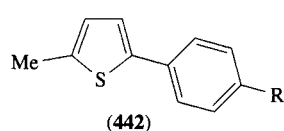
Conversion of furans to the corresponding thiophenes is one of the classical thiophene syntheses. Combination of hydrogen sulfide and an acid catalyst is the reagent most commonly used for this purpose. Thus, thiophenes (442) <84KGS1606> and (443) <89KGS1046> were synthesized from the corresponding furans on treatment with this reagent. Lawesson's reagent was employed for the synthesis of the benzo[c]thiophene (446) from (444) via furan (445) (Scheme 91) <92AM(4)564>.



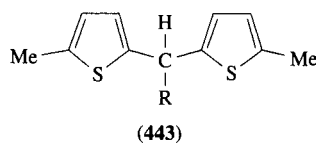
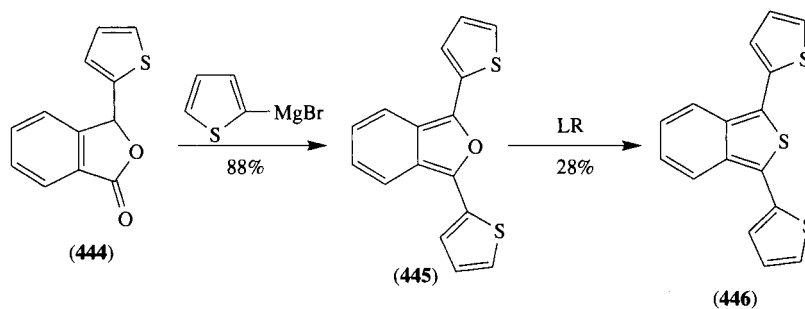
Scheme 89



Scheme 90



R = H, Me, OMe, Cl, Br

R = Me, Et, Ph, 4-MeOC₆H₄, 4-BrC₆H₄

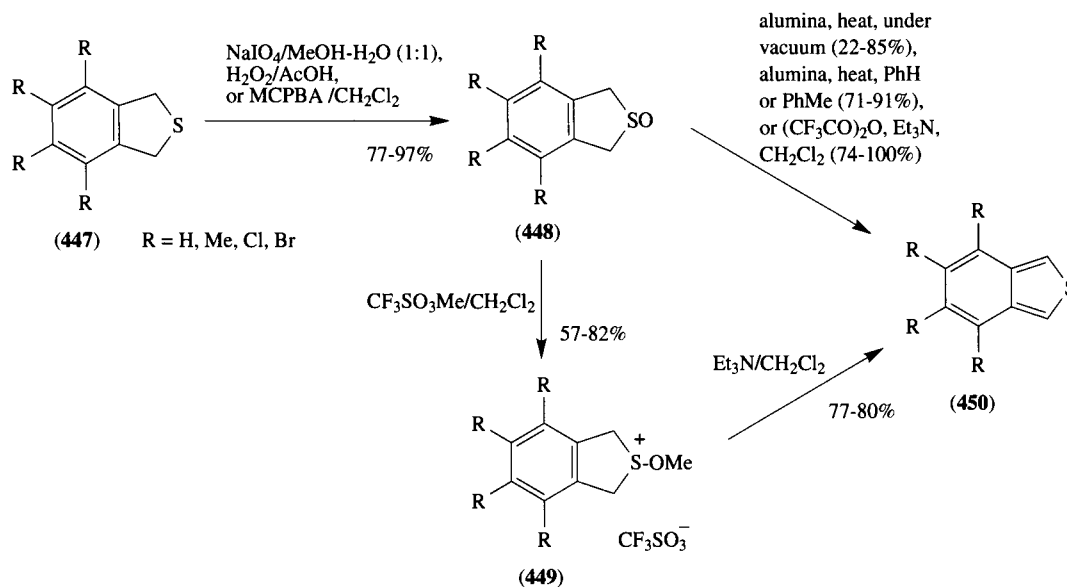
Scheme 91

2.11.5.2.2 From tetra- and di-hydrothiophenes and related compounds

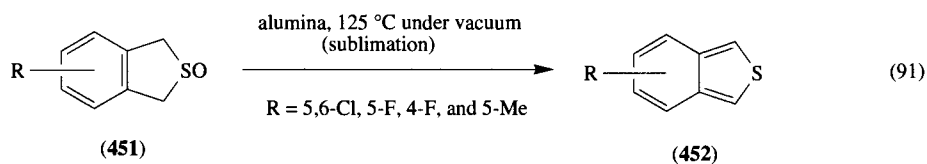
Although conversions of tetrahydrothiophenes (thiolanes), dihydrothiophenes (sulfolenes), thiophenones, and the related compounds to the corresponding thiophenes do not involve a process of ring transformation, they are important in the synthesis of thiophenes.

Benzo[*c*]thiophene and its tetramethyl, tetrachloro, and tetrabromo derivatives (**450**) were prepared from (**447**) via sulfoxides (**448**) (Scheme 92) [91CB645]. The dehydration of (**448**) was satisfactorily attained under a variety of conditions: (a) dehydration by heating with alumina under vacuum results in the sublimation of (**450**) on a cold finger in 22–85% yields, (b) dehydration by heating with alumina in boiling benzene or toluene affords (**450**) in 71–91% yields, (c) Pummerer dehydration by treatment with trifluoroacetic acid anhydride also gives (**450**) in 74–100% yields, and (d) methylation followed by treatment of the resulting sulfonium salts (**449**) with Et₃N also produces (**450**) in reasonable overall yields. 5,6-Dichloro-, 5-fluoro-, 4-fluoro-, 5-methyl-benzo[*c*]thiophenes (**452**) were also satisfactorily obtained on heating the corresponding sulfoxides (**451**),

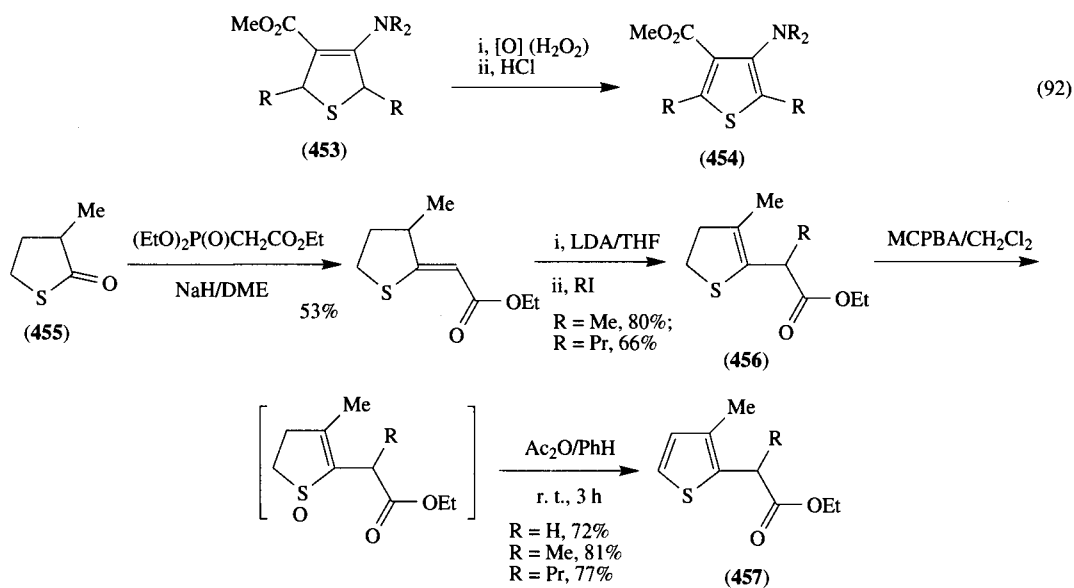
intimately mixed with activated alumina, under vacuum $\langle 94\text{CC}825 \rangle$ (Equation (91)). Preparation of 4-*t*-butylbenzo[*c*]thiophene in a similar manner was also reported $\langle 90\text{PS}(53)429 \rangle$.



Scheme 92

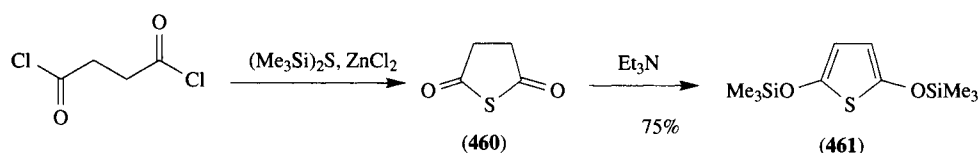
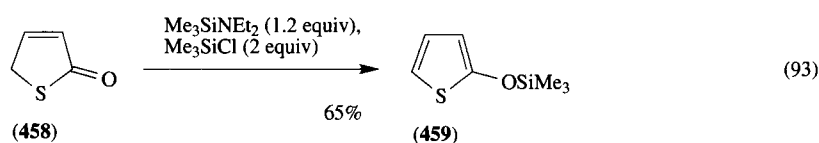


2,5-Dihydrothiophenes (453) were also converted to the corresponding thiophenes (454) via sulfoxide intermediates (Equation (92)) $\langle 89\text{GEP}(\text{O})3804794 \rangle$. Wittig–Horner reaction of (455) followed by alkylation leads to 2,3-dihydrothiophenes (456), which are oxidized and subjected to Pummerer reaction to give thiophenes (457) in reasonable overall yields (Scheme 93) $\langle 93\text{NKK}1263 \rangle$. Pummerer dehydration was also applied to the preparation of thieno[3,4-*c*]thiophenes $\langle 88\text{CC}959, 91\text{JOC}78, 92\text{BCJ}2821 \rangle$.

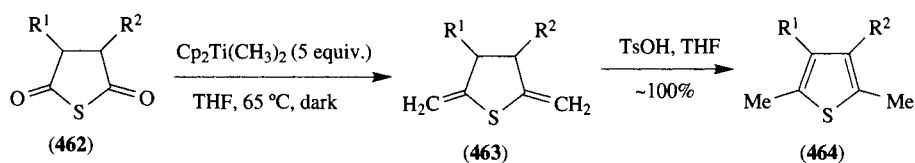


Scheme 93

Thiophenones, thiolane-2,5-diones, and related compounds are converted to thiophenes in many ways. Silylation of thiophen-2(5H)-one (**458**) with $\text{TMS-NEt}_3/\text{TMS-Cl}$ affords the thiophene (**459**) (Equation (93)) $\langle 84\text{JOC}551 \rangle$. The same conversion is also attained by use of $(\text{TMS})_2\text{S}$ or TMS-SMe as the silylating agent. Treatment of succinyl chloride with $(\text{TMS})_2\text{S}$ in the presence of ZnCl_2 and then addition of Et_3N to the mixture results in the direct formation of thiophene (**461**) in one pot via thiolan-2,5-dione (**460**) (Scheme 94) $\langle 84\text{JOC}3408 \rangle$. The intermediate (**460**) is isolated in high yield if Et_3N is not added. Methylenation of thiolan-2,5-diones (**462**) with excess dimethyltitanocene produces dimethylenation products (**463**), which rearrange to thiophenes (**464**) by treatment with an acid catalyst (*p*-toluenesulfonic acid) (Scheme 95) $\langle 94\text{JOC}494 \rangle$. Monomethylenation of (**462**) is also possible by use of a limited amount of dimethyltitanocene. Action of oxygen $\langle 84\text{JCS}(\text{P}1)2711 \rangle$, sulfur $\langle 86\text{JCR}(\text{S})168 \rangle$ and nitrogen $\langle 87\text{JHC}1301 \rangle$ nucleophiles on 3-thiophenone derivatives (**465**) produces thiophenes (**467**) via intermediates (**466**) (Scheme 96). Condensation of thiolan-3-one with methyl cyanoacetate leads to the compound (**468**), which is converted to the thiophene (**469**) in high yield (Scheme 97) $\langle 84\text{JHC}1231 \rangle$. Heating (**470**) with hydroxylamine affords the aminothiophene (**471**) (Equation (94)) $\langle 89\text{EUP}298542 \rangle$.

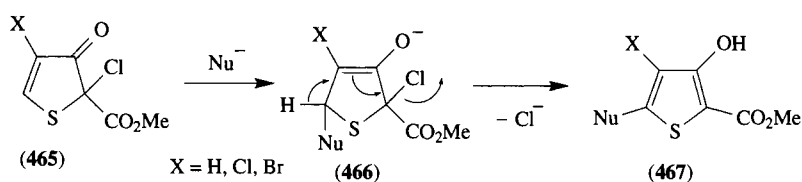


Scheme 94

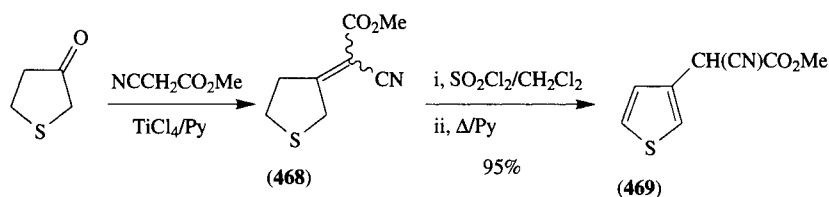


$\text{R}^1 = \text{R}^2 = \text{H}$, 40%; $\text{R}^1 = \text{Me}$, $\text{R}^2 = \text{H}$, 75%;
 $\text{R}^1 = \text{Ph}$, $\text{R}^2 = \text{H}$, 78%; $\text{R}^1, \text{R}^2 = -(\text{CH}_2)_4-$ (*cis*), 84%;
 $\text{R}^1, \text{R}^2 = -(\text{CH}_2)_4-$ (*trans*), 81%; $\text{R}^1, \text{R}^2 = -(\text{CH}_2)_2-$ (*cis*), 74%

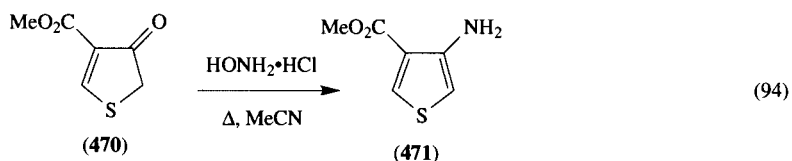
Scheme 95



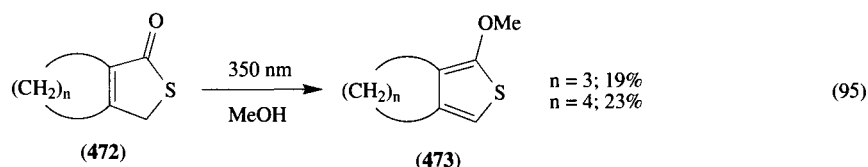
Scheme 96



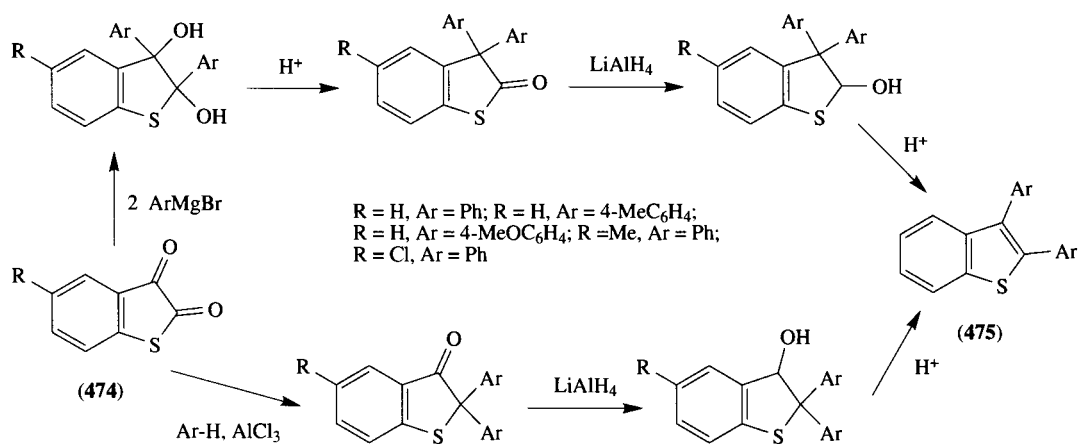
Scheme 97



Irradiation of the thiophenones (472) in methanol affords the condensed 2-methoxythiophenes (473) though in low yields <89HCA83, 92CB2311> (Equation (95)).

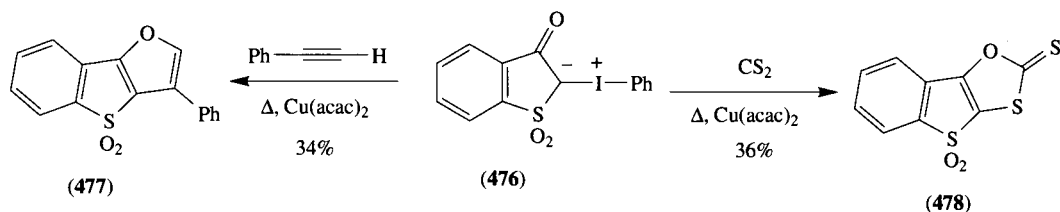


Efficient preparation of 2,3-diarylbenzo[b]thiophenes (475) is accomplished in two ways starting from thioisatins (474) (Scheme 98) <83TL3381>. A series of reactions, that is, Grignard reaction, pinacol rearrangement, hydride reduction, and acid-catalyzed rearrangement, converts (474) to thiophenes (475). Another series of reactions, Friedel-Crafts reaction, hydride reduction, and acid-catalyzed rearrangement, also convert (474) to (475).



Scheme 98

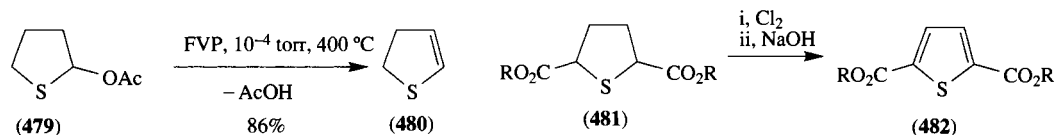
The ketocarbene, generated by Cu(acac)₂-assisted decomposition of the iodonium ylide (476), is trapped by phenylethyne and carbon disulfide to give thiophene dioxides (477) and (478), respectively, in moderate yields (Scheme 99) <89TL6673>.



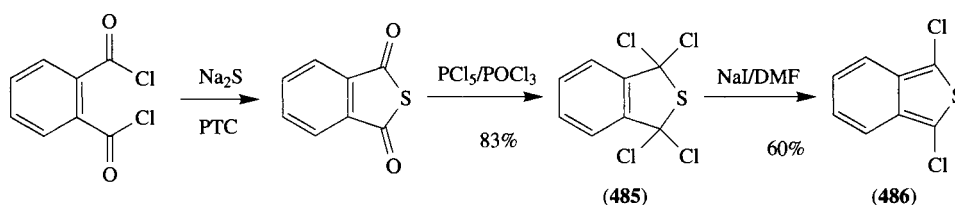
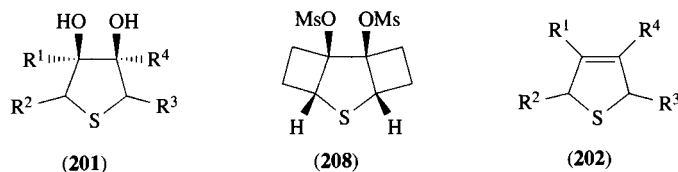
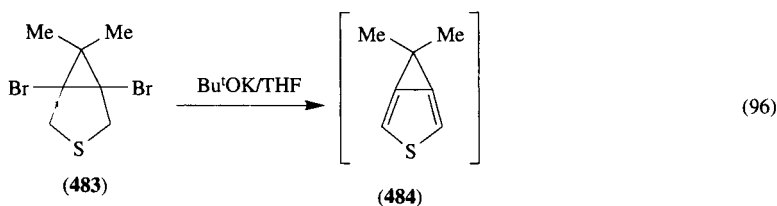
Scheme 99

FVP of 2-acetoxythiolane (479) provides a practical synthesis of 2,3-dihydrothiophene (480) <86JOC113> and is superior to the previous synthesis by heating (479) neat (Scheme 100). Thiolane is converted to thiophene on oxidation catalyzed by MoS₂ <89MIP1490120>. Chlorination and dehydrochlorination convert thiolanes (481) to thiophenes (482) <90EUP387725>. Treatment of the bicyclic compound (483) with Bu^tOK affords the angle-strained thiophene (484), which is not isolable but trapped by Diels-Alder reaction with isobenzofuran (Equation (96)) <90TL1315>. Conversion of thiolane-3,4-diols (201), the mesylate (208), and 2,5-dihydrothiophenes (202) to the corresponding

thiophenes by acid-catalyzed dehydration, base-catalyzed elimination, and oxidation with DDQ, respectively, has already been described (see Section 2.11.2.3.1). Reductive dechlorination of (485) with sodium iodide provides a facile synthesis of 1,3-dichlorobenzo[*c*]thiophene (486), from which a series of benzo[*c*]thiophene derivatives can be derived (Scheme 101) <91JOC6024>.



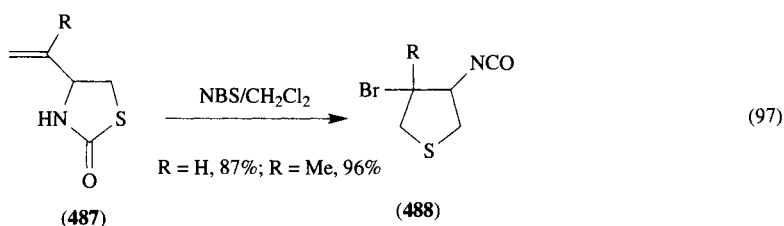
Scheme 100



Scheme 101

2.11.5.2.3 From sulfur- and nitrogen-containing five-membered heterocyclic compounds

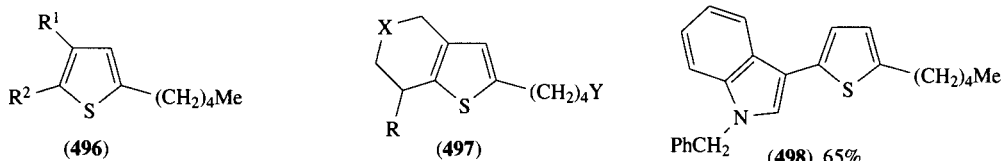
Reaction of readily available thiazolidinones (487) with NBS affords 3,4-disubstituted tetrahydrothiophenes (488) (Equation (97)) <87CC1577>. No explanation for the mechanistic pathway of this reaction was given except that it might involve the initial electrophilic attack of NBS on the sulfur atom.



The base-catalyzed intramolecular condensation of the pyridine (489) affords the compound (491)

Scheme 102

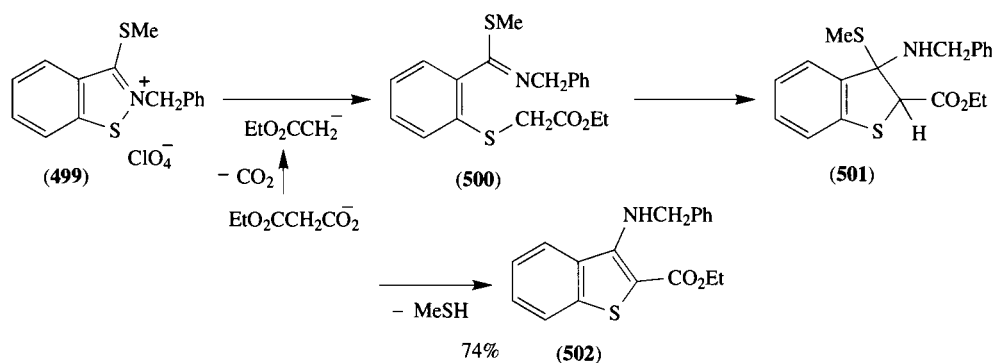
Scheme 103



$R^1 = H, R^2 = Ph$; 80%
 $R^1 = Me, R^2 = Bu^t$; 89%
 $R^1 = Ph, R^2 = Bn$; 71%
 $R^1 = H, R^2 = 1\text{-adamantyl}$; 70%

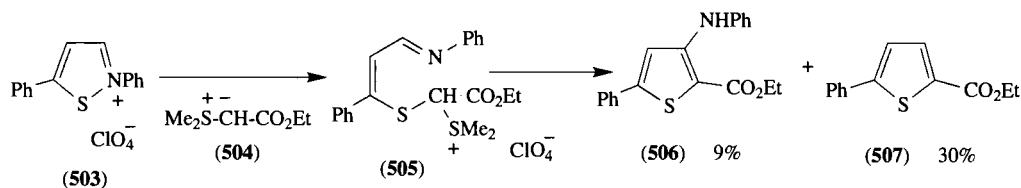
R = H, X = CMe₂, Y = Me; 87%
R = Y = Me, X = CH₂; 60%
R = H, X = S, Y = CO₂Et; 50%

Reaction of the isothiazolium perchlorate (**499**) with the potassium salt of 3-ethoxy-3-oxopropanoate (ethyl acetate anion equivalent) affords thiophene (**502**) probably via intermediates (**500**) and (**501**) (Scheme 104) ^{<84CJC1580>}. Some other aminothiophenes were prepared in this way in moderate to good yields. Reaction of the isothiazolium salt (**503**) with the sulfonium ylide (**504**) produces a mixture of thiophenes (**506**) and (**507**) (Scheme 105). A mechanism involving the initial formation of (**505**) was presented.



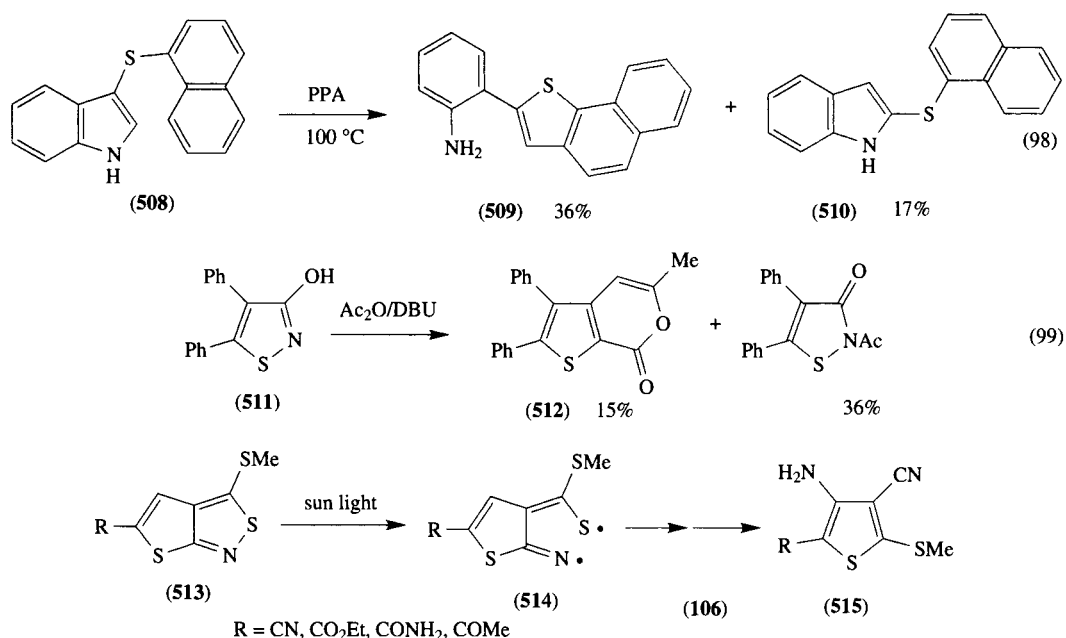
Scheme 104

3-(1-Naphthylthio)indole (**508**) undergoes a novel structural rearrangement to give the thiophene (**509**) in addition to the indole (**510**), the product of migration of the naphthylthio group (Equation

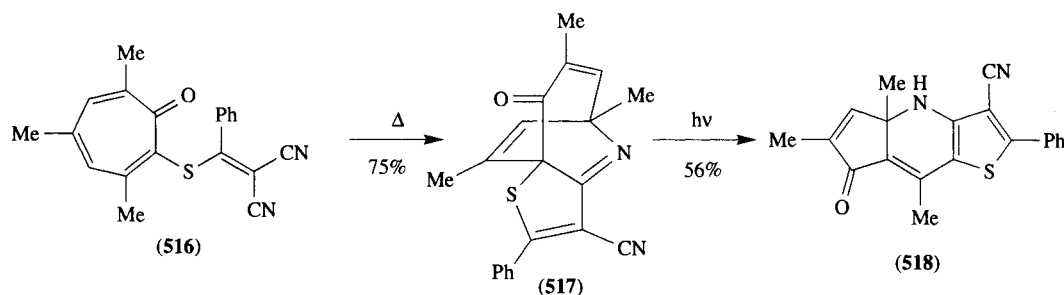


Scheme 105

(98)) <90CC1072>. The rearrangement seems to be general with 3-(aryltio)indoles, in which the aryl group is an electron-rich system. Treatment of the isothiazole (511) with acetic anhydride in the presence of DBU affords the thiophene (512) in low yield (Equation (99)) <85JHC1497>. Thieno[2,3-*d*]isothiazoles (513) undergo sunlight-induced sulfur extrusion to give thiophenes (515) (Scheme 106) <88H(27)2539>. The initial step of this transformation probably involves nitrogen—sulfur bond cleavage to give diradical intermediates (514). The intramolecular Diels–Alder adduct (517) obtainable from (516) photochemically isomerizes to (518) (Scheme 107) <89CB657>.



Scheme 106



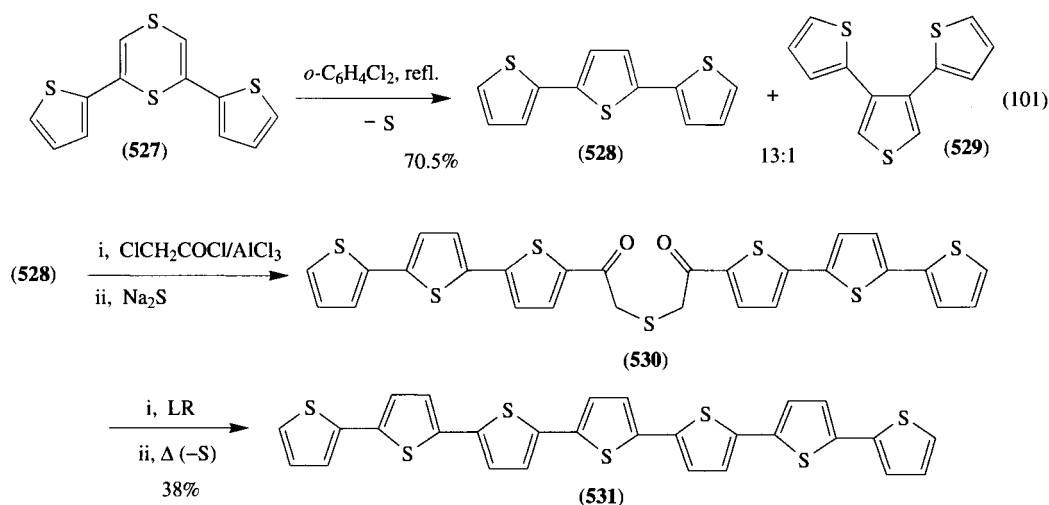
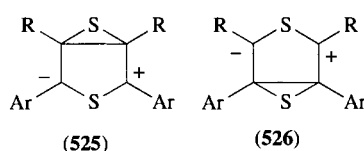
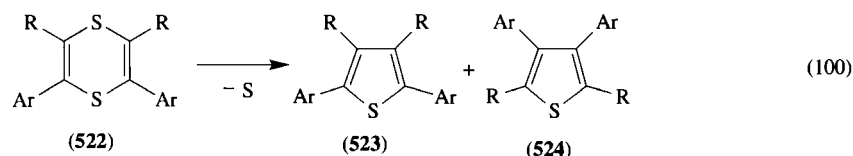
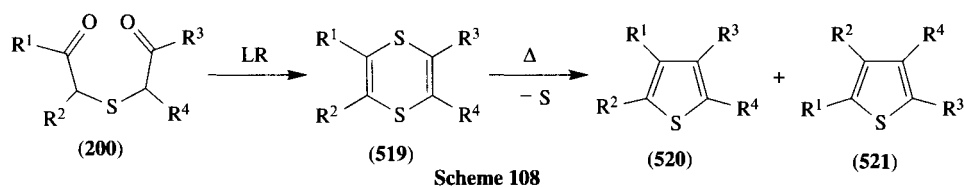
Scheme 107

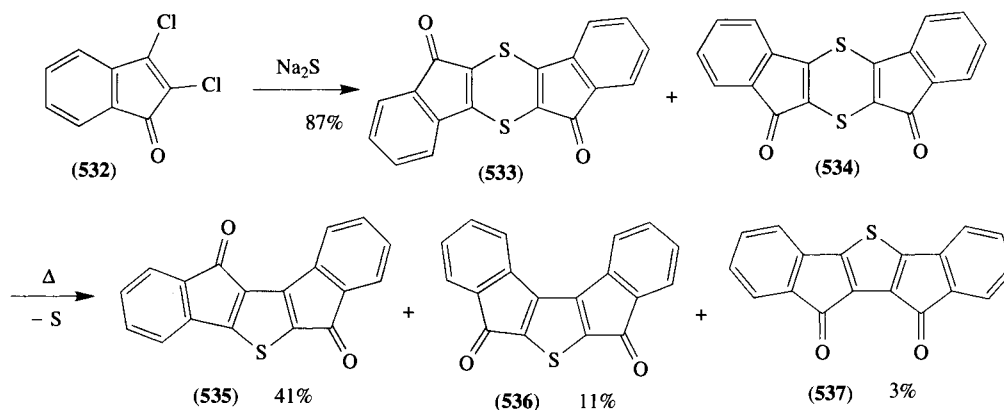
2.11.5.3 From Six-membered Heterocyclic Compounds

2.11.5.3.1 Extrusion of sulfur or sulfur monoxide from 1,2- and 1,4-dithiins

Formation of thiophenes by extrusion of sulfur is described for a number of substituted 1,2- and 1,4-dithiins. Particularly, sulfur extrusion of the latter compounds is often useful for the synthesis

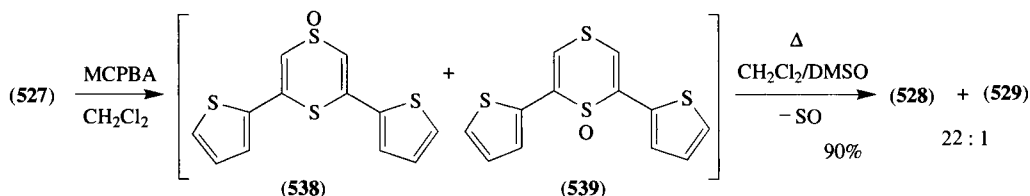
of thiophenes (87MI 211-01). Polysubstituted 1,4-dithiins (519) have become readily obtainable by sulfuration of diketo sulfides (200) with Lawesson's reagent or P_4S_{10} (Scheme 108) (84H(22)1527). Thermal extrusion of sulfur from these dithiins is expected to give a mixture of thiophenes (520) and (521), thereby their ratio depending on the nature of the substituents. Actually, sulfur extrusion of 2,6-diaryl-1,4-dithiins (522; R = H) and 3,5-dialkyl-2,6-diaryl-1,4-dithiins (522; R = alkyl) in refluxing *o*-dichlorobenzene affords a mixture of thiophenes (523) and (524) in 70.5–92% yields with predominant formation of thiophenes (523) (Equation (100)) (85H(23)1907). The observed regioselectivity of the sulfur extrusion can be explained by comparing the relative stability of the probable ylide intermediates (525) and (526) leading to (523) and (524), respectively. Application of the present reaction to the 1,4-dithiin (527) allows a large-scale synthesis of α -terthiophene (528) in a reasonable yield; in this case, the (528) and (529) are formed in the ratio 13:1 and the less soluble (528) is isolable in pure form by a single recrystallization (Equation (101)) (86H(24)637). Chloroacetylation of (528) followed by reaction with sodium sulfide gives the diketosulfide (530) (Scheme 109). Sulfuration of (530) and sulfur extrusion of the resulting 1,4-dithiin produce α -septithiophene (531) in one pot (87H(26)939). In a similar way, α -quinquethiophene was synthesized starting from α -bithiophene. A mixture of 1,4-dithiins (533) and (534) is obtainable by reaction of the indenone (532) and Na_2S . Heating this mixture neat at 280°C affords thiophenes (535)–(537) (Scheme 110) (83LA1598).





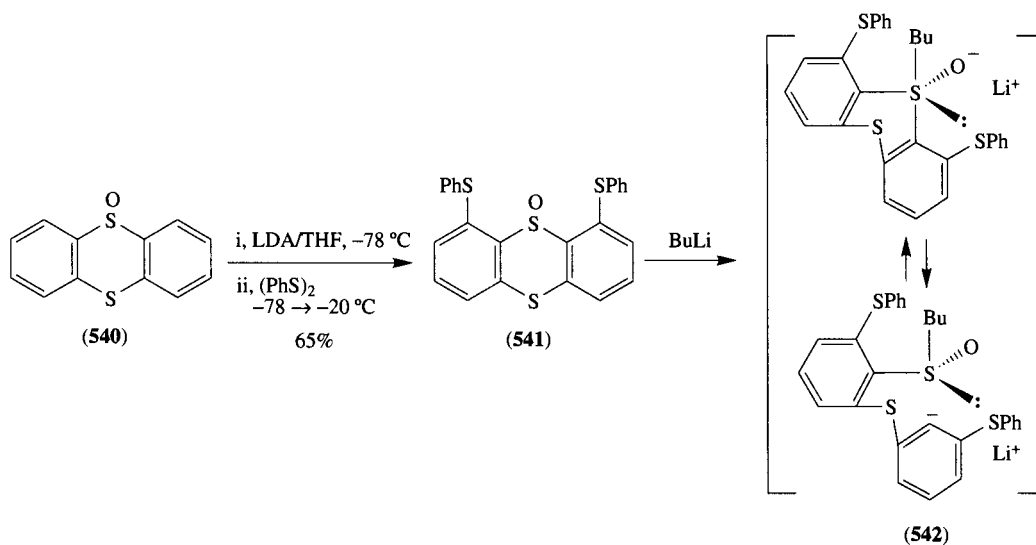
Scheme 110

Formation of thiophenes by extrusion of sulfur monoxide from 1,4-dithiin S-oxides is also known $\langle 86\text{SR}123 \rangle$. This enables a more selective synthesis of (528) from (527). Thus, when the crude sulfoxide mixture obtained by oxidation of (527) with MCPBA was heated in refluxing dichloromethane in the presence of DMSO, a mixture of (528) and (529) was obtained in 90% yield in the ratio 22:1 (Scheme 111) $\langle 87\text{H}(26)939 \rangle$. In this case, the isomer ratio is determined by the regioselectivity of the oxidation. A unique method for elimination of sulfur monoxide from thianthrene 4-oxide derivatives was also developed (Schemes 112 and 113) $\langle 91\text{H}(32)675, 92\text{TL}1489 \rangle$. Regioselective dilithiation of thianthrene-5-oxide (540) followed by treatment with diphenyl disulfide produces compound (541). Reaction of (541) with BuLi gives the congested dibenzothiophene (543) and the sulfide (544). The reaction is initiated by the attack of BuLi on the sulfinyl sulfur to form the sulfurane (542) as the probable intermediate which gives (543) and (544) via the intramolecular ligand coupling or ligand-exchange processes. Several congested dibenzothiophenes were synthesized in moderate yields in this way.

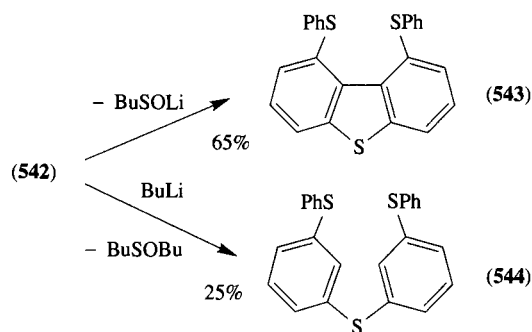


Scheme 111

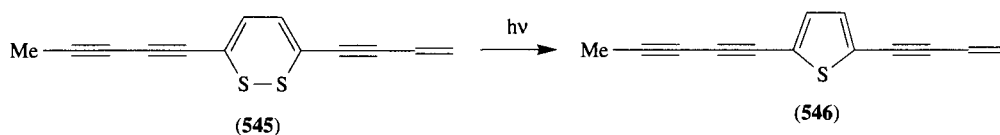
The chemistry of 1,2-dithiins has been attracting considerable interest in connection with their bioactivities $\langle 93\text{RHC}1 \rangle$. They are usually thermally and photochemically labile and extrude sulfur to give the corresponding thiophenes; for example, thiarubrine B (545), the antibiotic principal of giant ragweed, photochemically loses sulfur to give the thiophene (546) (Scheme 114) $\langle 94\text{JA}9403 \rangle$. The reaction of thiobenzophenone with tetracyanoethylene affords the 1,2-dithiin (547) and the thiophene (548) (Equation (102)); the conversion of (547) to (548) is fast on exposure to sunlight, but slow in the dark at 80°C $\langle 85\text{TL}1849 \rangle$. Meanwhile, the 1,2-dithiin (549) is highly thermally stable and is converted to the thiophene (550) only when heated with copper powder at elevated temperatures (Equation (103)) $\langle 94\text{CB}401 \rangle$. The compound (551) whose dithiin ring is surrounded by rigid σ -frameworks and kinetically stabilized is also very thermally stable and converted to the structurally interesting thiophene (552) when heated with copper powder (Equation (104)) $\langle 94\text{TL}1973 \rangle$.



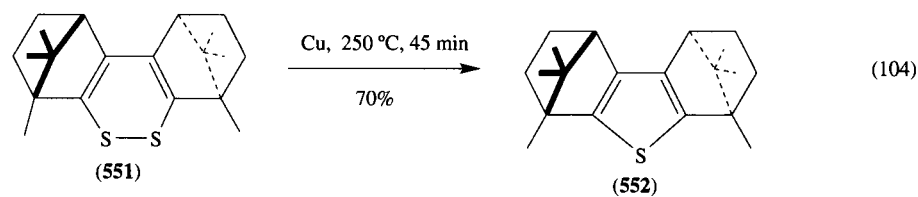
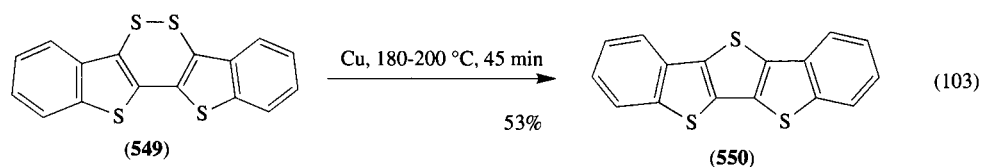
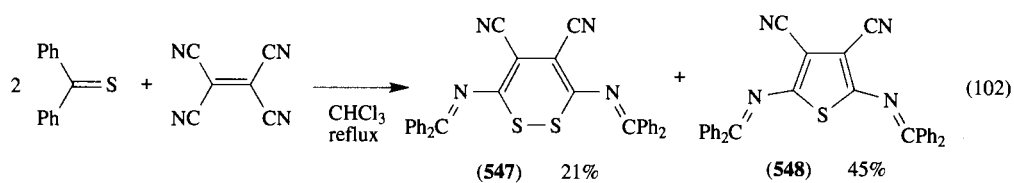
Scheme 112



Scheme 113

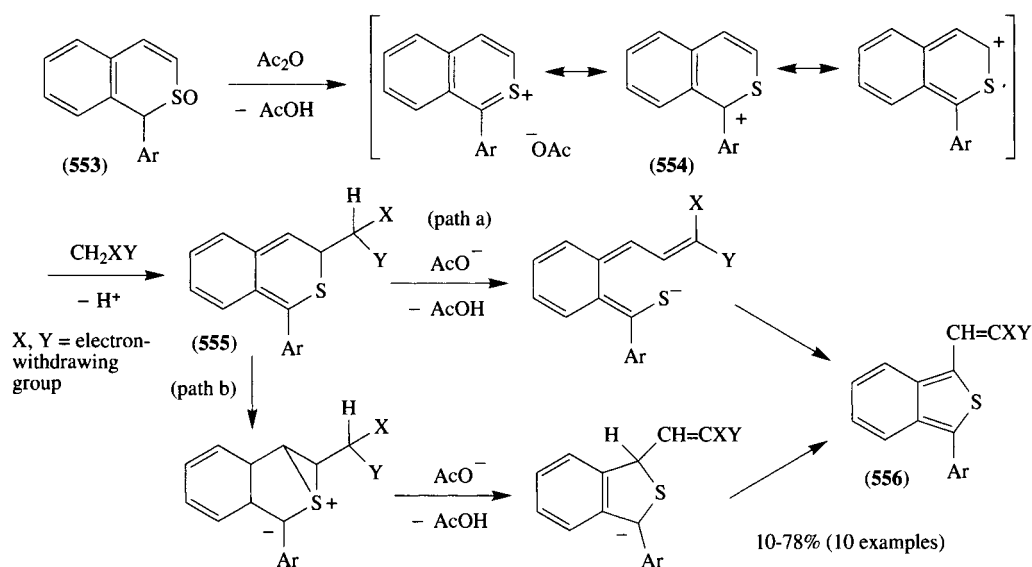


Scheme 114



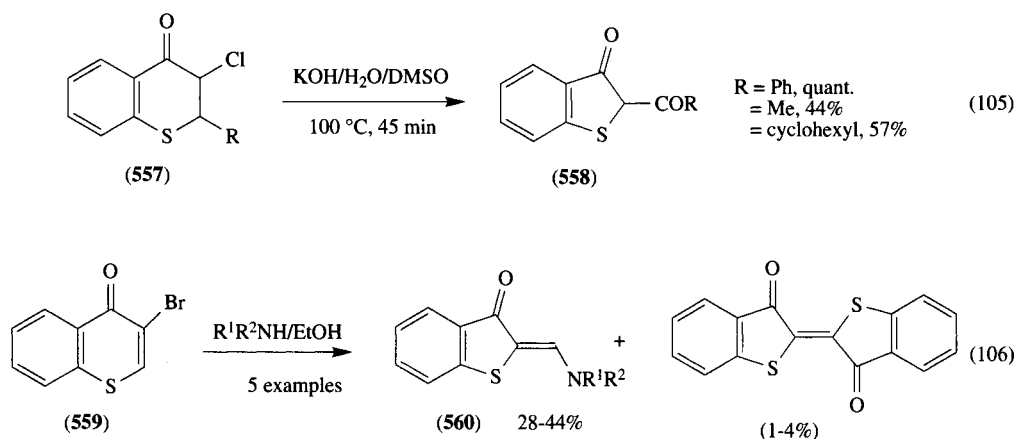
2.11.5.3.2 Ring contraction of other sulfur-containing six-membered heterocyclic compounds

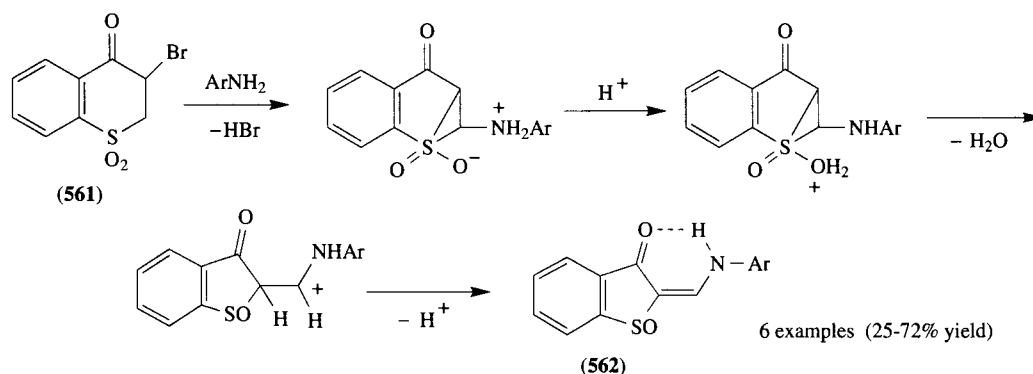
1-Aryl-1*H*-2-benzothiopyran 2-oxides (**553**) react with active methylene compounds in acetic anhydride to undergo a novel ring contraction, thus providing an interesting synthesis of benzo[*c*]thiophenes (**556**) in moderate yields (Scheme 115) <89JCS(P1)1611>. The initial step of the present reaction may involve the Pummerer reaction leading to the stable carbenium ion intermediates (**554**), to which active methylene compounds add to give another intermediate (**555**). Then, (**555**) collapses via either path (a) or (b) to give the thiophenes (**556**) as the final products. Incidentally, reaction of 1-unsubstituted 1*H*-2-benzothiopyran 2-oxide with active methylene compounds, under similar conditions, affords no benzothiophenes, but instead gives 1-substituted 1*H*-2-benzopyrans in good yields.



Scheme 115

2-Substituted 3-chlorothiochromen-4-ones (**557**), on treatment with base, rearrange to 2-acylbenzo[*b*]thiophen-3(2*H*)-ones (**558**) (Equation (105)) <83JCR(S)56>. Compound (**559**), an unsaturated derivative of (**557**), reacts with primary and secondary amines to give ring-contracted products (**560**) in moderate yields along with thioindigo in a small amount (Equation (106)) <90CL679>. On the other hand, compound (**561**), the sulfone derivative of (**557**), reacts with aromatic primary amines to give compounds (**562**) in acceptable yields <92NKK1223>. A mechanism for this ring contraction is proposed in Scheme 116 (the authors incorrectly assigned the unrearranged thiopyran structure for these compounds in the previous paper) <89JCS(P2)993>.



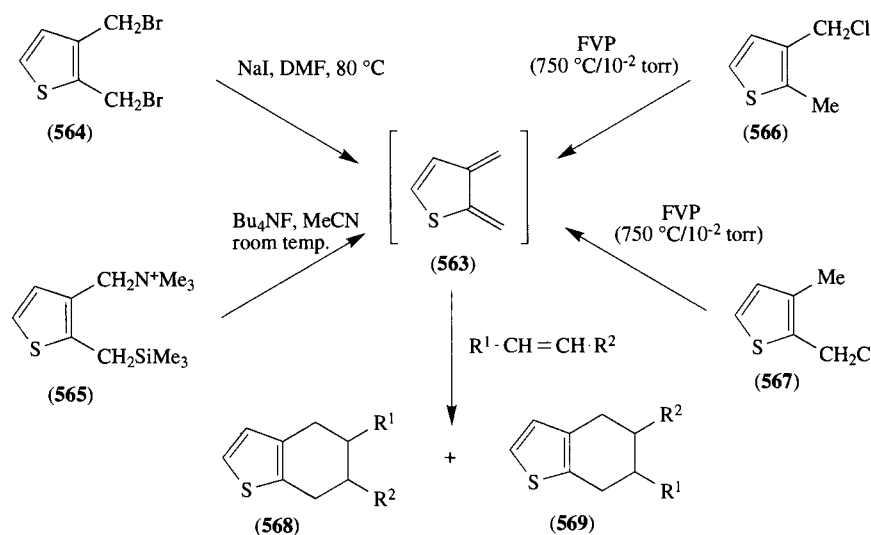


Scheme 116

2.11.6 BENZO[b]THIOPHENES BY ANNEALATION OF THIOPHENES

2.11.6.1 [2 + 4] Cycloaddition of Thiophene-2,3-quinodimethanes (2,3-Dimethylene-2,3-dihydrothiophenes) and Related Compounds

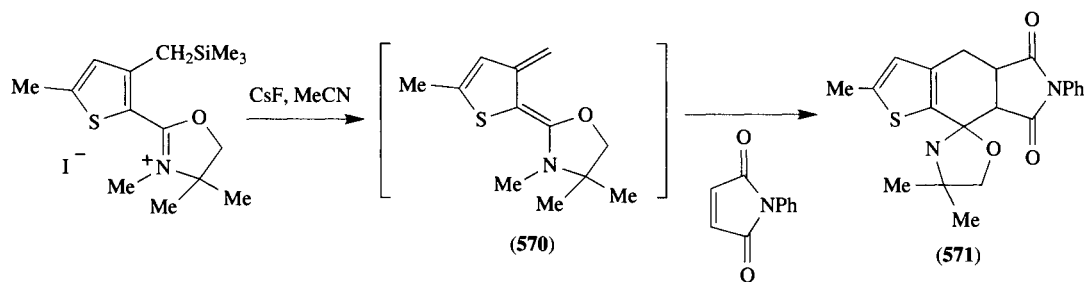
Thiophene-2,3-quinodimethane (**563**) is satisfactorily generated from a variety of precursor compounds such as (**564**)–(**567**) under appropriate conditions (Scheme 117) <87TL6085, 88TL117, 88TL2689, 91TL7751, 93RTC7> and can be trapped by a series of dienophiles to give six-membered fused thiophenes in good yields. In many cases, isomeric mixtures of (**568**) and (**569**) were formed on trapping with unsymmetrically substituted dienophiles. A quinodimethane such as (**570**) is generated and can be effectively trapped by dienophiles to give cycloadducts including (**571**) in good yields (Scheme 118) <88TL2689>.



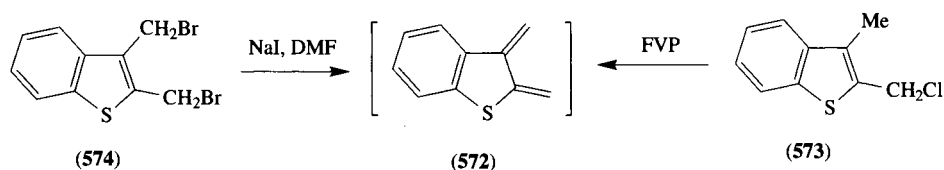
Scheme 117

Generation and trapping of benzothiophene-2,3-quinodimethane (**572**) was also reported; (**572**), generated by FVP of the benzothiophene (**573**), is trapped by methyl vinyl ketone to give (**575**) in 45% yield <88TL117> and (**572**), generated from (**574**) by treatment with sodium iodide, affords (**576**) by cycloaddition with 1,4-naphthoquinone followed by dehydrogenation (Scheme 119) <88CB1203>.

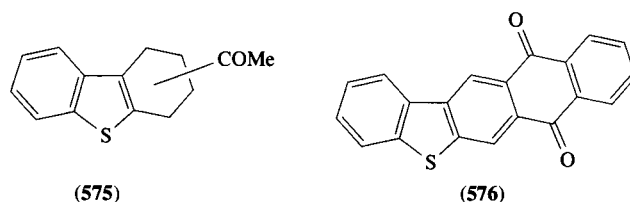
The pyranothiophenes (**577**), prepared in reasonable yields from commercially available thiophene-3-acetic acid in a single step, are stable thiophene-2,3-quinodimethane equivalents <88TL5817, 90JCS(P1)681>. Thus, they undergo Diels–Alder reaction with DMAD to give benzo[b]thiophenes (**578**) after loss of carbon dioxide, and with ethyl propiolate to give isomeric mixtures of (**579a**) and



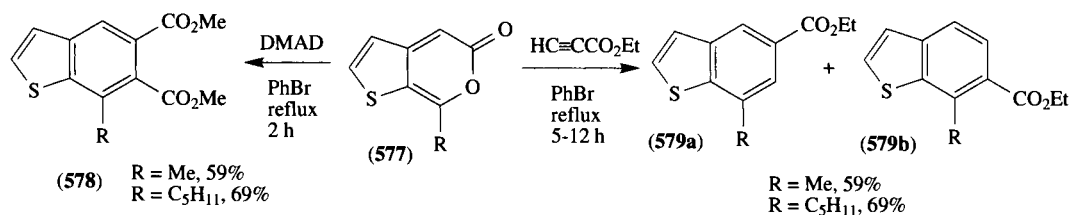
Scheme 118



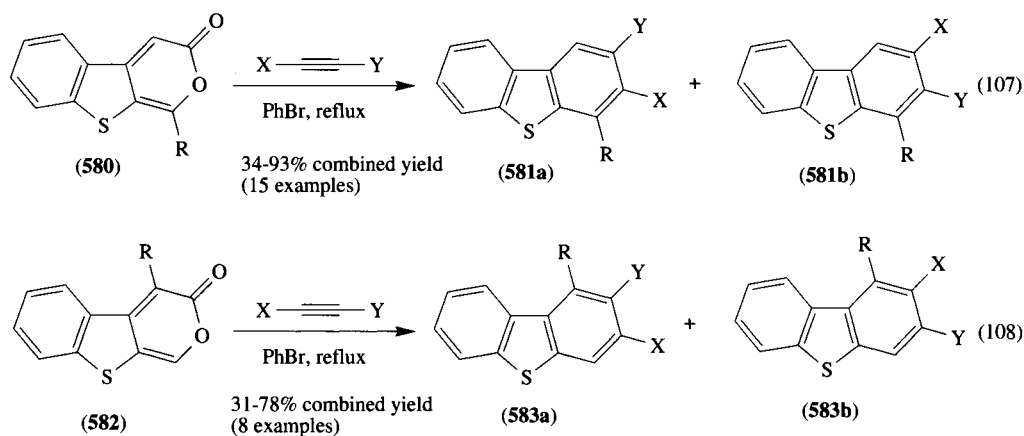
Scheme 119



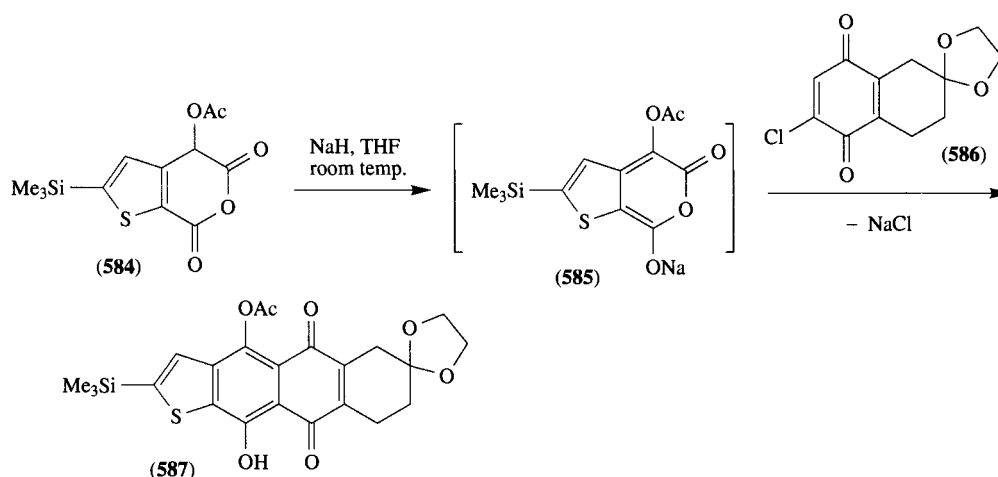
(579b) in the ratio 1 : 1 (Scheme 120). The benzothieno[2,3-*c*]pyran-3-ones (580) and (582) behave similarly to give isomeric mixtures, (581a) and (581b), and (583a) and (583b), respectively (Equations (107) and (108)), on reactions with unsymmetrically substituted alkynic dienophiles; an excellent regioselectivity was observed by use of dienophiles, such as ethyl trimethylsilylpropynoate, carrying a bulky substituent.



Scheme 120

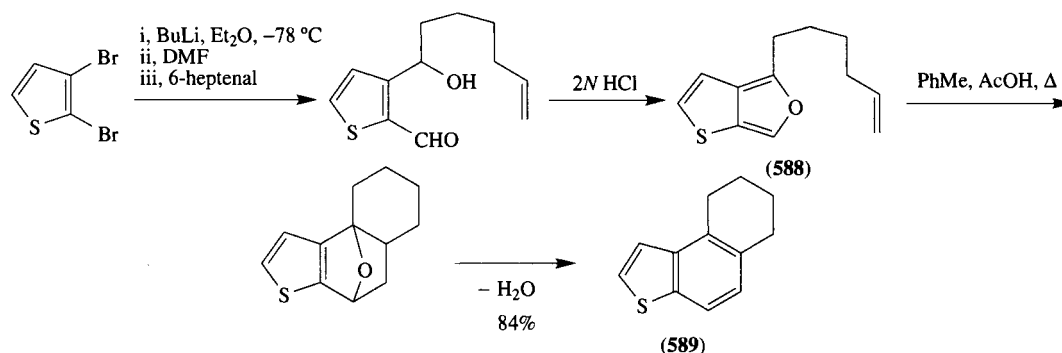


The enolate (**585**), generated by treatment of the thiophene analogue (**584**) of homophthalic anhydride with sodium hydride, serves as a diene and reacts with the benzoquinone (**586**) to produce the thiophene (**587**) in 57% yield in one pot (Scheme 121). This clever methodology was applied to the preparation of the D-ring thiophene analogue of daunomycin <87TL3971>.

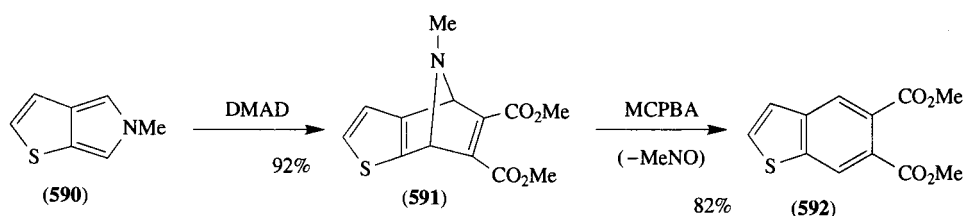


Scheme 121

Thieno[2,3-*c*]furan and thieno[2,3-*c*]pyrrole also behave as thiophene-2,3-quinodimethane equivalents. Thus, intramolecular Diels–Alder reaction of the furan (**588**) followed by acid-catalyzed dehydration affords the benzo[*b*]thiophene (**589**) in good overall yield (Scheme 122) <88TL1137>. The Diels–Alder reaction of the pyrrole (**590**) with DMAD followed by oxidation of the resulting adduct (**591**) affords the benzo[*b*]thiophene (**592**) with loss of nitrosomethane (Scheme 123) <90JOC2446>.



Scheme 122

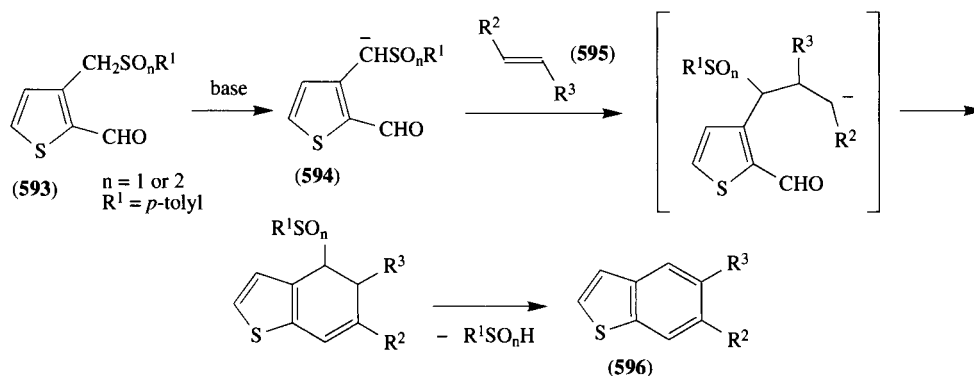


Scheme 123

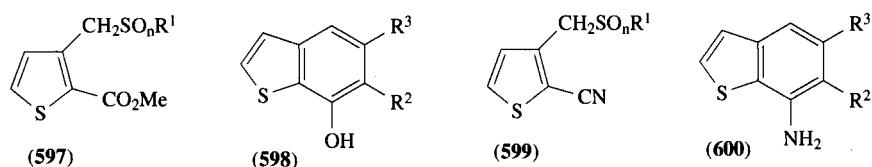
2.11.6.2 Condensation of 2,3-Disubstituted Thiophenes with Michael Acceptors

A full detail of the widely applicable practical synthesis of benzo[*b*]thiophenes was reported <81TL5097, 86JOC230>. The thiophenes (**593**) with two substituents, one electrophilic and one poten-

tially nucleophilic, react with Michael acceptors (**595**) in the presence of base to give benzo[*b*]-thiophenes (**596**) in reasonable yields in one pot (Scheme 124). The reaction proceeds via Michael addition of the carbanion (**594**) to the acceptors (**595**) followed by cyclocondensation and aromatization by loss of *p*-toluenesulfenic or toluenesulfinic acid. Application of the reaction to the ester derivatives (**597**) allows the preparation of the hydroxy derivatives (**598**) in 13–74% yields (17 examples) and application to the nitrile derivatives (**599**) allows the preparation of amino derivatives (**600**). Fumaric acid, dimethyl fumarate, dibenzoyl ethylene, chalcone, methacrylic acid, methyl methacrylate, and methyl cinnamate are the Michael acceptors employed in the present synthesis.

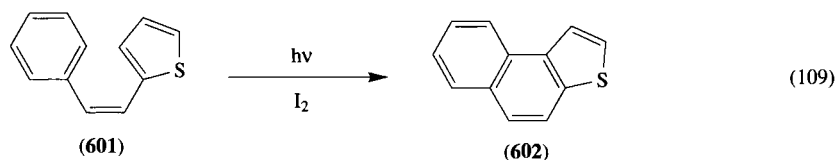


Scheme 124



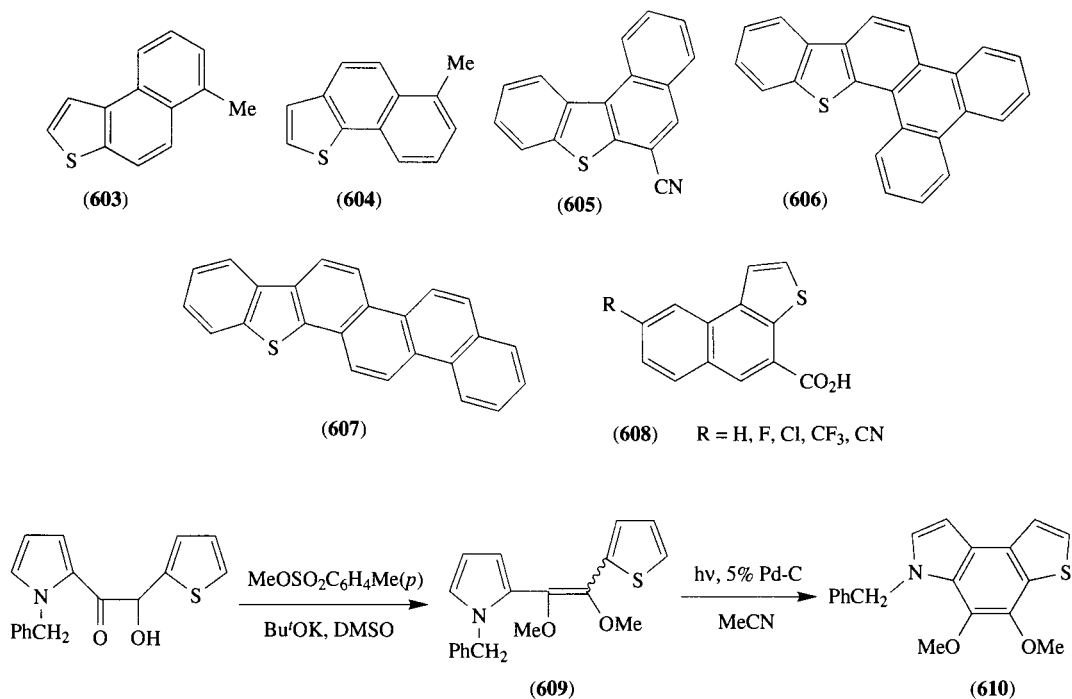
2.11.6.3 Oxidative Photocyclization of 1-Aryl-2-(thienyl)ethylenes

1-Aryl-2-(thienyl)ethylenes undergo an oxidative photocyclization to give benzoannulated thiophenes as is exemplified in the conversion of **(601)** to **(602)** (Equation (109)). The use of the *cis*-isomer is not necessarily required because the *trans*-isomer isomerizes to the *cis* on irradiation. The irradiation is commonly carried out in the presence of iodine which serves as the hydrogen acceptor (oxidant). By application of this method, condensed thiophenes such as **(603)**–**(608)** were synthesized <83JHC487, 83JHC861, 83JHC1453, 84JHC1215, 84JHC1775, 84JHC1833, 85JHC1229>. An interesting application is the preparation of the thiophene **(610)** by irradiation of **(609)** in the presence of palladium-charcoal as the dehydrogenating agent (64% yield based on the hydroxyketone) <86CC826>. The thiophene **(610)** serves as the precursor compound of the antitumour agent CC-1065.



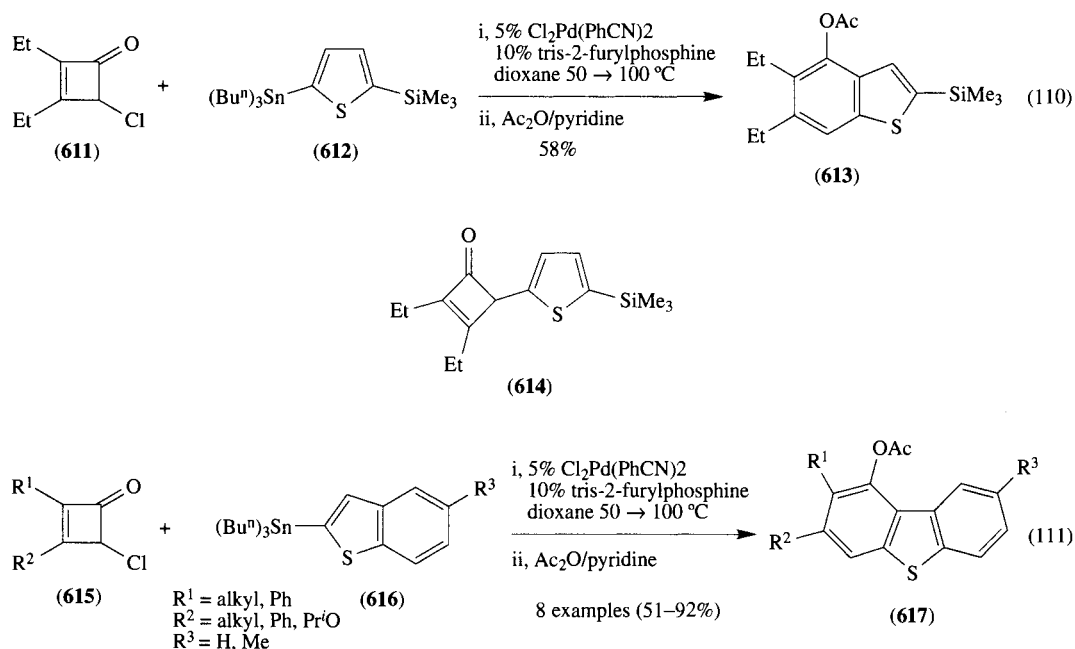
2.11.6.4 Condensation of 2-(Tri-*n*-butyl)stannylthiophene with 4-Halo-2-cyclobutenones

4-Chloro-2,3-diethyl-2-cyclobutenone (**611**) undergoes a palladium-catalyzed cross-coupling with 2-(tri-*n*-butyl)stannyl-5-trimethylsilylthiophene (**612**) to give the intermediate compound **(614)** (Equation (110)). Without isolation, **(614)** is converted to the benzothiophene **(613)** on thermolysis at 100°C followed by acetylation. Although this procedure seems not to be generally applicable to benzo[*b*]thiophene synthesis, it provides a general synthesis of dibenzothiophenes, and thus a series



Scheme 125

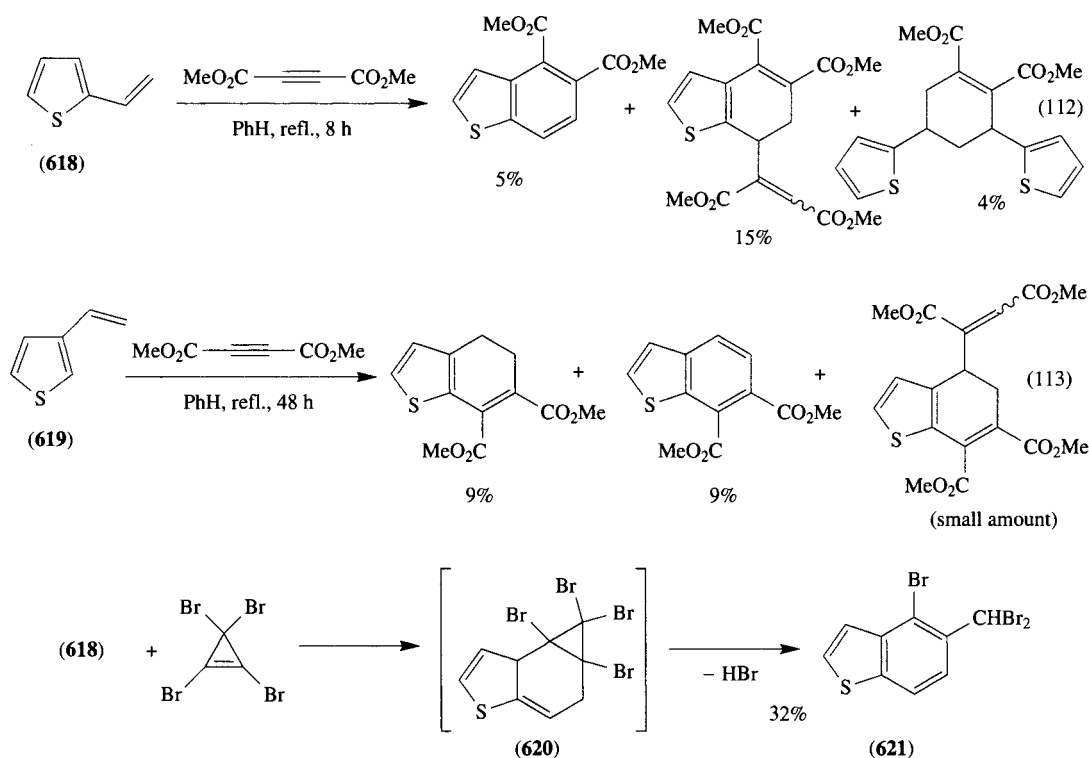
of dibenzothiophenes (617) could be prepared via coupling of (615) and (616) in good yields in a one-pot procedure (Equation (111)) <93JOC3550>.



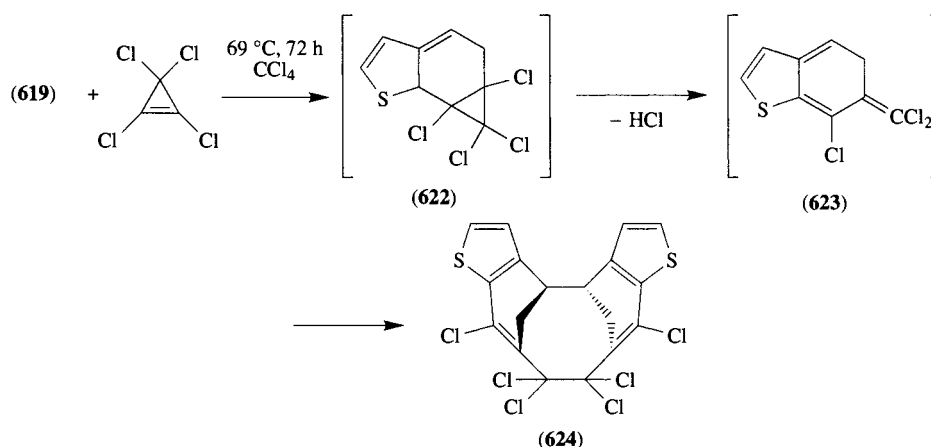
2.11.6.5 Diels–Alder Reactions of Vinylthiophenes and Related Compounds

2-Vinyl- and 3-vinyl-thiophenes (618) and (619) behave as a diene on reaction with dienophiles to give six-membered ring fused thiophenes (Equations (112) and (113)). The reaction generally gives a complex mixture as exemplified by reactions of (618) and (619) with DMAD <85T2435, 87T269,

87T991). Cycloadditions with cyclopropenes are of particular interest. Thus, the reaction of (618) with tetrabromocyclopropene affords the benzothiophene (621) via rearrangement of the initial adduct (620) (Scheme 126) <90TL4581>, while the reaction of (619) with tetrachlorocyclopropene produces the structurally interesting thiophene (624) via intermediates (622) and (623) (Scheme 127) <94TL7923>. [2 + 4] Cycloadditions of 2-vinylbenzo[*b*]thiophene with dienophiles <89IJC(B)724> and those of 2-vinyl-4,5-dihydrothiophene 1,1-dioxides with *p*-benzoquinones <89ZOR1231> were also reported.



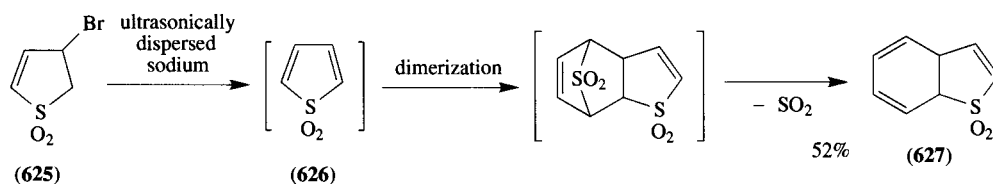
Scheme 126



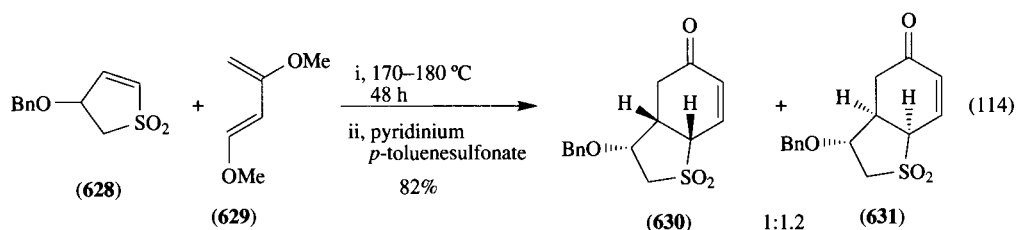
Scheme 127

Many thiophene 1,1-dioxides including the parent compound (626) are thermally labile and undergo dimerization to give six-membered ring fused thiophene 1,1-dioxides. Thus, the parent compound, generated by dehydrobromination of (625), dimerizes to give the compound (627) along with other products (Scheme 128) <87H(26)2829>. Treatment of the cycloadduct of the dihydro-

thiophene dioxide (**628**) and the diene (**629**) with pyridinium *p*-toluenesulfonate results in desilylation with concomitant loss of methanol to give compounds (**630**) and (**631**) (Equation (114)) <93TL4281>.

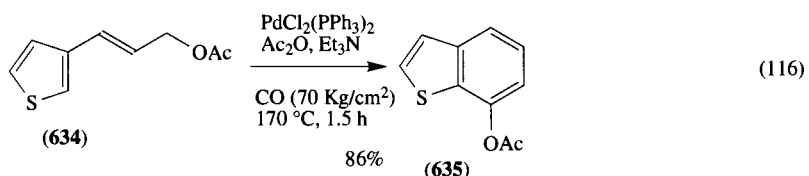
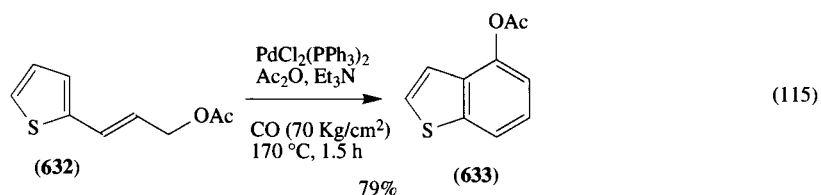


Scheme 128

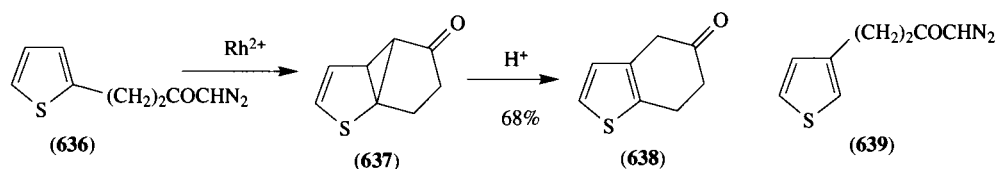


2.11.6.5.1 Miscellaneous cyclizations

Palladium-catalyzed cyclocarbonylation of 3-(2-thienyl)allyl acetate (**632**) and 3-(3-thienyl)allyl acetate (**634**) produces acetoxybenzothiophenes (**633**) and (**635**), respectively, in good yields (Equations (115) and (116)) <89TL95>.



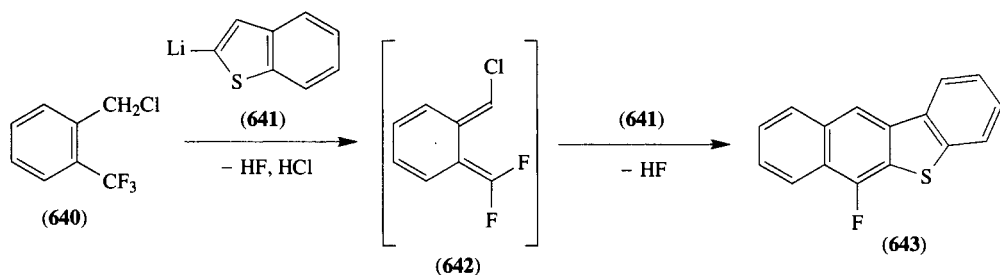
Rhodium-catalyzed decomposition of the diazoketone (**636**) affords the three-membered ring compound (**637**), which isomerizes to (**638**) on attempted purification with silica-gel column chromatography (Scheme 129) <89JOC299>. Thermolysis of the isomeric diazoketone (**639**) was also reported.



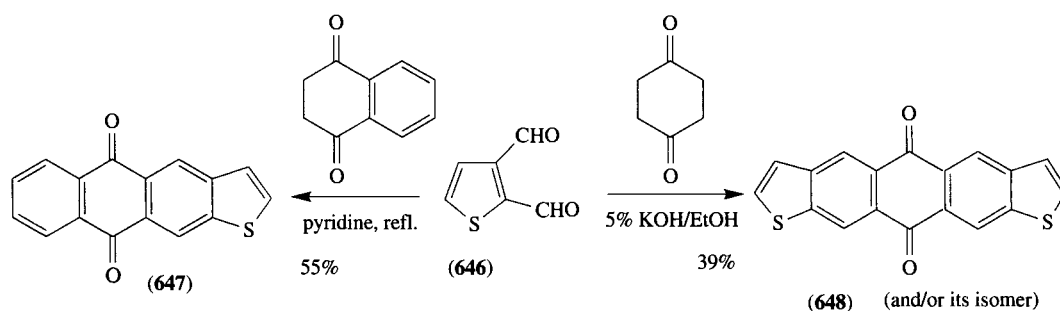
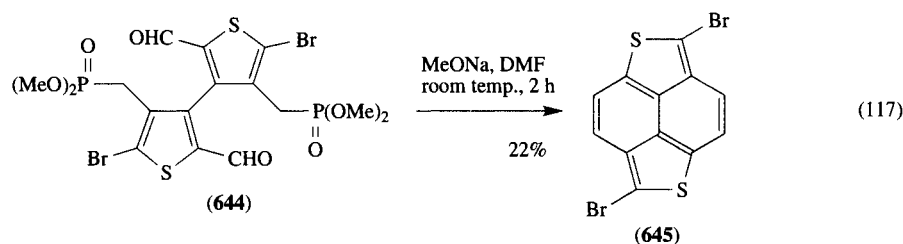
Scheme 129

The quinodimethane (**642**), generated by reaction of (**640**) with 2-benzo[*b*]thienyllithium (**641**), condenses with (**641**) to give the polycyclic thiophene (**643**) (Scheme 130) <94TL7597>. Intramolecular Horner reaction of (**644**) affords the polycyclic thiophene (**645**) (Equation (117)) <94CC1859>. Condensation of thiophenedialdehyde (**646**) with 1,4-dicarbonyl compounds leads to benzothiophenes

carrying two carbonyl functionalities (Scheme 131). Thus, condensation of (646) with 1,4-dihydroxynaphthalene affords the quinone (647) and that with cyclohexane-1,4-dione the quinone (648) <92JOC6192>. Condensation of (646) with (649) in acetic acid gives compound (650) <87SUL99>.

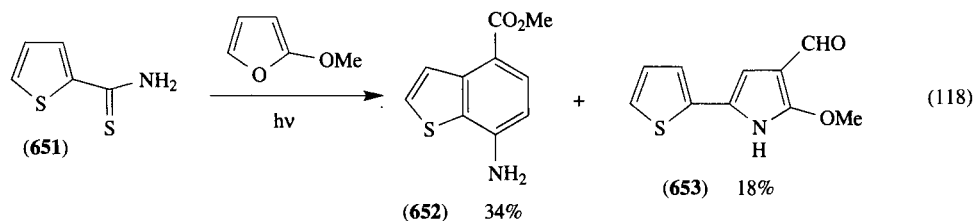


Scheme 130

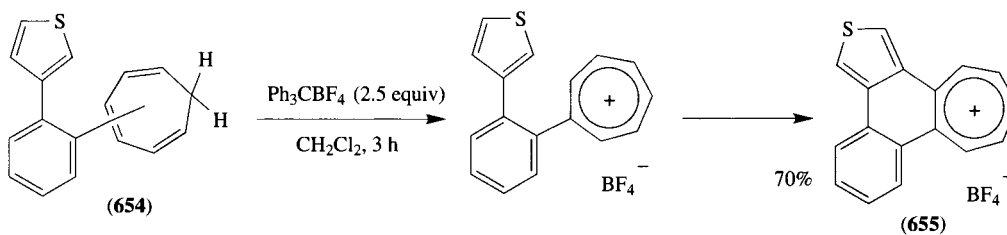


Scheme 131

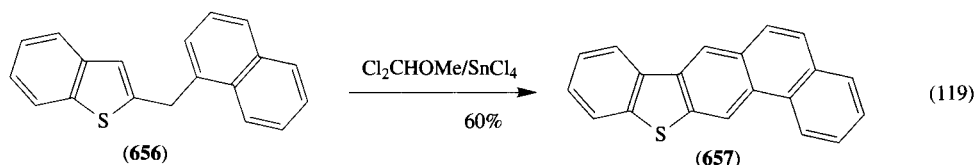
Photoirradiation of the thioamide (651) with 2-methoxyfuran in benzene solution affords the benzo[*b*]thiophene (652) along with the thienylpyrrole (653) (Equation (118)) <94CC1477>. The reaction is applicable to the preparation of other benzoannelated aromatic compounds though in moderate yields.



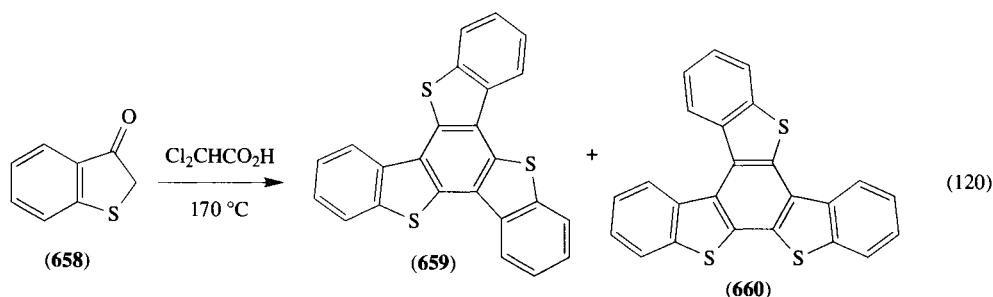
Intramolecular Friedel–Crafts reactions have been often employed for annelation of benzene ring as exemplified in the conversion of (654) to the compound (655) with an 18 π -electron system (Scheme 132) <92CL1213> and conversion of (656) to the polycondensed thiophene (657) (Equation (119)) <83JHC861>.



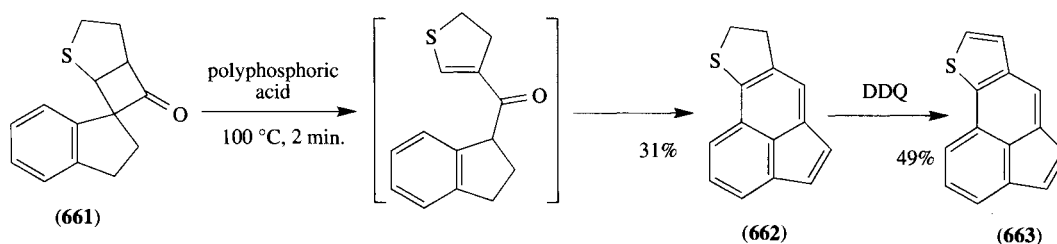
Scheme 132



The cyclocondensation of the benzothiophenone (**658**) under optimized conditions affords the cyclotrimers (**659**) and (**660**) in acceptable yields (Equation (120)) $\langle 86\text{T}763 \rangle$. Cyclotrimerization of 2-acetoxymethylbenzo[*b*]thiophene was also reported $\langle 86\text{CS}287 \rangle$.



Treatment of the compound (**661**) with hot polyphosphoric acid results in the rearrangement to give the dihydrothiophene (**662**), which is converted to the thiophene (**663**) by dehydrogenation (Scheme 133) $\langle 86\text{JHC}1551 \rangle$.



Scheme 133

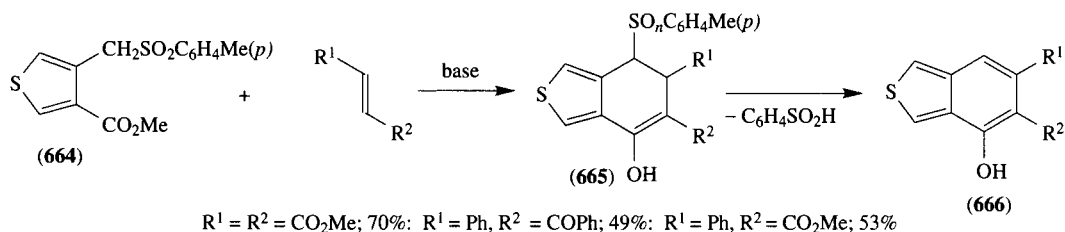
2.11.7 BENZO[*c*]THIOPHENES BY ANNEALATION OF THIOPHENES

Michael addition of the carbanion, generated from the thiophene (**664**), to the electron-deficient alkenes followed by cyclocondensation leads to compounds (**665**), which expel *p*-toluenesulfinic acid to produce the benzo[*c*]thiophenes (**666**) in one pot (Scheme 134) $\langle 86\text{JOC}230 \rangle$. Application of the procedure to the thiophene (**667**) allows the preparation of the corresponding amino derivatives.

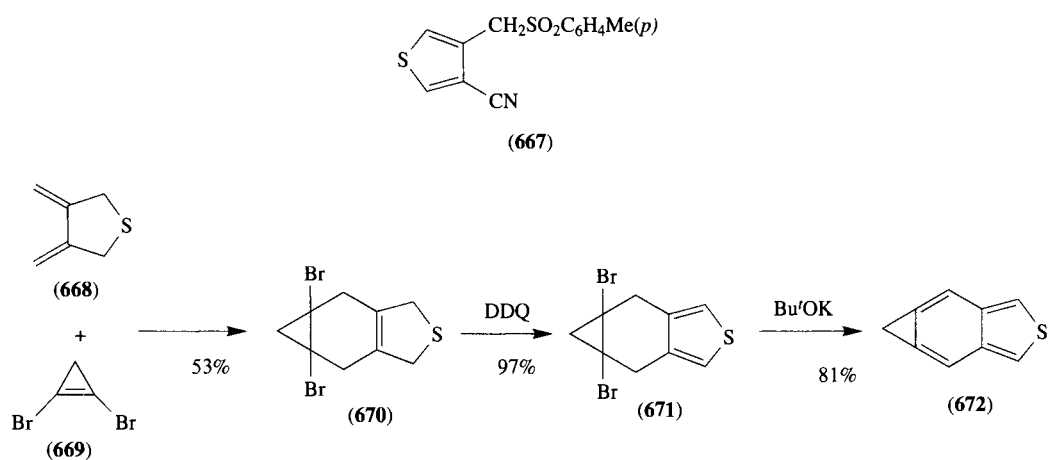
Diels–Alder reaction of the diene (**668**) and the cyclopropene (**669**), both generated *in situ*, affords compound (**670**), whose dehydrogenation and dehydrobromination produces the thermally labile benzo[*c*]thiophene (**672**) as a yellow solid (Scheme 135) $\langle 87\text{TL}4217 \rangle$. The singlet biradical (**673**) also

behaves as a diene and reacts with dienophiles to give six-membered ring-fused thiophenes such as **(674)** (Scheme 136) <88JA4050, 89JA3659, 89JA8732>.

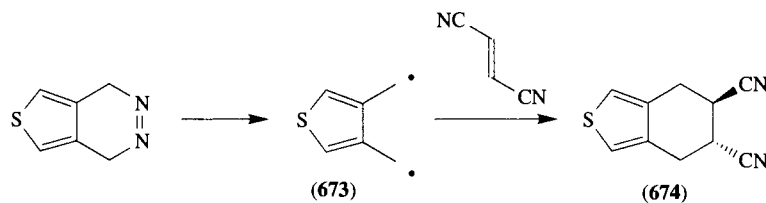
The Wittig reaction of the quinone **(675)** with ylide generated from **(676)** gives the benzo[*c*]-thiophene **(677)** in moderate yield (Equation (121)) <86JCS(P1)415>. Conversion of the diol **(678)** to the benzo[*c*]thiophene **(679)** was also reported <85MI 211-01>.



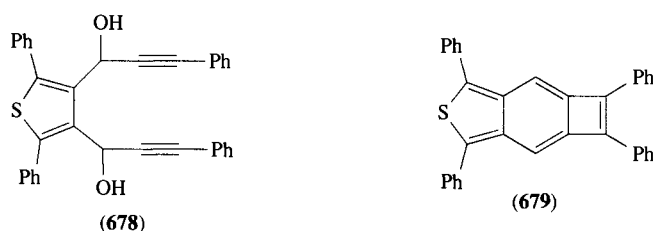
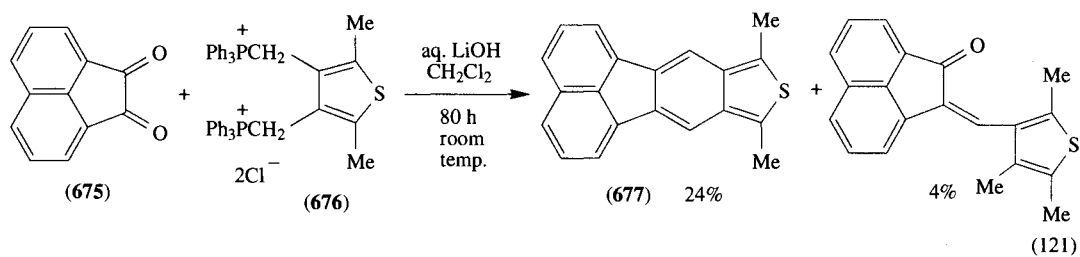
Scheme 134



Scheme 135



Scheme 136



2.12

Thiophenes and their Benzo Derivatives: Applications

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2.12.1 INTRODUCTION

The applications of thiophene and benzothiophene derivatives were not well exemplified in the first edition of *Comprehensive Heterocyclic Chemistry* (CHEC-I). There have been numerous examples of utilizing thiophene derivatives, both of natural and synthetic origins, for a variety of purposes since the 1950s. In fact, the number and variety of uses preclude an encyclopedic account of all examples, even within the scope of this expanded chapter. In addition, the complexity of the literature search suggests that there are numerous examples of applications of thiophenes that were probably not found.

The search strategy utilized both "keywords" and "section" searches to produce approximately 8700 "hits" between 1982 and 1993. Refinement reduced this number to around 2000 references and manual scanning further reduced the number to about 1200. Very few reports of synthesis results are included except where these reports clarify the topic. Review articles that focus on specific areas are covered in this chapter and should be consulted for more detail.

From the standpoint of molecular complexity, the most intriguing applications of thiophene and its derivatives focus either on the occurrence of the thiophene moiety in naturally occurring molecules or in pharmacological agents. Transformation of thiophene into other moieties also produces some remarkable molecules. From a commercial view, the unique electronic and physical properties of thiophene make it ideal for applications in polymers, liquid crystals and dyes. Examples of thiophene-containing cyclophanes and other macrocycles as well as organometallic derivatives complete this chapter.

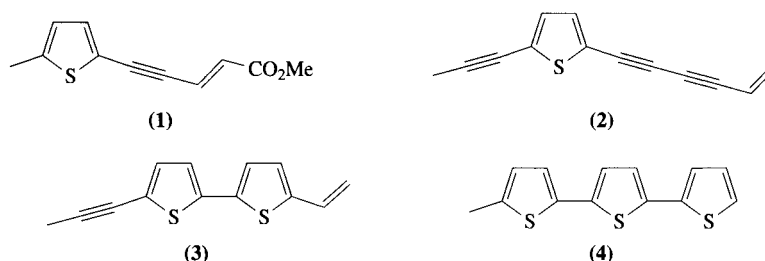
While no further discussion follows in this chapter, it should be noted that the unique coordination and stabilization properties of thiophene have made it essential as a nontransferable ligand in the chemistry of "higher order" organocuprates <84T5005, 87S325, 87TL945>. An excellent method of preparation of these reagents is reported <88JOC5366>. The direct formation of the synthetically useful thienyl-based organocopper reagent avoids the use of phosphine ligands which frequently interfere with product isolation and purification <93JOC2492>.

Another aspect of thiophene chemistry that also fails to fit into the above-mentioned categories merits mention. Thiophene-derived zero-valent copper is described as superior to phosphine-based organocopper reagents. Direct reduction of lithium 2-thienylcyanocuprate gives an intermediate that reacts with alkyl halides in high yields at -78°C . These alkyl copper reagents cross-couple with acyl halides, as well as undergoing other reactions with much easier product isolations than those derived from phosphine-based chemistry <89SC1833>.

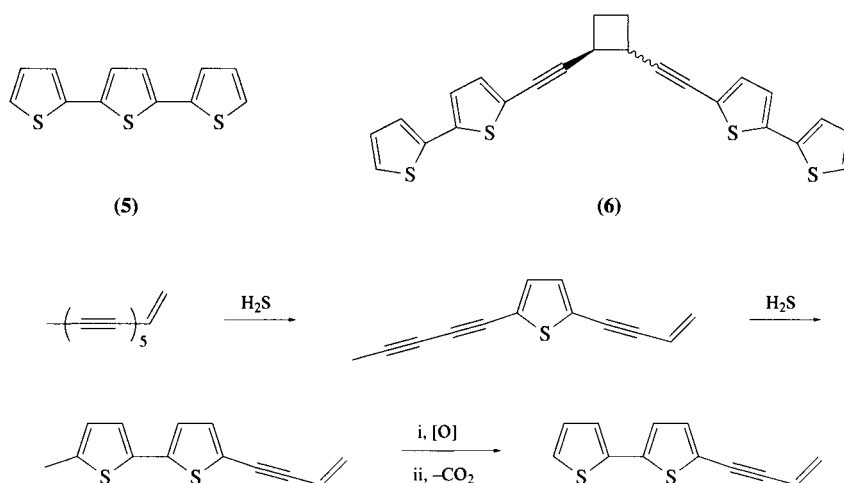
2.12.2 NATURALLY-OCCURRING DERIVATIVES

Thorough reviews of naturally-occurring thiophene derivatives isolated from coals, petroleum and shale oil <85HC(44)325> as well as from plants and fungi <85HC(44)261> establish the backgrounds in these fields of research. Focusing on biologically produced thiophene derivatives, the review <85HC(44)261> identifies more than a 150 thiophene derivatives from *Tagetes erecta* L. Monothiophenes such as (1) form from C_{10} acetylenes while monothiophenes (such as (2)), bithienyls

(such as (3)) and terthienyls (such as (4)) form from C_{13} acetylenes as well as from C_{13} and C_{14} triynes. Some of these materials are postulated to arise from dehydromatricaria ester and crepenynic acid, as determined by feeding experiments with labeled precursors. These compounds are believed to be part of the chemical defense mechanisms in plants having phototoxic, cytotoxic and insecticidal activities.



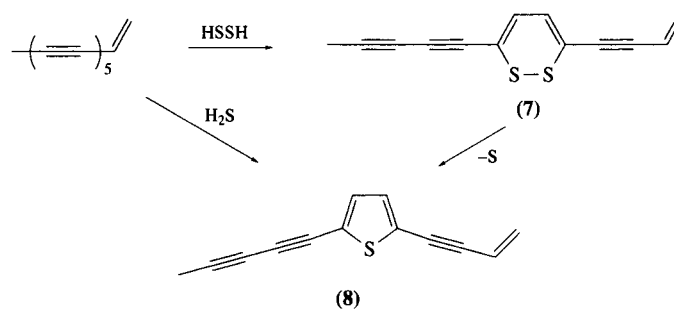
A thorough enumeration of all related unsaturated compounds from the tribe Cyanareae (Asteraceae) as well as their biosynthesis and biological properties appeared in 1990 <90P2753>. Among the numerous compounds in this report, the UV-light-induced phototoxicity of α -terthienyl (5) is noteworthy and has been more thoroughly studied since its first report <72MI 212-01>. Compound (5) is toxic to a variety of organisms, plants and even human erythrocytes through acting on membrane proteins. One of the more unusual compounds in this family of compounds is the cyclobutane (6). Several biosynthetic pathways are described; the formation of the C_{11} -alkynic thiophenes is representative (Scheme 1).



Scheme 1

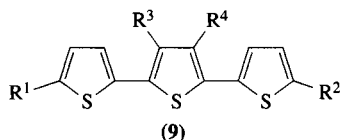
In a subsequent detailed report, alkynes and related compounds isolated from Heliantheae were discussed <91P11>. The Heliantheae are among the largest tribes of the Asteraceae. While there are many compounds in this group similar to those mentioned for Cyanareae, there are several examples of 1,2-dithiins which differ from those compounds. Most interesting is the formation of thiarubrine B (7) which forms as part of the proposed biosynthetic pathway of C_{13} -thiophenes including (8) (among many other products) (Scheme 2). Thiarubrine B (7) has recently been the subject of interest because of its light-enhanced activity against human immunodeficiency virus (HIV) as well as its antiviral, antibacterial and nemocidal activity. A total synthesis of (7), a light sensitive, deeply colored 8π -electron antiaromatic compound also isolated from giant ragweed, has been achieved <94JA9403>.

Tagetes spp. (marigolds) have been used in various applications such as dyes, perfumes and medicinal agents since antiquity <93MI 212-01>. Studies of one constituent, α -terthienyl (5), have progressed since its first synthesis <41LA180> and isolation from plants <47JA273>. The biocidal activity of (5) and related compounds has led to the study of cellular mechanisms both to identify biological model systems as well as to investigate improved synthetic therapeutic agents. One of the most interesting issues posed by the activity of these compounds is how a class of known photo-

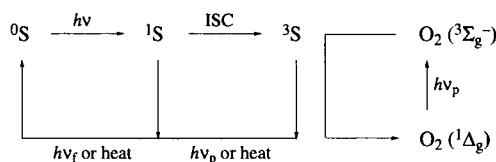
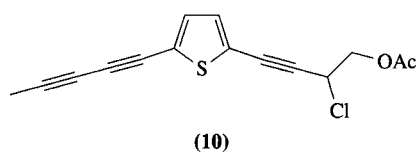


Scheme 2

sensitizers can have activity in the root systems of plants where there is little or no light exposure. A synthesis program has examined potentially useful phototoxic insecticides and model phototoxins for plant-insect interactions based upon substitution effects on alpha-T (**5**) as outlined (**9**) <89MI 212-01>. Conclusions from this study show that these derivatives have high quantum yields for production of singlet oxygen, a contributor to their toxicity. However, the toxicity of the compounds is not fully predicted solely on singlet oxygen yields.



The antiviral properties of several of these compounds including alpha-T, the nonthiophene-phenylheptatriyne, thiarubrine-A (an isomer of (**7**)), ACBP-thiophene (**10**) and related compounds arise from markedly different mechanisms of action <88MI 212-01>. A quantitative structure-activity relationship analysis of the phototoxic thiophenes and acetylenes from *Crysanthemum* spp. has attempted to describe the relationship between physicochemical properties and phototoxicity <91MI 212-01>. The *in vitro* and *in vivo* phototoxicity of these thiophene derivatives involves an oxygen-dependent type II mechanism in which a ground-state sensitizer (such as (**5**)) is excited by photon absorption and ultimately forms a triplet excited state which then produces reactive singlet oxygen as depicted in Scheme 3 <84MI 212-01>. A simple SAR model involving rates of formation of singlet oxygen and hydrophobicity accounts for 80% of the activity of these derivatives.



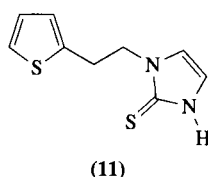
Scheme 3

2.12.3 BIOLOGICALLY ACTIVE MOLECULES

The design and synthesis of pharmacologically active agents is the task of medicinal chemists in academia and industry. The technologies of the 1990s including molecular modeling, molecular biology, and biotechnology as a means to produce pure receptors in larger amounts have allowed

the pursuit of sophisticated experimental approaches to drug discovery. Despite these powerful new tools, the end result, a selective pharmacological agent which is systemically effective, stable, deliverable and nontoxic remains a daunting goal.

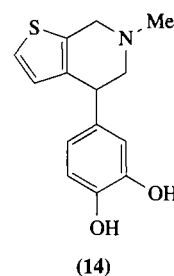
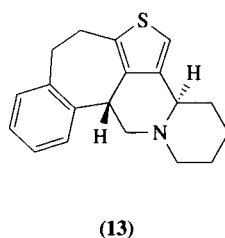
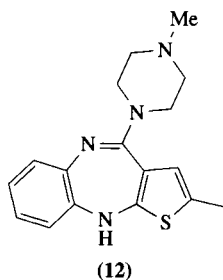
In this context, thiophene derivatives have been studied for decades using the concept of bioisosterism <79CSR563, 92C323> wherein the aromatic moiety of known drug agents is replaced by the heteroaromatic thiophene ring. Comparison of both the electronic and geometric differences of the thiophene ring as compared with the benzene ring suggests that replacement with thiophene may produce agents that add to the SAR studies of active series by more than simple isosteric replacement. A most interesting discovery along these lines is the dopamine β -hydroxylase inhibitor (**11**) which causes reduction of blood pressure after oral administration in rats while the benzene derivative has significantly less activity. What makes this compound unique is the conclusion that thiophene may not always be a bioisostere for phenyl <90JMC1866>.



Several very extensive reviews of biologically active thiophene derivatives <85HC(44)353, 91HC(44)397> as well as some reviews of lesser scope <83SR171, 84PHA4, 87MI 212-01> have appeared since the first edition of *Comprehensive Heterocyclic Chemistry*. In light of these reviews, this section will focus on discovery research reports of biologically active molecules during the first half of the 1990s as well as thiophene derivatives that have been approved for use by regulatory agencies since 1983.

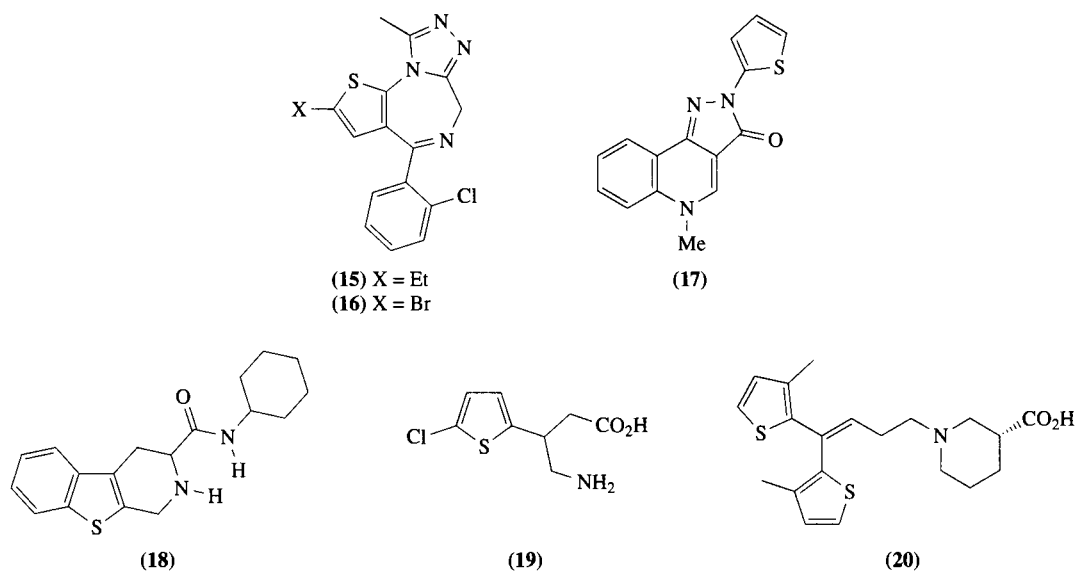
2.12.3.1 CNS Agents

A preponderance of reports of biologically active thiophene derivatives arises in the area of CNS research. In the area of antipsychotic (neuroleptic) therapy, olanzepine (**12**) is nearing NDA submission as a therapeutic agent with reduced extrapyramidal side-effects (EPS) similar in profile to clozapine. Studies of conformational restriction of thieno[2,3-*b*][1,5]benzodiazepine analogues of (**12**) show that antidopaminergic and anticholinergic activities of the series are modulated by steric interference with the piperazine ring <82JMC1133>. A number of thieno[3,2-*b*][1,5]benzoxazepine and -benzothiazepine derivatives, the thiophene positional isomers of (**12**), were made as analogues of clozapine. 7-Chloro substitution on these derivatives reduces propensity for undesired EPS <92AF896>. The taclamine thiophene analogue (**13**), QM-7184, has interesting neuroleptic activity while also producing α -adrenergic blockade. Although (**13**) is less potent than typical neuroleptic drugs such as haloperidol, its effects on the adrenergic system are speculated to be particularly interesting for further study in schizophrenia <83AF1417>. SKF 89145 (**14**) was discovered as a part of a program investigating selective D_1 agonists <87MI 212-02, 87MI 212-03>. Careful studies of (**14**) and its thiophene isomer SKF 89641 show it is a partial D_1 agonist with greater efficacy than the benzene isostere <89MI 212-02>.



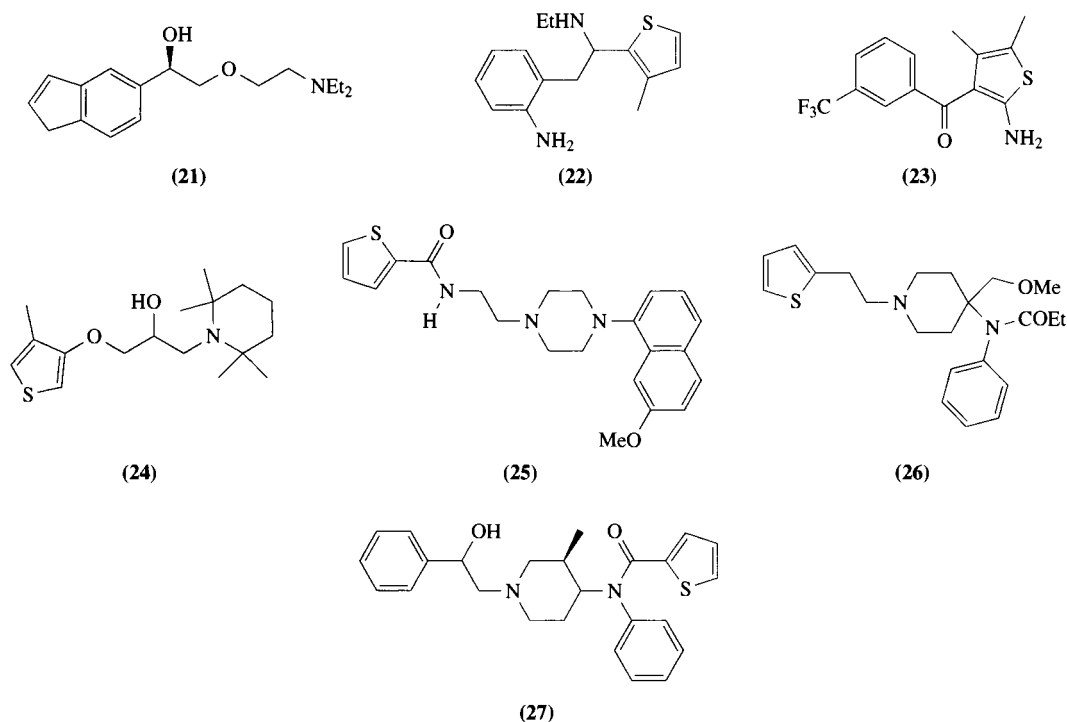
Etizolam (**15**) was approved for use by Yoshitomi in 1984 with an antianxiety (anxiolytic) therapeutic profile similar to that of diazepam. Studies of the behavioral effects of brotizolam (**16**) suggest that it has anxiolytic effects with potency between that of diazepam and nitrazepam <84MI

212-02). These effects include antagonism of pentylenetetrazol-induced convulsions and electroshock seizures as well as sleep potentiation (84MI 212-03). Compound (16) is effective at improving poor sleep induced by high altitude (84MI 212-04). Nonbenzodiazepine derivatives also have anxiolytic potential. A series of thienylpyrazoloquinoline derivatives shows that (17) and its isostere have equal affinity for the benzodiazepine (BZ) receptor but that they cause opposite behavioral effects. This phenomenon is postulated to result from inverse agonism at the receptor (88JMC1738). Of a series of benzothieno[2,3-*c*]pyridines, AP159 (18) is among the most potent in anticonflict and memory impairment behavioral assays. Despite its bioisosterism with β -carboline, (18) binds not to BZ but rather to 5-HT_{1A} receptors (90JMC3110). Only the (*R*)-isomers of these derivatives selectively bind to the 5-HT_{1A} receptor (93JMC3526). The BZ receptor is an allosteric regulatory site of the γ -aminobutyric acid (GABA) receptor. 3-Aryl-GABA congeners are specific ligands for bicuculline-insensitive GABA_B receptors. The racemic β -heteroyl-GABA analogue (19) is a particularly effective ligand (91JMC2557). Derivative (19) antagonizes ileal GABA_B receptors but is a full agonist at central receptors (92MI 212-01) and discriminates between GABA_A and GABA_B sites (93MI 212-02). Identification of a novel class of GABA uptake inhibitors has led to the developmental candidate, tiagabine (20), as an anticonvulsant (93JMC1716).

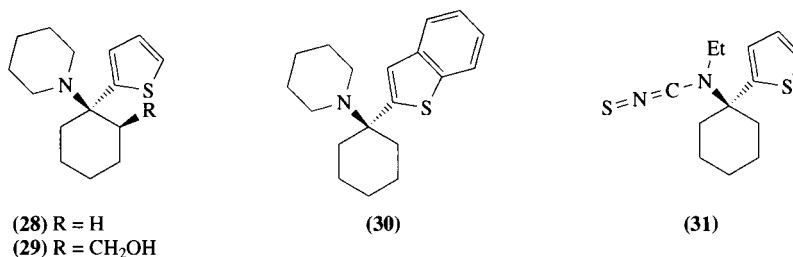


Compound (21) (T-588) is a cerebral activator with effects on normobaric hypoxia, KCN-induced anoxia, and ischemia (93MI 212-03). Activation of the CNS cholinergic system is at least one of the mechanisms of action of (21). Compound 8319 (22) is a novel noncompetitive *N*-methyl-D-aspartic acid (NMDA) antagonist and has neuroprotective and anticonvulsant properties in preclinical biological screens (90MI 212-01). A series of 2-amino-3-benzoylthiophene derivatives were prepared as adenosine A₁ agonists, of which PD 81,723 (23) was the most interesting (90MI 212-02). Thiophene analogues of the local anesthetic lotucaine such as (24) also have platelet antiaggregatory properties (87FES267). Derivative S-14671 (25) is an exceptionally potent 5-HT_{1A} antagonist with 5-HT_{1C} antagonist properties (91MI 212-02). Sulfentanil (26) is a narcotic analgesic with greater potency and better therapeutic ratio than fentanyl; it was first marketed in the mid-1980s. SAR studies of (26) examining effects of replacing the methoxy moiety with varying acyl groups have been reported (89JMC968). The 2-thiophenecarboxamide derivative (27) as well as its 3-isomer have high μ -opioid vs. δ -opioid selectivity (92MI 212-02).

As part of an extensive study of phencyclidine (PCP), which is one of the most widely abused CNS drugs, the thiophene isostere (TCP, (28)) has become the preferred ligand for characterization of the PCP-binding site. Continuation of these studies has shown that *cis*(29) is an NMDA receptor antagonist which is similar to TCP in affinity for the PCP binding site and 38-fold more potent than the *trans*-isomer (91CPB1581). The benzo[*b*]thiophene isostere of PCP (BTCP, GK13, (30)) is a noncompetitive NMDA antagonist and a potent dopamine (DA) uptake inhibitor but has low affinity for the PCP receptor (89MI 212-03). Isocyanate (31) is the most efficient alkylating agent of a series of irreversible affinity ligands for the PCP recognition site of the NMDA receptor (92MI 212-03). [³H]-(28) labels two affinity binding sites in the human cortex (91MI 212-03, 86MI 212-01). [³H]-(30) also binds to two brain sites and further study shows that PCP interacts with DA-uptake sites



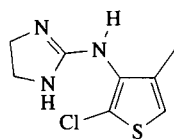
but that these sites are not the PCP receptor <88MI 212-02>. Some molecular modeling investigations led to the synthesis of several piperazine analogues of (30) which have high binding affinity <93EJM893>. Other replacements for the piperidine moiety of (28) include pyrrolidine and morpholine derivatives <88MI 212-03>. Several papers discuss the synthesis and biological evaluation of homologues of (30) at dopamine-uptake, PCP- and σ -binding sites <93JMC1188, 93JMC4075>.



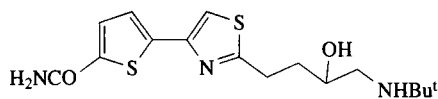
2.12.3.2 Pharmacodynamic Agents

Advances in understanding the mechanistic basis of cardiovascular disease have led to significantly more potent and selective therapeutic agents. Drugs which have gross systemic effects have been replaced with agents such as selective potassium channel agonists, calcium channel antagonists, α_1 -antagonists, α_2 -agonists, 5-HT₁ antagonists, etc. Thiophene derivatives have been prepared for many of these therapies. Tiamenidine (32) is a centrally-acting, α_2 -agonist antihypertensive agent approved for marketing in the late 1980s for mild to moderate hypertension. Arotinolol (33) is a selective β -adrenergic blocker with weak α -antagonistic activity which was also marketed in the late 1980s as an antihypertensive agent. Azosemide (34) was marketed in the mid-1980s as a high ceiling diuretic agent with less potency than furosemide; it is indicated for the therapy of congestive heart failure (CHF).

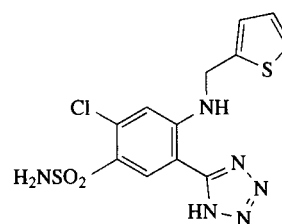
One of the most dramatic discoveries in the 1980s in this therapeutic area was the role of potassium channel openers (KCOs) as smooth muscle relaxants. These agents have potential for therapy in hypertension, angina, and asthma. Racemic RWJ 26629 (35) is among the most potent KCOs



(32)

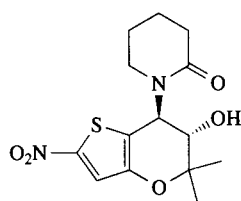


(33)



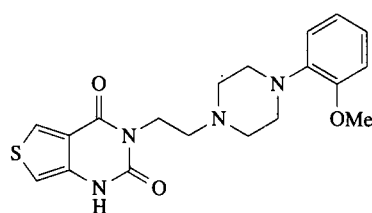
(34)

reported <92JMC4425>. Replacement of the piperidone moiety of (35) with other amide substituents also produces potent potassium channel openers <93BMC1385>. While the thieno[2,3-*b*] isomers of (35) are at least as potent as the [3,2-*b*] derivatives, the thieno[3,4-*b*] isomers are significantly less interesting <93BMC423>.

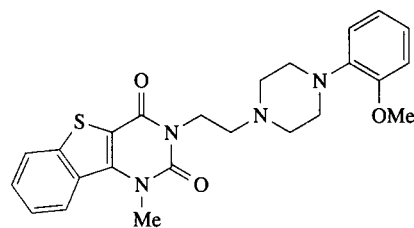


(35)

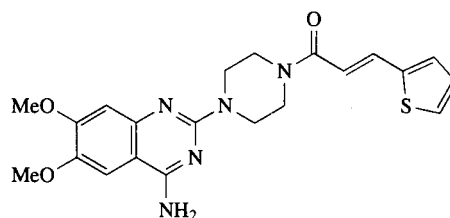
Although α_1 -antagonists have been known for a number of decades to lower blood pressure, their rapid onset of peripheral vasodilation with concomitant postural hypotension has limited their therapeutic potential. The success of prazosin has led to renewed interest in this therapy. Compound (36) was prepared as part of a program examining thieno[3,4-*b*]-, thieno[3,2-*b*]- and thieno[2,3-*b*]pyrimidinediones; it is significantly more potent than prazosin in reducing blood pressure <88JMC1786>. Benzo[*b*]thiophene (37) not only has potency at the α_1 receptor, but also is very active at the 5-HT_{1A} receptor <93EJM499>. Interestingly, although *N*-alkylation of (36) produces less potent derivatives, *N*-methylation does not potentiate the α_1 antagonism of (37). Derivative (38) was part of an SAR study of antihypertensive effects of quinazoline compounds <83JMC411>.



(36)



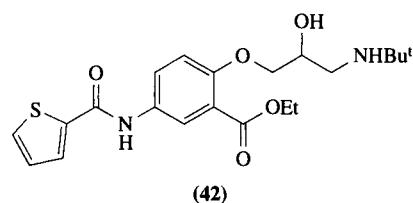
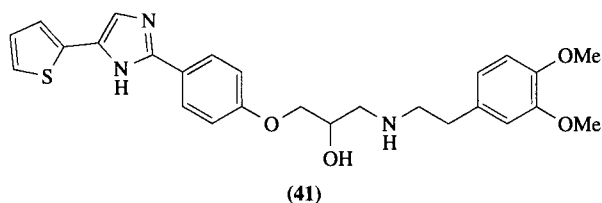
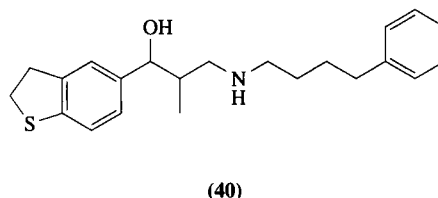
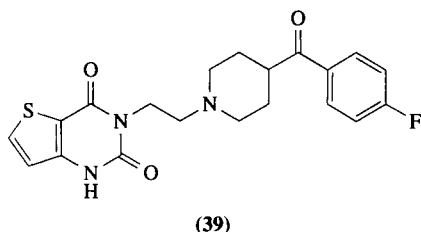
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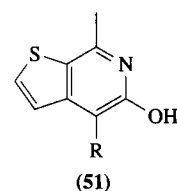
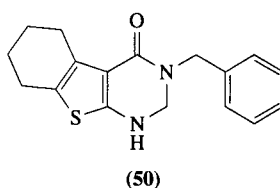
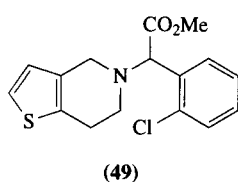
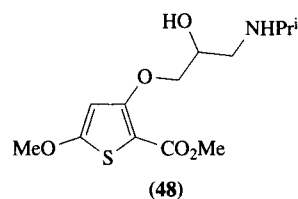
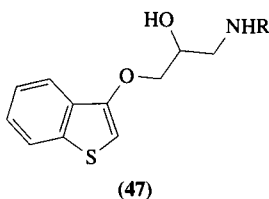
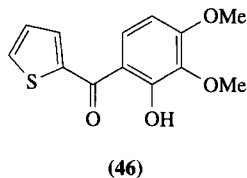
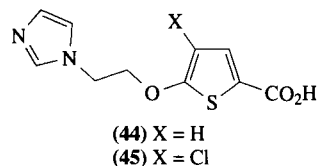
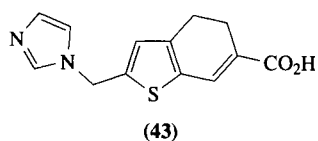
(38)

The role of serotonin (5-HT) in the cardiovascular system is also an active subject of investigation. Synthesis of the thiophene isosteres of ketanserin shows that the thieno[3,2-*d*] isomer (39) is ≈ 4 -fold more selective for 5-HT₂ vs. 5-HT_{1A} or α receptors than is ketanserin <91EJM807>. Tibalosine (CP 804 S, (40)) is a highly selective α_1 -adrenoceptor antagonist which is significantly less potent than prazosin <83MI 212-01>. Compound (41) is more than 100 times more selective than atenolol

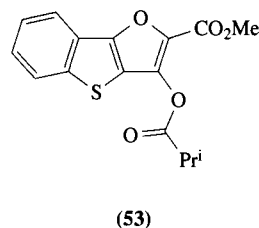
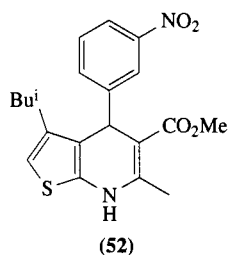
for the β_1 -adrenergic receptor and was chosen for development studies <83JMC950>. Tienoxolol (**42**) is an effective diuretic and β -adrenergic receptor blocking agent <86JMC100>.



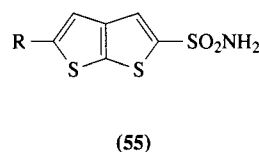
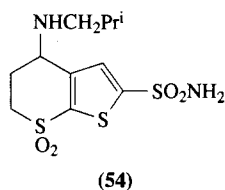
Control of thrombosis has been a research target for many years. Thromboxane synthetase inhibition represents one approach to such antithrombotic therapy. Compound CS-518 (**43**) reversibly inhibits TXB_2 production in whole blood in a concentration-dependent manner more effectively than ozagrel <91AF506>. Two thiophene isosteres (**44**) and (**45**) of dazoxiben are specific inhibitors of thromboxane synthetase in human platelets with approximately the same potency and activity as dazoxiben <85MI 212-01>. Aryl thienyl ketones have antiplatelet effects as a consequence of interfering with cyclooxygenase in the arachadonic acid cascade <92MI 212-04>; (**46**) is 25 times more active than aspirin <86EJM255>. Some pyrazoline isosteres of the ketone moiety of (**46**) also have *in vitro* antiplatelet activity <88MI 212-04>. Structural features of β -adrenergic antagonists incorporated in compounds such as (**47**) produce platelet aggregation inhibition <86FES478>. Derivative (**48**) has remarkable antiplatelet effects of 1.5–15 times the potency of known agents such as aspirin or ticlopidine <90MI 212-03>. Ticlopidine and PCR 4099 (**49**) interfere with platelet aggregation by interfering with fibrinogen binding <90MI 212-04>. Some thieno[2,3-*d*]pyrimidinone derivatives related to ticlopidine include (**50**) which prevents ADP-induced aggregation <92PHA754>. Thienopyridinol derivatives (**51**) have good phosphodiesterase (PDE) activity <88JHC1571>.



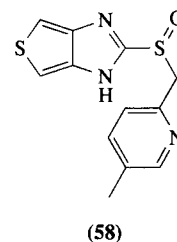
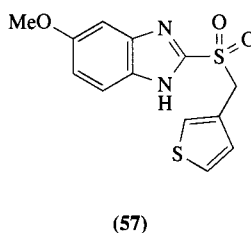
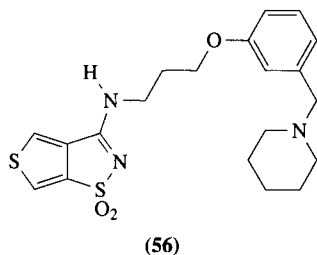
Dihydropyridines are a major class of calcium channel antagonists and continued study on these derivatives has led to identification of S-312 (**52**) as a promising cardiovascular agent <88CPB4389>. Studies of the racemate and two enantiomers of (**52**) show the *d*-form to be responsible for the biological effects <93AF1270, 93AF1276, 93AF1282, 93AF1291>. This derivative has a direct action on neuronal cells which produces neuronal protection from ischemic injury <93MI 212-16>. Compound LY806303 (**53**) is a potent, selective thrombin inhibitor <92BMC1025> which directly interacts with α -thrombin <93JMC2350>. Thienylalkylimidazoles such as (**11**) have already been mentioned as inhibitors of dopamine β -hydroxylase inhibitors which lower blood pressure <90JMC1866>.



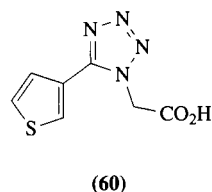
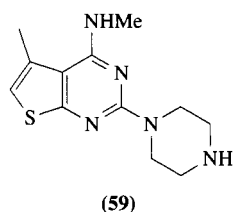
Thienothiopyran-2-sulfonamides have potent topical carbonic anhydrase inhibitory activity which makes them candidates for the treatment of glaucoma. The *S*-enantiomer of (**54**) (MK 417, *S*-enantiomer of MK 927) is the most potent compound in a series and is a development candidate <89JMC2510, 92JHC627>. Thieno[2,3-*b*]thiophene-2-sulfonamides (**55**) also have carbonic anhydrase activity <91JMC1805>. Other thiophenes discovered for this use include arylsulfonylthiophenes <90BJP59> as well as 6-hydroxybenzo[*b*]thiophene-2-sulfonamide <87MI 212-04>.



The discovery of H₂-receptor antagonists such as cimetidine and ranitidine was a major advance in gastrointestinal therapy. Thieno[3,4-*d*]isothiazole dioxide derivatives such as (**56**) are competitive H₂-antagonists <87MI 212-05>. More recently, in the early 1990s, antisecretory therapy has advanced with the discovery of H⁺,K⁺-ATPase inhibitors such as omeprazole. The thiophene analogue of omeprazole (**57**) does not prevent histamine-induced secretion, thus demonstrating the importance of the pyridine moiety of omeprazole for biological activity <92MI 212-05>. On the other hand, thiophene may replace the dimethoxybenzene moiety of omeprazole to produce potent H⁺,K⁺-ATPase inhibitors such as S 1924 (**58**) <88MI 212-05>. Subsequent studies of the related thieno[3,4-*d*]imidazole derivatives, Hoe 731 and S 4216, reveal that this class of agent may be less stable resulting in reduced duration of antisecretory activity <90MI 212-05>.

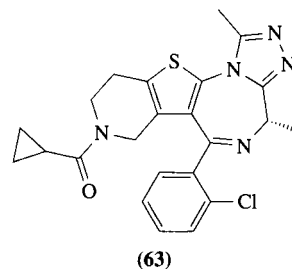
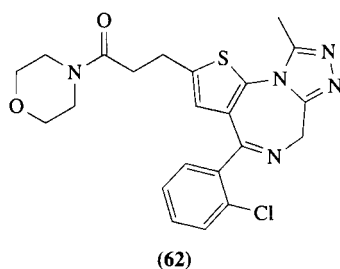
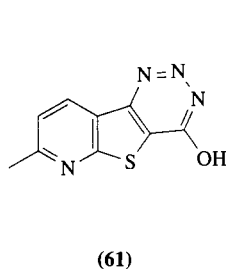


Diabetes has also continued as a target for drug discovery. Compound (**59**) has a hypoglycemic profile of activity produced by an extra pancreatic mechanism of action <86AF1770>. TAT (**60**) is a highly potent aldose reductase inhibitor which should be useful in treating the complications of diabetes such as neuropathy <93MI 212-04>.

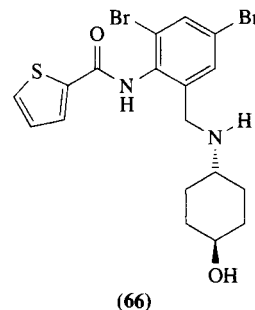
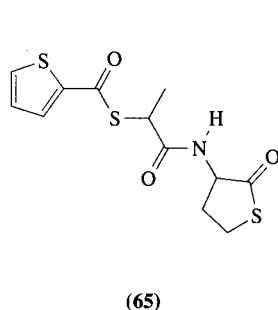
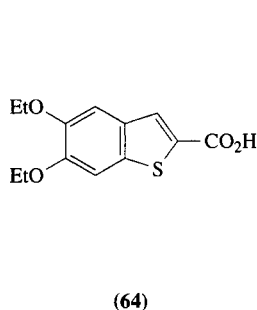


2.12.3.3 Agents to Treat Inflammatory Processes

Reduced lung function may arise from an asthmatic inflammatory response. Evaluation of a series of derivatives in three different *in vitro* models of anaphylaxis shows that RHC 2963 (**61**) is a potent inhibitor of histamine release from mast cells with a mechanism of action similar to disodium cromoglycate <83MI 212-02>. Antagonism of platelet-activating factor (PAF) is another approach to treating anaphylaxis. An excellent review of the role of thienotriazolodiazepines as PAF antagonists has appeared <91MI 212-04>. WEB 2086 (**62**) is a potent, specific PAF antagonist in both *in vitro* and *in vivo* assays without causing sedation or anticonvulsant effects <87MI 212-06>. Design aspects used in the discovery of (**62**) included the need to obviate the CNS effects inherent in the benzodiazepine derivatives. Designed using similar concepts, E6123 (**63**) is also a potent PAF antagonist which may have a better pharmacodynamic profile than (**62**) and may have therapeutic utility in the treatment of asthma <90AF1201>. Subsequent work shows that the (*S*)-(+) isomer of (**63**) is the active enantiomer <91AF1255>. Studies with [¹⁴C]-labelled (**63**) show the compound has good bioavailability in rats, guinea pigs, and dogs <93MI 212-05>.

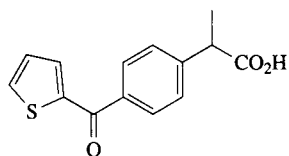


An alternative therapy for anaphylactic shock and asthma is represented by tibenelast (**64**) which is orally effective by a poorly defined mechanism of action <90LS917>. A later study showed (**64**) to be a selective phosphodiesterase inhibitor <90MI 212-06>. Elastase inhibition also has potential in the treatment of insufficient lung function; MR 889 (**65**) is an inhibitor of human leukocyte elastase and may have therapeutic utility for pulmonary emphysema <89BBR(165)568>. Neltexine (**66**) has recently been approved for the treatment of cystic fibrosis.

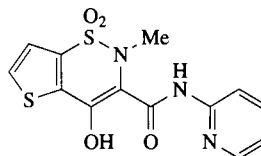


Nonsteroidal antiinflammatory agents (NSAIDs) have been the basis of treating many inflammatory diseases for more than a century. During the 1980s, suprofen (**67**) was approved as an NSAID with potency greater than that of indomethacin or ketoprofen; (**67**) was later removed from the market as a consequence of unexpected toxicity. Tenoxicam (**68**) has been approved as a therapy for rheumatoid arthritis with a profile of action similar to that of piroxicam. Tenidap (**69**) is an exciting new agent with a profile of activity which extends beyond inhibition of the cyclooxygenase

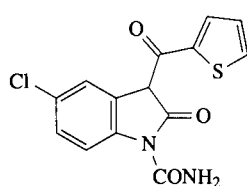
(CO) in the arachadonic acid cascade (typical of most NSAIDs) <91MI 212-05>. Inhibition of both CO and 5-lipoxygenase (LO) is purportedly the dual mechanism of action of L-652,343 (**70**); inhibition of both enzymes may produce antiinflammatory effects similar to the corticosteroids but with fewer side effects <86MI 212-02, 88AF372>.



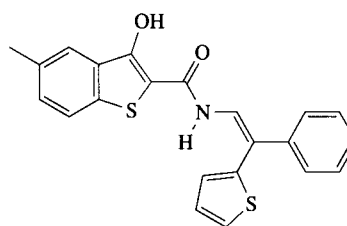
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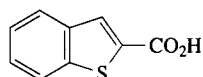
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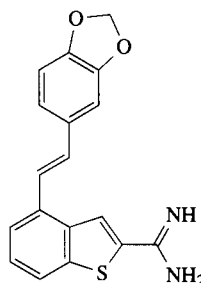
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2.12.3.4 Metabolism Modifiers

Based upon reports of the hypocalcemic effects of thiophene-2-carboxylic acid, benzo[*b*]thiophene-2-carboxylic acid (**71**) was studied and found to inhibit bone resorption in tissue cultures <84MI 212-05>. Benzo[*b*]thiophene-2-carboxamidines such as (**72**) are selective urokinase-type plasminogen activators which may regulate a variety of physiological processes such as wound healing, angiogenesis, arthritis, inflammation and tumor metastasis <93BMC403, 93MI 212-06>.

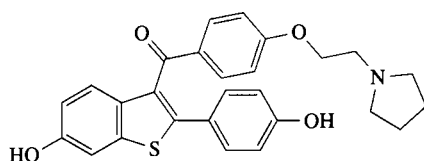


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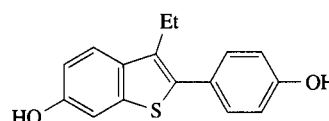


(72)

Antiestrogen therapy is useful for treating diseases of the endocrine system as well as breast cancer, as evidenced by the literature for tamoxifen. A conformational analysis of the estrogen receptor ligand, LY117018 (**73**), as well as several photoaffinity labeled compounds, has produced a model of the bioactive conformation of this class of compounds <93JMC3910>. Earlier work showed the piperidine analogue of (**73**) (LY156758) causes a pronounced regression of rat mammary tumors <84MI 212-06>. Benzo[*b*]thiophene (**74**) strongly inhibited growth of mouse mammary tumors <92MI 212-06>.



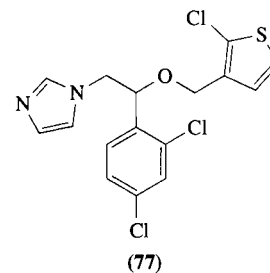
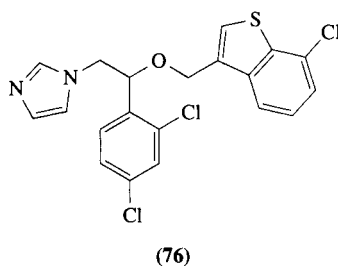
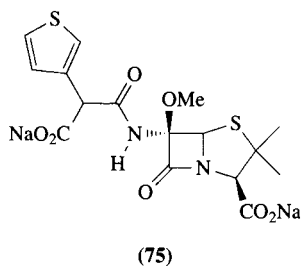
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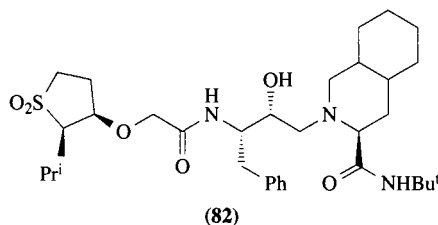
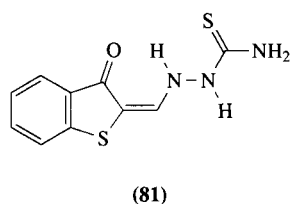
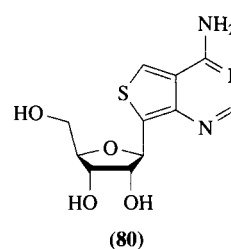
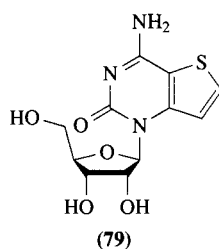
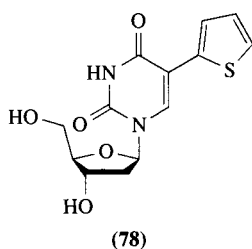
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2.12.3.5 Chemotherapeutic Agents

Chemotherapy commonly targets bacterial, viral and fungal infections as well as the treatment of cancers. During the past decade, temocillin (**75**) has been approved for marketing as a broad spectrum injectable penicillin antibiotic with β -lactamase resistance. Two thiophene-containing topical antifungal agents have also been approved during that time period, tioconazole (**76**) and sertaconazole (**77**). Tioconazole is more potent than miconazole vs. *Candida* and *Trichophyton* species. Both agents presumably act by inhibiting ergosterol synthesis which thus interferes with membrane permeability. The thiophene analogues of the antifungal agent bifunazole have comparable activity to clinical standards <92AP(325)199>.

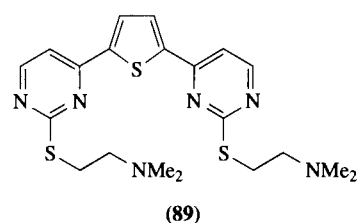
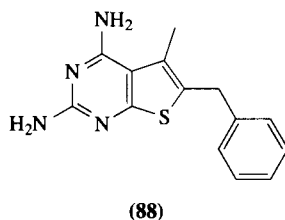
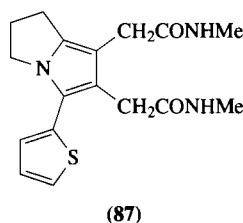
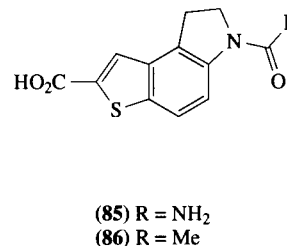
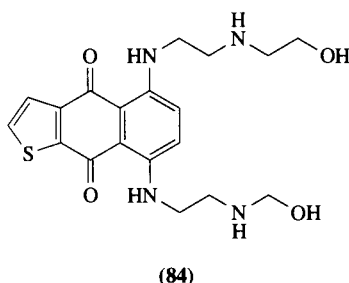
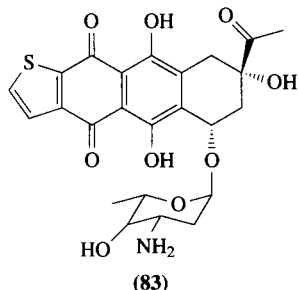


One approach to antiviral therapy has been the synthesis of 2'-deoxyuridines such as (**78**) which has potent anti-herpes simplex virus type 1 (HSV-1) activity <91MI 212-06>. The 5-bromothiophene derivative of (**78**) also has notable activity <91JMC2383>. The 3-thienyl isomer of (**78**) is formed by organotin chemistry <93JMC538>. Thieno[3,2-*d*]pyrimidine nucleoside uridine and cytidine analogues including (**79**) have insignificant activity against HIV-1 <93MI 212-07>. Thieno[3,4-*d*]pyrimidine C-nucleosides, especially the adenosine analogue (**80**), have high cytotoxicity for a number of tumor cell lines while the 5'-iodo derivative is a good inhibitor of purine nucleoside phosphorylase (PNP) <93JHC509>. Hydrazides also have antiviral activity; thiosemicarbazone 643 (**81**) inhibits replication of polio-1, Coxsackie B4, ECHO-2 and adeno-17 viruses <83MI 212-03>. 1,2,4-Triazoles (cyclic hydrazides) are active against a number of pathogenic organisms <89SUL209>. The tetrahydrothiophene 3(*R*)-hydroxy-2(*R*)-isopropylsulfolane embedded in (**82**) produces the best P₂ ligand among a series of HIV-1 protease inhibitors <93JMC292, 93JMC924>.

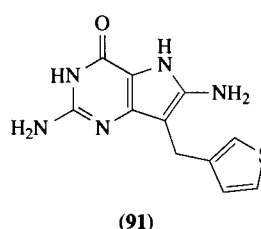
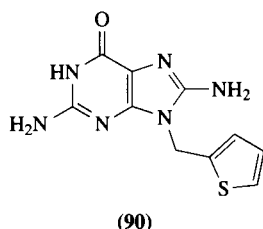


Anthracycline antibiotic chemotherapy is an established method to control cancer. However, the cardiotoxicity of these agents has led to continued efforts to prepare new analogues with reduced side effects such as the thiophene analogue of daunomycin (**83**) <87TL3971>. Thiophene isosteres of the anthracene-9,10-dione antitumor agent mitoxantrone have been prepared and (**84**) has good activity against L1210 leukemia *in vitro* and *in vivo* <90JMC2651>. Thiophene isosteres of PDE-I (**85**) and PDE-II (**86**) as well as their furan analogues have been prepared for incorporation into analogues of the antitumor agent CC-1065 <87JOC19>. 5-Thienyl pyrrolizidine bis(alkylcarbamates) such as (**87**) also have antileukemic activity <89JMC456>. Some 2,4-diaminothieno[2,3-*d*]pyrimidine analogues of

trimetrexate (TMQ) and piritrexim (PTX) including (88) were prepared but none of the compounds are as potent against *Pneumocystis carinii* and *Toxoplasma gondii* as TMQ or PTX <93JMC3103>. As a class of anticancer agents, thiophene derivative (89) was discovered as the best amplifier of bleomycin-mediated degradation of DNA <87JMC1415>.



PNP inhibitors are potential immunosuppressive agents as a consequence of their ability to prevent purine metabolism. A series of guanine derivatives was prepared and PD 119,229 (90) was identified as the most potent PNP inhibitor at the time of the report <87MI 212-11>. Further studies led to the identification of CI-972 (91) as a development compound; it is a reversible inhibitor of PNP with selective cytotoxicity to MOLT-4 but not MGL-8 lymphoblasts <92JMC1605>.



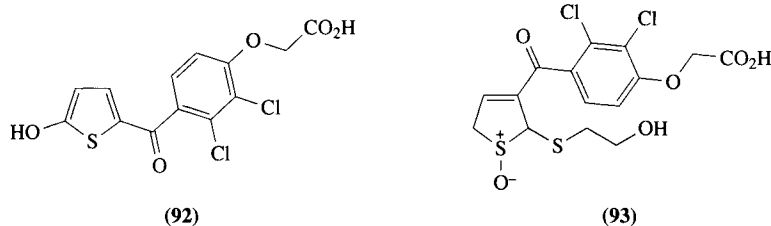
2.12.3.6 Metabolism of Thiophene Derivatives

In general, little is known about the metabolism of thiophene derivatives. Thiophene-containing drugs substituted with 2-arylketone moieties such as suprofen (67) or tienilic acid, a uricosuric diuretic agent, are 5-hydroxylated by cytochrome P450 in rat liver microsomes. In the case of tienilic acid, the result is (92) <90MI 212-07>. Earlier work showed that 3- or 4-hydroxylation does not occur <84MI 212-07>. Interestingly, the metabolism of the 3-substituted isomer of tienilic acid leads to the formation of an S-oxide which subsequently may be trapped by nucleophiles such as glutathione or thioethanol to give (93) which may subsequently oxidize and thus rearomatize the system <91JA7825>.

2.12.4 THIOPHENES AS INTERMEDIATES

2.12.4.1 Introduction

The discussion of thiophene and its derivatives as intermediates is somewhat arbitrary both in definition of topics as well as in which literature reports exemplify these topics. We have chosen to

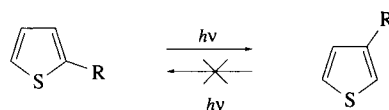


examine the intermediacy of thiophene in photochemical and electrocyclic processes; in some of these cases, the thiophene ring remains intact while in others it is destroyed. Beyond these examples, thiophene derivatives may be transformed into other 5-membered ring heterocycles as well as into smaller and larger ring systems. Finally, there is great commercial interest in petroleum chemistry; through this it has been shown that hydrodesulfurization (HDS) processes of thiophene (which is a component of petroleum) as catalyzed by metal coordination chemistry lead to linear molecules.

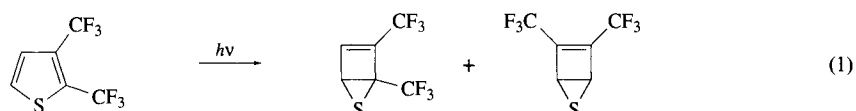
Reviews discussing some of these topics have appeared in the recent literature. These include an excellent discussion of 5-membered heteroaromatic rings (including thiophene) as intermediates <86CRV795>, a review of photoisomerization of furan, pyrrole and thiophene <86MI 212-03>, as well as a specific review of the cycloaddition and ring-opening reactions of thiophenes <83H(20)1127>.

2.12.4.2 Photochemical Isomerism

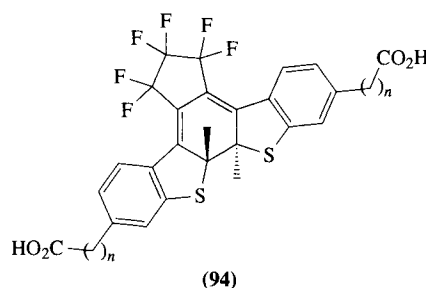
In addition to the photochemical isomerization of 2-substituted thiophenes to their 3-substituted isomers which was extensively studied and reported in the 1960s (Scheme 4) <66JA5047, 67JA3487, 67JA3495, 67JA3498, 67JA3501, 70JOC2737>, photochemically-derived intermediate Dewar thiophenes may be isolable if properly substituted. These compounds may react as the alkene component in [4 + 2] cycloaddition reactions (Equation (1)) <84TL1917>.

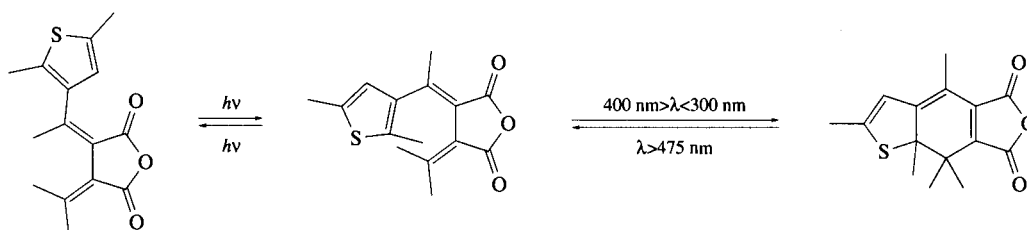


Scheme 4



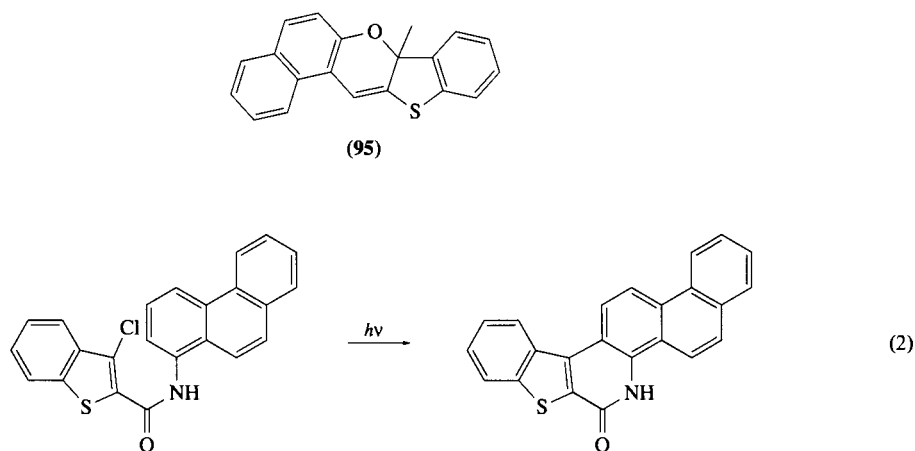
In ongoing studies of thermally irreversible photochromic systems, (94) undergoes more than 10^4 coloration/decoloration cycles without loss of performance <92BCJ430, 92CC206, 92JA8715>. Incorporating fulgide dyes into macromolecules creates photoswitchable bioactive materials. The basis of this chemistry is that the thiophenefulgide isomerizes from the *Z*- to the *E*-isomer and ultimately undergoes *con*-rotatory ring-closure (Scheme 5) <92JA3150>. Photochemically-induced [4 + 2] thiophene–benzene coupling forms naphthothiophenes <91H(32)2323> while intramolecular photochemical closure of 3-chlorothiophene derivatives with polyaromatic systems produces thienoquinolines <91JHC1309, 91JHC1825> and thienonaphthyridines <91JHC1997>.



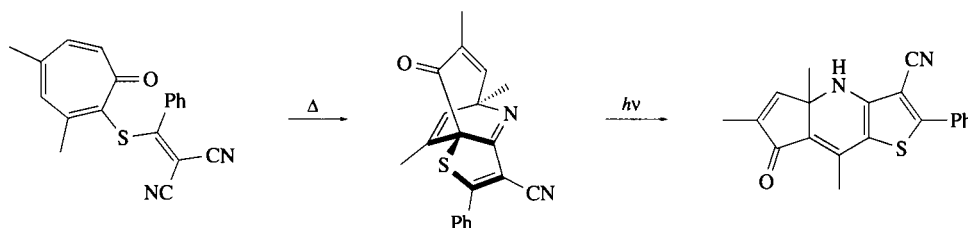


Scheme 5

Chromene (**95**) undergoes a photochromic reaction at room temperature which is dependent on the thiophene bond structure <92CL2257>. A series of papers have reported photochemical ring closure of 3-chlorothiophenes to produce complex polycycles (Equation (2)) <92JHC1613, 93JHC453, 93JHC487, 93JHC653>.



An extremely interesting example of electrocyclic thiophene formation is the intramolecular closure of a tropolone to form a thieno-fused bicyclic derivative; this compound may be induced to undergo photochemical isomerization to produce a novel thienopyridine derivative (Scheme 6) <89CB657>.

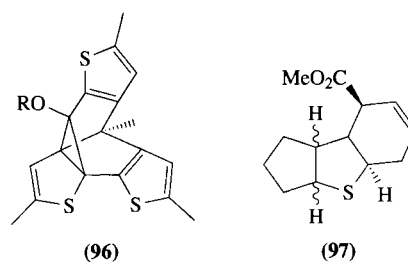


Scheme 6

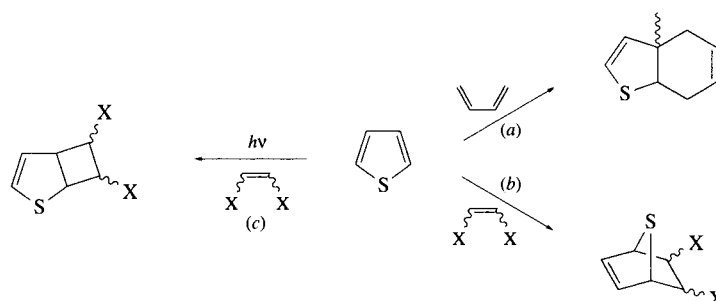
Other interesting fused thiophene derivatives include the photorearrangement of thiophenetriptocene to form semibullvalene (**96**) <93CL681>. Photochemically induced intramolecular reaction of thiophenones with alkenes in methanol produces novel tricyclic thiolane derivatives such as (**97**) <88HCA502>.

2.12.4.3 Electrocyclic Additions

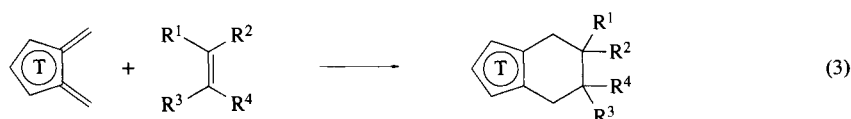
The thiophene ring is less aromatic than benzene; this allows for a rich potential of chemistry in electrocyclic reactions. Thiophene derivatives may be induced to react as monoene (*a*) or diene (*b*) components in these reactions by proper control of functionalization and activation (Scheme 7). Photochemical [2 + 2] cycloadditions are also possible if ethylene is sufficiently activated by



substitution (*c*, Scheme 7). In addition, thiophene *o*-quinodimethane isosteres form more easily than the benzene analogues as a consequence of the less aromatic character of thiophene vis-à-vis benzene; however, the rearomatization of these derivatives provides a strong driving force for [4 + 2] reactions (Equation (3)).



Scheme 7



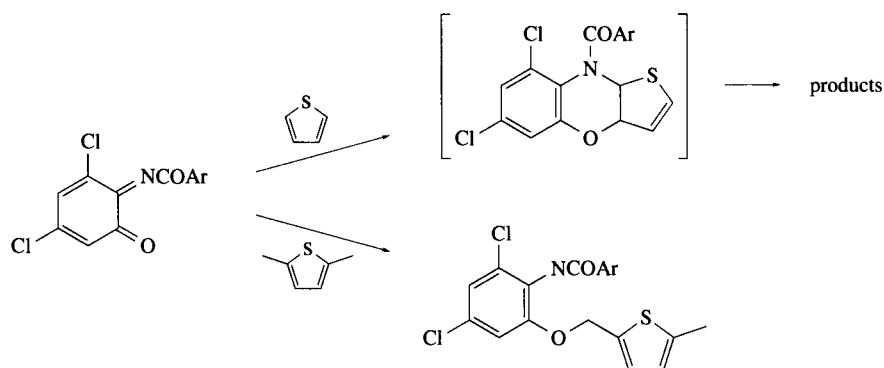
2.12.4.3.1 [2 + 2] Additions

Reaction of thiophene derivatives with benzyne results in predominant [4 + 2] cycloaddition to produce naphthalenes; however, 2-halogen substituted thiophenes react in a [2 + 2] fashion presumably as a consequence of the enhanced 2π enamine-like character of these thiophene derivatives <88JOC5799, 89JOC2142>.

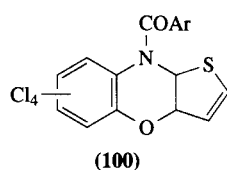
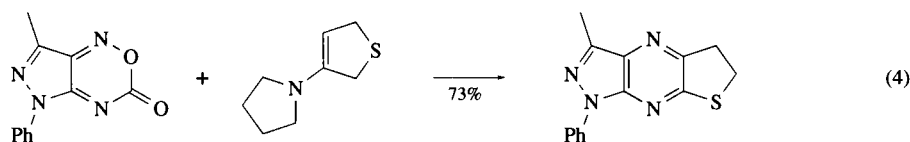
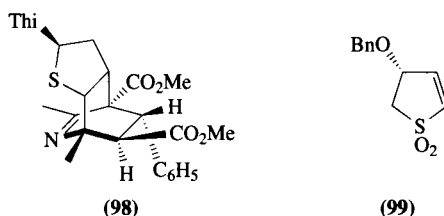
2.12.4.3.2 [2 + 4] Additions

Inverse electron demand Diels–Alder reactions allow reaction with a localized alkenic moiety of thiophene. Unsubstituted thiophene reacts with *o*-quinone monoimides to form tricyclic derivatives but 2,5-dimethylthiophene reacts on the methyl substituent (Scheme 8) <93H(35)1125>. These results differ from those of furan, presumably as a result of the increased aromaticity of thiophene. Iminium ion-mediated cycloaddition of 2-[2-(2,3-dihydrothienyl)]thiophene with 1,4-dihydropyridine gives (98); while furan undergoes a similar addition reaction, unsubstituted thiophene is not reactive enough to react in this condensation <89JOC4134>. Alternatively, a dihydrothiophene ring may serve as a dienophile (99) and react to form intermediates for the synthesis of natural products such as breynolide <93TL4281>. Similarly, in the synthesis of a molybdenum cofactor degradation product, an enaminothiophene reacts with an oxadiazinone to form a tricyclic derivative (Equation (4))

<93JOC6155>. Thiophene itself reacts in an inverse electron demand Diels–Alder with *o*-quinone monoimide to give products derived from intermediate **(100)** <93H(35)1125>.

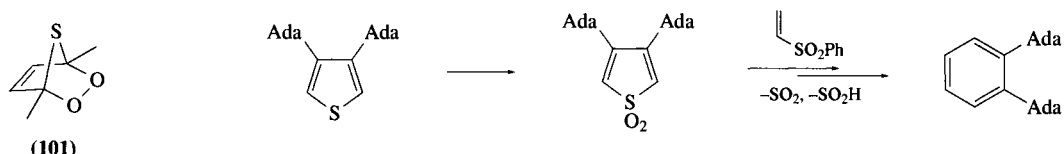


Scheme 8

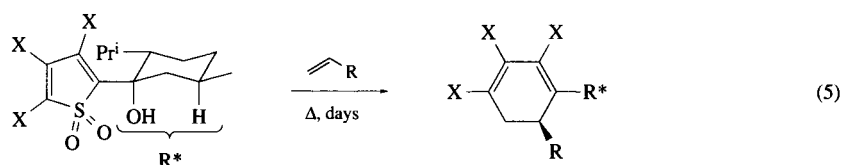


2.12.4.3.3 [4 + 2] Additions

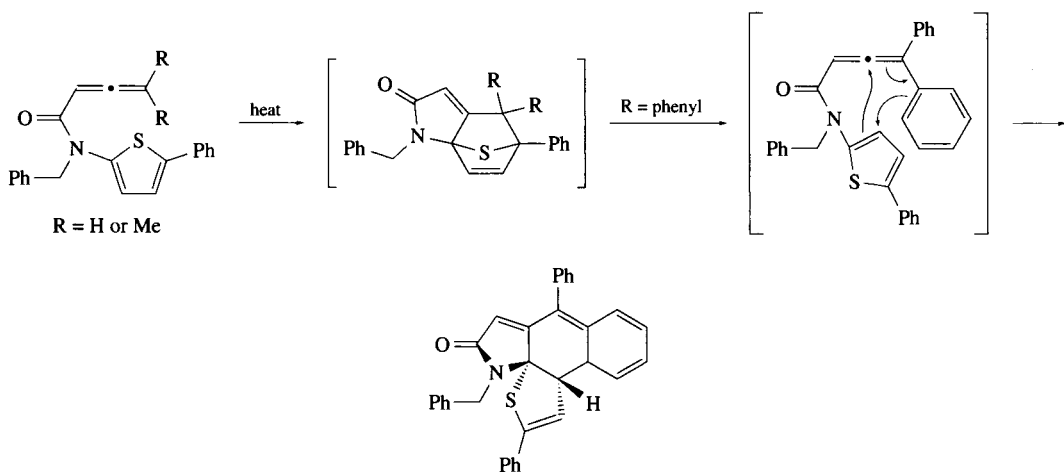
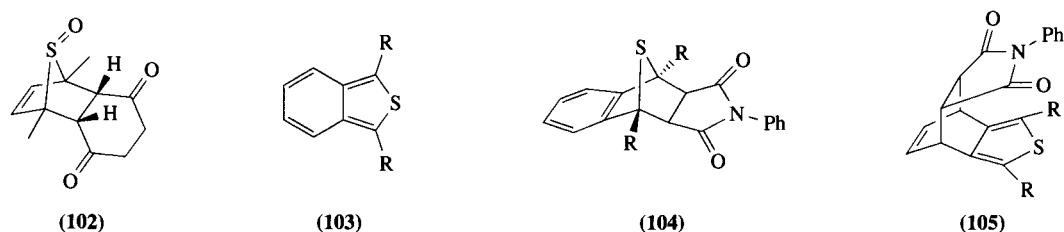
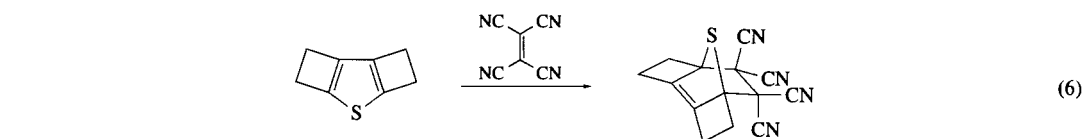
2,5-Dimethylthiophene undergoes a [4 + 2] cycloaddition reaction with singlet oxygen to give **(101)** <84TL4921>. Diels–Alder reaction of 3,4-diadamantylthiophene 1,1-dioxide (and loss of SO₂) with phenyl vinyl sulfone (and loss of benzenesulfonic acid) produces a sterically-demanding *ortho*-disubstituted benzene derivative (Scheme 9) <91PS(59)243>. Menthyltrihalothiophene *S,S*-dioxides are efficient sources of asymmetric dienes for Diels–Alder reactions which produce regio- and stereoisomerically pure adducts (Equation (5)) <88T3127>. The role of thiophene as a Diels–Alder diene has already been mentioned. 2,5-Disubstitution of thiophene using butyllithium and alkylation, MCPBA oxidation to the dioxide, and reaction with fumaronitrile (with loss of sulfur dioxide) produces phthalocyanines <90JCS(P1)1169>. Anthracene derivatives form by reaction of benzo[*c*]thiophene with quinones and by subsequent loss of hydrogen sulfide <90AP(323)295>.



Scheme 9

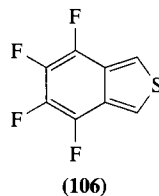


Steric activation also allows thiophene to react as a dienyl component in a $[4 + 2]$ reaction. Thus, 1,2,4,5-tetrahydrodicyclobut[*b,d*]thiophene smoothly reacts with TCNE (Equation (6)) or maleic anhydride (93JA4612). The diene system of unsubstituted thiophene also reacts if sufficiently activated. For example, oxidation of 2,5-dimethylthiophene produces a sulfoxide which reacts with quinone to give (102) (89TL5077). Dienophiles may react at either diene of benzo[*c*]thiophene (103) as controlled by steric effects. Thus, *N*-phenylmaleimide reacts normally to give (104) (when *R* = 4-methoxyphenyl) but unexpectedly to give (105) (when *R* = 2,4,6-trimethylphenyl) (90CB2065). Thiophene reacts either as an intramolecular diene or as a monoene component depending on the nature of the substituents at the ω -position of the side chain (Scheme 10) (90CC405).



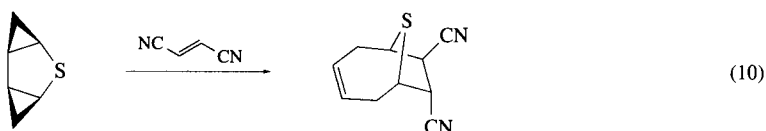
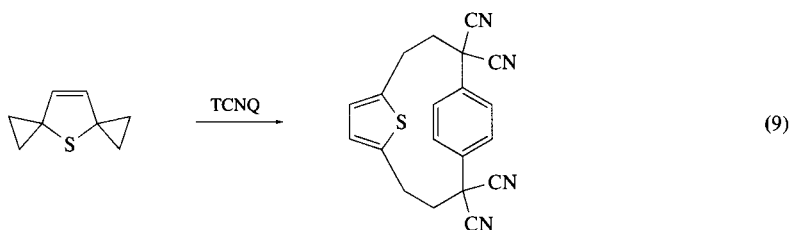
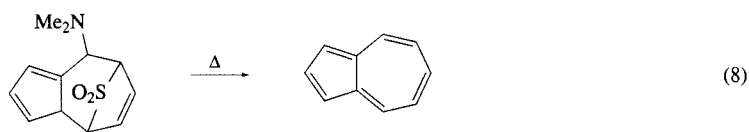
Scheme 10

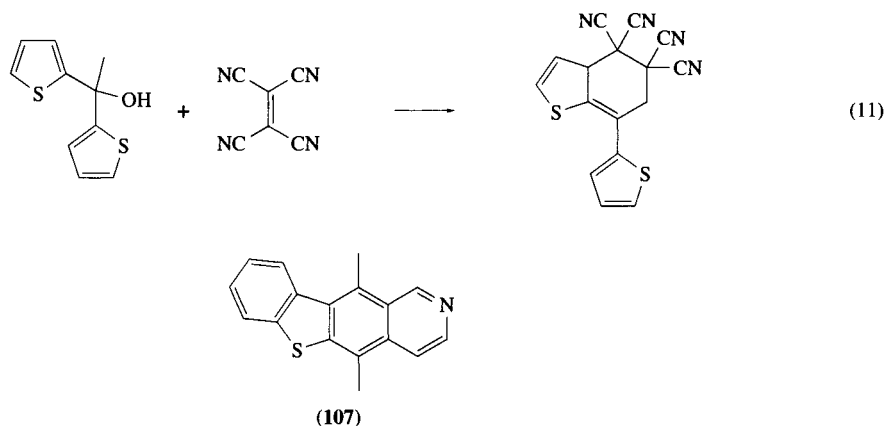
Less activated thiophenes may also react in a Diels–Alder fashion. For example, **(106)** reacts with dimethylacetylene dicarboxylate or benzyne to give naphthalene or anthracene derivatives <90JCS(P1)1919>.



2.12.4.3.4 [4 + others], Other additions

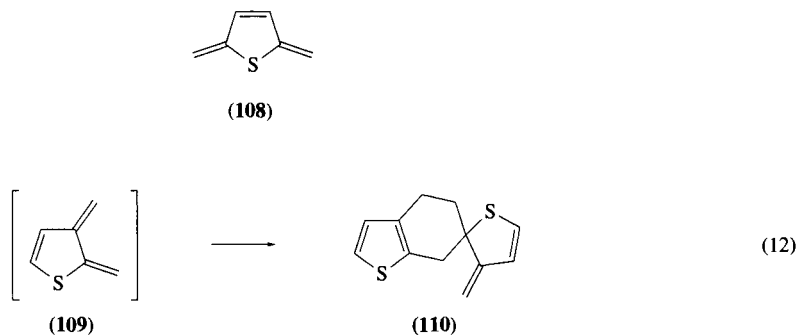
Thiophene derivatives also react as a 4 π component of electrocyclic reactions beyond the well-studied [4 + 2] reactions. For example, 1*H*-cyclopropylbenzene reacts with thiophene 1,1-dioxides to form annulenes (Equation (7)) <89HCA1311>. In a related example, 6-dimethylaminofulvene reacts with fully aromatic thiophene 1,1-dioxide to form a cycloaddition product which undergoes cheletropic loss of SO₂ to form azulene (Equation (8)) <92JCE262>. 4-Thiadispiro[2.1.2.2]non-8-ene reacts with TCNQ to form (2,5)thiophenophanes (Equation (9)) <93JOC1601>. Bishomothiophene forms by diazomethane addition to homothiophene and undergoes rearrangement to form 2,3-dihydrothiepine, which then dimerizes and reacts with fumaronitrile to form cycloadducts (Equation (10)) <93CB485>. Bis(2-thienyl)ethylene forms from 1,1-bis(2-thienyl)ethanol and reacts with TCNE or ddq to give [4 + 2] cycloadducts via a radical ion pair (Equation (11)) <90H(31)1873>. An analogous intramolecular reaction has been utilized to prepare benzo[*b*]naphtha[2,3-*d*]thiophene **(107)** <82H(19)1033>.



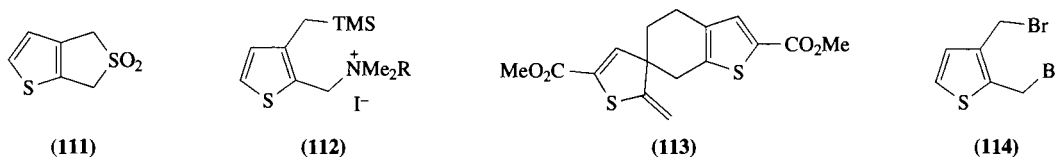


2.12.4.3.5 *o*-Quinodimethanes

Investigation of the synthetic utility of the thiophene isosteres of *o*-quinodimethanes has steadily increased as new synthetic methodologies have become available. In the mid 1980s, precursors of these compounds were studied as a means to understand their theoretical implications. Thus, flash vacuum pyrolysis of 2-chloromethyl-5-methylthiophene gives **(108)** which was characterized by variable temperature photoelectron spectroscopy (VTPES) <88TL6239>. In a similar way, **(109)** was generated and characterized only as its dimer **(110)** (Equation (12)) <88TL117>. Use of deuterium labeling has provided insight into the structure of the [4 + 2] dimer of this theoretically interesting molecule **(109)** <92MI 212-07>. 2,3-Didehydrothiophene is potentially generated by either flash vacuum pyrolysis or photolysis of 2,3-thiophenedicarboxylic acid anhydride as measured by formation of two unusual by-products <92CB423>.

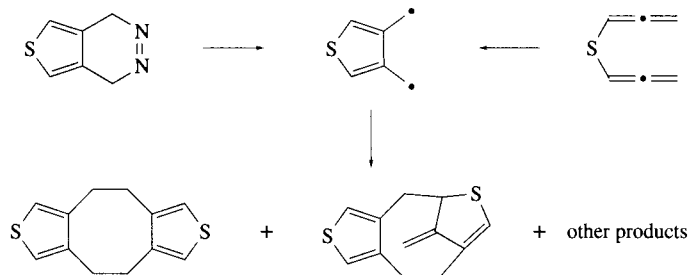


Other precursors of these compounds have been prepared. Two precursors of 2,3-dimethylene-2,3-dihydrothiophene **(109)** include cyclic sulfone **(111)** which loses SO₂ at 130°C <91CC1287> and trimethylsilyl derivative **(112)** which produces **(109)** upon treatment with fluoride ion <90S915>. 4,6-Dihydrothieno[3,4-*b*]thiophene 5,5-dioxide also serves as a precursor to 2,3-bis(methylene)thiophene <90TL1491>. The unique spiro dimer **(113)** forms through the intermediacy of the 5-carboxylate derivative of **(109)** <91JOC6948>. Pyrolysis of **(114)** produces the “hereto unknown” 2,3-dimethylene-2,3-dihydrothiophene which reacts *in situ* with a variety of dienophiles <87TL6085>.

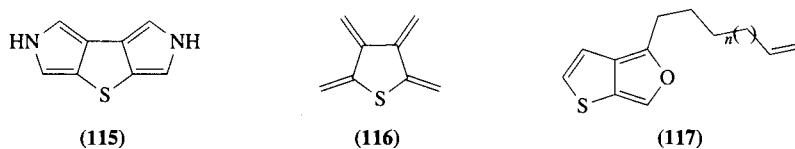


The biradical intermediate of 3,4-dimethylenethiophene forms by intramolecular cyclization of bisallenyl sulfide or by pyrolysis of a diazene precursor and dimerization to give a variety of products (Scheme 11) <87TL4263>. Studies of transient signals in ¹³C NMR spectra show that the isomeric biradical 3,4-dimethylenethiophene forms in rigid matrices at 77 K and exhibits an intense purple

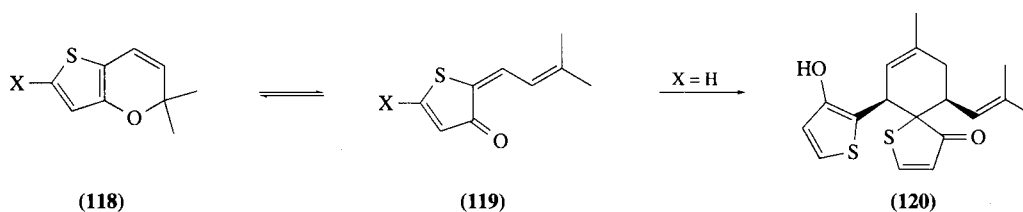
color which disappears upon warming <91JA2318>. Reduction/ring closure of a bisazide provides (115) which may be viewed as a synthetic equivalent of radialene (116) <93T6831>. Intramolecular Diels–Alder reaction of thienofuran (117) provides a route to thiamarmelerin <89LA405>. This compound also directly forms tetrahydronaphtho[2,1-*b*]thiophene <88TL1137>.



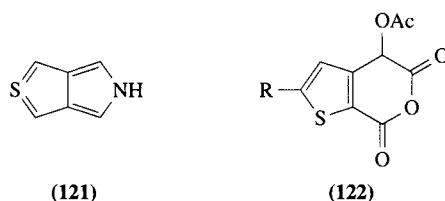
Scheme 11



Related to the dimethylenethiophenes, an example of a thiophene isostere of an *o*-quinomethide (119) is reported to exist in equilibrium with the ring-closed thieno[3,2-*b*]pyran (118). This is in contrast to chromenes (which ring-open only upon photolysis) and the isomeric thieno[2,3-*b*]pyran (which ring-opens exclusively) <93JOC4631>. Interestingly, (119) undergoes further isomerization and Diels–Alder dimerization to form (120) (Scheme 12) <94JOC5088>. The theoretically interesting 4*H*-thieno[3,4-*c*]pyrrole (121) is formed by an intramolecular azide cycloaddition on an alkene and subsequent loss of diazomalonate. The compound undergoes trapping reaction with *N*-phenylmaleimide <88CC320>. *N*-Aryl derivatives of (121) as well as the [2,3-*c*]isomers may be formed by condensations of anilines with thiophenedialdehydes <88JHC1531>. Treatment of (122) with strong base generates a functionalized 2,3-dimethylene-2,3-dihydrothiophene which is used in the regio-specific synthesis of the D-ring thiophene isostere of daunomycin <90CPB1836>.

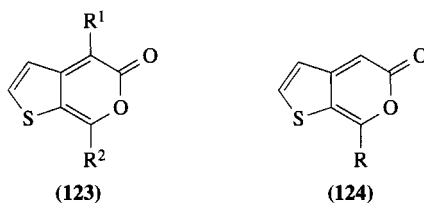


Scheme 12

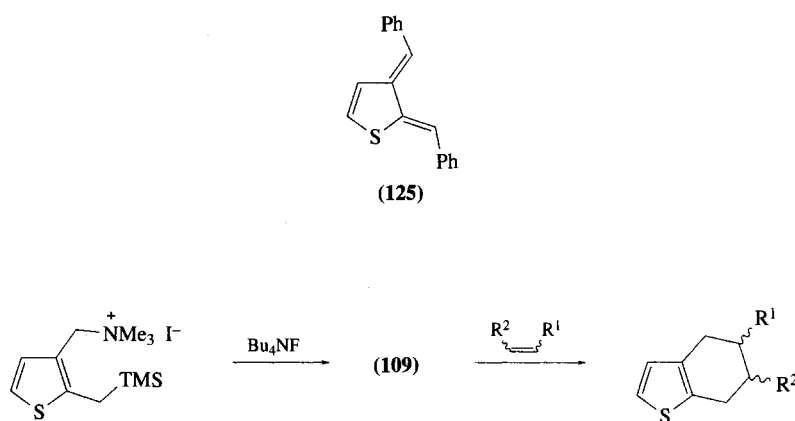


Other cyclic 2,3-dimethylenethiophene derivatives react to produce some other unique compounds. Derivative (123) is either an equivalent of the intermolecular Diels–Alder diene 2,3-dimethylenethiophene (if $R^1 = R^2 = H$) or an intermediate for intramolecular reactions (if R^1 con-

tains an alkyne) <90JCS(P1)2909>. Diels–Alder reaction of pyrano[3,4-*b*]thiophene-5-one (**124**) with alkynes gives ester derivatives <88TL5817>.



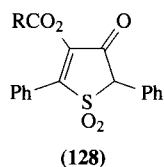
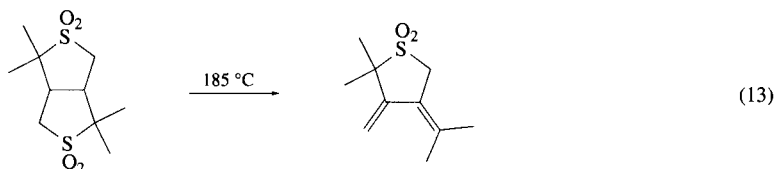
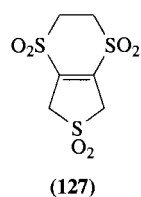
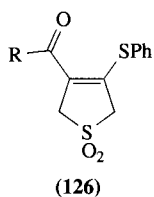
As more powerful methodologies for forming these reactive intermediates are discovered, more reports utilizing thieno *o*-quinodimethanes to prepare fused thiophene ring systems have appeared. *N*-Phenylmaleimide, 1,4-naphthoquinone and several other dienophiles react with (**109**) as generated by extrusion of sulfur dioxide to form benzo[*b*]thiophenes <92T8101>. 2,3-Dibenzylidene-2,3-dihydrothiophene (**125**) forms by base-induced elimination of methanol and reacts with norbornene in a similar fashion <91ACS919>. As mentioned, (**109**) also forms by fluoride-induced desilylation/elimination of trimethylammonium iodide and reacts with chiral 2(*5H*)-furanones with high enantioselectivity <91TL7751>. Fluoride-induced 1,4-elimination of (trimethylammoniomethyl)- or (trimethylsilylmethyl)thiophene forms (**109**) and reacts with dienophiles to form a series of [4 + 2] Diels–Alder adducts (Scheme 13); this reaction also occurs on the 3,4-disubstituted thiophene isomers <93RTC7>.



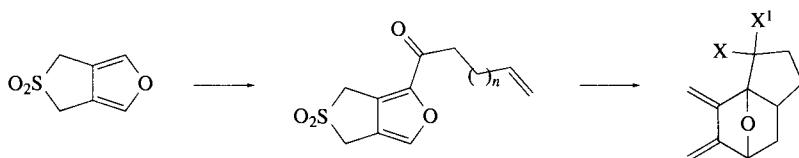
Scheme 13

2.12.4.3.6 Sulfolanes as precursors to butadienes

Sulfolanes have been used to great advantage as synthetic equivalents or precursors of butadienes for electrocyclic reactions. Alternatively, 1,3-dihydrobenzo[*c*]thiophene 2,2-dioxide thermally eliminates sulfur dioxide to produce a diene which reacts with alkenes <89SC2765>. Other reports utilizing the thiophene ring to produce a reactive diene include that of (**126**) as a precursor to 3-phenylthio-1,3-butadienes <89SC1593>. Oxidation of (**126**) to the corresponding phenylsulfone allows for substitution using organometallic reagents to produce precursors to 3-substituted-1,3-butadienes <90TL1035>. Derivative (**127**) serves as the precursor to electron deficient 2,3-ethylenedisulfonyl-1,3-butadiene <89CC1020>. Sulfonium salts of C-2 symmetric thiolane derivatives may be used to form optically active *trans*-stilbene oxides in up to 83% *e.e.* <90TL35>. Derivatives of 3,4-dialkylthiolanes have utility as precursors to 1,3-butadienes and may be considered as precursors to tetramethylene diradicals (Equation (13)) <87TL5913>. The related sulfolane (**128**) does not eliminate SO₂, but serves as an effective Friedel–Crafts acylating agent with alkenes and activated aromatic compounds <89MI 212-04>.

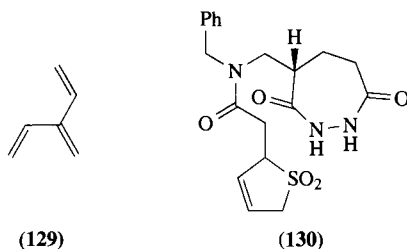


An interesting application of this approach utilizes the Diels–Alder reactivity of the furan moiety of 4*H*,6*H*-thieno[3,4-*c*]furan with subsequent loss of SO₂ to reveal a subsequent reactive diene. Three asymmetric centers with known relative stereochemistry are developed using this strategy (Scheme 14) <93JCS(P1)2387>.



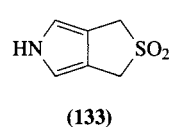
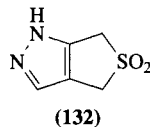
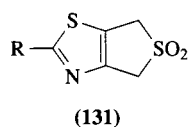
Scheme 14

Regiospecific substitution of 3-sulfolenyl dianions leads to synthetically useful di- and tri-substituted 3-sulfolenes <90JOC5410>. 3-Vinylsulfolene undergoes thermal cheletropic extrusion of SO₂ to produce [3]dendralene (129) which functions as a tandem annelating reagent <91CC114>. Compound (130) provides an intramolecular example of a latent *cis*-1,3-butadiene derivative which subsequently undergoes azidocarbonyl Diels–Alder reaction <91SL31>.

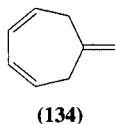


Other 3-sulfolene derivatives have led to the synthesis of a variety of molecules. Thiazole-fused 3-sulfolene (131) is a synthetic equivalent to *o*-dimethylene thiazole <92TL4201>. Similarly, (132) is the precursor to the pyrazole analogues of *o*-xylylene <91TL7609>. Reaction of (133) with dimethyl acetylenedicarboxylate gives not only [4 + 2] reaction of the 1,3-butadiene but also [4 + 2] reaction of the pyrrole to produce novel 7-azanorbornene derivatives <92CC549, 92CC1100>. Compound (127) is a ready precursor of 2,3-(ethylenedisulfonyl)-1,3-butadiene which reacts with a number of electron-

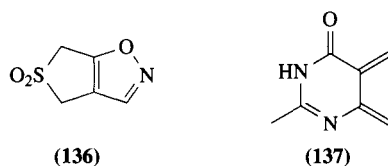
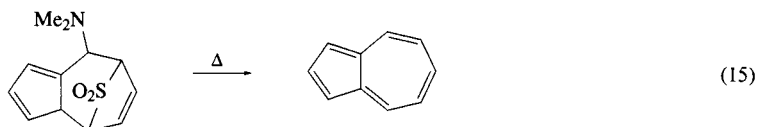
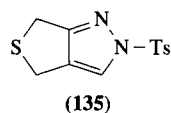
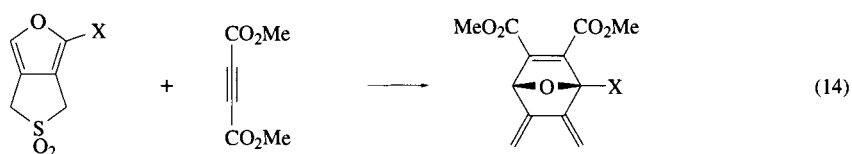
deficient and electron-rich alkenes <92CB499>. Both the furo[*b*]- <92H(34)663> and furo[*c*]sulfolene derivatives provide access to the *o*-quinodimethane analogues <91CC1765>.



An enantioselective synthesis of the tetracyclic quassinoid skeleton requires reaction of a derivatized 3-sulfolene with (*S*)-carvone <92CC341>. A general approach to alkaloids uses an intramolecular reaction of an amide derivative <92SL272>. 3-Sulfolenes are not only precursors for 1,3-butadienes; cycloheptadiene (**134**) forms by α,α' -alkylation of 3-sulfolene and subsequent thermal loss of SO₂ <91JOC4560>. Sulfolenes may also serve as protecting groups for preexisting dienyl systems. In one instance, addition of SO₂ to the diene of vitamin D₂ allows improved synthesis of 24,24-difluoro-1 α ,25-dihydroxyvitamin D₃ <92CPB1662>. Microbial hydroxylation of monoterpenes is enhanced by SO₂ protection of dienes <92T6681>.

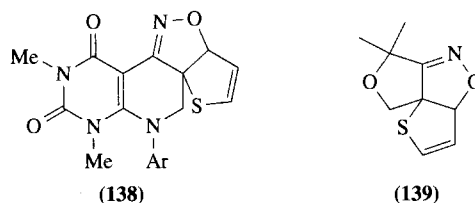


Extrusion of sulfur dioxide from oxidized dihydrothiophene derivatives is an exceptionally powerful method to prepare *cis*-dienes as components in Diels–Alder reactions. A good example of this approach utilizes the Diels–Alder reactivity of the furan ring in 2-substituted 4,6-dihydrothieno[3,4-*c*]furan 5,5-dioxides to react with a variety of dienophiles such as DMAD, dimethyl maleate, and dimethyl fumarate. The adducts subsequently lose SO₂ to form another reactive diene (Equation (14)) <93H(35)57>. Alkylation of the sulfolene ring allows for substitution in this reaction sequence <93JCS(P1)2263>. Careful control of conditions allows formation of *N*-substituted pyrazolo-3-sulfolene (**135**) which is a synthon for pyrazolo-*o*-quinomethane <92T8101, 93H(36)2839, 93JOC493>. Pyrrolizidines or indolizidines form by thermolysis and extrusion of SO₂ and N₂ from azidosulfolenes (Equation (15)) <93MI 212-08>. *o*-Dimethylene isoxazole is generated by pyrolysis of sulfolene (**136**) <92TL8121>. The very unusual pyrimidine *o*-quinodimethane (**137**) is formed similarly from its corresponding precursor <93SL347>. Tethering two sulfolenes by alkylative procedures leads to the precursor of bis(1,3-dienyl)alkanes <92T8963>. 2-Alkyl-3-(trimethylsilyl)-1,3-butadienes may also be prepared using this technology <93JOC967>.

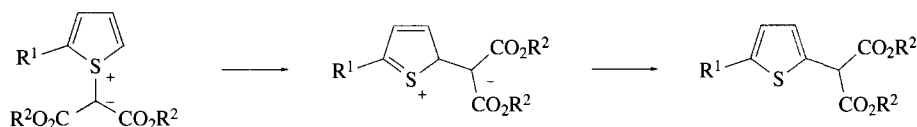


2.12.4.3.7 1,3-Dipolar additions

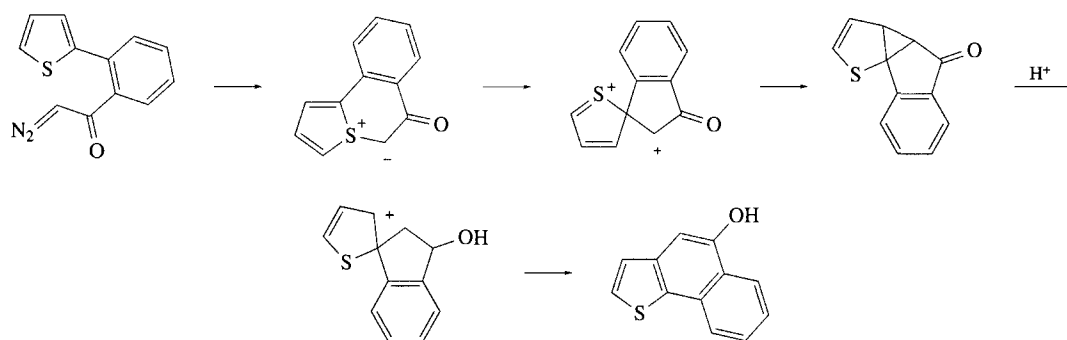
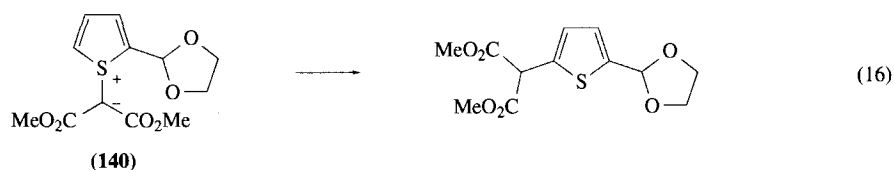
Intramolecular azide cycloaddition to a thiophene double bond leads to 4*H*-thieno[3,2-*b*]pyrrole derivatives <89TL1655>. 1,3-Dipolar intramolecular cycloaddition of a nitrile oxide on to the thiophene moiety gives **(138)** <88S342>. In a similar fashion, one double bond of thiophene acts as a dipolarophile to produce derivative **(139)** <89JOC5277>.



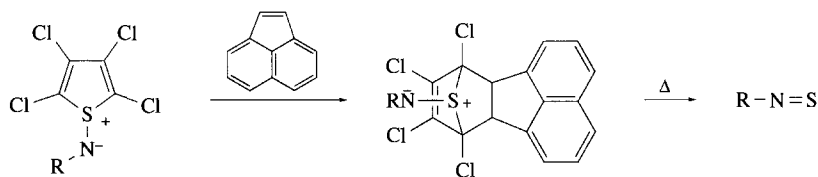
Ylide rearrangements result in some novel thiophene substitution reactions (Scheme 15) <85CC1590>. In specific, thiophenium ylide **(140)** rearranges cleanly to give the 2-thienylmalonate derivative (Equation (16)) <89CS97>. Some novel ring systems arise utilizing this chemistry to form aromatic thiophenium ylides (Scheme 16) <86ACS178, 86ACS303>. Use of *S,N*-ylide chemistry leads to *in situ* generation of synthetically useful amounts of thionitroso compounds (Scheme 17) <84CC114>.



Scheme 15

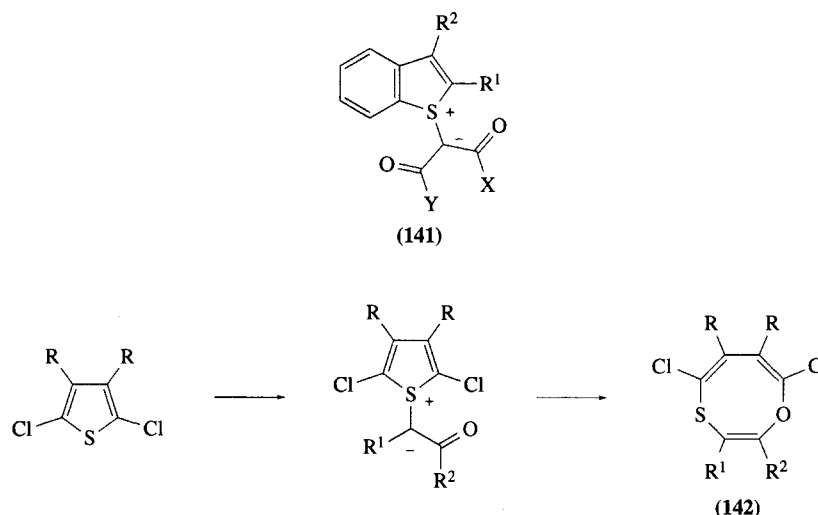


Scheme 16



Scheme 17

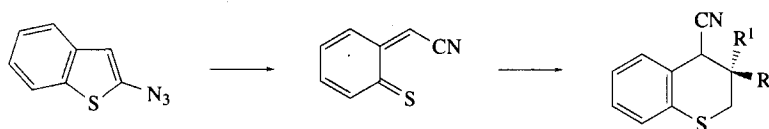
S-Substitution of thiophene results from the reaction of benzo[*b*]thiophenes with β -dicarbonyl-diazo compounds and forms the *S,C*-ylide (**141**) <91T8611>. In another example, 2,5-dichlorothiophene derivatives react with diazo compounds using rhodium catalysis to produce *S,C*-thiophenium methylides which undergo smooth thermal rearrangement to the 1,4-oxathiocines (**142**) (Scheme 18) <88CC138, 89JOC4822>. The oxathiocines are heteroanalogues of eight-membered ring 10π systems but have no aromatic character.



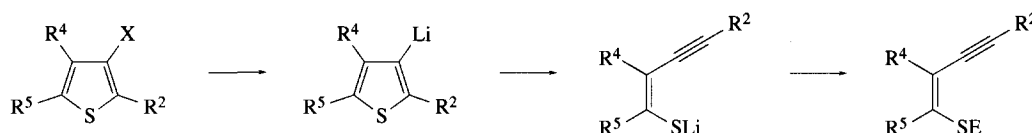
Scheme 18

2.12.4.4 Transformation to Non-thiophene Derivatives

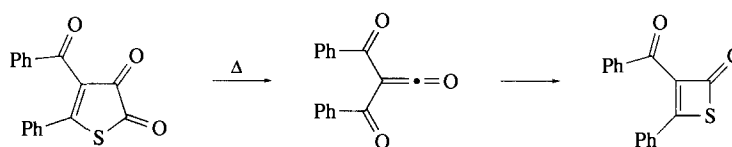
Fragmentation of thiophene rings leads to linear noncyclic derivatives as well as compounds with smaller as well as larger rings. Thermolysis of 2-azidobenzo[*b*]thiophene produces an *o*-quinoidal enethione which reacts in the presence of electron-rich alkenes to form thiochromans (Scheme 19) <88JCS(P1)3375>. A relatively general ring-opening reaction of 3-lithiothiophenes is a novel route to the preparation of enyne derivatives (Scheme 20) <83MI 212-04>. Thermolysis of 2,3-dihydrothiophene-2,3-dione leads to extrusion of CO and subsequent ring closure to a thiet-4-one derivative (Scheme 21) <82BSB997>.



Scheme 19



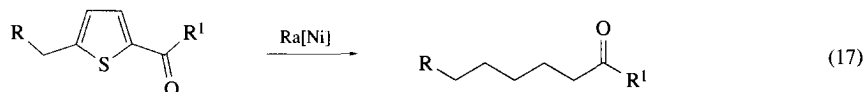
Scheme 20



Scheme 21

2.12.4.5 Hydrodesulfurization (HDS)

The removal of sulfur from thiophene derivatives has been an effective strategy for the preparation of a variety of useful compounds as outlined in Equation (17). A thorough review of this approach has appeared <84BAU178>. For example, Friedel–Crafts acylation of thiophene, Wolff–Kishner reduction, a second Friedel–Crafts acylation and finally Raney nickel reduction is used for a new synthesis of (*R*)-(+)- γ -dodecanolide <88IJC(B)257>. Electroreduction of the thiophene ring is used in the preparation of methoprene <83BAU1241>. Enantiomerically pure alkanols form by derivatization (and resolution) of alkylthienylcarbinols with chiral acetals and, ultimately, desulfurization <91M705>.

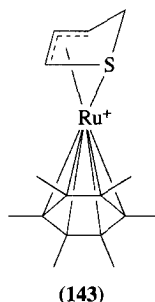


The study of hydrodesulfurization (HDS) has had tremendous impact on the petroleum industry <88ACR387, 88ACR394>. The mechanism of hydrodesulfurization has theoretical and commercial importance and has been the subject of numerous reports investigating mechanism, variation of catalyst and substrate, and product composition. In general, the role of the metal catalyst as well as the structures of thiophene–metal coordination complexes will be discussed in Section 2.12.11.2. In this section, we focus on specific desulfurization to produce isolable products.

Several easily prepared nickel-containing complex reducing agents (NiCRAs and NiCRALs) are nonpyrophoric alternatives to Raney nickel and nickel boride for desulfurization reactions <89JOC4848>. Desulfurization of benzo[*b*]thiophene may be accomplished using aqueous platinum metal species <91CJC590> or nickel boride <90CC819>.

The products of thiophene complexes from HDS have also been identified in some cases. Homogeneous hydrogenolysis of thiophene using rhenium catalysts leads to the formation of 1-butane-thiolate <92JA10767>. Nickel boride effects desulfurization of benzo- and dibenzothiophenes to alkylbenzenes and biphenyls, respectively <92JOC1986>. Adsorption and surface reactions of thiophene on zeolites show that fragmentation of C–S bonds is quite facile <92JPC2669>. Molybdenum catalyst activation during the formation of butane from thiophene is the subject of some semi-empirical molecular orbital calculations <92JMOC65, 92JCS(F1)3225>. Inclusion of nickel strongly promotes HDS on molybdenum catalysts <92CCC1033>. Aluminum phosphate lowers the selectivity of thiophene conversion on molybdenum catalysis <92CCC836>. Iron-containing catalysts supported on carbon interfere with butene to butane conversion in the HDS of thiophene <92MI 212-08>.

A host of transition metal complexes may effect HDS. Anionic ruthenium and molybdenum carbonyl complexes supported on alumina are effective in desulfurizing dibenzothiophene <92CL2285, 93CL589>. Ruthenium(II) thiophene complexes react with hydroxide at sulfur to produce an intermediate which reacts with additional hydroxide to form sulfoxides or rearranges to give 2-hydroxythiophene complexes <93OM3273>. A similar reaction with benzothiophene ultimately produces 2-ethylbenzenethiol <93JA7505>. The first example of homogeneous hydrogenolysis of thiophene was recently reported using $\text{ReH}_7(\text{PPh}_3)_2$ <92JA10767>. Proton-induced carbon–sulfur cleavage of coordinated thiophene ruthenium complex (**143**) provides a new mechanism for thiophene hydrogenolysis <93JA4943>. Tris(triethylphosphine)platinum(0) may effect HDS of dibenzothiophene <93JA12200>, while osmium complexes ring open tetrahydrothiophene <93JA8207>.



2.12.5 THIOPHENE-CONTAINING POLYMERS/CHARGE-TRANSFER COMPLEXES

2.12.5.1 Introduction

Since the first report that the unsaturated polymer of polyacetylene could conduct a weak electric current <77MI 212-02>, the science of conducting polymers (CPs) has attracted the interest of many investigators resulting in numerous publications. Polyalkyne is one of the best CPs (1.7×10^5 amps/V cm) <94SCI1700>; however, other polymers that contain alternating single-double bonds (e.g., poly(*p*-phenylene), polyaniline, polypyrrole or polythiophene) also perform well (Figure 1). These polymers can replace metals in applications where weight, flexibility, processability or resistance to corrosion is required for a particular application. Polythiophene (PT) has excellent environmental stability, structural versatility, and electronic properties that make it the subject of intense study directed toward use in applications such as conductors, electrodes, electronic devices, and nonlinear optical devices as well as other electrochemical applications. The preparation <86CJC76, 88H(27)1731, 88JCP(88)5535, 88MI 212-10, 89JPS(A)1891, 89MI 212-07, 89MI 212-08, 89MI 212-09, 90PHC(2)50, 91PHC(3)70, 92PHC(4)62, 93MI 212-09, 93PHC(5)82, 94PHC(6)88> and use of PTs have been reviewed <88CRV183, 91MI 212-07, 92CRV711>.

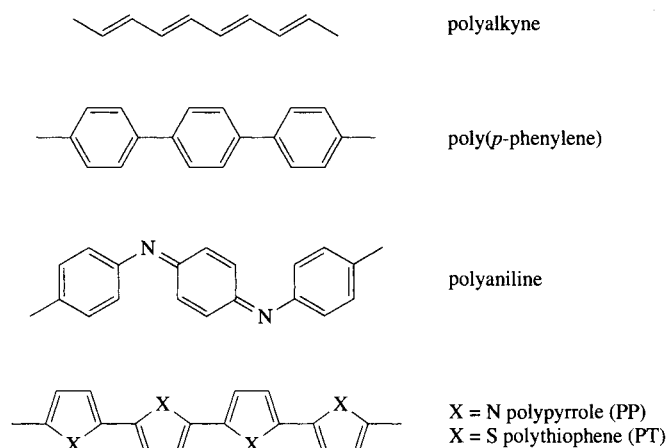


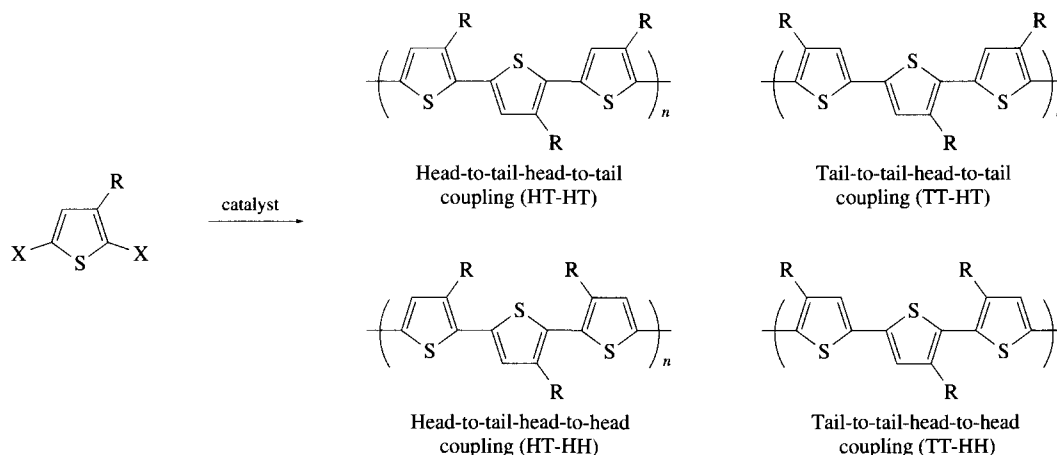
Figure 1 Structures of conducting polymers.

A review of all possible combinations of preparation methods (chemical, electrochemical or plasma), structural properties, results from neutral or doped (polarons or bipolarons) <90MI 212-08, 90SM125>, polymer film thickness, temperature, applied voltage, and polymer blends (copolymerization <87MI 212-08, 87MI 212-09, 87SM277, 88MI 212-11, 89MI 212-12, 90JEC275> or composites <88JEC(247)351>) is far beyond the scope of this review. In addition, the mechanisms of polymerization and of polymer conduction (neutral vs. doped) are also relevant. Interested readers are referred to the references above as well as to others <87MI 212-10, 89JPC5354, 91JA600, 93MI 212-10> to gain further insight into the complexity of the subject. This review is organized by the structural features of the various polythiophenes.

2.12.5.2 Poly(3-alkylthiophenes)

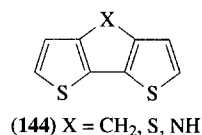
Polymerization of thiophene occurs at the α -position leaving the 3- and 4-positions available for substitution. By far the largest number of publications have described a host of different substituents at these sites. Depending on the substituent and polymerization conditions, there are four possible products (Scheme 22). A few papers report the isolation of the HT-HT coupling product <93JOC904, 93JA11608> while other reviews discuss specific properties of these poly(3-alkylthiophenes) <91MI 212-08, 93SM71>. Instead of providing details from each publication, polymer units and references are tabularized (Table 1). Of particular interest, note that the last three entries in Table 1 utilize chiral moieties attached at the 3-position. Electrodes or membranes comprised of these chiral polymers might be capable of producing stereoselective analysis and/or electrosynthesis. In fact, the last two

entries show modest stereoselectivity when a chiral anion such as (+)- or (−)-camphorsulfonic acid is used as a doping agent during voltammetric cycles.



Scheme 22

Instead of preparing polymers directly from monomers, various bi- <87CC1476, 91JEC57, 91MI 212-11, 93SM295>, ter- <89MI 212-10, 91CM888, 91SM3037, 93CC962, 93CM1456, 93MM3295> or quaterthiophenes may be used as precursors to polymerization <88MI 212-12, 92CM1097, 93SM420>. By doing this, the polymerization step can be done at lower oxidation potentials to thus afford a polymer in higher yield with better repeating unit regularity. The preparation and analysis of substituted sexithiophenes is also reported <93JA8716, 93JEC185, 93SM239>. These sexithiophenes are used as models for studying the physical properties of the repeating units in 3-substituted polythiophene backbones. Modification of the bithiophene moiety by ring fusion with either a CH_2 , S or NH unit produces (144); this tricyclic ring alters the flexibility of the monomeric units and allows for control of the redox potentials of the corresponding conducting polymers <91MI1841>.

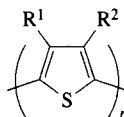


2.12.5.3 Polythiophene Containing “Spacers”

A variation on the bithiophene theme is the preparation of thiophene polymers that contain alkane, alkene, alkyne or aryl spacers <92SM287>. These conducting polymers are found to be especially useful as nonlinear optical devices or in constructing molecular electronic devices <91MI 212-12, 92MI 212-12, 93SM1632>. Poly(thienylenemethylene) forms from (2-thiophene)methanol and produces a conducting polymer when doped with iodine <89CE841, 93SM833>. The anodic coupling of bi- and terthiophene derivatives containing an ethylene spacer is reported to give variable results depending on the number of thiophene rings <93SM4796>.

2.12.5.3.1 Vinylene

Vinylene linkers can be introduced into a polymer by a number of methods. One method is to first prepare a saturated masked linker that can be later oxidized to the alkene. Poly(2,5-thienylenevinylene) is prepared from the 580°C pyrolysis of [2.2](2,5)thiopheneophane <89MI 212-11>. When this film is treated with ddq the vinylene polymer is obtained (Scheme 23) <89CL729>. Several publications report the preparation of sulfonium precursors starting with 2,5-bis(methylenedimethylsulfonium halide)thiophene <87CC1448, 87MI 212-11, 90MCLC169, 90MCLC211, 90SM95, 91SM327>. This material is converted to its methoxy polymer (also of interest) and then to the vinylene product (Scheme 24). Thiophene-2-carbaldehyde derivatives also serve as starting materials.

Table 1 Various 3-substituted polythiophenes.

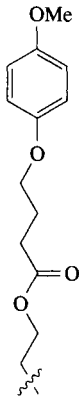
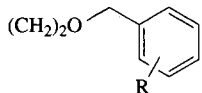
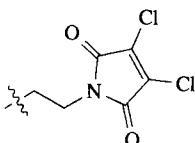
R^1	R^2	Ref.
H	H	87MI 212-12, 87SM233, 88JEC467, 89MI 212-05, 90SM107, 92JPS(A)1667, 92MM1214
H	Me	87JPC6706, 87MI 212-12, 87SM233, 87SM277, 88SM153, 90SM107, 92MM1214, 92SM109, 93MAC2483, 93MM3462, 93PS(74)471
H	Et	87SM233, 87SM277
H	Bu ⁿ	87SM233, 87SM277, 89NJC863, 93JOC904
H	<i>n</i> -C ₆ H ₁₃	91SM1045
H	cyclopentyl	93MM3462
H	<i>n</i> -C ₈ H ₁₇	92MI 212-09
H	OMe	87SM71, 88MI 212-08, 88SM277, 89MAC1649, 89SMC487
H	OC _n H _{2n+1} <i>n</i> = 4, 6	88SM153
	7	93SM582
	2, 3, 10-15	89SMC487
H	OCH ₂ (CF ₂) _n CF ₃ <i>n</i> = 2, 6	92JEC357
H	OCH ₂ OC ₄ H ₉	93SM582
H	O(CH ₂) ₂ O(CH ₂) ₂ OMe	93SM582
H	TMS	93JEC447
H	CH ₂ OMe	88SM153, 93JA11608
H	CH ₂ OC _n H _{2n+1} <i>n</i> = 4, 6	88SM153
	6	93SM582
H	CH ₂ OCH ₂ CH ₂ O-Me	93JA11608, 88SM153
H	CH ₂ (OCH ₂ CH ₂) ₂ OMe	93JA11608, 88SM153
H	CH ₂ SMe	93JA11608
H	CH ₂ NHCO-C _n H _{2n+1} <i>n</i> = 1, 7, 11	88SM153
H	CH ₂ CO ₂ Et	91JEC279
H	CH ₂ CO ₂ C ₁₇ H ₃₅	93MI 212-11
H		92SM109
H	(CH ₂) ₂ OSO ₂ -C ₆ H ₄ -CH=CH ₂	89SMC495
H	(CH ₂) ₂ OSO ₂ -C ₄ H ₉	91SM583
H	(CH ₂) ₂ O(CH ₂) ₂ -OMe	89CC679, 92MM6425
H		90CC414
	R = H, 4-F, 4-CF ₃ , 2,3,4,5,6-F ₆	
H	(CH ₂) ₂ O(CH ₂) _n O-C(O)Fc <i>n</i> = 6, 8, 10, 12, 16 Fc = ferrocene	92L959
H		93SM315

Table 1 Continued.

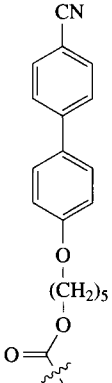
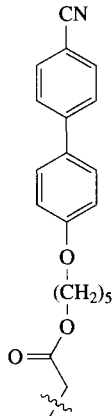
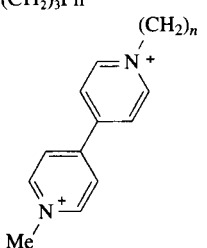
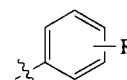
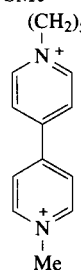
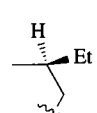
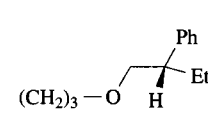
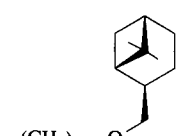
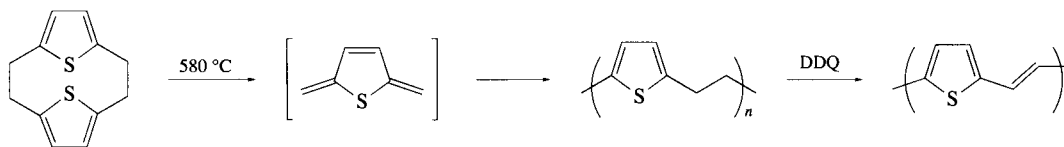
R^1	R^2	Ref.
H	$(\text{CH}_2)_n\text{SO}_3\text{H}$ $n = 2, 4$ 3 $n = 2, 6, 10$	88SM153 91SM333 93MM7108
H		92SM109
H	CO_2H	92MI 212-11
H	CHO	89MAC1649
H	CHNOH	89MAC1649
H	CN	89MAC1649, 92MM1214
H	SMe	87SM277, 88SM277, 89SMC481
H	SiMe_3	87MI 212-12
H	GeMe_3	87MI 212-12
H	SnMe_3	87MI 212-12
H	Me	87SM277
H	$n\text{-C}_9\text{H}_{19}$	89NJC863
H	$n\text{-C}_{12}\text{H}_{25}$	87MAC1763, 88SM369
H	CH_2Ph	87SM233
H	$\text{C}_n\text{H}_{2n+1}$ $n = 2-18$ 4, 6, 8, 10 6, 8 2, 4, 6, 12 5, 8, 12, 16 5, 12 6, 8, 12, 18 6, 8, 12 5, 7, 11, 17, 19 12, 18, 22 6, 12, 22	87JPC6706 93JOC904 93MAC2483 89MI 212-05 91MI 212-09 88MI 212-06 90MI 212-09 92MM1214, 92MM4364 88SM153 88MI 212-07, 93SM4168 90MI 212-10
H	CHF_2	93CM752
H	CF_3	93CM752
H	$(\text{CH}_2)_3\text{R}_f = \text{C}_4\text{F}_9 \text{ or } \text{C}_6\text{F}_{13}$	90JEC355
H		92SM109
H	$(\text{CH}_2)_2\text{OH}$	92MI 212-10
H	$(\text{CH}_2)_2\text{O}(\text{CH}_2)_3\text{-Me}$	91SM583, 92SM109
H	$(\text{CH}_2)_2\text{OC}_5\text{H}_{11}$	93SM582
H	$(\text{CH}_2)_2\text{OCH}_2\text{-CH=CH}_2$	91SM583

Table 1 Continued.

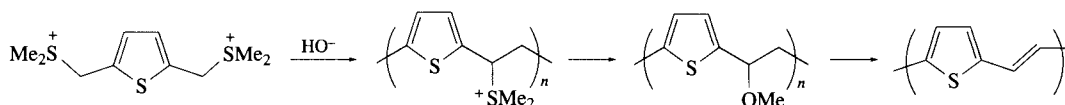
R^1	R^2	Ref.
H	$(\text{CH}_2)_n\text{O}(\text{CH}_2)_m\text{Me}$ $n = 1-3$ $m = 0, 1, 4-6$	89NJC863
H	$(\text{CH}_2)_2\text{O}_2\text{CC}_5\text{H}_{11}$	92MI 212-10
H	$(\text{CH}_2)_2\text{O}_2\text{C-CH=CH}_2$	91SM583
H	$(\text{CH}_2)_n\text{SO}_3\text{M}$ $\text{M} = \text{H, Li, Na, etc.}$ $n = 2, 4$	B-89MI 212-06
H	$(\text{CH}_2)_n\text{CO}_2\text{H}$ $n = 2$	91PP196
H	4, 5, 7, 10, 14	90AM(2)490
H	$(\text{CH}_2)_2\text{Ph}$	91MI 212-10
H	$(\text{CH}_2)_3\text{Ph}$	91MI 212-11
H	 $n = 5$	91MI 212-10, 91SM3037
H	 $R = \text{H}$	90AM(2)185, 91MM2694, 93MM3462
H	4-10	93CM1755
H	H, 4-OMe, 4-CF ₃	89MAC1233
H	2-, 3- or 4-OMe	
H	2-, 3- or 4-OC ₈ H ₁₇	
H	4-C ₈ H ₁₇	92MM4297
H	4-C ₆ H ₁₃	93SM1260
H	4-C ₈ H ₁₇	92MI 212-11
Me	CO ₂ Me	93SM582
Me	OC ₇ H ₁₅	93SM582
Me	O(CH ₂) ₂ OC ₄ H ₉	89MAC1649
OMe	OMe	91MI 212-11
OC ₄ H ₉	OC ₄ H ₉	89SMC481
SMe	SMe	
H	 	91SM3037
H		88CC917
H		88CC658
H		87MI 212-13

These are either pinacol-coupled using low-valent titanium or reacted with thiophene phosphonates under Horner–Wittig reaction conditions. The *E*-alkene is the major isomer in either reaction sequence. With the proper choice of starting materials and reaction conditions, thiophene derivatives such as (**145**) can be prepared and subjected to polymerization conditions <88MI 212-13, 91H(32)991, 92MI 212-13, 93SM4777>. Poly(3,4-dibutoxy-2,5-thienylenevinylene) (**146**) is also prepared by titanium-induced coupling of 3,4-dibutoxythiophene-2,5-dicarbaldehyde <93CL1085>. The Stille coupling

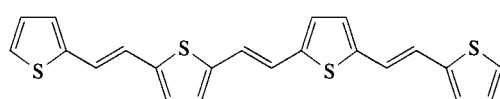
<86AG(E)508> of *E*-1,2-bis(tri-*n*-butylstannyl)ethylene with a 2-iodo-3-substituted thiophene affords monomer **(147)** <93SM1188>. When 2,5-diiodothiophene derivatives are used, the polymer **(148)** is obtained directly <93MI 212-12, 93SM1255>. Thiophene-2,5-dicarbaldehyde can also be subjected to benzoin condensation conditions to afford **(149)** after methylation with dimethylsulfate <93MI 212-13>.



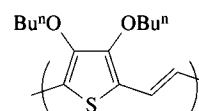
Scheme 23



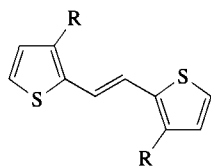
Scheme 24



(145)

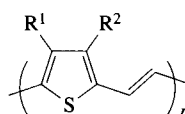


(146)



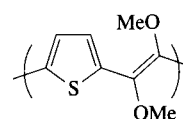
(147)

R = Me, Buⁿ, *n*-C₈H₁₇



(148)

R¹ = R² = Buⁿ
R¹ = *n*-C₁₀H₂₁; R² = H



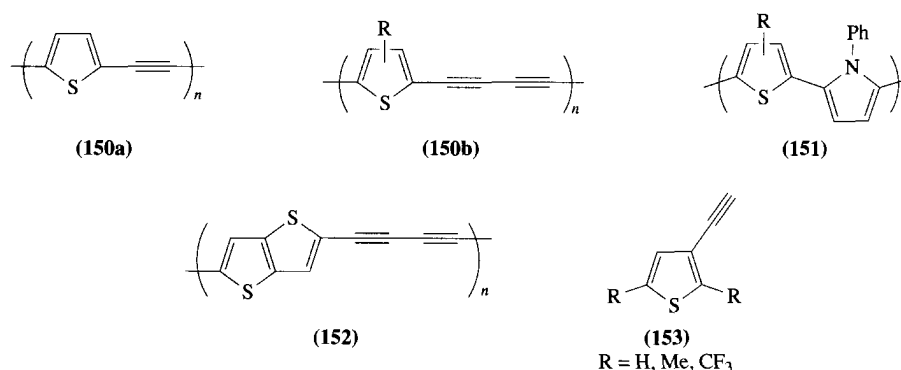
(149)

2.12.5.3.2 Alkyne

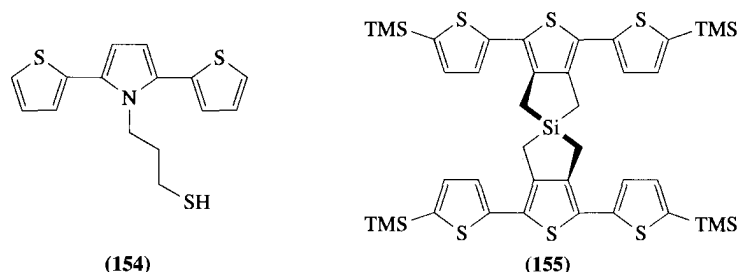
Thiophene polymers that contain alkyne spacers have also been investigated. In the simplest cases, the coupling of 2,5-diiodothiophene with 2,5-diethynylthiophene affords poly(2,5-thiophenediyl)ethynylene (**(150a)**) <89G309, 92MI 212-14, 93SM271>. When 2,5-diethynylthiophene is oxidized with CuCl/O₂, the polymer **(150b)** is isolated <88MM3528, 88MM3530, 89G309, 92MM2294>. This diyne polymer can be subjected to either thermal or photochemical reactions to afford a new polymer with high cross-linked density. The reaction of **(150b)** with aniline/CuCl at 150 °C produces polymer **(151)** <88MM3530>. Poly(2,5-thieno[3,2-*b*]thiophenylene) (**(152)**) <90PP643, 92MM2294> as well as the polymer obtained from **(153)** <90JFC445> have been reported. Selected thienylethynylene oligomers have properties similar to those of the corresponding poly(2,5-thiophenediyl)ethynylenes <92JEC123, 93SM81, 93SM271>.

2.12.5.3.3 Aryl

A number of examples exist wherein aryl and heteroaryl moieties are interposed between two thiophene units. Oligomers that contain a furan or selenophene between two thiophenes are reported <89PS(42)171, 90PS(48)239> as well as the polymers shown in Table 2. Of particular interest is the incorporation of an *N*-alkanethiol group to produce **(154)**. This thiol group may serve as an anchor to gold or platinum surfaces and allows for electropolymerization directly on the metal surface.



Another unique modification of the aryl spacer is the preparation of **(155)** <90JA5662, 92MI 212-16, 93JA1869>. This orthogonal arrangement of polythiophene units produces a conducting polymer that can act as a molecular switch <88JA5687>.

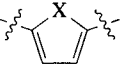
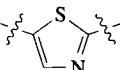
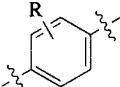
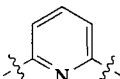
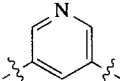
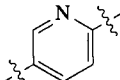
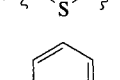
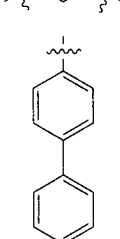
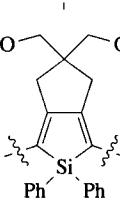


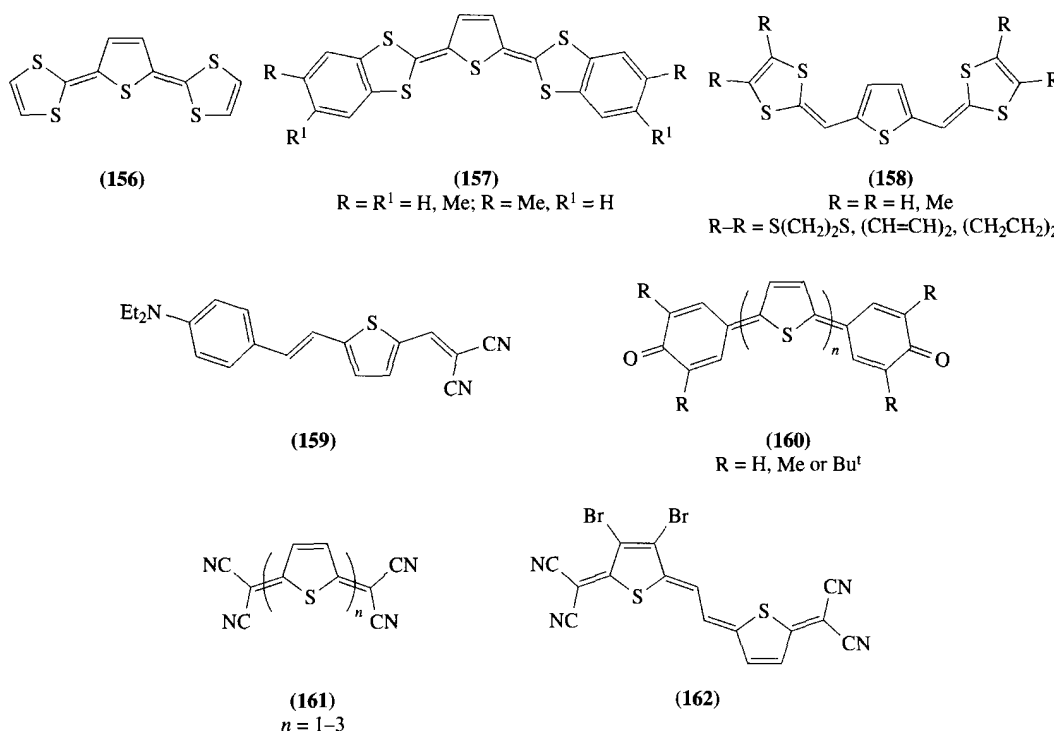
2.12.5.4 Charge-transfer Complexes

Thiophenes are not only used to prepare conducting polymers but are also used as the backbone to various conducting charge-transfer complexes. There is intense interest in preparing new types of highly conductive π -donor complexes. The 1,3-dithiolylienes **(156)** and **(157)** are “Wertz-type” donors with a coplanar configuration and produce stable radical cations and dication when treated with tetracyanoquinodimethane, ddq or iodine <89TL2091, 93CC1617>. These tetrathiafulvalene cations are stabilized by the conjugative effect of the thiophene sulfur atom; this extended conjugation may be applicable to the discovery of nonlinear optical materials, liquid crystals, and transistors as well as organic metals. Conjugation-extended tetrathiafulvalene analogues **(158)** <92TL6457, 93BCJ2330> show higher oxidation potentials than **(156)** and form charge transfer complexes with iodine and ddq that have relatively high conductivities [$(1.0\text{--}1.1) \times 10^{-3} \text{ S cm}^{-1}$] <93BCJ2330>. Donor-acceptor analogues of stilbene, such as **(159)**, show that thiophene rings increase second-order optical hyperpolarizability <93CC90>. Quinones **(160)** exhibit half-wave oxidation and reduction potentials indicative of a new type of amphoteric redox molecule <89JA5483, 91JA4576>. The amphotericity is enhanced by heteroatom radical stabilization. Related molecules that incorporate an additional quinoid ring into conjugation using a cyclopropane linkage also have amphoteric redox properties <91TL3507>. Electron acceptors such as **(161)** show very small on-site Coulomb repulsion and produce highly conductive charge transfer complexes with electron donor molecules such as hexamethylenetetrafulvalene <87CC1816, 89BCJ1539>. The unsymmetrical vinylogous compound **(162)** also forms highly conductive charge transfer complexes with donor molecules such as tetrathiotetracene <93SM2136>.

The fusion of thiophene rings to 5- or 6-membered rings or to other thiophene rings also produces molecules with interesting conductivity. Cyclopenta[2,1-*b*;3,4-*b'*]dithiophen-4-one <91CC752> and the derived dicyanomethylene analogue **(163)** <91CC1268> are both monomers that form electroactive polymers. The polymer of **(163)** displays a very low band gap ($E_g \sim 0.8 \text{ eV}$). Metal complexes of molecules with extended π -frameworks are also of interest because of their metallic behavior with high conductivities at low temperatures. A new acceptor benzodithiophene molecule **(164)** forms a Cu(I) complex with extremely interesting semiconducting properties <91CL1033, 91SM1847>. A series of thiophene-fused tetracyanoquinodimethanes (TCNQ) papers report the synthesis and physico-

$$\left(\text{S} \text{---} \text{[Aryl]} \text{---} \text{S} \right)_n$$

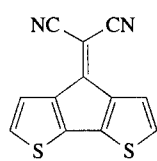
<i>Aryl</i>	<i>Reference</i>
	89CC1318, 90MI 212-11, 92MI 212-15
X = O, NH, NR	
	88MI 212-09
	87CC764, 87MI 212-14, 87MI 212-15, 90MI 212-12, 90MI 212-13, 91MM6787
R = H	
Me	90MI 212-12, 91MM678
OH	91MI 212-13
2,5-diOMe	90MI 212-12, 91MI 212-13, 91MM678
2,5-di(C ₁₆ H ₃₃)	91MI 212-14
	88MI 212-09, 93SM879
	88MAC1755
	88MI 212-09
	93SM1251
	88MAC1755
	87CC764, 88MAC1755
	92JA5867



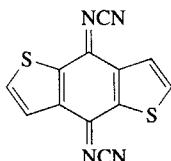
chemical properties of (164) as well as its three benzodithiophene isomers (92BCJ2168, 92BCJ2173, 92BCJ2180, 92BCJ2187). The thieno[3,2-*b*]thiophene derivative (165) ($R = H$) is a multistage redox system consisting of both the Wurster and Weitz types (90CC1196). A related derivative [$R = 3,5$ -bis(*t*-butyl)-4-hydroxyphenyl], prepared from a cyclopropenethione, is a novel three-color electrochromic system (dependent on the donor metal) (88AG(E)560). Other thieno[3,2-*b*]thiophene quinone derivatives, for example (166) ($R = H$), are electron-acceptors under redox conditions (91CL1117, 91TL4367, 91ZN(B)353). By varying the substituent R on the 2,5-bis(cyanimino)-2,5-dihydrothieno[3,2-*b*]thiophene moiety (166), a wide range of redox potentials may be achieved (92CB1235). The crystal structure of (166) ($R = Br$) shows it to form stacks of donor and acceptor molecules in an unusual segregated pattern (92CB1919). Bis(dicyanomethylene) derivatives of the 2,5-dihydrothieno[3,2-*b*]thiophene ring system are reported along with the tricyclic compounds (167) (89BCJ1547). Bis(thiodimethylene)tetrathiafulvene (168), (BTDM-TTF) has a low intramolecular Coulombic repulsion energy (89TL7249). The TCNQ and ddq charge transfer complexes of (168), isomer (169) as well as unsaturated analogues are described (91SM2205). In a related series, cation radical salts of bis(2,5-dimethylthieno)tetratellurafulvalene (170) have high conductivity (92CC1198). Further tailoring of the TTF and thiophene units produces compound (171) and its vinylogue (172) which form conductive charge transfer complexes with TCNQ and iodine (93CC431, 93JCS(P1)905). The crystal structure of the tetracycle thieno[2',3'':4',5']thieno[2',3'-*d*]thieno[3,2-*b*]thiophene (173) ($R = H$) as well as its charge-transfer complex with TCNQ show that the molecules stack in a zig-zag fashion with short intermolecular $S-S$ distances (92JCS(P2)761, 92JCS(P2)765). These hydrogen-poor condensed polythiophenes form inclusion compounds containing DMSO whose conformation is dependent on whether the number of condensed thiophene rings is odd or even (92CC1381). When R is a 9-hydroxyfluorenyl substituent, these polythiophenes crystallize with 4-amino-2,2,6,6-tetramethylpiperidine 1-oxyl (ATEMPO) to form stable radicals that have interesting magnetic properties (92CC1661).

2.12.6 BENZOTHIOPHENE- AND OTHER BICYCLIC(THIOPHENE)-CONTAINING POLYMERS

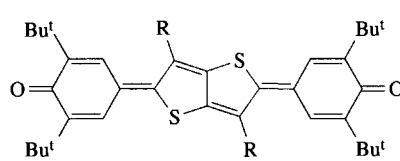
Conductive polymers made of poly(benzo[*b*]thiophene) are the subject of only a few publications (87MI 212-16, 88SM33, 91MI 212-15). More papers discuss the preparation and theoretical nature of



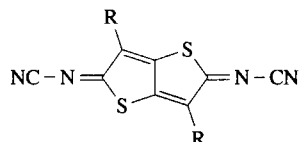
(163)



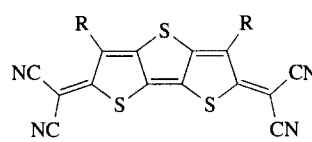
(164)



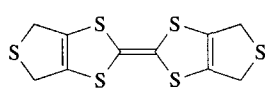
(165)



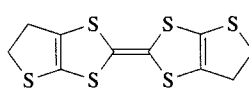
(166)



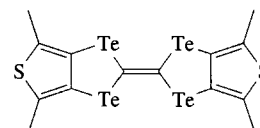
(167)



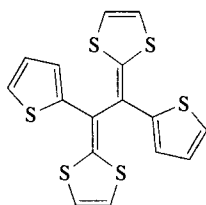
(168)



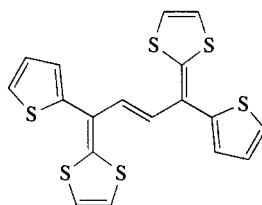
(169)



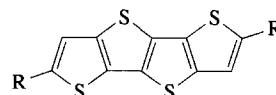
(170)



(171)



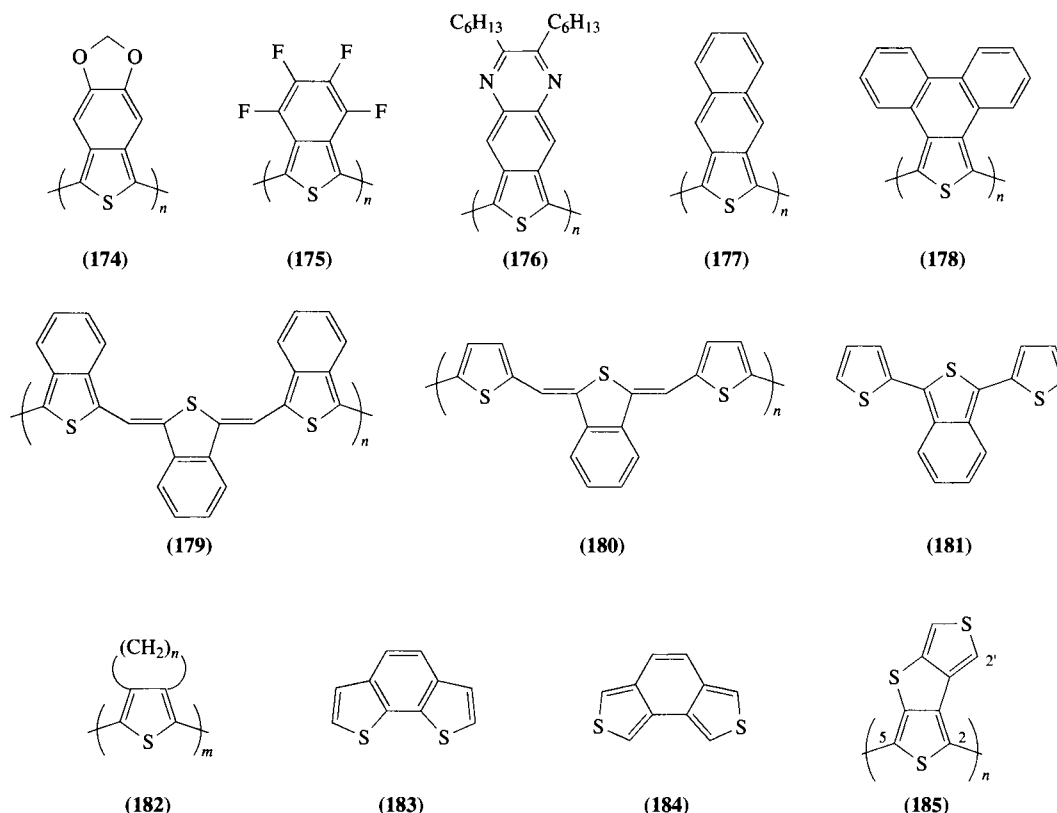
(172)



(173)

various poly(benzo[*c*]thiophene) [also known as poly(isothianaphthene), PITN]. Poly(benzo[*c*]thiophene) is of particular interest since it is a conductive polymer with a small band gap ($E_g \sim 1.0$ eV) (90MM2237, 91CC1618, 92MM7347, 92SM367). Low band gap polymers do not require doping to produce high conductivities since, at room temperature, there is sufficient thermal energy to excite electrons to the conduction band ($\pi \rightarrow \pi^*$). Modification of PITN has produced some interesting results. The poly(5,6-dioxymethyleneisothianaphthene) (174) was prepared in attempts to improve the oxidative stability of PITN (91SM1) while the perfluorinated polymer (175) was found to have excellent solubility in several solvent systems (90JCS(P1)1919, 93SM281). The dihexylpyrazine analogue (176) is reported to have a low band gap ($E_g = 0.95 \pm 0.1$ eV) (93SM960). In efforts to further reduce the band gap, poly(naphtho[2,3-*c*]thiophene) (177) (B-88MI 212-13, 90SM263) and poly(phenanthro[9,10-*c*]thiophene) (178) (93JEC305) were prepared. Compound (178) exists in the quinoid form whereas the other polymers are benzenoid in nature. This electronic difference was analyzed by MNDO and Hückel calculations (87MI 212-17, 90CM526, 92MM4652) as well as by the synthesis of known quinoid compounds such as poly(biisothianaphthene-methane) (179) (91JA9865) and polymer (180) (91CB1597, 93S634, 93SM1193). The isothianaphthene oligomer (181) was also prepared and polymerized (92AM(4)562, 93CC172).

The preparation of thiophene bi- and tricyclic derivatives provides another means to further modify polymer solubility and/or band gap. Poly(cyclopenta[*c*]thiophene) (182; $n = 3$) (87CC1500) as well as the higher homologues ($n = 4, 5, 10$; $m = 3$) are reported (90SM79); however, the desired increase in polymer conductivity does not occur. Polymerization of the annelated bithiophenes (183) and (184) is reported (89SMC521). Several publications describe the preparation and properties of poly(dithieno[3,4-*b*:3',4'-*d*]thiophene) (185) (88CC246, 89MI 212-13, 89SMC507, 89SMC527, 90MCLC519, 91SM579, 92MI 212-17). This thiophene polymer displays a strong electrochromic effect between semitransparent (conducting) and opaque (resistive) states. The band gap of the neutral polymer is 1.1 eV which is similar to PITN and 0.9 eV lower than that of polythiophene. The 2,5-attachment (vs. 2,2' or 2,5' or 5,5') of this polymer is suggested by molecular modeling using either MM2 (89SMC527) or valence effective Hamiltonian (VEH) (93MM1260) calculations.



2.12.7 LIQUID CRYSTALS

A majority of compounds with liquid crystalline properties are comprised of a ridge core (mesogenic) group that has a flexible hydrocarbon chain attached at one or both ends. Other combinations of these groups are also possible; for example, two mesogenic groups attached by a flexible chain with each mesogenic group containing a hydrocarbon chain attached (or not) to it (Figure 2) <93ZN(B)826>.

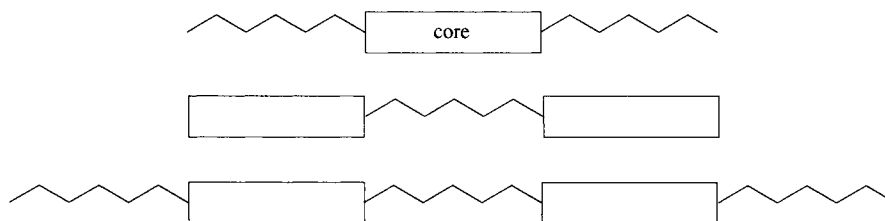
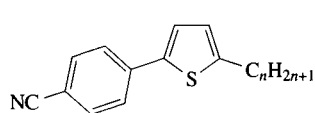


Figure 2 Diagrammatic structures of liquid crystals.

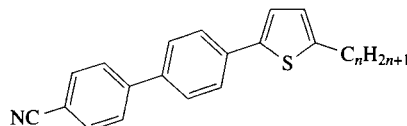
A 1,4-substituted phenyl moiety is often used as the mesogenic group and, when this moiety is replaced with a thiophene moiety, the sulfur heteroatom has a strong influence on mesomorphic phase formation. The sulfur atom not only changes the polarity of the local bonds, but also the polarizability and the geometry of the mesogenic group. This results in a modulation of the phase transition temperature and the dielectric constant, as well as in many other physiochemical parameters. Seemingly small changes in the mesogenic group or spacer can make significant changes in the liquid crystalline properties. An in-depth review of each article is beyond the scope of this review.

The use of the nonlinear cyanophenylthiophene moiety (186) provides liquid crystals with only monotropic phases whereas the corresponding cyanobiphenylthiophenes (187) have wide nematic ranges <92CL613, 93LC515>. An application of Figure 2 is reported wherein two thiophene mesogenic groups are linked by an alkane group (188) <93ZN(B)826>. The liquid crystalline properties of this series depends largely on the mesogenic tail and the alkane spacer. For example, a 4'-butoxybiphenyl-

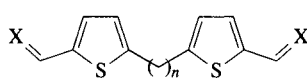
4-yl imino series shows enantiotropic smectic and nematic mesophases while a 2-fluorenylvinyl series has only enantiotropic nematic mesophases for $n = 4$ or 6. Another series investigated contains two thiophene mesogenic groups which are separated by an ethylene moiety (**189**) <93LC383>. In general, these compounds do not display liquid crystalline properties when the mesogenic tail is another 2,5-substituted thiophene. The only exception to this was *E,E*-5-[5-(5-butyl-2-thienylvinyl)-2-thienylvinyl]-2-thiophenecarbaldehyde which displayed a monotropic nematic phase.



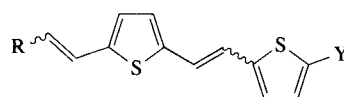
(186)
 $n = 3-11$



(187)
 $n = 4-6$

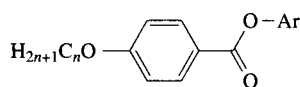


(188)
 $n = 3-12, 16$
 $X = \text{ArN or ArCH}$

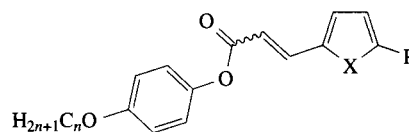
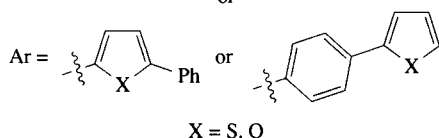


(189)
 $R = 4\text{-butylphenyl or } 2,5\text{-substituted thiophene}$
 $Y = \text{CHO or H or } \text{C}_4\text{H}_9$

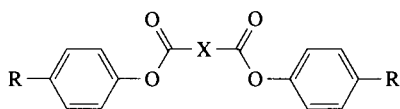
There are a number of examples where a thiophene carboxylic ester is linked to a phenoxy ether moiety. The liquid crystalline properties of a homologous series of heterocycle-containing esters (**190**) are compared with the corresponding all-benzenoid esters ($X = \text{CH=CH}$) <89MCLC121>. The related series (**191**) which uses an acrylate spacer has been reported <84BCJ2404>. Additional examples of thiophene-containing esters are (**192**) and (**193**) <83ZN(B)1669, 91LC617>.



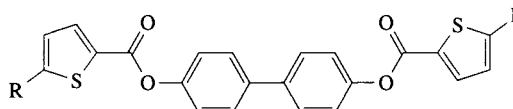
(190)
 $n = 1-8, 10, 12$
or



(191)
 $n = 3-8$
 $R = \text{Br, CN}$
 $X = \text{S, O}$



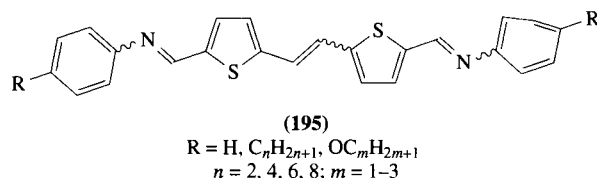
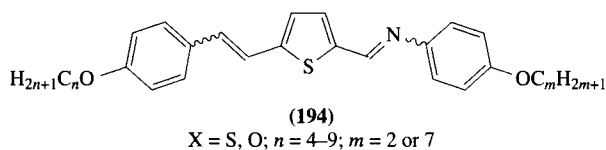
(192)
 $R = \text{C}_n\text{H}_{2n+1}$
 $n = 1-10$
 $X = 2,45\text{-thiophene or } 2,2'\text{-bithiophene-5,5'}$



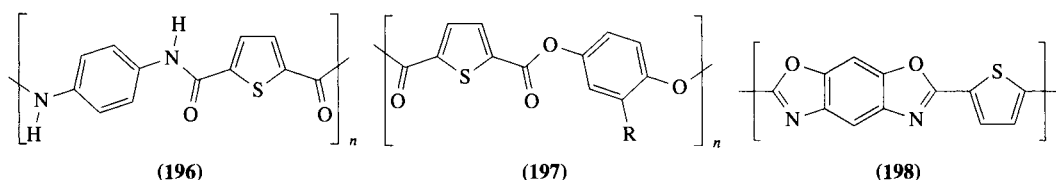
(193)
 $R = \text{C}_n\text{H}_{2n+1}$
 $n = 0-8$

The imine derivatives (**194**) were investigated and it was determined that the styrylthiophene series exhibited liquid crystalline properties while the corresponding styrylfuran series did not <93LC115>. A series of papers report the liquid crystalline properties of thiophene derivatives that contain bisimine (e.g. (**195**)) or bisethylene groups <85ZN(B)1199, 86ZN(B)751, 87ZN(B)478>. The thermal and optical characteristics of these compounds compare to their analogous all-benzenoid compounds.

Thiophenes have also been used to make liquid crystalline polymers. Polyamides (**196**) are reported as potentially useful high performance polymers which compare to poly(*p*-phenylene terephthalamide) (PPTA or Kevlar) <91PP213, 92PP1194>. The polyesters (**197**) were investigated to examine the effects of nonlinearity on mesophase stability <92MM563>. The insertion of these



nonlinear 'defects' into the polymer produced a lower melting temperature. A polysiloxane ester that contains styrylthiophene units is also reported <92ZN(B)567>. The modified poly(benzoxazole) **(198)** is reported where the 2,5-thiophene unit replaces the 1,4-phenylene unit of the high performance PBX polymers <91PP211, 93MM1793>.



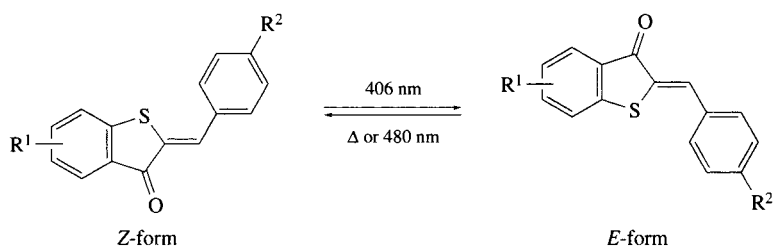
2.12.8 DYES

2.12.8.1 Coloring Agents

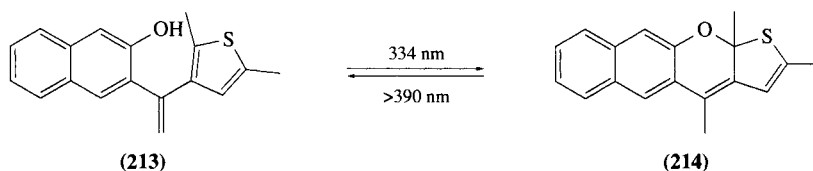
Several reviews describe the use of thioindigo **(199)** and 2,3'-bithioindigo **(200)** as dyes <82MI 212-01, 84CHEC-I(4)863, 86PAC1207, 88MI 212-14>. A significant number of thiophene and benzothiophene compounds have been prepared and studied as styryl or azo disperse dyes, fluorescent dyes and/or fluorescent brighteners. Color is only one important aspect of a dye; it must also have good light and sublimation fastness. Styryl compounds such as Foron Blue SR **(201)** and its cyano analogue **(202)** are dyes with a color range from yellow to greenish-blue <89DP37>. In general, incorporation of the thiophene moiety in dyes often produces a color deepening effect. The derivative **(203)** is reported <93MI 212-14> and may be viewed as the open form of 4-(coumarin-3-yl)thiophene **(204)**. Electron-withdrawing group substituents ($R = CO_2Et$ or CN) at the 3- and 5-position on **(204)** results in deepening of the dye hues on polyester fibers <92PS(71)1, 93SUL263>. Azo compounds such as **(205)** are dyes that display colors from brilliant red ($Ar = 5\text{-cyano-2,6-dihydroxy-4-methylpyridin-3-yl}$) to blue [$Ar = 2\text{-chloro-4-bis(2-hydroxyethyl)aminophenyl}$] <90MI 212-14>. The electronic effect of the thiophene ring was studied by CNDO/S and by the PPP method <85DP31>. Azo dyes of benzo[*b*]thiophene **(206)** are reported where R is an electron-withdrawing group (CN , CO_2Et or $CONH_2$) <90MI 212-15, 90MI 212-16, 92MI 212-18>. The tetrahydrobenzo[*b*]thiophene azo compounds **(207)** are reported ($R = CO_2Et$ or CN) <89DP295> as well as the thieno[2,3-*b*]pyridines **(208)** <92MI 212-19> and **(209)** <91MI 212-16, 92MI 212-20>. Some additional azo examples are the 3,4-dihydroxy derivatives **(210)** <91MI 212-17> and **(211)** <92MI 212-21> and the hydrazone series **(212)** <92MI 212-22>. All of these compounds have good to excellent sublimation fastness with varying degrees of light fastness (depending on Ar).

2.12.8.2 Optical Devices

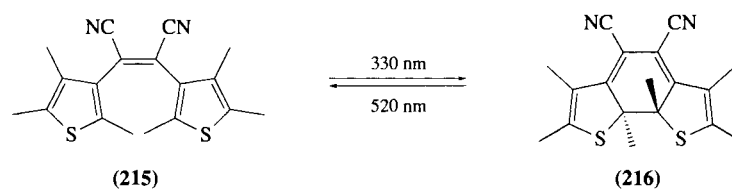
The use of thiophene or benzothiophene in polyester dyes is only one aspect of the interest in these compounds. They absorb light at a specific wavelength that causes photochemical (see Section 2.12.4.2) or photophysical changes that either alter their structure or the structure of the surrounding



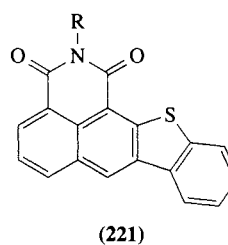
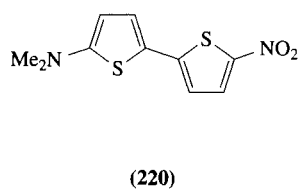
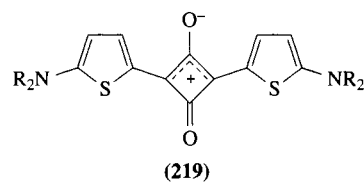
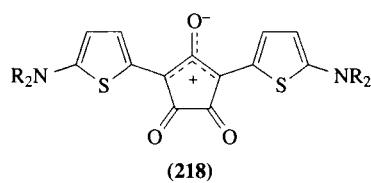
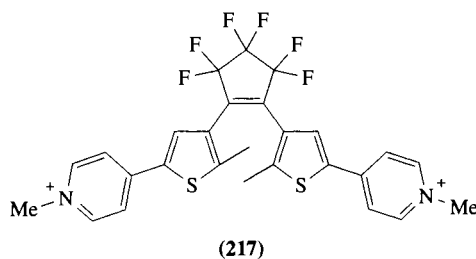
Scheme 25



Scheme 26



Scheme 27



2.12.9 CYCLOPHANES

Several examples exist wherein a thiophene ring is incorporated into a cyclophane framework <91PHC(3)70, 92PHC(4)62>. The [2]metacyclo[2](2,4)- and -(2,5)thiophenophanes, (222) ($R^2 = \text{CH}_3$) and (223), are subjected to reductive ring-opening to afford [7]- and [8]metacyclophanes (Equation (18))

<91CB411>. Thiophenophane (**222**) ($R^2 = H$) is best prepared by vacuum pyrolysis. A mixture of phenaleno[1,9-*bc*]thiophene derivatives is isolated from (**224**) (Equation (19)) <92JOC746>. The synthesis of the [2]metacyclo[2](*m,n*)thiophenophane isomers is reported (Figure 3) <91JOC2837> as well as the dynamic behavior of [2]metacyclo[2](3,4)thiophenophane <92JPO617>. The [3]metacyclo[3](2,5)thiophenophane (**225**) was prepared and studied by ^1H NMR <86JOC1551>. Dioxazolo[3 ^{2}]metacyclo(2,5)thiophenophane was synthesized by alkaline condensation of thiophene-2,5-dicarbaldehyde and 1,3-bis(2-isocyano-2-tosylethyl)benzene (Equation (20)). This thiophenophane has unexpected rigidity as a consequence of the 'large' sulfur atom as well as the longer C—S bonds <89CPB2303>. Synthesis of dithia[3]paracyclo[3](2,4)- and -(2,5)thiophenophanes (**226** and **227**, respectively) allows for the study of desulfurization as well as ring inversion <91CB1403>. The octamethyltetrasila[2.2](2,5)thiophenophane (**228**) was synthesized and studied by photoelectron spectroscopy to measure through-space vs. through-bond interactions. These measurements compare to those of the all-carbon analogue <88JA4117>. The trimethylene-bridged [2.2](2,5)thiophenophane (**229**) forms by a cobalt mediated [2 + 2 + 2]cycloaddition (Equation (21)) <88TL4529>.

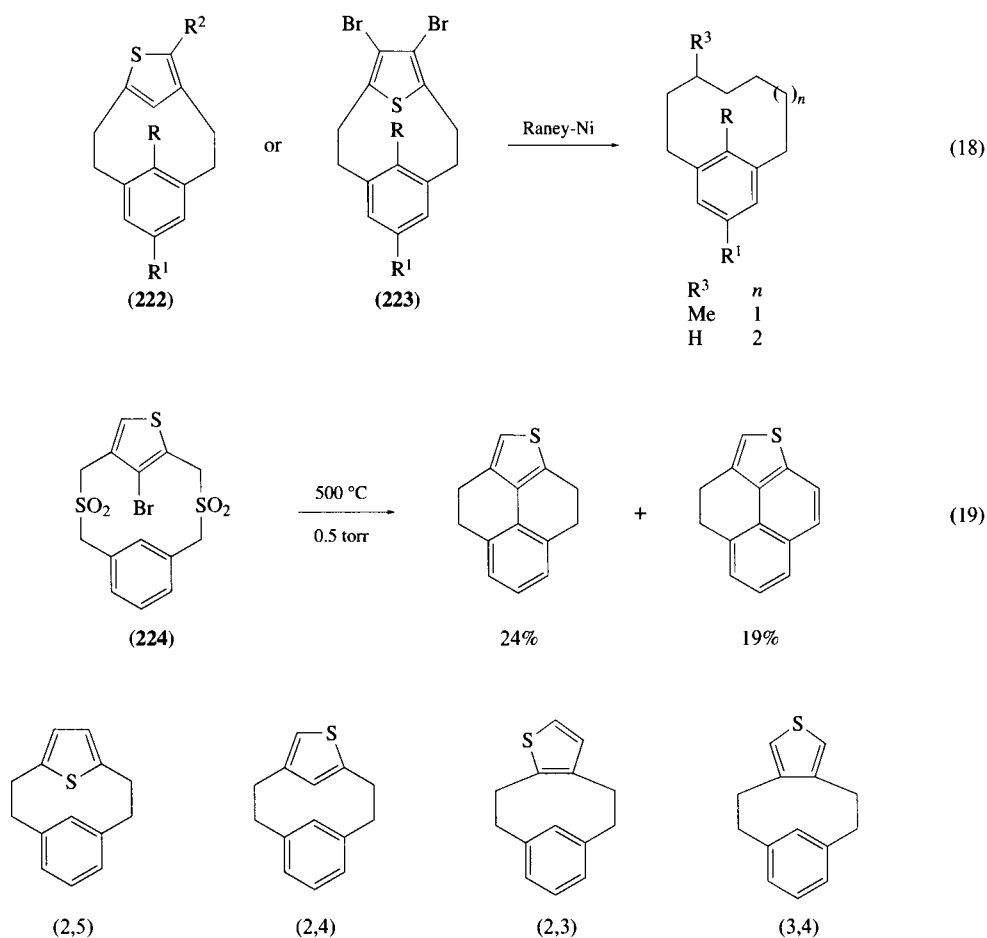
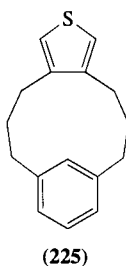
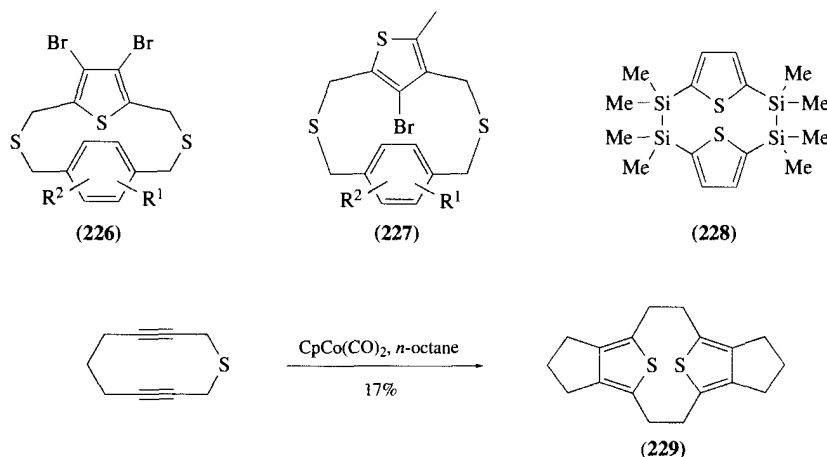
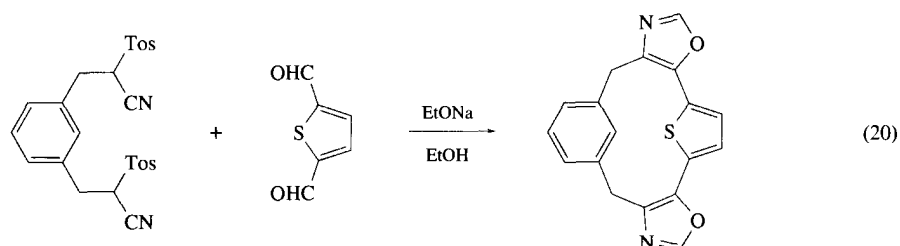


Figure 3 Isomeric [2]metacyclo[2](*m,n*)thiophenophanes.

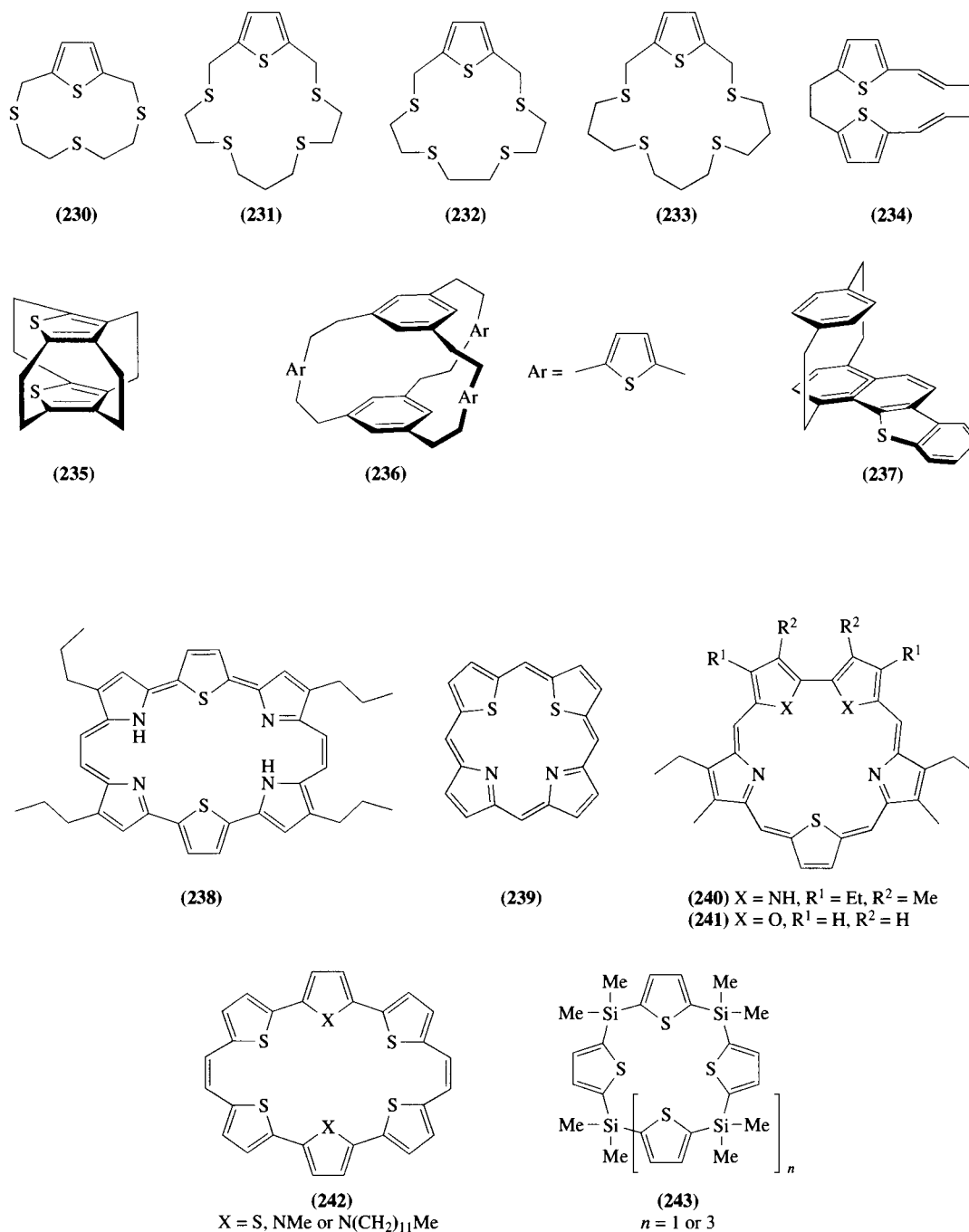




Thiophene ring-containing cyclophanes also demonstrate unique properties. The copper complexing properties of 2,5,8-trithia[9](2,5)thiophenophane (**230**) and its homologue (**231**) (also known as thiocrown ethers) were studied $\langle 88\text{CJC}1506, 89\text{CJC}639 \rangle$. 1,6-Bis-(2-thienyl)-2,5-dithiahexane and its trithianonane analogue form $\text{Mo}(\text{CO})_4$ and $\text{W}(\text{CO})_4$ complexes. 2,5,8,11-Tetrathia[12](2,5)-thiophenophane (**232**) also forms molybdenum and tungsten complexes $\langle 92\text{IC}2354 \rangle$ as well as a binuclear silver complex $\langle 90\text{CJC}644 \rangle$. The preparation of (**233**) is also reported; however, metal complexation is not described $\langle 89\text{MI} 212-14 \rangle$. (*E,E*)-[6.2]-(2,5)Thiophenophane (**234**) forms by pyrolysis of a trimethylammonium hydroxide precursor; (**234**) has structural features which are favorable for cyclopolymerization to form polymers with stacked aromatic rings $\langle 92\text{TL}2633 \rangle$. [2₄](2,3,4,5)Thiophenophane (superthiophenophane, (**235**)), the first reported “ultimate” heterophane, forms from 3,4-bis(chloromethyl)-2,5-dimethylthiophene and exhibits compression effects between facial *p*-orbitals of the thiophene ring carbons in the ^{13}C NMR $\langle 92\text{JOC}4654 \rangle$. The bicyclopheane (**236**) is another example of [2₄]cyclophane where the thiophene ring defines an interesting cavity $\langle 82\text{ACS(B)}661 \rangle$. The ^1H NMR, visible and infrared spectra of dibenzothiophene (e.g., (**237**)) charge-transfer complexes with ddq, TCNE or 9-dicyanomethylene-2,4,7-trinitrofluorone are reported and compared with those of the corresponding phenanthrene system $\langle 87\text{ZN(B)}1142 \rangle$.

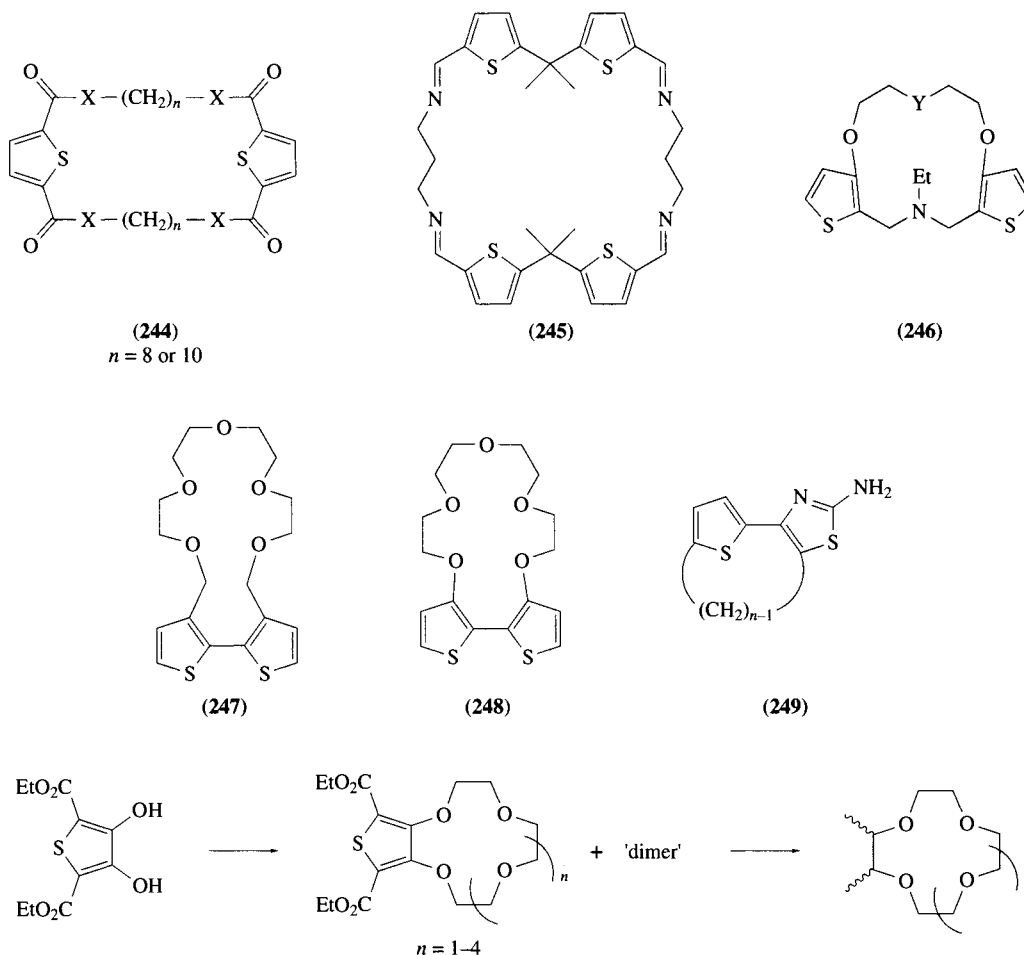
2.12.10 MACROCYCLES

The preparation of porphyrin-like macrocyclic molecules incorporating a thiophene nucleus (such as (**238**) or (**239**)) is reported $\langle 92\text{JHC}523, 92\text{JOC}4414 \rangle$. These macrocyclic ligands have potential as drugs for photodynamic therapy, as metallic chelates for catalysis, or as electrical conductors. Sapphyrins, members of a class of macrocycles known as “expanded porphyrins,” also have potential in the fields of photodynamic therapy and magnetic resonance imaging. The 27-thia- and 25,29-dioxa-27-thiasapphyrin analogues (**240**) as well as (**241**) are prepared from 2,5-bis(chloromethyl)thiophene and subsequent conversions $\langle 92\text{T}9661 \rangle$. The 28- π annulene (**242**) is formed by McMurray coupling of α -terthiophene-5,5''-dicarboxaldehyde $\langle 93\text{TL}1879 \rangle$. Electrochemical studies demonstrate that, when $\text{X} = \text{S}$, a dication is formed while the other annulene forms only a radical cation. The tetrathiophene macrocyclic molecule (**243**) forms when dimethyldi(2-thienyl)silane is treated with two equivalents of LDA followed by one equivalent of dichlorodimethylsilane $\langle 92\text{H(34)}1085 \rangle$.



Thiophene-containing macrocycles are designed as potential inclusion hosts for organic molecules or ligands for selected metals. 2,5-Thiophenedicarbonylchloride is converted by [2 + 2]-cyclizations into ester and amide macrocycles (**244**) <92PS(73)41>. This reaction did not require the use of templates or high dilution techniques. Nontemplate synthesis is also used to prepare the macrocyclic tetraimine Schiff base (**245**) <89BCJ1346>. A variety of aza crown ethers (**246**) are derived by condensation of 3-hydroxythiophene with α,ω -bifunctional alkanes and subsequent ring closure by formation of Mannich base linkages <93CC1733>. These macrocycles form complexes with various mono- and divalent metals. Interesting crown ethers form from 2,5-bis(ethoxycarbonyl)-3,4-thiophenediol and are subjected to Raney nickel desulfurization to afford a new crown ether (Scheme 28) <89BCJ838>. The crown ethers (**247**) and (**248**) are prepared from 2,2'-bithienylene <93ACS184>. Design of con-

ducting polymer-based sensors takes advantage of the metal coordination properties of these bithienylene crown ethers, the ease of polymerization of thiophene as well as the excellent conductivity properties of such derivatives (93JA12214). The macromolecule (249) has design features to enhance transport through a cell wall membrane by incorporating a 2,5-linked thiophene moiety.

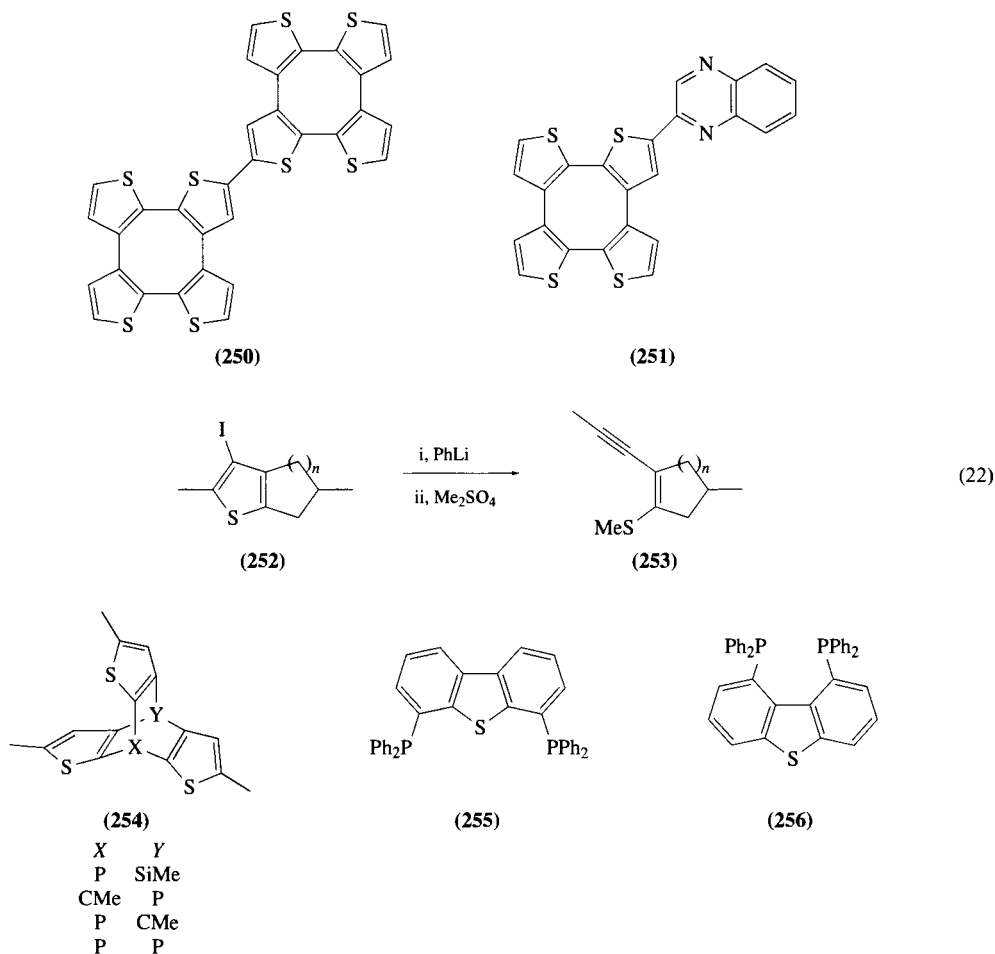


Scheme 28

2.12.11 ORGANOMETALLIC AND COORDINATION CHEMISTRY

2.12.11.1 Lithium and Magnesium

The preparation of various thiophene and benzothiophene organometallic derivatives was reviewed in several articles during the years between 1982 and 1993. These reviews discuss the preparation of organolithium compounds and organomagnesium compounds (83H(20)1127, 84CHEC-I(4)713, 84CHEC-I(4)741, 84CHEC-I(4)863, 86HC(44)159, 92HC(44)257) as well as organocopper compounds (84CHEC-I(4)741, 86HC(44)159, 92HC(44)257). Concerning the last group of references, thiophene plays a special role in the higher order mixed organocuprates (86ACS(B)466, 87JOM(334)255, 87S325). The lithiation of (all- α -S)-cyclotetrathiophene provides the 2-lithio derivative that can be used to prepare, *inter alia*, (250) or (251) (85CB2343). The ring opening of 3-lithio derivatives, described in Section 2.12.4.4, is further exemplified for the bicyclic compound (252) to afford (253) (Equation (22)) (83ACS(B)895). The organolithium derived from 4-bromo-2-methylthiophene is used to prepare triptycenes (254) (93TL2347, 93TL8259). The two-fold lithiation of dibenzothiophene with *n*-butyllithium produces the bidentate phosphines (255) and (256) (93TL2107).



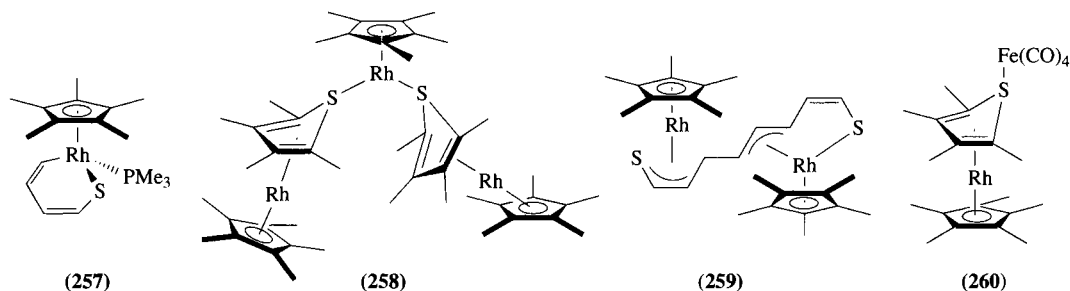
2.12.11.2 Hydrodesulfurization (HDS)

As discussed briefly in Section 2.12.4.5, thiophene compounds, mercaptans, and disubstituted sulfides are the most prevalent sulfur impurities in fossil fuels. These impurities are removed by treating crude oil with hydrogen at 400°C over catalysts such as a cobalt–molybdenum sulfide that is supported on alumina. The mechanism of this hydrodesulfurization (HDS) has theoretical <92JMOC65, 93CPL(212)525, 93JMOC219> and commercial importance and is the subject of numerous reviews that investigate mechanism, variation of catalyst and substrate, as well as product composition <85HC(44)457, 87CCR279, 90CCR61, 90PHC(2)50, 91PHC(3)70, 92CCC2509, 92PHC(4)62, 93CCR237, 93PHC(5)82, 94PHC(6)88>. Two general models for homogeneous HDS have evolved. The first model involves π coordination of one double bond or the entire π system (η^4 - or η^5 -C₄H₄S), while the second model involves insertion of the metal into the carbon-sulfur bond through an *S*-bound complex <92JA151>. A large number of metals have been investigated as HDS catalysts.

2.12.11.2.1 Rhodium

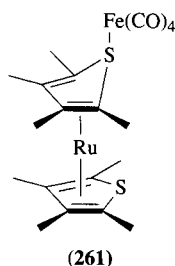
The oxidative addition of the thiophene C—S bond to the metal center of (C₅Me₅)Rh(PMe₃)PhH is postulated to occur by the sequential *S*-coordination to the metal with subsequent migration of the α -carbon to the metal to produce (257) <91JA559, 92JA151, 93MI 212-15>. Similar reactions occur with substituted thiophenes, benzothiophene or dibenzothiophene. The diene portion of (257) undergoes Diels–Alder reaction with dienophiles such as dimethyl acetylenedicarboxylate to afford dimethyl phthalate <91JA559>. When the rhodium species is [(COD)Rh][BF₄], a η^5 -2,5-dimethylthiophene complex is formed <93OM3504>. A η^6 -complex is formed with benzothiophene or di-

benzothiophene. The thermolysis of $(C_5Me_5)Rh(\eta^4-C_4Me_4S)$ causes elimination of tetramethylthiophene and formation of $[(C_5Me_5)Rh]_3(\eta^4, \eta^1-C_4Me_4S)_2$ (**258**) <92JA1732>. On the other hand, the reaction of thiophene with $(C_5Me_5)Rh(C_2H_4)_2$ leads to ring-opening and C—C bond formation to afford dimer (**259**) via the intermediacy of $(C_5Me_5)-(Rh-\eta^4-[CH=CHCH=CHS])$ <92AG(E)357, 92JA9851>. Protonation studies of rhodium-diene complexes which contain η^4 -thiophenes suggest a method to effect hydrogen transfer to thiophene during the course of HDS <92JA8515>. Base hydrolysis of thiophene coordinated to $[(C_5Me_5)Rh(C_4Me_4S)]^{2+}$ leads to the formation of rhodium-coordinated acyl thiolates <92JA8521>. The preparation of the related dicationic benzo[b]thiophene complex is reported <88OM686>. Thermolysis of $(C_5Me_5)Rh\{\eta^4: \eta^1-C_4Me_4S\}Fe(CO)_4$ (**260**) provides insight into the dual role of transition metals in the HDS process.



2.12.11.2.2 Ruthenium

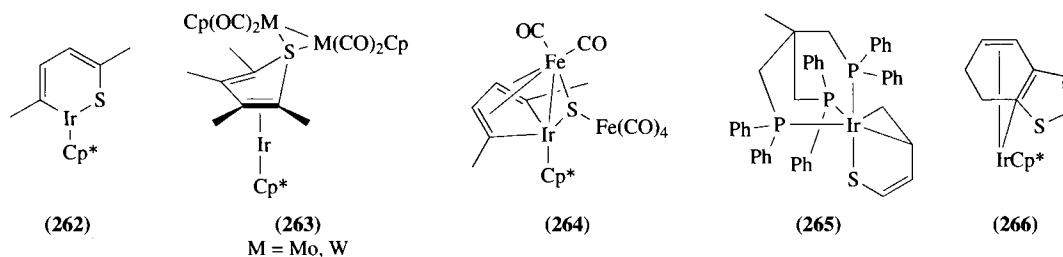
Metals other than rhodium also complex thiophene and provide insight as to thiophene absorption on to the catalyst surface. Transition metals function in a dual role wherein bis(tetramethylthiophene)ruthenium(0) reacts with $Fe(CO)_4$ to form a stable symmetrical compound (**261**) with the two pairs of C=C groups eclipsed <92OM3497>. In an effort to further elucidate the HDS process, studies of the C—S cleavage of various ruthenium (η -thiophene) complexes by deuteration in either acidic <91OM270, 92JA8515, 93JA4943> or basic media <85JA5569, 87OM1146, 93OM3273> are reported. Nucleophiles such as MeO^- , MeS^- <87OM1897> or hydride <87AG(E)909, 88JOM(355)359> also cleave the C—S bond of these complexes. The cluster $[Ru_3(CO)_{12}]$ oxidatively cleaves 2-methylthiophene by C—H as well as S—C bond cleavage <92JCS(D)2423>. An alumina-supported ruthenium carbonyl-alkali metal hydroxide system provides superior HDS activity towards dibenzothiophene than the corresponding nonalkali metal hydroxide system <92CL2285>. Deuteration studies of ruthenium benzo[b]thiophene complexes are also reported <88MI 212-15, 93IC1871>.



2.12.11.2.3 Iridium

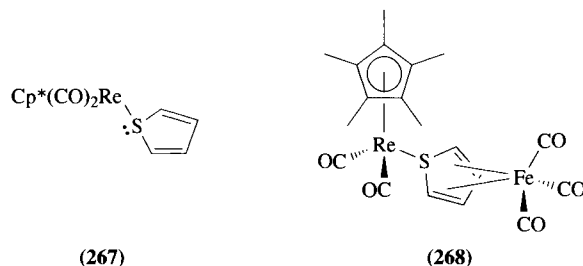
Two-electron reduction of $Cp^*Ir(\eta^5-2,5-Me-thiophene)$ produces a η^4 -thiophene complex which forms the more stable, ring-opened C,S-isomer (**262**) <90JA199, 90POL1883>. Ultraviolet photolysis of the same $Cp^*Ir \eta^5$ -complex also produces (**262**). Reaction of (**262**) with molybdenum or tungsten triple-bond dimers produces complex (**263**) wherein sulfur is coordinated to three metals <90OM879, 92MI 212-23>. Reaction of (**262**) with alkynes forms novel chelated bicyclocarbene complexes <92OM992>. Compound (**262**) also reacts with iron carbonyls <91JA2544> to produce (**264**) as well as other Lewis acids <89OM2277, 90OM849>. *In situ* formation of the iridium complex ' $Ir(PMe_3)_3Cl$ ' reacts

with benzene, pyridine and furan to give products of oxidative C—H addition whereas thiophene and benzothiophene give C—S ring-opened addition <93OM1583>. The cationic iridium species, $(\text{COD})_2\text{Ir}^+$, affords the η^5 -thiophene complex <93OM3504>. Homogeneous iridium complexes react with thiophene to give **(265)** which subsequently forms butadienethiolate complexes that undergo further transformations <93JA2731>. In a similar fashion, reaction with benzothiophene ultimately produces 2-ethylbenzenethiol <93JA7505>. Hydride addition to $\text{Cp}^*\text{Ir}(\eta^6\text{-benzothiophene})^{2+}$ produces a cyclohexadiene complex **(266)**. Dibenzothiophene reacts in a similar fashion <92JOM(428)415>.



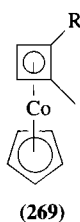
2.12.11.2.4 Rhenium

The S-bound thiophene complex **(267)** forms by the reaction of $\text{Cp}^*(\text{CO})_5\text{Re}(\text{THF})$ with thiophene; **(267)** may further react with $\text{Fe}_2(\text{CO})_9$ to produce **(268)** <89JA8753, 91OM2436>. Benzo[*b*]thiophene and dibenzothiophene gives a similar S-bound complex <91JA4005, 91OM2436, 92OM922, 92OM3328>. Homogeneous hydrogenolysis of thiophene using $\text{ReH}_7(\text{PPh}_3)_2$ leads to the formation of 1-butanethiolate <92JA10767>. A similar reaction occurs for benzo[*b*]thiophene <91JA4005>.



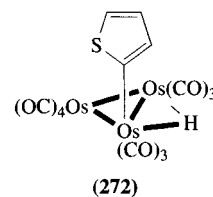
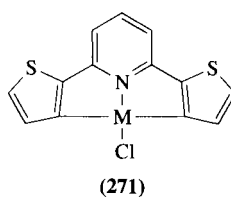
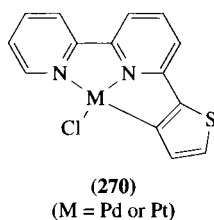
2.12.11.2.5 Other metals

Other examples of transition metal complexes with thiophene include cobalt <92OM2698>, nickel <89JOC4848>, manganese <84JA2901, 87OM591>, iron <86JOM(316)335, 87IC3424, 88OM1171, 91IC3957>, nickel boride <90CC819, 92JOC1986> and platinum <93JA12200>. Many of these same reports include complexes with benzo[*b*]thiophene or dibenzothiophene, as well as a report on HDS of benzo[*b*]thiophene with an aqueous platinum species <91CJC590>. The HDS of dibenzothiophene by silica–alumina supported molybdenum gives higher yields of reduced compounds, for example biphenyl, than does conventional sulfided molybdena–alumina catalysts <93CL589>. Thiophene is also reduced to unsaturated thiol-like species with ZSM5 zeolites <92JPC2669>. Iron complexes of thiophene 1,1-dioxide has been explored <90JOM(399)163>. The flash vacuum pyrolysis of $\text{CpCo}(\eta^4\text{-thiophene 1,1-dioxide})$ effects the loss of SO_2 and the formation of **(269)** <82OM1545, 85OM389>. The preparation of a $[\text{Cr}(\eta^6\text{-benzo}[3,4\text{-}c]\text{thiophene})(\text{CO})_3]$ is reported <93JA6448>.



2.12.11.3 Cyclometalation

A number of examples exist wherein the transition metal reacts with thiophene by carbon–hydrogen bond cleavage and, by so doing, provides a reactive thiophene derivative. The cyclopalladation of *N*-(3-thienyl)pyrazole provides both the 2- and 4-substituted compounds in a 3 : 1 ratio <82JOM(229)287>. This result is contrary to that for lithiation which provides only the 2-metalated product. Cyclopalladation of *N,N*-dimethylthiophene-3-carboselenoamide affords only the 2-palladated material <92ICA183>. Cyclometalation of 6-(2-thienyl)-2,2'-bipyridine produces (270) <91AG(E)1363, 92JCS(D)2251> and the cyclometalation of 2,6-bis(2-thienyl)pyridine affords (271) <92JCS(D)2467>. Electrophilic thallation of thiophene is also reported <92OM4352>. The osmium compound $[\text{Os}_3(\text{CO})_{10}(\text{MeCN})_2]$ oxidatively adds to thiophene to give the hydrido product (272) <90CC1568, 92JOM(436)351>.



2.13

Selenophenes

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2.13.1 INTRODUCTION

The scope of this chapter was first defined at the beginning of Chapter 3.16 in the first edition of *Comprehensive Heterocyclic Chemistry* (CHEC-I). Since then, a general review on selenium heterocycles has been published (B-86MI 213-01). A few more specific works have also appeared

concerning: the chemistry of selenophenes <82AHC(30)127, B-85MI 213-01, B-86MI 213-03, 88PS(38)149, 91PHC(3)70>, the ring opening of chalcogenophenes by means of lithiation reactions <B-83MI 213-01>, the molecular motions in metal carbonyl derivatives <83MI 213-04>, and the electrical and optical properties of conductive polychalcogenophenes and their applications <87MI 213-01>. This chapter will cover the literature through to 1993 (e.g., *Chemical Abstracts*, volume 118) with the same order of appearance of the material as in CHEC-I.

2.13.2 THEORETICAL METHODS

Numerous theoretical studies have been realized on selenophenes in order to compare them with their chalcogeno congeners. Indeed in this case the series is complete from oxygen to tellurium derivatives. One of the most fascinating and controverted parameters is the aromatic character. A new index (I_5) based upon a statistical evaluation of the deviations in peripheral bond orders (derived readily from experimental bond length) has been proposed for aromaticity quantization <85T1409>. The values for the furan series are: O: 43; S: 66; Se: 59; Te: 48, and so show a reasonable parallelism with A, the aromaticity parameter obtained from the dilution shifts method. More recently, this index has been improved <92T335>, and the values of I_5 are the following: O: 53; Te: 59; Se: 73; S: 81.5 (compared with benzene: 100).

The characterization by *ab initio* molecular orbital theory of chalcogen isosteres of benzene <88JA4204> has allowed the prediction of structures with a precision of 0.04 Å for the bonds and 2° for the angles. In this approach, the selenophene series shows important delocalization. New theoretically derived and energy-based criteria for aromaticity were proposed <90IJQ843, 90PHC(2)50>.

The extent of transmission of substituent effects in selenophene and several condensed derivatives was deduced from CNDO/2 quantum chemical calculations on C-protonated forms or σ complexes <82CS75, 82CS208>. Quantum chemical calculations (CNDO/S) of spectroscopic parameters of selenophene and several condensed derivatives have been used to determine their ionization potentials <86IZV2251>.

Several physicochemical parameters (first electron affinity, valence ionization potential, and photoionization cross-section) were determined by the Green's function <82MI 213-02>. The SCF-INDO perturbation theory calculations established the contributions of Fermi contact, orbital-dipole, and spin-dipole to $J^{13C-77Se}$ in several selenophene derivatives <82MI 213-03>. The PRMO-SCPT-INDO of chalcogenophenes indicates that spin couplings are transmitted through the σ network and that orbital dipole interactions play a lesser role than in benzene <83MI 213-02>. In a similar way, SCPT-INDO calculations of spin-spin coupling constants in selenophene show a good correlation with the experimental values <85MI 213-02>. Force field calculation procedures were developed with the aim of getting information about the influence of substituent effects on the aromatic ring force field in 2-bromo- and 2-chloroselenophenes <87CPL(138)244, 88MI 213-01>. Numerical values of force field constants, refined from experimental frequencies, show as previously established, a noticeable variation of aromaticity in the chalcogenophene series <85SA(A)679>.

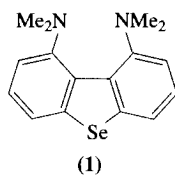
Comparison of the calculated geometries, ionization energies, dipole moment, and net atom charges indicated that the 3-21G basis should include a d polarization function on selenium <89MI 213-01>. Physicochemically-based semiempirical MO calculations showed a fair correlation between the average $\nu_{C=O}$, the average ionization energy of the carbonyl lone pair orbitals, and the $\delta^{13C=O}$ for seleno and tellurophthalic anhydrides. Their dipole moments in benzene at 25°C are respectively, 3.30 D (Se) and 2.71 D (Te) <82SA(A)185>.

2.13.3 EXPERIMENTAL STRUCTURAL METHODS

2.13.3.1 Molecular Structure

Several x-ray structures of complexed selenophene derivatives have been published: $(C_4H_8Se)_2 \cdot Hg^{II}Cl_2$ <86AX(C)1449>; $(C_4H_8Se)_2 \cdot HgBr_2$ <88AX(C)253>; $Cp(CO)_2 Re$ selenophene <90JA7811, 91JA5651>. Complete characterization of a selenium analogue (1) of proton sponge has been achieved by a crystal structure determination <88TL1905>.

X-ray diffraction shows that the 2-lithiobenzo[*b*]selenophene-TMEDA complex is dimeric, where the diamine acts as chelating ligand between the lithium-bridged benzo[*b*]selenophenes <89OM(1)688>. Oxygen and sulfur analogues behave similarly.



Trinuclear carbonyl clusters of Ru, Fe, and Os react with selenophene in an insertion manner to give products whose structures have been determined by x-ray diffraction <91JOM(419)63> and which decompose at high temperature.

Dimethyl ethynedicarboxylate and elemental selenium react with η^5 -cyclopentadienyl-dicarbonylcobalt to give several selenophene derivatives, whose structures have been confirmed by x-ray diffraction, among them the unexpected 2,3,4,5-tetramethoxycarbonyl-selenophene <92PS(68)37>.

Electronic and structural modifications of polyselenophenes during electrochemical doping were studied by *in situ* x-ray spectroscopy <86MI 213-05>. Microwave spectra of various α -deuterated selenolanes allowed the establishment of their molecular structure <92MI 213-01>.

2.13.3.2 NMR Spectroscopy

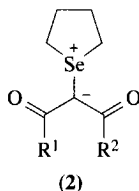
Besides routine structure determinations, NMR spectroscopy has been used for structure correlations and chemical shift predictions.

The total structure of selenophene has been determined by NMR (^1H , ^{13}C , and ^{77}Se) on the liquid crystal medium <82OMR(19)173>. The same study carried out on benzo[*b*]selenophene evidenced, besides its structure, a significant solvent effect on ^1H chemical shifts <84JOM(277)359>.

As expected, substituent effects on $\delta^{13}\text{C}$ and $\delta^1\text{H}$ in the chalcogenophene series led to a qualitative determination of four different substituent classes depending on the heteroatom, and its interaction with the substituent. This method allows the prediction of chemical shifts <90MRC397>.

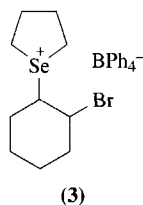
From values of $J^{13}\text{C}-^{13}\text{C}$ in 25 selenophene derivatives, it appeared that the σ -electronic effects are the most important factors governing the observed values <88ZOR2268>. Moreover, a quantitative measurement of π -electron deficiency or excessiveness $^n\Delta$ was proposed from $\delta^{13}\text{C}$ values. For the chalcogenophene series $^n\Delta$ is as follows: O: 1.31; S: 1.35; Se: 1.43; Te: 1.83 (compared with benzene: 1). It allows a more accurate prediction of ^{13}C chemical shifts in substituted heteroaromatic compounds <82OMR(19)192>. Spin-spin coupling pathways in chalcogenophenes were analyzed by the polarization propagation method <86MI 213-02>.

There is a good correlation between the NMR chemical shifts (^1H , ^{13}C , and ^{77}Se) of selenophenes and those of their [*b*]benzocondensed analogues <81CS147> as well as with the CNDO calculated electron densities <82OMR(18)33>. An increments-based evaluation of substituent effects in other heterocondensed selenophenes was made by means of the same technique <83CS22>. An excellent linear relationship of $\delta^{77}\text{Se}$ and $\delta^{125}\text{Te}$ in several heterocycles is found and its slope is discussed on a theoretical basis. Experimental ^{77}Se chemical shifts and $J^{77}\text{Se}-^{77}\text{Se}$ in alkylselenoselenophenes <87MRC129> are discussed in relation to aromaticity and substituents <87CS359>. The structure of pivaloylchalcogenophenes was established by routine NMR techniques <85ZN(A)1329>. Selenolane ylides (2) were studied by ^{77}Se NMR <82ZOR119>.



Data from ^1H NMR show that selenonium salt (3) exists in solution as diequatorial conformers <86ZOR1553>. Complexes of selenolane with PdX_2 were examined by dynamic NMR <86JCS(D)205>. Surprisingly, ^{19}F chemical shifts of trifluoromethylsulfone derivatives of chalcogenophenes are rather insensitive to the substitution pattern and to the nature of the heterocycle <87JFC(36)439>. A NMR kinetic study of the Meisenheimer adducts formed by nitroselenophenes and methoxide anion showed the following order of decreasing stability $\text{S} > \text{O} > \text{Se}$ <88CS349>. The conformation of nitro-

3,3'-biselenienyls was deduced from NMR measurements using induced cholesteric mesophases <86JST307>. Proton and ^{13}C NMR spectroscopy are also useful for the characterization of polymeric selenophenes <82JPS(A)1697>.



2.13.3.3 Ultraviolet, Infrared, Raman, and Photoelectron Spectroscopy

Ultraviolet spectra of chalcogenophenes were investigated with the aim of exploring the Rydberg transitions terminating at the $3s$ orbital <88CS331>. Pivaloylselenophenes and their oxygen and sulfur containing congeners were routinely examined by IR and UV spectroscopy <85ZN(A)1329>. Low lying excited electronic states have been identified for the dibenzochalcogenophene series <89MI 213-03>. The phosphorescence spectra of benzo[*b*]- and dibenzochalcogenophene series were analyzed at 77 K in ethanol and *n*-pentane <89ZN(A)205, 89ZN(A)1116>. The rate constants of both radiative (K_{PT}) and nonradiative (K_{GT}) deactivation of the lower triplet correlate linearly with $\Sigma k\text{Ck}^2\zeta^2$ (where Ck denotes the Hückel AO coefficients in the HOMO of the carbon atoms bound to the heteroatom, and ζ is the spin orbit coupling constant of the heteroatom present). A linear correlation is observed between K_{PT} and K_{GT} as an example for the Orlandi-Siebrand rule.

The UV and luminescence spectra of benzo[*b*]selenophene-doped naphthalene show that the properties of localized excitation states are different <83MI 213-03>. Charge transfer complexes of selenophene and some condensed derivatives were examined by UV, IR, and photoelectron spectroscopy and the experimental ionization potentials are in agreement with the calculated ones <82CS214>.

The isomerization potential of selenoindigo was obtained from its electronic spectra <87JPC1317>. The fluorescence decay of the same molecule as an impurity in benzoic acid crystals has been observed and probably involves a double proton transfer <86CPL(131)44>.

Oxidation potentials of oligomeric 2-thienyl and selenienyls, obtained by classical palladium(II) coupling, were determined by UV spectroscopy <88CC988, 89PS(42)171, 90PS(48)239>. Photoluminescence has been observed also in polyselenophenes <89SMC293>. Luminescence spectra of polychalcogenophenes depend on the temperature and for polyselenophene the phenomenon disappears at high temperatures <90MI 213-01>. Fundamental vibrations in the IR and Raman spectra for benzo[*b*]selenophene and tellurophene were completely assigned and modes proposed. Their evolution with chalcogen change has been analyzed <83SA(A)1033>. Molecular vibrational constants and chemical bonding allowed the establishment of a ground state aromaticity ($\text{S} > \text{Te} > \text{Se} > \text{O}$), whereas this order is modified when the reactivity in electrophilic substitution is considered ($\text{O} > \text{S} > \text{Te} > \text{Se}$) (see Section 2.13.2 for comparison) <85MI 213-04>.

Due to the high quality of the spectra, FT-Raman is claimed as the preferred technique for routine vibrational spectroscopy <92JOM(430)37>. Dynamic disorder in tricarbonylchromium selenophenes has been characterized by low frequency vibrational spectra <82SA(A)1163>. The principal bands for polyselenophenes (H or D) have been assigned <89SM317>. *Ab initio* calculations of electron bonding energies obtained from photoelectron spectroscopy of substituted selenophenes in the gaseous phase are well correlated with the Hammett substituent parameters <83CS209>.

The electron transmission spectra of selenophene have been observed and discussed <84CPH455>. The nonplanarity of 2-arylselenophenes has been demonstrated by photoelectron spectroscopy <88ZOB450>. This technique has also shown an inversion of the HOMO upon [*b*]benzo condensation of selenophene <88IZV2616>. The system $\text{C}=\text{C}-\text{Se}-\text{C}=\text{C}$ has been studied by photoelectron spectroscopy and quantum chemical methods. There is a linear correlation for $\sigma_1-\sigma_2$ -MO energies for $\text{C}=\text{C}-\text{Se}-\text{C}=\text{C}$ and selenophene <91IZV2317>. Comparison of the photoelectron spectra of dibenzochalcogenophenes and LCAO calculations shows as expected the planarity of these systems <83JST(102)183>. Photoelectron spectra of the dibenzochalcogenophene series <88ZOB1409> and MO calculations identified a b_1 symmetry for the first HOMO for these compounds. The first ionization potential decreases in the order $\text{O} > \text{S} > \text{Se} > \text{Te}$. The second HOMO has a_2 symmetry and the

second ionization potential is fairly constant. In order to evaluate substituents effects in selenophene, selenium 3d core electron shifts were correlated with the CI shifts of the ring carbon atoms and with Hammett σ constants <86MI 213-07>.

2.13.3.4 Mass Spectrometry

Polyselenophenes were analyzed by laser desorption/FT mass spectrometry <86MI 213-04, 86MI 213-06>. Appearance potentials for the formation of negative ions in resonance electron capture of chalcogenophenes have been reported <91IZV590>.

2.13.3.5 Electron Spin Resonance

Anion radicals generated electrochemically from 29 derivatives of chalcogenophenes were examined by ESR and the results were discussed <83JST(102)183>. An ESR study of radical ions in the 2,5-diphenylselenophene series has been published <90JOM(389)301>. These spectra belong to systems conformationally rigid on the ESR timescale as evidenced by the nonequivalence of the *ortho* and *meta*-proton hyperfine coupling constants. For the O, S, and Se congeners, the spectra show the expected symmetry of the parent LUMO and antisymmetry for the HOMO.

2.13.3.6 Dipole Moments

Anisotropic molecular polarizabilities, dipole moments, and quadrupole moments of the selenophene series have been discussed in terms of comparison of experimental results and *ab initio* calculations. The results elucidate the electronic charge distributions in these molecules <92JPC7301>. The group dipole moments of 2- and 3-substituted selenophenes, depend upon the σ -acceptor and π -donor ability of the substituted heteroaryl moieties <85ZOR203>. The preferred conformation of some 2-acylselenophenes was deduced from their dipole moment analysis <85ZN(A)1338>. Measurements of the infinite dilution (in cyclohexane) molar field gradient birefringence constants, Kerr constants, and Cotton–Mouton constants for the chalcogenophenes series led to an estimate of the position of the effective quadrupole center of these molecules <83JCS(F2)539>.

2.13.3.7 Miscellaneous

Theoretical and experimental investigations into resonance hybridization between benzo[*b*]selenophene and naphthalene in benzoselenophene-doped naphthalene systems provided evidence for two nonequivalent centers for the impurity <85MI 213-03>. Chromatographic structural analysis by adsorption on graphitized thermal carbon black was used for the determination of conformation in 2-phenylchalcogenophenes <87MI 213-02>. The adsorption of selenophene on various solid phases allowed its direct observation by EXAFS <91MC6>.

Polyselenophenes were examined by NEXAFS and compared with the ISEELS spectra of selenophenes <89CJC1819, 90MI 213-04>. The presence of polarons and bipolarons may explain the experimental data on the evolution of optical and magnetic properties upon doping polyselenophenes <88MI 213-02>. A compilation (250 pages) of spectral parameters associated with triplet–triplet absorption of organic molecules and in condensed media has been published <86MI 213-08>.

2.13.4 THERMODYNAMIC CONSIDERATIONS

Several thermodynamics constants ($\Delta H^0_{\text{comb.}}$, $\Delta H^0_{\text{form.}}$, $\Delta H^0_{\text{evap.}}$, $\Delta H^0_{\text{atom.}}$) were determined by calorimetry for selenophene and its benzo[*b*]derivatives <89DOK(307)1139>. The existence of stable and metastable phases in crystalline selenophene has been demonstrated by thermodynamic evidence <91CR(C)701>.

The glass transition temperatures (T_g) were measured for selenophene copolymers and their

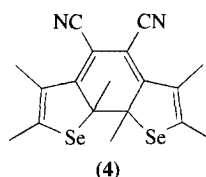
dependence upon the length of the lateral chain was discussed <89MI 213-04>. Low energy resonance states of molecular anions were determined for the chalcogenophene series <90KGS276, 90MCLC459>.

A compilation of thermodynamic parameters for heterocycles has been published <90MI 213-05>. Selenophene has been observed in the gasification of spiked coal by ICP <86JC465>.

2.13.5 REACTIVITY OF FULLY CONJUGATED RINGS

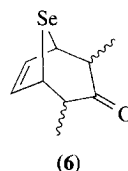
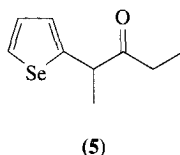
2.13.5.1 Thermal and Photochemical Reactions

Electron withdrawing groups on di-1,2-(3-selenienyl)ethenes show a thermally irreversible photochemical cyclization with relative high quantum yield <90JOC2592> giving (4).



Photochemical reaction of selenophene with 3-iodocoumarin afforded the 3-(2-selenienyl)coumarin <90KGS1469>. Selenophene has been used for MOCVD deposition of ZnSe <84EUP106537, 84MI 213-02>. The structural quality of these thin films has been studied by cross-sectional high resolution transmission electron microscopy and cathodoluminescence <85MI 213-05>. In the presence of $\text{Fe}_2(\text{CO})_9$ and $\text{Me}-\text{CHBrCO}-\text{CHBrMe}$, selenophene undergoes photochemical addition leading to the 2-substituted derivative (5) and the 1,5-dihydrobicyclic compound (6) <85PS245>.

Theoretical and experimental investigations into resonance hybridization between benzo[b]-selenophene and naphthalene in benzoselenophene-doped naphthalene systems provided evidence for two nonequivalent centers for the impurity.



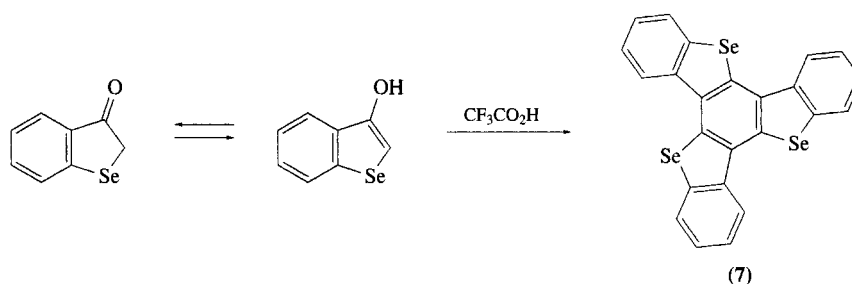
2.13.5.2 Electrophilic Substitutions

The perchloric acid catalyzed acylation of selenophene with acetic anhydride is claimed to give solely 2-acetylselenophene <81MI 213-01>. Ethoxalyl chloride reacts at the same position <83BEP896054>. Reaction with trifluoromethylsulfenyl chloride and stannic chloride gives 2-trifluoromethylthioselenophene, and addition of trifluoromethanesulfonic acid promotes formation of the 2,5-bis(trifluoromethylthio) derivative <83JFC(23)525>.

Electrophilic substitution on 2-(2-selenienyl)benzimidazole <88JCS(P1)1033> takes place in the selenophene moiety <83KGS1531, 87KGS1636>. New selenium containing D,L-amino acids were obtained by reacting selenium containing substrates with α -hydroxy Cbz glycine. With benzo[b]-selenophene, only the 3-isomer was isolated and the reaction failed with selenophene <81JHC1605>.

Treatment of selenoindoxyl in its enol form (3-hydroxybenzo[b]selenophene) with trifluoroacetic acid (Scheme 1) leads to a trimeric condensed compound (7) beside dimeric compounds <86CS287>.

Conventional palladium(II)-catalyzed coupling methodology allowed the formation of a number of oligo 2-thienyl or selenienyls <90H(30)645>. The thallation reaction of selenophene affords the 2-thallated species which give 2-iodoselenophene upon iodination <90MI 213-06>.

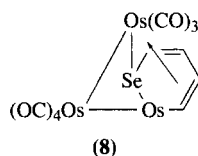


Scheme 1

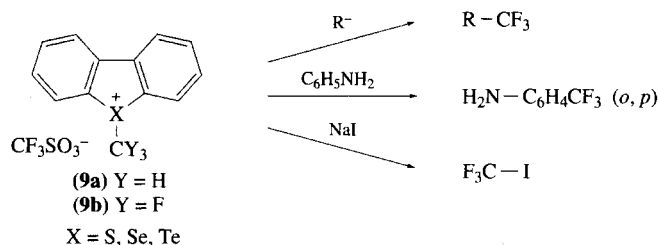
2.13.5.3 Reactions on the Selenium Atom

Much work has been devoted to the formation of complexes where selenophene acts as a ligand. For example, with first row transition metals selenienylthienylmethane forms stable molecular species <83MI 213-05>. The effect of substitution of either cyclopentadienyl or selenophene rings on the structure of η^2 - and η^1 -selenophene (sel) complexes $\text{Cp}(\text{CO})_2\text{Re}(\text{sel})$ has been discussed <91JA5651>.

Clusters are formed of $\text{Os}_3(\text{CO})_{10}(\text{MeCN})_2$ with selenophene of composition $\text{Os}_3(\text{CO})_{10}\text{C}_4\text{H}_4\text{Se}$ by both C—H and C—Se insertions, the latter affording compounds (8) <90CC1568>. Substituted selenophenes were also studied <92JOM351>.

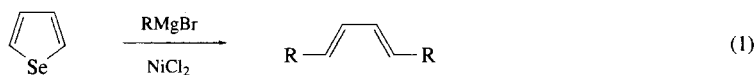


Salts readily obtained from dibenzochalcogenophenes (9) are powerful methylation agents (9a), even in water as solvent <89TL7293>, or trifluoromethylating electrophiles (9b) (Scheme 2) <90EUP382206, 90TL3579>.



Scheme 2

The reactivity depends on the aromaticity of the heterocycle and decreases in the order $\text{S} > \text{Se} > \text{Te}$ <93JA2156>. The NiCl_2 catalyzed reaction of Grignard reagents induces the ring opening of chalcogenophenes, with the loss of the chalcogen atom, giving the corresponding *s-cis*-1,3-butadiene (Equation (1)) whose structure has been proved by high resolution ^1H and ^{13}C NMR spectroscopy. The yields decrease in the series from Te to O <84CC617>.

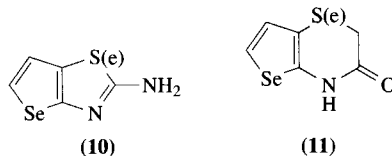


Some benzo[*b*]selenophenes have been used as models for catalytic dihydrodesulfuration studies <91DIS(B)5302>.

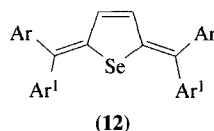
2.13.5.4 Nucleophilic Substitutions

Nucleophilic substitution of halogen in 2-(3)halo-3-(2)formylbenzo[*b*]selenophenes by nitrogen-containing nucleophiles is the first step for the formation of new tricyclic compounds <83JHC49>.

The role of Meisenheimer complexes was studied in the nucleophilic substitution of the nitro group by methoxide anion in 2- and 3-trifluoromethylsulfonyl-4- and 5-nitroselenophenes <83JCR(S)58>. The use of potassium thiocyanate and selenocyanate as nucleophiles with 3-bromo-2-nitroselenophene has allowed the elaboration of several condensed bicyclic selenophenes <83JHC113> as shown in Structures (10) and (11).

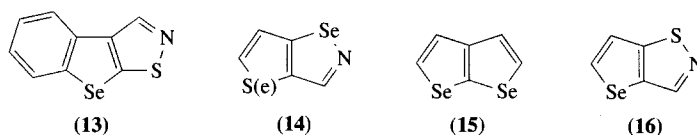


As an example of general methods for the synthesis of arylacetic acids, selenophene has been metallated by Bu^nLi . Transmetallation afforded the corresponding zinc compound which was coupled with good yields, with ethyl bromoacetate <83OM598> or bromoacetonitrile <87S40>. A new class of compounds (12) have been synthesized, starting from 2,5-dilithioselenophene, as potential electron donors or acceptors <91TL4313>.



The classical nucleophilic ring opening of condensed selenophenes has been generalized to more complex compounds. The C—Se bond is preferentially cleaved by MeLi in heterocycles (13)–(16) <83BSB53, 83CS177>.

However, heterocycle (16) undergoes ring opening with S—N bond cleavage <87BSB407>. These reactions afford diversely substituted selenophenes that can be further transformed.

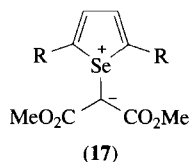


2.13.5.5 Radicals and Electrochemical Reactions

One electron electrochemical reduction of 2- NO_2 chalcogenophenes becomes progressively easier when going from oxygen to selenium <83MI 213-07>. Selenophene-3-carboxylic acid is electrochemically reducible to its 2,3-dihydroderivative but the corresponding furan is not reduced under the same conditions <83IZV944>.

Oxidation of benzo[*b*] and dibenzoselenophene in nonaqueous media leads to the formation of the corresponding oxide <92MI 213-02>, whereas 1,2-dibenzodiselenin first loses a selenium atom forming dibenzoselenophene before undergoing oxidation <92MI 213-03>.

Like thiophene, selenophene derivatives react with dimethyl diazomalonate in the presence of rhodium acetate to give ylides (17) which are less stable than their sulfur-containing congeners <84CS253>.



Due to potential industrial applications, important studies (see Section 2.13.9) have been made on polyselenophenes and similar compounds which can be prepared by electrochemical polymerisation <83MI 213-08>. Electrochemical study (cyclic voltametry and chronoamperometry–coulometry) shows that poly(3-methylselenophene) is a very good electrode material <88SM77>.

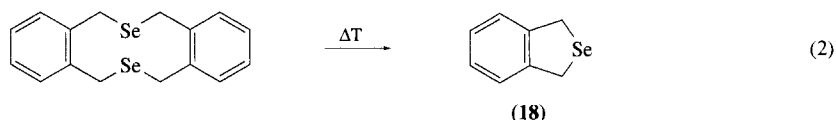
2.13.5.6 Cycloaddition Reactions

The mechanism of the photochemical addition of benzophenone to selenophene derivatives has been studied and in some cases an oxetane is isolable <82MI 213-01, 83MI 213-06>. The [2 + 2] photoaddition of 2,3-dimethylmaleic anhydride to benzo[*b*]selenophene and heteroaryl-condensed selenophenes furnished cycloadducts whose structure was established. Selenophene does not react under these conditions <83JHC1465>. 1,2,3-Triphenylnaphthalene is obtained when reacting selenophene with E-stilbene <91ZOR1364>, probably through a cycloaddition pathway. Inverse electron demand [4 + 2] cycloadditions of selenophene across 1,2,4,5-tetrazines gave 5,6-diazabenzob[*b*]selenophene derivatives <89CZ17>.

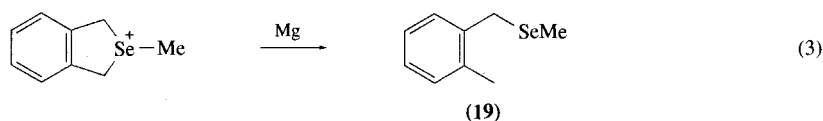
2.13.6 REACTIVITY OF NONCONJUGATED RINGS

Investigation of the kinetics and mechanism of ligand substitution in the formation of complexes $\eta^5\text{-C}_6\text{H}_5\text{Nb(CO)}_3\text{C}_4\text{H}_8\text{X}$ shows that the stability of these complexes increases from X = O to Te <91OM256>.

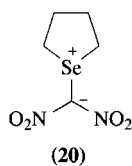
The comparative thermolysis of several benzylselenides has shown that the 1,3-dihydrobenzo[*c*]selenophene (**18**) is more stable than expected for a dibenzylselenide since it is produced by the reaction shown in Equation (2) <82BCJ182>.



Reduction of 1,3-dihydrobenzo[*c*]selenophenium salts with magnesium leads to ring opening and the formation of selenide (**19**) (Equation (3)) <89TL981>.



Vinylselenonium salts derived from selenolane were obtained by hydrobromic acid elimination from β -bromoalkylselenonium salts <88ZOR355>. Generation of dinitrocarbene is possible through electrochemical oxidation of the ylide (**20**) <89IZV356>.



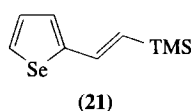
Classical reactions of 2,3-dihydrobenzo[*b*]selenophene-3-one (selenoindoxyl) with various amines followed by cyclization have been used to give several condensed polycyclic compounds <85IZV861>. The oxidation of selenoindoxyl gives its rather unstable selenium oxide <89LA811>. The attempted photochemical rearrangement of 2-diazoselenoindoxyl afforded the 2-methoxy derivative instead of the expected benzoselenene <89JOC240>. Charge transfer complexes of 2,5-dihydroselenophene have been described <87JOM(330)337>.

Oxidation of selenolane by chloramine T in the presence of amides or sulfonamides afforded the corresponding selenenimides <81PS(10)153>. The Se—Br bond of Se-dibromoselenolane adds across double bonds in a regio- and stereospecific manner <84ZOR2480, 87ZOR949>.

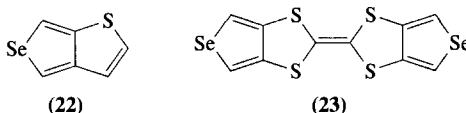
2.13.7 REACTIVITY OF SUBSTITUENTS

The (*E*) alkene (**21**) was obtained from 2-iodoselenophene as an example of a general coupling reaction between iodoarenes and vinylsilanes <86JOC5286>.

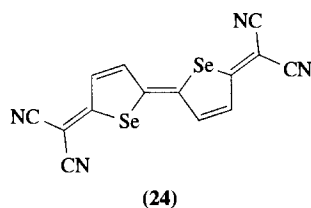
Bromine–lithium interconversion reactions of 3,4-dibromoselenophene were the starting steps for



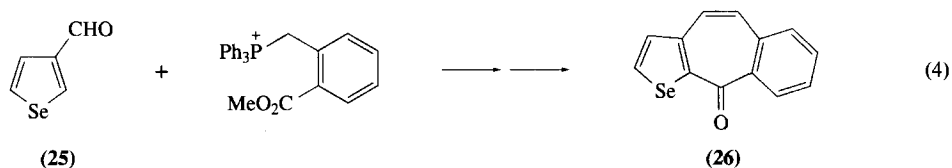
the synthesis of nonclassical selenolothiophenes (**22**) <82CS176> as well as selenolotetrathiafulvalene (**23**) <84JOC1117>.



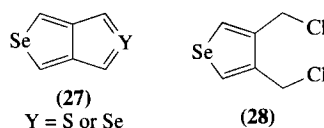
The selenium analog (**24**) of TCNQ was synthesized from 2,5-dibromoselenophene <88CL1179>.



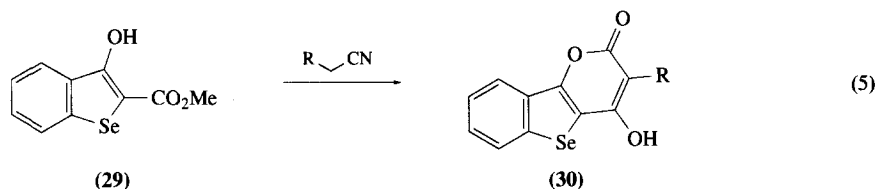
Selenophene-3-carbaldehyde (**25**) has been transformed by classical ways into the tricyclic compound (**26**) (Equation (4)) <83T819>.



The formation of nonclassical selenothiophene and selenophenes (**27**) has been achieved by traditional transformation of bis-3,4-chloromethylselenophene (**28**) <84CS53>. The Schollkopf methodology and rhodium catalyzed reduction allowed the enantioselective synthesis of 2- and 3-selenienylalanines <87CS249>.



Application of a general synthesis of 4-hydroxycoumarins allowed the synthesis of tricyclic compound (**30**) from ester (**29**) (Equation (5)) <86UKZ1082>.

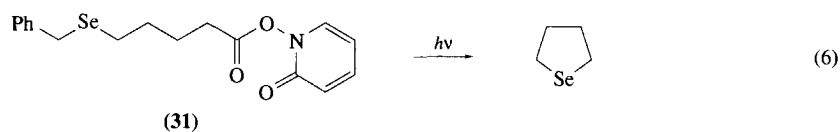


2.13.8 RING SYNTHESIS

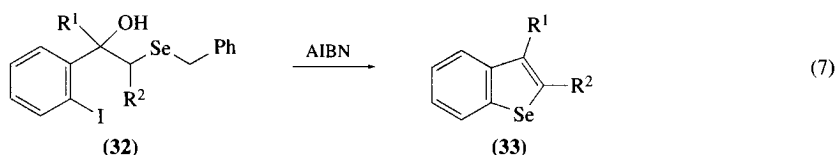
2.13.8.1 Formation of One Bond

2.13.8.1.1 C_α—Se

When applied to benzylselenides of the type shown in Structure (**31**), the Barton radical based methodology afforded selenolane (Equation (6)) <92CC57>.



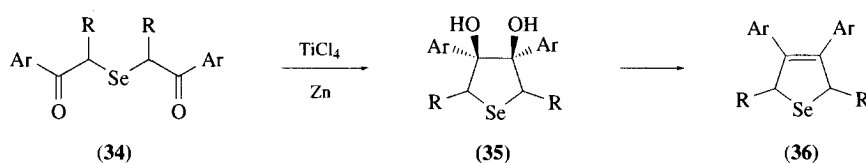
The easy generation of radicals from benzylselenides also leads to benzo[*b*] derivatives (33) from compounds (32) contained in (Equation (7)) <92TL5137>.



2.13.8.1.2 C_β—C_γ

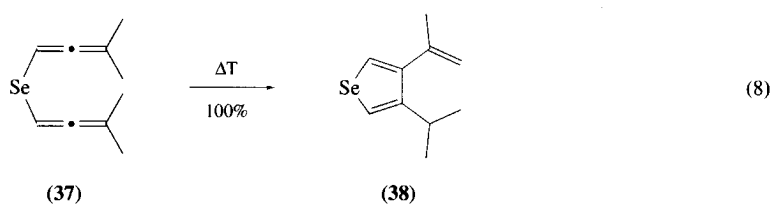
When thermolysed at 400°C, divinylselenide forms selenophene with 50% yield <82ZOR223>.

The low valence titanium coupling reaction allows the formation of 3-selenolenes (36) from β-carbonylselenides (34) with yields up to 60% (Scheme 3).

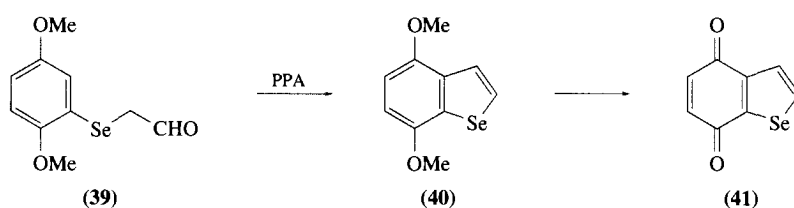


Scheme 3

The *cis*-3,4-diol (35) is isolable <87CC1072> and can be aromatized <88TL1399>. The cycloaromatization by thermal rearrangement of allenic selenides (37) leads to unusual selenophene derivatives (38) (Equation (8)) <90JA5830>. A pericyclic mechanism is proposed.



The electrophilic cyclization of selenide (39) led to the benzo[*b*]selenophene derivative (40) which has been transformed into the corresponding quinone (41) (Scheme 4) <92H(34)1749>.

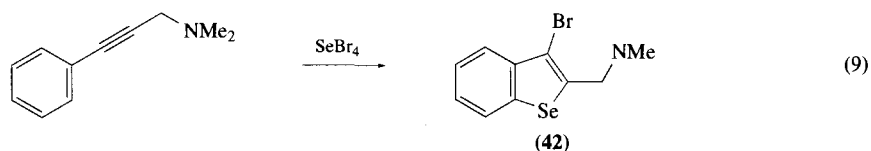


Scheme 4

2.13.8.2 Formation of Two Bonds

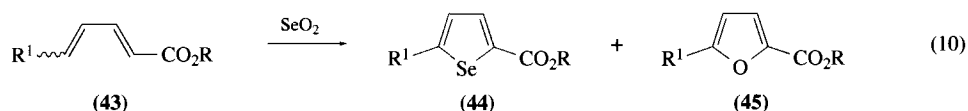
2.13.8.2.1 Selenium plus C_4 unit

This is the easiest and most studied route because it uses inorganic derivatives of selenium. New examples of the ring closure of arylalkynes by means of SeX_4 have been published <81KGS1283>. By this method 2-aminomethylbenzo[*b*]selenophenes (**42**) are obtained (Equation (9)) <90KGS1331>.

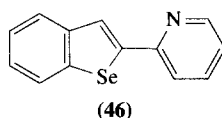


In a similar way, $SeBr_4$ adds across allylbenzene in an anti-Markovnikoff manner and gives 2-bromomethyl-2,3-dihydrobenzo[*b*]selenophene <82KGS911>.

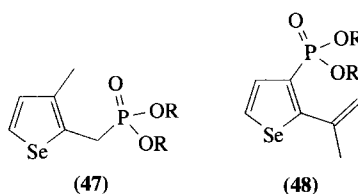
A new synthesis of selenophene derivatives (**44**) has been developed by Japanese chemists (Equation (10)). It consists of the oxidation by SeO_2 of conjugated alkenes (**43**).



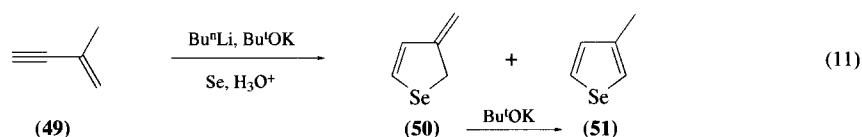
The furan analogue (**45**) is formed at the same time with higher yields <86TL2643, 87BCJ1807>. Similar oxidation of isoprenoid dienes affords mixtures of substituted selenolanes as well as six- and seven-membered selenium-containing heterocycles <86DOK(289)114, 86IZV2514>. The SeO_2 oxidation of stilbazole with the formation of 2-(2-pyridyl)benzo[*b*]selenophene (**46**) is claimed to be an analytical procedure for the quantization of SeO_2 , even in water <89MI 213-02>.



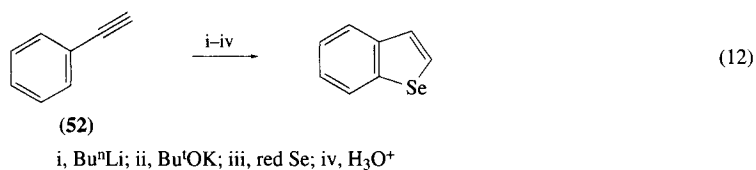
Phosphorylated allenes react with methylselenenyl chloride to give, after cyclization, phosphonic acid derivatives (**47**) <83PS(15)373, 88PS(35)35> or (**48**) <85H(23)43> with 60–70% yields.



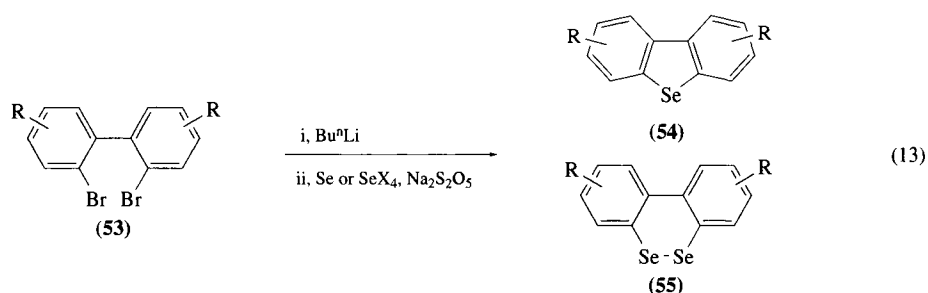
Numerous examples of an organometallic based methodology for the formation of selenophenes have been discussed. In these cases, a selenolate anion is formed *in situ* followed by cyclization. The application to selenophenes is due to the Brandsma group <83TL2203>. Indeed, when hydrocarbon (**49**) is first metallated and then treated with selenium, a mixture of (**50**) (46%) and (**51**) (42%) is formed after hydrolysis (Equation (11)). The base catalyzed isomerization of (**50**) to (**51**) is quantitative.



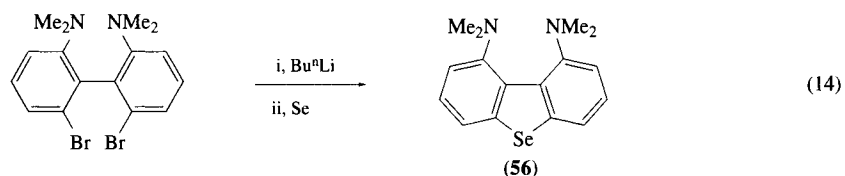
In a quite similar reaction, benzo[*b*]selenophene is available from phenylethyne (**52**) via a double metallation reaction (Equation (12)) <85RTC226>.



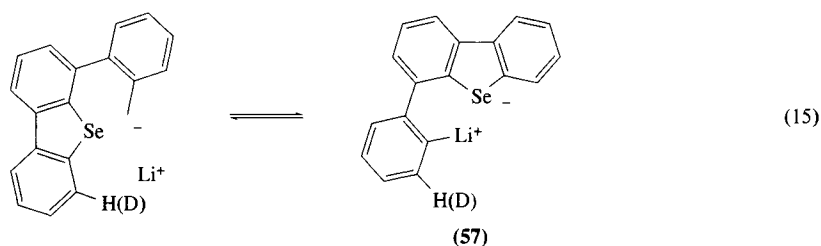
The dianionic intermediate is quenchable with several electrophiles. Dibenzoselenophene derivatives (**54**) are also available from suitably substituted 2,2'-dibromobiphenyls (**53**) (Equation (13)). In that case, Engman has shown that the chalcogen may be introduced in its elemental form as well as its halide (SeX₄) <84JHC413>. The corresponding diselenide (**55**) is isolable and can be transformed into dibenzoselenophene (**54**).



The same reaction has been extended to 2,2'-dibromo-1,1'-binaphthyl <91JHC433> which affords dinaphthoselenophene (Equation (14)). The dibenzoselenophene analogue (**56**) of proton sponge has been obtained (7% yield) following the same methodology (Equation (14)) <88TL1905>. Its pK_a has a value of 11.8 ± 0.1 close to that of proton sponge itself (pK_a: 12.1).



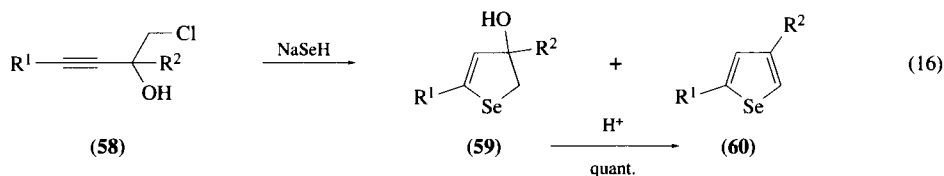
The first organoselenoate complex (**57**) has been obtained by a Br-Li exchange and fully characterized (Equation (15)) <92JA7937>.



The transmetallation of zirconium five-membered heterocycles by elemental selenium also affords selenophene derivatives with yields up to 50% <88JA2310>. Classical thermal displacement (210°C) of SO₂ by selenium allowed the synthesis of 3,4-di-*t*-butylselenophene from the corresponding thiophene dioxide <90TL4473>.

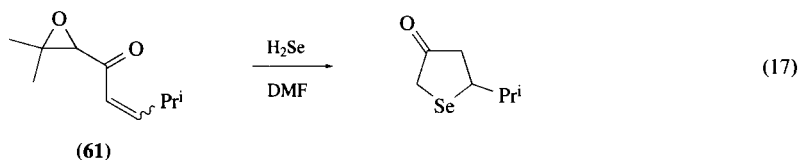
Selenophene derivatives are also available from selenide anion. Phase transfer catalysis improved the yield of selenolane from Na₂Se and 1,4-dibromobutane <89SR1931>. The reaction of sodium

selenolate with alkyne (**58**) affords a mixture of two compounds (**60**) and (**59**) which can be transformed easily into (**60**) by acidic treatment (Equation (16)) <87PS(34)119>.

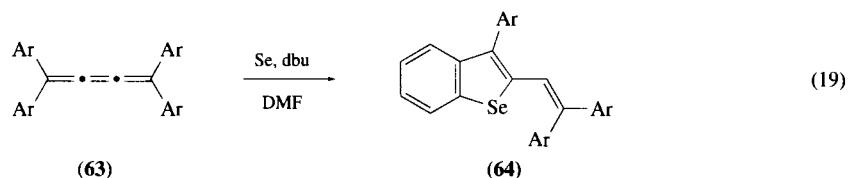
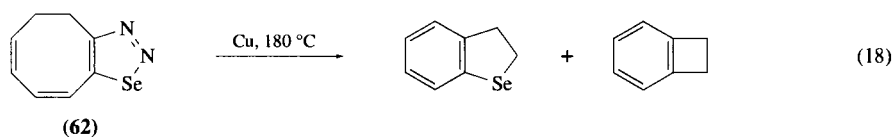


Using the Diels adduct between anthracene and maleic anhydride, it is possible to obtain a complex selenolane. Its flash thermolysis affords the previously unknown 3-selenolene, which has been fully characterized <86JCR(S)98>.

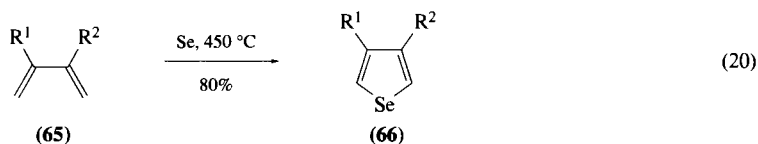
Lithium selenide reacts with *o*-chloroacetophenone in aprotic medium to give unexpectedly a significant amount (25%) of selenoindoxyl <88TL6119>. Reaction of H_2Se with ketoepoxide (**61**) gives 12% of the selenolane derivative besides a tetrahydroselenopyran derivative (Equation (17)) <91ZOR942>.



The thermolysis of the selenadiazole derivative (**62**) follows an unexpected course and affords 15% yield of 2,3-dihydrobenzo[*b*]selenophene (Equation (18)) <85CB198>. Bis(allylenic) compounds (**63**) even react with selenium to give benzo[*b*]selenophenes (**64**) (Equation (19)) <88TL1935>.



Bis(alkynes) react similarly <90MI 213-08, 92URP1721053>. Less reactive 1,3-butadienes (**65**) require more drastic conditions to afford selenophenes (**66**) (Equation (20)) <89S181>.

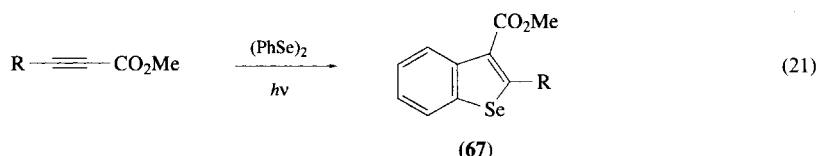


The selenation of cinnamaldehyde with a substituted borylselenide leads to a 2-selenolene derivative <92CL1843>.

2.13.8.2.2 $\text{Se}-\text{C}_\alpha-\text{C}_\beta$ plus $\text{C}_\gamma-\text{C}_\delta$

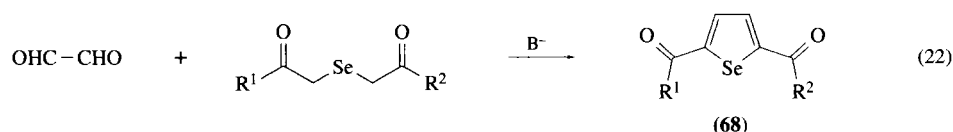
Several products were formed by addition of phenylselenolate anion to DMAD <82ZOR281>. Among them, modest yields of 2,3-di(methoxycarbonyl)-4-phenylselenophene and 3-methoxycarbonyl-2,4-diphenylselenophene are obtained. The study of the photochemical addition of

diphenyldiselenide to alkynes (Equation (21)) has shown the formation of benzo[*b*]selenophene derivatives (**67**) among other photoproducts <88JOC2533>.



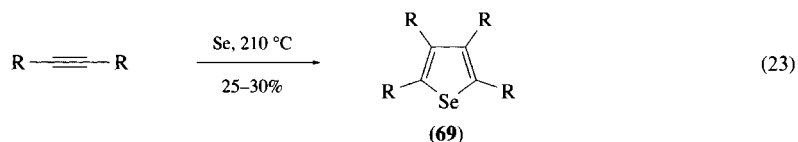
2.13.8.2.3 $\text{C}_\alpha\text{---Se---C}_\delta$ plus $\text{C}_\beta\text{---C}_\gamma$

The classical Hinsberg synthesis of thiophenes has been applied to the preparation of 2,5-dicarbonylselenophenes (**68**) <87H(26)909> as shown in Equation (22).

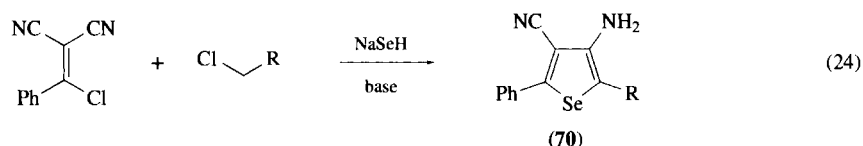


2.13.8.3 Formation of Three Bonds

Russian chemists have developed the synthesis of selenophenes from alkynes and (or) alkenes and elemental selenium. A 25% yield of selenophene is obtained when selenium and KOH are treated with a mixture of ethyne and ethene at 120°C in DMSO <82T713>. In the transformation HMPT may also be used as a cosolvent <84ZOR484>. An improved yield of 58% of selenophene (Se, Al₂O₃, ethyne) has been reported <83JOC3544> and the method has been patented <83JAP(K)59227881>. All the parameters of the reaction <86ZOR276> have been optimized. It has also been claimed that using dimethylselenide instead of elemental selenium gives higher yields <85URP1231055>, even up to 96% <87KGS279>. With diphenyldiselenide, benzo[*b*]selenophene derivatives are also formed <89MI 213-06, 90MI 213-07>. In the same way, when heated neat at 210°C with elemental selenium substituted, alkynes afford substituted selenophenes (**69**) <87H(26)2215, 87NKK1424, 92H(34)1487>.



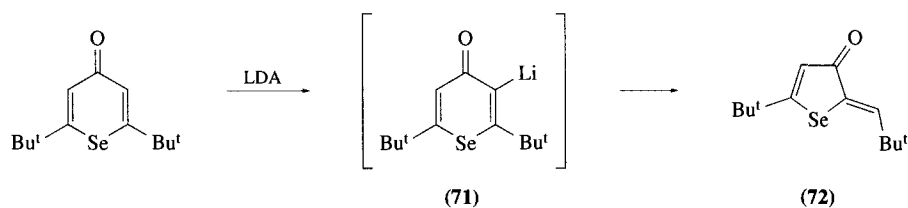
The general synthesis of chalcogenophene derivatives has been extended to aminoselenophenes (**70**) (Equation (24)) <92M455>.



2.13.8.4 Ring Transformations

The ring opening reaction of 3-lithioselenopyran-4-one (Structure (**71**)) gives a 10% yield of the 2,3-dihydroselenophene (**72**) (Scheme 5) <88JOC1203>.

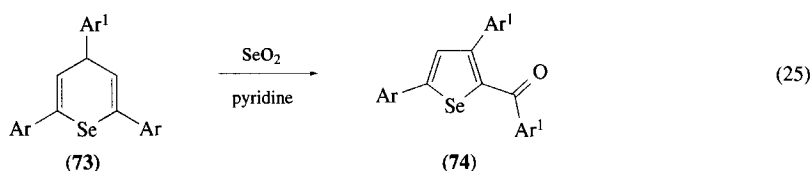
A general transformation of furan into selenophene is possible by action of hydrogen selenide on the oxygen heterocycle <82KGS418, 83KGS219> in the presence of strong acid (HClO₄). The mechanism has been elucidated <89KGS1458>. Yields of up to 80% are attainable with 2,5-disubstituted seleno-



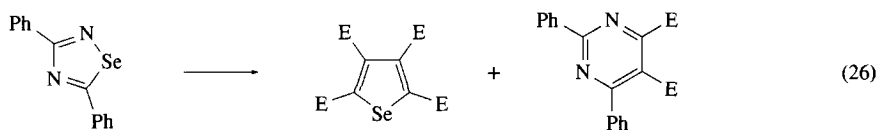
Scheme 5

phene <84KGS1606>. Even with 2,3-dihydrofuran, 2-selenolene is obtained in 20% yield by the action of H_2Se at 300°C on Zeocarb 2 <86KGS1478>.

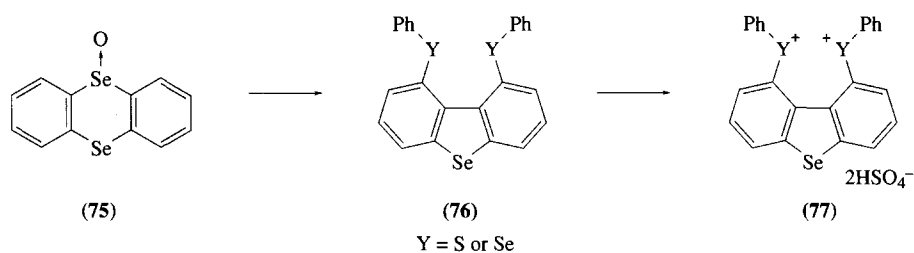
Ring contraction is observed as a result of classical oxidation of selenopyran derivatives (73) with formation of selenophenes (74) (Equation (25)) <89KGS767>.



Some selenazoles may be transformed into selenophenes by dipolar additions, for example, Equation (26) <91CL2043>.



The preparation of sterically highly congested 1,9-disubstituted dibenzoselenophenes (76) has been described from selenanthrene Se-oxide (75). When treated in concentrated H_2SO_4 , these compounds afforded new dications (77) (Scheme 6) <92CL675>.



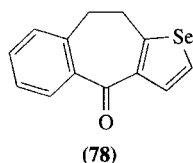
Scheme 6

2.13.9 IMPORTANT COMPOUNDS AND APPLICATIONS

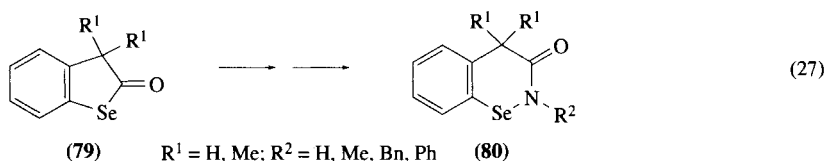
2.13.9.1 Compounds of Biological Interest

A large variety of compounds have been prepared in which a selenium atom replaces an oxygen, a sulfur, or a nitrogen one. These compounds were synthesized in order to try to improve the existing bioactivity. Among them selenolevamisole analogues are described <83JHC523>. The palladium(0) catalyzed coupling of selenienylboronic acids has been realized with halopyrimidines <86CS305>, halouracils <90JHC2165>, halocytosines <91JHC1613>, halo-2-deoxyuridine, and cytidine <92MI 213-04>. The antiviral activity of all these molecules has been evaluated.

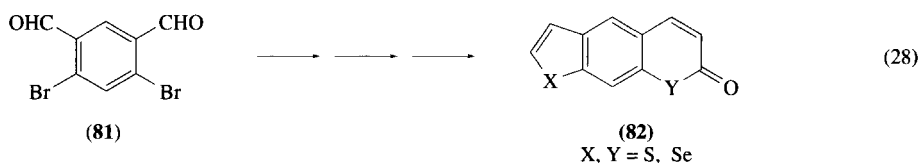
The antiviral character of 2-selenienyl nucleosides has been patented <90MIP8912061> as well as the antiallergic activity of ketone **(78)** <90GEP(O)4037187>.



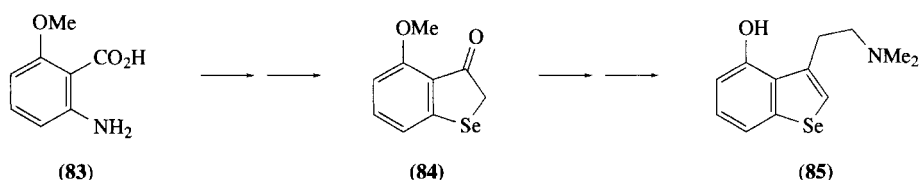
Homo-Ebselens **(80)** were synthesized from selenolactones **(79)** in a multistep pathway (Equation (27)). Unfortunately these molecules are highly photosensitive and their antiinflammatory activity is too low compared with Ebselen <92TL3863>.



Starting from dibromoisophthalaldehyde **(81)**, a new multistep methodology (Equation (28)) has been designed for the synthesis of all the sulfur and selenium analogues of psoralene **(82)** <92H(34)1119>. These compounds seem to be promising with regard to their bioactivity <94MI 213-01>.

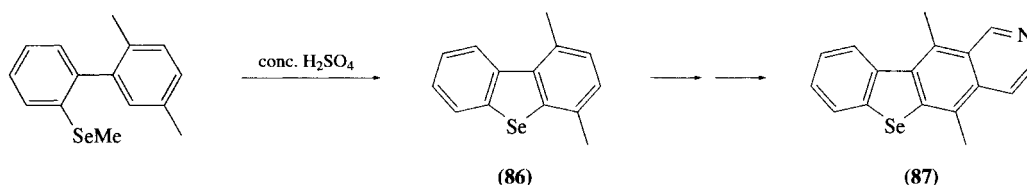


The synthesis of the selenium analogue **(85)** of the hallucinogen psilocin is described from 6-methoxyanthranilic acid **(83)**. The key intermediate is 4-methoxyselenoindoxyl **(84)** (Scheme 7). The bioactivity is not mentioned <92H(34)1737>.



Scheme 7

A new synthesis of dibenzoselenophene **(86)** is the key step for the formation of selenoellipticine **(87)** (Scheme 8) <93ACS(B)208>.



Scheme 8

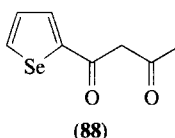
The auxin-like activity of benzo[*b*]selenienyl-3-acetic acid has been confirmed <83P2657, 85MI 213-06>. The phototoxicity of α -terthienyl is claimed to be higher against yeast alcohol dehydrogenase than α -terselenienyl <84MI 213-03>.

2.13.9.2 Compounds with Potential Technical Utility

Since the mid-1980s, more than 60 patents concerning selenophene derivatives have been filed. They generally deal with polyselenophenes that can be used in two important directions: electroconducting films and photosensitive materials. In such a matter, it is not possible to be exhaustive and we will summarise the more significant features. The polymerization of selenophene may be effected chemically <84SM389, 85MI 213-07, 87JAP(K)62106923>, or electrochemically <83JAP(K)58187432, 84JAP(K)59191727, 85JAP(K)6050194, 86SM281, 87MI 213-04>.

New long chain alkyl selenophenes were synthesized <90PS(47)35> and the effects of the position <85MCLC235, 87MI 213-05>, and of the length of the alkyl chain <89MI 213-05> have been established. An optimization of the electrosynthesis of polyselenophene has been claimed <90MCLC459>. Mixed thienyl-selenienyl polymers were prepared and their properties investigated by cyclic voltametry and chronocoulometry <93SM115>. Poly-2-vinylselenophenes are claimed to have a higher conductivity than their sulfur analogues <92CL1551>, whereas iodine doped 2,5-polyselenophenes show an unexpectedly low conductivity <84MI 213-01>. Some interesting applications seem to be possible with such polymeric materials. They are useful in: electric devices <84JAP(K)5943060>, dry batteries <88JAP(K)63224151>, microporous membranes with selective gas permeability <89JAP(K)0138125, 89JAP(K)0111139>, printed circuits <90EUP413109>, and antistatic coatings <90MIP463516>. It is claimed that 2-azoderivatives of selenophenes are photosensitive <84JAP(K)59223433, 91JAP(K)03141362>, as well as selenophene ketohydrazones <89JAP(K)0165557>. The incorporation of selenophene derivatives in low percentages improves the storage properties of photographic materials <88JAP(K)63175853>.

Charge transfer complexes of dibenzoselenophene and naphthoquinones have colouring properties <88DP343>. A new catalyst containing the selenophene moiety (**88**) has been devised for the formation of polyurethanes <87MI 213-03>.



2.14

Tellurophenes

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University of Liège, Belgium

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2.14.1 INTRODUCTION

The scope of tellurophenes was well defined at the beginning of Chapter 3.16 in the first edition of *Comprehensive Heterocyclic Chemistry* (CHEC-I). Since that publication, several general reviews have been published on this subject <B-86MI 214-01, B-90MI 214-01, 91S793, 91S897>. More specific works have also appeared <B-86MI 214-02, B-86MI 214-03, B-87MI 214-01, 90PHC(2)50, 91PHC(3)70>. In this chapter, the literature will be covered through to 1993 with the same approach for the form and the material as in CHEC-I.

2.14.2 THEORETICAL METHODS

A new index (I) of aromatic character has been devised <85T1409> based upon a statistical evaluation of the deviations in peripheral bond orders (derived readily from experimental bond lengths). The I_5 index for the chalcogenophene series is O: 43, S: 66, Se: 59, and Te: 48 showing a reasonable parallelism with the resonance energies and a good linear relationship with A , the aromaticity parameter observed with the dilution shift method. More recently, this index has been improved <92T335> and an excellent linear relationship is found between I_A and A . For chalcogenophenes, the new index I_A is O: 53, Te: 59, Se: 73, and S: 81.5 (compared with benzene: 100). Principal component analyses and partial least squares analyses have been used to study ^{13}C and ^1H 2-substituent effects in the chalcogenophene series <90MRC397>. A valence force field for in-plane vibration of seleno- and tellurophenes has been derived from experimental frequencies

<85SA(A)679>. It shows, as previously established, a noticeable variation of aromaticity in that series. The characterization by *ab initio* molecular orbital theory of chalcogen isosteres of benzene <88JA4204> allowed the prediction of the structures with a precision of 0.04 Å for the bonds and 2° for the angles. Only the chalcogenophene series shows delocalization.

The transmission mechanisms of chalcogen-carbon coupling constants in selenophene and tellurophene according to the PRMO-SCPT-INDO approach indicate a *J* transmission through the σ network and a lesser orbital dipole interaction than in benzene <83MI 214-01>. *Ab initio* calculations using a 3-21 G basis set indicate that the system should include a *d* polarization function on tellurium <89MI 214-01>. Force field calculation procedures have been developed with the aim of getting information on the influence of substituent effects on the aromatic ring force field in 2-bromo and 2-chloro derivatives of seleno- and tellurophenes <87CPL244, 88MI 214-01>. Physicochemically based semiempirical MO calculations have shown a fair correlation between the average $\nu_{C=O}$, the average ionization energy of the carbonyl lone pairs orbitals, and the $\delta^{13}C=O$ for seleno- and tellurophthalic anhydrides. Their dipole moments in benzene at 25°C are, respectively, 3.30 D (Se) and 2.71 D (Te) <82SA(A)185>.

2.14.3 EXPERIMENTAL STRUCTURAL METHODS

2.14.3.1 Molecular Structure

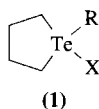
The crystal structure of the 1:1 complex of dibenzotellurophene and tetracyanoquinodimethane shows a quasihexagonal packed mixed stack of alternating donors and acceptors whose structures are nearly identical to the uncomplexed molecules <84JCS(D)1267>. A rather close contact Te-N interaction is observed. This poor electron transfer is confirmed by the Mossbauer effect and IR techniques. Some 1,3-dihydrobenzo[*c*]tellurophenes behave similarly <90POL847>. Disc conductivity measurements show a semiconducting behaviour related to the temperature <91IJC(A)720> and allow a classification of the donor ability of some tellurophene related heterocycles <87JOM(330)337>. The x-ray structure of 3-iodo-2,5-diphenyltellurophene has been reported <92AX(C)767>.

Several tellurium disubstituted 1,3-dihydrobenzo[*c*]tellurophenes including the β form of 1,1-diiodo <85IC1814>, 1-allyl-1-bromo, 1-phenacyl-1-bromo, 1-deuteriomethyl-1-iodo <82JOM(234)299>, 1-methyl-1-tetraphenyl boride <84JOM(269)11>, 1,1-bis(diethyl)dithiocarbamoyl <88JA6753>, 1,1-dihalo <88JOM(349)95>, and 1,1-diacetate <92PS(71)13> have similar structures. The octahedral tellurium atom bearing a lone pair lies on a twofold axis, and the two substituents occupy nearly axial positions with respect to the carbons. The substituents of neighboring molecules are nearly equatorial and complete the geometry. Conductivity measurements lead to the conclusion that this type of compound exists as dimeric ion pairs in low polarity solvents (CHCl₃) <82JOM(234)287, 84JOM(277)365, 85JOM(287)321, 88JOM(356)49, 92MI 214-01> but displays rather more ionic behavior in high polar solvents (DMSO) <91HAC417>.

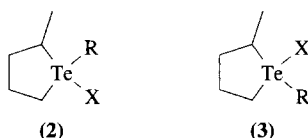
2.14.3.2 NMR Spectroscopy

Proton, ^{13}C , and ^{125}Te NMR measurements (δ and *J*) in several nematic phases afforded a solvent-independent structure of tellurophene <81MP877, 86MP959, 87MRC244>. Tellurophene-2-carbaldehyde shows a Te—O *syn* conformation as indicated by NMR data in semiliquid crystalline phase <88G529>. From $J_{^{13}C-^{13}C}$, it has been deduced that the σ electronic effect is the most important for ^1J in benzo[*b*]tellurophenes <89ZOR2268>. A quantitative determination of π electron excess or deficiency, obtained from $\delta^{13}C$ in tellurophene series, shows a good correlation with electrophilic substitution <82OMR(19)192>. Different coupling pathways (J_{C-H} , J_{H-H} , J_{C-C}) were studied for the same compounds by application of the IPPP method <87MI 214-02>. Excellent ^1H and ^{13}C correlations were made between substituted benzo[*b*]selenophenes and tellurophenes. Transmission of substituent effects by the chalcogen is similar in those series which exhibit a heavy atom effect <81CS147>. The effect of the lone pair of the hetero atom in 2-substituted tellurophenes has been discussed in relation to the $J_{^{13}C-^{125}Te}$ <82JMR(47)504>. Due to their easy synthetic access, numerous derivatives of 2,5-dihydro-2,3,4,5-tetrahydrotellurophene and 1,3-dihydrobenzo[*c*]tellurophene have been obtained and their structures confirmed by routine NMR data. From ^{125}Te data the Te—R bond in (1) is

shown to be covalent and the $\delta^{125}\text{Te}$ are rather insensitive to X but strongly affected by the solvent $\langle 85\text{JOM}(287)321 \rangle$.



Compounds (2) and (3) are obtained synthetically as a mixture where the 'trans' form (3) is predominant but depends upon the size of the alkyl R $\langle 85\text{JOM}(294)315 \rangle$. Complexes with palladium or platinum behave similarly $\langle 92\text{POL}2165 \rangle$.



Most of these compounds are subject to ligand exchange in solution. This has been demonstrated by ^{125}Te NMR measurements (a $J_{^{125}\text{Te}-\text{F}} = 1030 \text{ Hz}$ is mentioned) $\langle 82\text{JOM}(234)287, 88\text{JA}6753, 88\text{JOM}(353)35, 88\text{JOM}(349)95 \rangle$.

2.14.3.3 Ultraviolet, Infrared, and Raman Spectroscopies

The structures of the aforementioned telluranes or telluronium salts have also been proved by routine IR techniques $\langle 82\text{JOM}(234)287, 84\text{JOM}(277)365, 85\text{POL}1223 \rangle$. The complete attribution of the fundamental vibrations in benzo[*b*]tellurophene have been proposed based on IR and Raman measurements and the evolution with change of chalcogen analysed $\langle 83\text{SA}(A)1033 \rangle$. The UV spectra of tellurophene and its congeners were investigated with the aim of exploring the Rydberg transitions terminating at the $3s$ orbital $\langle 88\text{CS}331 \rangle$. Due to the high quality of the spectra, FT-Raman is claimed as the preferred technique for routine vibrational spectroscopy $\langle 92\text{JOM}(430)37 \rangle$.

2.14.3.4 Mass Spectrometry

Routine mass spectrometry has been used for the confirmation of structures, but no specialist report has been published. Several 1,3-benzo[*c*]tellurophenes were identified through their fragmentations under electron impact $\langle 84\text{JOM}(277)365 \rangle$.

2.14.3.5 Thermodynamic Considerations

A ground state aromaticity ranking has been deduced from molecular vibrational constants and chemical bonding for the chalcogenophene series $\langle 85\text{MI} 214-01 \rangle$. It follows the order $\text{S} > \text{Te} > \text{Se} > \text{O}$ whereas the electrophilic reactivity follows $\text{O} > \text{Se} > \text{Te} > \text{S}$. The relative positions of tellurium and selenium are at variance with the new index of aromatic character described in Section 2.14.2. Thermal decomposition of mixtures of (2) and (3) first affords $\text{R}-\text{X}$. The heterocycle loses the tellurium atom at higher temperatures and gives substituted cyclobutanes $\langle 85\text{MI} 214-02 \rangle$. The potential of 2,5-dihydrotellurophene as a tellurium source for metal organic chemical vapor deposition (MOCVD) growth of CdTe at 250°C has been assessed $\langle 88\text{MI} 214-02 \rangle$. The compound was found to be thermally more stable than anticipated. Heat capacities, entropies, and thermodynamic parameters for phase transitions of some organotellurium compounds are included in a compilation $\langle 90\text{JPC}881 \rangle$.

2.14.3.6 Miscellaneous

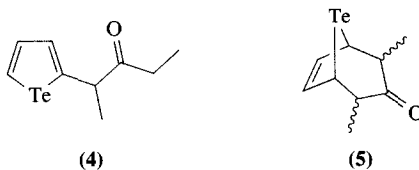
An ESR study of radical ions in the 2,5-diphenylchalcogenophene series has been published $\langle 90\text{JOM}(389)301 \rangle$. These spectra belong to systems conformationally rigid on the ESR timescale as

indicated by the nonequivalence of the *ortho*- and *meta*-proton hyperfine coupling constants. For the O, S, and Se congeners, the spectra show the expected symmetry of the parent LUMO and the antisymmetry for the HOMO. For tellurophene, a coupling to ^{125}Te is observed for the radical anion, while the spectrum of the radical cation suggests a change in the nature of the HOMO. The phosphorescence spectra of the benzo[*b*]- and dibenzochalcogenophenes series were analysed at 77 K in ethanol and *n*-pentane (89ZN(A)205). The rate constants of both radiative (K_{PT}) and nonradiative (K_{GT}) deactivation of the lowest triplet state correlate linearly with $\sum kC_k^2\zeta^2$ (where C_k denotes the Hückel AO coefficients in the HOMO of the carbon atoms bound to the heteroatom and ζ is the spin orbit coupling constant of the heteroatom). A linear correlation is observed between K_{PT} and K_{GT} as an example of the Orland-Siebrand rule. Photoelectron spectra of the dibenzochalcogenophene series (88ZOB1409) and MO calculations identified a_1 symmetry HOMO of these compounds. The first ionization potential decreased in the order $\text{O} > \text{S} > \text{Se} > \text{Te}$. The second HOMO has symmetry a_2 and the second ionization potential is fairly constant.

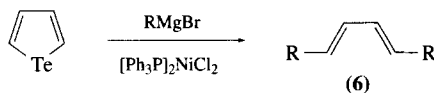
The electron transmission spectra of tellurophene and its analogs were recorded in the 0–6 eV energy range. Two resonances were identified for furan and thiophene and three were observed for selenium and tellurium analogues. The latter indicated a strong heteroatom 'd' character (84MI 214-01).

2.14.4 REACTIVITY OF FULLY CONJUGATED RINGS (REACTIVITY AT RING ATOMS)

In the presence of $\text{Fe}_2(\text{CO})_9$ and $\text{CH}_3\text{—CHBr—CO—CHBr—CH}_3$, tellurophene undergoes photochemical additions leading to 2-substituted derivatives (4), and a mixture of the stereoisomeric 2,3-dihydrobicyclic compounds (5) (85PS(25)245).

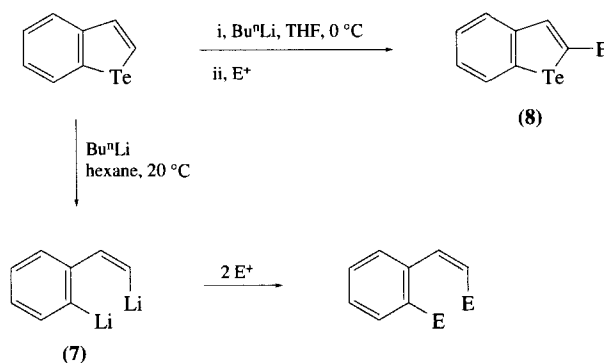


Tellurophene derivatives may be considered as organic intermediates. Indeed the treatment of tellurophene (84CC617) with Grignard reagents in the presence of nickel catalysts affords 88% of *Z,Z*-1,3-butadiene (6), as determined by high resolution ^1H and ^{13}C NMR spectroscopy. This yield decreases from Te to O in the series.

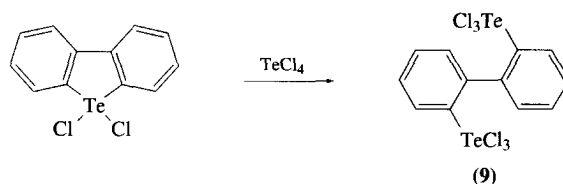


Similar behaviour is observed with benzo[*b*]tellurophene. When reacted at 20°C with *n*-butyllithium in hexane, the heterocycle loses tellurium and gives the *o*-lithiostyryllithium (7) which can be quenched by various electrophiles (92AG(E)1339). In THF at 0°C, the same reagent effects a metallation reaction which gives rise to 2-substituted benzo[*b*]tellurophenes (8).

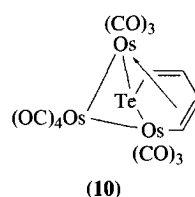
Tellurium tetrachloride brings about the electrophilic ring opening of Te—dichloro-



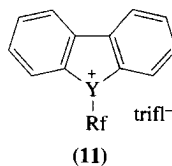
dibenzotellurophene. The intermediate bistelluronium compound (9) can be reduced to polymers and 8% of dibenzotellurophene <87KGS420>.



$\text{Os}_3(\text{CO})_{10}(\text{MeCN})_2$ forms clusters with heterocycles of the chalcogenophene series by C—H insertion; with tellurophene, C—Te insertion is also observed <90CC1568> leading to the formation of (10).



A series of dibenzochalcogenium salts of type (11) have been synthesized by cyclization of the corresponding chalcogenides with triflic anhydride and DMSO <93JA2156>.



They are powerful electrophilic perfluoroalkylating agents and react with a large variety of anions. The reactivity depends on the aromatic character of the dibenzochalcogenophene ($\text{S} > \text{Se} > \text{Te}$).

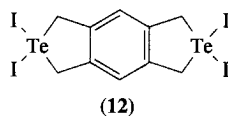
2.14.5 RING SYNTHESIS

The most widely used method for the synthesis of tellurophene derivatives is the introduction of tellurium into a substrate already bearing the four necessary carbon atoms. The introduction may be either nucleophilic or electrophilic.

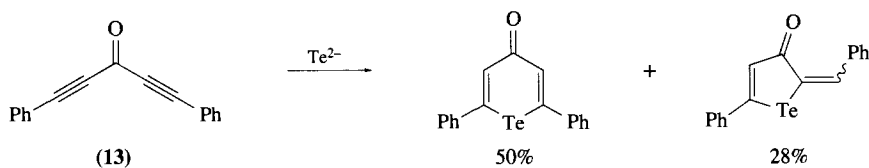
Due to their easy access from haloalkane derivatives, the chemistry of 2,3,4,5-tetrahydrotellurophenes and 1,3-dihydrobenzo[c]tellurophene is very well documented (see above). Improvements upon the nucleophilic character of the telluride anion were made either by the use of tin telluride <90TL6291> or by use of phase transfer catalysis conditions <89SRI931>.

It is worthy of note that the bistelluronium salt (12) is not reducible. The probable explanation may be related to insolubility <88JOM(338)1>.

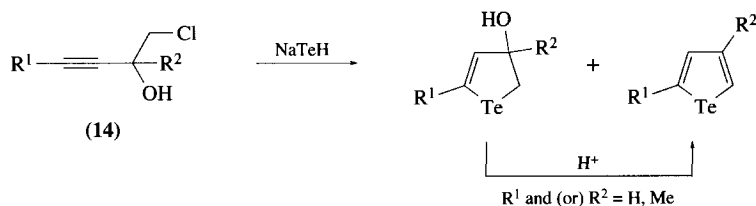
The telluride anion also adds to triple bonds to give tellurophene derivatives. With the



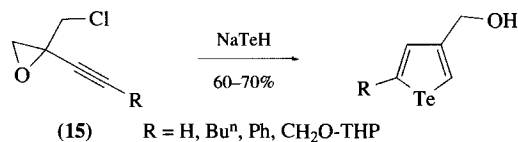
bis(phenylalkynyl) ketone (**13**), the yield of the two heterocycles depends highly upon the reaction conditions <82JOC1968>.



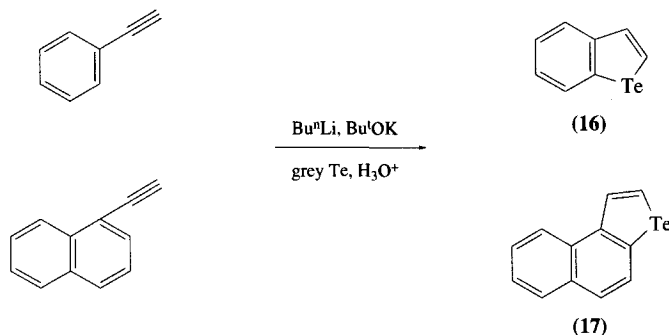
A fairly similar reaction is observed with the alkyne (**14**), a nucleophilic displacement of Cl^- with formation of a transient telluroate probably being the first step <87PS(34)119>.



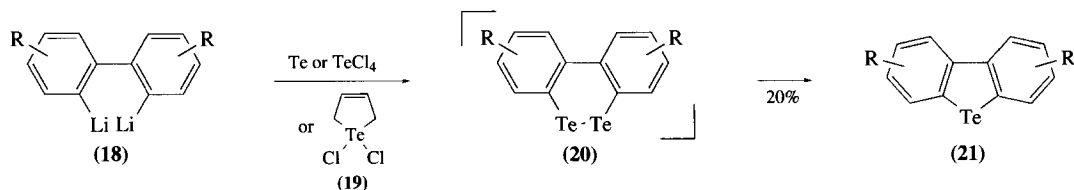
Ring opening of the oxirane (**15**) gives a similar result <88TL4923>.



The telluroate anion may also be generated in situ by reaction of elemental tellurium with carbanions. This reaction has been used for the synthesis of benzo- (**16**) <85RTC226> and naphtho[2,1-*b*]tellurophene (**17**) <89SC2691>. The dianionic intermediate is quenchable with several electrophiles.

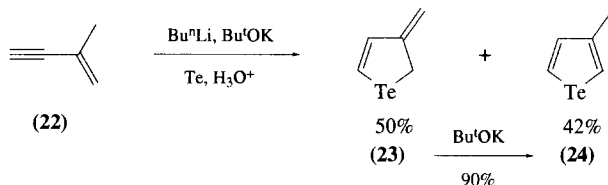


Engman describes a similar procedure for the synthesis of dibenzotellurophene but starting from 2,2'-dilithiobiphenyls (**18**). The cyclization is realized with elemental tellurium, tellurium tetrachloride, or the dichlorotelluroylene (**19**) which acts as a source of electrophilic tellurium.



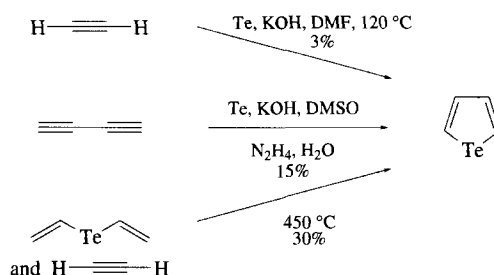
With tellurium, the postulated intermediate (**20**) is a 1,2-ditellurin which loses a tellurium atom to give dibenzotellurophene (**21**). In the selenium series, the corresponding diselenide is isolable <84JHC413>.

A similar pathway is observed with 2,2'-dilithiobinaphthyl <91JHC433>. The application in the acyclic series has been explored by Brandsma <83TL2203>. When compound (22) is subjected first to metallation and then reacted with tellurium, a mixture of (23) and (24) is obtained.



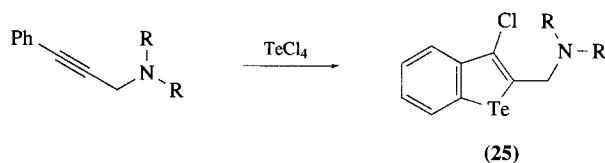
The *exo*-methylene derivative may be isomerized in 90% yield to (24). The reaction is sensitive to the nature of the cosolvent.

The formation of tellurophene derivatives through vigorous basic conditions is more complex but probably proceeds through tellurolate anions (Scheme 1) <89ZOR39, 90MI 214-02, 90MI 214-03>.



Scheme 1

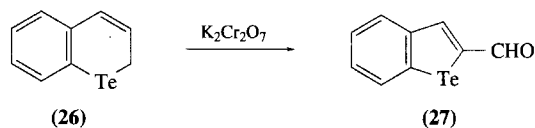
One synthesis of this type has been patented <92MIP1721053>. Dibenzotellurophene Te—dichloride is formed by an electrophilic route when biphenyl and tellurium tetrachloride are reacted neat <95SR1709>. The already known electrophilic cyclization of suitable alkynes is also realized by the same reagent giving amine (25) <90KGS1331>.



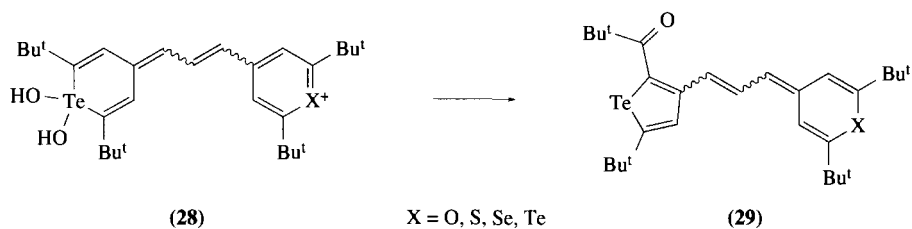
2.14.6 HETEROCYCLE SYNTHESIS BY RING TRANSFORMATION

Just like for the selenium series, (2*H*)benzo[*b*]tellurin (26) undergoes ring contraction by oxidation <88KGS1050> giving benzo[*b*]tellurophene-2-carbaldehyde (27).

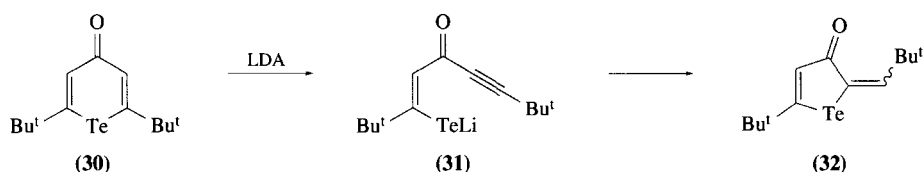
Similarly, dihydroxytellurane (28) can thermally lose hydrogen peroxide which acts as an



intermolecular oxidizing agent giving the tellurophene derivative (29). The reaction is strongly pH dependent <91OM702> and a four-step mechanism is proposed.



The metalation of telluropyrone (30) with LDA generates the 3-lithio derivative which equilibrates with the ring-opened anion (31). The latter furnishes traces of the tellurophene derivative (32) by Michael addition <88JOC1203>.



2.14.7 COMPOUNDS OF BIOLOGICAL INTEREST

The tellurium dichloro derivative of dibenzotellurophene is claimed to have antiviral activity <85SRI709>. Some derivatives of 1,3-dihydrobenzo[*c*]tellurophene have been shown to act as anti-microbials <83MI 214-02>.

2.14.8 COMPOUNDS WITH POTENTIAL TECHNICAL UTILITY

Two major applications have emerged between 1983 and 1993: the first is the use of tellurophene derivatives for the synthesis of electroconducting polymers, and the second is the thermolysis of organotellurium compounds for the production of thin films of cationic tellurides. Semiconductors derived from 3,4-disubstituted tellurophenes have been patented for photosensors <90JAP(K)0241317>. Electrochemical polymerization of benzo[*c*]tellurophene derivatives provides useful semiconductors <90JAP(K)02263823>, applicable among other uses, for solar cells <88EUP273643>.

Polymerization of tellurophene derivatives in the presence of a mild oxidizer (FeCl_3) affords conducting oligomers <84JAP(K)59191727, 84JAP(K)59202226, 85JAP(K)61276818>. Thermolysis of 2,5-dihydrotellurophene in the presence of metal dialkyls (e.g., dimethylcadmium, dimethylmercury) affords thin films of metal tellurides useful in optical recorders <87JAP(K)62104833, 87JAP(K)62109820, 87JAP(K)62196652, 87EUP240844, 87MIP8706275, 91JAP(K)04243879>.

Finally heterocyclic telluroxides have been patented as selective oxidants in organic syntheses <81BRP2058758>.

2.15

Phospholes

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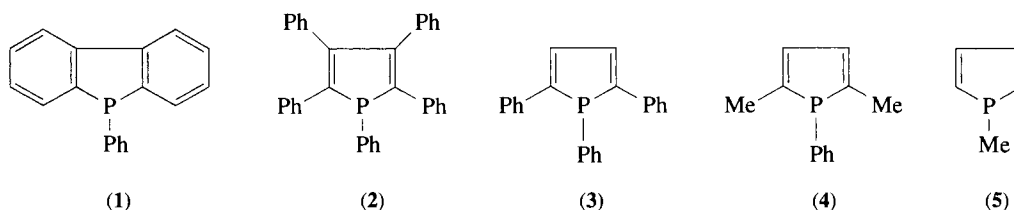
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2.15.1 INTRODUCTION

The fully unsaturated five-membered ring system containing a phosphine unit (phosphole) did not make an appearance in the chemical literature until 1953, when Wittig and Geissler announced the synthesis of the dibenzophosphole derivative (1) <53LA(580)44>. Another six years were to pass before two groups independently reported the construction of the pentaphenyl derivative (2) of the monocyclic parent <59CI(L)1250, 59JA3163>. The complexity of these derivatives prevented detailed study of the ring system, and the immediate question that arose of the presence of cyclic delocalization could not be addressed. The few reports that appeared in the next 8–10 years showed that simpler derivatives, notably (3) <62CI(L)359>, (4) <67AG(E)86>, and (5) <67JA5984> could be

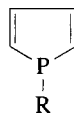
prepared; the character of the phosphole ring then began to emerge and the study of the chemistry of the phosphole system commenced in earnest.



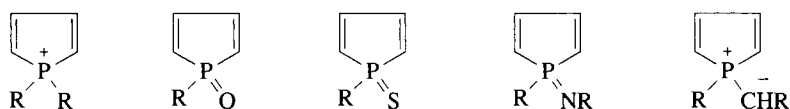
Interest has remained high, and many phospholes are now known, including some with phosphorus in other states of coordination (number of atoms directly attached to phosphorus) and with various rings fused to the parent. A major development has been the synthesis and study of a fascinating array of metallic derivatives and coordination complexes, some of which exhibit unique and valuable properties. Research in phosphole chemistry continues unabated, and it is a major part of contemporary phosphorus chemistry. Recognition of phospholes as having importance in heterocyclic chemistry has been slower to develop, however, and in the first edition of *Comprehensive Heterocyclic Chemistry* (CHEC-I) <84CHEC-I(1)518> only four pages were devoted to the field. Even some later monographs on heterocyclic chemistry fail to mention the existence of this field. It is hoped that this chapter will aid in heightening the interest of general heterocyclic chemists in this area. To this end, the chapter is written insofar as possible with that audience in mind, and some of the intricacies of phosphorus chemistry are not allowed to dominate the presentation. There are, in fact, many reviews of phosphole chemistry that provide great detail on the subject, and are recommended to those wishing further insight. For a complete coverage of the early work (to about 1976) on phospholes, three reviews are available <70JHC1, B-70MI 215-01, 76JHC1>. Later reviews were published by Hughes in 1979 <B-79MI 215-01>, by Mathey in 1980 <80MI 215-01> and Quin in 1981 <B-81MI 215-01>. Reviews from the late 1980s and early 1990s include those of Mathey in 1988 <88CRV429>, Quin and Hughes in 1990 <B-90MI 215-01>, and lastly Hughes in 1992 <B-92MI 215-01>.

A complexity, yet at the same time a source of fascinating chemistry, of systems based on phosphorus is that the phosphorus may appear in functional groups where this atom is attached to anywhere from one to six other atoms. This has led to a description of the group by its "coordination number" (C.N.), which refers to this number of attached atoms. Thus simple phospholes are designated as having C.N.-3, but the coordination number may range from two to six. However, some of these coordination numbers are rather rare, and most of phosphole chemistry is based on C.N.-3 and C.N.-4, which includes especially the oxides, sulfides, and salts of the phosphines. Structures for the various coordination numbers are given below.

C.N.-3. This is considered to be the parent ring system, with analogy to pyrroles, and accorded the most attention as possibly possessing the cyclic delocalization found in the N, S, and O counterparts.

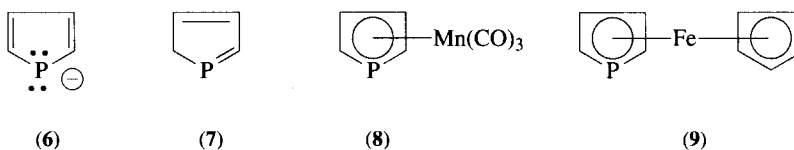


C.N.-4. Salts, oxides, sulfides, imines, and ylides of the parent. Here cyclic delocalization is not possible since the lone pair on phosphorus is used in a fourth bond. Most of the oxides and some of the other functions are unstable at or slightly above room temperature, forming Diels-Alder dimers in the manner of thiophene oxides.

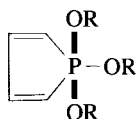


C.N.-2. Phosphole anions (6) and 2H-phospholes (7). Very different species, the former is quite stable and possesses significant cyclic delocalization, while the latter readily undergoes dimerization. Phosphole anions can take the place of cyclopentadienide ion in metallocene complexes, forming

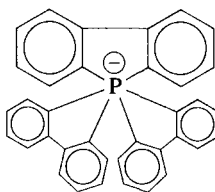
another family of delocalized heterocycles, such as the phosphacymantrene (8) and phosphoferrocene (9) shown below.



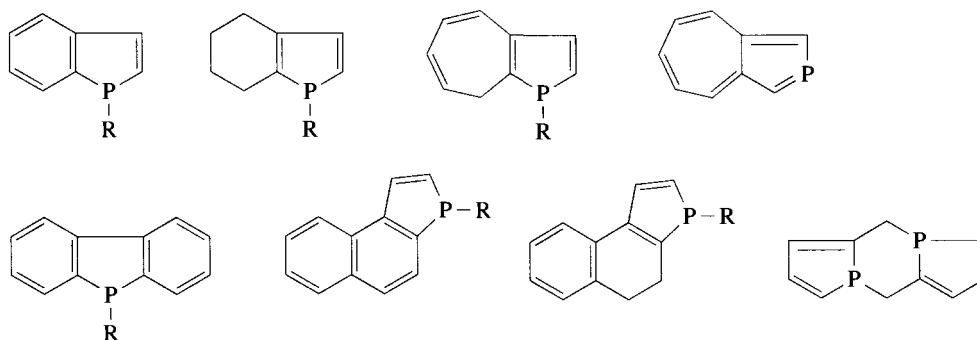
C.N.-5. Phosphoranes. These are uncommon derivatives, following the usual rules of phosphoranes that stability requires the presence of multiple electronegative substituents. Unlike the lower coordination numbers, this condition develops two sets of bond angles around phosphorus, 120° in the equatorial plane and 90° in the two apical positions.



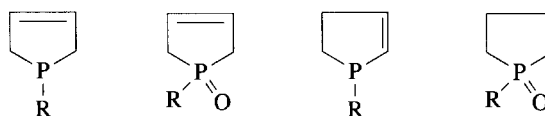
C.N.-6. Hexacoordinate phosphorus. This is the rarest of the coordination numbers, and can only exist in anionic form. The geometry at phosphorus is square pyramidal.



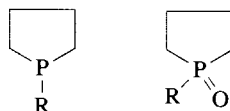
The parent phosphole ring can be fused to a number of other cyclic structures; for many of these compounds the coordination number can be varied, but most of the known compounds are found in phosphole form, or as the oxide derivative. The ring systems that are the most common are shown below. By far the most widely studied is the dibenzo derivative, because it is easily synthesized and worked with. However, the system displays few properties of a phosphole and is more like a diphenylphosphine. This system is accorded abbreviated attention; for current interest, a series of papers by Cornforth *et al.* starting in 1982 should be consulted (82JCS(P1)2289).



Dihydro derivatives of phospholes, commonly referred to as phospholenes, are easily prepared and known in great number. The oxides are especially well studied, the result of a highly versatile and simple synthetic procedure (the McCormack reaction) being available.



Tetrahydro derivatives are known as phospholanes and well known as the phosphine and the oxide.



From the above introductory survey of structural possibilities, it is easily seen that a very great variety of phosphole derivatives is known. However, many remain to be synthesized, and even the information on some of those that exist is sketchy. Unanswered questions abound, and it is a field that can be expected to thrive and grow in the years ahead, as it slowly approaches the degree of maturity that exists for the related N, S, and O heterocyclic systems.

2.15.2 THEORETICAL STUDIES OF PHOSPHOLES

Knowledge of the electronic structure of the phosphole ring is far less advanced than it is for the related heterocyclopentadienes, and in spite of the years of study of this ring system the degree of cyclic electron delocalization remains unclear. No thermochemical measurement of the resonance energy has ever been made with appropriate compounds, and the chemical and physical properties, while sometimes suggestive of a degree of delocalization, are not compelling. Nor have purely theoretical treatments given much insight into the electronic structure; indeed, many have merely contributed uncertainty, with some studies speaking to a significant degree of delocalization, others denying it almost completely. The last theoretical treatment of the electronic structure was reported in 1976, before the advent of the supercomputer. It is hoped that a detailed reexamination of the electronic structure with the advanced computational techniques of today might at last lead to a clearer understanding of this matter. Indeed steps in this direction are already being taken; beginning in 1988, there have been a series of publications on calculations of the geometry of pyramidal and planar phosphole, and of the energy difference between the two forms. Also, for the first time, theoretical calculations of the ^{31}P NMR shift of phospholes (see Section 2.15.3.2.1), and of the relative stabilities and energy of interconversion of 1*H*-, 2*H*-, and 3*H*-phosphole, have been made. Some of these theoretical treatments are discussed in more detail later in this section. It does seem obvious that the new techniques can make a major contribution to our understanding of the true nature of phospholes.

Three years after phospholes first became available (as the pentaphenyl derivative $\langle 59\text{CI(L)1250, 59JA3163} \rangle$, Brown published a theoretical treatment of the unsubstituted system that was based on the relatively simple molecular orbital theory of the time $\langle 62\text{JCS929} \rangle$. With the assumption that the system was planar, it was concluded that the conjugation energy for the phosphole ring was considerable, 1.49β in a calculation giving 1.27β for pyrrole. Also in 1962 appeared thermochemical measurements of the oxidation of pentaphenylphosphole, showing that, relative to the *p*-oxide, the phosphole was more stable by $39 \pm 13 \text{ kcal mol}^{-1}$, and this was taken as a measure of the conjugation energy $\langle 62\text{JCS2932} \rangle$. However, studies with such a highly substituted derivative cannot give much information on the true nature of the ring, and it was not until synthetic work was initiated by three groups $\langle 67\text{AG(E)86, 67JA5984, 69CR(C)1066} \rangle$ that simple phospholes without the interfering ring phenyls became available and made it possible to begin serious experimental examination of the question of cyclic delocalization in the ring. Some spectral properties pointed to a degree of delocalization (see Section 2.15.3), and an x-ray diffraction analysis of the solid 1-benzylphosphole showed bond order changes that supported the concept $\langle 70\text{JA5779, 73JCS(D)1888} \rangle$. That phosphorus had retained the pyramidal character of a phosphine was first revealed by stereochemical studies with phospholes lacking symmetry $\langle 70\text{JA1442, 71JA6205} \rangle$, and confirmed by the x-ray analysis of 1-benzylphosphole. The latter analysis showed that the phosphorus was slightly out of the plane of the four ring carbons, but more importantly that the angle at P between the benzylic C and the ring C was 106° . These observations voided the early treatment of Brown, who had assumed full planarity of the system. The stereochemical studies showed that the inversion barrier in 1-isopropyl-2-methyl-5-phenylphosphole (16 kcal mol^{-1}) was some 20 kcal mol^{-1} below that of a phosphine model, and this was interpreted as meaning the planar transition state for the inversion was stabilized by cyclic electron delocalization $\langle 71\text{JA6205} \rangle$. Some of the theoretical studies that followed were greatly concerned with this point, and questioned the extent of electron delocalization that could occur from pyramidal phosphorus. Several such studies were made over the period 1970–1976 but gave conflicting results,

some stating that conjugation was virtually absent in the system $\langle 73\text{AG(E)145, } 73\text{T469, } 75\text{JCS(P2)974} \rangle$, others that it could be considerable $\langle 71\text{JA6507, } 74\text{JA6904, } 74\text{PS(4)231, } 76\text{JA407, } 76\text{JA4365} \rangle$. Advances in computational techniques were made during this period, and are reflected in the approaches taken by the authors. Thus the Pople CNDO/2 semiempirical MO theory was applied by Rauk *et al.* $\langle 71\text{JA6507} \rangle$, and an advanced semiempirical method was also used by Andose *et al.* $\langle 74\text{JA6904} \rangle$, that supported the concept of delocalization in the planar transition state. Kaufmann and Mathey $\langle 77\text{4PS(4)231} \rangle$ employed LCAO–MO–CNDO/2 calculations and found that $2p$ – $3d$ π interaction played a role both in the pyramidal state and in the planar transition state of inversion. However, Palmer and Findlay $\langle 75\text{JCS(P2)974} \rangle$ performed a treatment by linear combination of Gaussian orbitals (LCGO) which showed no delocalization, and all of the known experimental data were reinterpreted on that basis. However, within a year Epiotis and Cherry $\langle 76\text{JA4365} \rangle$ published the results of SCF–MO–CNDO/2 calculations that found considerable “aromaticity” to be present. With the exception of the last paper, the salient points of all of these theoretical evaluations have been reviewed in 1976 by Hughes and Kleemola $\langle 76\text{JHC1} \rangle$. The theoretical inquiry rested at this inconclusive stage for 12 years. Probably the consensus opinion of the phosphorus community during that period was that expressed by Mathey in a 1988 review: the phosphole ring may have only weak delocalization, less than that in furan, and ought not to be considered as an aromatic system $\langle 88\text{CRV429} \rangle$. This position relative to furan was emphasized by Bird who devised an “index of aromaticity” in heterocyclic rings $\langle 85\text{T1409} \rangle$ that was applied to some phosphorus systems $\langle 90\text{T5697} \rangle$. The index is based on published experimental bond lengths. Using a scale of 0 (none) to 100 (high), the heterocyclopentadiene series had the indices (I_s) S, 66; Se, 59; NH, 59; O, 43; PhCH_2P , 35.5; 1,2,5- Ph_3 phosphole, 20. This accords with the view that some phospholes may have a measure of delocalization but less than that in furan.

Great advances in computational techniques and in computer power were made in the years following the 1976 theoretical treatment of Epiotis and Cherry, and now the phosphole problem is again a subject for theoretical investigation. In the first of the new round of publications, Baldrige and Gordon reported the optimized geometries for pyramidal 1*H*-phosphole, using the 3-21G* basis set at the SCF level of computation, employing GAUSSIAN 82 $\langle 88\text{JA4204} \rangle$. Hughes and Edgecombe showed in 1992 that with an *ab initio* method using the 6-311G* basis set and the GAUSSIAN 86 program series it was possible to compute optimized geometries for both the pyramidal and the planar state of phosphole, as well as their total energies $\langle 92\text{H(33)563} \rangle$. From the latter, the energy difference, and thus the inversion barrier, was calculated to be 24.4 kcal mol^{−1}. There is no experimental value for the inversion barrier in phosphole itself, but as noted a value of 16 kcal mol^{−1} was found for a 1,2,5-trisubstituted phosphole $\langle 71\text{JA6205} \rangle$. The authors conclude that this reduced inversion barrier is consistent with some electron delocalization in the planar transition state. In 1993 Bachrach also reported geometry calculations for pyramidal phosphole using *ab initio* calculations at the MP2/6-31G* level $\langle 93\text{JOC5414} \rangle$, and Chesnut and Quin in 1994 reported the geometries of both pyramidal and planar 1*H*-phosphole $\langle 94\text{JA9638} \rangle$. In the latter two studies, P was displaced from the four-carbon planar segment by 0.18 and 0.167 Å, respectively. Bachrach and Derriott have calculated parameters for all possible isomers from methyl substitution on the 1*H*-, 2*H*-, and 3*H*-phosphole isomers and on some vinyl derivatives as well $\langle 94\text{JOC3394} \rangle$. Steric interaction causes a preference for P- over C-substitution.

Table 1 Calculated and experimental molecular parameters for phosphole.

	Phosphole ^a					Thiophene ^b		
	Pyramidal					Planar		
	A	B	C	D	E	B	D	
P–C ₂ (C ₅)	1.815	1.817	1.7998	1.817	1.783 ^c	1.716	1.716	1.714
C ₂ (C ₅)–C ₃ (C ₄)	1.334	1.331	1.3618	1.332	1.343	1.371	1.373	1.370
C ₃ –C ₄	1.475	1.473	1.4506	1.471	1.438	1.423	1.420	1.423
$\angle$ C ₂ –P–C ₅	81.6	89.5	90.8	89.68	90.7	98.2	98.2	92.17
$\angle$ P–C–C	110.9	111.2	110.6	110.63	110 ^c	105.6	105.5	111.47
$\angle$ C–C–C	113.9		114.1	114.19	114.1		115.4	112.45
$\angle$ X–P–C ₂ (C ₅) ^d	99.2	99.3			106 ^c	130.9		

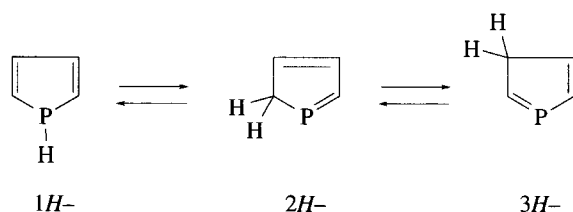
^aA = Baldrige and Gordon $\langle 88\text{JA4204} \rangle$; B = Hughes and Edgecombe $\langle 92\text{H(33)563} \rangle$; C = Bachrach $\langle 93\text{JOC5414} \rangle$;

D = Chesnut and Quin $\langle 94\text{UP 215-01} \rangle$; E = experimental for 1-benzylphosphole $\langle 70\text{JA5779} \rangle$.

^b $\langle 94\text{JA9638} \rangle$. ^cAveraged value. ^dX = H or CH₃.

The good agreement between the geometries calculated by the various groups, and with the experimental values for 1-benzylphosphole, is shown in Table 1. It is seen that the experimental bond lengthening at C_2-C_3 in 1-benzylphosphole is between the values for planar and pyramidal phosphole, as it is also for C_3-C_4 . If it can be concluded that no delocalization is present in the calculated pyramidal 1*H*-phosphole, then these experimental values are in the correct direction to indicate some delocalization in 1-benzylphosphole, as of course was concluded by Bird in his treatment of this compound. There is a striking agreement for the lengths of these same carbons between the experimental (microwave) values for thiophene and the planar form of 1*H*-phosphole. The thiophene parameters are also included in Table 1 for comparison. This might suggest that the degree of delocalization in planar phosphole would be the same as that in thiophene, which is generally accepted to be considerable. This is supported by a calculation of the Bird indices $\langle 94PC215-01 \rangle$ for planar 1*H*-phosphole and 1-methylphosphole using the computed bond-length values of Chesnut and Quin. The indices (I_s) are 75 and 83, respectively, compared to the experimentally based value for thiophene of 66. It follows that a natural phosphole which for steric reasons adopts a shape with an angle intermediate between the extreme values of pyramidal and planar might partake of an intermediate degree of delocalization, which need not be trivial. The view that natural phospholes may vary in their degree of delocalization depending on their structure was early emphasized by Kaufmann and Mathey $\langle 74PS(4)231 \rangle$, thus making a flat statement that, as a family, phospholes lack delocalization seem incautious. Qualitative variation in the degree of delocalization is a point that has already appeared in the literature concerned with the chemical character of phospholes, as is noted in Section 2.15.5.

Bachrach has also considered the relative stabilities of the isomeric forms resulting from placement of the "extra hydrogen" at ring positions 1, 2, and 3 of phosphole (Scheme 1).



Scheme 1

These forms are related through [1,5] sigmatropic hydrogen shifts. An interesting discovery is that 2*H*-phosphole is the most stable of the three, in particular some $6.53 \text{ kcal mol}^{-1}$ more stable than the 1*H*-isomer. The activation energy for the 1*H* to 2*H* conversion is only $16.0 \text{ kcal mol}^{-1}$. Indeed, it is seen in Section 2.15.5.8 that 1*H*-phosphole exists only at low temperature and rearranges to 2*H*-phosphole as the temperature rises, then undergoing dimerization. Activation energies for the cycloaddition of ethyne to the phosphole isomers were calculated in later work; the 2*H*-isomer had the lowest energy in this process which was confirmed to be of the concerted $[4 + 2]$ type $\langle 94JOC5027 \rangle$.

The situation regarding delocalization in phospholide ions is quite different and not controversial. The problem of pyramidal structure at P is of course absent, and the parent phospholide ion is isoelectronic with thiophene. Some early calculations of Kaufmann and Mathey $\langle 74PS(4)231 \rangle$ suggested that the ion would be highly delocalized. Calculations by Malar $\langle 92JOC3694 \rangle$ using the all-valence SCF-MO method SINDO1, which includes some *d*-orbital contribution, suggest that the delocalization is better described as only "moderate"; they concluded that the ion possesses 90.7% of the aromaticity of cyclopentadienide ion. Optimized geometries for the system are given in Table 2, where it will be seen that the C_2-C_3 and C_3-C_4 bonds are nearly equal (1.402 and 1.405 Å, respectively) from the delocalization. The P—C bond is shortened to 1.749 Å. The C—P—C bond angle is 94.2° and thus not greatly different from that in phospholes. Chesnut and Quin $\langle 94JA9638 \rangle$ included phospholide ion in their theoretical calculations and found C—C bond lengths that are not quite so equalized (1.376 and 1.424 Å, respectively), with C—P at 1.764 Å. Their results are, in fact, in very close agreement with the experimental structural parameters reported for lithium phospholide as a TMEDA solvate $\langle 89AG(E)1367 \rangle$; the data are compared in Table 2. This paper makes no conclusions on the extent of delocalization, except to note that it is present and important.

Chesnut and Quin have extended their calculations to include a treatment of the cause of the very strong deshielding of ^{31}P that characterizes all known phospholide ions. This theoretical work is presented in Section 2.15.3.2.1. Relevant to the present discussion is the conclusion that, although

Table 2 Calculated and experimental molecular parameters for phospholide ion.

	<i>A</i> ^a	<i>B</i> ^a	<i>Experiment</i> ^b
P—C ₂ (C ₅)	1.749	1.764	1.715 ^c
C ₂ (C ₅)—C ₃ (C ₄)	1.405	1.376	1.396
C ₃ —C ₄	1.402	1.416	1.424
∠ C ₂ —P—C ₅	94.2	89.71	90.5
∠ P—C—C	108.5	112.23	112.3 ^c
∠ C—C—C	114.4	112.92	112.6

^aA = Malar <92JOC3694>; B = Chesnut and Quin <94JA9638>. ^bFor LiC₄H₄P·TMEDA <89AG(E)1367>. ^cAveraged.

delocalization is extensive in the phospholide ring, this is not the direct cause of the deshielding, and there is no connection with the increased P=C double bond character from the delocalization as was first suggested when the strong deshielding was discovered <79CC401>. The deshielding arises from an orbital effect of the lone pair on phosphorus that lies in the plane of the ring, placed there by the conjugation in the ring.

2.15.3 EXPERIMENTAL STRUCTURAL METHODS

2.15.3.1 X-ray Diffraction Analysis

The structures of six phospholes (two with a metal σ -bonded to P) have been determined by x-ray diffraction analysis (Table 3). In five of these, the C—P—C internal angle was determined, and found to be quite small (89.2–91.8°), as it is in thiophene (92.2°) <61JSP(7)58>. The phosphorus atom is slightly out of the plane of the ring carbons, and retains its pyramidal configuration, a point of relevance in discussion of the degree of delocalization in the phosphole ring (Section 2.15.2). Thus, in 1-benzylphosphole, the angle at phosphorus between C₂ or C₅ and the benzyl substituent is 106°, which places the substituent below the plane of the ring atoms by 66.9°. This bond angle is greater with the tungsten substituents, and the pyramidal shape at P is diminished.

Delocalization is characteristically associated with increases in double bond lengths and decreases in single bond lengths, and in one structure, that of 1-benzylphosphole, these effects are clearly apparent. C-substitution, especially where leading to steric crowding, appears by other criteria to be associated with diminution of delocalization, and to some extent this can be detected in the crowded structures of Table 3. Thus, the C—C single bond is smallest in 1-benzylphosphole (1.438 Å) and increases to as much as 1.472 Å with substituents at positions 2, 3, and 4. Unfortunately, obtaining structural information by x-ray analysis in the phosphole field has been hampered by the low melting points of most of the simple compounds, and further exploration of the presence of delocalization effects by structural analysis has not yet been possible. (The bonding in the tungsten derivatives is not clear and is not included in this discussion.)

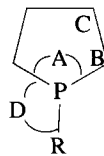
Structures for several phospholes with higher coordination numbers at phosphorus have also been reported and are included in Table 3, as are some reports on benzo derivatives. A structure for the 2-coordinate phospholide ion (as a lithium salt) is also included; the effects of delocalization are pronounced in this case, consistent with other observations on such ions. The C—C single bond is decreased to 1.424 Å, and the C=C double bond is increased to 1.396 Å.

A number of structures of phosphole coordination complexes have been determined but are not included in this review. Many of them are described elsewhere <B-83MI 215-01>.

2.15.3.2 NMR Spectroscopy

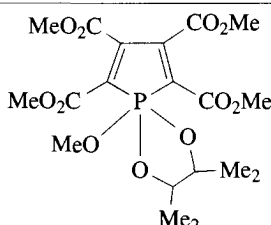
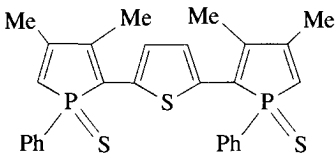
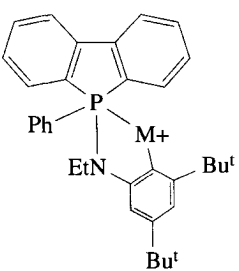
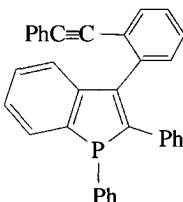
2.15.3.2.1 ³¹P NMR spectroscopy

The immediate determination of the ³¹P NMR shift is virtually routine in the characterization of new phosphorus compounds or analysis of reaction mixtures, and this is certainly the case in the

Table 3 Structural parameters for phospholes.

Compound	Bond angles ^a				Bond lengths ^a				Ref.
	A	B	C	D	P—C(R)	P—C ₁	C ₁ —C ₂	C ₃ —C ₄	
	90.7	110.0	114.1	106.0	1.858	1.783	1.343	1.438	73JCS(D)1888
					1.822	1.822	1.349	1.440	71CC1062
	89.2	111.5 112.9	112.8 113.1	102.9	1.840	1.804 1.789	1.360 1.332	1.472	80CSC905
						1.785	1.374	1.442	83JCS(D)841
	90.7	111.3	113.0	116.2 112.7	2.571	1.771	1.37	1.447	93OM98
	91.4	109.7	114.5	110.8 112.9	2.448	1.795	1.363	1.439	93OM98
	93.3	108.7	114.6	108.1	1.796	1.810	1.355	1.505	76CB877
	91.4	110.5	113.9	108.1	1.807	1.813 1.786	1.353 1.336	1.494	80CSC901

Table 3 Continued.

Compound	Bond angles ^a				Bond lengths ^a				Ref.
	A	B	C	D	P—C(R)	P—C ₁	C ₁ —C ₂	C ₃ —C ₄	
	87.9	111.1	115.5		1.837 1.885	1.334 1.340	1.462		82AX(B)3024
		92.6 ^b 92.2							91HAC377
 Li ⁺ (TMEDA)	90.5	112.3	112.4		1.751	1.396	1.424		89AG(E)1367
	86.3	113.7	112.2		1.85	1.37	1.47		83CB1914
	90.1	111.4	114.2	102.7	1.833	1.826 1.805	1.352 1.402	1.466	83CB3271

^a Averaged values reported for two bonds in a molecule in some cases. ^b See ref. for additional data.

subfield of phosphole chemistry. The ³¹P NMR shift of phospholes is significantly displaced in the downfield direction relative to model tertiary or secondary phosphines, and substitutions can produce additional profound modifications of the shifts. With a proper understanding of the structure-shift relations, the technique can be of enormous importance in structure assignments, and is given special emphasis in this section. Wherever possible, the considerable NMR data available are organized around the shift changes that take place when structural modifications are made on the simplest phospholes with tertiary structure at phosphorus, 1-methylphosphole or 1-phenylphosphole. Much less is known about 1*H*-phospholes, but effects there are treated as well. All ³¹P shifts are referenced to 85% H₃PO₄ as 0 ppm; downfield shifts bear a positive sign. Literature before about 1978–1980 used the opposite sign convention, but these have been converted to the current convention. The shifts for phospholes with C.N.-3 are listed in Table 4; data for phosphole oxides, phosphole sulfides, phospholium salts, and ylides are given in Table 5. References are given in the tables, and are not repeated in the discussion which follows.

Table 4 ^{31}P NMR shifts of phospholes.

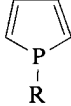
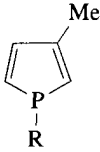
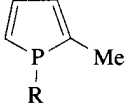
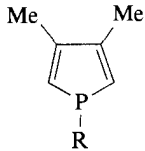
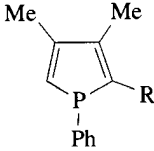
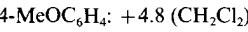
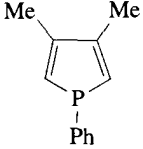
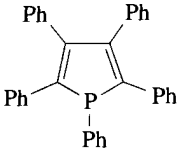
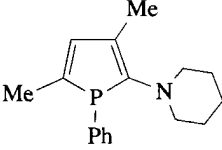
Compound		Ref.
	R = H: -49.2 ($\text{C}_4\text{D}_8\text{O}$) Me: -8.7 (neat) Bu ⁿ : $+5.5$ Ph: $+6.4$ PhCH ₂ : $+8.0$ (neat) PhCH ₂ CH ₂ : $+5.8$ (neat) 4-Cl—C ₆ H ₄ CH ₂ : $+5.9$ (neat)	87TL5025 69JA3308 72OMR(4)171 81S983 73JOC1858 73JOC1858 73JOC1858
	R = Me: -6.9 (neat) Bu ⁿ : $+8.5$ Bu ^t : $+40.5$ (neat) PhCH ₂ : $+11.5$ (neat) Ph: $+7.5$	73JOC1858 72OMR(4)171 72T4171 73JOC1858 81S983
	R = Me: -7.3 (neat)	73JOC1858
	R = H: -59.4 ($\text{C}_4\text{D}_8\text{O}$) Me: -20.2 (neat) Bu ⁿ : -6.5 Bu ^t : $+27.5$ PhCH ₂ : -3.0 (neat) CN: -54.7 W(CO) ₃ Cp: -105.2 (CD_2Cl_2) Me ₃ CO: $+82.6$ 9-fluorenyl: $+1.0$ Ph: -4.4	87TL5025 73JOC1858 76T2395 72T4171 73JOC1858 88OM1796 93OM98 88OM1796 88OM1796 81S983
	R = Me: $+6.6$ COOH: $+5.7$ (CH_2Cl_2) COOEt: $+5.0$ CHO: $+1.3$ (CH_2Cl_2) COMe: $+5.0$ COPh: $+11.3$ (THF) Br: $+10.2$ (toluene) SiMe ₃ : $+15.3$ (THF)	94OM925 92BSF486 76T2395 92BSF486 76T2395 92BSF486 92BSF486 92BSF486 90JOC2494
	: $+6.6$ (CH_2Cl_2)	90JOC2494
	: $+4.8$ (CH_2Cl_2)	90JOC2494
	: $+16$ (C_5H_{12})	92BSF486
	$+13.9$	82TL511
	-6.3	88TL4581

Table 4 Continued.

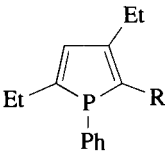
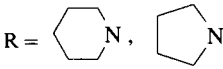
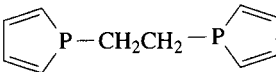
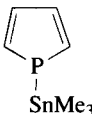
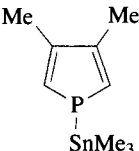
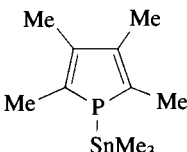
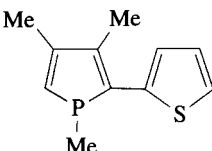
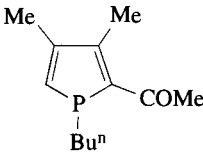
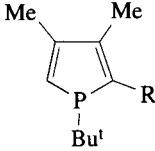
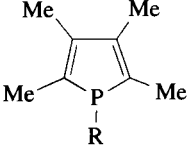
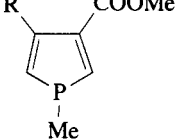
Compound		Ref.
		88TL4581
	+9.96 (CD ₂ Cl ₂)	87TL5025
	-50.09	88CC770
	-65.09	88CC770
	-45.96	88CC770
	-8.4	90JOC2494
	+6.8	76T2395
	R = COMe: +35.1 COOEt: +34.3	76T2395 76T2395
	R = Ph: 12.71 Cl: +80.69 (C ₆ D ₆) H: +63.02 (C ₄ D ₈ O)	88OM921 88OM921 87TL5025
	R = H: +3.0 Me: -12.6	73JOC1858 73JOC1858

Table 4 Continued.

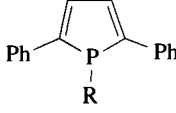
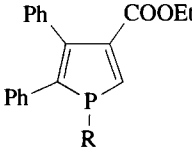
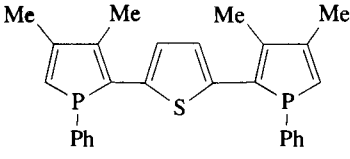
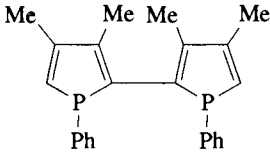
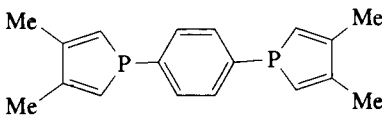
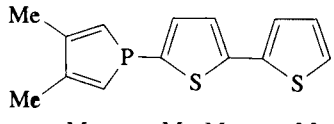
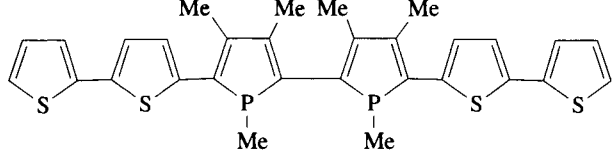
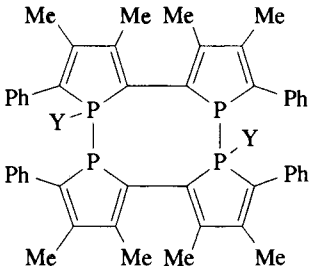
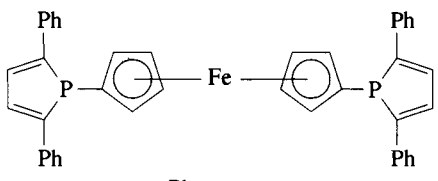
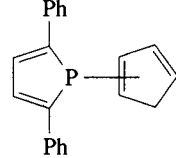
Compound		Ref.
	R = H: +78.75 (C ₄ D ₈ O) Ph: +0.32 Br: +31.1 MeO: +112.9 EtO: +107.4 Cp(CO) ₃ W: -98.8 (C ₆ D ₆)	87TL5025 87TL5025 82JOM(231)361 82JOM(231)361 82JOM(231)361 93OM98
	R = Me: -0.4 (CH ₂ Cl ₂) CO ₂ EtCH ₂ CH ₂ : +11.9 (CH ₂ Cl ₂)	93BSF695 93BSF695
	+4.0	91HAC377
	+12.5	86JOM(316)271
	-3.4 (CD ₂ Cl ₂)	90HAC187
	-25.2 (C ₆ D ₆)	90AG(E)655
	+7.5	90AG(E)655
	Y = l.p: -11.6 S: -10.3	81JA4595 81JA4595
	-5.5 (CH ₂ Cl ₂)	87JOM(332)141
	isomers: -13.3 (CH ₂ Cl ₂) and -15.7 (CH ₂ Cl ₂)	87JOM(332)141

Table 4 Continued.

Compound		Ref.	
	$P_A + 28.7, P_B - 1.7$ ($^1J = 370$ Hz)	83JA6871	
	-14.6	83JA6871	
	-25.3	83OM1234	
	-27.9	82JOM(231)361	
	-11.8	82JOC2376	
	isomers: -29.06, -33.32 R = Ph: -11.6	94JA3306 82JA2077	
		-2.1	90AG(E)655
	R = Ph, R' = H: -8.3 R = H, R' = Me: -13.3	93PS(85)207 93PS(85)207	

Table 4 Continued.

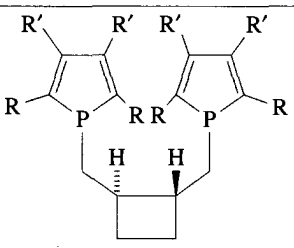
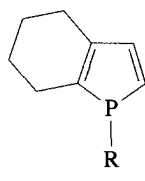
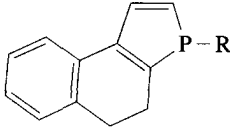
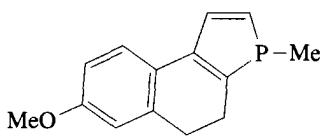
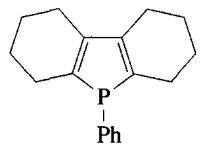
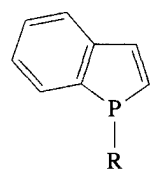
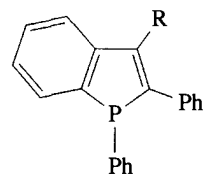
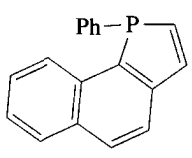
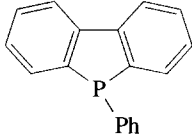
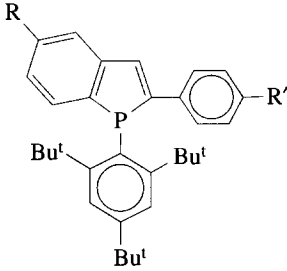
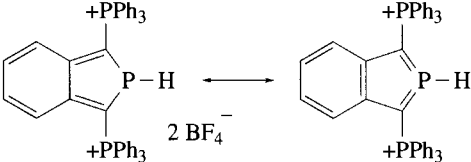
Compound		Ref.
	R = Ph, R' = H: -0.8 R = H, R' = Me: -9.0	93PS(85)207 93PS(85)207
	R = Me: -7.6 Ph: +10.5	79CC1100 79CC1100
	R = Me: -4.1 Ph: +12.8	82PS(12)161 82PS(12)161
	-4.9	82PS(12)161
	+7.6	78CJC1952
	R = Ph: -2 Bu ^t : +23.5	82PS(13)259 82PS(13)259
	R = H: -1.1 Cl: +0.6  : +2.4 (CHCl ₃) C≡CPh	84CB990 84CB990 77CB2168
	+0.1	82PS(12)161

Table 4 Continued.

Compound		Ref.
	-10.2	65CB576
	R = R' = H: -13.92 R = OMe, R' = H: -15.74 R = H, R' = OMe: -14.22 R = R' = OMe: -15.67	93TL3103 93TL3103 93TL3103 93TL3103
	+41.7 (also +23.1, $^2J = 12.3$)	91AG(E)308

^aIn CDCl₃ unless otherwise noted.

(i) Effect of substitution at the P-methyl group

Tertiary phosphines (and other classes of phosphorus compounds with carbon substituents) respond to the effect well known in ¹³C NMR that replacement of H of a methyl group by a methyl group causes a significant downfield shift (the β -deshielding effect), and replacement of H on the methyl group of ethyl causes a smaller upfield shift (the γ -shielding effect). Average values for saturated tertiary phosphines are +13.5 and -4 ppm, respectively, and are additive (<73OMR(5)17>). An examination of the data available for phospholes clearly reveals these effects to be operating, but data are insufficient to calculate appropriate constants. Thus, 1,3-dimethylphosphole has a shift of δ -6.9; 3-methyl-1-*t*-butylphosphole has a shift of δ +40.5, which implies a quite reasonable β -deshielding of 15.8 ppm per methyl in the *t*-butyl group. An identical value is found in the replacement of the 1-methyl group of 1,3,4-trimethylphosphole by *t*-butyl (δ +27.5). However, the crowded *t*-butyl is not the best model for making this calculation and the β -constant of 15.8 ppm must be considered as a tentative value. Nevertheless, this reasoning allows an interpretation of the shift of δ +5.5 reported for 1-*n*-butylphosphole; here there is a combination of the large β -deshielding and small γ -shielding, and if the former value is taken as +15.8 ppm from the *t*-butyl group calculation, then the γ -shielding is seen to be -1.6 ppm. Similar values for γ -shielding are found in the 1,3,4-trisubstituted compounds (1,3,4-trimethyl, δ -20.2 (neat); 3,4-dimethyl-1-*n*-butyl, δ -6.5; γ -shielding of -2.1 ppm) and in the 1,3-disubstituted compounds (1,3-dimethyl-, δ -6.9; 3-methyl-1-*n*-butyl-, δ +8.5; γ -shielding of -0.4 ppm). Using this logic, the values of δ +5.8 for 2-phenethylphosphole, and δ +9.96 for 1,2-bis(2-phospholyl)ethane are understandable.

Replacement of a methyl H in 1-methylphosphole by phenyl causes a substantial downfield shift (1-benzylphosphole, δ +8.9) implying that a β -phenyl has a constant of +17.6 ppm. The constancy of this value is seen in the comparison of, for example, 1,3-dimethylphosphole (δ -6.9) to 1-benzyl-3-methylphosphole (δ -3.0; $\Delta\delta$ 18.4 ppm).

(ii) Effect of replacement of methyl by other substituents

In tertiary phosphines, replacing methyl by phenyl causes a substantial downfield shift, and this is found also among phospholes (1-phenyl, +6.4 from -8.7, $\Delta\delta$ +15.1 ppm; 3-methyl-1-phenyl,

Table 5 ^{31}P NMR shifts of phosphole derivatives with C.N.-4 and C.N.-5.^a

Compound	$\delta^{31}\text{P}$	Ref.
<i>A. Oxides</i>		
	+46.6	71JA4541
	+42.3	91HAC359
	+55.7	78CJC1952
	R = Me: +46.3 Ph: +38.7 MeO: +36.88 EtO: +35.28 Pr ⁱ O: +34.0	88JCS(P1)175 88JCS(P1)175 84JCS(P1)1627 84JCS(P1)1627 88JCS(P1)175
<i>B. Sulfides and selenides</i>		
	R = Bu ⁿ : +52.2 Bu ^t : +70.5 Ph: +46.1	76T2395 76T2395 76T2395
	R = Bu ⁿ : +55.3 Bu ^t : +73.8 Ph: +48.0	76T2395 76T2395 76T2395
	R = EtOOC: +48.8 HOOC: +53.2	76T2395 76T2395
	+70.5	76T2395
	+28.5	72T4171
	+35.7	86JCR(S)392

Table 5 Continued.

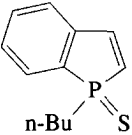
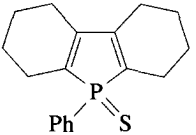
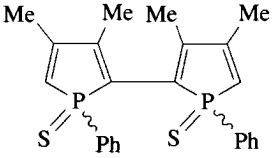
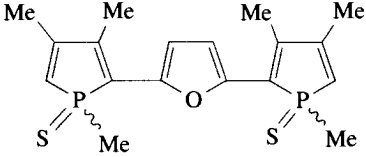
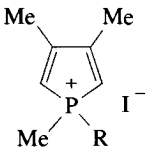
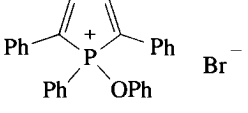
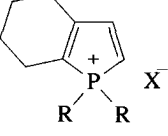
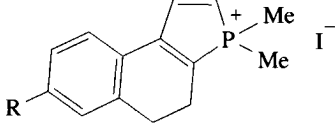
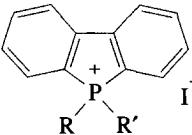
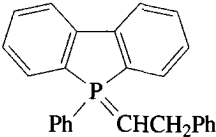
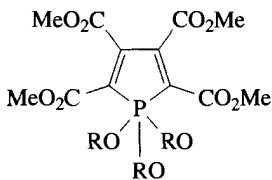
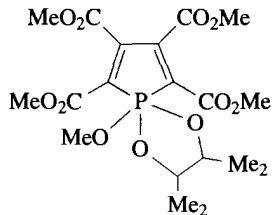
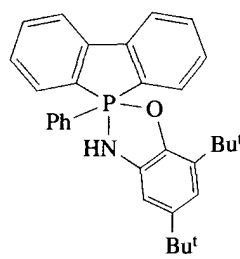
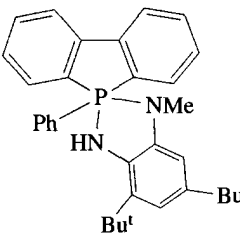
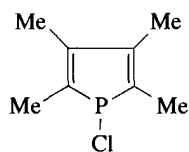
Compound	$\delta^{31}\text{P}$	Ref.
	+ 5.1	82PS(13)259
	+ 50.9	78CJC1952
	+ 47.1 and + 47.7	86JOM(316)271
	+ 44.73 and + 45.03	91HAC377
<i>C. Salts</i>		
	R = Me: + 34.0 (D ₂ O) Bu ⁺ : + 52.8	73JOC1954 72T4171
	+ 74.6	77JCS(P1)1044
	R = Me (I ⁻): + 44.1 PhCH ₂ (Br ⁻): + 50.1	79OMR442 79OMR442
	R = H: + 48.3 MeO: + 46.4	79OMR442 79OMR442
	R = R' = Me: + 27 (TFA) R = Me, R' = Ph: + 21.5(?)	70JCS(B)1527 72JCS(P1)2713
<i>D. Ylides</i>		
	+ 17	72JCS(P1)2713

Table 5 Continued.

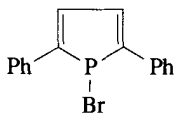
Compound	$\delta^{31}\text{P}$	Ref.
<i>E. C.N.-5</i>		
	R = Me: -37.4 (toluene) Et: -41.84	84JCS(P1)1627 84JCS(P1)1627
	-35.5	81TL3533
	-57.5 (acetone)	83CB1914
	-68.94 (toluene)	83CB1914

^aIn CDCl₃ unless otherwise noted.

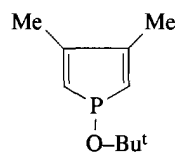
+ 7.5 from -6.9 , $\Delta\delta + 14.4$, etc.). Replacing methyl by Cl, Br, or OR causes much more pronounced downfield shifts ((10), $\delta + 80.69$; (11), $\delta + 31.1$; (12), $\delta + 82.6$). On the other hand, a very strong shielding takes place when methyl is replaced by cyano ((13), $\delta - 54.7$). These effects of P-substituents are common among phosphines in general.



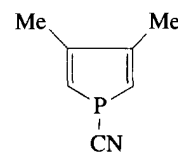
(10)



(11)



(12)



(13)

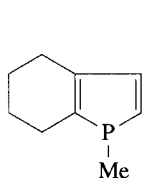
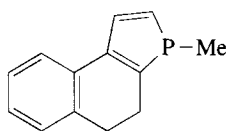
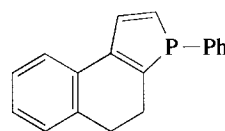
(iii) Effect of alkyl or aryl substitution on ring carbons

A peculiarity noted in ^{31}P values of 3-phospholenes <72PS(2)55> is very evident also in the phosphole family: replacement of a 3H by methyl causes a small downfield shift (to $\delta - 6.9$) from the value for the 1-methyl derivative ($\delta - 8.7$), but a second methyl placed at the 4-position causes a very strong

upfield shift (to $\delta -20$, $\Delta\delta -11.5$ ppm). The same effect of 3,4-dimethyl substitution is seen in the *n*-butyl- ($\Delta\delta -15.0$ ppm), P-phenyl ($\Delta\delta -11.9$ ppm) and P-benzyl series ($\Delta\delta -11.9$ ppm), and elsewhere. No satisfactory explanation of this effect seems to have been published but one intuitively thinks of the steric crowding accompanying *vic*-dimethyl substitution as being involved. The existence of this effect needs to be remembered when examining the literature, since many known phospholes in fact have 3,4-dimethyl substitution, and the reported shifts are no doubt some 10 ppm more upfield than less crowded, more representative derivatives. As will be noted, the 3,4-dimethyl effect is found in phospholes with higher coordination numbers, and even in phospholide ions.

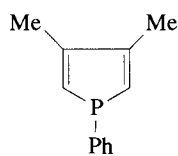
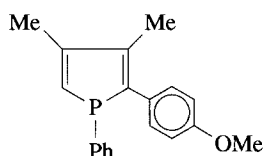
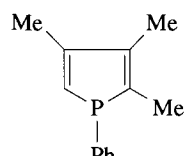
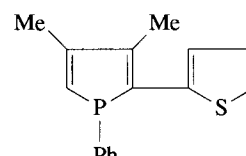
It might be expected that 2,3,4-trimethyl substitution would lead to even further upfield shifting if simple steric crowding were involved, but this does not seem to occur. 1-Phenyl-2,3,4-trimethylphosphole has $\delta +6.6$, whereas the 3,4-dimethyl derivative has $\delta -4.4$. Thus the third methyl has caused a *downfield shift* of 11.0 ppm.

Along the same line, placement of substituents at the 2,3 positions does not cause an upfield shift; 1,2-dimethylphosphole has almost the same shift ($\delta -7.3$) as 1-methylphosphole, and 1-methylcyclohexano[*b*]phosphole (**14**) has a shift of $\delta -7.6$. Similar values are found for the tricyclic phospholes (**15**) and (**16**).

(14), $\delta -7.6$ (15), $\delta -4.1$ (16), $\delta +12.8$

Even 2,3,4,5-tetramethyl substitution does not cause pronounced shift effects. The 1-phenyl derivative has $\delta +12.71$, compared to $\delta +6.4$ for 1-phenylphosphole.

Data are insufficient to evaluate the effect of replacing ring H by aryl groups. Compared to 1-phenylphosphole ($\delta +6.4$), the replacement of both 2 and 5 hydrogens by phenyl leads to a small net shielding (to $\delta +3$). Just as did a methyl substituent, an aryl group placed at the 2-position of a 3,4-dimethyl derivative switches off the shielding caused by this arrangement; relative to (**17**), phospholes (**18**) and (**19**) have significantly more downfield shifts. A 2-thienyl group has a similar effect ((**20**), $\delta +6.6$), and does not seem to introduce any significant shift influence from its electron-releasing character. Thus, (**20**) has exactly the same shift as the 2,3,4-trimethyl derivative (**19**).

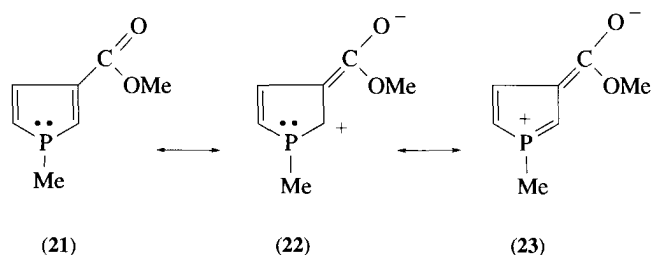
(17), $\delta -4.4$ (18), $\delta +4.8$ (19), $\delta +6.6$ (20), $\delta +6.6$

(iv) Replacement of ring H by conjugating groups

The 3-carbomethoxy derivative of 1-methylphosphole (**21**) was the first functionalized phosphole to be prepared (Scheme 2), and it was discovered that the substituent had a very pronounced deshielding effect (of $+11.7$ ppm) on the ^{31}P NMR shift ($\delta +3.0$). In some way, the conjugation of the ester with a ring double bond (resonance form (**22**)) is strongly influencing the phosphine group, and the explanation offered was that the phosphorus lone pair was also involved in the conjugation (resonance form (**23**)).

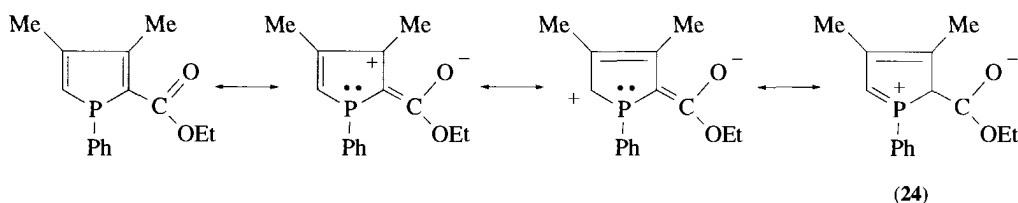
As will be seen, the ^1H NMR and UV spectra are in accord with the conjugation of form (**22**), but do not speak to form (**23**). Substitution of 4-H by methyl caused an upfield shift ($\delta -12.6$), of the same magnitude ($\Delta\delta -15.6$) as seen on converting 1,3-dimethylphosphole to 1,3,4-trimethylphosphole ($\Delta\delta -11.5$ ppm), and thus providing a further extension of the 3,4-disubstitution effect.

The effect of a conjugating group at the 2-position can only be analyzed by examining compounds with 3,4-dimethyl substitution, since simpler models are not yet known. A 2-carboethoxy group



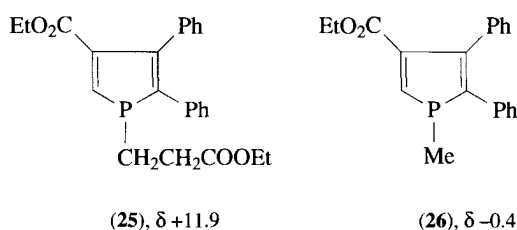
Scheme 2

placed on 3,4-dimethyl-1-phenylphosphole caused a downfield shift of 9.4 ppm ($\delta + 5.0$), an effect like that of a 2-methyl group ($\Delta\delta + 11.0$ ppm). Similarly, a 2-acetyl group led to a shift also of $\delta + 5.0$. The acetyl group had the same effect when placed on 1-*n*-butyl-3,4-dimethylphosphole ($\delta + 6.8$, $\Delta\delta + 13.5$ ppm). Other conjugating groups ($-\text{CHO}$, $-\text{COPh}$, $-\text{COOH}$) show similar effects. These results suggest that the net effect is again simply switching off the shielding of 3,4-dimethyl substitution, with no obvious conjugating effect being transmitted to phosphorus as might be associated with resonance forms such as (24) (Scheme 3).



Scheme 3

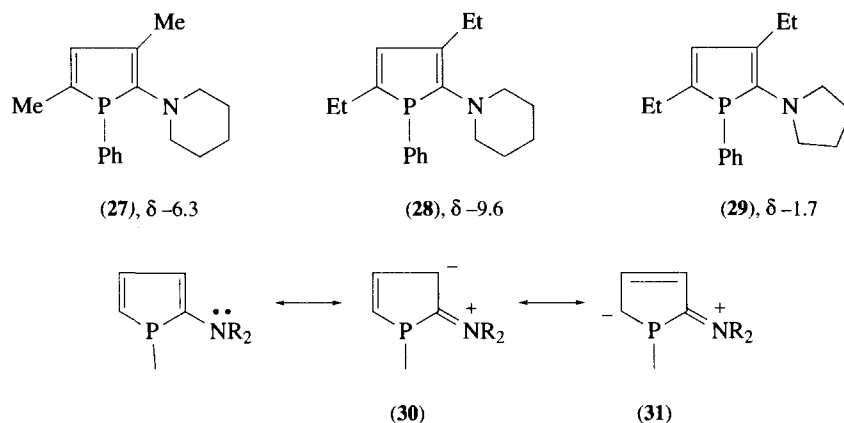
Substitution on 1-*t*-butyl-3,4-dimethylphosphole by 2-carboethoxy caused a net upfield shift of -6.8 ppm, and the same effect was observed for 2-acetyl ($\Delta\delta - 7.6$ ppm). The upfield shift may be more a consequence of steric influences from the bulky *t*-butyl group, and not indicative of a conjugative effect. Other ester derivatives are known ((25) and (26)) but are not useful in ascertaining conjugative effects without related model compounds.



(v) Effects from halo, silyl, and amino substituents

Only one C-halophosphole (2-bromo-3,4-dimethyl-1-phenyl) is known; its shift of $+10.2$ suggests a stronger deshielding than the switching off of the 3,4-dimethyl effect. This was true also when TMS replaced Br ($\delta + 15.3$).

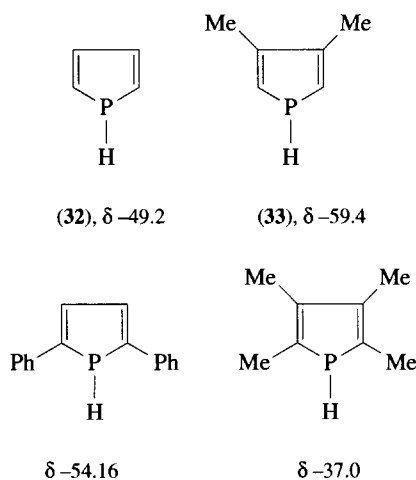
There are three 2-amino-substituted phospholes ((27), (28), and (29)), but with an unusual 2,3,5-substitution pattern that makes it difficult to assess the effect of the amino groups. There is a suggestion, however, of upfield shifting coming from this substituent; if one ignores the 5-methyl substituent of (27), then a comparison can be made to 3-methyl-1-phenylphosphole ($\delta + 7.5$). The shift of (27) is $\delta - 6.3$, thus suggesting an upfield shift of -13.8 ppm due to the amino group. It will be of interest to confirm this value with other aminophospholes, since a shift of this magnitude is of considerable importance; it suggests that there is strong electron release into the ring (resonance forms (30) and (31)) that in some way has an influence on phosphorus (Scheme 4). This point clearly deserves further study, as it may bear on the nature of electron delocalization in the phosphole ring (see Section 2.15.2).



Scheme 4

(vi) Replacement of methyl on P by hydrogen

In simple tertiary phosphines, replacement of one carbon substituent by hydrogen causes a very strong upfield shift, and this is seen also among phospholes. The shifts of *1H*-phospholes have been recorded at low temperatures (-90°C), since they rapidly rearrange by [1,5]H-shifts and then dimerize (see Section 2.15.5.8). Structures (32) and (33) also clearly show the operation of the 3,4-dimethyl shielding effect ($\Delta\delta -10.2$ ppm) in the *1H*-phospholes.

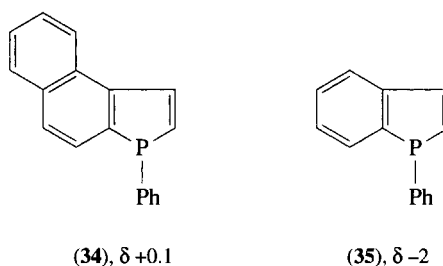


The ^{31}P - ^1H coupling constants for the *1H*-phospholes range from 207.3 to 233.1 Hz, which is within the range for typical secondary phosphines, although on the high side. This is suggestive of low *s*-character in the P—H bond [83JA6871].

(vii) Effects of benzo fusion

The fusion of a benzo group on the 2,3-positions of 1-phenylphosphole causes an upfield shift of 8.4 ppm, to $\delta -2$. The effect seems to be additive; the shift for dibenzophosphole ($\delta -10.2$) is an additional 8.2 ppm upfield. The shift for 1-*t*-butylphosphindole is also found in a relatively shielded position ($\delta +23.5$; compare to $\delta +40.5$ for 3-methyl-1-*t*-butylphosphole).

The only phosphole with a naphtho group fused to the *b*-face (34) has a shift of $\delta +0.1$, not surprisingly quite close to the value for 1-phenylphosphindole (35).



(viii) *Conversion to C.N.-4 and C.N.-5 species*

All conversions of phosphines to derivatives with C.N.-4 are accompanied by sizeable downfield shifts, and this is true of phospholes. Phosphole oxides are rare species, since they tend to dimerize unless sterically protected (see Section 2.15.6.1). Nevertheless, some data are available and collected in Table 5. The magnitude of the downfield shifting that takes place is not unlike that known for simple phosphine to oxide conversions. Phosphole sulfides and phospholium ions (both Table 5) also give downfield shifts of expected size. The 3,4-dimethyl shielding effect is evident in all of these classes on examining the tables.

Phospholes with C.N.-5 (phosphoranes) are quite rare, but give the highly characteristic upfield shifts always associated with this coordination state. Trioxyphosphoranes with the phosphole nucleus, for example, have shifts around $\delta -37$ (Table 5).

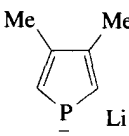
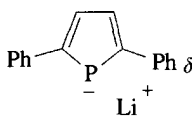
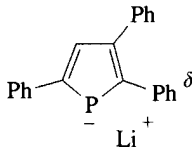
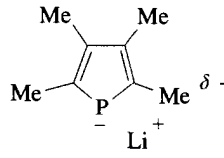
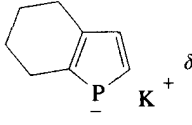
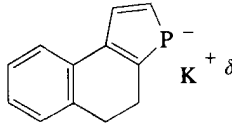
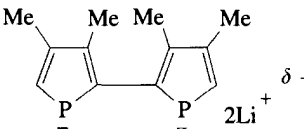
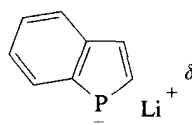
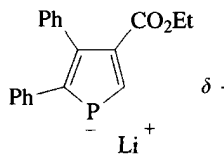
(ix) *Phospholide ions*

Dramatic downfield shifts accompany the cleavage of P-substituents with formation of phospholide ions. These shifts are considerably further downfield than those for simpler phosphide ions, and have been taken to be a reflection of strong electron delocalization in the phospholide ion (see Section 2.15.2). Data are collected in Table 6, where it will be seen that typical shifts fall in the region $\delta +70$ to $+100$. Once again the 3,4-dimethyl shielding effect is present and indeed is of the largest size ($\Delta\delta$ 21.4 ppm) of all families examined. Phospholide ions with this substitution have shifts in the range $\delta +50$ to $+60$.

(x) *Theoretical aspects*

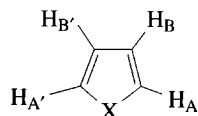
It was noted in Section 2.15.3.2.1 that the ^{31}P NMR signal for phospholes is generally downfield-shifted relative to model (unsaturated) phosphines. This effect was early attributed to cyclic delocalization in the phosphole ring, which could reduce the shielding effect by diminishing the contribution of the lone pair on phosphorus and give it more 4-coordinate character. However, later work has emphasized the low level of delocalization in phospholes that is occasioned by its pyramidal shape. New computational techniques have now made it possible to explore this point by calculating the ^{31}P NMR shifts for 1*H*-phosphole in its natural pyramidal form and in its forced planar form, where delocalization can be appreciable (see Section 2.15.2) <94JA9638>. The surprising result was obtained that the ^{31}P shifts were quite similar, differing by only 9.0 ppm, and thus speaking against any important role for delocalization in establishing the chemical shift. Furthermore, the calculations showed that a large downfield shift of 110.9 ppm should occur on removing the proton on phosphorus, just as is observed experimentally (126.4 ppm). However, this shift does not arise directly from the delocalization in the phospholide ion, but from the presence of the *second* lone pair on phosphorus which is oriented in the plane of the ring. It is this lone pair then that is the cause of the deshielding; it has an orbital energy value significantly closer to that of the HOMO–LUMO gap than does the lone pair in planar 1*H*-phosphole, which is perpendicular to the plane of the ring. This was shown to lead to a significant paramagnetic contribution to the net shielding. This strong deshielding effect was also calculated to be present (147.7 ppm) when pyrrole was deprotonated to form the pyrrolide ion; the reverse effect, shielding by 100 ppm, was found when a lone pair on thiophene was protonated to form the thiophenium ion.

Table 6 ^{31}P NMR shifts of phospholide ions^a.

Compound	Ref.
 $\delta + 77.2$	87TL5025
 $\delta + 55.8$	87TL5025
 $\delta + 78.8$	87TL5025
 $\delta + 102.5$	82JOM(231)361
 $\delta + 63.02$	87TL5025
 $\delta + 73.3$	79CC401
 $\delta + 56.7$	86JOM(316)271
 $\delta + 56.7$	86JOM(316)271
 $\delta + 40$	82PS(13)259
 $\delta + 116.2$	93BSF695

^aIn THF.**2.15.3.2.2 ^1H NMR spectroscopy**

Most of the compounds listed in Table 4 have also been characterized by ^1H NMR spectroscopy, and this table therefore stands as a source of references for obtaining detailed spectral information for particular phospholes.

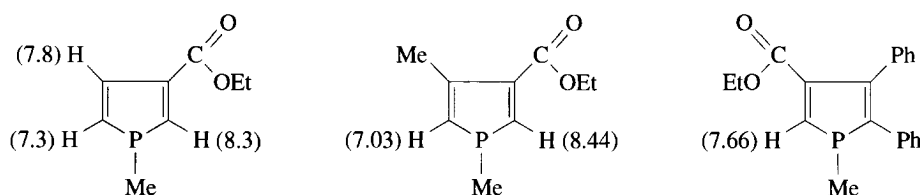
Table 7 Proton NMR spectra of *P*-substituted phospholes.

	$P-CH_3^a$	$P-Ph^b$	$P-H^b$	$(P-CH_2)_2^b$	$P-Li^+{}^b$
δH_A	(6.5–7.5)	6.94	7.10	6.76	6.78
δH_B	(6.5–7.5)	7.00	7.33	7.03	6.62
$^2J_{P-H_A}$	38.49	38.7	39.8	37.1	40.76
$^3J_{P-H_B}$	13.77	14.5	14.33	15.3	6.42
$^3J_{H_A-H_B}$	7.24	7.2	7.05	7.2	5.61
$^4J_{H_A-H_B}$	1.12	1.1	1.04	1.1	1.23
$^4J_{H_A-H_{A'}}$	3.01	2.7	2.78	2.8	3.56
$^3J_{H_B-H_{B'}}$	1.96	2.2	2.3	2.3	2.43

^a At 100 MHz. From calculated spectrum; error limits not shown (69JA3308). ^b (87TL5025).

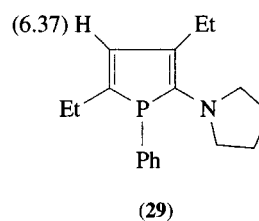
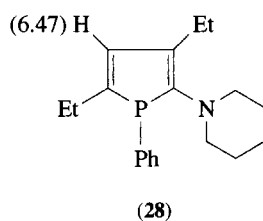
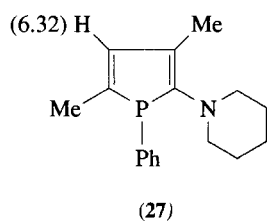
The ^1H NMR spectra of phospholes without C-substituents are of the AA'BB'X type and are quite complex. The first measurement was made on 1-methylphosphole at 100 MHz; the spectrum had to be interpreted with the aid of computer simulation (69JA3308). Later measurements at higher field gave spectra that agreed well with that for 1-methylphosphole (Table 7). The chemical shifts of the ring protons are of considerable interest, for they exhibit strong deshielding relative to model phosphines and appear in the same region as do the signals for thiophene. Also pointing to the strength of the downfield shifting is the observation that, in 1-benzylphosphole, the phosphole and phenyl proton signals actually overlap in the region δ 6.31–7.29 (73JOC1858). In thiophene, this downfield shifting is taken to imply the existence of an aromatic ring current, and this interpretation was applied to 1-methylphosphole in the 1969 study. However, later studies of phospholes revealed that the cyclic delocalization cannot be very extensive (see Section 2.15.2), and was considered to be *less* than that in thiophene. This apparent contradiction has not yet been satisfactorily explained; it cannot be attributed to experimental differences in the measurements of the two compounds as suggested by Palmer and Findlay (75JCS(P2)974).

Spectra for C-methyl phospholes are much easier to interpret since the differences in the chemical shifts of the ring protons are of significant size, and observed chemical shifts for the α - and β -protons (73JOC1858) are quite consistent with the values seen in Table 7. When the conjugating ester group is placed at the 3-position, the expected additional deshielding takes place (numerals are δ values) (73JOC1858, 93BSF695):

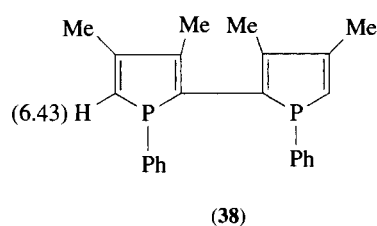
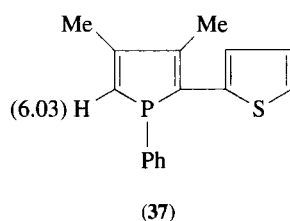
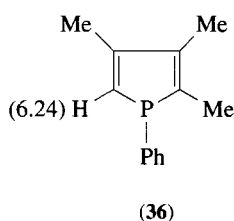


The effects of a conjugating group at the 2-position are not yet fully known, since all compounds of this type so far studied have methyls also at positions 3 and 4. Any shift effect at H-5 is not pronounced; typical shifts for 2-acetyl and 2-carboethoxy are in the range δ 6.5–6.75 (76T2395).

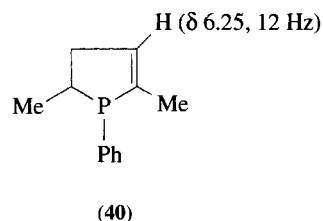
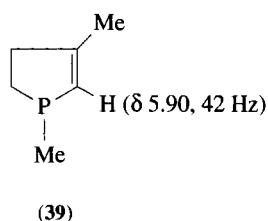
The effect of electron-releasing groups on the ring protons cannot yet be interpreted, due to the scarcity of structures available. There is a strong suggestion, however, that a 2-amino group introduces significant shielding at H-4. Assuming that the 3,5-dialkyl substitution in compounds (27)–(29) introduces no appreciable effect (not yet confirmable), the signal for H-4 in these amino derivatives falls in the range δ 6.32–6.47 (88TL4581), whereas the proton in this position in the unsubstituted phospholes has a signal around δ 7.0.



2-(2-Thienyl) derivatives of 3,4-dimethylphospholes (e.g. (37)) also may exhibit small shielding of H-5 relative to the 2,3,4-trimethyl compound (36). A 2-(2-phospholyl) substituent (38) <86JOM(316)271> shows the opposite effect.

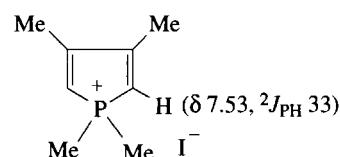
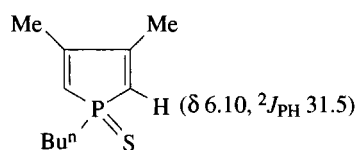


The ^{31}P - ^1H coupling constants for the 2- and 3-protons are typical of cyclic vinyl phosphines and while they are rather large they are not reflective of any special property of the phosphole ring system. For all of the monocyclic phospholes listed in Table 7, the $^2J_{\text{PH}}$ values for H-2 fall in the range 34–42 Hz; the $^3J_{\text{PH}}$ values for H-3 are around 12–17 Hz. These should be compared to the 2-phospholene model compounds (39) <71JOC1297> and (40) <B-81MI 215-03>, which have constants in the same ranges.



Methyl groups attached to the phosphorus atom (34) <B-81MI 215-03> typically have signals around δ 1.3–1.4, values noticeably more downfield than found for simple methylphosphines (generally δ 0.7–1.0) but not dissimilar to those for cyclic vinyl phosphines (e.g. 1,3-dimethyl-2-phospholene, δ 1.23). However, the introduction of a conjugating carbonyl substituent in the 3-position clearly leads to considerable additional deshielding at a P-methyl group; placement of a 3-carboethoxy group on 1-methylphosphole and on 1,3-dimethylphosphole leads to shifts of δ 1.92 and 1.96 respectively, for the P—Me group. The coupling constant for the ^{31}P interaction with the methyl group is frequently zero, and does not exceed 2 Hz. Such small $^2J_{\text{PH}}$ values are not unique to the phosphole ring, however; benzylic protons have signals around δ 3.0, and generally show no detectable coupling with ^{31}P .

It has been difficult to establish the effects on ^1H NMR spectra when phospholes are converted to derivatives with higher coordination numbers, since these species (especially oxides) are prone to dimerize (see Section 2.15.6). Those that are stable are multiply substituted, and the chemical shifts will be subject to modification by the substituents. Some structures that show the effects at the α -position are given below. The effects are in fact not great, both with regard to shifts and coupling to ^{31}P , although there is some diminution of the latter value <76T2305>.

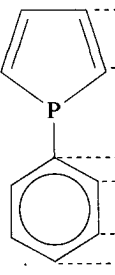
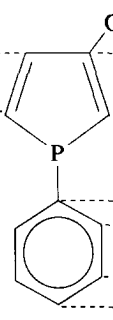
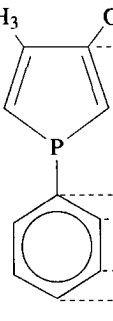


2.15.3.2.3 ^{13}C NMR spectroscopy

^{13}C NMR has been of great value in characterizing phospholes, and data are available for several of the compounds recorded in Table 4. Not only are the chemical shifts of value but so also are the ^{13}C – ^{31}P coupling constants. Influences on the magnitude of the coupling constant are frequently of steric origin, where the relative disposition of the carbon and the lone pair on phosphorus seems to be important (B-87MI 215-01).

Detailed considerations of the ^{13}C NMR spectra of phospholes (72TL3353, 80OMR(14)14) and of dibenzophospholes (80OMR(14)14, 87MRC774) have been published, and some correlations with the extent of cyclic delocalization in the ring system have been suggested. Thus, the 1972 paper proposed that, in a series of P-phenylphospholes, the relative extent of cyclic delocalization could be revealed by the chemical shifts of the *ipso* carbon of the phenyl ring, with delocalization increases associated with increased shielding of this carbon. Another indication of the degree of delocalization was said to be provided by the magnitude of $^1J_{\text{CP}}$ for C-2 of the ring, where an increase in this value correlated with an increase in delocalization. The data in Tables 8–10 show differences in the two parameters, but since the degree of electron delocalization in the compounds is not a measurable quantity by other techniques, the interpretation of these relations remains subjective. A useful observation of structural importance that came from the 1980 paper was the increase in shielding of a 3-methyl carbon when a second methyl was placed at C-4. The increase, while small (Table 9; 0.78 ppm in the P-phenyl compounds (42) and (43); 0.93 in the P-methyl compounds (45) and (46)) may be associated with the operation of the γ -shielding effect which arises from steric proximity of the groups.

Table 8 ^{13}C NMR of some P-phenylphospholes.^{a,b}

	
(41)	(42)
	
(43)	

^a Values in parentheses are J_{PC} , in Hertz. ^b Refs: (41) (72TL3353); (42) and (43) (80OMR(14)14).

The representative spectra presented in Tables 8–10 reveal some other characteristics of phosphole spectra:

- (i) In C-unsubstituted compounds, there is little difference in the shifts of the α - and β -carbons; coupling constants are larger for the latter, which aids in assignments.

Table 9 ^{13}C NMR of some P-alkylphospholes.

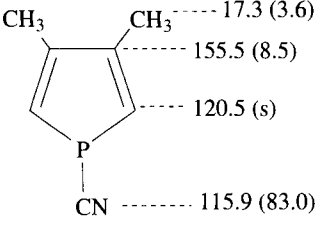
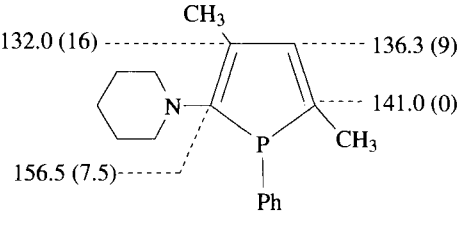
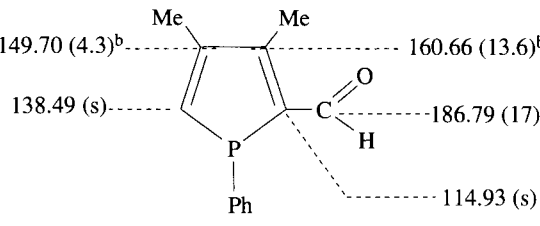
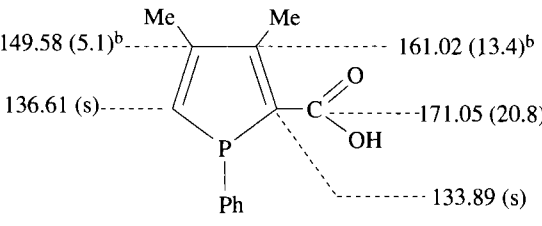
(44)^c	(45)^c
(46)^c	(47)^c
(48)^c	

^aValues in parentheses are J_{PC} , in Hertz. ^bThese assignments require confirmation. ^cRefs: (44) and (45) <73OMR(5)161>; (46), (47) and (48) <80OMR(14)14>.

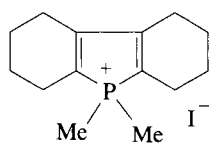
- (ii) Coupling constants (both ^1J and ^2J) are much larger for exocyclic carbons than for endocyclic carbons. The ^2J values are controlled by the relation of the lone pair with respect to the coupled carbon <B-87MI 215-01>. The pyramidal shape at phosphorus has the effect of placing the lone pair close to a 2-substituent, a relation giving strong coupling (compound (44), 22 Hz for 2-Me). This effect operates on substituents attached to P also (compound (47), 22 Hz for C-2 of *n*-butyl; compounds (41), (42), and (43), 18–19 Hz for the *ortho*-phenyl carbon).
- (iii) ^1J to an exocyclic carbon seems to be quite variable, and not obviously related to the structure. Thus, P-methyls have values consistently of 17–20 Hz, but the value for a *t*-butyl substituent is 8.6 Hz. For phenyl carbons, values range from –8.63 to 12.2 Hz. A very large value was observed for a P-cyano substituent (83.0 Hz).

As in the case of ^1H NMR of phospholes, few model compounds are available to assess the effect of a change in coordination number on ^{13}C NMR spectra. However, some striking changes do take place; the β -carbon in sulfides and salts is strongly deshielded, and the coupling constants to ^{31}P are greatly increased, especially at the α -carbon. None of these effects are unique to phospholes, however, and are known also for derivatives of other cyclic, α,β -unsaturated phosphorus compounds in these coordination states <B-81MI 215-02>. Compound (53) illustrates these spectral effects <78CJC1952>.

Table 10 ^{13}C NMR of some phospholes with functional groups.^a

 (49)^c	 (50)^c
 (51)^c	 (52)^c

^aValues in parentheses are J_{PC} , in Hertz. ^bCould be reversed. ^cRefs: (49) <88OM1796>; (50) <88TL4581>; (51) and (52) <92BSF486>.



(53)

$\beta\text{-C}$: δ 157.1, $^2J_{\text{PC}}$ 22 Hz
 $\alpha\text{-C}$: δ 122.9, $^1J_{\text{PC}}$ 79.2 Hz

2.15.3.3 Mass Spectrometry

Following the various NMR techniques in value for structural characterization is mass spectrometry. It was early recognized <69JA3308> that the phosphole ring was of good stability under electron impact; the molecular ion (m/z 98) of 1-methylphosphole was the most abundant peak (R.A. 100), followed closely by $M^+ - 1$ (R.A. 98). The ring was retained in some other fragments (m/z 83, $M^+ - \text{Me}$, R.A. 47; m/z 96, $M^+ - 2$, R.A. 26). Ring fragmentation with loss of ethyne accounted for a peak at m/e 72 ($\text{C}_3\text{H}_3\text{P}^+$, R.A. 11). Numerous later studies have confirmed that the base peak is usually the molecular ion in a variety of compounds (e.g. in methyl 1-methylphosphole-3-carboxylate <73JOC1858>; in 1-cyano-3,4-dimethylphosphole <88OM1796>; in 1-phenyl-2,3,4-trimethylphosphole <94OM925>; in ethyl 2,3-diphenyl-1-methylphosphole-4-carboxylate <93BSF695>; in 2-acetyl-3,4-dimethyl-1-phenylphosphole <76T2395>; in 3,4-dimethyl-1-phenyl-2-(2-thienyl)phosphole <90JOC2494>; in bis(3,4-dimethyl-1-phenyl-2-phospholyl) <86JOM(316)271>; in 3,5-dimethyl-1-phenyl-2-piperidinophosphole <88TL4581>; in 2-bromo-3,4-dimethyl-1-phenylphosphole <92BSF486>; and in others). A notable exception occurs with P-benzyl compounds, where the familiar cleavage of benzyl with formation of the C_7H_7^+ ion accounted for the base peak, and the molecular ion had R.A. 39% <70JA5779>. Certain carbonyl derivatives are also exceptional in giving base peaks from fragmentation of this function: 3,4-dimethyl-2-ethoxycarboxyl-1-phenylphosphole, M^+ (65%), $M^+ - \text{C}_2\text{H}_4$ (100%), $M^+ - \text{C}_2\text{H}_4 - \text{CO}$ (53%), $M^+ - \text{C}_2\text{H}_4 - \text{CHO}$ (75%) <92BSF486>; 2-benzoyl-3,4-dimethyl-1-phenylphosphole, M^+ (6%), $M^+ - \text{PhCO}$ (100%) <92BSF486>.

2.15.3.4 Ultraviolet Spectroscopy

Simple P-alkylphospholes possess a unique absorption band, of moderate intensity, in the region 280–290 nm. First noted with 1-methylphosphole (λ_{max} 286 nm in isoctane, $\log \epsilon$ 3.89, <69JA3308>),

this band cannot be associated either with the diene component of the ring, or with the phosphine unit, considered separately, since neither have absorption bands near this region. The band is clearly descriptive of the phosphole ring system, but its electronic origin, and relevance to delocalization phenomena, remain to be evaluated. Other phospholes have also given this absorption; some representative data are given in Table 11.

As noted in this table, P-phenylphospholes also give characteristic absorption, and the 1,2,5-triphenyl derivative absorbs at such long wavelength (372 nm) that it is yellow. It also has the rare property of exhibiting triboluminescence <89MI 215-01>, and a reversible color change (yellow to red) on boiling in CCl₄. This has been attributed to a reversible formation of a phosphole trichloromethide salt <82CJC138>.

Table 11 UV spectra of some phospholes.

Phosphole	Solvent	λ_{max}	$\log \epsilon$	Ref.
1-Me	isooctane	286	3.89	69JA3308
1-PhCH ₂	95% EtOH	286	3.56	73JOC1858
1-PhCH ₂ CH ₂	95% EtOH	284	3.93	73JOC1858
1-PhCH ₂ -3,4-diMe	95% EtOH	280	3.26	73JOC1858
1-Me-3-COOMe	C ₆ H ₁₂	307		73JOC1858
1,4-diMe-3-COOMe	C ₆ H ₁₂	300		73JOC1858
1,2,5-triPh	EtOH	372, 221	4.26, 4.37	74PS(4)279
2-MeCO-3,4-diMe-1-Ph	MeOH	211.5	4.12	76T2395

2.15.3.5 Infrared Spectroscopy

Little use has been made of the IR spectra of phospholes. A few reports exist on such spectral measurements, but primarily they have been made for the purpose of characterizing functional groups on the ring. From the spectrum reported for methyl 1-methylphosphole-3-carboxylate (ν_{CH} 3110, $\nu_{C=C}$ 1535, $\nu_{C=O}$ 1250 and 1186, unassigned at 1075, 1062, 888, 793, 740, 700 cm⁻¹ <73JOC1858>), it is deduced that the ring double bonds absorb around 1535 cm⁻¹, although in this model compound the conjugating group should introduce as usual a shift of the double bonds to lower frequency. In 1-cyano-3,4-dimethylphosphole <88OM1796>, ν_{CN} has been reported at 2160 cm⁻¹, which is at the low frequency end of the C—CN range but like that of S—CN (2170 cm⁻¹). In 2-acetyl-3,4-dimethyl-1-phenylphosphole $\nu_{C=O}$ appeared at 1655 cm⁻¹ <76T2395>.

2.15.3.6 Photoelectron Spectroscopy

The photoelectron spectrum of 1-phenylphosphole contains bands of equal intensity at 8.45 and 9.25 eV <73AG(E)145>; other phospholes give similar bands <76JA407>. The theory of these signals has been discussed and interpreted in various ways, and they have figured in discussions favoring the view that phospholes can be considered as "aromatic" <76JA4365>, as being non-aromatic <75JCS(P2)974>, and as having a form of hyperconjugative delocalization involving the σ -orbital of the exocyclic P-substituent <76JA407>. A reinterpretation of the data would be timely, since there is fresh interest in the nature of bonding in phospholes (see Section 2.15.2).

2.15.3.7 Dipole Moments

The dipole moments (in D units) of a few phospholes have been determined <72CR(C)100>, with the following results: 1-*n*-butyl, 1.23; 1-*n*-butyl-3,4-dimethyl, 2.00; 3-methyl-1-phenyl, 1.39; 3,4-dimethyl-1-phenyl, 1.52; 1,2,5-triphenyl, 1.45. These values are close to those found for saturated phosphines (e.g. 1-*n*-butylphospholane, 1.93; 1-phenylphospholane, 1.52). The negative end of the dipole in the phospholes is the phosphorus atom, which is the same as found for thiophene and the opposite of the case for pyrrole <72JHC817>.

2.15.3.8 Electron Spin Resonance Spectroscopy

The only application of this technique in phosphole chemistry has been in the detection of radical intermediates in the cleavage of P-substituents with alkali metals at -80°C . Radical anions at phosphorus were formed as the initial products from 1-methyl-2,5-diphenylphosphole, 1,2,5-triphenylphosphole, and pentaphenylphosphole. The ^{31}P nucleus exhibited strong coupling with the electron $\langle 71\text{T}5705 \rangle$. Radical anions were also detected in the reaction of 1,2,5-triphenylphosphole oxide and pentaphenylphosphole oxide with alkali metals $\langle 71\text{CC}782 \rangle$.

2.15.4 THERMODYNAMIC ASPECTS

2.15.4.1 Physical Properties

In general, the simple phospholes are malodorous, colorless materials that require great care in handling because of their ease of oxidation. Those with more complex structures, especially where conjugating substituents are present, are less volatile and oxidizable, and can be relatively inoffensive. These characteristics figure prominently in the design of experiments to determine physical and other properties of phospholes.

Most of the simple phospholes are liquids that can be distilled. Distillations must be conducted under air-free conditions; generally reduced pressure is used to avoid thermal decomposition. Some typical b.p. ($^{\circ}\text{C}$) are: 1-Me, 83–85 $\langle 69\text{JA}3308 \rangle$; 1,3,4-triMe, 60 at 15 mm Hg $\langle 72\text{OMR}(4)171 \rangle$; 1-benzyl, 71–72 at 0.2 mm Hg, m.p. 34–34.5 $^{\circ}\text{C}$ $\langle 73\text{JOC}1858 \rangle$; 1-Ph, 55 at 0.1 mm Hg $\langle 81\text{S}983 \rangle$; 3,4-diMe-1-Ph, 78 at 0.1 mm Hg $\langle 81\text{S}983 \rangle$; 1-cyanoethyl-3,4-diMe, 80 at 0.1 mm Hg $\langle 76\text{CJC}2402 \rangle$; Me 1,4-diMe-phosphole-3-carboxylate, 67 at 0.3 mm Hg $\langle 73\text{JOC}1858 \rangle$; 1-Me-cyclohexano[b]-phosphole, 60 at 0.03 mm Hg $\langle 82\text{PS}(12)161 \rangle$; 1-Ph-2,3,4,5-tetraMe, 105–110 at 0.5 mm Hg $\langle 88\text{OM}921 \rangle$; 1,2-diPh-5-isopropyl, 100–105 at 0.025 mm Hg, m.p. 73–76 $^{\circ}\text{C}$ $\langle 71\text{JA}6205 \rangle$; 1-cyano-3,4-diMe, 80 at 20 mm Hg, m.p. 52 $^{\circ}\text{C}$ $\langle 88\text{OM}1796 \rangle$.

As noted in the above listing, a few simple phospholes are liquids at room temperature, but those with higher molecular weight are generally solids (m.p. $^{\circ}\text{C}$): 1,2,5-triPh, 184–186 $\langle 67\text{AG}(E)86 \rangle$; 2,2'-bis(3,4-diMe-1-Ph-phospholyl) $\langle 86\text{JOM}(316)271 \rangle$; Me 3,4-diMe-1-Ph-phosphole-2-carboxylate, 61–63 $\langle 80\text{CSC}905 \rangle$; 1-Ph-phosphindole, 66–68 $\langle 72\text{CC}496 \rangle$; 1,2-diPh-3-*n*-Bu-benzophosphindole, 84–84.5 $\langle 67\text{JA}5732 \rangle$; 1-Ph-dibenzophosphole, 93–94 $\langle 53\text{LA}(580)44 \rangle$.

In general, unless polar substituents are present, phospholes are to be viewed as rather non-polar substances, and show little or no solubility in water. They do have good solubility in most common organic solvents. Frequently used for extraction purposes are chloroform, methylene chloride, benzene, toluene, and others. Reactions such as cleavage of P-substituents with metals are usually performed in THF. Simple P-alkyl and P-phenylphospholes are soluble in alkanes, and some 2-aminophospholes are soluble in ether–petroleum ether. Examples of systems used for the crystallization of phospholes include: CH_2Cl_2 for 3,4-dimethyl-1-phenyl-2-carboxylic acid $\langle 92\text{BSF}486 \rangle$; aqueous MeOH for methyl 3,4-dimethyl-1-phenyl-1-phosphole-2-carboxylate $\langle 80\text{CSC}905 \rangle$; chloroform for 1,2,5-triphenylphosphole $\langle 65\text{JCS}2184 \rangle$.

Liquid chromatography on silica gel has been employed in the isolation and purification of a number of less volatile and less easily oxidized phospholes. Some solvent systems employed include: toluene for 3,4-dimethyl-1-phenyl-2-carbaldehyde $\langle 92\text{BSF}486 \rangle$; hexane– CH_2Cl_2 for a 2,2'-biphospholyl $\langle 86\text{JOM}(316)271 \rangle$; ether–petroleum ether for some 2-aminophospholes $\langle 88\text{TL}4581 \rangle$; toluene–hexane (1 : 4) for 1-methyl-2,3,5-triphenylphosphole $\langle 82\text{JOC}2376 \rangle$; hexane–ether (95 : 5) for 1-phenyl-2-(2-thienyl)-3,4-dimethylphosphole $\langle 90\text{JOC}2494 \rangle$, 2,5-bis(2-phospholyl)thiophene $\langle 91\text{HAC}377 \rangle$, and 2-bromo-3,4-dimethyl-1-phenylphosphole $\langle 92\text{BSF}486 \rangle$; pentane– CH_2Cl_2 for 1-phenyl-2,3,4-trimethylphosphole $\langle 94\text{OM}925 \rangle$.

3-Methyl-4,5-dihydro-(3*H*)-benzo[e]phosphindole was chromatographed on alumina (neutral) with benzene as eluent $\langle 82\text{PS}(12)161 \rangle$; Florisil with pentane as eluent was used for 1-isopropyl-2-methyl-5-phenylphosphole, and also for a phosphindole $\langle 71\text{JA}6205 \rangle$.

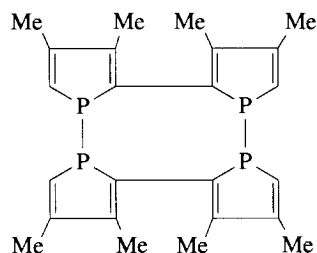
The high-pressure liquid chromatography technique does not appear to have been useful in phosphole isolation or purification. However, the more volatile phospholes can safely be subjected to gas chromatography. For 1-methylphosphole, a column of silicone oil SE-30 at 50 $^{\circ}\text{C}$ was used $\langle 69\text{JA}3308 \rangle$; the 3-carboxymethyl derivative required a temperature of 150 $^{\circ}\text{C}$ on this column $\langle 73\text{JOC}1858 \rangle$. In this paper, C-methyl phospholes were chromatographed on OV-17 at 100–125 $^{\circ}$.

Conversion of phospholes to derivatives with coordination number 4 completely changes their

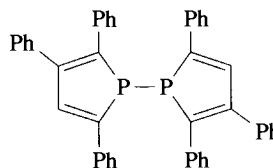
properties. As noted in Section 2.15.6, such derivatives frequently dimerize, even on formation. Those that remain monomeric are invariably more polar than the phosphole, and are non-distillable but chromatographable solids, recrystallizable from common solvents when thermally stable.

2.15.4.2 Thermal Stability

1*H*-Phosphole and some of its C-substituted derivatives are stable only at low temperatures (-70 to -90°C), rapidly isomerizing at higher temperatures and then undergoing dimerization (see Section 2.15.5.8). Replacement of H by methyl or phenyl gives the common phospholes that are stable for considerable periods at room temperature, and as noted above are distillable. Placement of a carboalkoxy group at the 3-position decreases the stability, and a distilled sample turned into a solid brown mass after a few hours at room temperature $\langle 73\text{JOC}1858 \rangle$. Prolonged thermal treatment of phospholes produces profound chemical change. Some (e.g., 1-benzyl-3,4-dimethylphosphole in refluxing xylene for 25 h) form insoluble, unidentifiable solids $\langle 73\text{JOC}1858 \rangle$; 1,3-dimethylphosphole, on the other hand, in part undergoes Diels–Alder dimerization on heating at 120°C for 17 h in xylene $\langle 85\text{PS}(25)117 \rangle$. Heating 3,4-dimethyl-1-phenylphosphole at temperatures around 150 – 170°C leads to rearrangement to a 2*H*-isomer, as in the case of 1*H*-phospholes, which then dimerizes (see Section 2.15.5.9). When the phosphole is heated at 170°C for a prolonged period, a tetramer (**54**) is formed along with other products $\langle 82\text{JA}2077 \rangle$. A different reaction course is followed by 1,2,5-triphenylphosphole; at 230°C for 10 days, a 1,1'-biphospholyl (**55**) is formed, probably via a 2*H*-phosphole intermediate.



(54)



(55)

Evident in the above discussion is the great instability of the 2*H*-phosphole ring; this structure lacks any stabilization by cyclic delocalization, and as true of the $\text{C}=\text{P}$ double bond in general (except those with special stabilizing substituents) is prone to dimerize upon formation. The 2*H*-phosphole ring system can be stabilized, however, in some specialized forms that are described in Section 2.15.7.1.

2.15.4.3 Basicity of Phospholes

A significant property of phospholes is their reduced basicity relative to other types of cyclic phosphines. This was first noticed in work on 1-methylphosphole, where it was found that some phospholene impurities could be selectively extracted from a pentane solution of a phosphole with 2 N HCl $\langle 69\text{JA}3308 \rangle$. With 6 N HCl, the phosphole was extracted from the pentane, but could not be recovered on neutralization; the products were unrecognizable. The $\text{p}K_{\text{a}}$ of 1-methylphosphole was then determined to be approximately 0.5 by following the changes in the strength of the UV maximum at 284 nm in various concentrations of HCl. This $\text{p}K_{\text{a}}$ is approximately 5 units lower than might be expected for a divinyl phosphine, implying that the ring structure has a profound effect on basicity at phosphorus. This property is in common with that of pyrrole and the first interpretation given to this property was that the phosphole system was similarly highly delocalized. As other evidence accumulated over the years that the system probably has rather weak delocalization (see Section 2.15.2), the low basicity seems to require another explanation. A possible one is that the small internal $\text{C}-\text{P}-\text{C}$ bond angle (about 90°) imposed by the ring structure is probably retained in the protonated form, which however is sp^3 -hybridized, thus creating an energy barrier to the protonation $\langle 78\text{JCS}(P)98 \rangle$. It has also been suggested that the low basicity arises from the possible degeneracy of the n - and highest π -orbitals $\langle \text{B-90MI } 205\text{-01} \rangle$.

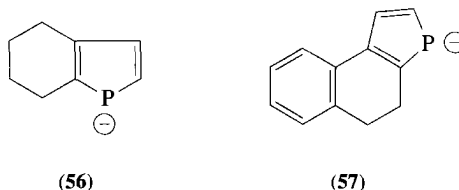
The site of protonation of phospholes in nonaqueous solvents by dry HCl or CF₃SO₃H has been found to be the phosphorus atom when the reaction is conducted at -90°C <86JCS(P2)629>; in pyrroles it is a ring carbon that undergoes protonation. The 1*H*-phospholium ions are not stable with Cl[−] as counterion, and consequences of the P-protonation by HCl are discussed in Section 2.15.5.3. When the counterion is nonnucleophilic, the 1*H*-phospholium ions are sufficiently stable to be directly observable <71JHC877, 72CJC1008>.

2.15.4.4 Resonance Energies

Missing in phosphole chemistry are adequate thermochemical measurements that would provide experimental determinations of resonance energies. The only thermochemical measurements so far reported were made on pentaphenylphosphole and its oxide; heats of combustion were -4349.17 ± 3.4 and -4308.26 ± 5.2 kcal mol^{−1}, respectively <62JCS2932>. Heats of formation were calculated to be $+93.7 \pm 3.4$ and $+52.8 \pm 5.2$, respectively. It was possible to determine the bond dissociation energy *D* (P=O) for the oxide, which was found to be 100.4 ± 9.5 kcal mol^{−1}. This value is significantly smaller than those for triphenylphosphine oxide (128.4 ± 5.5 kcal mol^{−1}) and for trimethylphosphine oxide (139.3 ± 3.0 kcal mol^{−1}). It was concluded that the difference of 39 ± 13 kcal mol^{−1} between *D* (P=O) for trimethylphosphine oxide and pentaphenylphosphole oxide gave an indirect measure of the conjugation energy of the system. At the time, this value seemed acceptable as an indicator of quite high resonance energy in the phosphole ring. Unfortunately, pentaphenylphosphole is a very poor model for studies of the properties of the parent phosphole ring, and as noted in Section 2.15.2, over the years the view has developed that the resonance energy is probably rather small, perhaps less than that in furan. This is now being substantiated by application of modern sophisticated computational techniques to phospholes. Some calculated values for 1*H*-phosphole with pyramidal phosphorus are: 12.9 kcal mol^{−1} <75JCS(P2)974>, 12.23 kcal mol^{−1} <88JA4204>, and 13.18 kcal mol^{−1} <94JA9638>. The latter two values were derived from isodesmic bond separation methods, used for the first time in studies of phosphole.

2.15.4.5 Basicity of Phospholide Ions

Compared to noncyclic phosphide ions, phospholide ions show a definite decrease in basicity that is consistent with the high degree of electron delocalization evident from other properties (see Sections 2.15.2 and 2.15.3.2). The ions (56) and (57) in THF solution were found not to be protonated by ethanol, implying an acidity for the conjugate acid (the 1*H*-phosphole) greater than that of ethanol, *pK*_a 15.9 in water <79CC401>.



No other measure of the basicity of phospholide ions has yet been published. For comparison, the *pK*_a of diphenylphosphine is 21.7.

2.15.5 REACTIVITY OF C.N.-3 PHOSPHOLES

2.15.5.1 General

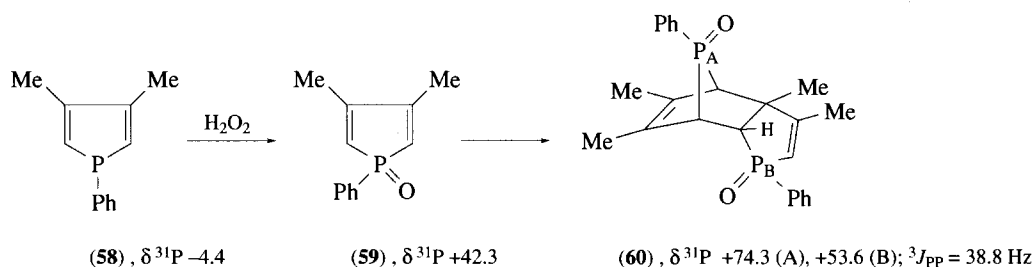
Studies of the reactivity of phospholes have revealed that the system behaves very differently from the related S, N, and O ring systems. In the first place, all attempts at performing electrophilic

substitution of the ring protons, a commonplace process for the other systems, have so far resulted in failure; attack occurs instead at the nucleophilic phosphorus atom. This is also a site of reactivity when Diels–Alder reactions are attempted with certain alkynic dienophiles, although the expected cycloaddition to a diene moiety can be realized. In fact, it is quite common for the phosphole ring to be destroyed in its reactions rather than preserved in the characteristic way of the related heterocyclic systems. However, these structural modifications are of considerable value as a source of other phosphorus heterocyclic systems. Another differentiating property of phospholes, and of the related phosphole anion (phospholide), is that an extraordinarily rich chemistry based on metal coordination complexes of various types has developed. In short, the phosphole ring, while disappointing in not giving some of the expected reactions of a truly cyclically delocalized system, has nevertheless displayed its own unique reactivity that has not only led to fascinating results for the researcher but also to the creation of some quite novel heterocyclic phosphorus compounds.

A point of caution needs to be made in interpreting phosphole chemistry: there are distinct differences in reactivity that are imposed by the pattern of substituents, so much so that a study made with one phosphole as a model may give specific results that are not extendable to other phospholes. In the early days of phosphole chemistry, 1,2,5-triphenylphosphole was frequently the model used because it was readily available. It is, in fact, a rather poor model. Even the placement of methyl groups on the 3 and 4 positions can have significant effects on reactivity at phosphorus. An excellent example of the influence of structure on the outcome of a reaction is seen in Section 2.15.5.5.1 for the case of addition of diethyl acetylenedicarboxylate.

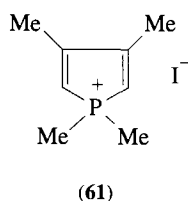
2.15.5.2 Reactions on Phosphorus with Conversion to C.N.-4

Some of the expected simple reactions of phosphines occur also with phospholes. Phosphorus can be oxidized with common oxidizing agents to form P-oxides, but with few exceptions these are unstable, usually spontaneously so, and give crystalline dimers (see Section 2.15.6.1). Intermediate stability was exhibited by 3,4-dimethyl-1-phenylphosphole (**58**) which when oxidized with *t*-butyl hydrogen peroxide at room temperature gave an oxide (**59**) detectable by ^{31}P NMR (δ +42.3). However, dimer (**60**) begins to form in a few hours and after several days is the only product that can be observed (Scheme 5) <91HAC359>. Pentaphenylphosphole <61JA4406> and 1,2,3-triphenylphosphole <65JCS2184> give oxides that can be isolated and preserved. Sulfides are more stable; they are prepared by direct sulfuration of phospholes (see Section 2.15.6.2).

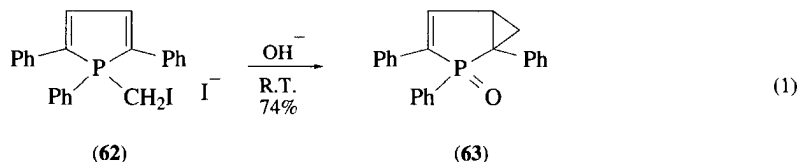


Scheme 5

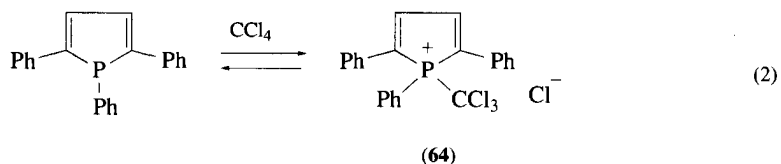
Simple alkyl halides give crystalline quaternary salts, although these seem to form more slowly than those of other cyclic phosphines <69JA3308>. They too will dimerize on formation or on heating <73JOC1954> but less readily so than oxides, so that some simple phospholium salts (see Section 2.15.6.3) are known (e.g. (**61**), <73JOC1858>).



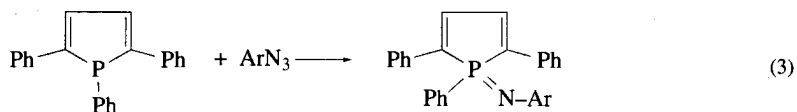
Methylene iodide gives an iodomethyl salt (**62**) with 1,2,5-triphenylphosphole which is of interest because it reacts with base to undergo rearrangement to the homophosphole oxide (**63**) (Equation (1)) <71CJC879>. The mechanism was not confirmed.



It has also been noted that, while stable at room temperature, a solution of triphenylphosphole in carbon tetrachloride undergoes a distinct color change (yellow to red) which is reversible on cooling <82CJC138>. Benzotrichloride also reacts on heating but the color change is not reversible. The phosphorus species formed in these reactions are not fully understood, but may stem from an initially formed phospholium salt, for example (**64**) (Equation (2)).

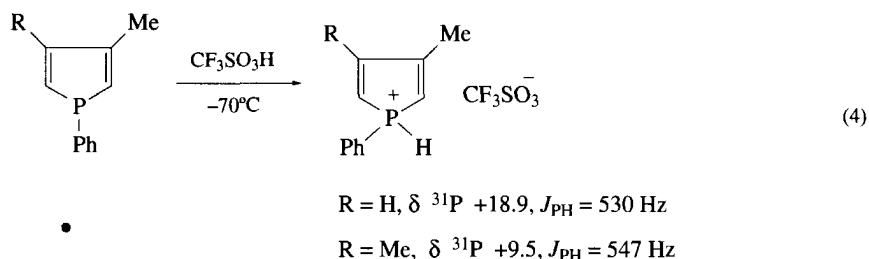


Azides have been found to attack the phosphorus atom of 1,2,5-triphenylphosphole to give imino derivatives in a version of the well-known Staudinger reaction of phosphines <72CC1242>. Yields of the crystalline imines (see also Section 2.15.6.4) are in the range 40–95%.

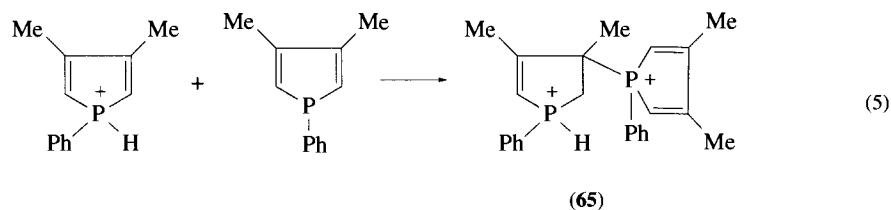


2.15.5.3 Protonation of Phospholes

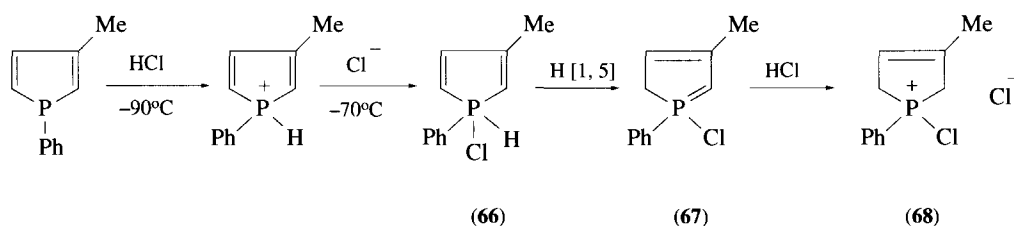
1-Alkylphospholes have been shown to be much weaker bases than other tertiary phosphines; a pK_a of 0.5 was recorded for the 1-methyl derivative <69JA3308>. The site of proton attachment in nonaqueous systems has been investigated <86JCS(P2)629> and it was conclusively shown that if the protonation is allowed to occur at low temperature (-70°C) and with an acid whose anionic part is nonnucleophilic (e.g. $\text{CF}_3\text{SO}_2\text{OH}$) the proton attaches to phosphorus to form a 1*H*-phospholium ion. These ions are quite stable up to about -30°C , but decomposition occurs at room temperature (Equation (4)).



The ions were found to react with phosphole in the solution to form a dimer-like structure (**65**) (Equation (5)).



With an acid whose anionic part has some nucleophilic character, such as HCl, a secondary reaction occurs. The P-protonation in CD_2Cl_2 at -90°C can be detected by ^{31}P NMR but on warming to -70°C the P—H form disappears and a signal for the 1-chloro-2,5-dihydrophospholium ion (**68**) appears. This substance is the same as formed in the McCormack cycloaddition of dienes and RPCl_2 (see Section 2.15.8.2) and is easily recognized (Scheme 6).

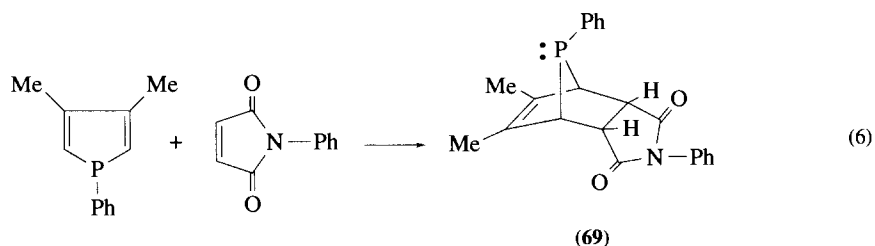


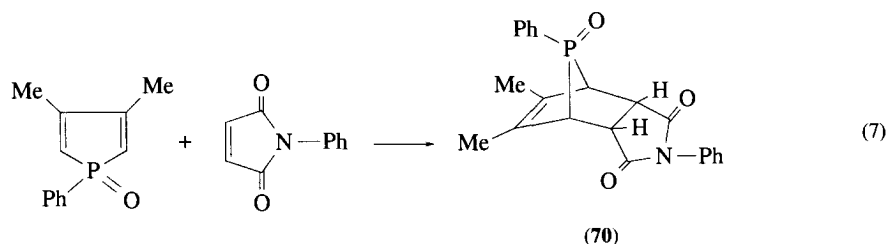
Scheme 6

Apparently the nucleophilic chloride ion first added to the phospholium ion to form a five-coordinate species (**66**), which undergoes a [1,5] sigmatropic hydrogen shift to give a 2*H*-phosphole derivative (**67**), then adding HCl. Such [1,5] shifts have been encountered in other types of phosphole reactions and are discussed in Section 2.15.5.9. In an earlier study $\langle 81\text{PS}(12)161 \rangle$ protonation of phospholes with dry HCl had been performed at room temperature; this formed immediately the chlorodihydrophospholium ion, and was considered at that time to indicate that carbon was the site of protonation. On the other hand, 1,2,5-triphenylphosphole is not protonated when dry HCl is passed into a benzene solution, although a stabilized 1*H*-phospholium TaCl_6^- salt can be obtained when HCl is passed into a solution of the TaCl_5 complex of the phosphole $\langle 71\text{JHC}877 \rangle$. Protonation of 1,2,5-triphenylphosphole by triflic acid also occurs on P $\langle 83\text{JOM}(256)103 \rangle$.

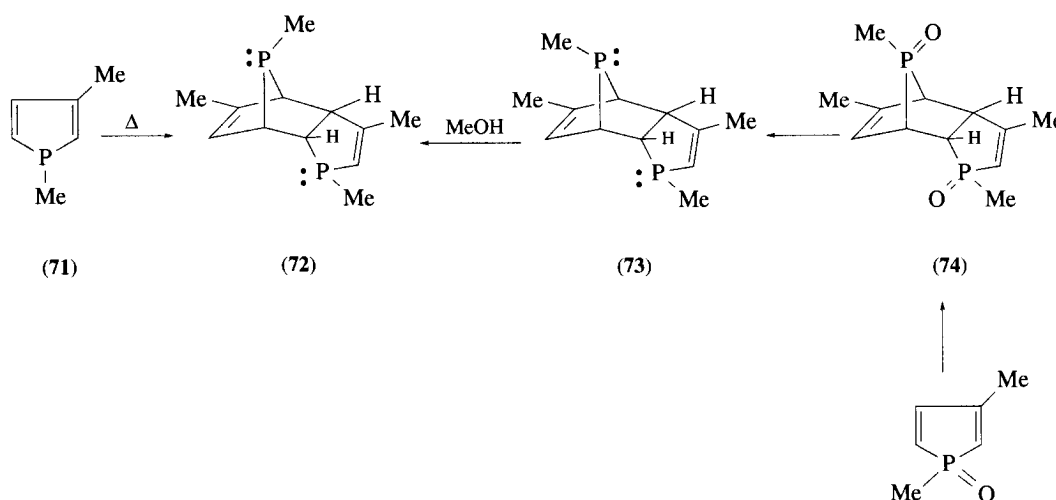
2.15.5.4 Cycloadditions with the Diene Moiety

The first successful Diels–Alder reaction of a phosphole was accomplished with the 3,4-dimethyl-1-phenyl derivative, which gave the crystalline cycloadduct (**69**) with *N*-phenylmaleimide (50%) (Equation (6)) $\langle 81\text{TL}319 \rangle$. The structure at phosphorus is of interest in this adduct; the phenyl substituent is *anti* to the double bond, while in cycloadditions of the corresponding phosphole oxide with the same dienophile the structure (**70**) with *syn* phenyl is the exclusive product (Equation (7)) $\langle 91\text{HAC}359 \rangle$. In reactions of both systems, only the *endo* fused product is formed; these stereochemical points have been discussed fully $\langle 90\text{RHA}39 \rangle$.





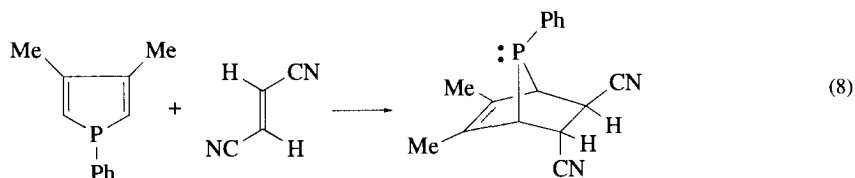
Another example of a Diels–Alder reaction giving the *anti* isomer is seen in the dimerization of phosphole (71) [85PS(25)117]. This occurred on heating in xylene solution at 120°C for 17 h. The dimer (72) was observed by ^{31}P NMR spectroscopy; it was not isolated but has been made indirectly by isomerization at P of the *syn* dimer (73), made from the oxide dimer (74) (Scheme 7).



Scheme 7

As noted in Section 2.15.9, however, thermal treatment of phospholes can also lead to other products, and the simple dimerization process is probably of little value. The dimer framework is much more easily formed from phosphole oxides.

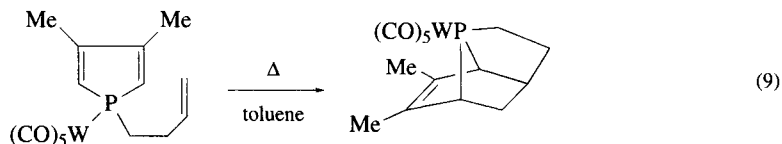
An important new development is the demonstration that Diels–Alder reactions with phospholes and fumaronitriles are greatly accelerated under pressure, and this has led to the synthesis of a number of new 7-phosphanorbornene derivatives. Thus, 3,4-dimethyl-1-phenylphosphole reacted with fumaronitrile at 30°C under 9 kbar for 24 h to give an adduct in 67% yield (Equation (8)) [89T7083]. The stereochemistry at phosphorus was confirmed by x-ray analysis.



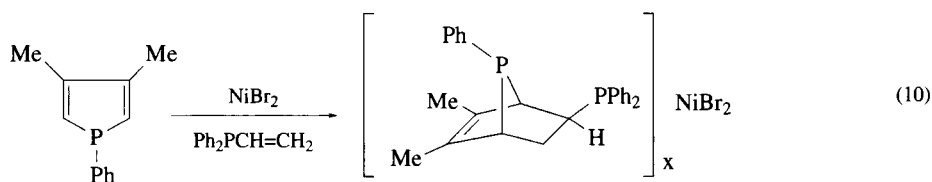
1-Phenylphosphole and 3-methyl-1-phenylphosphole also gave such adducts, but they were difficult to separate from some accompanying P-oxides. Other dienophiles (maleic anhydride, tetracyanoethylene, benzoquinone) were not useful in this process.

A fascinating aspect of phosphole chemistry is revealed in consideration of a different form of Diels–Alder cycloadditions. It is feasible to conduct reactions with a phosphole while it is coordinated to a metal center, and this has made possible the performance of several Diels–Alder reactions with dienophiles that similarly are held in a state of coordination or are bonded to

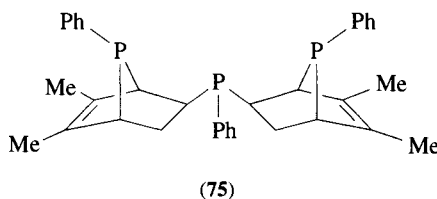
phosphorus. In the absence of the metal, no reaction occurs between the phosphole and the dienophile. An excellent example is the intramolecular cycloaddition of a 3-butenyl group with the phosphole diene in a complex with $\text{W}(\text{CO})_5$ (Equation (9)) <86OM1161>.



Another metal-promoted cycloaddition is found when, in the presence of NiBr_2 , diphenylvinylphosphine acts as a dienophile towards 3,4-dimethyl-1-phenylphosphole (Equation (10)) <89IC3453>.

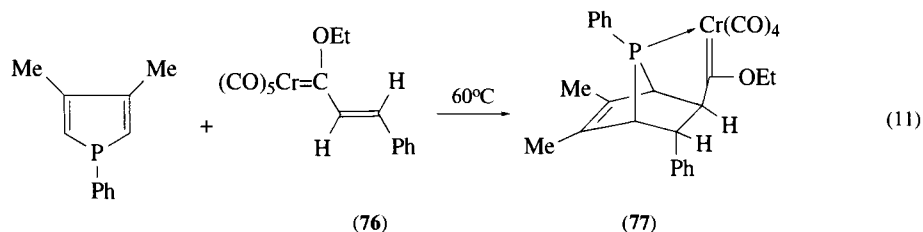


Since the cycloaddition occurs within the coordination sphere of a NiBr_2 complex of the two ligands, it was considered to be an intramolecular process. A similar reaction occurred with phenyldivinylphosphine to form triphosphine (**75**), isolated in free form by treating the complex with NaCN .



Similar intramolecular Diels–Alder reactions between phospholes and vinylphosphines were also effected with complexes of ruthenium <89IC4132, 92OM1840>, palladium <85JA6396>, molybdenum <89IC4536>, platinum <89IC217>, and iron <92OM4069>, with varying levels of reactivity and diastereoselectivity to various vinylphosphine derivatives.

A vinylcarbene complex (**76**) also is capable of acting as a dienophile towards 3,4-dimethyl-1-phenylphosphole, again considered to be an intramolecular cycloaddition within the coordination sphere <88OM2233>. A novel 7-phosphanorbornene derivative (**77**) was formed (Equation (11)).

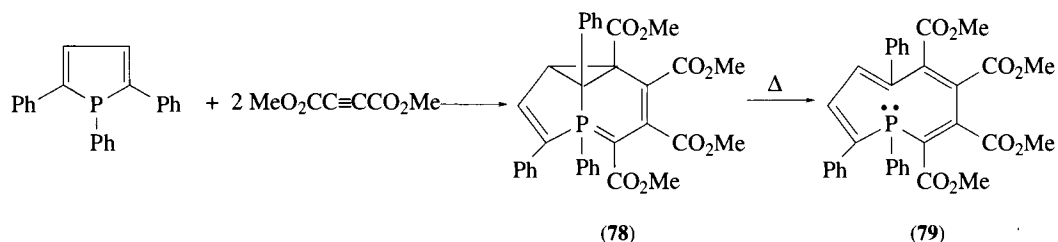


2.15.5.5 Reactions on Phosphorus Followed by Attack on Ring Carbon

2.15.5.5.1 Acetylene dicarboxylates

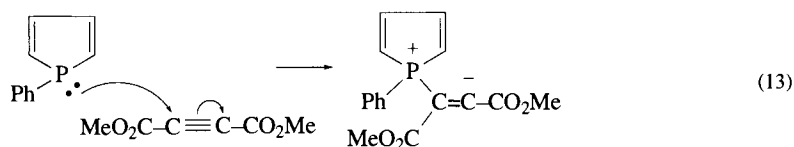
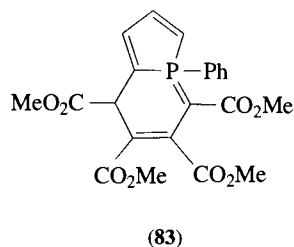
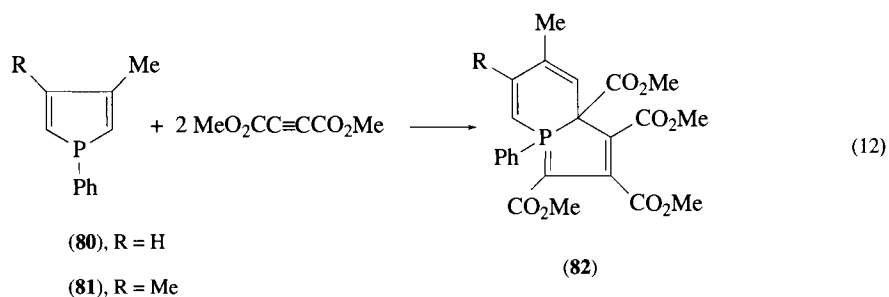
One of the earliest reactions performed on a phosphole is that with diethyl acetylenedicarboxylate, carried out with 1,2,5-triphenylphosphole <68T3437>. This reaction is best accomplished by exposing

the phosphole to excess carboxylate for two days at room temperature. It was obvious to the investigators that it was not a Diels–Alder cycloadduct; rather it resulted from a 1 : 2 reaction with the ester. Its true structure was later found to be that of a novel tricycle with phosphorus in the ylidic condition ((**78**) <70JCS(C)386>). This compound is of some interest as on refluxing in chloroform it gave a rearrangement product believed to be the phosphonin derivative (**79**) (Scheme 8); this still constitutes the only report of a monocyclic phosphonin with P in the C.N.-3 condition.

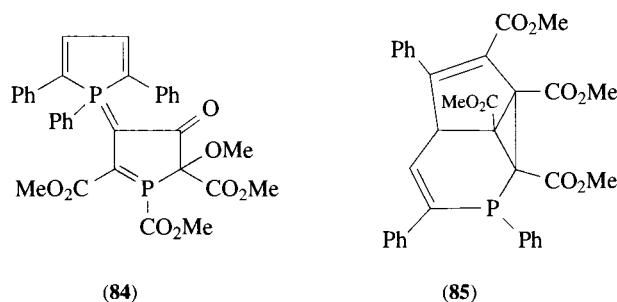


Scheme 8

The importance of the substitution pattern on the phosphole in this reaction was obvious from continued studies <78JHC1319> that showed that a much higher order of reactivity and the formation of a quite different 1 : 2 adduct (**82**) could be experienced with phospholes of structure (**80**) and (**81**). Structure (**82**) also is novel in heterocyclic phosphorus chemistry and does not seem to have been approached by any other method (Equation (12)). 1-Phenylphosphole also reacts rapidly with the ester, but gave a mixture of products in low yield. One product is probably of the same type as (**82**); the other is tentatively assigned structure (**83**). All of these early results with acetylenedicarboxylate have been summarized in a review <81H(15)637>. In terms of mechanism, in every case it appears that the initial step is the action of the phosphorus atom as a typical nucleophilic phosphine in attacking on the triple bond (Equation (13)). Subsequent reactions involve attack on the ring carbons and on a second alkynic group, but details of these processes have not been fully established.



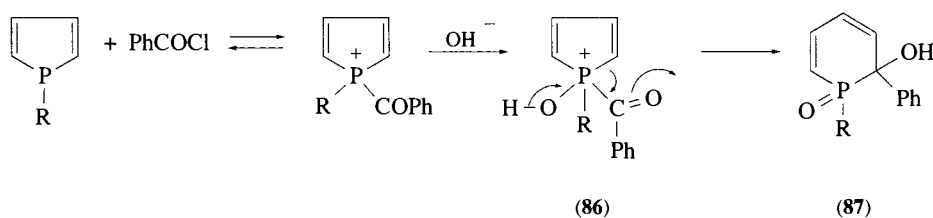
A report that includes structural proofs by x-ray crystallography has served to clarify some of the complex chemistry of these reactions <93CJC1873>. In particular, structure (78) was confirmed, and a new structure (84) was determined as a reaction product from 1,2,5-triphenylphosphole. The phosphonin structure (79) was not confirmed, and structure (85) was found instead.



When 3,4-dimethylphenylphosphole is heated in xylene at 140°C with propiolic esters, quite different products are formed. These are Diels–Alder adducts of 2*H*-phospholes, which are formed from the phospholes by [1,5] phenyl migrations. A similar result has been obtained with 1,2,5-triphenylphosphole and alkynes at higher temperatures. These reactions are described in Section 2.15.7.1.

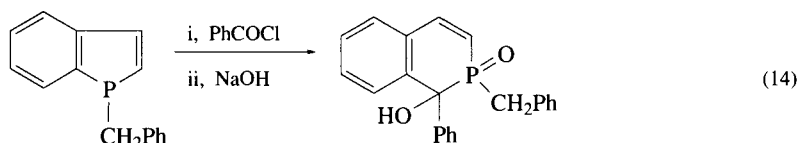
2.15.5.5.2 Aromatic acid chlorides

A remarkable and valuable reaction takes place when aromatic acid chlorides are reacted with phospholes and the mixture then treated with water. The products possess the 1,2-dihydrophosphinine ring structure. Apparently the nucleophilic phosphorus displaces chlorine from the acid chloride; water then adds to the phosphonium ion to form a pentacoordinate intermediate (86) which undergoes the ring expansion to the phosphinine derivative (87) (Scheme 9).



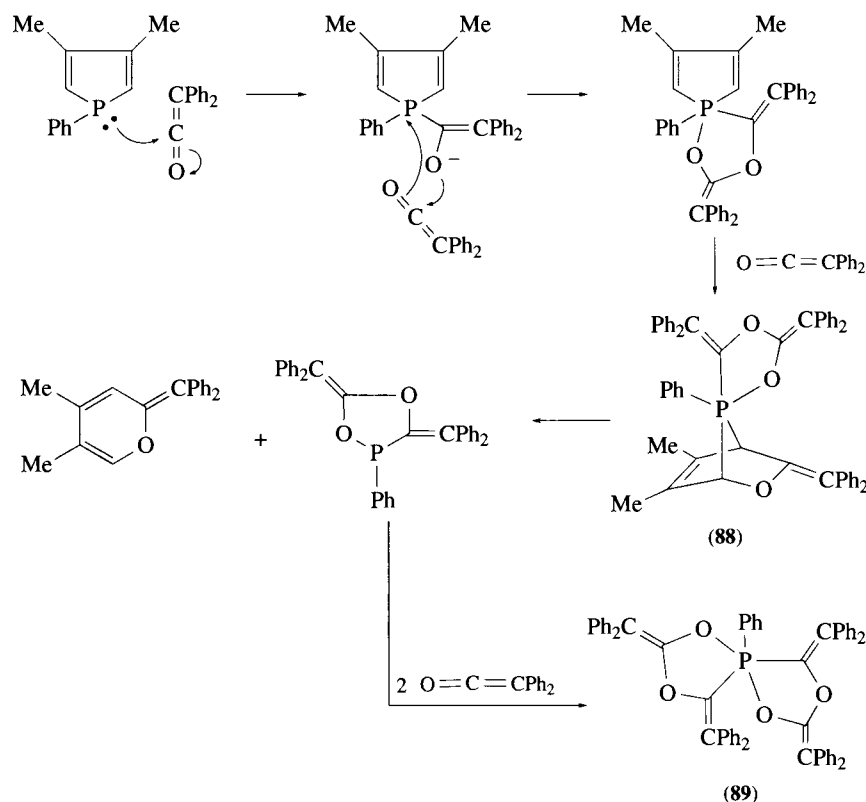
Scheme 9

In the first paper on this reaction <75CJC855>, some eight aryl acid chlorides were reacted with 3,4-dimethyl-1-phenylphosphole, giving yields generally in the range 60–80%. Thenoyl chloride also reacted to give low yields of the same type of product, but nicotinoyl chloride gave no product. It was later found that picolinoyl chloride could be used and provided access to the 2-(2-pyridyl)phosphinine derivative <82TL1565>. Various other phospholes, including 1-phenyl and 1-benzyl <84PS(19)45>, and 1-benzyl-3,4-dimethyl <79TL1753> could also be used in the process. Application of the ring expansion process to phosphindoles is also possible (Equation (14)), and has provided dihydroisophosphinolines <81NJC187>. The dihydrophosphinines can be converted to the phosphinines, and this has provided a route to some valuable members of this family as well as the isophosphinoline system <79TL1753, 84PS(19)45>.



2.15.5.5.3 Diphenylketene

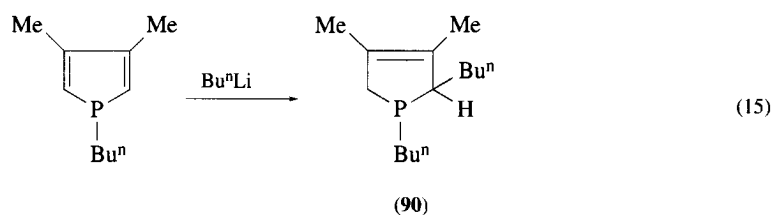
In a study of the effect of pressure on phosphole reactions, the combination of 3,4-dimethyl-1-phenylphosphole with diphenylketene was investigated (89RTC120, 89T7083). The reaction was conducted at 25°C at 9–10 kbar for 10 h and yielded a yellow crystalline product. The best yield was about 30%. The solid was proved by x-ray diffraction analysis to have the unusual spirodioxaphosphorane structure (89) (Scheme 10) and thus the Ph—P moiety has been completely removed from the carbon atoms of the phosphole ring. The product contains four moles of the ketene relative to the Ph—P unit. A plausible explanation for the course of the reaction is shown in Scheme 10. A critical step is seen to be the expulsion of the bridging phosphorus from (88), proposed by analogy to the 7-phosphanorbornenes with five-coordinate phosphorus, which undergo such fragmentations (83TL5831). This still remains as the only example of the complete destruction of the phosphole ring, but others may be encountered. The influence of pressure conditions on other phosphole reactions certainly deserves continued exploration.



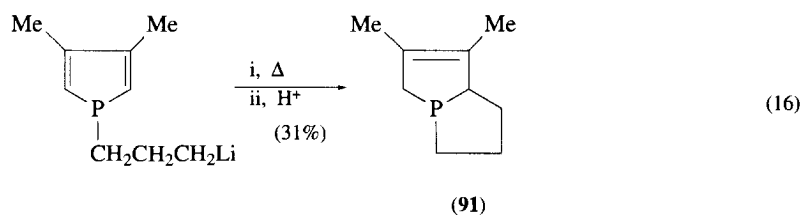
Scheme 10

2.15.5.6 Additive Reactions to a Double Bond

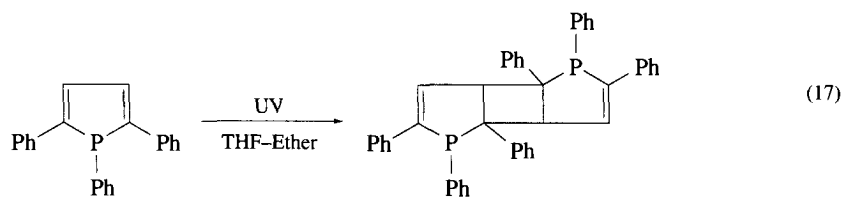
Relatively few reactions are known where only the equivalent of one double bond is involved in a reaction. Carbanion addition to the phosphole ring is of this type. *n*-Butyllithium add to 1-*n*-butyl-3,4-dimethylphosphole to give 3-phospholene (90) (Equation (15)) (72OMR(4)171).



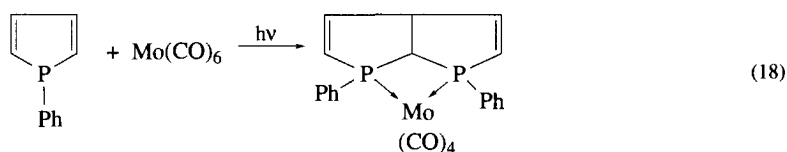
An extension of this reaction type was the intramolecular addition of a carbanion to the ring, which provided a rare type of bicyclic compound (91) with phosphorus at a bridgehead position (Equation (16)) <87OM586>.



A photochemical dimerization is known for phospholes that can be described as a [2 + 2] cycloaddition to a double bond (Equation (17)) <69TL5037>.



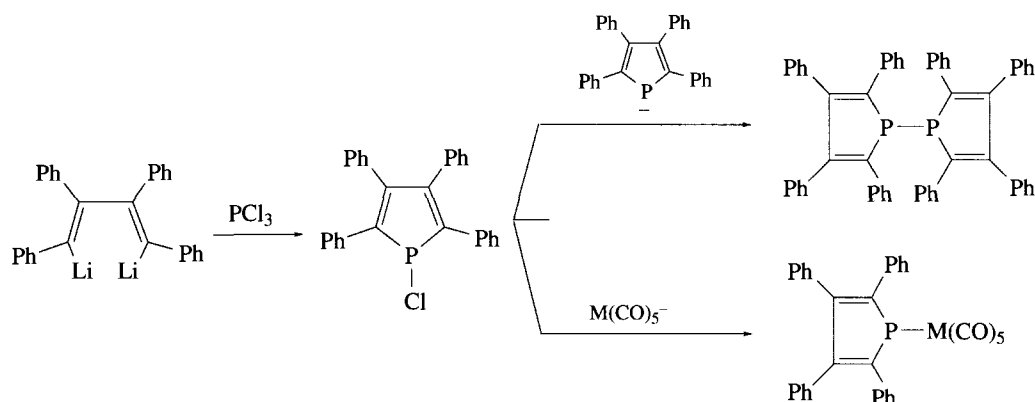
A [2 + 2] head-to-head adduct also has been detected when 1-phenylphosphole is photolyzed in the presence of $\text{Mo}(\text{CO})_6$. The reaction takes place in the coordination sphere of Mo, and the product appears in the form of a Mo complex (Equation (18)) <80JA5809>.



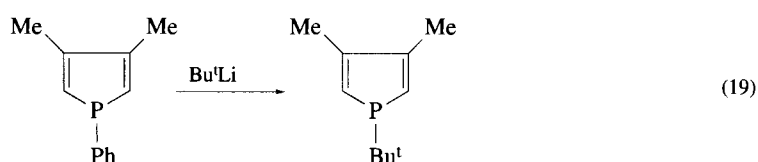
2.15.5.7 Substitution and Cleavage Reactions on Phosphorus; Phospholide Ions

1-Chlorotetraphenylphosphole was synthesized as early as 1961 from the reaction of 1,4-dilithiotetraphenylbutadiene with PCl_3 <61JA4406>, but only in the late 1970s have simpler 1-halophospholes been synthesized (from cleavage of the P—P bond in bipospholyls; see below). These halides undergo the expected replacement reactions with simple nucleophiles (see Section 2.15.12.8). Of special interest are the formation of a biposphole with a phospholide ion, and a σ -bonded metal derivative from, e.g. the anion of $\text{M}(\text{CO})_5^-$ where $\text{M} = \text{Mn}$ or Re (Scheme 11) <79JCS(D)814>.

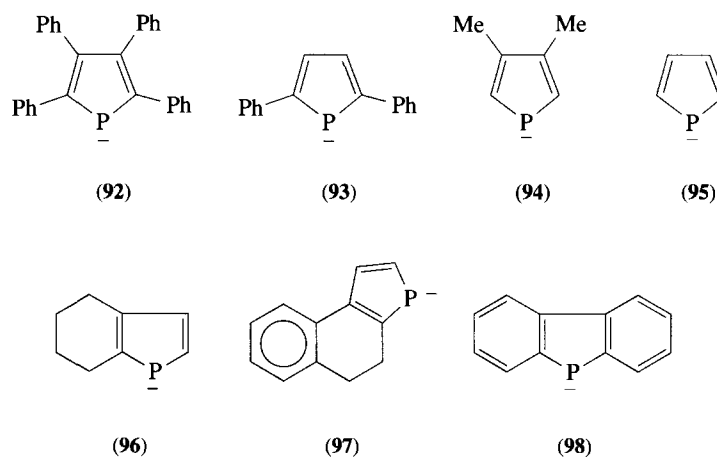
More surprising is the replacement of a carbon substituent on P, as has been observed in the reaction of 3,4-dimethyl-1-phenylphosphole with *t*-butyllithium (Equation (19)). This is an excellent method for the formation of *t*-butyl phospholes <72OMR(4)171, 72T4171>.



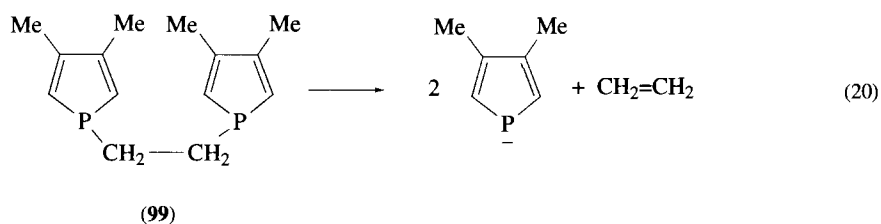
Scheme 11



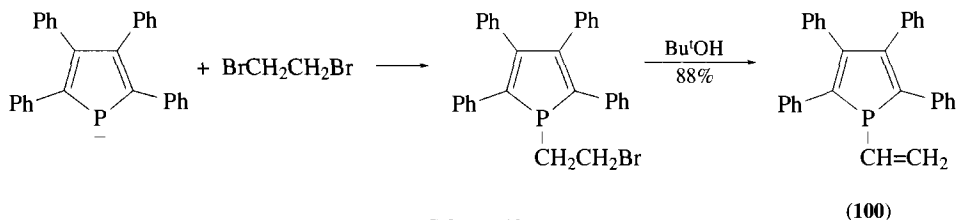
Carbon substituents (phenyl, alkyl, benzyl) on phosphorus can be cleaved by direct reaction with metals (potassium, lithium, sodium) and this constitutes an outstanding method for the generation of phospholide ions. The reactions are easily conducted in inert solvents such as THF or DME and proceed with nearly quantitative formation of the phospholide ion. This reaction was first observed by Braye with pentaphenyl- and 1,2,5-triphenylphospholes, to give (92) and (93), but has since been extended to include the formation of a great variety of phospholides, such as (94)–(97); dibenzophosphole also readily forms a phospholide ion (98): (92) and (93) <71T5523>; (94) and (95) <82TL511>; (96) and (97) <79CC401>; (98) <74PS(4)199>.



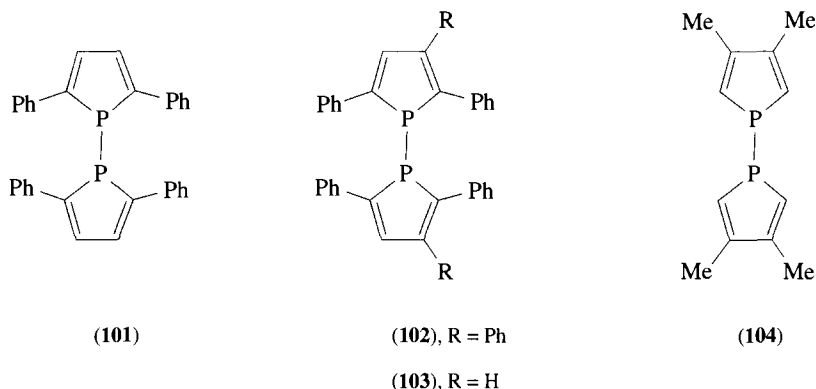
To avoid the presence in the reaction medium of the cleaved carbon fragment, the bis(phospholyl)-ethane (99) is useful, since no such fragment remains <87OM586> (Equation (20)).



Phospholides are extremely important in synthesis; they act as highly nucleophilic species that can be alkylated with simple and functional alkyl halides, and other electrophiles, to produce new phosphole derivatives. An excellent example is provided by the early synthesis of a *P*-vinylphosphole (**100**) (Scheme 12) <71T5523>.

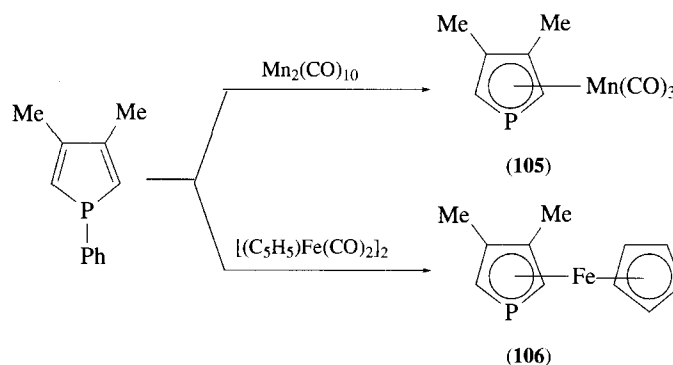


Phospholide ions are also of value in the formation of 1,1'-biphospholyls; the coupling can be effected with phosgene or iodine <82JOM(231)361>. The latter reaction, in the case of the 3,4-dimethyl derivative, is best accomplished in a medium free of the phenyl anion that accompanies the P—C cleavage. This anion was removed by conversion to triphenylaluminum with AlCl_3 <83OM1234>. Some biphospholyls synthesized in this way are shown as (**101**) <82JOM(231)361>, (**102**) and (**103**) <79JCS(D)814, 82JOC2776>, and (**104**) <83OM1234>.



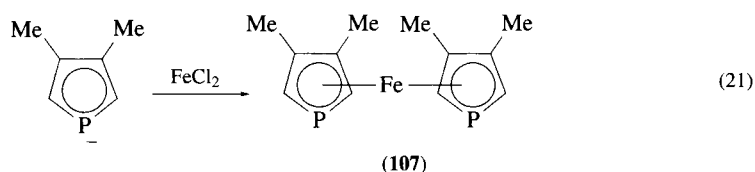
Another extremely important property of phospholide ions is their behavior as ligands in coordination chemistry. This subject has been reviewed in detail <B-83MI 215-01, 87NJC585>, and for the most part is beyond the scope of this chapter. However, complexes of the metallocene type deserve comment here, since in them the phospholide ion displays some chemistry of interest from the heterocyclic chemist's view. The first phosphametallocenes (phosphacymantrenes, (**105**)) were formed when 1-phenylphospholes were heated with $\text{Mn}_2(\text{CO})_{10}$ <75JOM(93)377, 78JA5748>. In this reaction, the P—phenyl bond is cleaved to form the phospholide ion, which proceeds to complex with manganese. Phosphaferrocene (**106**) is formed similarly (Scheme 13) <77JA3537>.

Phospholide ions can also be made separately by the cleavage reaction with metals, and then



Scheme 13

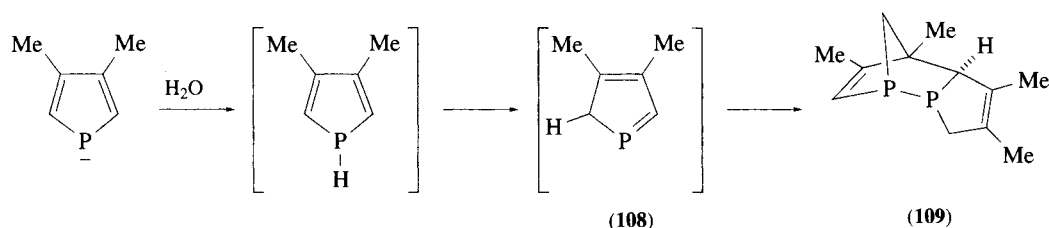
complexed with the proper metallic compounds. Diphosphoferrocene (**107**) was first made this way, by reacting the phospholide ion with FeCl_2 (Equation (21)) <78JOM(156)C33>.



These novel complexes are of interest because they possess some of the chemistry of a highly delocalized system, specifically in their giving C-substitution products with acyl halides in the presence of AlCl_3 (e.g. Mn <76TL4155, 78JA5748>; Fe <77JOM(139)77, 80NJC683, 87ICA93>). The high nucleophilicity at phosphorus is reduced in these complexes, and it no longer is the site of attack by electrophilic reagents. In addition to the Friedel–Crafts type of reaction, the Vilsmeier formylation has been observed. These reactions are of synthetic value for obtaining C-functional phospholes and are presented from that standpoint in Section 2.15.12.

2.15.5.8 1*H*-Phospholes from Protonation of Phospholide Ions

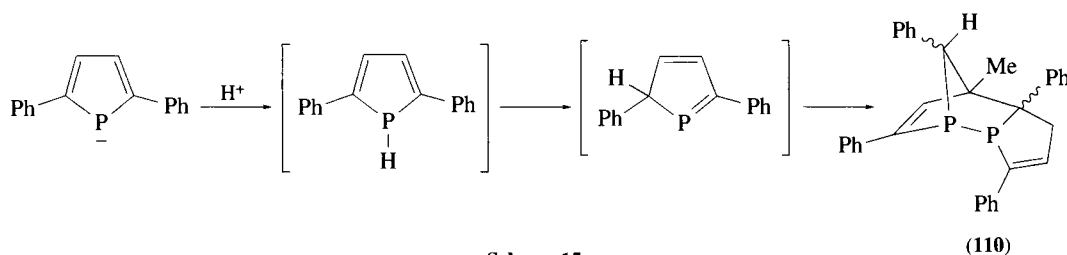
The phospholide ions are also used in the synthesis of 1*H*-phospholes. However, in a paper that is of great importance in phosphole chemistry, 1*H*-phospholes were found to be quite unstable, undergoing a [1,5] sigmatropic hydrogen shift to form 2*H*-phospholes (e.g. (**108**)) which then dimerize <82TL511> by a [4 + 2] cycloaddition (e.g. to (**109**)). This reaction can be so rapid that the parent 1*H*-phosphole and some substituted derivatives can only be observed at low temperatures, and dimerization is complete at room temperature (Scheme 14) <83PS(18)51>.



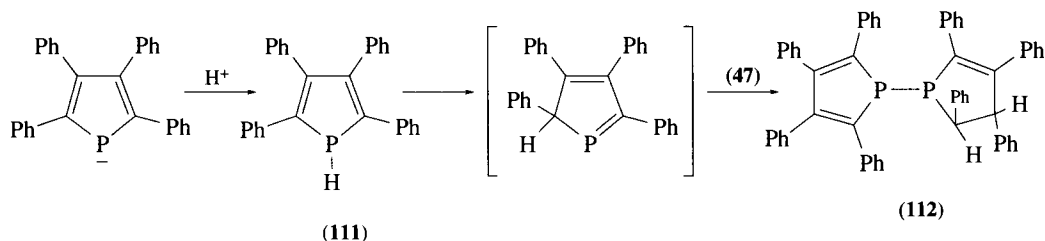
Scheme 14

The W(CO)_5 complex of the 1*H*-phosphole is more stable, lasting for 4 h at room temperature <86POL1413>. Even the polyphenylphosphole anions give 1*H*-derivatives that undergo ready rearrangement (in contrast to the earlier reports of Braye and of Freedman); thus, 1,2,5-triphenylphosphole forms the phospholide ion in 100% yield in THF at room temperature with potassium, and when the solution is quenched with acetic acid, the product observed is the dimer (**110**) (Scheme 15). Pentaphenylphosphole also gives the anion quantitatively; on acetic acid quenching at room temperature, the initially formed 1*H*-phosphole (**111**) can be observed spectroscopically ($\delta^{31}\text{P} - 40.9$, $J_{\text{PH}} = 218$ Hz) but this also undergoes the rearrangement. In this case, the 2*H*-phosphole forms more slowly, which allows a reaction between the unreacted 1*H*-form and the 2*H*-form to take place. The product is again a dimer, but of different structure ((**112**), <83JA6871>) (Scheme 16).

The Diels–Alder [4 + 2] structure of the 2*H*-dimers was first elucidated from spectroscopic data.

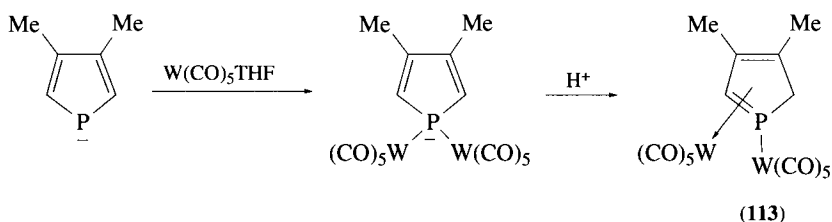


Scheme 15



Scheme 16

The P—P bond was evident from the ^{31}P NMR data, which gave a doublet of doublets with a large P—P coupling constant (e.g. for (110), $\delta -25.86$ and $+17.11$, $J = 200$ Hz). Other NMR data were interpreted on the basis of the dimeric structure. Conclusive proof of the [4 + 2] structure, with the endo fusion of the rings, came from the x-ray diffraction analysis of the product (109) from protonation of 3,4-dimethylphospholide ion (82CC1272). Another significant event was the stabilization of the 2*H*-phosphole (108) formed from this ion as a coordination complex; the anion was first coordinated to either tungsten or chromium carbonyl and then protonated, whereupon the [1,5] H-shift occurred to the 2*H*-phosphole (e.g. (113)), which was unable to undergo the dimerization (Scheme 17) (84JA826).



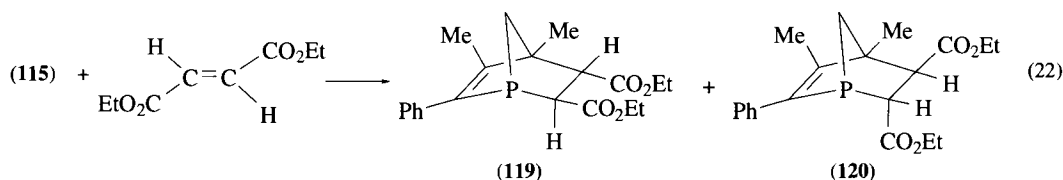
Scheme 17

The discovery that 1*H*-phospholes are unstable relative to 2*H*-phospholes is of theoretical significance, and represents a unique difference from pyrrole chemistry. The subject has received comment in Section 2.15.7.1.

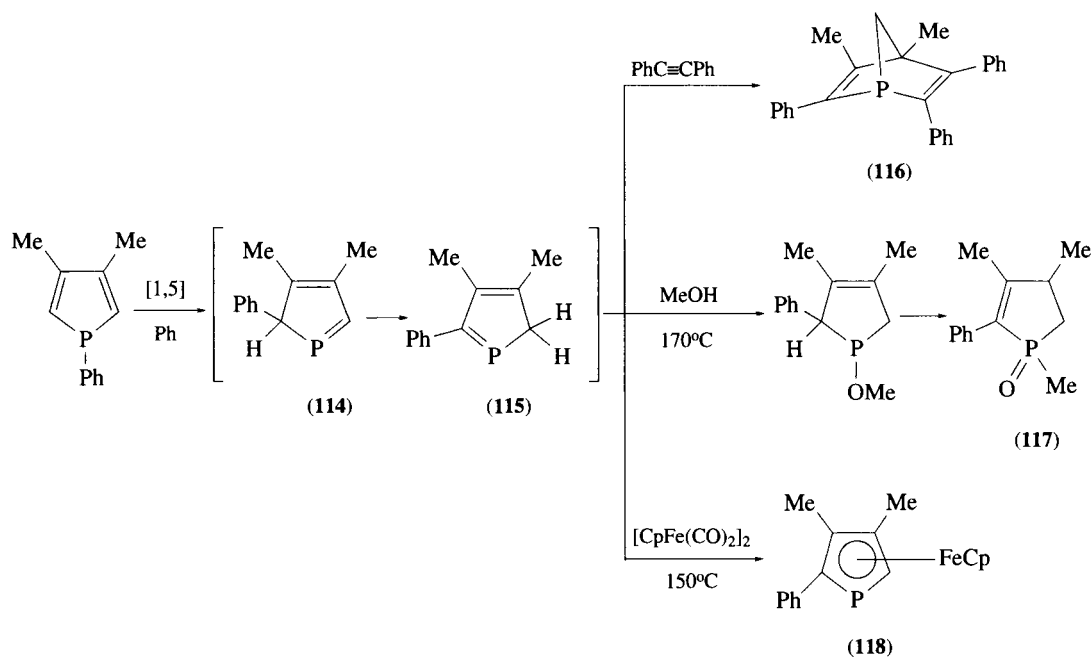
2.15.5.9 [1,5] Sigmatropic Rearrangement of P-Substituents

The important observation was made in 1981 that a P-phenyl substituent on a phosphole could undergo migration to carbon, creating thereby the highly reactive 2*H*-phosphole system (81JA4595). This reaction, a [1,5] sigmatropic rearrangement, occurred in 100% yield when conducted for 60 h at 170°C with 3,4-dimethyl-1-phenylphosphole. The initially-formed 2*H*-phosphole (114) rearranged to (115), which was detected by various trapping reactions (Scheme 18). Additions occur to the diene unit (with alkynes, forming (116); see also (89NJC369)) or at the $\text{P}=\text{C}$ bond (to (117)). Trapping as a coordination complex (118) was also possible.

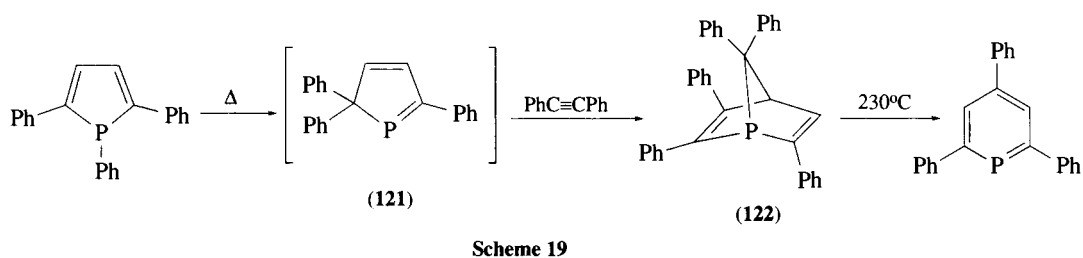
The intermediate 2*H*-phosphole (115) could also be trapped with alkenes to yield isolatable 1-phosphanorbornene derivatives (e.g. (119) major and (120) minor; (89JOC4754)) Equation (22).



1,2,5-Triphenylphosphole also gave a 2*H*-phosphole rearrangement product (121), which was trapped with toluene in 60% yield. These alkyne reaction products are of interest in possessing the rare bicyclo[2.2.1] framework with phosphorus at a bridgehead position. Also of interest was the observation that the adduct (122) was thermally unstable; the bridging Ph_2PC unit was eliminated at 230°C to form a phosphinine, and this constitutes a new and valuable entry to this ring system

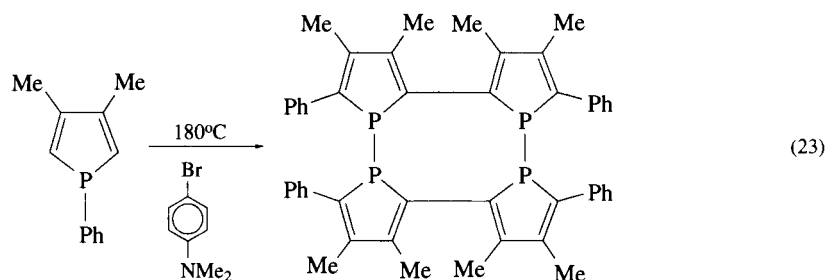


(Scheme 19). Other phosphinines have also been prepared by strong heating of the triphenylphosphole in the presence of an alkyne <82JOC2376>.

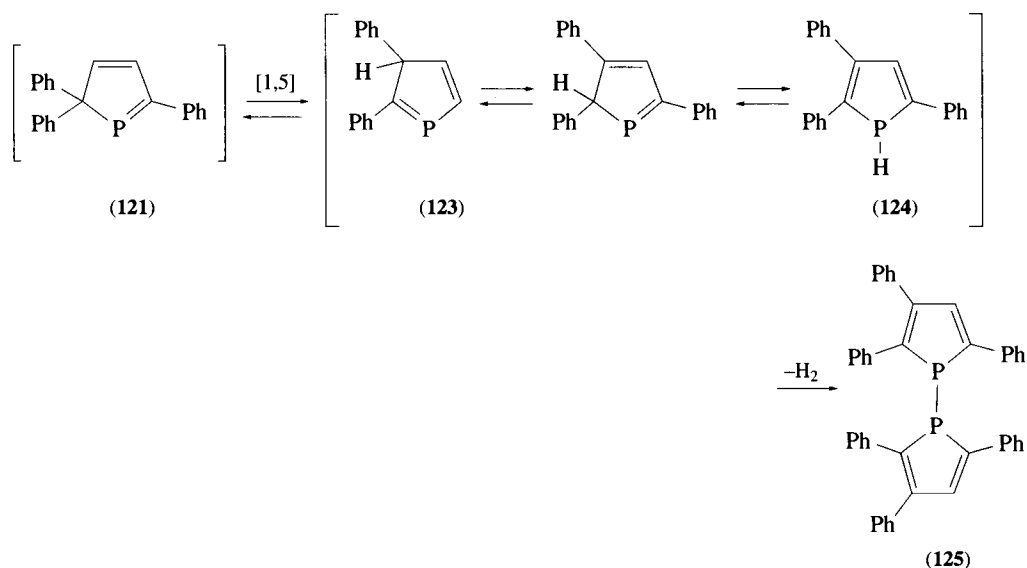


When the pyrolysis of 1,2,5-triphenylphosphole was conducted at a higher temperature (230°C) in the absence of a trapping agent, the product was found to have the rare biphospholyl structure (125) <82JOC2376>. This was proposed to result from a second [1,5] phenyl shift in (121) to form 3*H*-phosphole (123) followed by hydrogen shifts to a 1*H*-phosphole structure (124) which then lost hydrogen (Scheme 20).

2,2'-Diphospholes are themselves subject to dimerization, thereby forming cyclic tetramers containing four phosphole rings <82JA2077, 90AG(E)655>. The reaction with 3,4-dimethyl-1-phenylphosphole can be conducted at 170–180°C; *N,N*-dimethyl-4-bromoaniline greatly enhances the reaction rate, and leads to yields of the tetramer of 40–45% after 18 h (Equation (23)) <94JA3306>.



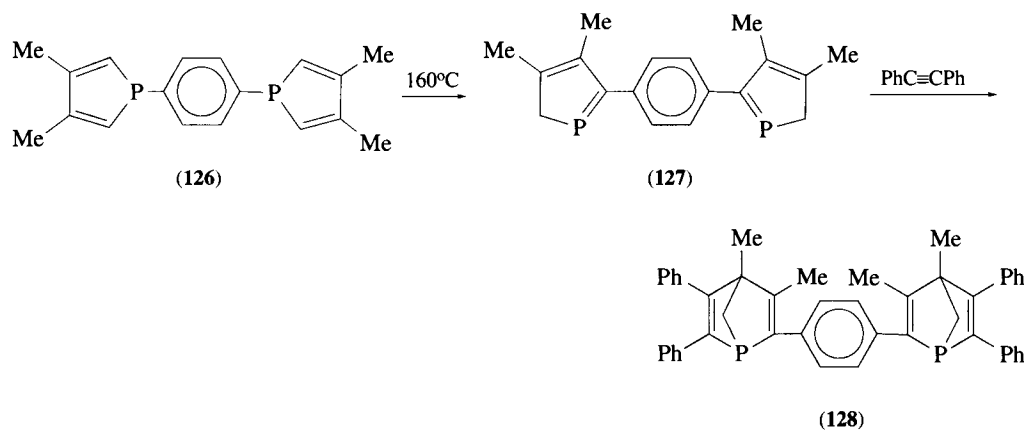
The P—P bond in the tetramers can be cleaved by sodium in THF, giving a diphospholide which can be used for elaboration to macrocyclic systems on reaction with dihalides. Cleavage of only one



Scheme 20

P—P bond is also possible. The macrocycles formed from the two types of diphospholide ions are of considerable interest as novel ligands in coordination chemistry.

A bisphosphole (**126**) has also been subjected to the rearrangement; the connecting *p*-phenylene group migrated in both rings to give the novel bis-2*H*-phosphole (**127**) trapped with toluene (giving (**128**)) as well as with metal complexes (Scheme 21) <90HAC187>.

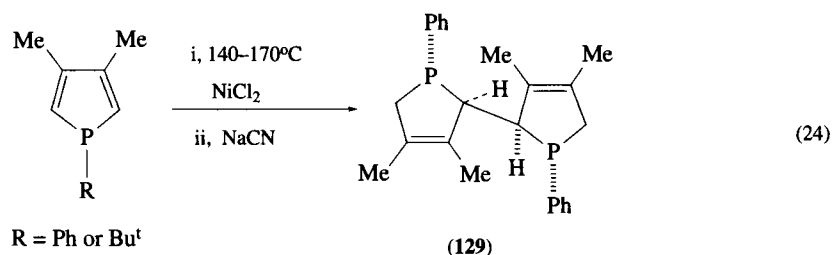


Scheme 21

The mechanism of [1,5] sigmatropic rearrangements of P-substituents has been treated by Mathey <83JA6871>. The reaction is also known for P in the four- and five-coordinate states, and in fact occurs more readily in the latter state. An important feature is hyperconjugative overlap of the σ -orbital of an exocyclic P-substituent with the π^* orbital of the diene unit, which is geometrically most favorable in the trigonal bipyramidal structure of C.N.-5.

2.15.5.10 Thermal Dimerization of Phospholes

Novel 2,2'-bisphospholenes can be obtained when certain phospholes are heated at 140–170°C in the presence of nickel(II) chloride <84JA425, 85IC4141>. The products are obtained in the form of a nickel complex, but the free bisphospholene can be liberated with sodium cyanide. This product has the exclusive stereochemistry shown in structure (**129**) (Equation (24)).

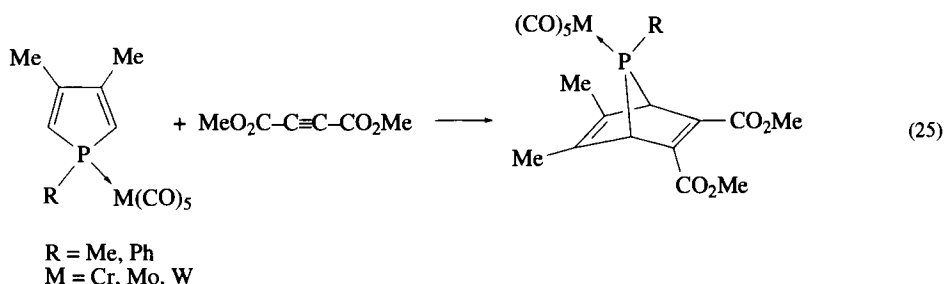


In the presence of the nickel chloride, the thermal [1,5] migration of the P-substituent described in Section 2.15.5.9 is completely avoided. The bisphospholenes are of considerable value as ligands in coordination chemistry.

2.15.5.11 Coordination with Metals

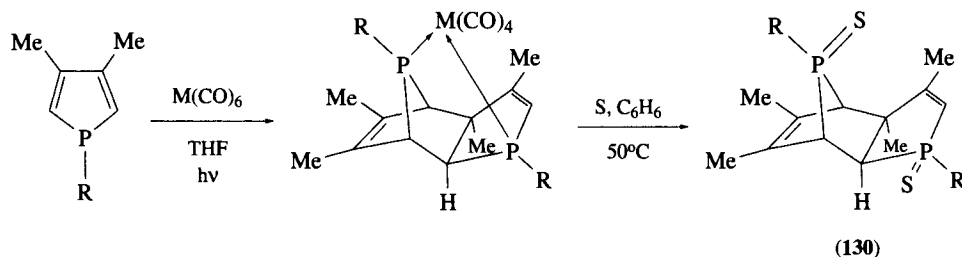
There is a vast and increasing literature on the formation, characterization, and practical applications of metallic complexes of phospholes. An excellent summary of the various types of complexes that can be prepared is provided in a review (B-83MI 215-01). Suffice it to say that complexation is a fundamental reaction of phospholes and has opened many pathways both in phosphole reaction chemistry and in coordination chemistry. In this section, only special reactions that occur with the phosphole moiety while in coordinated form are considered. Examples have, of course, already been encountered, as in the intramolecular form of Diels–Alder cycloadditions described in Section 2.15.5.4. Other important processes include:

(i) Diels–Alder reaction of σ -complexes of phospholes with $\text{Cr}(\text{CO})_5$, $\text{Mo}(\text{CO})_5$, or $\text{W}(\text{CO})_5$ (Equation (25)) (82CC667).



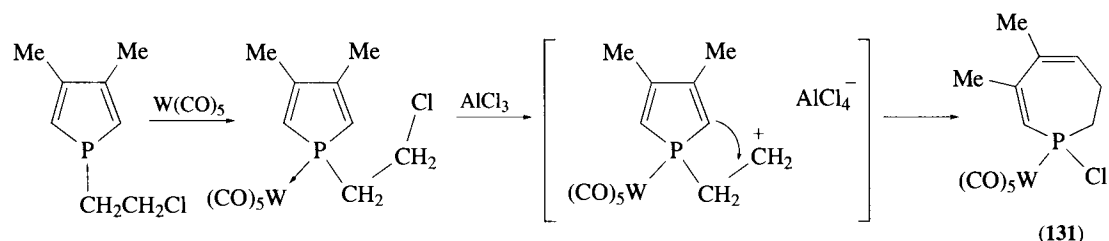
This reaction permitted the successful construction of a stable 7-phosphanorbornadiene derivative. While it was not possible to release the free phosphine from the complex, the complexes acted, on thermolysis, as precursors of the synthetically valuable phosphinidene complex, $\text{RPM}(\text{CO})_5$.

(ii) [2 + 2] and [4 + 2] dimerization of σ -complexes. UV irradiation of some phosphole $\cdot \text{M}(\text{CO})_5$ complexes (but not of the free phosphole) causes the formation of a Diels–Alder dimer, which acts as a bidentate ligand to the metal center. The dimer has the unique *exo* fusion of the two rings, and is the only dimeric product known to date to have this configuration proved by x-ray analysis (80JA5809). The complex where R = Ph was broken on heating with sulfur to form this novel *exo*-fused dimeric disulfide (130) (Scheme 22).



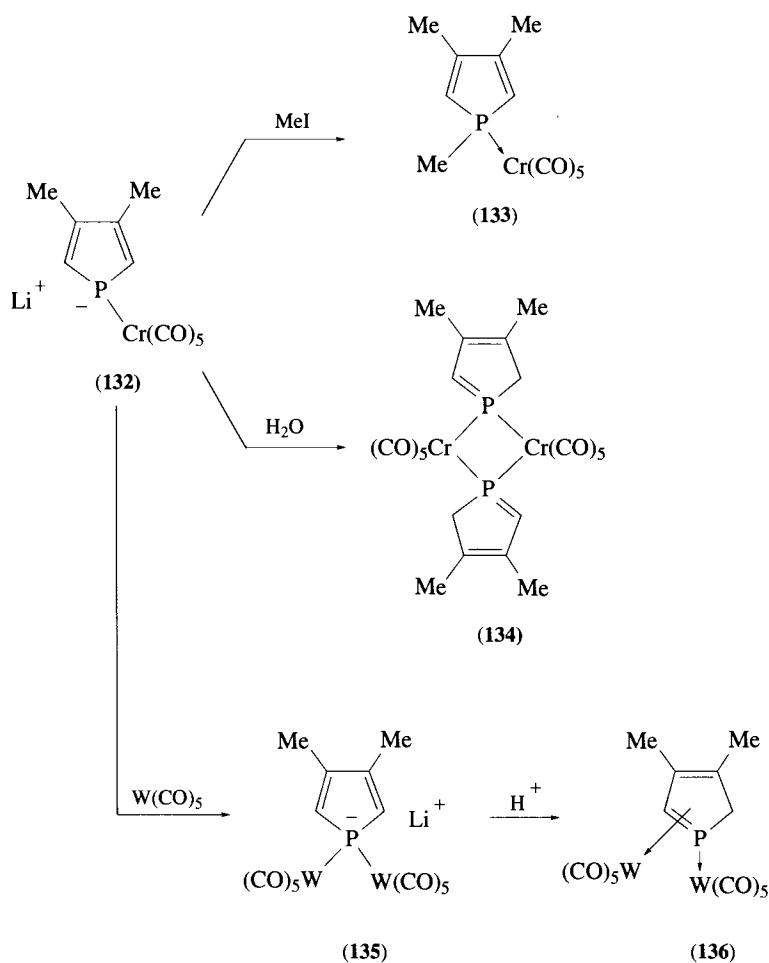
Scheme 22

(iii) Rearrangement of P- β -chloroethylphospholes to dihydrophosphepins. β -Chloroethyl-3,4-dimethylphosphole forms a complex with $W(CO)_5$ which on treatment with $AlCl_3$ at room temperature rearranges with the insertion of the ethylene unit into the ring. The product is a 1-chlorodihydrophosphepin (**131**) (Scheme 23(a)) <85TL3461>. The chlorine is reactive, and can be displaced on reaction with methanol at room temperature with retention of the metal group.



Scheme 23(a)

(iv) Isomerization to complexes of 2*H*-phospholes. The anionic complex (**132**), formed by addition of 3,4-dimethylphospholide ion to $Cr(CO)_5$, can be alkylated on P to form the stable 1,3,4-trimethylphosphole complex (**133**), but on reaction with water the protonated product undergoes a [1,5] sigmatropic H-shift to form the complex (**134**) of the 2*H*-phosphole <84JA826>. The same phospholide ion reacts with two moles of $W(CO)_5$ to give the anionic complex (**135**), which also gives a stable 2*H*-phosphole complex (**136**) on protonation (Scheme 23(b)).



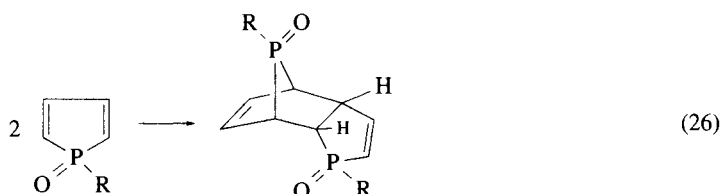
Scheme 23(b)

2.15.6 REACTIVITY AND SYNTHESIS OF PHOSPHOLES WITH C.N.-4

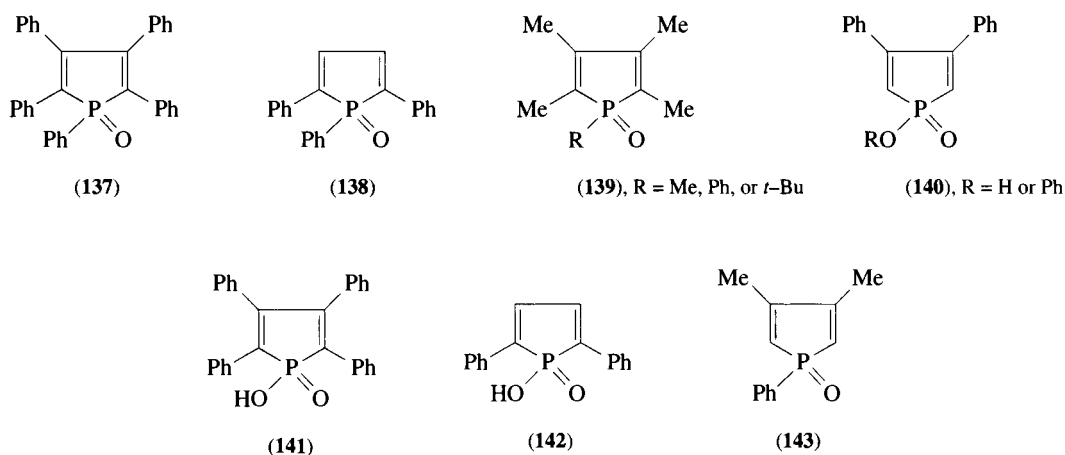
2.15.6.1 Phosphole Oxides

2.15.6.1.1 Synthesis

The great majority of phosphole oxides are unstable and rapidly undergo Diels–Alder dimerization upon formation (Equation (26)). This is true for all types of phosphoryl-containing functionalities studied to date (phosphine oxides, phosphinates, phosphinamides).



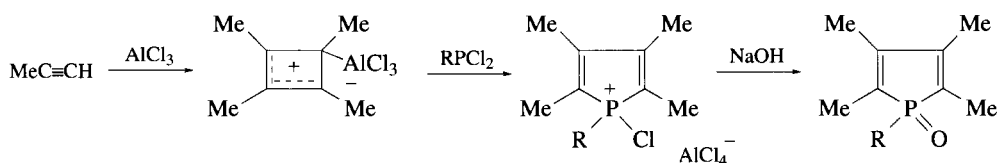
There are, however, some exceptions, which appear to be associated with steric crowding that may be experienced around the phosphorus atom by substitutions at C-2 and C-5 (as in (138) and (142)), or at C-3 and C-4 by phenyl as in (140). All known pentasubstituted oxides (137), (139), and (141) are stable. Compound (143) cannot be isolated but has reasonable stability in solution, no doubt associated with the presence of the 3,4-dimethyl substitution ((137) <61JA4406>; (138) <65JCS2184>; (139) <83TL1423>; (140) <71JA4541>; (141) <74PS(4)199>; (142) <74PS(4)199>; (143) <91HAC359>).



There are two general methods for the creation of phosphole oxides. The most obvious is the direct oxidation of phospholes, which is easily accomplished with hydrogen peroxide, *t*-butyl hydroperoxide, *m*-chloroperbenzoic acid, and the like. The stable compounds (137), (138), and (143) were prepared this way. The second method is indirect, and involves the elimination of hydrogen halide from halophospholane oxides. These substances are generally obtained by addition of halogen (usually bromine) to the double bond of 3-phospholene oxides, themselves readily available from the McCormack reaction (see Section 2.15.8.2), or by bromination at the 2-position of phospholane oxides. The elimination of hydrogen halide occurs at room temperature in the presence of a base, commonly triethylamine. This method is used for the synthesis of phosphole oxides that are easily dimerized or are to be trapped with dienophiles present in the reaction medium (see Section 2.15.13.2). The considerable literature on the generation of unstable phosphole oxides has been extensively reviewed by Mathey <88CRV429> and Quin <90RHA39>.

A more direct method, but of limited scope, consists of the direct formation of the phosphole ring in the chlorophosphonium condition, which with aqueous base gives the phosphole oxide <83TL1423> in 60–78% overall yield. The reactants are phenyl-, methyl- or *t*-butylphosphonous dichloride and the aluminum chloride σ -complex of cyclobutadienes (Scheme 24).

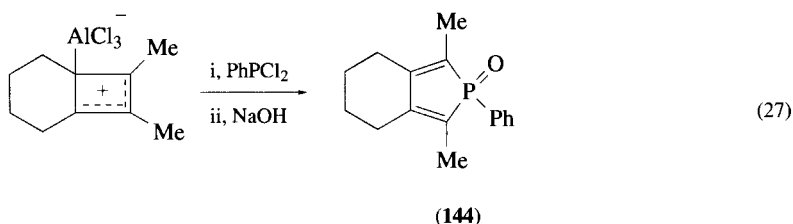
The starting complexes were formed from reaction of 2-butyne with aluminum chloride, and



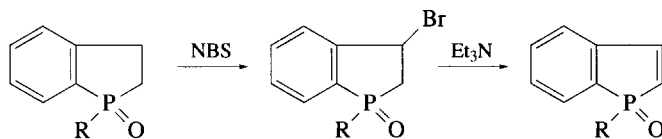
Scheme 24

accordingly the phosphole product is a tetramethyl derivative. With this degree of substitution, the phosphole oxides are stable, isolable solids.

This method has also been used to synthesize the bicyclic phosphole oxide ((**144**), obtained as a 3 : 1 mixture with the 2,3-cyclohexano isomer, but isolable) (Equation (27)).

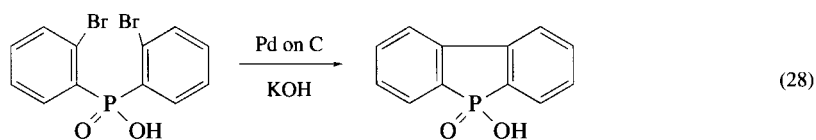


Benzo[*b*]phosphole oxides (phosphindole oxides) and dibenzo[*b,d*]phosphole oxides are stable as the monomer, since Diels–Alder dimerization would require disruption of the aromatic ring system. The former are prepared by the dehydrohalogenation method ($R = \text{Ph}$ <71CJC530>; $R = \text{MeO}$ <74AJC831, 82PS(13)259, 81NJC187>) (Scheme 25).



Scheme 25

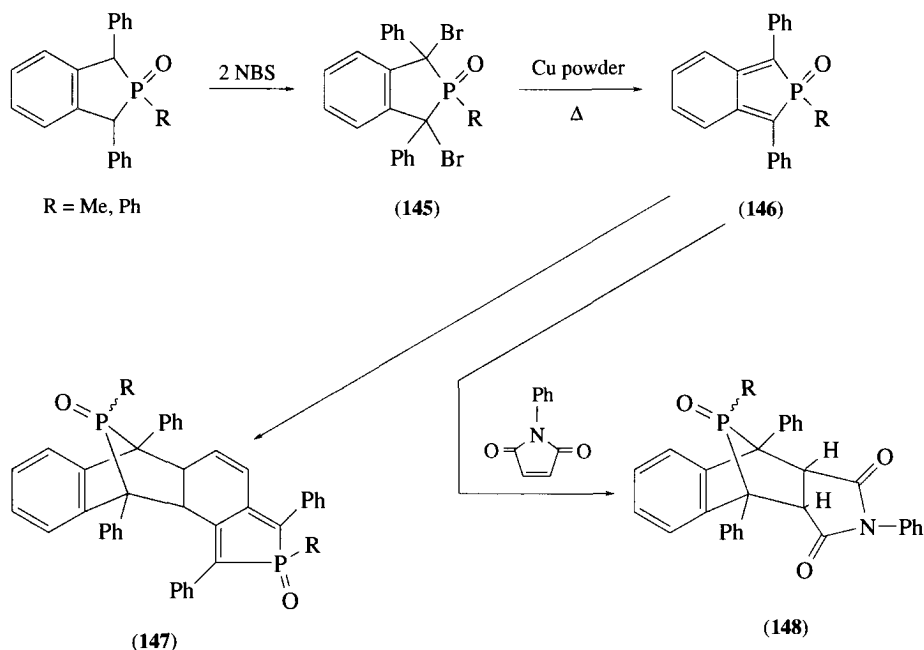
Dibenzophosphole oxides are prepared by the direct oxidation (H_2O_2) of the phospholes <69JOC1777>; a diarylphosphinic acid can also be used for generating the phosphole ring <56JOC238, 59JOC638> (Equation (28)).



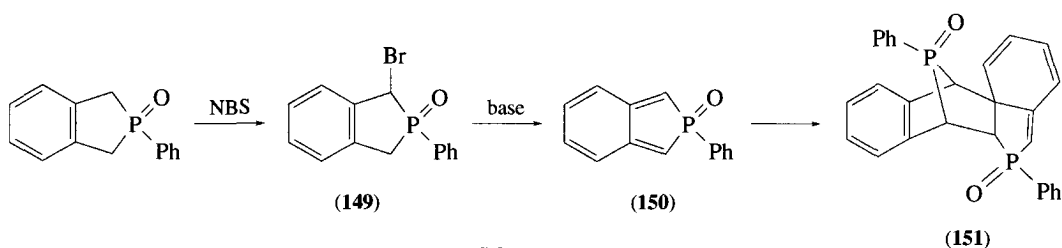
Benzo[*c*]phosphole oxides (isophosphindole oxides) have remained elusive; all attempts to generate them have led to dimeric products. The first approach to an isophosphindole oxide involved debromination of the dibromide (**145**) with copper powder and yielded a yellow solid with the composition of a dimer of the expected isophosphindole oxide (**146**) to which structure (**147**) was assigned <70CC122>. This was supported by the spectral properties, and the yellow color indicated the presence of the phosphole oxide ring. To account for this structure, the intermediate isophosphindole oxide acts as a diene in condensation with a double bond of the benzo group. When *N*-phenylmaleimide was present in the reaction medium, (**146**) was trapped as the Diels–Alder adduct (**148**), in the same way that monocyclic phosphole oxides can be trapped (see Scheme 26).

Another approach involved the dehydrobromination of bromide (**149**), which also led to a dimeric product, (**151**) (Scheme 27) <73TL4815, 75T2537>. In this case, the evidence supported a dimer structure of the same Diels–Alder type as observed from monocyclic phosphole oxides, and this was supported by later studies <85MRC929>. Dienophiles present as trapping agents gave the expected adducts of (**150**).

Functionalized phosphole oxides are quite rare; the only stable type that is known has four carboxylate groups, with phosphorus in phosphinate form <84JCS(P1)1627, 88JCS(P1)175> or in phos-



Scheme 26



Scheme 27

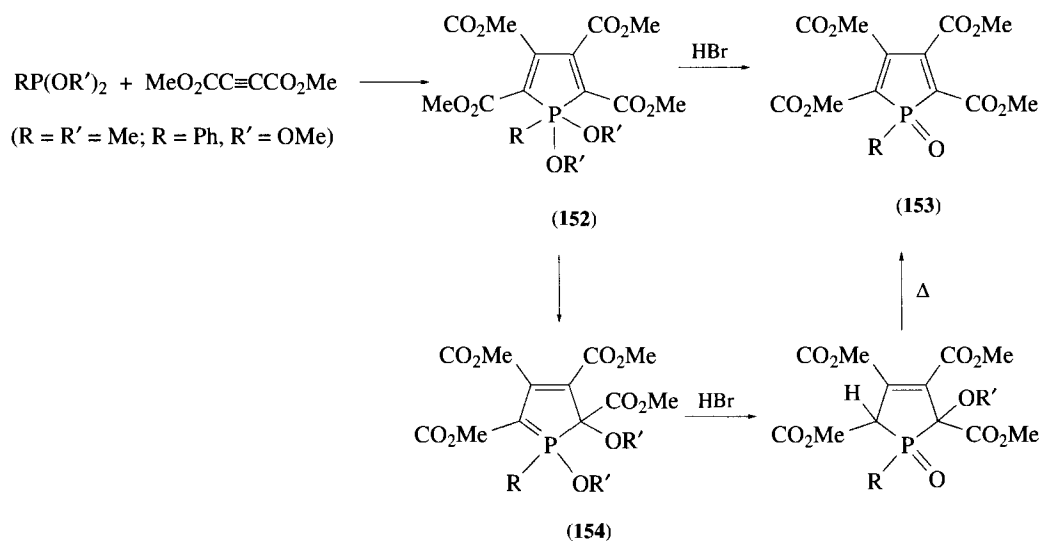
phine oxide form <88JCS(P1)175>. A five-coordinate adduct (152) from a phosphite or a phosphinite is converted with HBr to the phosphole oxide (153) or is first allowed to rearrange to a 2*H*-phosphole (154) which is then converted to (153) (Scheme 28). Since the starting phosphorus materials are the common trialkyl phosphites or dialkyl phosphonites, respectively, and the yields are quite high, the method is of considerable synthetic value for this type of phosphole derivative.

2.15.6.1.2 Reactions

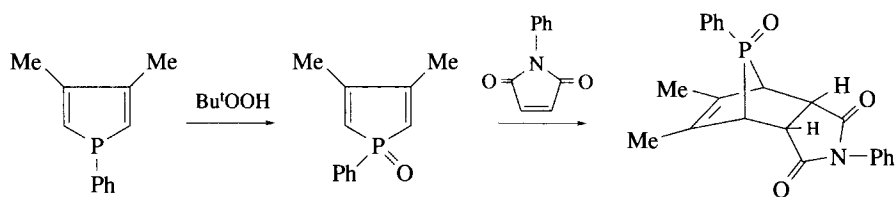
Because the few stable phosphole oxides that are known have special steric features that can modify reactivity, relatively little is known about the reaction chemistry of the ring systems. The unstable structures are of interest, however, for the dimers that form spontaneously are of value as derivatives of the 7-phosphanorbornene ring system, a subject reviewed in some detail elsewhere <88CRV429, 90RHA39>. Of more importance is the reaction of the unstable phosphole oxides with dienophiles when present as trapping agents in the reaction medium, which also gives 7-phosphanorbornene derivatives. Numerous examples of such compounds are known and are described in the reviews cited. The choice of dienophile is somewhat restricted, however. The most commonly used type has the maleimide or maleate structure; some examples are given in Schemes 29 <91HAC359> and 30 <85JA3389>.

Ethyne dicarboxylate has given cycloaddition reactions with some stabilized, highly substituted phosphole oxides, but the cycloadducts (7-phosphanorbornadiene derivatives) readily lose the phosphorus bridge under the reaction conditions and have never been characterized <65JCS2184, 72JA4761>. However, diaroyl ethynes have been reported to give stable 7-phosphanorbornadiene

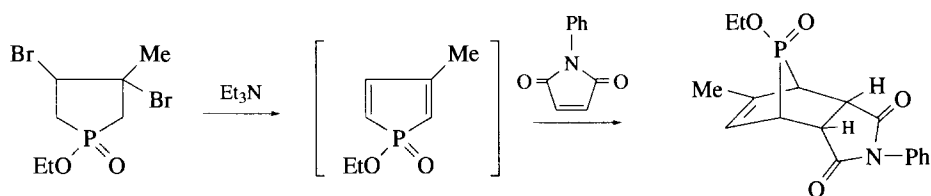
Phospholes



Scheme 28

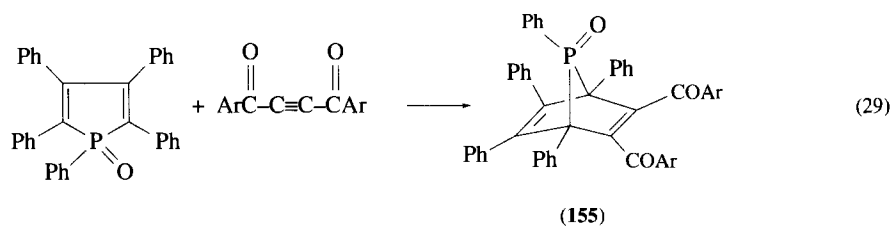


Scheme 29



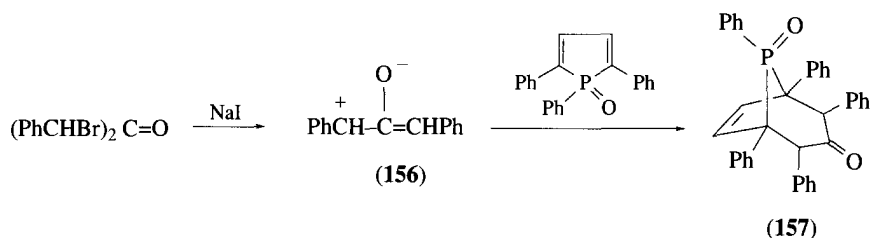
Scheme 30

derivatives (155) with pentaphenylphosphole oxide when the reaction was conducted under high pressure (Equation (29)) (90H(30)201).



Attempts to trap the less stable phosphole oxides by having ethyne derivatives present in the medium of their generation have not been successful, since the ethynes used so far have been sensitive to the reagents in the medium.

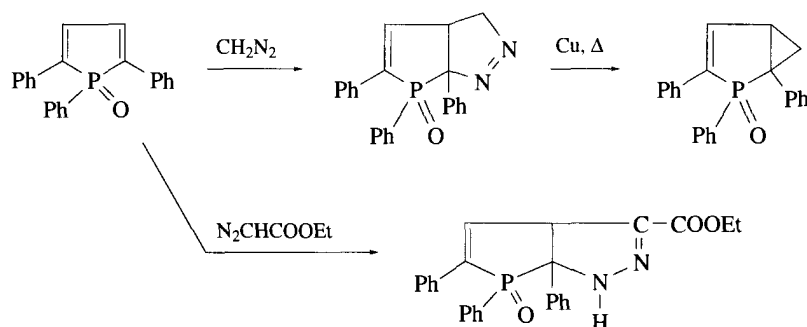
Phosphole oxides also participate in a cycloaddition reaction with oxyallyl cations $\langle 75T53 \rangle$, a process that gives rise to novel phosphatropolone structures. Preformed 1,2,5-triphenylphosphole oxide gave a derivative of the 8-phosphabicyclo[3.2.1]octan-6-ene-3-one ring system ((**157**), 91%). The oxyallyl cation (**156**) was prepared from α, α' -dibromodibenzylketone and NaI in acetonitrile (Scheme 31).



Scheme 31

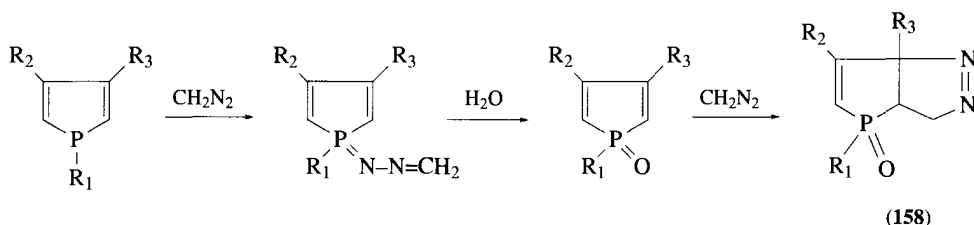
The reaction was also attempted on 3,4-dimethyl-1-phenylphosphole oxide generated in the reaction medium; the expected cycloadduct was obtained in only 6% yield, dimerization of the oxide being the major reaction pathway.

Additions to only one double bond of a phosphole oxide are also possible. Diazomethane gives a pyrazoline, thermally converted to a cyclopropano derivative, as has been observed with 1,2,5-triphenylphosphole oxide $\langle 65JCS2184, 71CJC874 \rangle$ and 3,4-dimethyl-1-phenylphosphole oxide $\langle 89T7083 \rangle$ (Scheme 32).



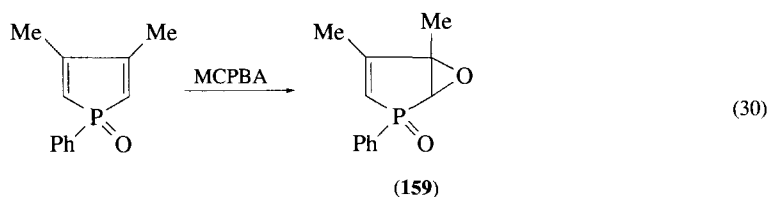
Scheme 32

A phosphole oxide may be an intermediate when diazomethane reacts with a phosphole in the presence of water, since the reaction product is the pyrazoline that would be expected from addition to the double bond of a phosphole oxide. The technique was applied in high yield to several phospholes (Scheme 33) $\langle 89TL3525, 93CB473 \rangle$.

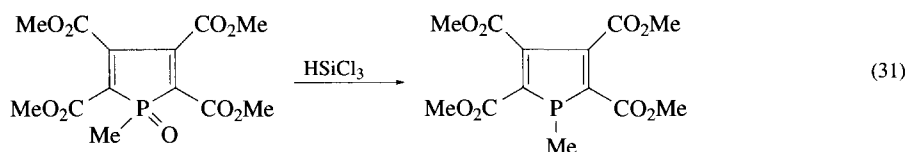


Scheme 33

The orientation of addition of diazomethane is the opposite of that proposed in the earlier work $\langle 65JCS2184 \rangle$. The ^1H NMR spectrum of (**158**) does support this structure, since large $^3J_{\text{PH}}$ values (14–19 Hz) were observed for the CH_2 group. It was also found possible to epoxidize one of the double bonds of 3,4-dimethyl-1-phenylphosphole oxide with *m*-chloroperbenzoic acid $\langle 91HAC359 \rangle$. The product (**159**) was a stable solid, a mixture of diastereoisomers (Equation (30)).



Phosphine oxides are normally reducible to the phosphines with trichlorosilane or phenylsilane (Equation (31)), and some examples with cyclic systems have recently been reviewed $\langle$ B-90MI 215-01 $\rangle$. This reaction probably can be applied to the stable phosphole oxides, and would have practical value as a phosphole synthesis when applied to phosphole oxides that are prepared by methods other than the direct oxidation method.

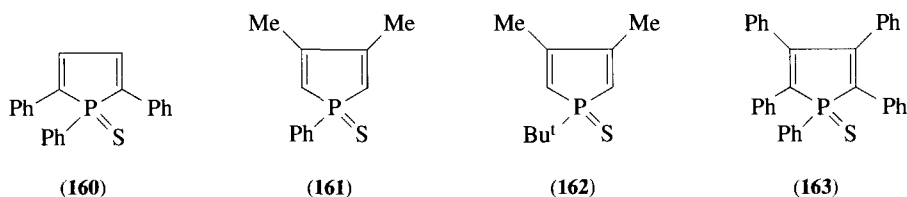


2.15.6.2 Phosphole Sulfides and Selenides

2.15.6.2.1 Synthesis

The only approach to phosphole sulfides involves direct addition of sulfur to preformed phospholes (e.g. $\langle$ 65JCS2184, 68TL1755, 69CR(C)1066, 70BSF4433 $\rangle$). The most commonly studied compound is 3,4-dimethyl-1-phenylphosphole sulfide, easily prepared in high yield from the phosphole on refluxing with sulfur in benzene. Unlike the corresponding oxide, the sulfide is a stable, isolable solid; stability of the phosphole sulfides is in fact one of their notable characteristics, and makes them quite valuable as starting materials in processes that are denied to most phosphole oxides. The known stable phosphole sulfides that are available as starting materials are shown below (**160**–**163**): (**160**) $\langle$ 65JCS2184 $\rangle$; (**161**) $\langle$ 69CR(C)1066 $\rangle$; (**162**) $\langle$ 70BSF4433 $\rangle$; (**163**) $\langle$ 61JA4406 $\rangle$. Many other phosphole sulfides have been derived from them as will be seen in the next section.

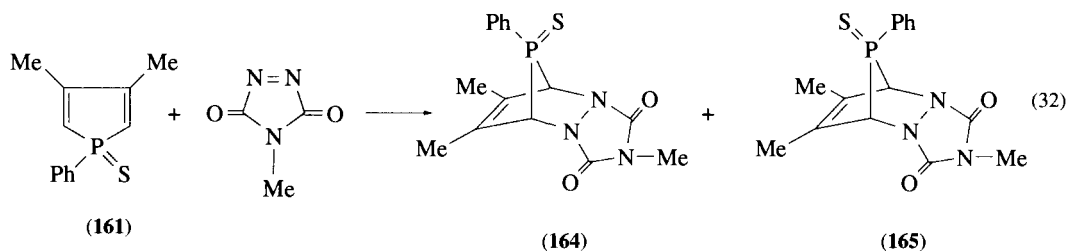
Selenides are also prepared from the phospholes by reaction with selenium $\langle$ 65JCS2184, 72T4171 $\rangle$. Stable compounds analogous to (**160**), (**161**) and (**163**) are known, but have been little studied.



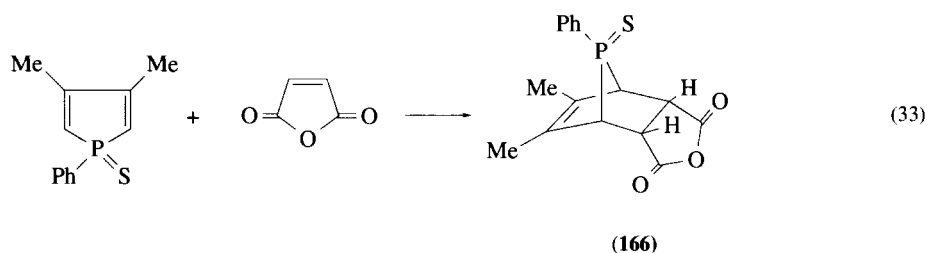
2.15.6.2.2 Reactions

Phosphole sulfides are generally not prone to dimerization by a Diels–Alder reaction, although exceptions are known (e.g. $\langle$ 81PS(10)175 $\rangle$), but they do participate in cycloaddition reactions with various dienophiles $\langle$ 76T2427 $\rangle$. Thus, the 3,4-dimethyl-1-phenyl derivative (**161**) gave an adduct with *N*-methyl-1,2,4-triazoline-3,5-dione consisting of two isomers, the major being obtained in pure form by recrystallization (Equation (32)). The isomerism was attributed to configurational differences at phosphorus. Yields were not reported in the original work, but a later preparation of the same adduct gave a yield after recrystallization of 34% of the isomer mixture (**164**)–(**165**) $\langle$ 85PS(25)201 $\rangle$. The *N*-phenyl derivative was prepared in 49% yield, also as an isomer mixture. The major isomer

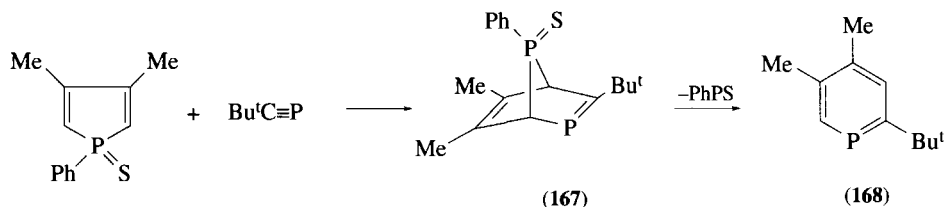
from these reactions appears from NMR studies to have the P-substituent *syn* to the C=C bridge, as in (165).



Diels–Alder additions (*endo*) were also reported to occur with maleic anhydride to give a single isomer (166), and with dimethyl ethynedicarboxylate, although in the latter case the adduct was unstable and lost the P bridge (Equation (33)) <76T2427>. Yields were not reported.



The triple bond of phosphalkynes also acts as a dienophile with phosphole sulfides, giving a cycloadduct (167) that readily loses the bridging phosphine sulfide group and is converted to a phosphinine derivative, (168) (Scheme 34) <86ZN(B)931>. The reaction has practical value as a phosphinine synthesis.

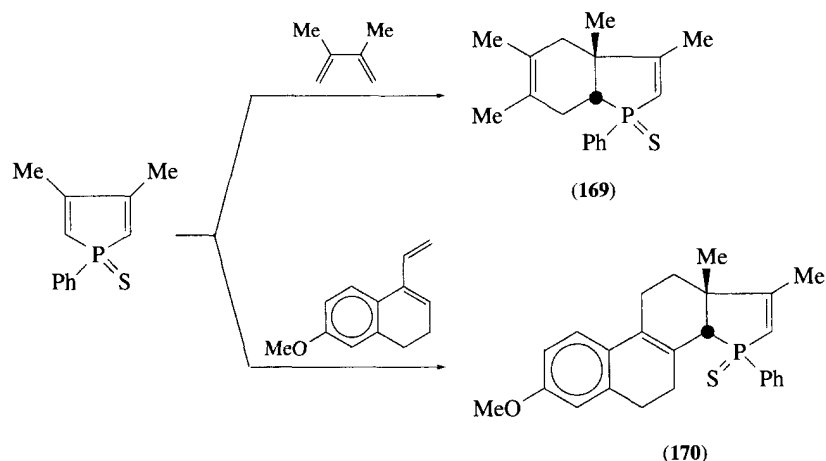


Scheme 34

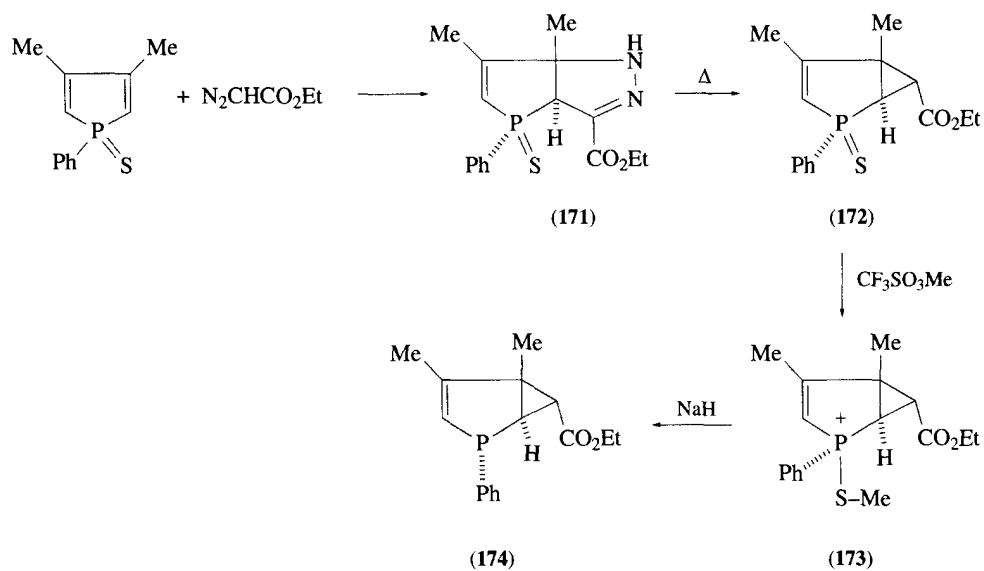
3,4-Dimethyl-1-phenylphosphole selenide gave adducts with maleic anhydride (49%) and N-phenylmaleimide (72%; <85PS(25)201>). With the triazolinediones, the addition products fragmented easily.

In behavior unlike that known for phosphole oxides, phosphole sulfides also act as dienophiles in reaction with dienes, thereby giving valuable cycloadducts such as (169) and (170) (Scheme 35) <76T2427>. The latter is a member of a rare class of phosphorus heterocycles describable as phosphasteroids.

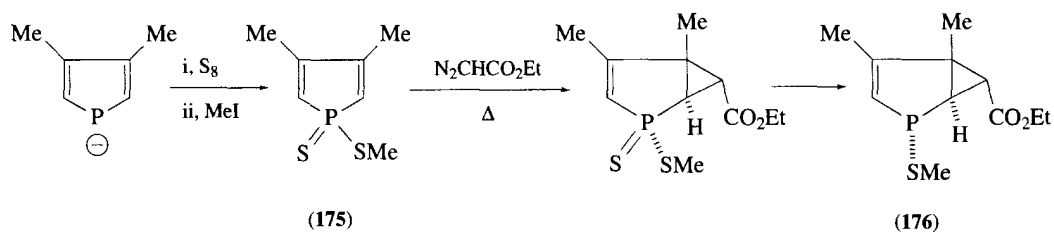
Reaction at one of the double bonds of phosphole sulfides also takes place with diazo compounds. Thus, ethyl diazoacetate condensed over a period of 45 days with phosphole sulfide (161) to give the cycloaddition product (171) in 54% yield <91JOC4031>. A single isomer was obtained; the regiochemistry was established by ¹H and ¹³C NMR techniques to be the same as that proposed in the work in 1989 on phosphole oxides <89TL3525>. When the reaction was attempted at a higher temperature (150 °C), nitrogen was lost and the cyclopropano derivative (172) resulted, in 69% yield. The same product was formed when the initial adduct (171) was heated under similar conditions (Scheme 36). The cyclopropano derivative (172) can be viewed as a sulfide of a homophosphole, and was converted to the homophosphole (174) in 74% yield by a reduction procedure that involved first conversion to the S-methyl derivative (173) and reaction with NaH. Reduction of (172) to (174) was also achieved with Bu₃P or Zn/Hg. A similar series of reactions was applied to the dithiophosphinate (175), giving homophosphole (176) (Scheme 37). The homophospholes are useful as precursors on thermolysis of phosphinines.



Scheme 35



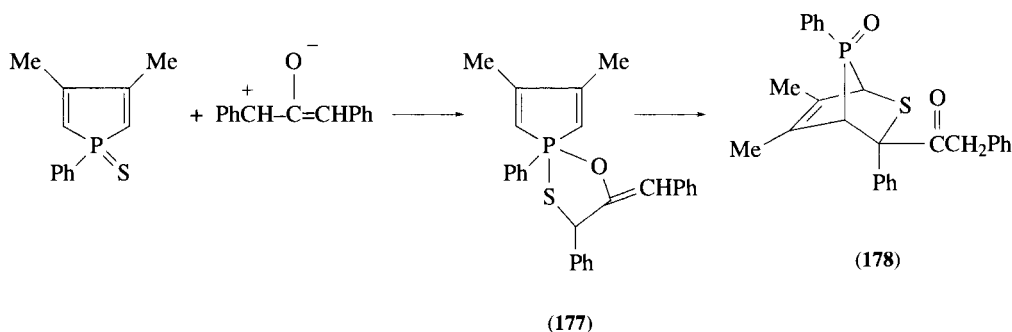
Scheme 36



Scheme 37

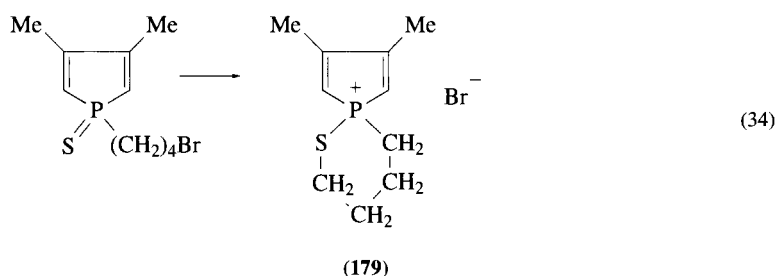
The sensitivity of S to electrophilic reagents proved to be a complication when an attempt was made to perform cycloaddition with the oxallyl cation, which had worked well with phosphole oxides. The reagent attacked on S, presumably to give a spiroposphorane, such as (177), which rearranged to leave oxygen on phosphorus as in (178) (Scheme 38) (31%; <75T53>). Other products

observed were the dimer of the phosphole oxide and the cycloadduct of the oxide with the oxyallyl cation.

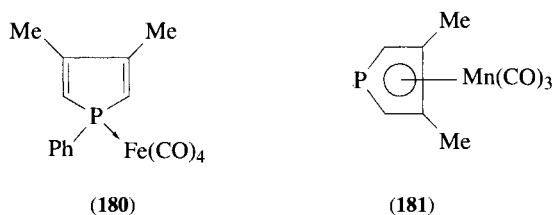


Scheme 38

The reactivity of the sulfur atom to attack by electrophiles is also revealed by the intramolecular quaternization of the bromoalkyl derivative (179) (Equation (34)) <81JOC4386>.



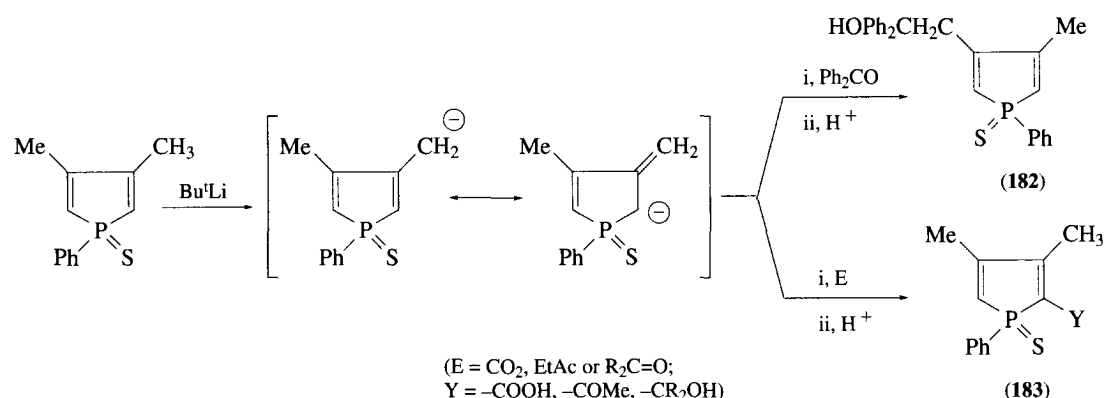
In addition to the simple removal of sulfur to generate the phosphole (Bu_3P or Zn/Hg), reaction at sulfur occurs with other reagents, and of special value is that taking place with certain coordination complexes. With $\text{Fe}_2(\text{CO})_9$, phosphole sulfide (161) underwent a reduction-complexation process that resulted in the formation of the σ -complex (180) <77JOM(136)241>. With $\text{Mn}_2(\text{CO})_{10}$, phosphole sulfide (161) underwent both loss of sulfur and of phenyl, giving rise to the phosphacycmanthrene derivative (181) <79JOM(165)129>.



One of the most valuable reactions of 3,4-dimethyl-1-phenylphosphole sulfide is that with strong bases such as the butyllithiums <73TL3255, 74T3127>. A proton is abstracted from a ring methyl group, and the resulting anion is resonance-delocalized. Attack of electrophiles can then occur either on the exocyclic carbon (as with $\text{Ph}_2\text{C}=\text{O}$ to form (182)) or at the 2-position of the ring to form (183) (Scheme 39). It is of particular importance when attack occurs on the ring (as with CO_2 , ethyl acetate, and certain ketones; a later report <92OM1411> showed that Ph_2PCl also reacted at this position) and a proton migration follows to regenerate the methyl group, for this constitutes a technique for the direct introduction of functional substituents on the phosphole sulfide ring. Removal of sulfur then gives the functionalized phosphole. The full scope of this process is described in Sections 2.15.12.2 and 2.15.12.3.

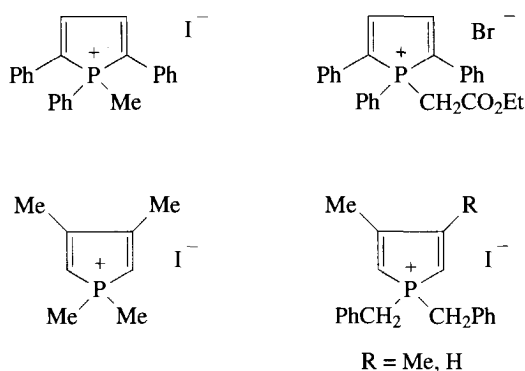
2.15.6.3 Phospholium Salts

Alkyl halides react with phospholes to give crystalline phospholium salts; many such derivatives have been prepared. While some of these are stable as monomers, others proceed to dimerize

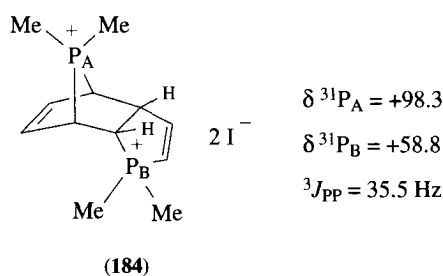


Scheme 39

during formation or on heating of the phospholium salts <73JOC1954, 79OMR442>. Some examples of monomeric phospholium salts are shown below <65JCS218, 73JOC1954>.

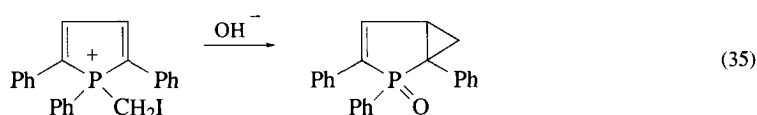


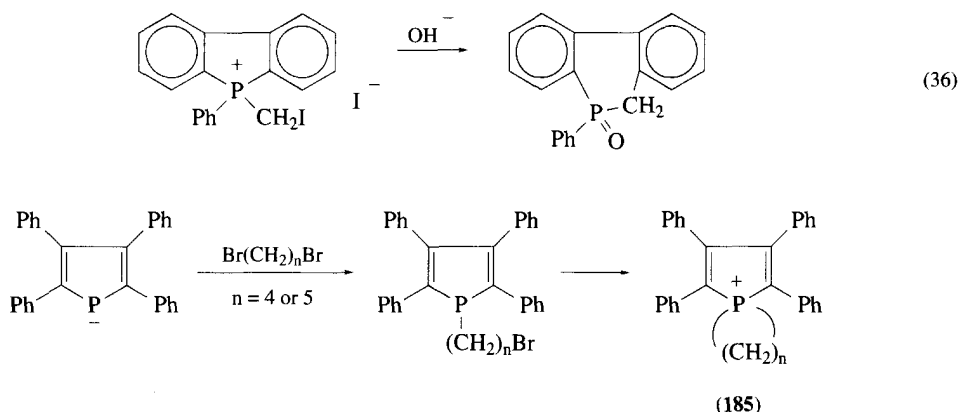
The dimeric products have the typical Diels–Alder structure as in (184); the bridging P atom possesses quite distinctive ^{31}P NMR characteristics.



As is discussed in Section 2.15.6.5, P-alkylphospholium ions react with strong bases to give ylides which participate in Wittig reactions. Other reactivity of the ring system is for the most part unexplored, an exception being the reaction that occurs when iodomethyl salts are treated with aqueous base (Equation (35)) <71CJC879>.

In the dibenzophosphole system, ring expansion to dihydrophosphinines occurs (Equation (36)) <69JCS(C)252>. A novel spirocyclic ion (185) has been formed by an intramolecular quaternization (Scheme 40) <71T3523>.





Scheme 40

2.15.6.4 Iminophospholes

The imino group may be attached to phosphorus of the phosphole ring by conventional methods of phosphine chemistry to form the family of iminophospholes, which may be considered to be nitrogen analogues of ylides (Equation (37)).



The best known of these methods is the reaction of azides with phosphines (the Staudinger reaction), and this has been applied both to dibenzophosphole <64CB741> and to several 1,2,5-triarylphospholes <72CC1242, 74JCS(P1)1694>. The reactions with the phospholes were conducted at 100°C in toluene and gave generally good yields of the iminophospholes. The triarylphospholes also reacted with nitrenes from various sources to give iminophospholes.

The iminophospholes are crystalline compounds, of widely varied color. They have good stability except on exposure to water. It is notable that the color differences disappear in solution, and in general the iminophospholes have strong UV absorption maxima at 210 and 380–405 nm.

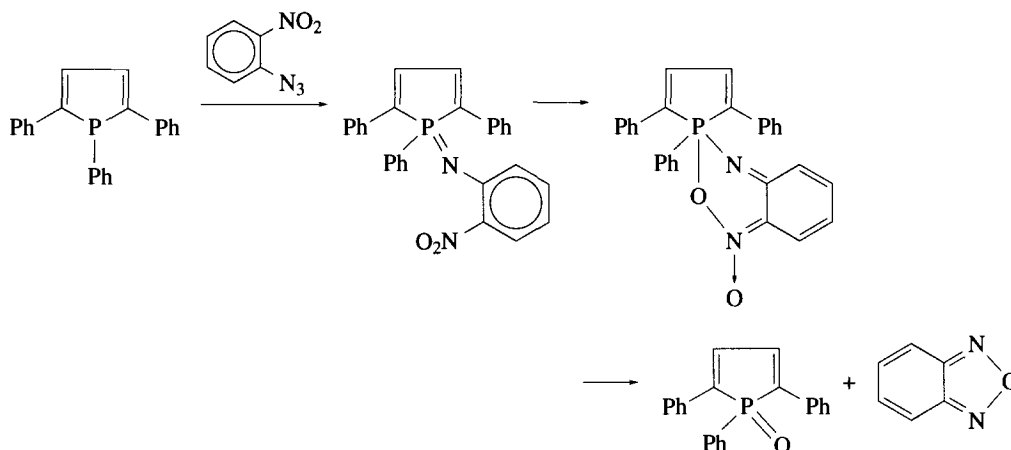
Iminophospholes have high electrophilicity at the phosphorus atom, and are easily hydrolyzed to phosphole oxides. The electrophilic activity also is evident in an unusual reaction discovered for iminophospholes with *o*-nitroaryl substituents on nitrogen. These compounds on being heated at 160°C in mesitylene gave benzofurazan derivatives (32–65% yields) along with the phosphole oxide <74JCS(P1)1694>. The mechanism proposed involved attack of an oxygen of the nitro group on the electrophilic phosphorus to form a five-coordinate intermediate which collapsed to products. The reaction seems to be peculiar to iminophospholes, since some noncyclic iminophosphines with *o*-nitroaryl N-substituents did not give these products. Ring strain in the phosphole derivatives, which is diminished in the five-coordinate state, may account for their higher reactivity (Scheme 41).

2.15.6.5 Phosphole Ylides

Phosphole ylides may be represented by the resonance structures of Equation (38).

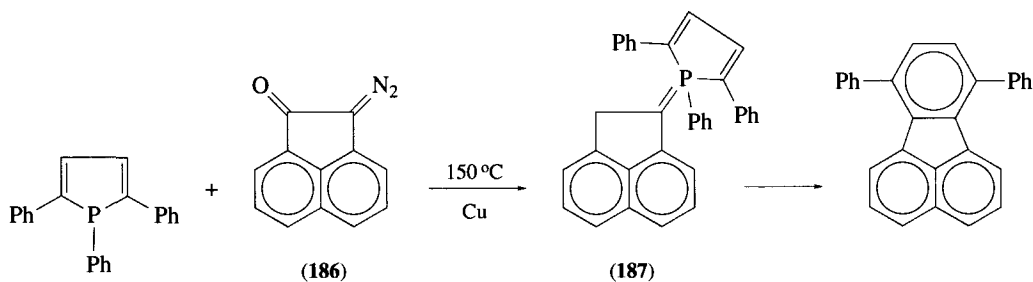


It was early recognized <72CC1242, 74JCS(P1)1694> that carbenes, generated from diazo compounds, added to phosphorus to produce the ylide structure. 1,2,5-Triphenylphosphole has been the usual participant in this reaction (Schemes 42 and 43). When some multicyclic diazo compounds were

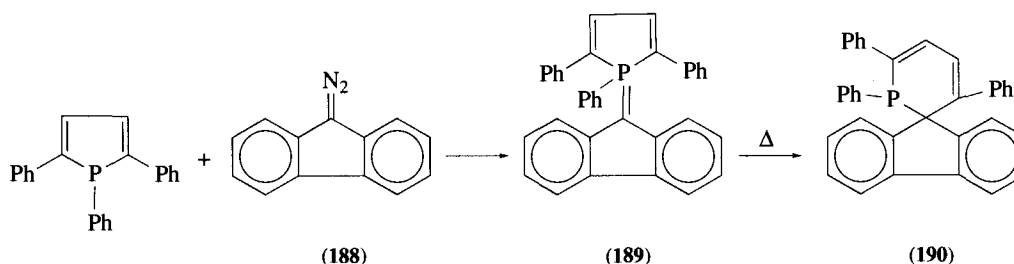


Scheme 41

used (e.g. **(186)** and **(188)**) as sources of carbenes, the ylides were thermally unstable. Compound **(187)**, formed from **(186)**, decomposed with loss of phosphorus to give 7,10-diphenylfluoroanthene <74CC902, 78LA74>; ylide **(189)** from **(188)** rearranged by a novel Stevens-like process to spiroposphinine **(190)** <84CC1217>.

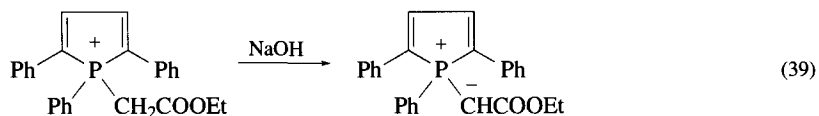


Scheme 42

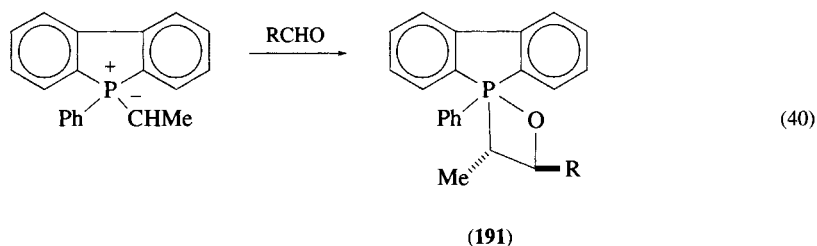


Scheme 43

Phosphole ylides have also been prepared by the common procedure of abstraction of an α -proton from P-alkyl phospholium salts (Equation (39)) <65JCS2184>.

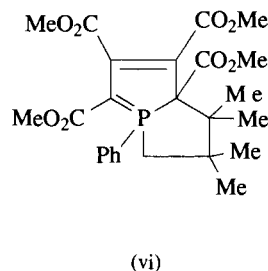
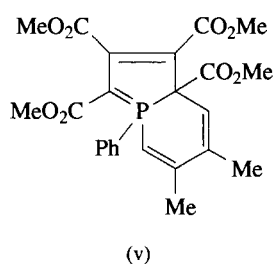
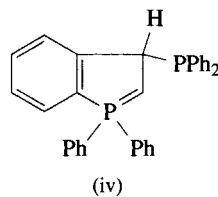
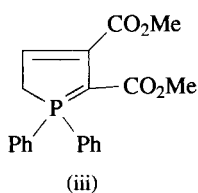
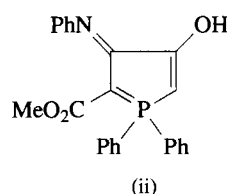
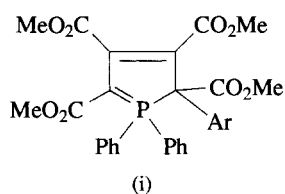


Ylides derived from benzophospholes have attracted interest as participants in the Wittig reaction, where they give exceptionally high ratios of *E*- to *Z*-alkenes (90:10 or better) <87TL3445, 93JOC1985>. They are also of interest in giving rather stable oxaphosphetane intermediates **(191)** in this reaction (Equation (40)); these five-coordinate phosphole derivatives are discussed in Section 2.15.7.3.



Most of the interest in phosphole ylides has rested in the reactivity external to the ring, and little is known about the character of the phosphole ring itself.

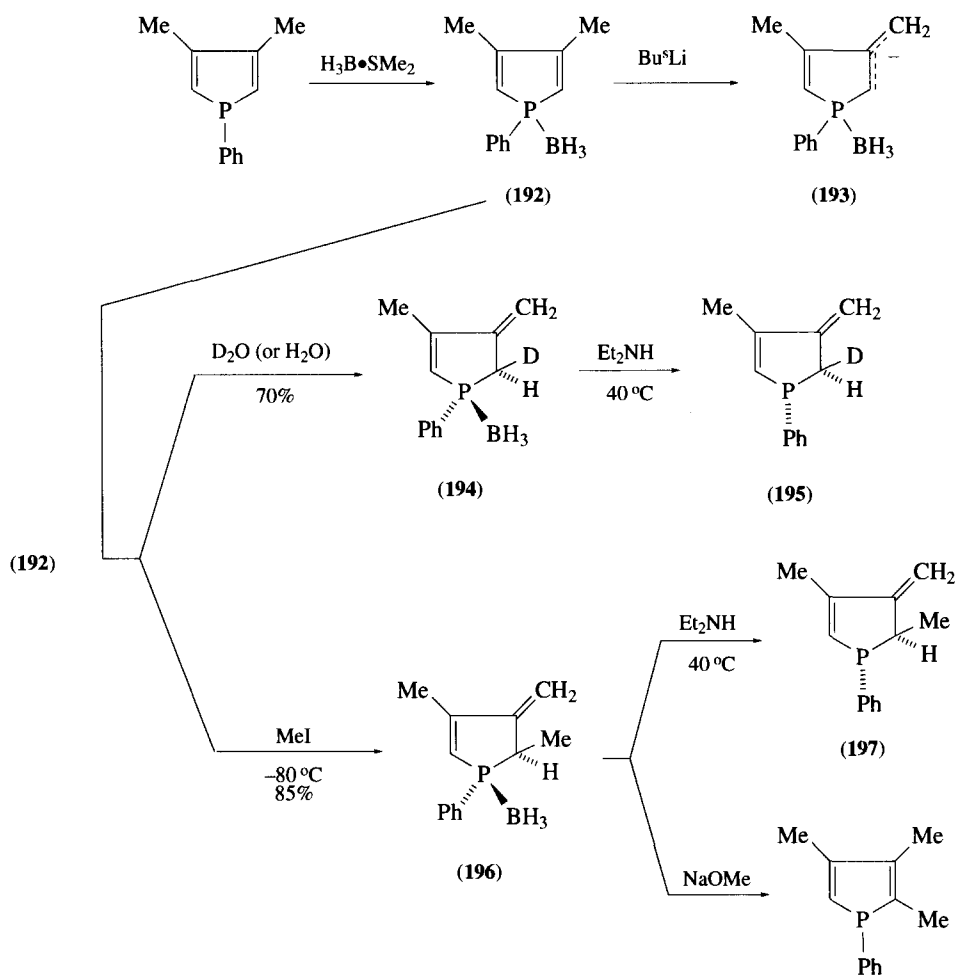
Ylides of another type are also known, where the functionality is incorporated within the ring. These endocyclic ylides, frequently the result of complex reactions, are highly reactive to water, and virtually nothing is known about the chemistry of the ring system. The structures of this type that are known are summarized in structures (i) to (vi), the leading references being: (i) <69JCS(C)1100>; (ii) <86CC1574>; (iii) and (iv) <72CJC3625>; (v) <78JHC1319>; (vi) <70JCS(C)1855>.



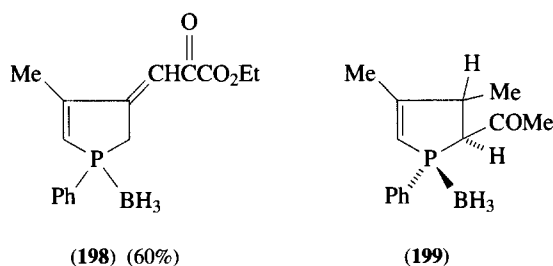
2.15.6.6 Phosphole-Boranes

A Lewis salt (192) of 3,4-dimethyl-1-phenylphosphole with BH_3 is formed by reaction with $\text{H}_3\text{B} \cdot \text{SMe}_2$. The reaction occurs rapidly at 0°C and is near quantitative <94OM925>. The salt can be deprotonated at a methyl group at -80°C with *sec*-butyllithium; the allylic ion (193) so produced reacts at the 2-position on protonation or methylation, giving the novel phosphine complexes (194) and (196) that are isomeric with phosphole complexes and can generate the free phosphole isomers (195) and (197) on decomplexation (Scheme 44).

Silylation and stannylation also occurred at the 2-position, but quenching allylic anion (193) with diethyl oxalate gave the product of γ -attack (198). With ethyl acetate, attack occurred at the 2-position, but the product underwent reduction to give the 2-phospholene complex (199).



Scheme 44

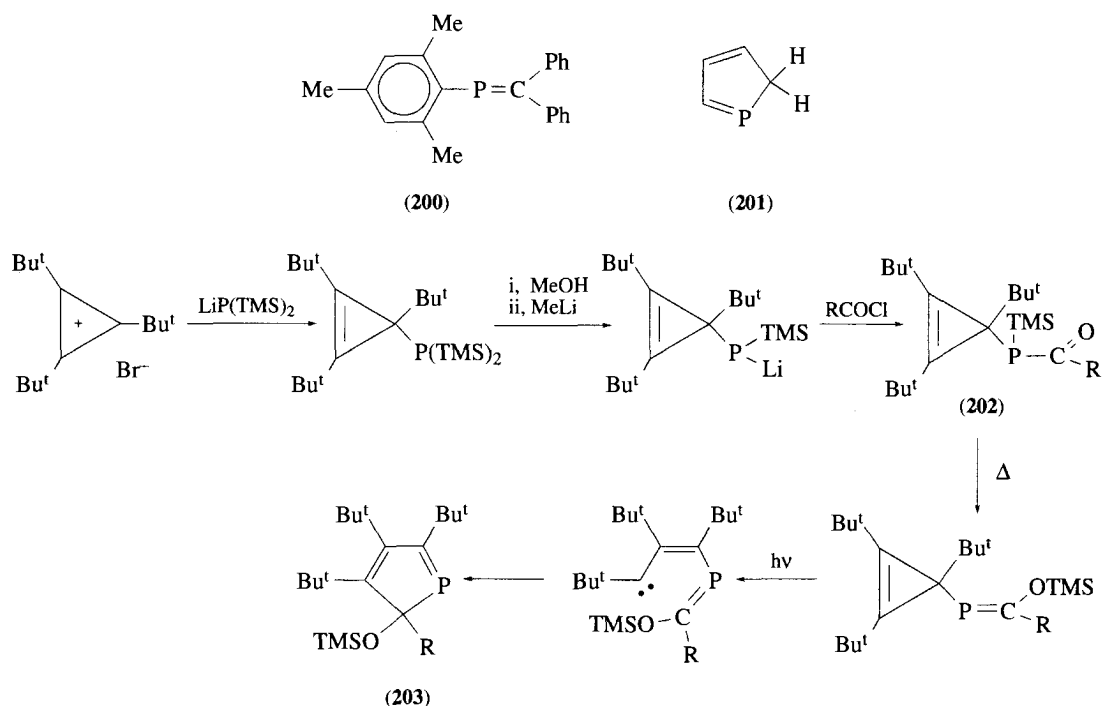


2.15.7 REACTIVITY AND SYNTHESIS OF PHOSPHOLES WITH C.N.-2 AND C.N.-5

2.15.7.1 2H-Phosholes

The $\text{C}=\text{P}$ double bond was unknown until the discovery of the delocalized phosphinines in 1966 [66AG(E)846], and it was not until 1978 that the first stable compound (200) with an isolated double bond was discovered [78JA4886]. The 2H-phosphole structure (201) requires such a bond, and although compelling evidence for the fleeting existence of 2H-phosholes accumulated after 1981 (see below), it was not until 1987 that the first example of a stable monocyclic 2H-phosphole (203) was reported [87JOM(332)C1]. As is common in studies of low-coordination phosphorus species, the ring was heavily substituted by very large substituents to endow the system with kinetic stability. The synthesis is outlined in Scheme 45; the migration of TMS from P to O in (202) is a known technique for creating the $\text{C}=\text{P}$ double bond.

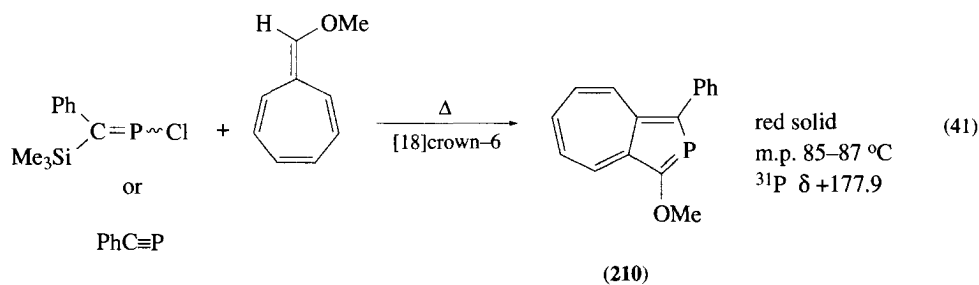
2H-phosphole (203) was a yellow oil that could be distilled by the Kugelrohr technique at 170–

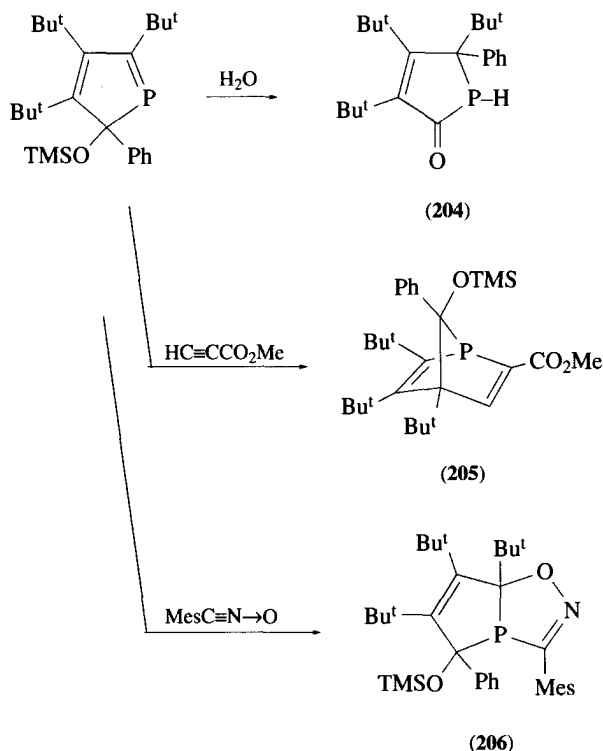


Scheme 45

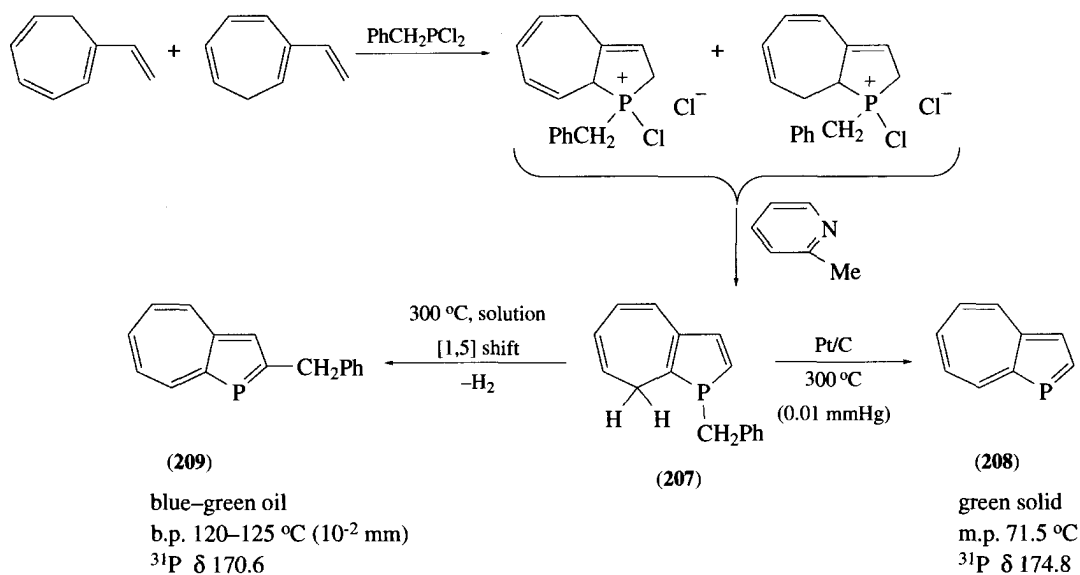
180°C (10⁻³ mmHg). In C₆D₆, the ³¹P NMR shift was $\delta +159$, entirely consistent with the presence of a C=P double bond, and this was confirmed by the ¹³C NMR spectrum, which showed phosphorus to be directly coupled to an *sp*² carbon (δ 194.7, ¹*J*_{PC} 68.7 Hz) and to an *sp*³ carbon (δ 37.1, ¹*J*_{PC} 51.4 Hz). The 2*H*-phospholes where the 2-phenyl was replaced by 2,4,6-trimethylphenyl, 2,4,6-trimethoxyphenyl, and methyl were also made in good yield by this method. These 2*H*-phospholes are thermally stable but very sensitive to water and were hydrated on attempted chromatography on "Kieselgel" to form the phospholanone (204). Other reactions observed (Scheme 46) included a Diels–Alder cycloaddition with methyl propiolate (giving (205)), and a 1,3-dipolar cycloaddition with mesityl nitrile oxide that formed (206).

The 2*H*-phosphole moiety has also been stabilized by incorporation into some phosphazulene derivatives synthesized in the Märkl laboratory. The structures ((208), (209), and (210)) are shown in Equation (41) and Scheme 47. These remarkable compounds were of good stability; in fact the molecular ion was the base peak in the mass spectrum of (209) <83AG(E)57>. All showed strong UV absorption that was appropriate for an azulene derivative, and strongly deshielded ³¹P <92TL5495> due to the double bond. Some other properties are noted by the structures. The methods of synthesis of the phosphazulenes are shown in Scheme 47. In the synthesis of phosphole (207), conventional reactions (McCormack cycloaddition, dehydrohalogenation with α -picoline) were remarkably applicable. When (207) was heated at 300°C (0.01 mm Hg) in the gas phase over Pt/C, the expected elimination of toluene occurred forming phosphazulene (208) <92TL5495>. Earlier it had been noted that when (207) was heated at 300°C in diphenyl ether or xylene, the familiar [1,5] sigmatropic shift of the P-benzyl group occurred; aromatization to phosphazulene (209) by elimination of H₂ followed. Phosphazulene (210) <83AG(E)879> was prepared more directly in a simple condensation of a phosphanitrile (or equivalent) with a heptafulvene.





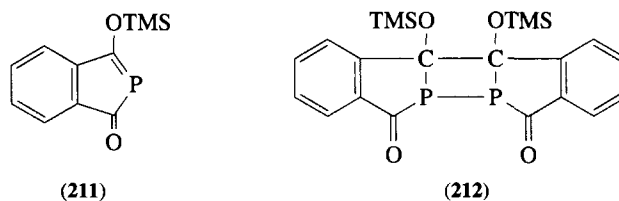
Scheme 46



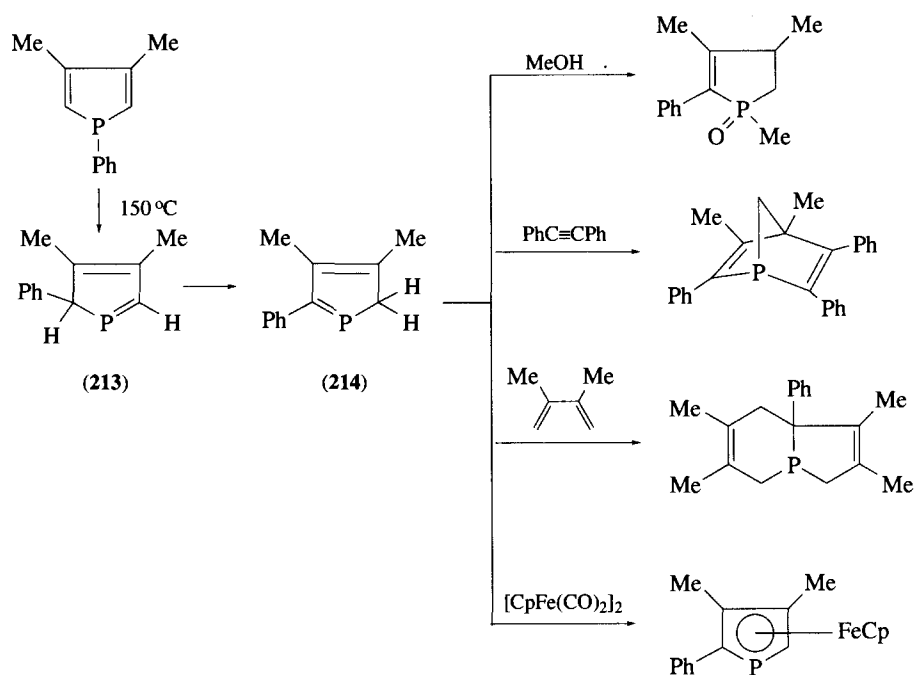
Scheme 47

Another type of bicyclic 2*H*-phosphole has the 2-keto structure (**211**). It was synthesized by two groups ^{83ZOB2796, 86CB2915}, and found to be stable and spectroscopically observable up to about –20 °C. A novel [2 + 2] dimerization ensued; the structure of this product (**212**) was elucidated by x-ray analysis in the later study.

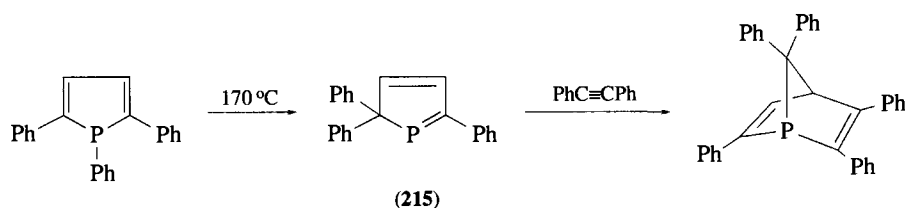
As noted, the existence of 2*H*-phospholes as fleeting intermediates had been placed on firm ground (by work of the Mathey laboratory) that preceded the actual isolation of a stable monocyclic 2*H*-phosphole. Some of this work is described in Section 2.15.7.1; to summarize, it was discovered that at high temperatures a phenyl group on phosphorus of a phosphole migrated in a [1,5] sigmatropic



shift to an adjacent carbon, giving the highly reactive, non-observable *2H*-phosphole (e.g. **(213)** and **(215)**); a proton shift ensued in **(213)** to give **(214)**) (Scheme 48) <81JA4595>. The *2H*-phospholes could be trapped as noted in Scheme 49. Computational studies by Bachrach support the notion that *1H*-phospholes rearrange to the *2H*-isomers, which are of lower energy <94JOC5027>.

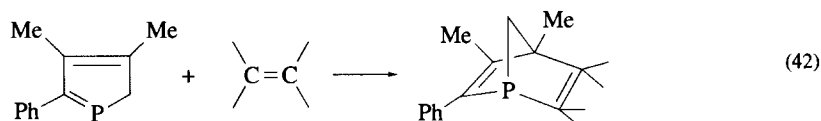


Scheme 48



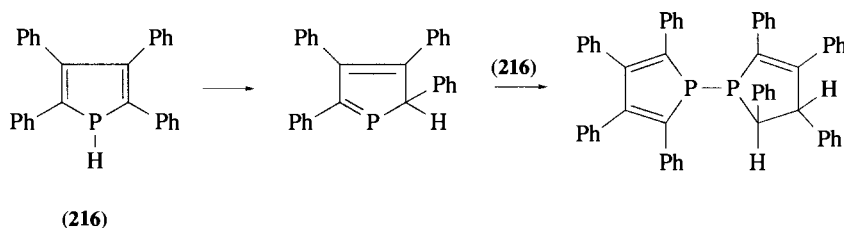
Scheme 49

It was later noted <89JOC4754> that activated alkenes reacted with the *2H*-phospholes (faster than with the *1H*-isomer) to form the very valuable 1-phosphanorbornene system (Equation (42)).



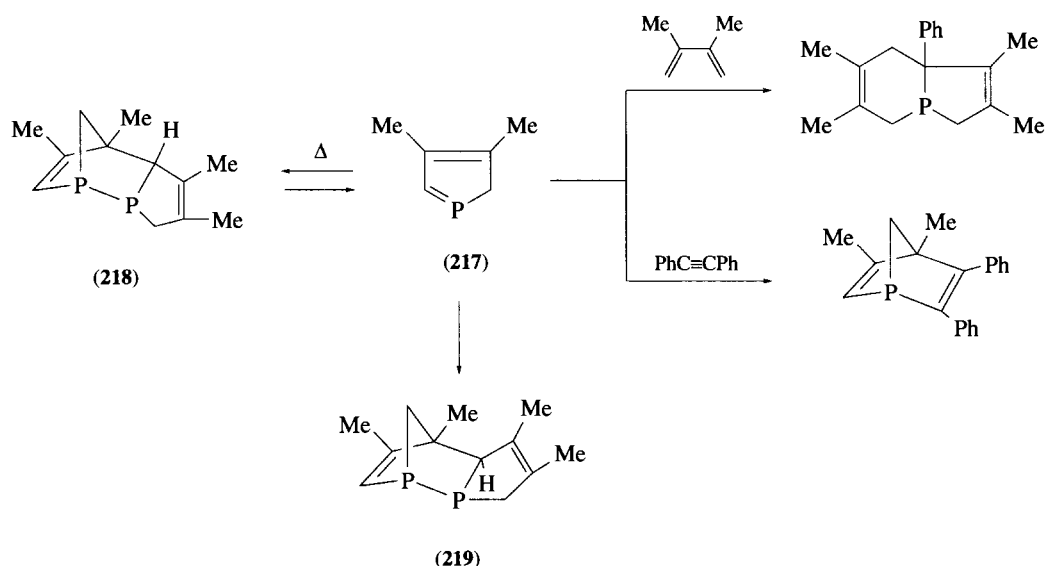
1H-Phospholes, prepared by protonation of phospholide ions at -90°C , behave in a similar manner; their rearrangement is so fast that it occurs on gradually raising the temperature of the protonation mixture <82TL511, 83JA6871>. The *2H*-phospholes rapidly form dimers as discussed in Section 2.15.5.8.

If the 1*H*-phoshole is heavily substituted, as in (216), stability and lifetime of the materials are increased, and an addition of the 1*H*-phoshole to the 2*H*-phoshole occurs (Scheme 50).



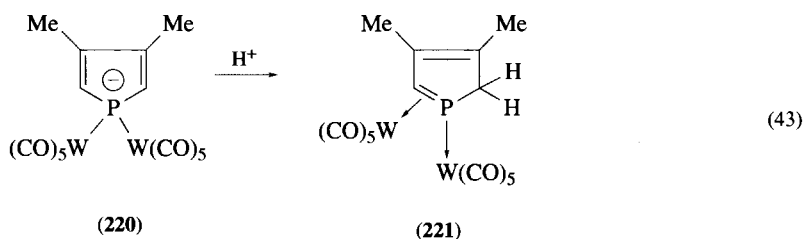
Scheme 50

The important observation was made that the dimer (e.g. (218) from 2*H*-phoshole (217)), whose structure was proved by x-ray analysis, thermally rearranged to the *exo* isomer (219), implying reversibility in the dimerization process <82CC1272, 83JA6871>. It was found possible to conduct the retro-dimerization in refluxing toluene, and when dienes or ethynes were present, the 2*H*-phoshole could be trapped (Scheme 51). Adducts of alkenes with 2*H*-phosholes showed similar reversibility <89JOC4754>.



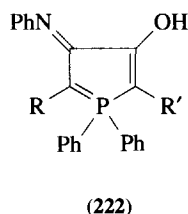
Scheme 51

It was also shown by the Mathey laboratory that the 2*H*-phoshole moiety could be preserved in the form of a chromium or tungsten (e.g. (221)) complex <84JA826>. This was accomplished by preparing a phospholide ion complex (e.g. complex (220)) and treating it with acid at room temperature; P-protonation occurred and the usual rearrangement to the 2*H*-phoshole followed (Equation (43)). This is an excellent example of the power of conducting reactions in the coordination sphere of a metal.



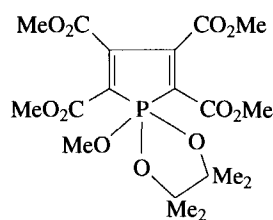
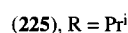
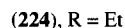
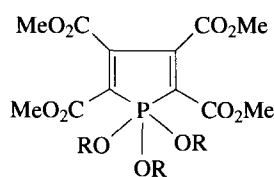
2.15.7.2 3*H*-Phospholes

There are no examples yet known of 3*H*-phospholes; in fact, the only stable structure based on a 3*H*-phosphole seems to have the ylidic structure **(222)** <86CC1574>.

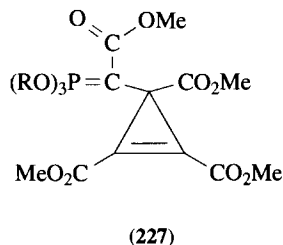


2.15.7.3 Five-Coordinate Phospholes (Phosphoranes)

The phosphole moiety has been incorporated in the trioxophosphorane structures **(223)**–**(225)**. In this coordination state, the ^{31}P shift is typically strongly negative and the experimental values for these compounds support the structures, as do other spectral data. Compounds **(223)**–**(225)** are stable in solution at -10°C but decompose at room temperature (**(223)** <81CC420, 81TL3533>; **(224)** and **(225)** <81CC420>). However, the bicyclic phosphorane **(226)** was a stable crystalline solid, and its structure was confirmed by x-ray analysis <82AX(B)3024>.



Such structures are not made from the phospholes as reactants but are prepared indirectly by condensation reactions of alkynic derivatives with trialkyl phosphites <81CC420, 81TL3523>, whereupon a cyclization process occurs that gives the phosphole ring. In the reaction of dimethyl ethynedicarboxylate with trialkyl phosphites, an intermediate has been detected at -50°C that has structure **(227)** <84JCS(P1)1627>.

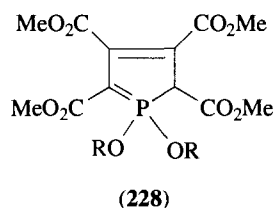


At room temperature, the monocyclic phosphoranes undergo rearrangement by a [1,5] sigmatropic migration of an alkoxy group from phosphorus to an α -ring carbon, thus creating an ylidic structure **(228)** <81TL3533, 84JCS(P1)1627>.

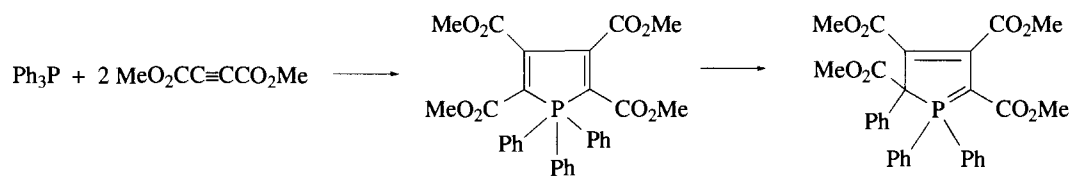
It has also been shown that dialkyl phosphonites give similar phosphoranes with dimethyl acetylene dicarboxylate <87PS(29)123>. These products are less stable than those from phosphites, and undergo the [1,5] rearrangement at -50°C .

Transient intermediates with five-carbon substituents attached to phosphorus have been proposed in related reactions between phosphines and acetylene dicarboxylates <69JCS(C)1100, 71PS(1)139> but

Phospholes

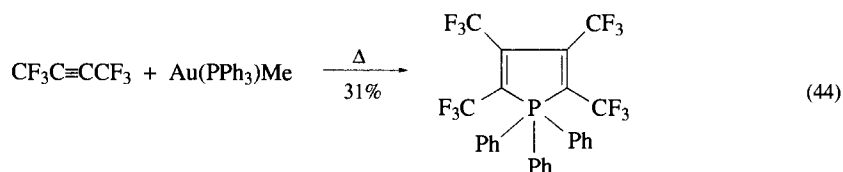


are not directly detectable $\langle 93\text{CJC}1873 \rangle$, such reactions giving the ylide from the [1,5] rearrangement process (Scheme 52).



Scheme 52

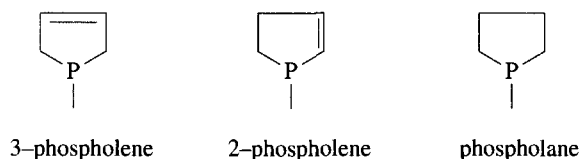
However, a C.N.-5 phosphole with all-carbon substituents has been prepared by another method $\langle 72\text{JCS(D)}102 \rangle$ (Equation (44)).



2.15.8 DIHYDRO- AND TETRAHYDRO-DERIVATIVES

2.15.8.1 General

Reduced forms of the phosphole ring system, especially the P-oxide and P-sulfide derivatives, are extremely well known and include the two possible dihydro derivatives in addition to the tetrahydro derivative.

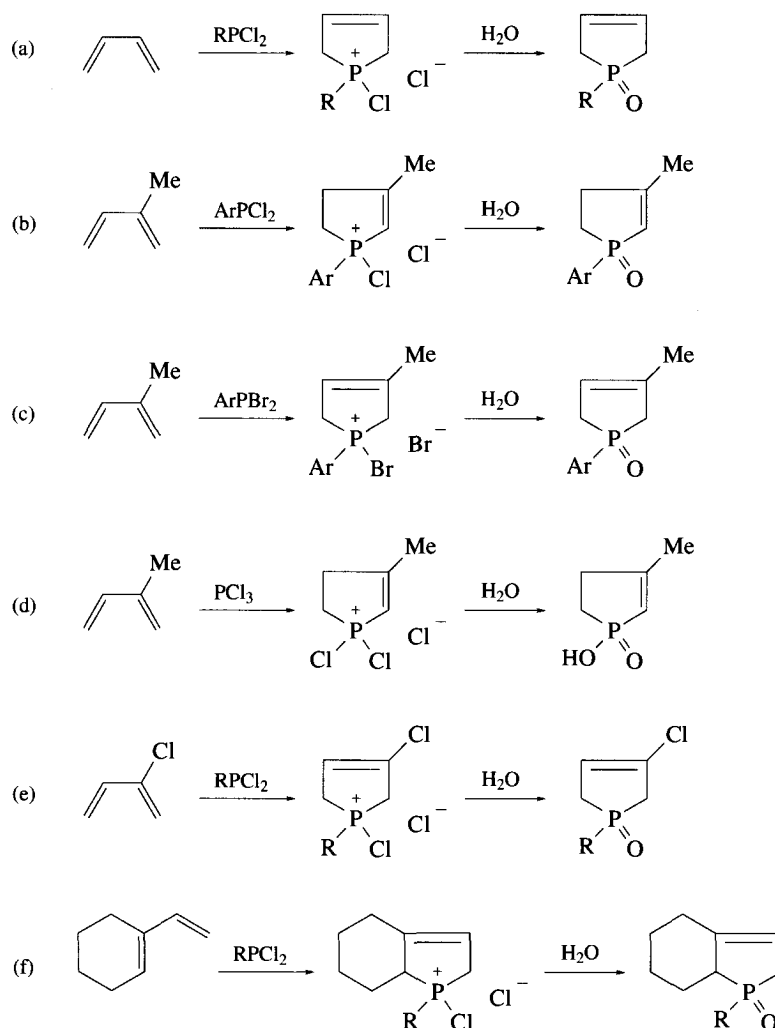


Since the chemistry is so extensive for the reduced phospholes, the expedient is taken here of presenting a broad picture with few specifics, and depending upon a 1981 review that was complete in describing synthetic methods, reactions, and ^{31}P NMR spectra up to that time $\langle \text{B-81MI 215-01} \rangle$. Some comments on new trends in syntheses and applications of the reduced phospholes are included.

2.15.8.2 Some Synthetic Approaches to Reduced Phospholes

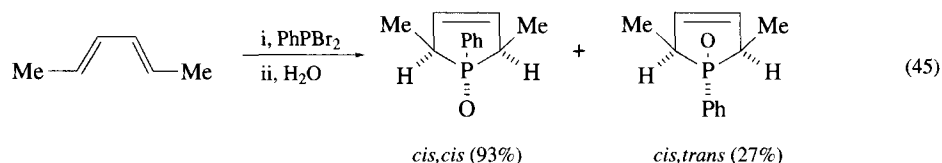
The reduced phospholes are never made directly by hydrogenation of phospholes; most commonly, the ring is created in the desired state of reduction while phosphorus is in the oxide or sulfide form, and the process is completed by removal of these atoms. For the synthesis of 2- and 3-phospholenes, the McCormack cycloaddition of trivalent phosphorus halides with dienes, followed by hydrolysis, is the method of choice for obtaining the requisite P-oxides. This process, first described in US patents $\langle 53\text{USP}2663737, 53\text{USP}2663738 \rangle$ by W. B. McCormack of the duPont Co., has

proved over the years to be of monumental importance in heterocyclic phosphorus chemistry. Some simple examples of typical syntheses are shown in Scheme 53.

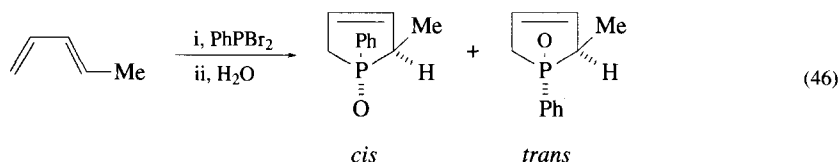


Scheme 53

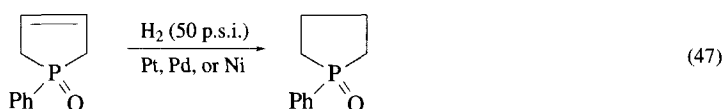
The McCormack reaction has very great versatility, and a considerable variety of phospholene oxides, monocyclic as well as multicyclic, have been prepared with it. Not only do phosphonous dihalides participate, but also PCl_3 and PBr_3 , and some ester derivatives of these, are useful reactants. As the examples show, with some combinations of reactants the 3-phospholene system is favored, but the intermediate cycloadduct and the 3-phospholene oxide can enter into rearrangement processes that lead to the 2-phospholene as the major product. Reaction mixtures are always dominated by one of the two regioisomers, and usually can be separated with ease. Some structural limitations in the McCormack cycloaddition do exist, however. Thus, branched-chain phosphonous dihalides react very slowly (e.g. isopropylphosphonous dihalides) or not at all (e.g. *t*-butylphosphonous dichloride), and dienes that contain two substituents on a terminal carbon of the diene fail to react. Branched-chain substituents can, however, be placed indirectly on phosphorus by reaction of *P*-halophospholene derivatives with organometallic reagents. Both of these limitations appear to have a steric origin, which is especially evident from the behavior displayed by the 1,3-pentadienes (piperylenes); the *trans*-isomer reacts readily but the more crowded *cis*-isomer is virtually inert. *Trans,trans*-2,4-hexadiene is of special interest in that a mechanistic feature of the McCormack reaction is revealed by the exclusive formation of a 3-phospholene oxide with the two methyls *cis*-oriented. This implies that the cycloaddition is concerted and disrotatory, and thus may be considered as a cheletropic process, a mechanism known also for the addition of SO_2 to dienes. The phosphorus atom constitutes a third chiral center and isomers are possible as in Equation (45).



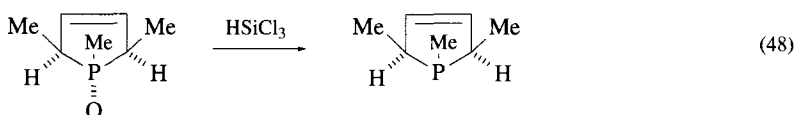
Isomers are also formed from the use of *trans*-piperylene; in Equation (46) the *cis* : *trans* ratio was 2 : 1.



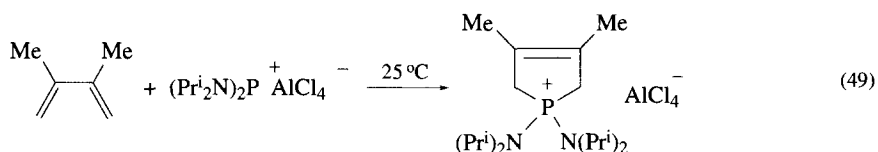
The phospholene oxides resulting from the McCormack reaction also serve as highly useful starting materials for the synthesis of phospholane oxides by simple hydrogenation techniques (Equation (47)).



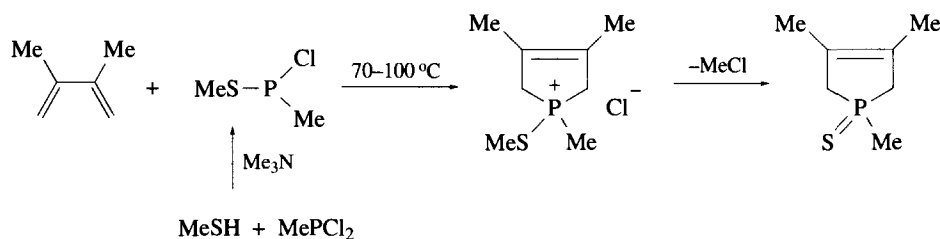
Many of the known phospholene oxides have been reduced to phosphine form, and this constitutes the best method at present for obtaining both 2- and 3-phospholenes. The latter are of special interest when the ring bears 2- or 2,5-substituents; with HSiCl_3 as the reducing agent (and some others), the stereochemistry at phosphorus in the oxide is preserved, and because of the high barrier to pyramidal inversion characteristic of phosphines there can be stable *cis,trans*-isomers where the tricovalent phosphorus acts as a chiral center (Equation (48)). Examples with several substitution patterns are known.



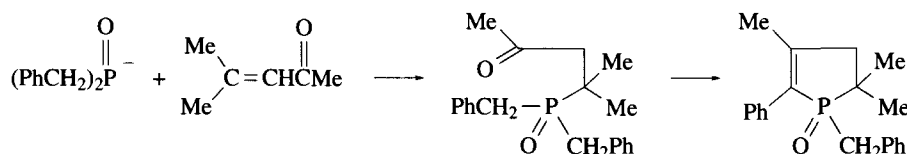
There have been some significant developments in the McCormack reaction since the 1981 review; one of some value is the discovery that performing the reaction under pressure $\langle 89\text{CC}967 \rangle$, greatly speeds up the cycloaddition and gives a more tractable solid product, and another is the application of the multivariate optimization approach to improving the yield of 1-methoxy-3-methyl-2-phospholene oxide from workup of the reaction product of isoprene and PCl_3 , which resulted in an overall yield of 90% $\langle 93\text{JCS}(\text{P}2)629 \rangle$. Other advances in the McCormack process include the introduction of two new types of dienophiles, phosphonium ions $\langle 83\text{JA}7443, 83\text{JA}7444, 86\text{IC}740, 92\text{JOC}5189 \rangle$ which react quite rapidly with dienes (Equation (49)), and alkyl (methylthio) chlorophosphine $\langle 88\text{ZN}(\text{B})257 \rangle$ (Scheme 54).



No other synthetic method for phospholene oxides approaches the McCormack reaction in simplicity and versatility. Some specialized methods do exist, however. One is shown here that allows 2,2-disubstitution in the phospholene ring, which cannot be obtained with the McCormack method (Scheme 55).

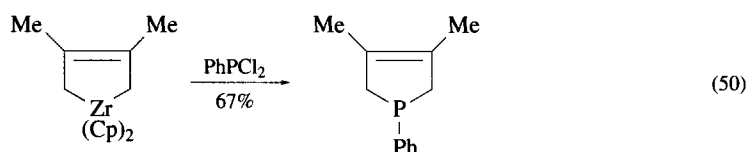


Scheme 54

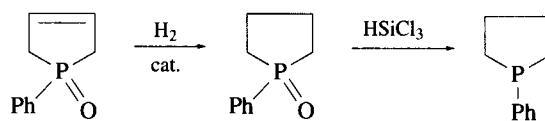


Scheme 55

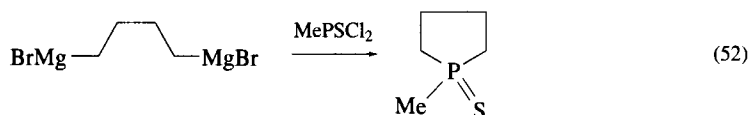
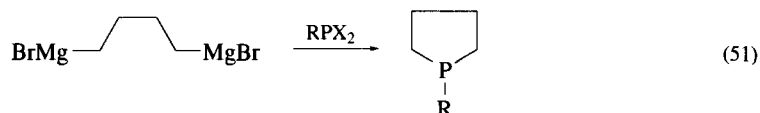
In 1988, it was discovered that 3-phospholenes can be prepared directly by the reaction of zirconium metallocycles with phosphonous dihalides (Equation (50)) <88JA2310>. The synthesis of only one phospholene (3,4-dimethyl-1-phenyl) has been reported so far, but the method appears to have a good chance of possessing valuable versatility.

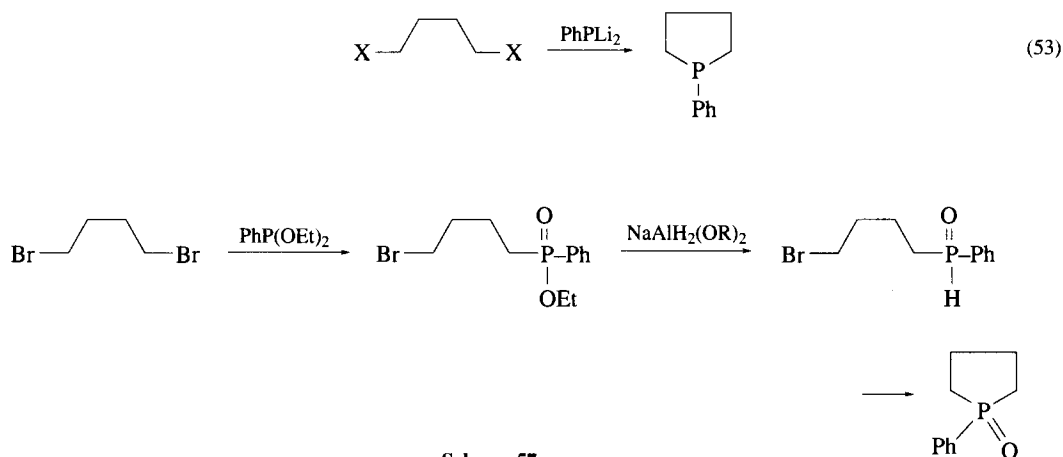


Phospholene oxides from the McCormack reaction also stand as useful precursors of phospholanes; as noted, the double bond can be easily hydrogenated, and then the oxygen removed from phosphorus with silanes, etc. (Schemes 56 and 57; Equations (51) to (53)). Phospholanes can be prepared by other cyclization methods, however, some of which are outlined below.

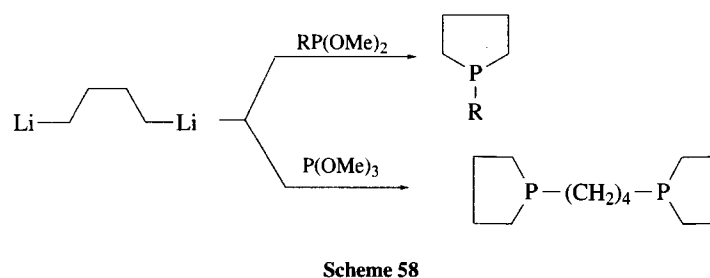


Scheme 56



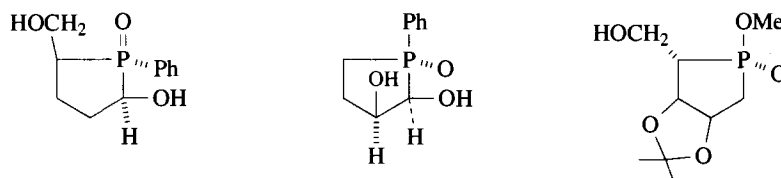


A method introduced in 1993 <93S39> involves reaction of phosphonites and phosphites with 1,4-dilithiobutane (Scheme 58).



Phospholanes and their oxides are known with a variety of functional groups on ring carbons (e.g. 2- and 3-keto, 2- and 3-hydroxy, 2,5- and 2,3-dihydroxy, 3-carboalkoxy, 3,4-epoxy, 3,4-dinitro, 3-CN, 3-R₂N, 3-OR, etc.).

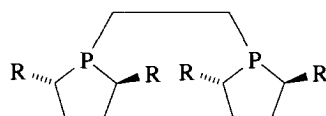
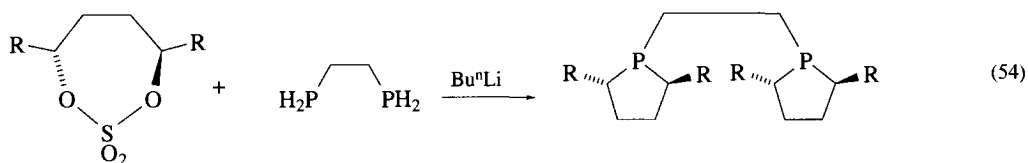
Since the late 1980s, phospholanes with appropriate multiple oxy functionalities have been considered as analogs of furanose sugars, wherein phosphorus takes the place of the ring oxygen. A number of phosphasugars have been synthesized, with defined stereochemistry at chiral carbons of the ring that in the following cases has been determined by x-ray analysis <80AX(B)2809, 88CC569, 94CL23>.



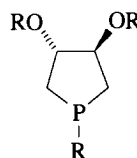
Other types of phosphasugars have also been described (e.g. in <84JOC1364, 91CAR(209)C9, 92JCS(P1)295, 93CL93, 94H(38)1449>).

Another identifiable trend in phospholane research is the construction of phosphines with chiral centers so as to create novel ligands for complexes used in asymmetric synthesis. Some examples of the new phospholanes of this type are shown in structures **(229)** <91JA8518>, **(230)** <91JOM(413)55>, **(231)** <91JOC3137>, and **(232)** <92JOC5189>. **(229)** and **(230)** are optically active and **(231)** and **(232)** are racemic, the latter two being new P-oxy structures with chiral centers.

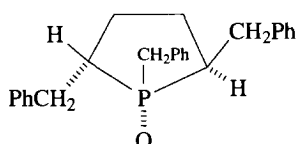
The phosphines of type **(229)** deserve special comment; they are prepared by a simple but novel route from an optically active cyclic sulfate (Equation (54)). Some complexes based on a rhodium center have been found to have excellent enantioselectivity in hydrogenation reactions.



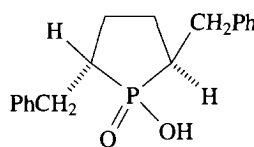
(229)



(230)



(231)



(232)

2.15.8.3 Reactivity of Reduced Phospholes and Their Oxides

The chemistry of the hydro derivatives of phospholes, with P in the three-coordinate state, is dominated by the very high sensitivity of the phosphorus atom to various oxidizing and electrophilic reagents that result in conversion to higher coordination states (usually C.N.-4). In this sense their chemistry is not exceptionally different from common tertiary phosphines. The attack of electrophiles normally occurs first at phosphorus, and the product may react further at the double bond.

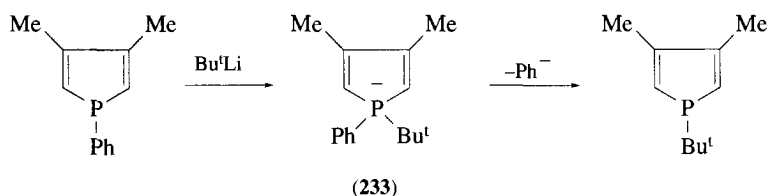
There is a rich chemistry known for phospholene oxides, too extensive to be included in this review. Most of the background information has been summarized (B-81MI 215-01). In general, the common reactions of alkene chemistry occur at the double bond, although there are some notable failures (hydration with sulfuric acid, hydroboration, oxymercuration, etc.). Some of the failures may be correctable with different conditions, as was demonstrated in the case of addition of dichlorocarbene from an NaOH-CHCl₃ mixture, which is only successful under phase-transfer conditions (87JOC3983). Another example is the failure of 2-phospholene oxides to add bromine; when the bromination is conducted in water or alcohols, bromohydrins or bromo alkoxy derivatives can be easily obtained (93HAC553). The 2,5 positions of the 3-phospholene oxide are activated and can be deprotonated by strong bases such as *n*-butyllithium; the resulting carbanions act in conventional manner and can be used in condensation reactions to prepare functionalized phospholene oxides. A novel type of compound is formed when the carbanion is reacted with Ph₂PCl; a phosphino group is installed at the 2-position of the 2-phospholene ring (85JHC513). The double bond in 2-phospholene oxides is activated by the phosphoryl group, and nucleophilic additions then are possible with such reactants as OH⁻, RO⁻, CN⁻, RNH₂, (RO)₂P(O)H, etc. The activating effect also makes it possible to perform Diels-Alder and 1,3-dipolar cycloadditions (94JOC1315) at the double bond, and permits the ready displacement of leaving groups from the 3-position by attack of nucleophiles (e.g. of Cl⁻ by NaOCH₃, H₂O, R₂NH, etc.).

2.15.9 REACTIVITY OF RING SUBSTITUENTS

2.15.9.1 Reactivity of P-Substituents

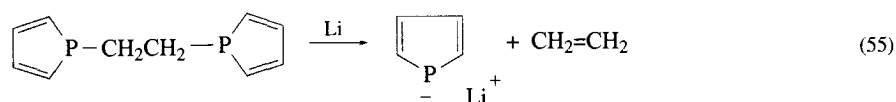
There are no examples of reactions occurring on a P-substituent that comprise retention of the substituent in a modified form. The only type of reactivity known consists of the displacement or cleavage of some P-substituents.

The phenyl group on phosphorus can be replaced by an alkyl group on reaction with alkyllithium reagents $\langle 72T4171 \rangle$. This has proved to be a process of considerable value for the introduction of the *t*-butyl group on phosphorus; 3,4-dimethyl-1-*t*-butylphosphole was prepared for the first time by this reaction. The reaction is conducted in hexane solution at room temperature in the presence of TMEDA; it is presumed to proceed by expansion of the coordination number of phosphorus with formation of a phosphoranyl anion (**233**). This anion returns to the three-coordinate phosphole by loss of phenyl anion. The phenyl group may also be displaced by *n*-butyl in similar fashion $\langle 72OMR(4)171 \rangle$ (Scheme 59).

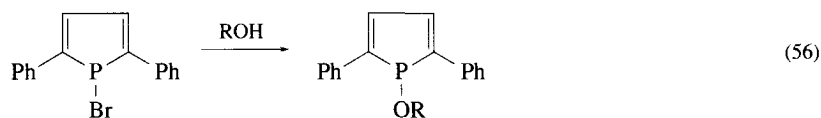


Scheme 59

Another type of reactivity displayed by P-phenyl substituents is their ready cleavage by alkali metals. This is a reaction of great importance in phosphole chemistry as the products are phospholide ions, widely used as precursors of other P-substituted phospholes (see Section 2.15.5.7). The P-methyl group can also be cleaved by metals, as can an ethylene linkage between two phosphole nuclei (Equation (55)).



Halogen on phosphorus may be displaced by alcohols (Equation (56)) $\langle 82JOM(231)361 \rangle$.



2.15.9.2 Reactivity of Substituents on Carbon

The phosphole literature in its present state is devoid of information on the reactivity of carbon-based substituents of any type attached to ring carbons. Phospholes are π -excessive heterocyclic systems, and C-alkyl substituents will not be activated; substituents such as ester and carbonyl should exhibit normal reactivity. At present there is only one known C-halo derivative of a phosphole. In this compound (2-bromo-3,4-dimethyl-4-phenylphosphole), the bromine may be replaced with lithium, and the lithio derivative used in conventional phosphine reactions with electrophiles $\langle 92BCF486 \rangle$ (see Section 2.15.12).

2.15.10 RING SYNTHESSES

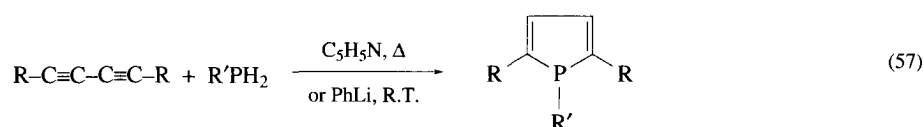
2.15.10.1 General

Construction of the phosphole ring, with one useful exception, is accomplished by methods that are quite different from those employed for the N, S, O ring systems. Because of fundamental differences in the chemistry of phosphines versus amines, none of the familiar carbonyl condensation processes are known to be applicable to phosphole synthesis. Thus an attempt to use the Paal-Knorr condensation of 1,4-dicarbonyl compounds with PhPH_2 $\langle 65JCS2184 \rangle$ and with PH_3 $\langle 88ZOB783 \rangle$ failed to give phospholes. The only successful method known to the present that is common to both the phosphole and pyrrole ring systems is the condensation of 1,3-diynes with primary phosphines

or amines. This method has some generality that makes it of value in phosphole synthesis, but the other methods that are used for direct ring synthesis are of limited scope. In fact, the best methods for obtaining phospholes are based not on direct ring closures but on the synthesis first of the 3-phospholene ring system, with subsequent conversion to phospholes. These methods are treated as ring transformations and are discussed in Section 2.15.11.

2.15.10.2 Condensation of Alkynic Compounds with Phosphines

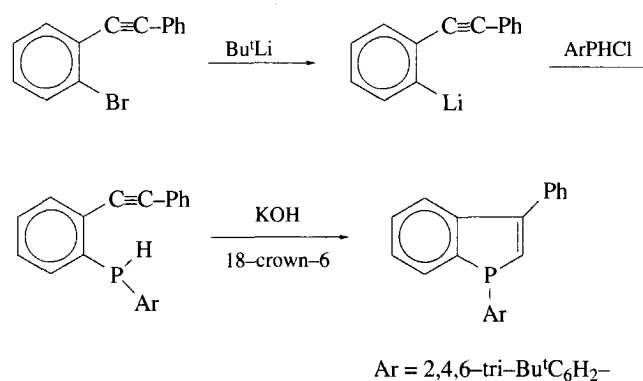
The first example of this method was discovered by G. Märkl in 1967 <67AG(E)86>, and was one of the earliest that provided variety in the substitution pattern on the phosphole ring; the others known at that time, to be discussed, were limited to the formation of C-phenyl derivatives. Märkl employed diynes with both 1- and 4-substituents, and thus created phospholes with 1,2,5-trisubstitution (Equation (57)).



The primary phosphine needed for this process may be replaced by the dimethylol derivative ($\text{RP}(\text{CH}_2\text{OH})_2$), a material that is far easier to handle but gives significantly lower yields than the free phosphine. Thus, 1,2,5-triphenylphosphole was formed in 60% yield from phenylphosphine and 1,4-diphenylacetylene, but only 20.5% from the dimethylol derivative.

Phospholes prepared in the original report as well as by later users of this method are shown in Table 12. In addition to bases, the reaction may be promoted by metal salts and by free radicals <71T5523>. Märkl had employed only symmetrical diynes in the reaction, but unsymmetrical diynes work quite well <71JA6205>.

The alkyne-phosphine condensation can be performed in intramolecular fashion, allowing the synthesis of the benzo[b]phosphole (phosphindole) ring system, as seen in Scheme 60. The phosphindoles were obtained in 90–96% yield <93TL3103>.



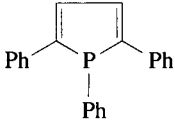
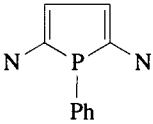
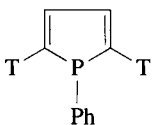
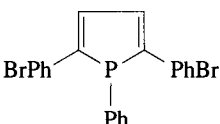
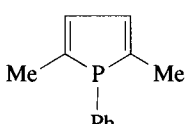
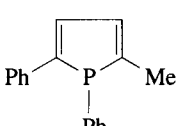
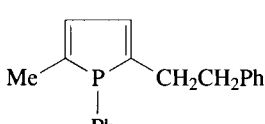
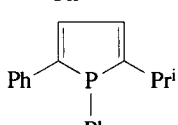
Scheme 60

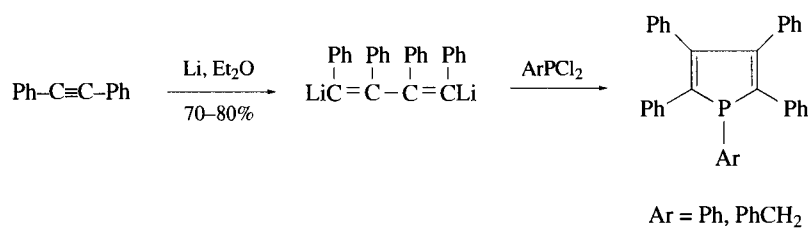
2.15.10.3 Reaction of 1,4-Dimetallic Derivatives of Dienic Systems with Phosphonous Dichlorides

This was the first approach used for the synthesis of a monocyclic phosphole, the pentaphenyl derivative, and stands as a landmark in heterocyclic phosphorus chemistry. In 1959, two laboratories reported the synthesis of this compound. In one synthesis, the dilithio derivative of tetraphenylbutadiene was reacted with phenylphosphonous dichloride (Scheme 61); the reaction proceeded in excellent yield (84% <59JA3163, 60JA5099>). Since the starting diene is easily prepared from diphenylethyne and lithium, it remains the best way to prepare this derivative, which has found use in many subsequent studies. In addition to the P-phenyl derivative, the P-benzyl compound was also reported (38% <61JA4406>).

The other synthesis of pentaphenylphosphole <59CI(L)1250> employs an iron carbonyl complex (234) as the metallic reactant with phenylphosphonous dichloride (Scheme 62).

Table 12 Phospholes from phenylphosphine with diynes^a.

$R(C\equiv C)_2R'$		Phosphole	Yield (%)	Ref.
R	R'			
Ph	Ph		60	67AG(E)86
2-Naph	2-Naph		89.5	67AG(E)86
<i>p</i> -Tol	<i>p</i> -Tol		59	67AG(E)86
2-BrC ₆ H ₄	2-BrC ₆ H ₄		—	67AG(E)86
Me	Me		51	67AG(E)86
Ph	Me		30	71JA6205
Me	PhCH ₂ CH ₂		70	71JA6205
Ph	Pr ⁱ		86	71JA6205

^aN = 2-naphthyl, T = *p*-tolyl, BrPh = 2-bromophenyl.**Scheme 61**


$$\text{Ph-C}\equiv\text{C-Ph} \xrightarrow{\text{Bu}^n\text{Li}} \text{Ph-C}(\text{Bu}^n)=\text{C}(\text{Li})\text{Ph} \xrightarrow{\text{PhPCl}_2} \text{Ph-C}(\text{Bu}^n)=\text{C}(\text{Ph})\text{P}(\text{Ph})_2$$

(235) (236)

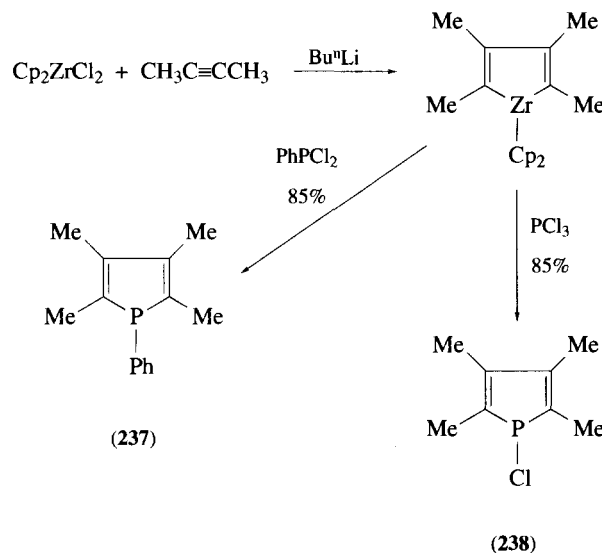
Scheme 63

Scheme 64

$$\text{C}_6\text{H}_5\text{Li}-\text{C}_6\text{H}_5 \xrightarrow{\text{ArPCl}_2} \text{C}_6\text{H}_5\text{P}(\text{Ar})\text{C}_6\text{H}_5 \quad (58)$$

A new one-step approach to main group heterocycles involves the reaction of the easily prepared zirconacyclopentadienes with halides (Scheme 65). Yields of heterocycles from zircona derivatives

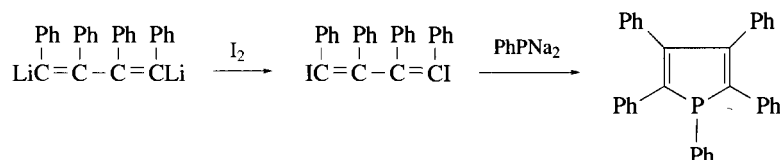
are high. The method has so far only been used for the synthesis of two phosholes ((**237**) and (**238**)), both fully substituted, but appears capable of extension to other types: (**237**) <88JA2310, 92OS(70)272>; (**238**) <89AG(E)1367>.



Scheme 65

2.15.10.4 Reaction of 1,4-Dihalobutadienes with Metallic Phosphides

The first example of this reaction type involved the conversion of the 1,4-dilithio derivative of tetraphenylbutadiene to the 1,4-diiodo derivative (70.5%) and then reaction with the disodium salt of phenylphosphine to form pentaphenylphosphole (88%) (Scheme 66) <61JA4406>.



Scheme 66

1-Phenylphosphole has been prepared by the related reaction of 1,4-dichlorobutadiene with PhPLi_2 (25%, <87AG(E)229>), and other extensions of the general method seem possible.

2.15.10.5 Reaction of Enamines with Phosponous Dichlorides

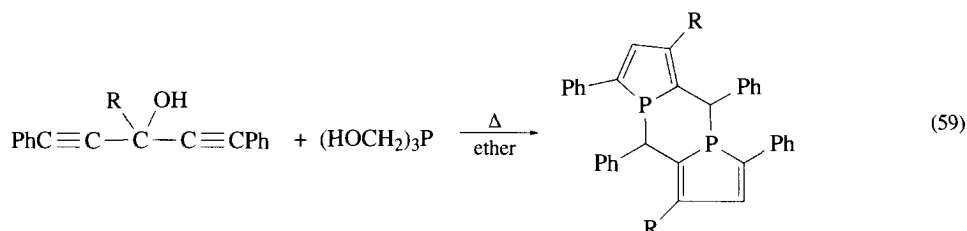
A discovery in phosphole synthesis in 1988 is that reacting a mixture of two moles of an enamine with a phosphonous dichloride at room temperature results in a one-step construction of the phosphole ring, bearing an amino substituent <88TL4581>. This reaction is considered in more detail in Section 2.15.12.5 treating functional phospholes; it constitutes an important advance in this area, and is the only source at present of aminophosholes. Further development of the method could make it of major significance in phosphole chemistry.

2.15.10.6 Creation of the Phosphole Ring in the Coordination Sphere of Metallic Derivatives

An entirely different approach for the creation of the phosphole ring is found in the field of coordination chemistry. A coordinating metal group can significantly modify the chemistry of a phosphine ligand, allowing novel and useful reactivity to develop without loss of the stabilizing metallic group. The principle has been exploited for phosphole synthesis in brilliant fashion by the

laboratory of F. Mathey. Thus, a palladium(0) complex catalyzed the insertion of an alkyne into a phosphirene when the latter was in the coordination sphere of a $W(CO)_5$ group <87TL5021>, thereby creating a phosphole. Here also the method is of special value as a source of C-functional phospholes and is discussed from that standpoint in Section 2.15.12. In the synthesis of six different phosphole complexes by this method, moderate to excellent yields were obtained (33–85%). After the phosphole complex has been created, the phosphole must be displaced from the coordinating metal; a useful procedure involves reaction with I_2 and 1-methylimidazole <84CC45>.

Tricyclic compounds containing two phosphole rings have been synthesized by the reaction of trisubstituted phosphine with certain 3-hydroxypenta-1,4-dienes (Equation (59)) <74PS(4)279>.



The products possess the novel diphosphadihydroindazene ring system. Derivatives with $R = H$, Me, and Et have been prepared in low yield (10–37%).

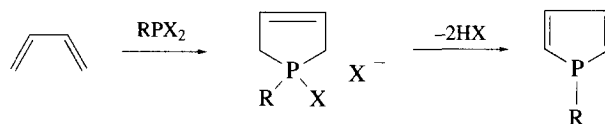
2.15.11 PHOSPHOLE SYNTHESIS BY RING TRANSFORMATION

2.15.11.1 General

The McCormack cycloaddition of dienes and phosphorus(III) halides (see Section 2.15.8.2) has made readily available a great variety of 3-phospholene derivatives, and these have figured heavily in the development of synthetic approaches to phospholes. Numerous techniques have been devised for transformations to the fully unsaturated ring system; they continue to be of great synthetic value, and in some cases these methods are clearly superior to those based on direct formation of phospholes by ring-closure methods. There are also some syntheses based on transformation of phosphole derivatives, but these methods are generally limited to modification of the substitution at phosphorus rather than at ring carbons. A useful exception is the formation of carbanions from phosphole sulfides which can then react with electrophiles; this method is of interest in forming C-functional phospholes and is described in Section 2.15.12.2.

2.15.11.2 Dehydrohalogenation of McCormack Cycloadducts (Halophospholenium Halides)

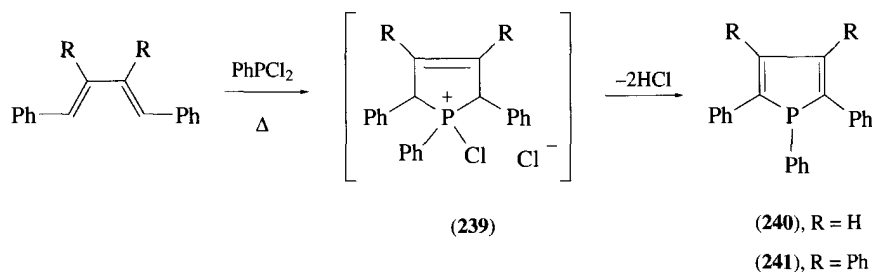
The initially formed product in the McCormack cycloaddition can be described as a halophospholenium halide (see Section 2.15.8.2). Depending upon the reactant combination, the double bond can be found in the 2- or 3-position. When it is preserved in the 3-position, these adducts can undergo dehydrohalogenation to form phospholes (Scheme 67).



Scheme 67

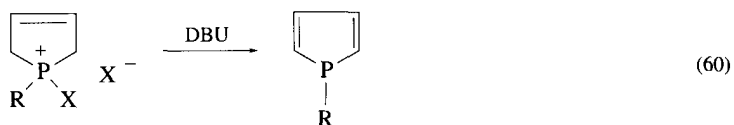
One of the earliest phosphole syntheses can be considered to be of this type, although the mechanism has not been confirmed: heating at high temperature (226–230°C) of a mixture of 1,4-diphenylbutadiene with phenylphosphonous dichloride gave 1,2,5-triphenylphosphole (**240**), a yellow solid, in 50–66% yield <62CI(L)359>. The yield can be improved to 70% by conducting the reaction in excess phenylphosphonous dichloride as solvent <83JOM(256)103>, and this remains the best method to obtain this compound. Similarly, treating 1,2,3,4-tetraphenylbutadiene with phenylphosphonous dichloride gave pentaphenylphosphole <65JCS2184>, and reacting 1,4-di-*p*-tolyl butadiene with *p*-tolyl PCl_2 gave a 13% yield of 1,2,5-tritolylphosphole <74JCS(P1)1694, 78LA74>. The

process can be expressed as proceeding through the McCormack cycloadduct (**239**) which then loses HCl (Scheme 68). However, an attempt to synthesize the McCormack cycloadduct from the same reactants at 100°C was unsuccessful, and the mechanism remains unconfirmed. An attempt to use this method for the synthesis of 1,3,4-triphenylphosphole was not successful [74PS(4)199].



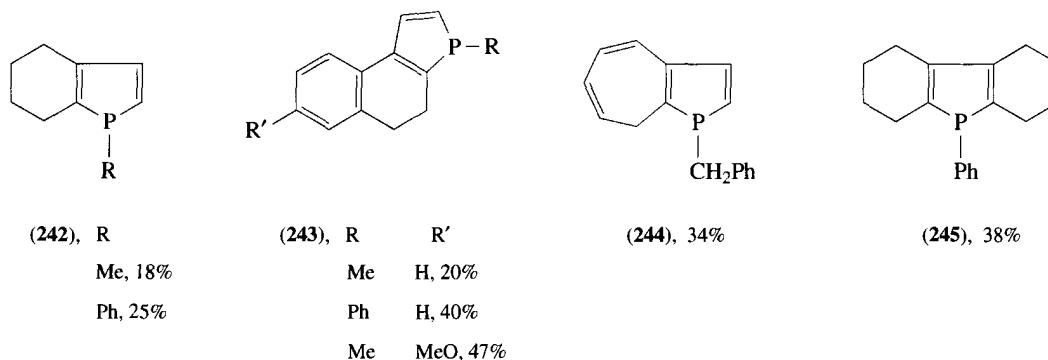
Scheme 68

A more clear-cut case of the dehydrohalogenation of McCormack cycloadducts was discovered by Mathey, who accomplished the synthesis of phospholes by the action of DBU as a base in benzene solution on the isolated cycloadduct (Equation (60)) [69CR(C)1066].

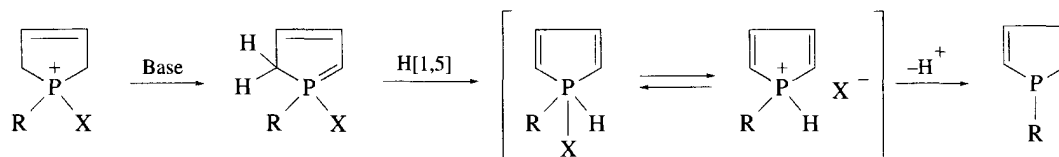


This was a major event in phosphole chemistry, as it allowed a number of phospholes, inaccessible by other methods, to be synthesized. This remains the best and most general of all phosphole syntheses, and has been widely used in this field. A review [88CRV429] provides a summary of the 25 phospholes prepared by dehydrohalogenation of halophospholenium halides (some prepared as discussed in Section 2.15.11.3). Yields are improved by the use of benzene-CH₂Cl₂ as solvent [70BSF4433] and of α -picoline as the base [81S983]. In the latter paper, it was reported that phenylphosphonous dichloride could be used instead of the less readily available dibromide in those cases where it gives a 3-phospholenium adduct rather than a 2-phospholenium adduct. These improvements in this "one-pot" method led to yields of 85% for 3,4-dimethyl-1-phenylphosphole, 75% for 3-methyl-1-phenylphosphole, and 50% for phenylphosphole. Some of the low yields reported in earlier work (many around 15–30%) could probably be improved by the newer techniques. Another valuable discovery is that the McCormack reaction can be greatly accelerated by the use of pressure in the range of 4–7 kbar [89CC967]. Thus, the reaction of 2,3-dimethylbutadiene with phenylphosphonous dibromide normally requires 7–10 days at room temperature for completion, but at 7 kbar it is complete in 2 h. Pressure also has the effect of giving a granular, more tractable form of the cycloadduct. The Mathey dehydrohalogenation is then performed directly on the reaction mixture with α -picoline to give a yield of 77%. Similarly were prepared 1-phenylphosphole from butadiene (75%) and 3-methyl-1-phenylphosphole from isoprene (75%).

The scope of the Mathey method is quite broad and can be extended to the synthesis of multicyclic phospholes (types (**242**) and (**243**) [82PS(12)161]; (**244**) [92TL5495]; (**245**) [78CJC1952]). Also, adducts from some phosphonous dichlorides fail to form phospholes, and the less accessible bromides must be used.



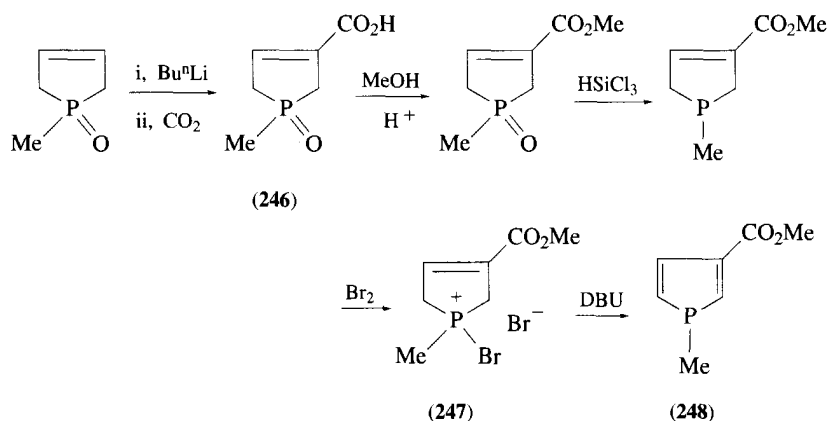
The mechanism of the dehydrohalogenation process has not been directly studied. A reasonable explanation is shown in Scheme 69; it is based on observations made on the course of events of the reverse process, the reaction of phospholes with anhydrous HCl <86JCS(P2)629> (see Section 2.15.4.3). A key feature is the formation of a transient 2*H*-phosphole derivative which undergoes a [1,5]-sigmatropic shift to the phosphole.



Scheme 69

2.15.11.3 Dehydrohalogenation of Halophospholenium Halides from Other Sources

Since the addition to the phosphorus atom of a 3-phospholene gives the same structure as formed in the McCormack cycloaddition, 3-phospholenes can be viewed as starting materials for the Mathey dehydrohalogenation procedure to form phospholes. Normally, the 3-phospholenes are themselves prepared from McCormack cycloadducts, either directly by dehalogenation <64JOC836> or by reduction of the phospholene oxide formed on hydrolysis of the adducts <65CB171>. There are, however, occasions when the 3-phospholene to phosphole conversion can be useful. Thus, 3-phospholene oxides or sulfides can undergo C-substitution reactions that are forbidden to phospholes; after such a reaction, the oxygen or sulfur can be removed, for example with trichlorosilane or phenylsilane, and the phosphine converted to the phosphole. This approach was used <73JOC1858> in the first synthesis of a C-functional phosphole ((**248**), see Section 2.15.12.2). The acid (**246**) was recovered after conversion of 1-methyl-3-phospholene oxide to its anion with *n*-BuLi, followed by carbonation. The phosphine oxide function in the methyl ester was reduced to the phosphine; bromine addition gave the bromophospholenium bromide (**247**) needed for the Mathey conversion to the phosphole (**248**) (Scheme 70).

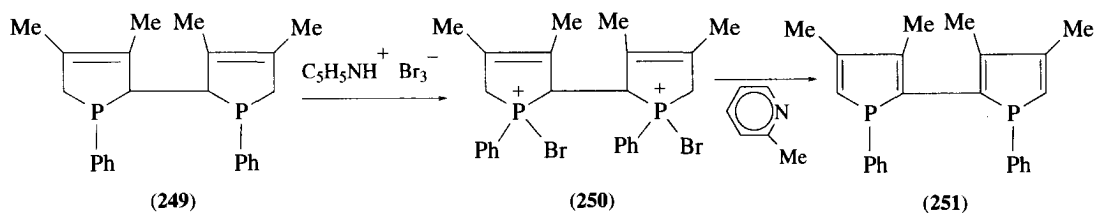


Scheme 70

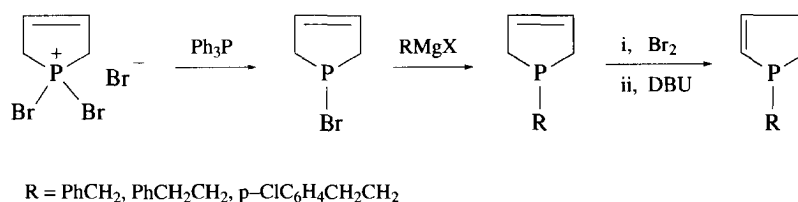
The reaction has been extended to the synthesis of a 2,2'-biphosphole <86JOM(316)271>. The biphospholene derivative (**249**) is directly available from the reductive dimerization of phospholes <84JA425>. Addition of bromine gave the salt (**250**) which on dehydrohalogenation with α -picoline gave the biphosphole (**251**) (Scheme 71).

It is also possible to place a variety of alkyl substituents on phosphorus of the phospholene system by preparing the P-halo derivative and reacting it with organometallic derivatives. Several phospholes have been prepared by this approach (Scheme 72) <73JOC1858>. An obvious advantage is that the need for the alkylphosphonous dichloride required in a McCormack reaction is eliminated. The dichlorides are difficult to prepare and handle.

McCormack type compounds can be generated by another approach, involving quaternization

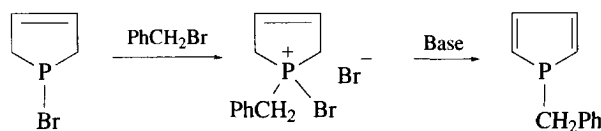


Scheme 71



Scheme 72

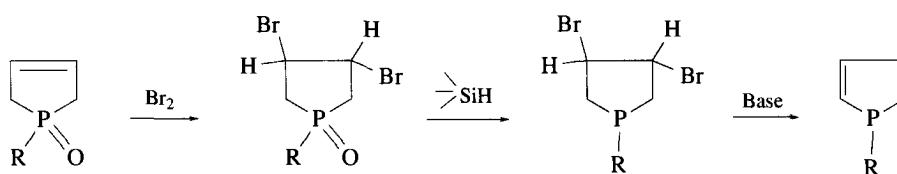
of P-bromophospholenes. Benzyl bromide is especially effective as the quaternizing agent, although methyl bromide may also be employed (73JOC1858). The bromophospholenium salts can then be treated in the usual way to form phospholes. By this method, 1-benzyl-3,4-dimethylphosphole was first synthesized. The yield in the final step was 28.6% with DBU as the base, but this was later improved to 75% with α -picoline (Scheme 73) (81S983). The method has also been used to prepare a series with XCH_2 - or XCH_2CH_2 - as P-substituents ($\text{X} = \text{OR}$, SR , $\text{C}(\text{O})\text{R}$, $\text{CH}(\text{OR})_2$, COOR , CONR_2 , and CN ; (76CJC2402)).



Scheme 73

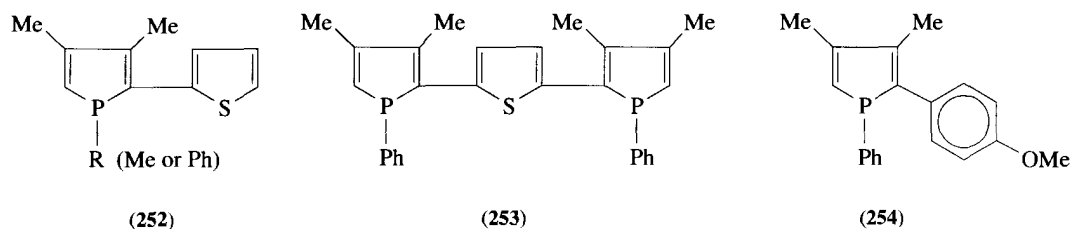
2.15.11.4 Dehydrohalogenation of C-Halo Phospholanes

The first synthesis of methylphosphole (67JA5984, 69JA3308) and of phenylphosphole (68TL1755) employed 3-phospholene oxides from McCormack cycloadducts as the starting materials. Bromine was added to the double bond to form the dibromophospholane oxides; these were deoxygenated with trichlorosilane and phenylsilane, respectively, to form the dibromophospholanes. Potassium *t*-butoxide was used for the dehydrohalogenation of the methyl derivative (13–15% from the dibromo oxide), and DBU for the phenyl derivative (47%) (Scheme 74). These two studies were important in providing the first examples of C-unsubstituted phospholes, which allowed the beginning of serious inquiry into the matter of cyclic delocalization in the phosphole ring.

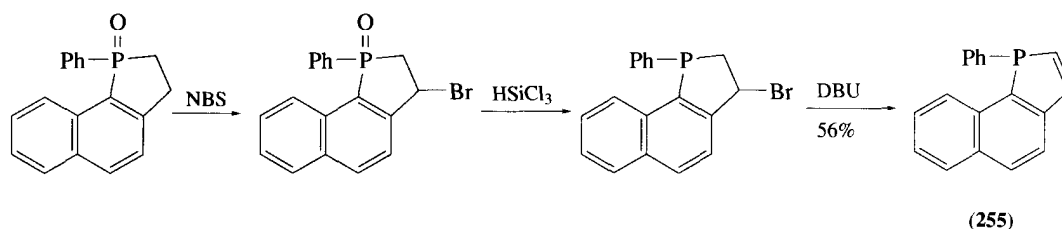


Scheme 74

This method is still of value and has been used to prepare 1-benzylphosphole (26%), 1-(2-phenylethyl)phosphole (10.1%), 1-[2-(*p*-chlorophenyl)ethyl]phosphole (17.3%) <73JOC1858>, the thienyl phospholes **(252)** <90JOC2494> and **(253)** <91HAC377>, and the 2-anisyl derivative **(254)** <90JOC2494>.

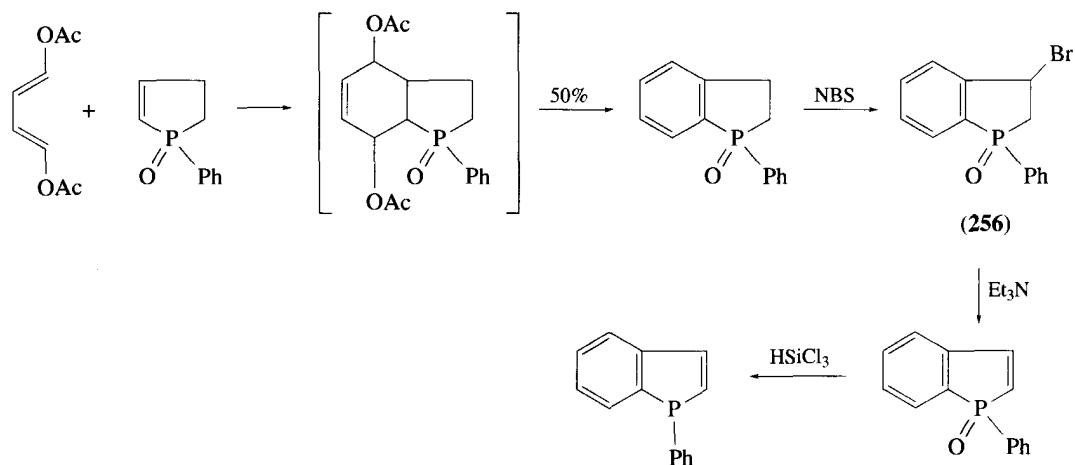


The concept of preparing a C-bromo P-oxide as an intermediate for conversion to a phosphole has appeared in modified form in the synthesis of fused-ring phospholes, for example **(255)** (Scheme 75) <79PS(5)349>.



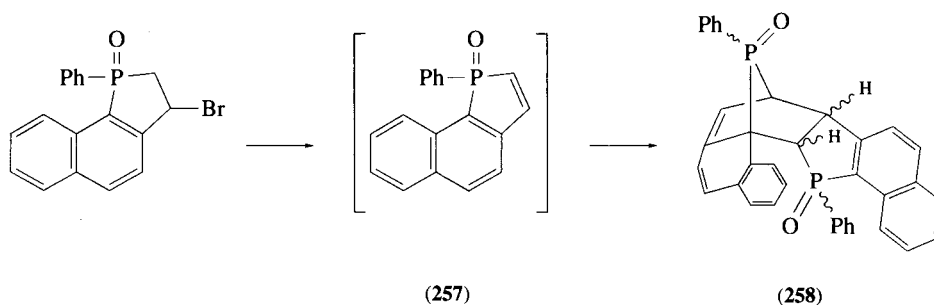
Scheme 75

In the case of the phosphindole system, a C-bromo P-oxide (**(256)**) may be generated as a precursor to the phosphole oxide, which is then reduced to the phosphole group by a silane <71CJC530>. In this fused ring system, the phosphole oxide moiety is stable and does not undergo the rapid dimerization characteristic of most monocyclic phospholes. The entire synthesis is shown in Scheme 76.



Scheme 76

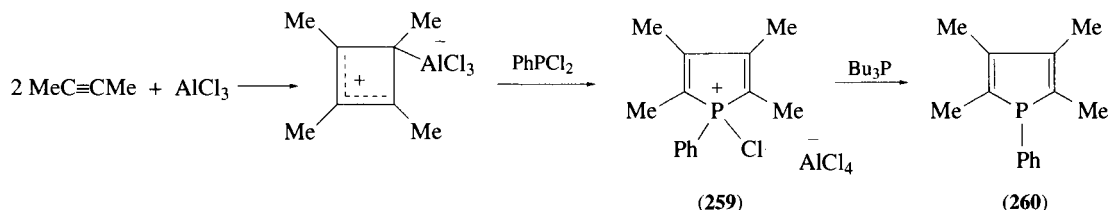
This was not the case for a benzo[*g*]phosphindole oxide (**(257)** <82PS(12)161>); application of the above reaction sequence gave a dimer, rather than the desired monomer, of the benzophosphindole oxide (**(257)**). It was necessary to reverse the reaction sequence so as to prepare the bromophosphine for the dehydrohalogenation step as in Scheme 77. The structure of the dimer was proposed, but not fully established, as having the usual Diels–Alder adduct framework.



Scheme 77

2.15.11.5 Dehalogenation of Halophospholium Tetrachloroaluminates

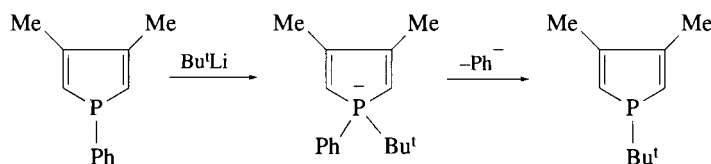
A new approach to tetrasubstituted phospholes is based on the observation that the tetrachloroaluminate salt of a tetrasubstituted cyclobutadiene- AlCl_3 σ -complex undergoes insertion of phosphorus into the ring when reacted with a phosphonous dichloride. A chlorophospholium salt is formed ^[83TL1423] and hydrolysis gives a stable phosphole oxide. Later workers ^[88OM921] showed that the salt **(259)** could be dehalogenated to the phosphole **(260)**, and thereby accomplished the first synthesis of 1-phenyl-2,3,4,5-tetramethylphosphole (Scheme 78). The method is particularly simple, and consists of the formation first of the cyclobutadiene salt from 2-butyne and AlCl_3 , addition of the phosphonous dichloride, and finally tri-*n*-butylphosphine as the reducing agent, without isolation of any intermediates. The yield was 68%. The method will have limited scope, however, due to restrictions in the alkyne that will form the cyclobutadiene complex.



Scheme 78

2.15.11.6 Displacement of P-Substituents

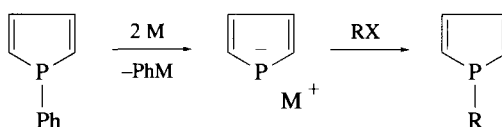
The P-phenyl group of a phosphole can be directly displaced by reaction with alkyl lithium reagents in TMEDA. Both *t*-butyl ^[72T471] and *n*-butyl ^[72OMR(4)171] have been placed on P by this method. With 3,4-dimethyl-1-phenylphosphole, the former reaction occurred in 70% yield, the latter in 31.5% (with some oxide) (Scheme 79). This method is of considerable value for the introduction of the *t*-butyl group on phosphorus, as this group cannot be used as the P-substituent in a phosphonous dihalide in the McCormack reaction because of steric restrictions.



Scheme 79

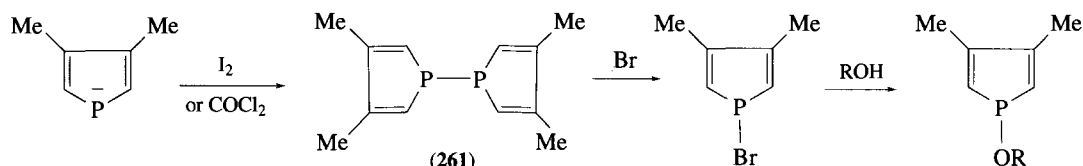
A more versatile method for a group interchange at P consists of cleaving a P-phenyl substituent with a metal (lithium, sodium, or potassium) to form the phospholide ion, which can then be reacted with electrophiles. In every case known the electrophile has attacked exclusively at P, even though electron delocalization in the phospholide ion is extensive (see Section 2.15.2). This reaction was

discovered early in the development of phosphole chemistry <71T5523>, and has been widely used over the following years (Scheme 80).



Scheme 80

A considerable number of alkyl halides, dihaloalkanes, and halides with other functional groups has been used for this purpose (for a summary, see <B-90MI 215-01>). Biphospholes (**261**) are created by reaction of the anion with phosgene <83OM1234> or iodine <82JOM(231)361> (Scheme 81). The biphospholes serve as precursors of other P-functional phospholes; reaction with bromine gives 1-bromophospholes, which with alcohols provide 1-alkoxyphospholes (Scheme 81). This is the only method presently available for synthesizing these derivatives <82JOM(231)361>.



Scheme 81

2.15.12 SYNTHESIS OF PARTICULAR TYPES OF PHOSPHOLES

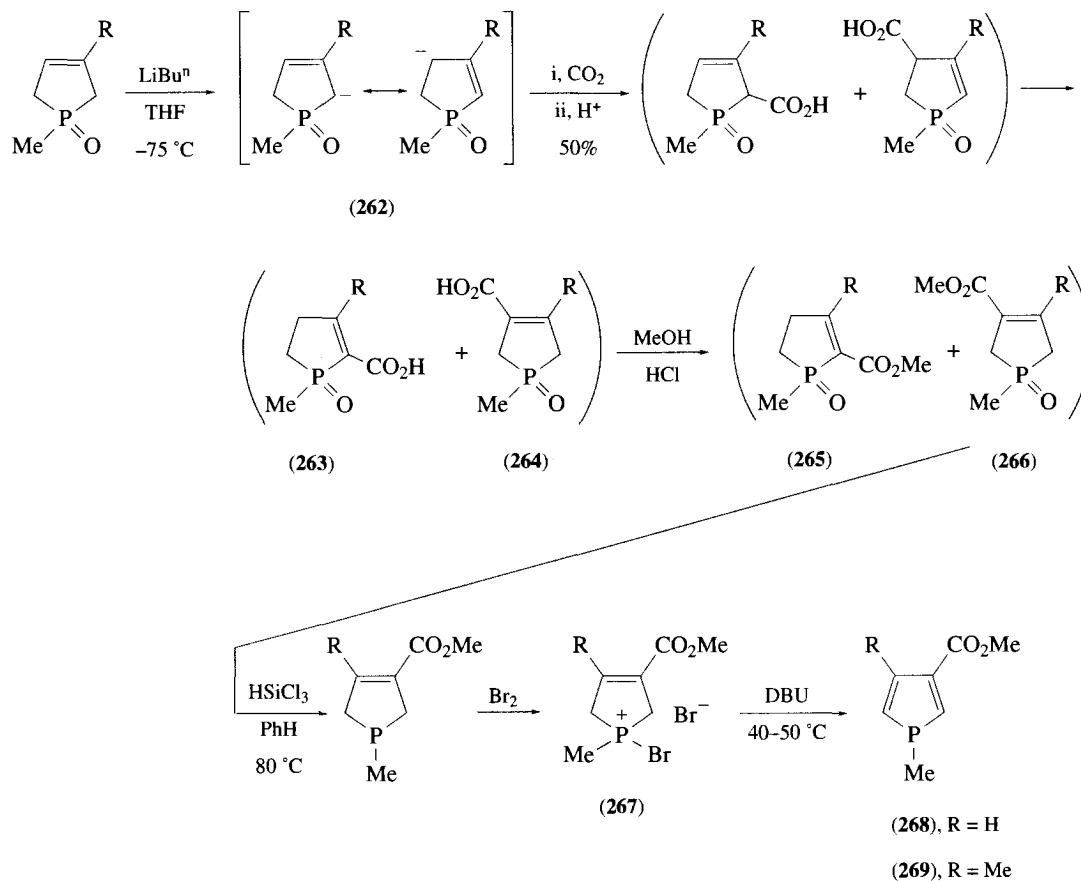
2.15.12.1 General

With the exception of the ring synthesis that provides aminophospholes (see Section 2.15.10.5), the reactions used for the construction of the phosphole ring are not amenable to the direct synthesis of phospholes with C-substituents other than alkyl or aryl groups. This is a drastic difference with regard to the related heterocycles with N, S, or O in the ring. Additionally, there are no known direct electrophilic substitution reactions that take place on phosphole ring carbons. Indeed, the first functionalized phosphole (a carboxylate <72TL299>) was not prepared until 1972. To address the need for functional phospholes, techniques have been devised that are based on appropriate substitutions on dihydro derivatives, followed by conversion to phospholes, or on phospholes where phosphorus bears additionally a blocking function such as O, S, or a metallic group, which can be removed at the end of the synthesis. For the most part, these synthetic procedures are multistep with low overall yields. For these reasons, functionalized phospholes remain rare. There remains much to be done in this area, both in the creation of practical new methods and in the generation of new structures.

2.15.12.2 Carboxylic Acids and Derivatives

The first synthesis of a carboxylate was based on the use of a phospholene oxide as starting material <72TL299>. Carbanion formation occurs relatively readily with these compounds; reaction of the resonating ion (**262**) with carbon dioxide installed the carboxy group on the ring at positions 2 and 4 with subsequent double bond rearrangement to (**263**) and (**264**). The mixture was then esterified; isomer (**265**) was unstable at room temperature and formed an insoluble polymer, making it possible to isolate (**266**). The oxygen on P in (**266**) was then removed, and the phosphine treated with bromine to provide a bromophospholenium ion (**267**). This is the same species as formed in the McCormack diene-phosphonous dibromide cycloaddition reaction, widely used as an immediate precursor of phospholes (see Section 2.15.11.2). By this approach (Scheme 82), two phosphole-3-carboxylates ((**268**) and (**269**)) have been synthesized <72TL299, 73JOC1858>. These phosphole car-

boxylates are not stable at room temperature; they can be distilled if this is performed rapidly at reduced pressures, but they require temperatures around -75°C for storage.

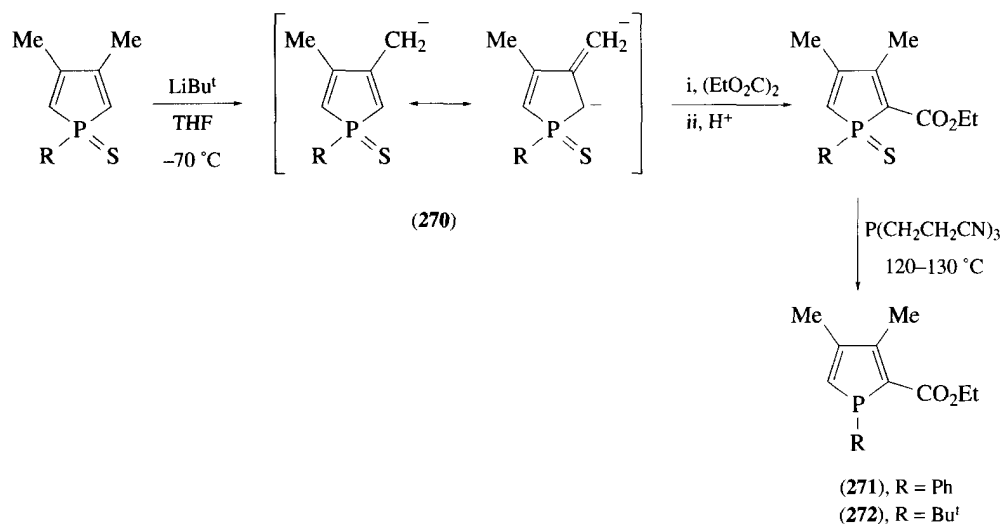


Scheme 82

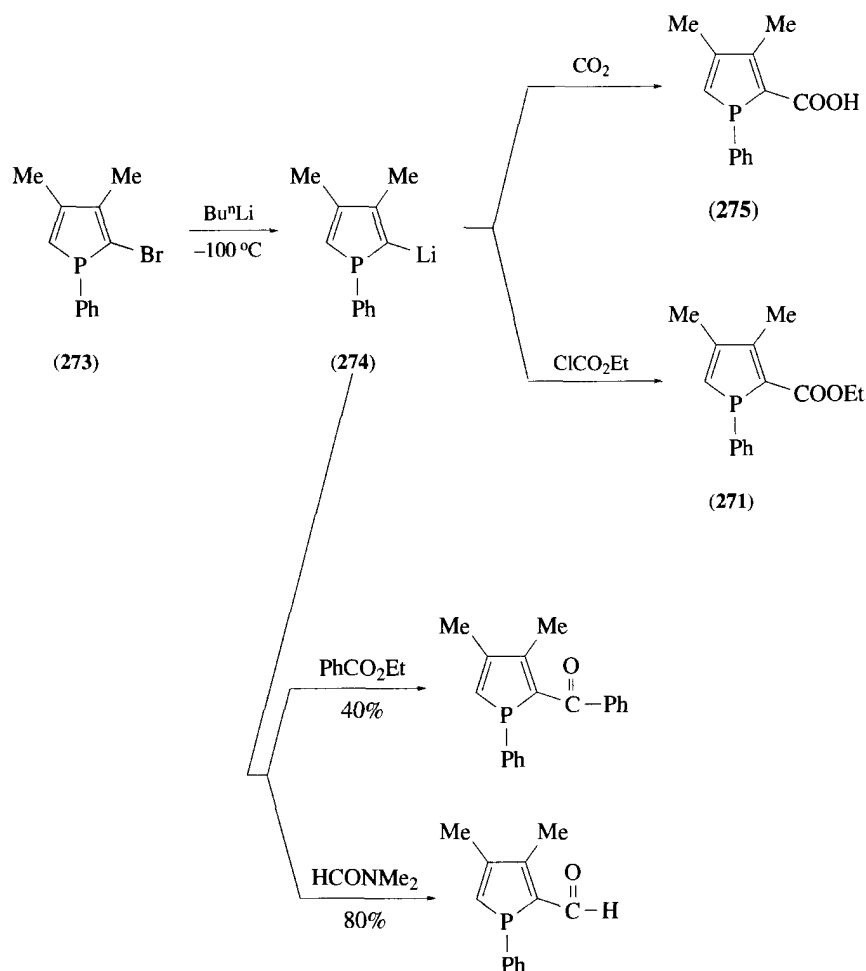
Phosphole sulfides are frequently stable, unlike oxides which rapidly dimerize, and can be used as precursors of phospholes by simple reduction methods. In another approach $\langle 80\text{NJC}683 \rangle$, this stability was taken advantage of for the introduction of the carboxylate group on the 3,4-dimethylphosphole ring (Scheme 83). The sulfide forms a carbanion (**270**) which can be quenched with diethyl oxalate to form a phosphole sulfide 2-carboxylate. This was converted to the phosphole by removing the sulfur with a more nucleophilic phosphine (tri-*n*-butylphosphine or tris-(2-cyanoethyl)phosphine). The phosphole-2-carboxylates prepared this way (**271**) and (**272**) were significantly more stable than the 3-carboxylates and could be stored at room temperature. 3,4-Dimethyl substitution is well known to increase the stability of phospholes, and at present it is unknown if this substitution pattern or the position of the carboxylate is responsible for the increased stability.

Until 1992, no C-metal derivative of a phosphole was known, and some of the conventional routes to carbonyl derivatives from this type of precursor were not practical. However, 2-bromo-3,4-dimethyl-1-phenylphosphole (**273**) has now been synthesized (see Section 2.15.12.8 $\langle 92\text{BSF}486 \rangle$) and this allowed the synthesis of the 2-lithio derivative (**274**) and its use in various processes (Scheme 84). Thus, the 2-COOH (**275**) and 2-COOEt (**271**) derivatives were prepared from this organometallic. Compound (**275**) is the first example of a phosphole carboxylic acid; its properties are yet to be evaluated.

Two phosphole-3-carboxylates with 4,5-diphenyl substituents (**276**) and (**277**) have been prepared by a rather specialized approach $\langle 93\text{BSF}695 \rangle$, outlined in Scheme 85. This method was designed to generate phospholide ions with a C-functional group (e.g. (**278**)), but the ions can be alkylated on P to form phospholes. The ions are formed by a base-induced retro-Michael reaction. While of value in producing an otherwise inaccessible substitution pattern on the ring, the method is less



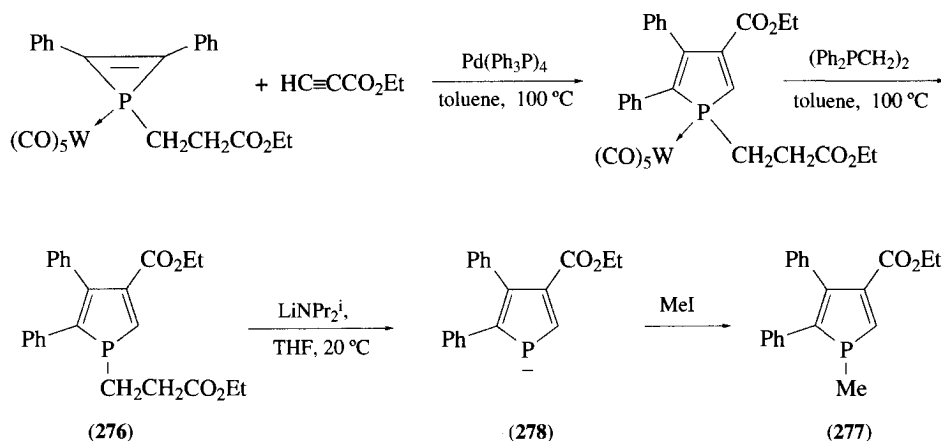
Scheme 83



Scheme 84

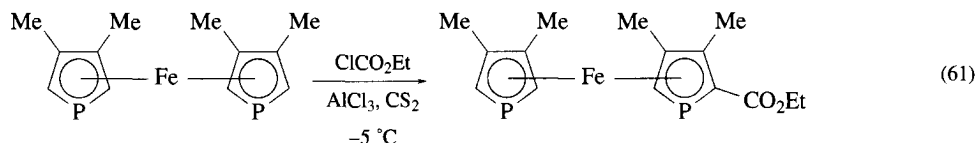
versatile than those discussed above. Unlike the 3-carboxylates (268) and (269), these more highly substituted compounds were quite stable.

A property of certain metallic complexes with the phosphole ring may allow them to become useful precursors of carboxylates (as indeed they have in the synthesis of ketones, see Section



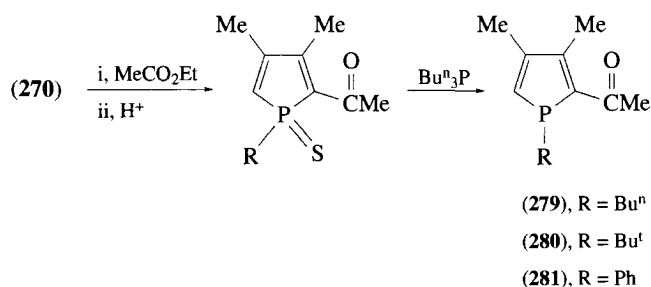
Scheme 85

2.15.12.3). In the phosphoferrocene ring, where the phosphole moiety is in the form of the phospholide ion, direct substitution reactions are possible, carboxyethylation among them (Equation (61)) <80NJC683>. There is, however, no report on the conversion of the complexes to the free phospholes.



2.15.12.3 Aldehydes and Ketones

A method similar to that used for phosphole carboxylate synthesis has been employed for the formation of ketones (279), (280), and (281) (Scheme 86); here the carbanion from a phosphole sulfide is reacted with ethyl acetate and the sulfur then removed with a phosphine <76T2395>.

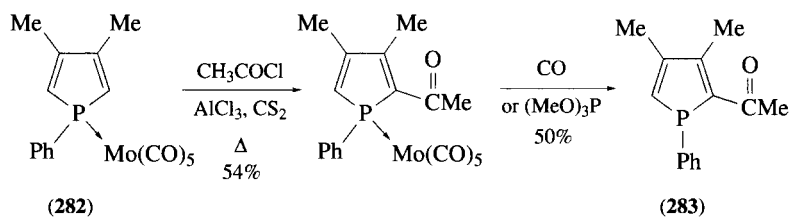


Scheme 86

These ketones are of good stability and can be purified by distillation. The method appears extendable to the introduction of other acyl groups.

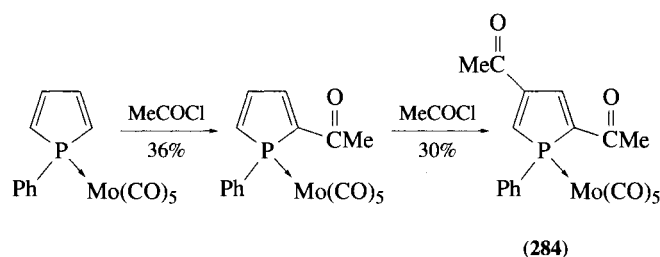
With the synthesis of the first organometallic derivative of a phosphole (274), a new approach to ketones (and also to the first phosphole carbaldehyde) became possible (see Scheme 84; <92BSF486>).

The sensitivity of metal complexes of phospholide ions to electrophilic substitution has been employed in another approach to phosphole ketones <85JOC467>. The molybdenum complex (282) was acetylated at the 2-position under Friedel–Crafts conditions, and the metallic group then removed with carbon monoxide under pressure. Trimethyl phosphite also can be used for this purpose.

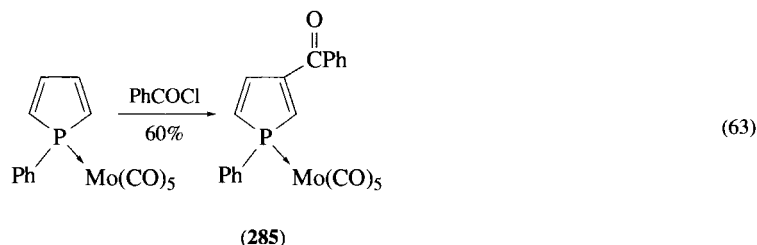
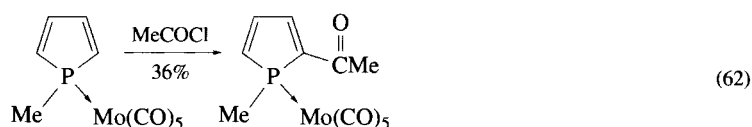


Scheme 87

Some other metallic complexes were also found in this study to be acetylated readily under the same conditions, but the metallic group has not yet been removed to give the free phosphole ketones. These reactions are summarized in Scheme 88 and Equations (62) and (63).

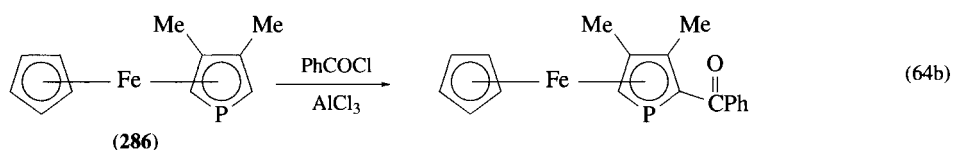
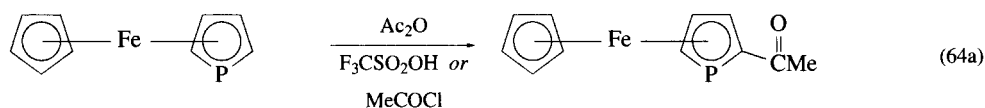


Scheme 88



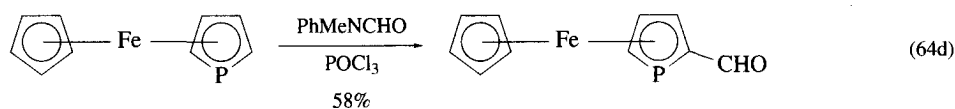
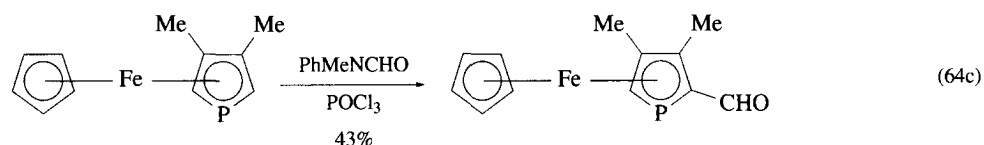
The first example of a diacetylated product (284) was also encountered in this study, as was the formation of a 3-benzoyl product (285). This orientation presumably arose from a steric effect associated with the use of the more bulky reagent benzoyl chloride for attack on the ring.

The phospholide moiety of a monophosphaferrocene was acetylated by treatment with acetic anhydride in the presence of trifluoromethylsulfonic acid $\langle 87\text{ICA}93 \rangle$. Again the acetylated phosphole was not released from the complex. Benzoyl chloride also was used to benzoylate the phospholide moiety of (286) (Equations (64a) and (64b)).



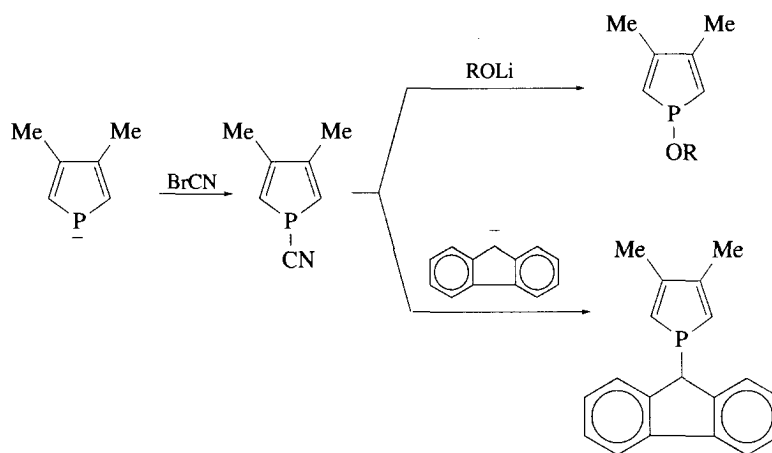
Diphosphaferrocenes can also be acetylated under similar conditions $\langle 80\text{JA}994 \rangle$.

The aldehyde function has been placed on the phospholide ring of phosphosphaferrocenes by a conventional Vilsmeier reaction $\langle 80\text{JA}994 \rangle$ (Equations (64c) and (64d)).



2.15.12.4 Nitriles

Phospholes with a C-nitrile group remain unknown at this time. A P-nitrile has been prepared and has considerable value for the introduction of other types of substituents on phosphorus (Scheme 89) <88OM1796>.

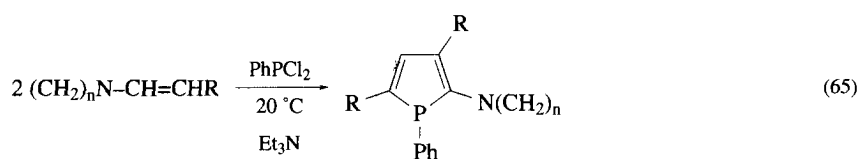


Scheme 89

A tungsten carbonyl complex of the 1-cyanophosphole has also been used with success in the synthesis of other P-substituted complexes (e.g. the P—OH complex (296) see below).

2.15.12.5 Amines

The only phospholes with amino substituents are compounds (287), (288), and (289) (Equation (65)). Unlike the C-functional phospholes discussed to the present, these compounds resulted directly from the ring-forming process <88TL4581>. The starting materials were simple enamines and phenylphosphonous dichloride, which were condensed in a single step. Because of the simplicity of the method, it could become the most important one for providing a phosphole with a C-function. The yields for the single step are 30–35%, surpassing the yields of all of the multistep functionalizations discussed in this section.



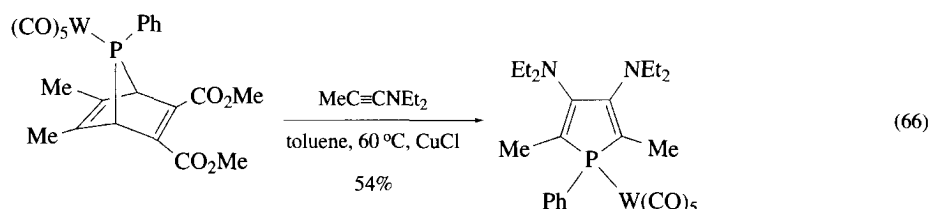
(287), R = R = Me, n = 5

(288), R = R = Et, n = 5

(289), R = R = Et, n = 4

The mechanism of this process is not obvious and has received no experimental work. The phosphole ring was indicated by the ^{31}P NMR spectra, and the compounds gave the correct molecular weight by mass spectral measurements. ^{13}C NMR spectral features were used to assign the position of the substituents.

Aminophospholes in the form of tungsten carbonyl complexes have been synthesized as follows (Equation (66)) <90PS(47)477>.

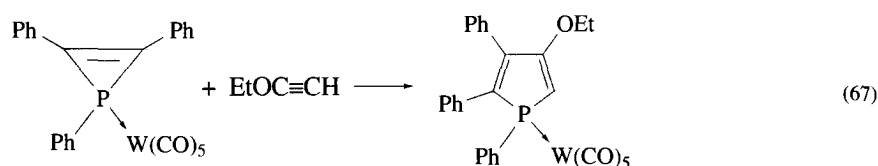


2.15.12.6 C-Aryl and C-Heteroaryl Phospholes

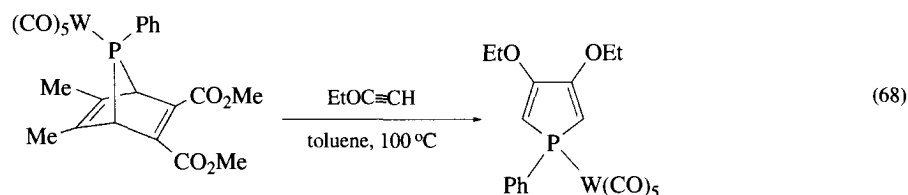
C-Phenyl phospholes may be prepared from the use of phenyl-substituted dienes in the McCormack reaction (see Section 2.15.8.2). However, there is only one technique available for the synthesis of C-aryl phospholes by a substitution process, and it is one that involves several steps <90JOC2494, 91HAC377>. Again a metal complex (with $\text{W}(\text{CO})_5$) of a phosphole is the starting material; in the presence of aluminum chloride, electrophilic character in the ring is increased, expressed by complex (290), and attack on electron-rich aromatic systems becomes possible. The complex after the arylation reaction is broken to generate a phospholene oxide (291), which is then converted to a phosphole in the same way (reduction to the phospholene, followed by bromine addition and dehydrohalogenation with base) as used in the first synthesis of phosphole-3-carboxylates <72TL299>. These reactions are summarized in Scheme 90. The compounds formed have structures (292)–(295).

2.15.12.7 Hydroxy and Alkoxy Derivatives

C-alkoxy derivatives of a phosphole have resulted from the insertion of ethoxyethyne into a phosphirene complex (Equation (67)) <87TL5021>.

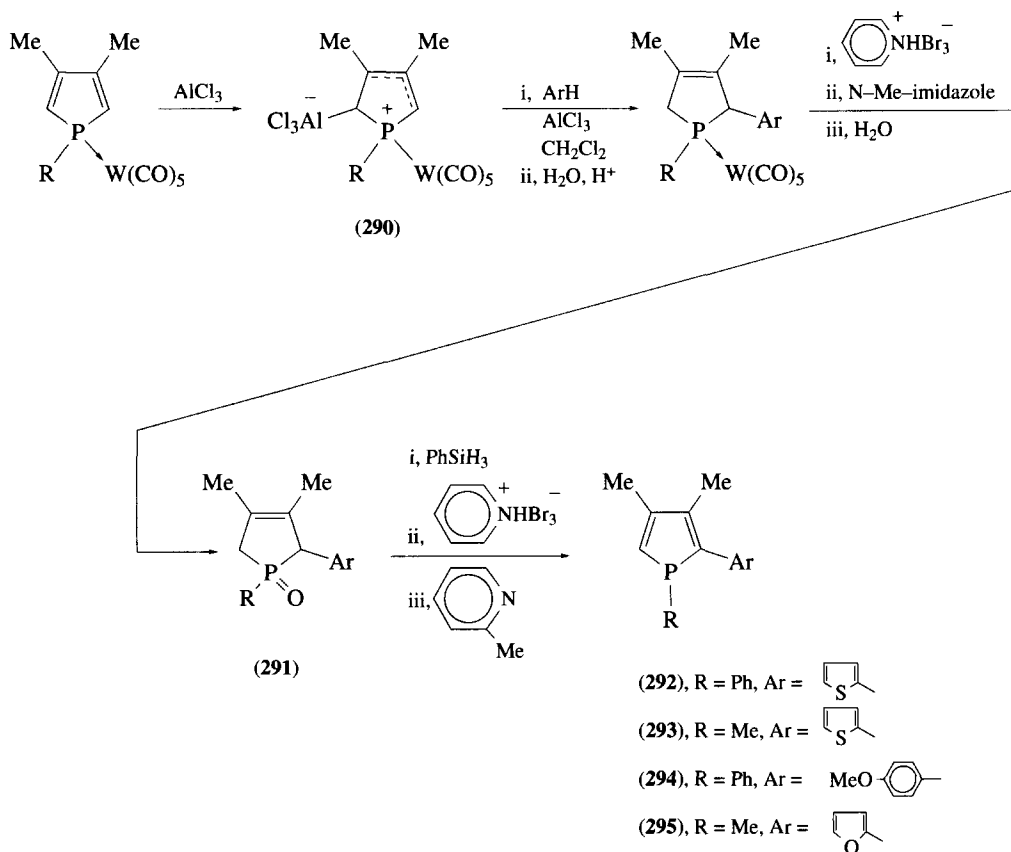


Related complexes have been formed from a norbornadiene complex (Equation (68)) <90PS(47)477>. In neither case has the decomplexed phosphole yet been reported, however.

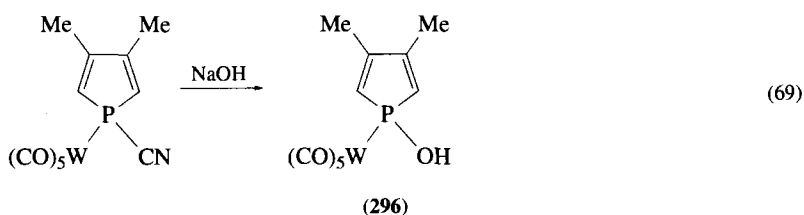


A C-hydroxy derivative of a phosphole would of course be involved in tautomerism with the keto isomer, with the latter probably the observable form (as with O, N, S cycles). However, no model compounds have yet been prepared to test this point.

A P-hydroxy phosphole has been reported in the form of a tungsten carbonyl complex (**296**) (Equation (69)) <88OM1796>. Normally P—OH groups in phosphines undergo rearrangement to —P(O)H forms, but the complex prevents this from occurring.

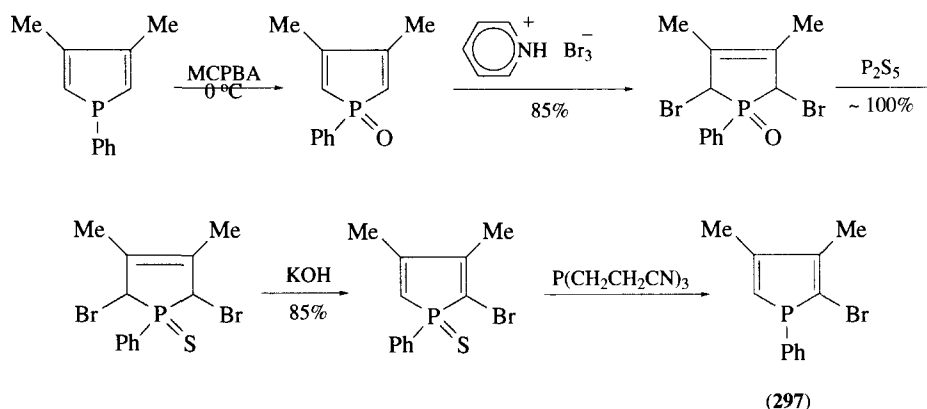


Scheme 90



2.15.12.8 Halo Substituents

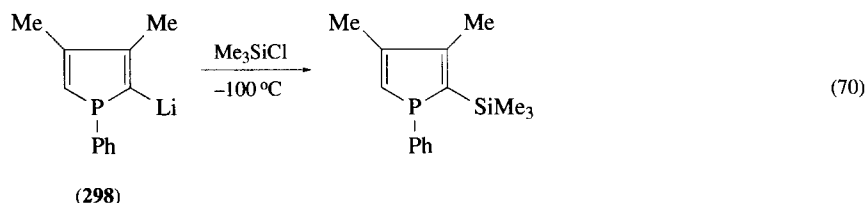
The only known C-halophosphole is 2-bromo-3,4-dimethyl-1-phenylphosphole (**297**) prepared as shown in Scheme 91 <92BSF486>. As noted in Section 2.15.12.3, this compound is important as a precursor of the 2-lithio derivative.



Scheme 91

2.15.12.9 Silyl Derivatives

The only C-silylated derivative of a phosphole was prepared from the lithio compound (298) (Equation (70)) (92BSF486).



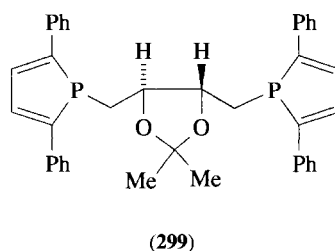
2.15.13 SOME PRACTICAL ASPECTS OF PHOSPHOLE CHEMISTRY

2.15.13.1 Phosphole Ligands in Metal Complexes for Catalysis

As has been noted in various places in this chapter, the coordination chemistry of phospholes is extensive and of considerable practical importance. The field continues to be a very active one, with new complexes being reported frequently as the list of metals forming complexes with phospholes is extended. Another current activity is the practical one of exploring the use of some of the complexes as homogeneous catalysts for organic reactions, and it is this aspect of phosphole complex chemistry that is summarized here.

In their important review entitled, "Complexing Modes of the Phosphole Moiety" (B-83MI 215-01), Mathey *et al.* pointed out that phospholes have unusually great variety in the way bonding to metals can occur, and are known to form many different types of complexes through the use of electrons on phosphorus and/or those in the diene unit. In general phospholes are better ligands than noncyclic tertiary phosphines such as the commonly used triphenylphosphine, but the substitution pattern on the phosphole ring can play a very important role in the reactivity to metals. It was noted in quite early studies of complexation that P-methyl- and P-benzylphospholes showed no tendency to form complexes with NiCl_2 , but the 3,4-dimethyl derivatives of these phospholes readily formed stable complexes (73PS(2)205). A similar effect was noted in a series of P-phenyl phospholes (B-83MI 215-01), which appear to be more reactive than P-methyl counterparts. In general, complexing ability is maximized in the compound 3,4-dimethyl-1-phenylphosphole, and this is the phosphole most frequently used in complexes prepared as possible catalysts. It is fortunate that this happens to be one of the most easily synthesized phospholes, and has been made in 30 kg batches (88CRV429). The process has been patented (81EUP41447). Dibenzophosphole is also frequently used as a ligand, but its mode of bonding is restricted to the two-electron type typical of a tertiary phosphine. Complexes containing 1-phenylphosphole, 1,2,5-triphenylphosphole, pentaphenylphosphole, and some 1-alkyl-3,4-dimethylphospholes have also been included in studies

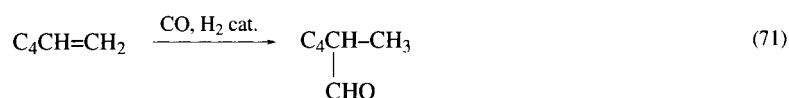
on potential catalyst systems. A diphosphole with an optically active P-substituent (**299**) has also been prepared <93PS(85)207> and used in coordination chemistry <93JOM(463)205>.



Phosphole complexes have been used effectively as catalysts for homogeneous hydrogenation of alkenes and alkenylbenzenes. The most commonly used metallic moieties are rhodium(I) and (III) hydrides <72CJC3714, 74CJC775, 74CJC3758, 87JMOC79> and ruthenium(II) chlorides and carbonyl derivatives <83IC2476, 89IC3831>. The efficiency of some of the catalysts based on phospholes exceeds that of the more commonly used systems such as the Wilkinson catalyst $\text{RhCl}(\text{PPh}_3)_3$, and it is an area of research that might be expected to lead to useful applications of phospholes.

In the case of aryl-substituted alkenes, double bond isomerization can be effected. Thus, rearrangement of allylbenzene to *cis*- and/or *trans*- β -methylstyrene has been effected with some ruthenium(II) complexes <89IC3831>.

Phosphole complexes based on rhodium, with the general formulation $\text{HRh}(\text{CO})_m(\text{PR}_3)_{4-m}$, have been found to be excellent catalysts for the hydroformylation of hexene, a process of great industrial importance, giving higher reaction rates and better regioselectivity than found for the more commonly used triphenylphosphine complexes. The first systematic study of the use of monocyclic phospholes as ligands was reported in 1989 <89JMOC219, 89JMOC153> (some earlier studies including the use of dibenzophosphole are referenced in these papers). The complexes are easily made *in situ* by adding the phosphole to a solution of $[\text{Rh}(\text{CO})_2\text{Cl}]_2$; that from 1,2,5-triphenylphosphole was especially efficient (Equation (71)).



In later work, hydroformylation of ethyl acrylate to give ethyl 2-formylpropanoate was also effected in good regioselectivity <91NJC279>; in the hydroformylation of styrene, 1,2,5-triphenylphosphole was a more effective ligand than triphenylphosphine <88CC1370>.

Another application found for phospholes in catalysis is in the insertion of carbon monoxide into a carbon-halogen bond <86POL911>. *trans*-Dichlorobis(1,2,5-triphenylphosphole)palladium(II) was effective as a catalyst at atmospheric pressure and was superior to the complex based on triphenylphosphine (Equation (72)).

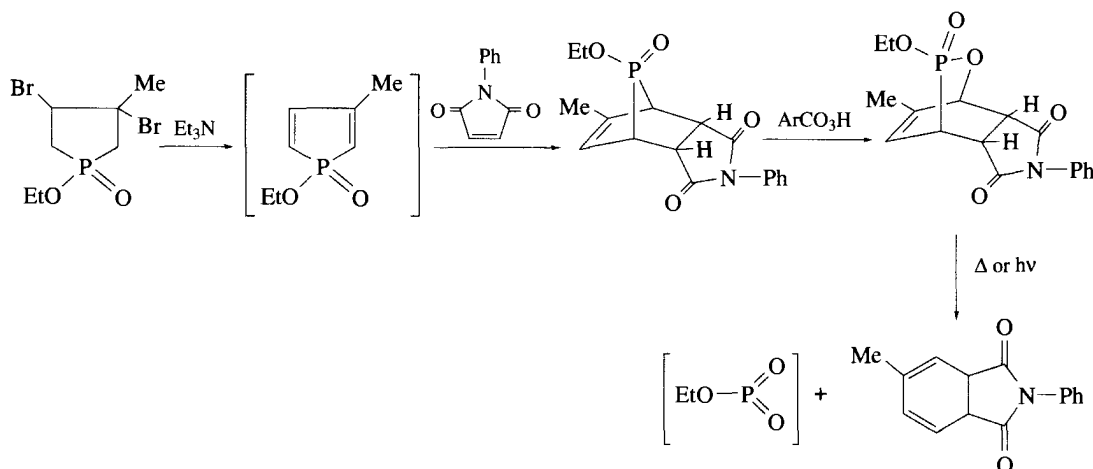


2.15.13.2 Phospholes as Precursors for Other Phosphorus Heterocycles

2.15.13.2.1 7-Phosphanorbornenes and 7-phosphanorbornadienes

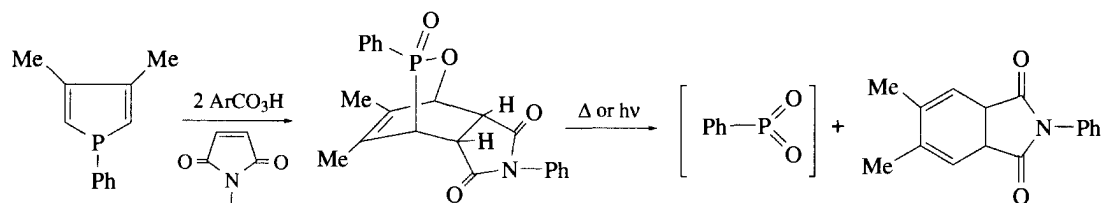
The only synthetic approach to the valuable 7-phosphanorbornene system is through Diels–Alder chemistry on phospholes, especially in the four-coordinate state (P-oxides and sulfides). Some of this chemistry has been described in Sections 2.15.6.1 and 2.15.6.2, and it has been reviewed elsewhere <90RHA39>. The 7-phosphanorbornenes are used as precursors for the 2,3-oxaphosphabicyclo[2.2.2]octene ring system, which finds value as a source, on thermal or photochemical

fragmentation, of 3-coordinate phosphoryl species (metaphosphates and dioxophosphoranes, <B-92MI 215-02>). An example of the synthesis of ethyl metaphosphate is shown in Scheme 92.



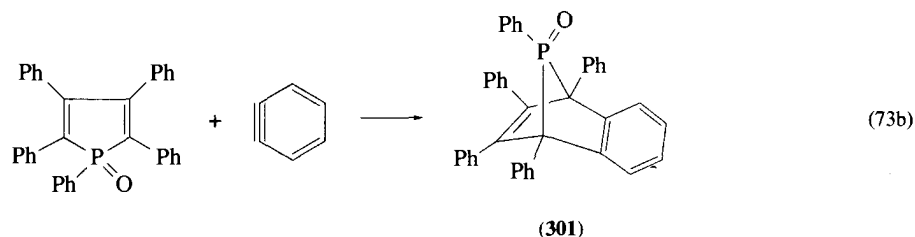
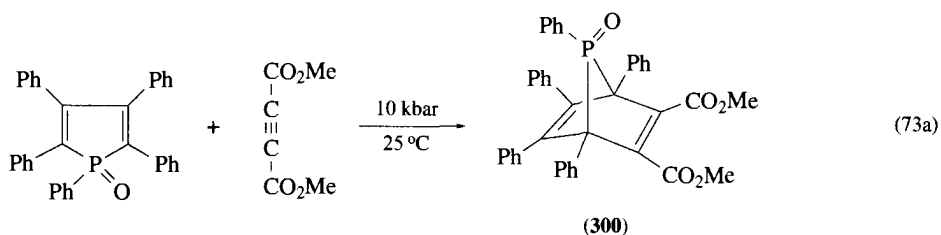
Scheme 92

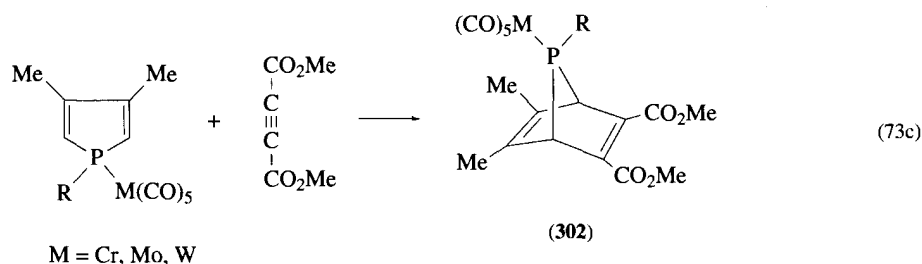
Phenyl dioxophosphorane is especially easily generated in a sequence that starts from 3,4-dimethyl-1-phenylphosphole <92HAC359> (Scheme 93).



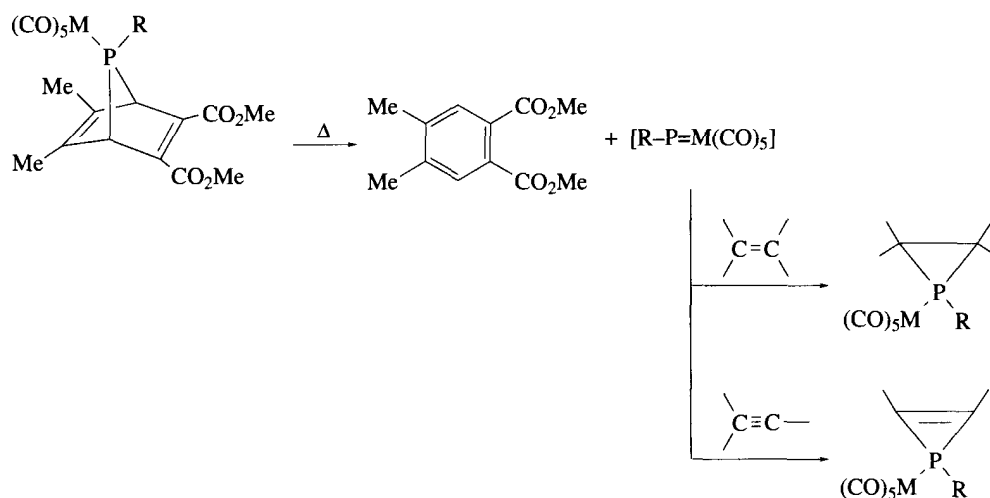
Scheme 93

There are no stable derivatives of the 7-phosphanorbornadiene ring system with phosphorus in the phosphine state, but one example of an observable, though rather unstable, oxide (**300**) has been reported, and the ring system is also known in the form of metal complexes (**302**) (Equations (73a-c)). A stable benzo derivative (**301**) is also known.





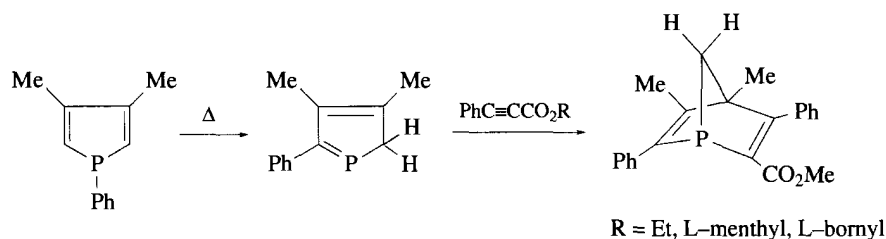
The complexes (302) have synthetic utility also; when heated in inert solvents in the presence of an alkene $\langle 84OM456 \rangle$ or an ethyne $\langle 82JA4484 \rangle$, the P-containing fragment is released from the bicyclic ring and transferred to the unsaturated reactant (Scheme 94).



This results in the formation of complexes of phosphiranes and phosphirenes, respectively, which can be decomposed to give the free phosphines. Phosphirenes were synthesized for the first time by this technique. A one-pot synthesis of phosphirene complexes from a 3,4-dimethyl-1-phenylphosphole complex with tungsten has even been developed $\langle 85JA4700 \rangle$. These applications based on phosphole chemistry are included in a review of the three-membered ring systems $\langle 87AG(E)275 \rangle$.

2.15.13.2.2 1-Phosphanorbornadienes

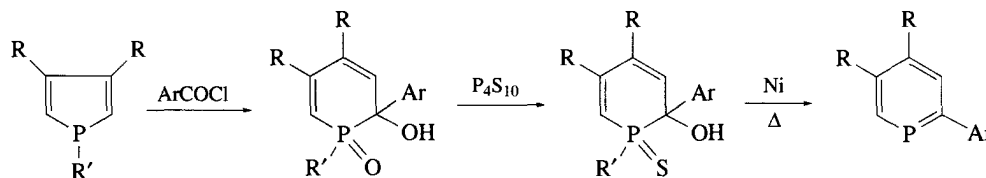
The sole source of the valuable 1-phosphanorbornadiene ring system is based on a novel feature of phosphole chemistry; as seen in Section 2.15.5.9, at high temperatures, some phospholes undergo [1,5] sigmatropic rearrangements to give 2*H*-phospholes, and these can participate in Diels–Alder reactions that serve to generate the bicyclic system having phosphorus at the bridgehead position. Again 3,4-dimethyl-1-phenylphosphole is a particularly useful reactant $\langle 81JA4595, 89NJC369 \rangle$, and has been reacted with several ethyne derivatives to form 1-phosphanorbornadienes (Scheme 95); the other regioisomer is formed in lesser amount.



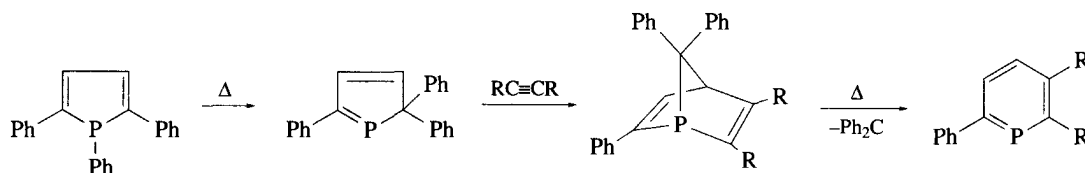
Scheme 95

2.15.13.2.3 Phosphinines

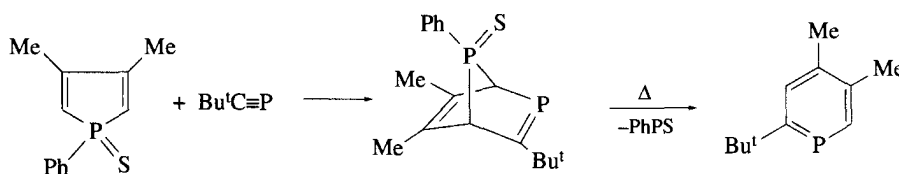
Several processes have already been noted that amount to conversion of the phosphole ring to the phosphinine ring. These are summarized in Schemes 96 to 98 and are included in a 1992 review of some work in phosphinine chemistry (92RHA1). The methods are of considerable synthetic value and are evaluated along with other methods in Vol. 5, Chapter 12. Various groups R and Ar may be present in the methods in Scheme 96 and Scheme 97, making some novel substitution patterns possible in the phosphinines.



Scheme 96



Scheme 97



Scheme 98

2.15.14 ADDENDUM

The papers listed below appeared after submission of the original manuscript. They are assigned to particular sections of the chapter, but for some there is considerable overlap with other sections, and the papers should be consulted for full appreciation of their contributions to phosphole chemistry.

2.15.2 Theoretical Studies of Phospholes

Aromaticity and antiaromaticity in five-membered C_4H_4X ring systems: "classical" and "magnetic" concepts may not be "orthogonal" (95AG(E)337).

Ab initio studies of the dipole polarizabilities of conjugated molecules. Part 5. The five-membered heterocyclics C_4H_4E (95JST(331)109).

Effects of substituents on the aromatization of phosphole (95JPC586).

2.15.3 Experimental Structural Methods

Synthesis and x-ray crystal structural analysis of a fully unsaturated tetraphosphole macrocycle (95CC1561).

Synthesis, crystal structure, dynamic behavior and reactivity of dinaphtho[2,1-b:1',2'-d]phospholes and related atropisomeric phosphacyclic derivatives (94JOC6363).

2.15.4 Thermodynamic Aspects

Gas-phase properties and reactivities of phospholide and arsolide ions (94OM4732).

2.15.5 Reactivity of C.N.-3 Phospholes

Palladium-complex-promoted asymmetric Diels–Alder reaction: stereoselective synthesis of a new sulfinyl-substituted phosphine ligand containing three carbon, one phosphorus and one sulfur stereogenic centres <95CC1747>.

Thermal dimerization of 1-substituted-3,4-dimethylphospholes within the coordination sphere of platinum(II) <94IC109>.

X-ray structures of triphenylphosphine and 1,2,5-triphenylphosphole products with dimethyl acetylenedicarboxylate tetramer <94CJC2428>.

A 2,5-di-(*tert*-butyl)phospholyl complex containing a molybdenum–molybdenum triple bond: synthesis, molecular structure, and coordination chemistry of $[\text{Mo}_2(\text{PC}_4\text{H}_2\text{Bu}^t)_2(\text{CO})_4]$ <94CC2459>.

Use of the 2,5-bis-(*tert*-butyl)phospholide anion as an η^5 -ligand: stabilization of 5-phospholyl complexes of ruthenium and rhodium <94JCS1167>.

A novel coordination mode for cationic phosphorus p systems: μ^2 -bridging coordination of a bis(phosphonio)isophosphindolium cation <95JCS1541>.

Latest advances in phosphole chemistry <94PS(87)139>.

Recent advances in the chemistry of phospholide and polyphospholide ions <94JOM(475)25>.

The chemistry of phospho- and polyphosphacyclopentadienide anions <94CCR1>.

2.15.7 Reactivity and Synthesis of Phospholes with C.N.-2 and C.N.-5

The equilibrium between 1H- and 2H-phospholes: influence of the substitution pattern and scope <93BSF843>.

2.15.10 Ring Syntheses

Reaction of diphenyl-2-bromoalkenylphosphines with dimethyl acetylenedicarboxylate <94ZOB1566>.

2.15.12 Synthesis of Particular Types of Phospholes

Toward polyphospholes: synthesis and structure of an α -linked quaterphosphole <95AG(E)1158>.

Dinaphtho[2,1-b; 1',2'-d]phospholes: a new class of atropisomeric phosphorus ligands <93CC1124>.

2.16

Arsoles, Stiboles, and Bismoles

KENNETH C. CASTER

Lord Corporation, Cary, NC, USA

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2.16.1 INTRODUCTION

2.16.1.1 Historical Background

The heterocyclic chemistry of arsenic, antimony, and bismuth has its roots in the medicinal chemistry of the early 1900s. With the discovery that an organoarsenic compound provided a cure for syphilis, many new arsenic compounds were prepared and tested for potential medicinal properties. This also spurred interest in the heavier antimony and bismuth analogues. However, their decreased stability relative to the arsenic derivatives and the discovery that penicillin easily cured syphilis precluded the large scale investigation of these materials as medicinal agents. Between 1923 and 1950, many 5-membered ring arsenic and antimony heterocycles were prepared (e.g., arsinolones, arsa- and stibafluorenes), but the parent arsoles and stiboles were still unknown. Furthermore, no bismuth derivatives were known (B-50MI 216-01). By 1970, heavily substituted derivatives of arsoles and stiboles had been prepared. Only three dibenzobismole derivatives were known (B-70MI 216-01). Much progress has been made in the synthesis of unsubstituted arsole, stibole, and bismole derivatives in the 25 years from 1970–1995.

2.16.1.2 Relationship to CHEC-I

This review expands on the chapter by Atkinson in the first edition of *Comprehensive Heterocyclic Chemistry* (CHEC-I) <84CHEC-I(1)539> by detailing certain areas and highlighting the higher coordination states available to arsenic, antimony, and bismuth. Some new topics, such as the 1,1'-biheteroles and coordination compounds, are added. Experimental structural methods and thermodynamic aspects are expanded and include new information.

2.16.1.3 Previous Reviews

Most of the literature between 1970 and 1995 dealt with arsenic and antimony, with very little new material being published on bismuth chemistry. There are earlier reviews by Atkinson <84CHEC-I(1)539>, Mann <B-50MI 216-01, B-70MI 216-01>, and Doak and Freedman <B-70MI 216-02>. σ, π -Organometallic complexes of arsoles and stiboles were recently covered by Sadimenko <93CCR237>. Ashe reviewed thermochromism of these heterocyclic systems in 1990 <90AOC(30)77>. General information on arsenic, antimony, and bismuth chemistry may be obtained by consulting Wardell <82COMC-I(2)681> and Patai <B-94MI 216-01>. Organobismuth chemistry has been reviewed as well <82CRV15>.

2.16.1.4 Scope of Review

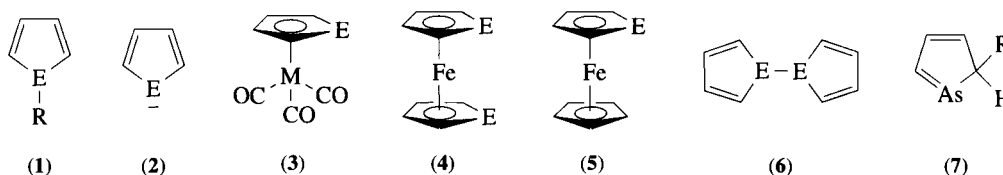
This review covers π -excessive five-membered ring systems containing one arsenic, antimony, or bismuth atom within the ring. Their benzo-, dibenzo-, unsaturated, partially unsaturated, and saturated analogues are also covered. These heteroatoms can have expanded valence shells and thus different coordination numbers (CN) are included as well. Although not strictly covered in this Chapter, mention is made of systems containing one of the above heteroatoms with another atom within the ring, but usually to show an important property of the system.

2.16.1.5 Survey of Structural Types

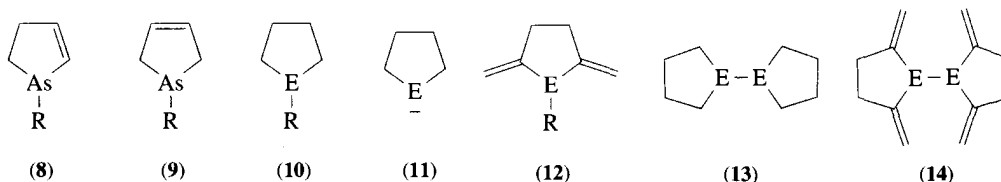
While many different structural types are known for heterocycles containing arsenic, antimony, or bismuth, structural diversity comes from the expanded CN that are available with these heteroatoms. Throughout the Chapter, the letter E, as used in the ring, represents As, Sb, or Bi and its use is explained in the text, Scheme, or Equation. In general, few ring systems exist that contain ring bound heteroatom substituents, and as a result, hydroxy-oxo, amino-imino, and thiol-thioxo tautomerism is nearly nonexistent.

2.16.1.5.1 Monocyclic compounds

The fully conjugated neutral (1) systems, known as arsoles, stiboles, and bismoles, and anionic (2) systems, known as arsoly, stiboly, and bismoly anions, exist for E = As, Sb, Bi. Many examples of both heavily substituted and unsubstituted systems are known, but the parents (1; R = H) do not exist. Derivatives include oxides and σ -organometallic complexes. These heteroles are poor nucleophiles and do not form quaternary salts. There is some debate as to the aromaticity of the neutral systems, whereas the anions are considered to be aromatic. This is clearly seen by examining the x-ray structures of π -complexes such as (3), diheteroferrocene (4), or monoheteroferrocene (5) where E = As, Sb, Bi. Dimeric systems such as (6) have been prepared (E = As, Sb, Bi) and are known as 1,1'-biheteroles. Of the 2*H*- and 3*H*-tautomeric species, two examples of the reactive 2*H*-arsoles (7) are known.

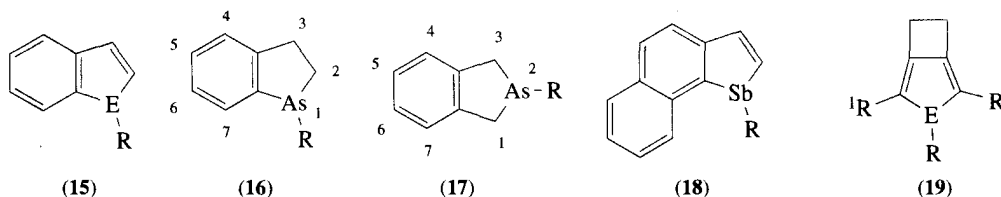


Of the partially unsaturated systems, only the 2- and 3-arsolenes (**8**) and (**9**) have been prepared, while no examples of stibolenes or bismolenes have been reported. Examples of the fully saturated systems (**10**), known as arsolanes, stibolanes, and bismolanes, and their anions (**11**), where E = As, Sb, and Bi, exist. Arsonium and stibonium quaternary salts are routinely found. Bis(methylene)-stibolane and -bismolane derivatives (**12**) and their anions have been prepared. Dimeric systems such as (**13**) and (**14**) have been prepared (for E = Sb, Bi) and are known as 1,1'-biheterolanes.

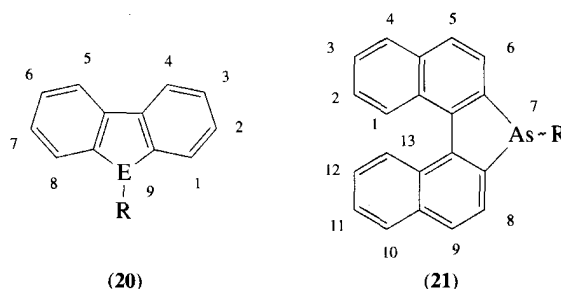


2.16.1.5.2 Benzo derivatives of CN-3

Monobenzo derivatives containing arsenic and antimony are known. For arsenic, multiple ring-fusion derivatives (i.e., benzo[*b*]-, benzo[*c*]-), which are commonly known as arsinindoles (**15**), arsinindolines (**16**), and isoarsindolines (**17**) have been studied in detail resulting in well-characterized quaternary salts, oxides, and organometallic complexes. For antimony, only the stibaindoles (**15**) are known. Isoindole type systems are unknown for any of the heteroatoms. The derivatives (**18**) and (**19**; E = Sb, Bi) exist.



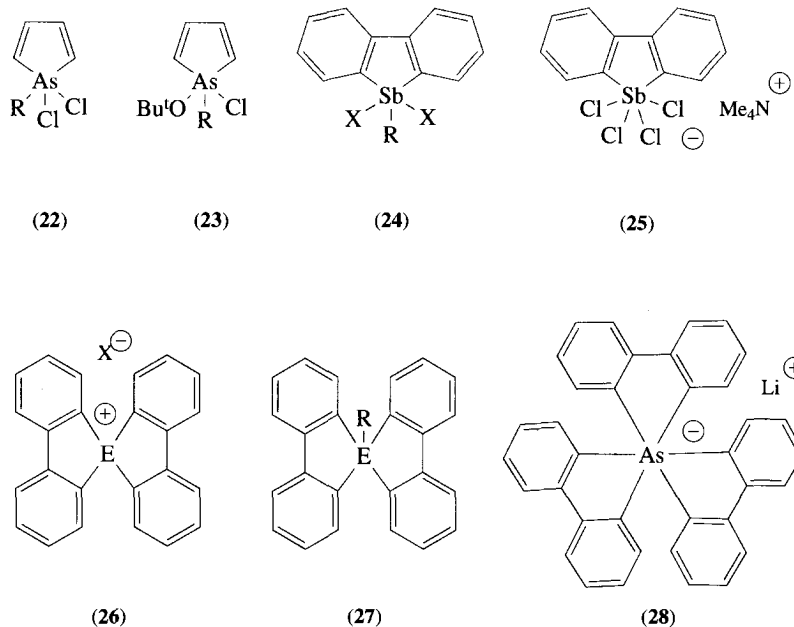
Dibenzo derivatives (**20**), which are commonly referred to as 9-heterofluorenes, exist (E = As, Sb, Bi). Quaternary salts, oxides, and organometallic complexes have been prepared. The dinaphth[2,1-*b*; 1'2'-*d*]arsole (**21**) has also been studied.



2.16.1.5.3 Compounds of CN-4, -5, and -6

The number of groups attached directly to the heteroatom defines its CN, and examples of CN-3 and -4 were discussed above. In this section, structurally different examples of CN-4, -5, and -6 are illustrated. Arsoles form the As^V adducts (**22**) and (**23**) with halogens and hypochlorites, respectively. The chlorostibole (**24**) is converted into the hexavalent anion (**25**). Spirocyclic deriva-

tives of CN-4 (**26**; E = As, Sb), -5 (**27**; E = As, Sb), and -6 (**28**; E = As) have been characterized. Anionic intermediates similar in structure to (**28**) are proposed intermediates in nucleophilic substitution reactions at arsenic.



2.16.2 THEORETICAL METHODS

Computational techniques are utilized heavily by the pharmaceutical industry to understand subtle structural changes to organic compounds. Great strides in applying these methods to heterocyclic compounds containing traditional atoms (i.e., C, H, N, O) have been made. However, application to the less common elements (i.e., P, S, As, Se, Sb, Bi) has been left to the specialist interested in specific properties of compounds containing these atoms. As a result, reports of computational techniques applied to these elements have been slow to emerge. No reports concerning the use of molecular mechanics (MM2) calculations to study structure or reactivity of arsenic, antimony, or bismuth heterocycles appeared up to 1995.

2.16.2.1 Semiempirical Calculations

Pyramidal inversion barriers of arsoles have been calculated using a modified version of CNINDO. Agreement between experimentally and computationally derived values is to within a few kcal mol⁻¹ over a wide range of barrier heights (10–60 kcal mol⁻¹) for a diverse set of structures that include arsoles and arsinholes. These results are used to argue the aromaticity of arsoles via (4*p*-2*p*) π electron delocalization <74JA6904>.

Molecular orbital theory provides an understanding of thermochromism and an explanation of the reversible color change for 1,1'-bistiboles <90AOC(30)77>. Hoffmann and coworkers used the tight binding (LCAO) method, which is based on extended Hückel calculations, to explain the thermochromic effect in terms of the one-dimensional electronic band structure of 1,1'-bistiboles and to account for the observed UV red shift. Inclusion of 5*d* orbitals is important in explaining thermochromism in tetraalkyldistibines <82JA3876>. Similar methodology finds the valance band localized on antimony and the conduction band localized on the carbon π -orbitals <87OM1185>.

Effective core potential, REX, and relativistically parameterized semiempirical molecular orbital calculations are used to understand inter- and intramolecular bonding and how it relates to thermochromism in bibismuthines. All the calculations suggest modest secondary intermolecular Bi $\cdots$ Bi bonding <93OM343>.

Modified CNDO/S calculations have been used to aid interpretation of UV PE spectra of 1*H*- and 3*H*-benzazarsoles. Good agreement is obtained between experimental and calculated ionization potentials and orbital energies <89JOM(373)49, 89JOM(373)57>.

2.16.2.2 *Ab Initio* Calculations

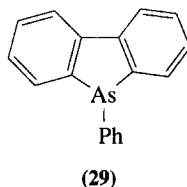
Bond separation and superhomodesmotic reaction energies, as determined from *ab initio* molecular orbital calculations at the SCF/3-21G* level, suggest there is only minor lone-pair electron delocalization for arsenic and antimony into arsole and stibole rings <88JA4204>.

Structures and relative stabilities of AsH₃, AsH₄⁺, AsH₄⁻, AsH₅, and AsH₆⁻ are determined by *ab initio* effective potential calculations at SCF and CI levels. The calculated Berry pseudorotation barrier in AsH₅ is 2.5 kcal mol⁻¹ <84JA4794>. *Ab initio* methods have also been used to calculate gas-phase proton affinities for simple arsines <84JST(109)195>.

2.16.3 EXPERIMENTAL STRUCTURAL METHODS

2.16.3.1 X-ray Diffraction

Structural information on simple, relatively unsubstituted arsoles, stiboles, and bismoles is lacking as the substances are usually oils; and they have not been analyzed by microwave spectroscopy. X-ray structures are solved for 9-phenylarsafluorene (**29**) <63JCS4414>, a dinaphtharsole (**21**) <93JOM(445)71>, and a stibaindole <90AG(E)771>. Ashe has reported on many x-ray analyses for the 1,1'-biheteroles <81JA207, 83OM1005, 90AOC(30)77, 92OM2743> and heteroferrocenes <91OM2068, 92JA372, 92OM1491, 92OM2743> and has related their structural similarities and differences. In general, x-ray structural data of the arsafluorene, stibaindole, and the 1,1'-biheteroles show the rings to be planar and the heteroatom to be highly pyramidalized.



2.16.3.2 NMR Methods

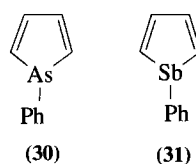
NMR data is nonexistent for parent arsoles, stiboles, or bismoles, but is available for more substituted derivatives. In this section, NMR shifts are tabulated to provide an idea of the differences between molecular types. Proton and ¹³C NMR data is available for trimethylsilyl substituted antimony and bismuth heteroles and their complexes <92OM1491>, a variety of arsenic complexes <79ICAL67, 87AG(E)229>, diheteroferrocenes <91OM2068, 92OM2743>, and for bis(methylene)heterolane derivatives <87OM1185>.

2.16.3.2.1 Proton NMR spectroscopy

Selected ¹H NMR data for arsoles, stiboles, bismoles, and their 1,1'-biheterole derivatives is displayed in Table 1. Relative to 1-phenylpyrrole and -phosphole, the C-2,5 ring protons of the arsole (**30**) and stibole (**31**) are deshielded, which has been related to anisotropy of the heteroatom and not necessarily to ring current effects <85OM1478>. Arsolyl σ-complexes are well-characterized by ¹H NMR <94JOM(467)67>. Dynamic NMR has been used to determine rotational barriers of the diheteroferrocenes, but inclusion here is beyond the scope of this review. Atropisomerism of the dinaphtharsole (**21**) has been studied by dynamic NMR <93JOM(445)71>.

Table 1 Proton NMR spectral data for arsoles, stiboles, bismoles, and 1,1'-biheteroles.^a

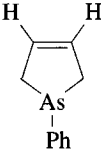
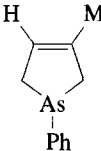
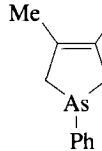
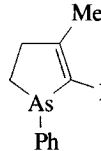
Compound	H-2,5	H-3,4	Other	Solvent	Reference
1-Phenylarsole (30)	7.20 ^b	7.25 ^b	7.29–7.35	CD ₂ Cl ₂	85OM1478
1-Phenylstibole (31)	7.53 ^b	7.57 ^b	7.25–7.50	CD ₂ Cl ₂	85OM1478
1-Phenyl-2,5-dimethylarsole (35)		6.51	1.99 (Me)	CDCl ₃	83JOM(249)335
1-Phenyl-2,5-dimethylstibole (125)		6.79	2.13 (Me)	CDCl ₃	91OM2068
1-Phenyl-2,5-dimethylbismole (44)		8.53	2.35 (Me)	CD ₂ Cl ₂	84OM495
1-Methyl-2,5-dimethylstibole		6.80	0.9 (SbMe)	CDCl ₃	80JOM(202)C95
			2.10 (Me)		
Tetramethyl-1,1'-biarsole (148)		6.49	1.99 (Me)	THF-d ₈	83OM1005
Tetramethyl-1,1'-bistibole (150)		6.70	2.10 (Me)	C ₆ D ₆	80JOM(202)C95
Tetramethyl-1,1'-bibismole (152)		8.43	2.41 (Me)	C ₇ D ₈	84OM495

^aUnits are in p.p.m. ^bTentative assignment.

The pyramidal geometry of arsenic in 3-arsolenes is revealed by the display of AB spin systems for the α -methylene protons in their ¹H NMR spectra. Some arsolene ¹H NMR data is shown in Table 2. Their salts and organometallic complexes are also characterized by ¹H NMR <90JOM(396)C8, 94JOM(467)57>.

The ¹H NMR data for saturated ring systems, as recorded in the original papers, lacks fine structural information and only gives a gross indication of ring proton chemical shifts and is therefore not included.

Table 2 Proton NMR spectral data for arsolenes.^a

				
	(128)	(134)	(135)	(146)
H-2	2.62, 2.79	2.48, 2.73 ^b	2.46, 2.72	6.04
H-3	6.02			
H-4	6.02	5.68		2.55
H-5	2.62, 2.79	2.54, 2.77 ^b	2.46, 2.72	2.55
Me		1.87	1.74	1.96
² J _{H-H}	15.3	16.0, 16.4	15.5	
Solvent	CDCl ₃	CDCl ₃	CDCl ₃	CDCl ₃
Reference	93JOM(450)C4	93JOM(450)C4	93JOM(450)C4	79TL3157

^aUnits are in p.p.m. and Hz. ^bUncertain assignment.

2.16.3.2.2 Carbon-13 NMR spectroscopy

Arsoles, stiboles, and bismoles are fairly well-characterized by ¹³C NMR, and this technique is used routinely. Select data for these compounds, as well as for 1,1'-bistiboles and -bibismoles, is included in Table 3. Occasionally, the bismuth α -carbon is broad or is not observed <92OM3492, 93JOM(447)197>. Table 4 contains ¹³C NMR data for tricoordinate arsolenes; data for other derivatives, their salts, and organometallic complexes may be found elsewhere <90JOM(396)C8, 94JOM(467)57>.

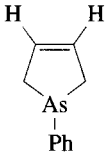
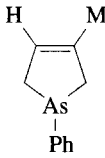
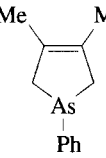
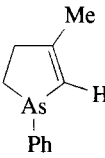
Virtually no ^{13}C NMR data has been published for saturated systems. The 1,1'-bibismolane (**32**) displays a broad α -carbon resonance at 17.8 p.p.m and its β -carbon at 40.5 p.p.m. in its ^{13}C NMR spectrum <83OM1859>.

Table 3 ^{13}C NMR spectral data for arsoles, stiboles, and 1,1'-biheteroles.^a

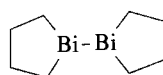
Compound	C-2,5	C-3,4	Other	Solvent	Reference
1-Phenylarsole (30)	140.0 ^b	142.2 ^b	134.8 (ipso)	CD_2Cl_2	85OM1478
1-Phenylstibole (31)	142.0 ^b	147.1 ^b	134.6 (ipso)	CD_2Cl_2	85OM1478
1-Phenyl-2,5-dimethylstibole (125)	154.5	141.6	21.9 (Me)	CDCl_3	91OM2068
1-Methyl-2,5-dimethylstibole	154.5	141.6	29.7 (SbMe) 21.9 (Me)	CDCl_3	80JOM(202)C95
Tetramethyl-1,1'-bistibole (150)	151.7	140.6	22.5 (Me)	C_6D_6	80JOM(202)C95
Tetramethyl-1,1'-bibismole (152)	162.8	152.3	28.5 (Me)	C_7D_8	84OM495

^aUnits are in p.p.m. ^bTentative assignment.

Table 4 ^{13}NMR spectral data for arsolenes.^a

				
	(128)	(134)	(135)	(146)
C-2	34.0	37.8	39.8	not recorded
C-3	131.0	140.5	130.8	not recorded
C-4	131.0	125.3	130.8	40.4
C-5	34.0	33.5	39.8	25.4
Me		19.3	17.7	20.1
Solvent	CDCl_3	CDCl_3	CDCl_3	CDCl_3
Reference	93JOM(450)C4	93JOM(450)C4	93JOM(450)C4	79TL3157

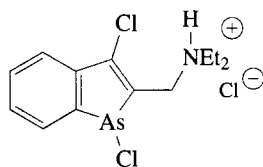
^aUnits are in p.p.m.



(32)

2.16.3.3 Mass Spectroscopy

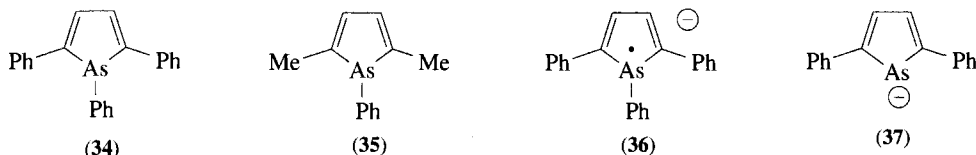
Arsoles, stiboles, and bismoles are analyzed by electron impact (EI, 70 eV) and chemical ionization ($\text{CI}-\text{CH}_4$) MS. Molecular ions are easily observed for all compound types included in this review. Similarities between fragmentation patterns of the arsendole (**33**) with other 5-membered ring aromatic heterocycles have been noted <80JHC1341>. Hellwinkel *et al.* have analyzed and compared fragmentation patterns for dibenzoarsoles, -stiboles, and -bismoles; and for arsenic and antimony derivatives of spirocyclic bis-2,2'-biphenylenes (**27**) <73PS(2)167>. Common EI fragmentation ions for 1-*t*-butyl-, 1-chloro-, and 1-bromoarsolane include the molecular ion and AsCH_2^+ <90PS(48)157>. General fragmentation patterns are known for 1*H*-benzazarsoles <89JOM(364)C17>.



(33)

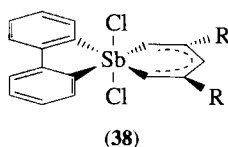
2.16.3.4 Ultraviolet Spectroscopy

UV absorption maxima ($\lambda_{\max}$ nm (log ϵ), pentane) are known for 1-phenylarsole (**30**) (274 (3.67), 223 (3.81)); 1-phenylstibole (**31**) (283 (3.45), 230 (4.00), 216 (4.22)) <85OM1478>; 1,2,5-triphenylarsole (**34**) (368 (4.18), 233 (4.23), 220 sh (4.26), 208 (4.41)); and 1-phenyl-2,5-dimethylarsole (**35**) (263 (4.56), 207 (4.52)). The $\lambda_{\max}$ for the arsolyl radical anion (**36**) and anion (**37**) in DME are near 610 nm and 650 nm, respectively <83JOM(249)335>. Diffuse reflectance and UV absorption spectra of the 1,1'-biheteroles are described by Ashe <90AOC(30)77>. UV absorption spectra of the heteroferrocenes (**4**) and (**5**) have been recorded <91OM2068, 92OM1491>.



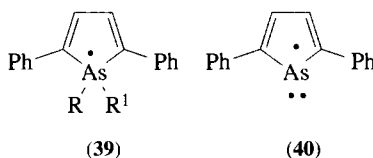
2.16.3.5 Infrared and Raman Spectroscopy

Scattered IR spectral data exists for many of the compounds illustrated in this review, but very little has been assigned to specific ring vibrations. Meinema *et al.* have characterized ring skeletal vibrations for stibolane derivatives <76JOM(110)183> and made conformational assignments for the hexacoordinate, octahedral species (**38**) and related stibolane derivatives using IR spectroscopic analyses <78JOM(160)435>. Raman spectroscopy of the 1,1'-biheteroles is described by Ashe <90AOC(30)77>.



2.16.3.6 Electron Spin Resonance Spectroscopy

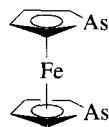
Radical chemistry has not played an important role in the heterocyclic chemistry of arsenic, antimony, or bismuth compounds. However, arsolyl radicals (e.g., (**39**), (**40**)) are proposed intermediates in alkylations (Section 2.16.5.3.2) and are observable by ESR during the formation of radical anions (Section 2.16.10.1) such as (**36**) <72AG(E)439, 83JOM(249)335>.



2.16.3.7 Electrochemistry

The arsole (**34**) has been studied by cyclic voltametry (Pt, MeCN, 0.1 M $\text{Bu}_4\text{N}^+\text{ClO}_4^-$). It undergoes reversible one-electron redox chemistry with the radical anion (**36**) ($E_0 = -1.77$ V vs the saturated calomel electrode (SCE)) <83JOM(249)335>. Cyclovoltammetry of the 1,1'-diarsaferrocene (**41**) gives redox potentials for the arsole/radical cation of $E_{1/2} = 0.730 \pm 0.015$ V (-38°C in

DME/tetra-*n*-butylammonium perchlorate (TBAP) vs SCE, $v \geq 20 \text{ mV s}^{-1}$, $I_{\text{pa}}/I_{\text{pc}} = 1.0$) and for the arsole/radical anion of $E_{1/2} = -2.190 \pm 0.01 \text{ V}$ (-40°C in DME/TBAP vs SCE, $v \geq 1 \text{ mV s}^{-1}$, $I_{\text{pa}}/I_{\text{pc}} = 0.98$) $\langle 87\text{AG(E)229} \rangle$.



(41)

2.16.4 THERMODYNAMIC ASPECTS

2.16.4.1 Boiling and Melting Points

Most simple heteroles are liquids or viscous oils that can be Kugelrohr distilled under reduced pressure. Many of the solid heteroles are isolated by sublimation. Physical property data for a few of the simple compounds are included in Table 5. Quaternary salts, metal complexes, oxides, and sulfides tend to be crystalline solids with definite melting points.

Table 5 Physical property data of some simple heteroles.

Compound	Color	<i>m.p.</i> ($^\circ\text{C}$)	<i>b.p.</i> ($^\circ\text{C}$)	Torr	Reference
1-Phenylarsole (30)	light yellow	oil	95 ^a	0.005	85OM1478
1-Phenylstibole (31)	yellow	oil	70 ^a	0.005	85OM1478
1-Methylarsolane (137)	colorless	liquid	65–66	15	77JCS(D)704
1-Phenylarsolane (138)			130–134	20	77JCS(D)704
1- <i>t</i> -Butylarsolane (139)			57	3	90PS(48)157
1-Methylstibolane (89)	colorless	liquid	67–68	30	76JOM(110)183
1-Phenylbismolane (142)	colorless	liquid	78–80	0.1	83OM1859
1,1'-Biarsole (148) ^c	yellow	74–75	40 ^b	0.05	83OM1005
1,1'-Bistibole (150) ^c	deep blue	99			80JOM(202)C95
1,1'-Bibismole (152) ^c	black-green	101–102	45–52 ^b	0.01	92OM2743

^aKugelrohr distilled. ^bSublimed. ^cAll derivatives are 2,2',5,5'-tetramethyl substituted.

2.16.4.2 Solubility and Chromatographic Behavior

Arsoles, stiboles, bismoles, and their nonpolar derivatives display solubility behavior typical of organic compounds and dissolve in common solvents (e.g., CHCl_3 , benzene, THF, ether, hexane). Many are purified by low temperature crystallization from pentane.

While many of these heterocycles are plagued by air-sensitivity, some may be chromatographed on a variety of supports using common solvents. This usually requires thorough degassing of the support and solvent prior to compound application, followed by isolation under inert atmosphere. Organometallic complexes are routinely purified in this manner (Table 6).

As-Alkyl, -chloro, and -bromo arsolanes can be analyzed by GLC using a packed column of SE-30 adsorbed on Chromosorb P (injection port temperature = 298°C , oven temperature = 150°C) $\langle 90\text{PS(48)157} \rangle$. Bismuth compounds, which tend to decompose on GLC, have been analyzed on 5% SE-30 adsorbed on Carbowax $\langle 83\text{OM1859} \rangle$.

Table 6 Compound class and chromatography system.

Compound class	Stationary phase	Solvent system	Reference
Arsoles	Alumina	Benzene	83JOM(249)335
As-Phenylarsoles	Alumina (neutral)	None listed	83JOM(248)269
As-Phenylarsole oxides	Silica gel	Benzene/MeOH	61JA4406
Sb-Phenylstiboles	Alumina (III)	Hexane	94JA1880
Sb-Phenyldibenzostiboles	Alumina	Benzene	59JCS3101
As-Phenylarsindoles	Alumina (II)	Hexanes/ether (90 : 10)	90AG(E)771
As-Chloroarsindoles	MgSO ₄	Hexanes/ether (90 : 10)	90AG(E)771
Arsindole As-oxides	Silica gel	Benzene/EtOH (95 : 5)	72JA2861
3-Arsolenes	Kieselgel	Petroleum ether	94JOM(467)57
3-Arsolene As-sulfides	Kieselgel	Acetone	94JOM(467)57
2- and 3-Arsolenes	Silica gel	Pentane	79TL3157
1,1'-Biarsolenes	Kieselgel	Petroleum ether	94JOM(467)57
Arsolyl σ -complexes	Silica gel	Benzene/pentane (50 : 50)	79ICAL67
3-Arsolene σ -complexes	Kieselgel	Petroleum ether	90JOM(396)C8
Arsaferrocenes	Silica gel	Pentane/benzene (80 : 20)	79ICAL67
Stibolyl π -complexes	Silica gel	None listed	80JOM(202)C95

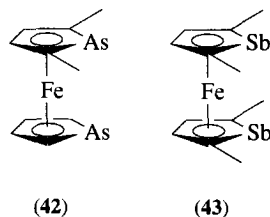
2.16.4.3 Aromaticity and Stability

2.16.4.3.1 Aromaticity and electron-pair delocalization

Defining relative “aromaticity” has always been difficult, particularly for heteroaromatic substances, and is no less difficult for arsoles, stiboles, and bismoles. Classical physical approaches provide contradictory evidence of aromaticity. In the ¹H NMR, ring proton deshielding increases with increasing heterole atomic number (**1**; E = N, P, As, Sb, R = Ph) and is related to anisotropy of the heteroatom and not necessarily to ring current effects <85OM1478>; a behavior also displayed by group 5 heterobenzenes <82JA5693>. X-ray structural data on stibaindoles <90AG(E)771>, 1,1'-bistiboles <81JA207, 91OM2068>, and bismoles <93JOM(447)197> shows the rings to be planar and the heteroatom to be highly pyramidalized. On the other hand, comparison of UV spectra and MS fragmentation patterns for arsindeoles with indoles and phosphindeoles suggests there is lone-pair delocalization into the π -system <80JHC1341>. The substantially lower pyramidal inversion barriers of arsoles (10–15 kcal mol⁻¹) compared with simple 3° arsines suggests extensive delocalization in the planar inversion transition state. It may be argued that little delocalization occurs in the pyramidal ground state, but Mislow and coworkers argue, on molecular orbital grounds, that some ground state (4p–2p) π delocalization does occur for arsoles. Phospholes display similar behavior. Semiempirically calculated inversion barriers show good agreement with the experimental values <74JA6904>.

Molecular orbital calculations at the SCF/3-21G* level, and bond separation and superhomodesmotic reaction energies suggest there is only minor lone-pair electron delocalization for arsole and stibole <88JA4204>.

Arsolyl, stibolyl, and bismolyl anions are considered aromatic. Proton affinities, as calculated by semiempirical methods, support this by showing that bismole (**1**; E = Bi, R = H) has higher relative acidity than BiH₃ and that this is consistent with aromatic stabilization of the anion (**2**; E = Bi) <93OM343>. The anions serve as 6 π -electron ligands in the metal complexes (**3**), (**4**), and (**5**). ¹J β -CH Coupling constants (**42**; 168 Hz, **43**; 164 Hz) are similar to ferrocene (175 Hz) suggesting similar coordination in the complex. The larger CCC bond angles explain the ¹J change <80JOM(202)C95>. Bismuth–carbon bond lengths in bismaferrocenes suggest multiple bond character as in other pnictogen analogues <92JA372>.



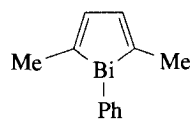
1*H*-Benzazarsoles are considered aromatic by comparative examination of their structures with those of other benzazaheteroles by x-ray <83ZAAC(501)146>, UV PE spectra, CNDO/S calculations <89JOM(373)49>, NMR, and UV spectroscopy. Little delocalization of the lone-pair electrons is observed for 3*H*-benzazarsoles by these techniques <78JOM(154)1, 89JOM(373)57>.

New measures of aromaticity such as Parr and coworkers, absolute and relative hardness <89JA7371> or Bird's aromaticity index <85T1409> have yet to be applied to these systems.

2.16.4.3.2 Stability

Stability follows the general trend for arsenic, antimony, and bismuth compounds in that it decreases down the column <82COMC-I(2)681>, for example, 1-phenylarsole is an air-sensitive, light yellow oil that is stable at room temperature under an inert atmosphere. 1-Phenylstibole darkens and slowly resinifies on standing at room temperature under an inert atmosphere <85OM1478>. 1-Phenylbismole has yet to be prepared, but 1-phenyl-2,5-dimethylbismole (**44**) is labile at room temperature and is used without extensive purification <84OM495>. Benzo and dibenzo derivatives tend to have higher thermal stabilities. Increasing ring substitution and substituting heteroatoms with alkyl and aryl groups rather than with halogens also increases stability. Quaternary salts, oxides, sulfides, and certain CN-5 compounds are very stable. Free radical processes are implicated in thermal decomposition of tetraalkylarsines, -stibines, and -bismuthines (e.g., 1,1'-bibismolane) <83OM1859>.

Most of the tricoordinate heterocycles are air-sensitive. Their handling requires at least inert atmosphere techniques, but in certain cases and particularly for bismuth compounds, Schlenk line techniques and glove boxes are required. Increasing CN decreases or eliminates air-sensitivity.



(44)

2.16.4.4 Conformations

Conformational properties of these heterocyclic systems usually result from the increased CN numbers of arsenic, antimony, and bismuth. Tricoordinate systems show interesting solid state conformational behavior as displayed in 1,1'-biheteroles (Sections 2.16.10.2 and 2.16.11.2) and organometallic complexes (Section 2.16.10.3). More detailed treatment of this subject is found in the references in these sections.

2.16.4.4.1 Geometry at the heteroatom and inversion barriers

The heteroatom in arsoles, stiboles, and bismoles has a pyramidal geometry. Barriers to inversion in arsendoles, which have been determined by NMR equilibration experiments, ($\Delta G_{151}^\ddagger = 35.2 \text{ kcal mol}^{-1}$) are lower than in comparable acyclic arsines ($\Delta G_{218}^\ddagger = 43.1 \text{ kcal mol}^{-1}$) and this is attributed to $(4p-2p)\pi$ delocalization of the electron-pair. Arsendolines, which lack the ability for such delocalization, have inversion barriers of 46–48 kcal mol^{-1} . These barriers are lowered by direct attachment of silicon substituents, which has been related to the substituent's electronegativity <72JA2859, 72JA2861>. Calculated inversion barriers (kcal mol^{-1}) are shown below with their arsole derivatives (Figure 1) <73M1 216-01, 74JA6904>. Although no inversion barrier data was found for

antimony and bismuth heterocycles, they too are expected to have high barriers based on their geometries.

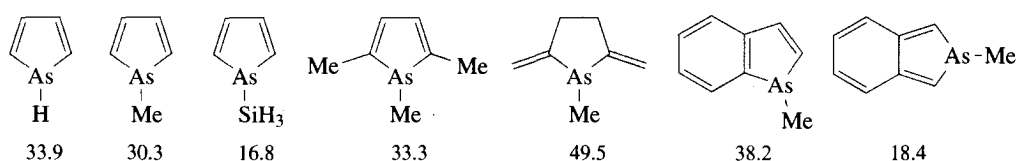


Figure 1 Inversion barriers of heteroatoms (kcal mol⁻¹) and arsole derivatives.

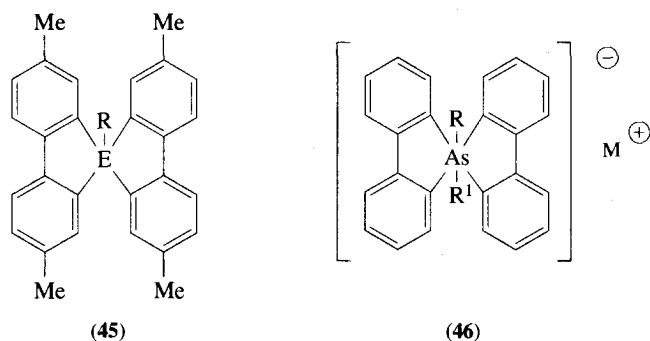
The stable pyramids associated with these heteroatoms should allow optically active compounds to be prepared. The dinaphtharsole (**21**), while potentially chiral at arsenic, undergoes rapid room temperature atropisomerism of the binaphthalene system which precludes isolation of individual enantiomers <93JOM(445)71>. Optically active dibenzoarsoles and -stiboles have been prepared <50JCS3109, 52JCS4448, 55JCS1662, 84CHEC-I(1)539>.

2.16.4.4.2 Conformational equilibria of trigonal bipyramid and octahedral compounds

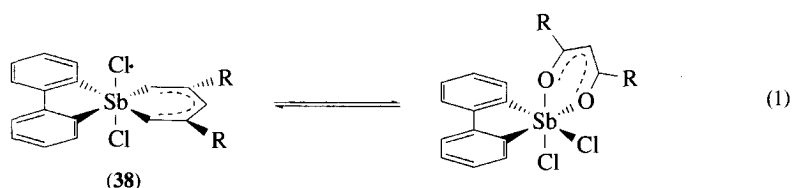
Attainment of high CN provides a pathway for polytopal isomerization. X-ray studies on spirocyclic arsoranes show them to have trigonal bipyramidal, rectangular pyramidal, and intermediate structures <85OM708, 85OM714>. Computational studies on arsoranes and phosphoranes provide relative structural energies between isomers and energetics of Berry pseudorotational processes <75JA5379, 84JA4794>.

Spirocyclic derivatives (**45**; E = As, Sb) have ground state trigonal bipyramidal geometries. Dynamic NMR studies reveal that intramolecular isomerization occurs readily at room temperature, and for E = As have free energies of activation of 12–15 kcal mol⁻¹ that are dependent on the bulkiness of the R group <69JOM(17)389, 72PS(2)129>.

The conformational properties of the anion (**46**) have been studied as they relate to nucleophilic addition to the precursor pentacoordinate species <64CB769, 68CB121>. The hexacoordinate anion (**28**) is conformationally stable and has been prepared as a chiral salt. Its protonation has been studied from a stereochemical standpoint <67LA(705)66>.



The stereochemistry of the hexacoordinate, octahedral species (**38**) has been determined from ¹H NMR, IR, and dipole moment studies. Freshly prepared solutions of *trans*-dichloro (**38**) slowly isomerize to the 1 : 1 mixture of the *cis*- and *trans*-dichloro compound through a twist mechanism (Equation (1)). Similar processes occur for stibolane derivatives. No exchange with free 1,3-diketone occurs <78JOM(160)435>. Mössbauer spectroscopy has been used to study the conformational properties of these molecules <77JOM(136)173>.



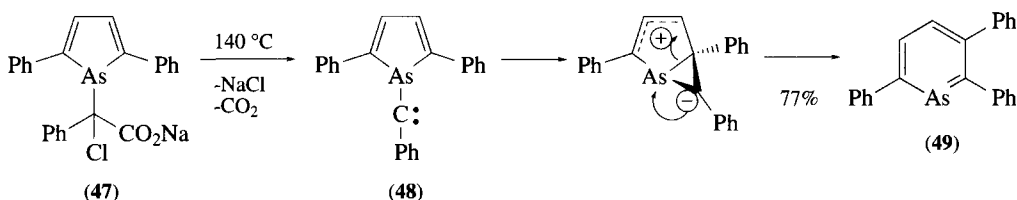
2.16.5 REACTIVITY OF FULLY CONJUGATED RINGS

2.16.5.1 General Survey of Reactivity

While heterocyclic chemistry of traditional atom systems is relatively mature, heterocyclic chemistry of the less common hetero systems is in its infancy. The chemistry of these ring systems is governed by the reactivity of the heteroatom. Very few examples of electrophilic or nucleophilic reactions at carbon or at substituents attached to ring carbon atoms, or of ring hydrogen deprotonation, are known. Arsines are less nucleophilic than phosphines, and this property carries over to the arsoles, which have chemistry closer to pyrroles than to phospholes <72AG(E)441, 72AG(E)441a>.

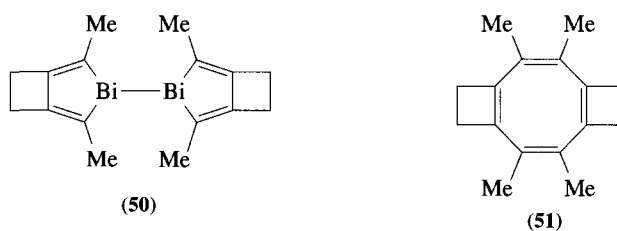
2.16.5.2 Unimolecular Thermal Reactions

Thermolysis of the arsole (47) at slightly above its melting point produces a good yield of the arsabenzene (49) by the pathway shown in Scheme 1. Generation of the carbene (48) by thermolysis is necessary since α -elimination pathways are precluded by reactivity of the ring with organometallic bases <72AG(E)441b>.



Scheme 1

The 1,1'-bibismole (50) decomposes at room temperature in benzene solution to give the tetraene (51) (Scheme 36). Thermal rearrangements of organometallic derivatives of arsoles, stiboles, and bismoles are discussed in Section 2.16.10.3.

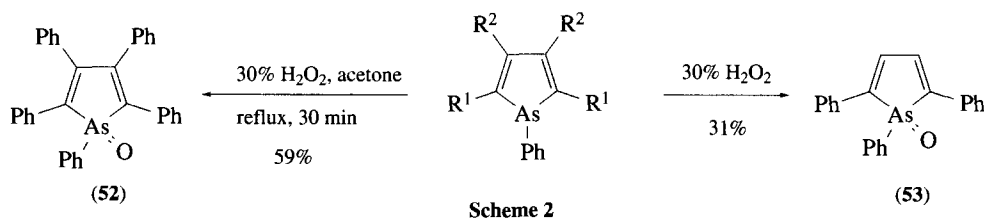


2.16.5.3 Electrophilic Attack at the Heteroatom

2.16.5.3.1 Neutral ring systems

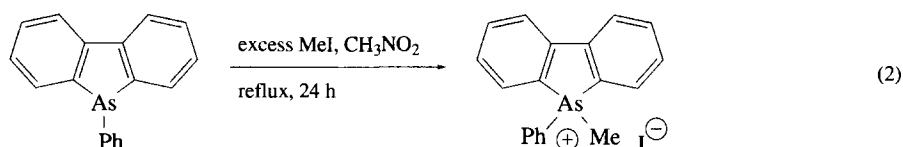
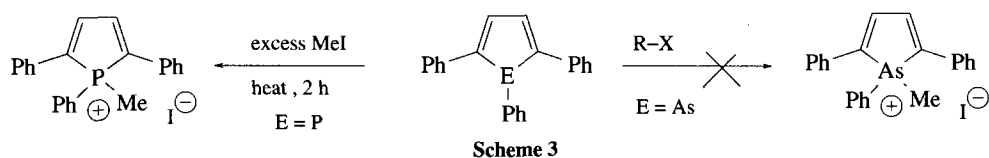
(i) Oxidation

Arsoles are oxidized to their *As*-oxides by 30% H_2O_2 in acetone solution (Scheme 2). Yields increase with increasing ring substitution, as is seen in the preparation of the oxides (52) <61JA4406> and (53) <68TL3257, 72AG(E)441a>. Arsindoles <72JA2861> and dibenzoarsoles <63JCS4414, 71CB1761, 76JOM(110)315> are also oxidized in this way. Stiboles are oxidized by air to their corresponding *Sb*-oxides <61JA4406>. 1,2,5-Triphenylarsole does not form its *As*-sulfide on reaction with sulfur <72AG(E)441a>.



(ii) Alkylation

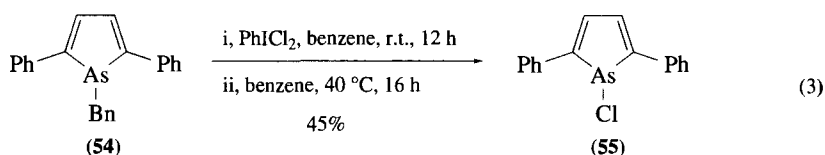
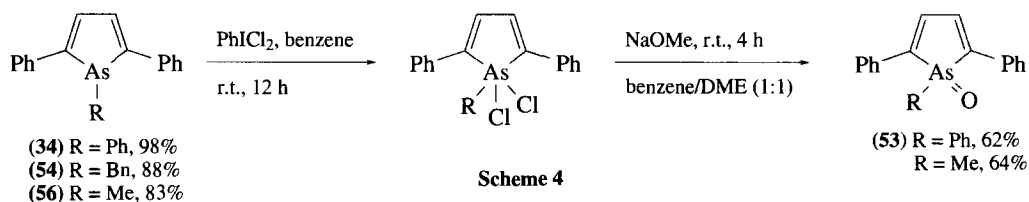
Unlike phospholes <65JCS2184>, simple arsoles do not form quaternary salts on reaction with alkyl halides (Scheme 3) <68TL3257, 72AG(E)441a>. Dibenzoarsoles are quaternized with alkyl iodides <59JCS3101, 76JOM(110)315> or bromides <63JCS4414> at elevated temperatures (Equation (2)).



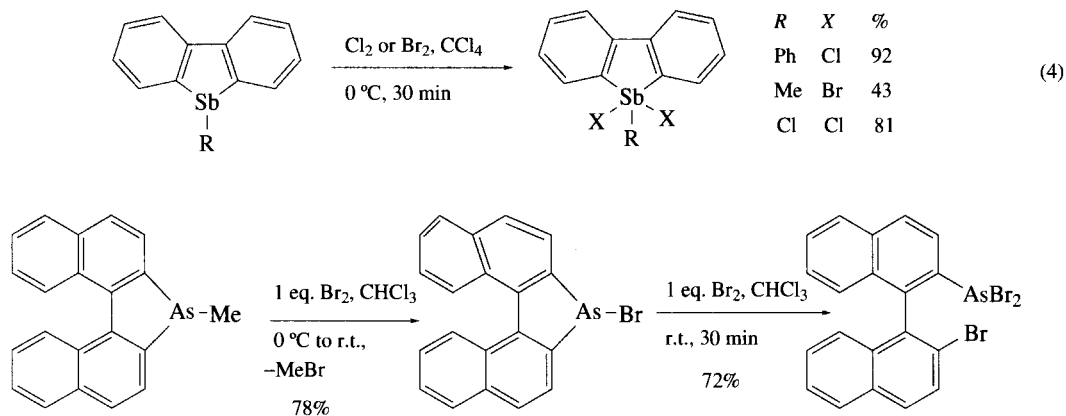
1-*t*-Butyl-2,5-diphenylarsole is alkylated by BnCl/Bu^tOK at 50–60 °C to give the arsabenzene (49) in 4% yield <74TL203>. 2-*t*-Butyl-1,3-benzoxarsole displays interesting reactivity by being alkylated with MeI and CCl₄ under forcing conditions to give λ⁵- and (λ³-dihydro)benzoxazoles, respectively <84PS(20)347>.

(iii) Halogenation

Arsoles add halogens to give stable 1 : 1 adducts that are pentacoordinate in nonpolar solution. Reaction of 1-substituted-2,5-diphenylarsoles with PhICl₂ gives stable, isolable adducts that are unreactive with H₂O or alcohols, but undergo reactions with alkoxides (Scheme 4). They are thermally stable, except for the *As*-benzyl derivative (54) which loses BnCl at room temperature to give the *As*-chloroarsole (55) (Equation (3)) <72AG(E)441a, 83JOM(248)269>.

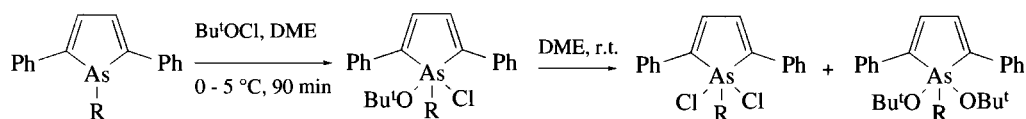


As in Equation (4), dibenzostiboles form similar adducts with Cl_2 , Br_2 <69JOM(17)389> and sulfonyl chloride <77JOM(136)173>. Arsoles appear to be more sensitive to overhalogenation as ring cleavage can result (Scheme 5) <93JOM(445)71>.



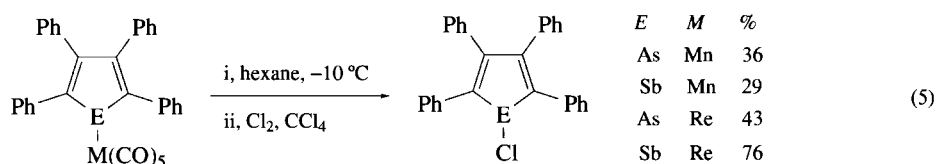
Scheme 5

Arsoles form voluminous 1:1 adducts with *t*-butyl hypochlorite that disproportionate at room temperature (Scheme 6). Although not isolated because of their hydrolytic and thermal instability, they may be trapped by nucleophiles (Section 2.16.6.5.3) to give new arsole derivatives <72AG(E)441a, 83JOM(248)269>.



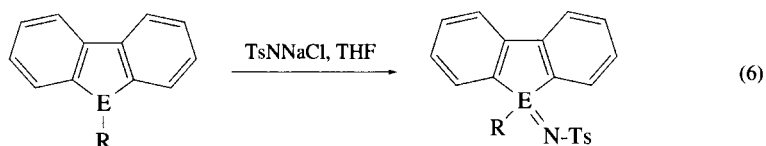
Scheme 6

σ -Organometallic derivatives are cleaved to chloro- or bromoarsoles and -stiboles with Cl_2 or Br_2 (Equation (5)) <79JCS(D)814>. More organometallic derivative reactions of fully conjugated systems are covered in Section 2.16.10.3.



(iv) Miscellaneous reactions

Dibenzoarsoles <68CB121> and -stiboles react with chloramine-T to give their respective imines (Equation (6)), which are used *in situ* to prepare pentacoordinate derivatives. Dibenzbismoles do not react in this manner <64CB789>.



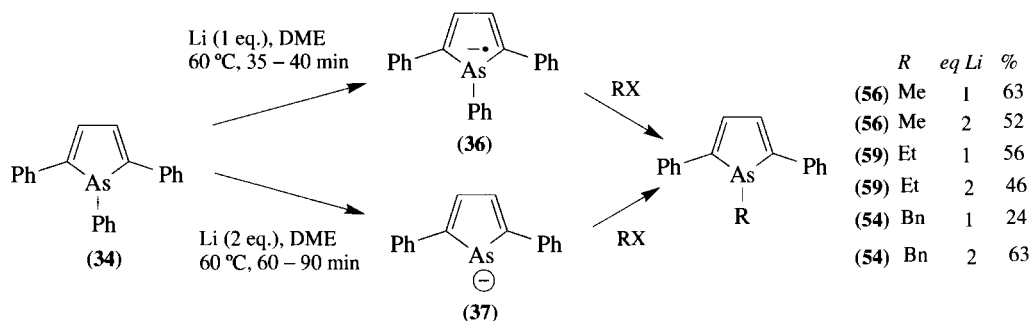
2.16.5.3.2 Anionic and radical anion ring systems

The preparation of anions is described in Section 2.16.10.1.

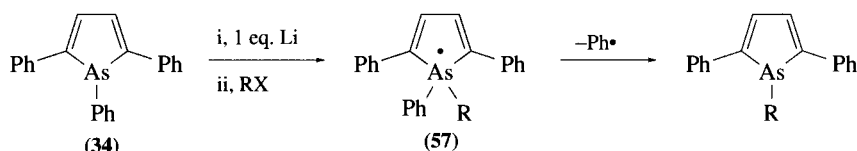
(i) Alkylation with carbon electrophiles

Arsole anions and radical anions are alkylated with alkyl halides to give the expected products in poor to moderate yields. Higher yields are obtained by alkylation of the radical anion (Scheme 7), which is thought to involve the As^V intermediate (57) followed by loss of phenyl radical (Scheme 8). The *As*-alkyl (56), opposed to the *As*-phenyl arsole (34), forms a stable dianion with two equivalents of lithium, which on alkylation is believed to proceed via the As^V species (58) to give the new arsole (59) (Scheme 9) <72AG(E)439, 83JOM(249)335>. Alkylation of arsoles with CH_2Cl_2 , benzal chloride, and chloroacetates yields the arsoles (47) and (60)–(64) <68TL3257, 72AG(E)441a, 72AG(E)439, 83JOM(249)335>.

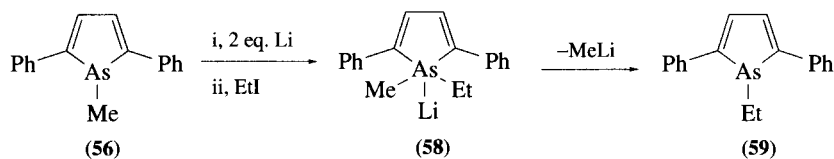
Arsindole <72JA2861> and stibole <80JOM(202)C95> anions are alkylated as well.



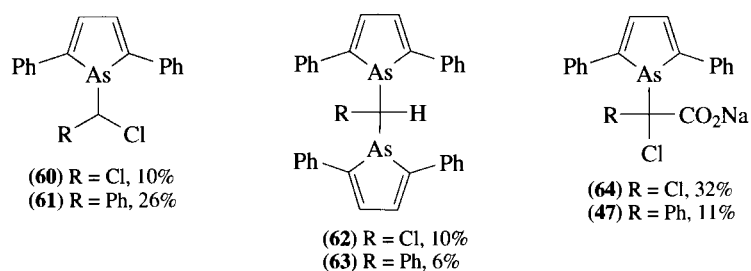
Scheme 7



Scheme 8

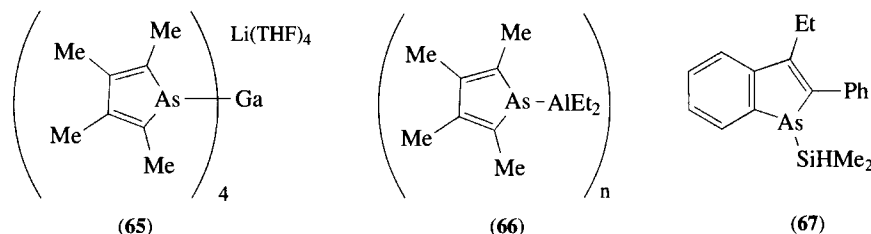


Scheme 9

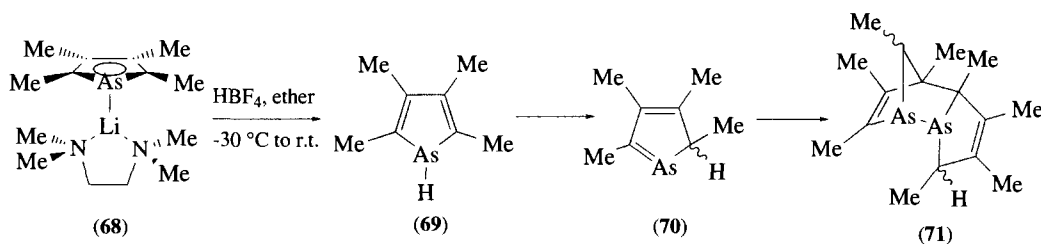


(ii) Reactions with other electrophiles

Arsole, stibole, and bismole anions react with I_2 to give 1,1'-biheterole systems (2.16.10.2) and with organometallic electrophiles to give either σ - or π -coordinated complexes (2.16.10.3). Reaction of $GaCl_3$ and Et_2AlCl with arsolyl lithium (**68**) gives the arsinogallate (**65**) and the head-to-tail dimer (**66**) in 53% and 70% yields, respectively <91CB2453>. The silylarsindole (**67**) results from reaction of the lithioarsindole anion with Me_2SiHCl <72JA2861>.



Protonation of the arsole anion (**68**) yields predominately the *endo*-bicyclic system (**71**). This reaction finds analogy in phosphorus chemistry and is believed to occur by initial formation of the 1*H*-arsole (**69**), which tautomerizes to the 2*H*-arsole (**70**) and then undergoes cycloaddition to give primarily the *endo*-product (Scheme 10). Some of the *exo*-isomer is observed by ^1H NMR. The *endo*-dimer is converted into the *exo*-dimer by heating at $95^\circ C$ for 16 h <91CB2453>.

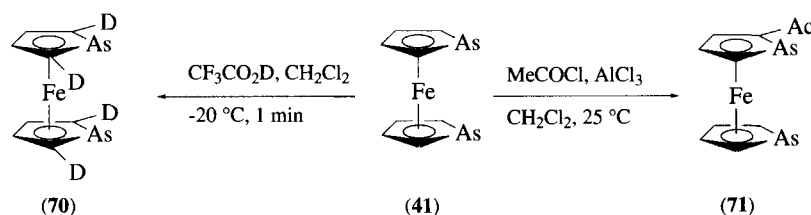


Scheme 10

2.16.5.4 Electrophilic and Nucleophilic Attack at Carbon

As mentioned above and to be described in later reactivity sections, the ring chemistry is dominated by the heteroatom. As a result, for CN-3 systems, only one example of electrophilic and none of nucleophilic attack at carbon are known. No examples of nucleophilic attack at hydrogen attached to carbon (i.e., deprotonation) are known.

The 1,1'-diarsaferrocene (**41**) undergoes rapid acid-catalyzed deuterium exchange at $-20^\circ C$ to give the fully α -deuterated derivative (**70**), while heating at $70^\circ C$ for 7 h produces only partial conversion to the β -deuterated compound. Competition experiments show the ferrocene (**41**) to be 165 times faster than ferrocene in α -exchange experiments, and four times slower than the C-4 proton in toluene in β -exchange experiments. Relative reactivity for the α/β positions is in the order of 10^6 . Acylation gives the monoacylated derivative (**71**) under mild conditions (Scheme 11) <80JOM(202)C95, 87AG(E)229>.



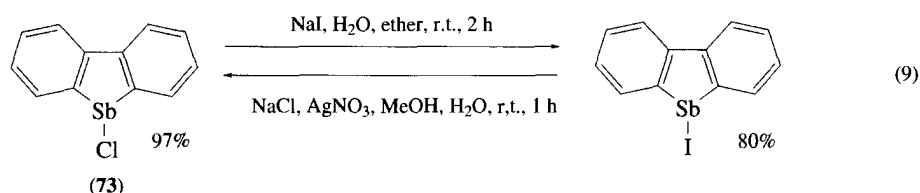
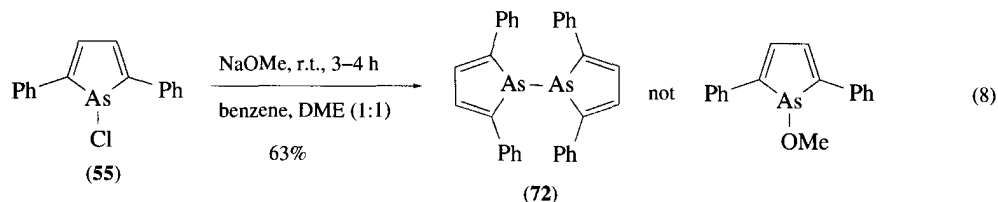
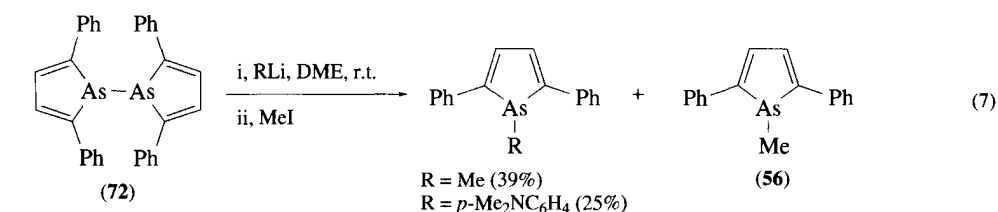
Scheme 11

2.16.5.5 Nucleophilic Attack at the Heteroatom

Many examples of nucleophilic attack at arsole arsenic are known. Most deal with heteroatom interconversion reactions, such as reaction of an *As*-chloroarsole with PhLi to give the *As*-phenylarsole (see Section 2.16.9.1), or formation of radical anions and anions by electron transfer from alkali metals to *As*-phenylarsoles (see Section 2.16.5.3.2).

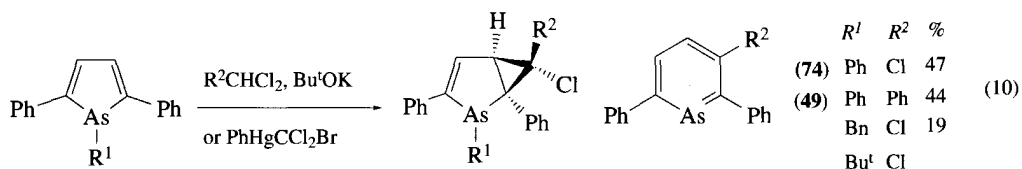
1,1'-Biarsole (72) reacts with alkyllithium reagents by nucleophilic addition to give mixtures of the 1-alkylarsole and the arsolyl anion, which is then quenched with MeI (Equation (7)). For these reactions, biarsole (72) conversion and reaction yields are low; there is a strong propensity to form the 1,1'-biarsole as shown in Equation (8) <83JOM(248)269>.

Attack by iodide on the dibenzostibole (73) occurs in good yield (Equation (9)). This reaction may be reversed by using aqueous chloride <69JOM(17)389>.



2.16.5.6 Reactions with Radicals and Electron-deficient Species

By analogy with pyrrole and phosphole chemistry, carbenes, which are generated from the dihalide and base or from PhHgCCl₂Br (R¹ = Bu^t), add to the arsole ring to give bicyclo[3.1.0] derivatives (Equation (10)), of which certain ones (R¹ = Bn, Bu^t) serve as precursors of the arsa-benzenes (49) and (74) <74TL203>.

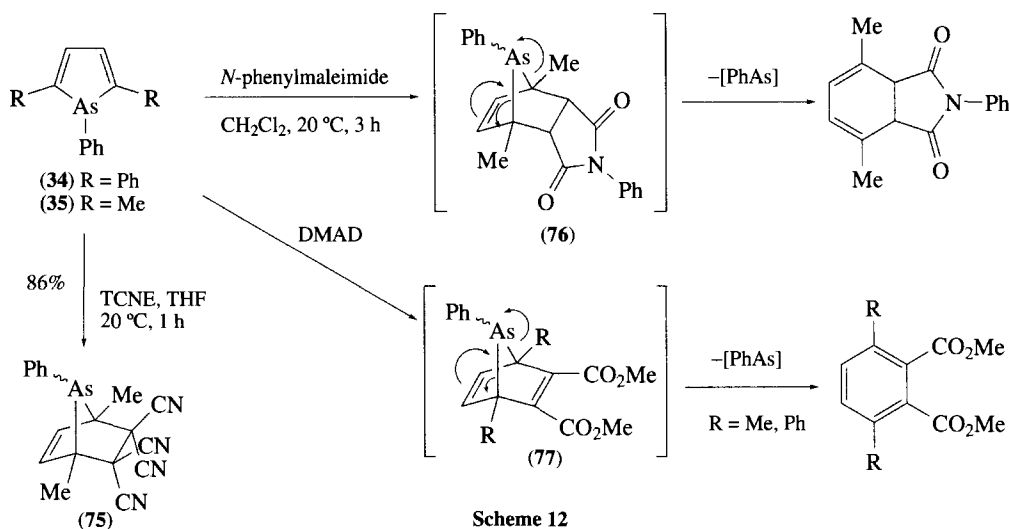


2-*t*-Butyl-1,3-benzoxarsole undergoes radical addition by the reagent Me₃SnH/AIBN to give the λ³-3-trimethylstannyl-2,3-dihydro-1,3-benzoxarsole <84PS(20)347>.

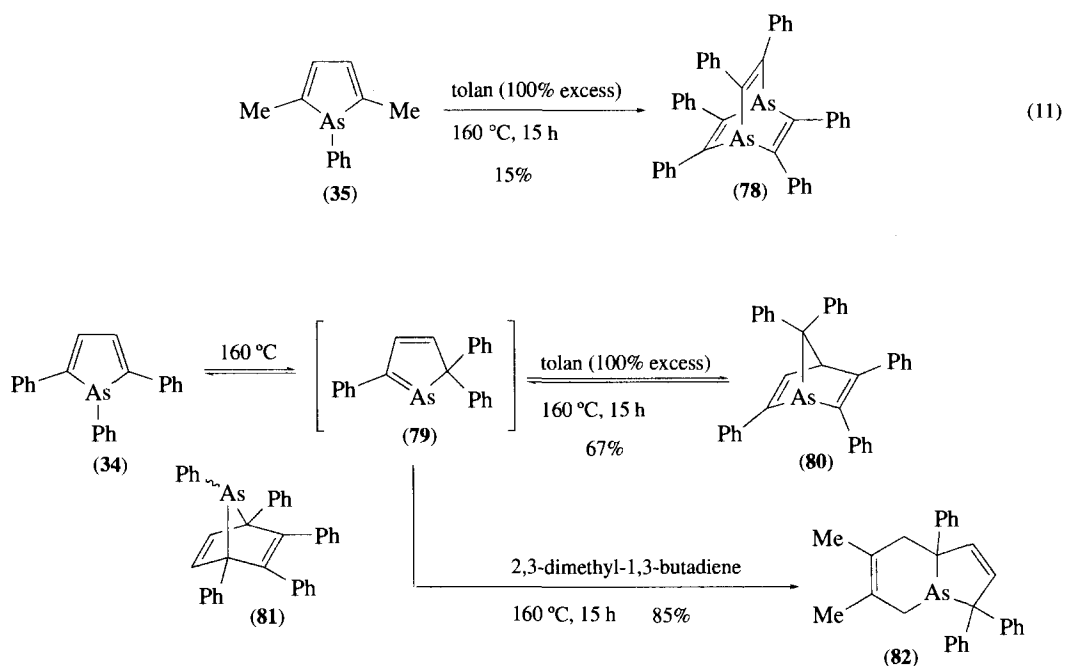
2.16.5.7 Cyclic Transition State Reactions with a Second Molecule

Arsoles exhibit rich cycloaddition chemistry. They react at room temperature with strongly electron-withdrawing alkenes to give stable Diels–Alder adducts (75), which transfer their arsenic moiety to dimethylbutadiene at elevated temperature to give diene derivatives. In some cases, the

intermediate cycloadducts (**76**) lose their bridging arsenic moiety to give dihydrobenzene derivatives (Scheme 12). Substitution plays an important role in reactivity as the arsole (**35**) participates in this chemistry at room temperature, whereas the arsole (**34**) requires elevated temperature $\langle 68\text{TL}3257, 81\text{TL}4713 \rangle$. Alkynes such as DMAD also undergo $[4 + 2]$ cycloaddition reactions with arsoles to give unstable intermediates (**77**) that, upon loss of their bridging groups, give substituted benzene derivatives (Scheme 12) $\langle 68\text{TL}3257 \rangle$. Arsole complexes react similarly with DMAD by losing an arsinidene complex from the unstable 7-arsanorbornadiene intermediate $\langle 94\text{JOM}(467)67 \rangle$.



The arsole (**35**) reacts with tolan at 160°C to give the bicyclo derivatives (**78**) (Equation (11)). The triphenylarsole (**34**) provides different products (**80**) under the same reaction conditions. It rearranges via a $[1,5]$ -shift to its $2H$ -isomer (**79**) prior to reaction with tolan. The 1-arsanorbornadiene (**80**) appears to be in equilibrium with the 7-isomer (**81**) from MS data. Further evidence for the $2H$ -arsole comes from its participation as a dieneophile in a reaction with 2,3-dimethylbutadiene to give the arsine (**82**) (Scheme 13) $\langle 81\text{TL}4713 \rangle$. $2H$ -Arsoles, obtained by tautomerism of the $1H$ -arsole, undergo Diels–Alder dimerization reactions (Scheme 10). Cycloaddition reactions of stiboles and bismoles appear to be unexplored. Benzoxarsoles $\langle 83\text{TL}5481 \rangle$ and diazarsoles $\langle 86\text{TL}2957, 92\text{HAC}151 \rangle$ participate in $[4 + 2]$ and $1,3$ -dipolar cycloaddition reactions.



2.16.6 REACTIVITY OF NONCONJUGATED RINGS

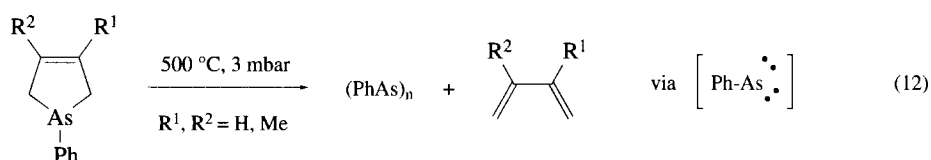
2.16.6.1 General Survey of Reactivity

The chemistry of the nonconjugated ring systems is dictated by the chemistry of the heteroatom. Typical reactions of 3°-arsines are seen with these ring systems. No examples of reactions of dihydrostiboles or -bismoles are included as these compounds do not exist.

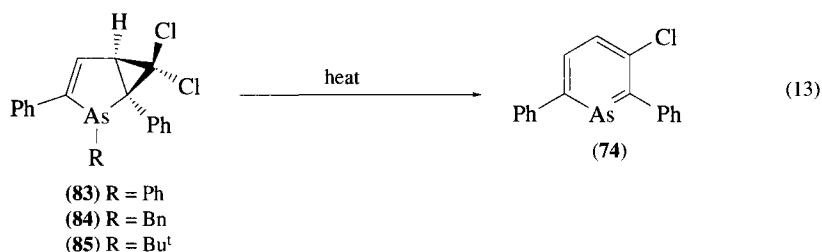
2.16.6.2 Dihydro Derivatives of CN-3

2.16.6.2.1 Unimolecular reactions

Gas-phase pyrolysis of 1-phenyl-3-arsolenes at $>500^{\circ}\text{C}$ gives, presumably by [4 + 1] cycloreversion, butadienes and cyclopolyarsines via the intermediate phenylarsinidene (Equation (12)) $\langle 93\text{JOM}(450)\text{C4} \rangle$. 3-Methyl-1-phenyl-3-arsolene rearranges to the 2-isomer when pyrolyzed at 200°C $\langle 79\text{TL}3157 \rangle$.



Arsabicyclo[3.1.0]hexenes, which are prepared by carbene addition to the parent arsole, may, depending on the arsenic substituent, thermally rearrange to arsabenzenes. The chemistry requires a group on arsenic that readily forms radical or cationic species on elimination; for example, thermolysis of the *As*-phenyl compound (**83**) at 135°C gives only a high molecular weight product and none of the arsabenzene (**74**). The *As*-benzyl derivative (**84**) displays the benzene (**74**) as a primary fragment in its mass spectrum, but, by the ^1H NMR, produces it only as a minor component on heating at $95\text{--}100^{\circ}\text{C}$ in nitromethane. The *As*-*t*-butyl derivative (**85**) readily forms the benzene (**74**) at room temperature in 24% yield within a few minutes during the carbene addition reaction (Equation (13)) $\langle 74\text{TL}203 \rangle$.

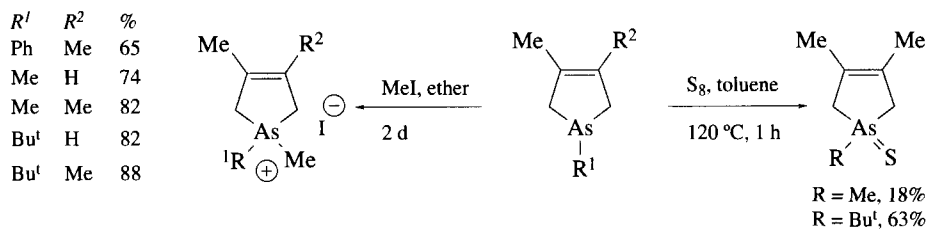


2.16.6.2.2 Attack at the heteroatom

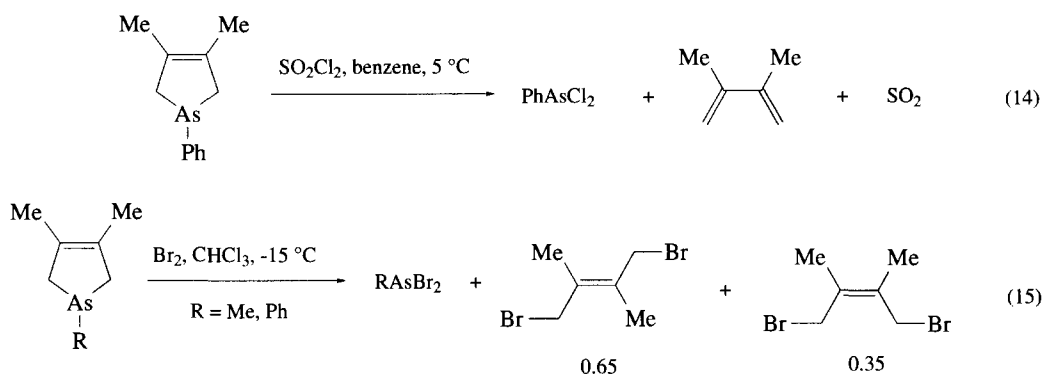
(i) Reactions with electrophiles

Reactions typical of 3° arsines are observed. Alkylation with MeI gives arsonium salts with 3-arsolenes (Scheme 14) $\langle 90\text{JOM}(396)\text{C8, } 94\text{JOM}(467)57 \rangle$, arsindolines $\langle 58\text{JCS}1719 \rangle$, and isoarsindolines $\langle 45\text{JCS}30 \rangle$. *As*-Sulfides are prepared by reaction of 1-alkyl-3-arsolenes with elemental sulfur (Scheme 14) $\langle 94\text{JOM}(467)57 \rangle$. Arsindoline *As*-oxides are produced by oxidation of the arsindoline with SeO_2 $\langle 58\text{JCS}1719 \rangle$. Many isoarsindoline derivatives, such as iodides, oxides, sulfides, dihydroxides, and hydroxynitrates, are prepared by reactions of the parent isoarsindoline $\langle 45\text{JCS}30, 47\text{JCS}662, 84\text{CHEC-I}(1)539 \rangle$.

Halogenation of 3-arsolenes with either Br_2 or SO_2Cl_2 causes ring cleavage and the formation of the alkyl- or arylarsonous dihalides (Equations (14) and (15)) $\langle 94\text{JOM}(467)57 \rangle$. Organometallic derivatives are readily formed with 3-arsolenes $\langle 90\text{JOM}(396)\text{C8, } 94\text{JOM}(467)57 \rangle$ and arsindolines $\langle 58\text{JCS}1719 \rangle$.

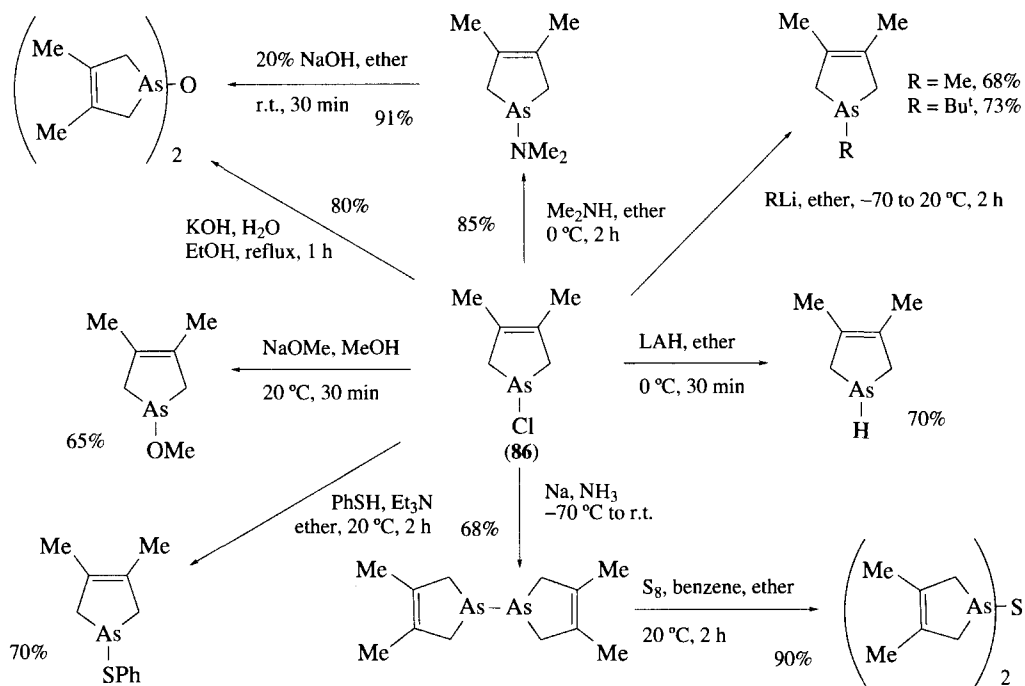


Scheme 14



(ii) Reactions with nucleophiles

The 1-chloroarsolene (**86**) reacts with many nucleophiles under mild conditions to give new arsolene derivatives (Scheme 15) <94JOM(467)57>. Similarly, 2-iodo-isoarsindoline reacts with MeMgI to give 2-methylisoarsindoline in 81% yield <47JCS662>.



Scheme 15

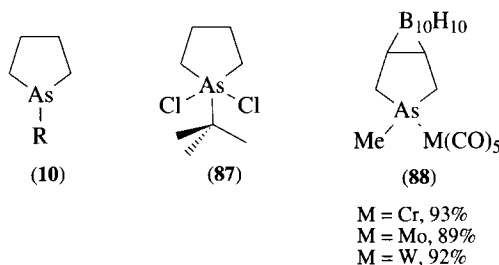
(iii) Miscellaneous reactions

All attempts to ring functionalize 1-phenylarsindoline failed. For example, dehydrogenation to its arsendole with Pd/C in ethylene glycol or with tetrachloro-*o*-benzoquinone failed. Ring halogenation with NBS and benzoyl peroxide in refluxing CCl₄ also failed. Finally, reaction with hot aqueous KMnO₄ or with SeO₂ in EtOH gave primarily the *As*-oxide and none of the desired 2- or 3-oxo derivatives <58JCS1719>.

2.16.6.3 Tetrahydro Derivatives of CN-3

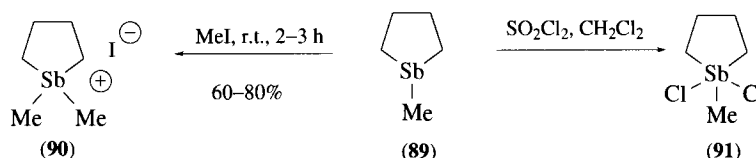
As with the fully and partially conjugated compounds, the chemistry of the fully saturated ring is dictated by the heteroatom's chemistry. Typical reactivity, for example, formation of anions (Section 2.16.10.1) and 1,1'-biheteroles (Section 2.16.10.2), is displayed by these heterocyclic compounds.

Arsolanes (**10**) are readily halogenated by Cl₂ at 0°C in CCl₄ to form the thermally stable As^V derivative (**87**) <90PS(48)157> and are alkylated by transition metals under thermal or photochemical conditions to form the σ -complexes (**88**) <83JGU73, 83ZOB(53)90>.

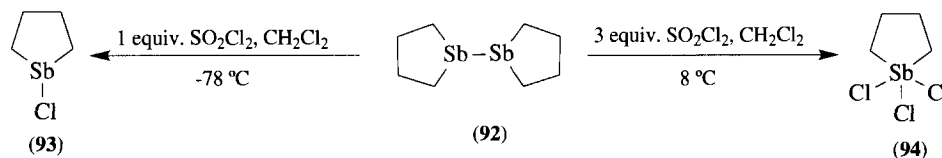


The air-sensitive 1-methylstibolane (**89**) readily forms the quaternary salt (**90**) on reaction with MeI and the Sb^V derivative (**91**) with sulfuryl chloride (Scheme 16) <76JOM(110)183>.

The 1,1'-bistibole (**92**) is converted into either the 1-chloro-(**93**) or the thermally unstable 1,1,1-trichloro-(**94**) derivative by controlling reaction stoichiometry (Scheme 17) <77JOM(136)173>.



Scheme 16



Scheme 17

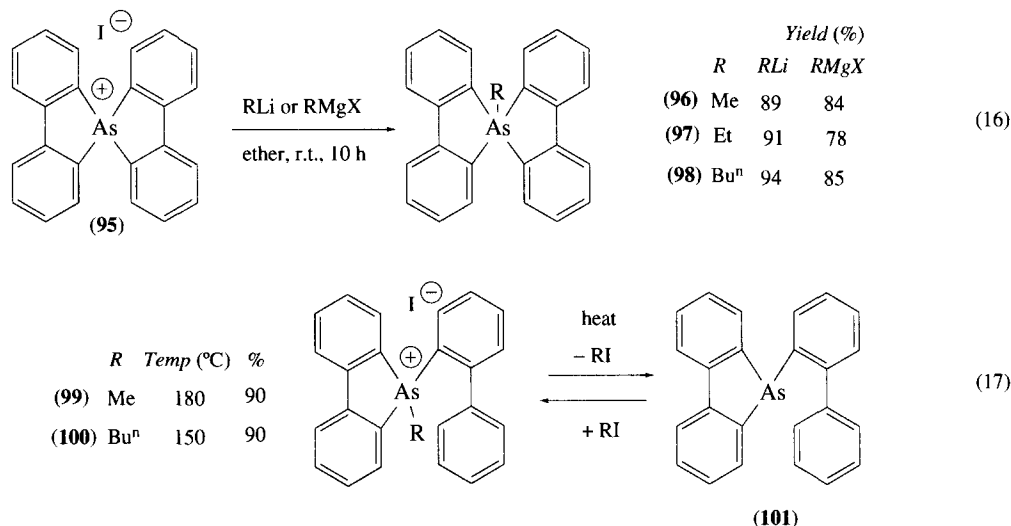
Very little is known about the chemistry of the bismolane system. Ashe *et al.* have reported on the fundamental chemistry of acyclic dibismuthines <83OM1859>.

2.16.6.4 Derivatives of CN-4

Arsonium salts (**95**) undergo nucleophilic addition by alkyl- or aryllithium or alkyl-Grignard reagents to form As^V derivatives (**96**), (**97**), and (**98**) (Equation (16)). Slightly higher yields are

obtained with the lithium reagents <64CB769, 68CB121, 71CB1761>. Sterically encumbered nucleophiles (e.g., neopentyl Grignard) give directly the As^{III} derivative (**101**). It is not well understood whether this occurs via pentacoordinate species, via hydride transfer, or via a radical process <71CB1761>.

Thermolysis of the salts (**99**), (**100**) at elevated temperatures causes reversible elimination of the alkyl group as its iodide and formation of the arsole (**101**) (Equation (17)) <68CB121>.



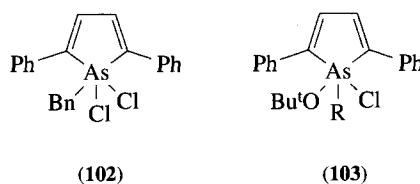
Dibenzoarsole-1-oxides are ring opened and cleaved to biphenyl derivatives on treatment with NaOH at high temperatures <71CB1761>. 2-Phenylisoarsindolidine-2-oxide is converted into the 2-sulfide by treatment of its intermediate dihydrate with H₂S <45JCS30>.

2.16.6.5 Derivatives of CN-5

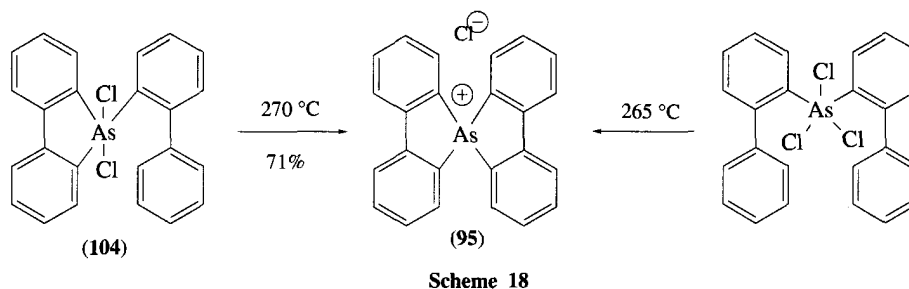
Many different reactions occur with As^V and Sb^V derivatives. In many cases, they exist as transient intermediates, and examples of these are scattered throughout this chapter. This section will focus on reactions of stable derivatives of CN-5.

2.16.6.5.1 Unimolecular reactions

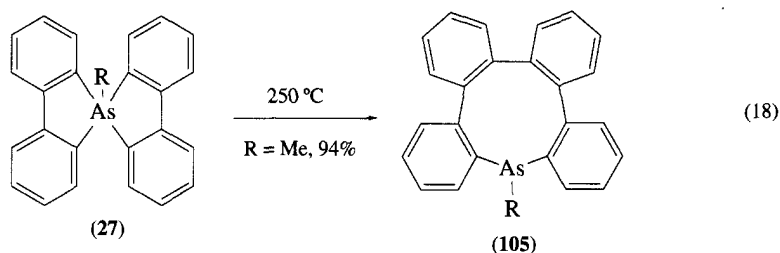
The dichloroarsole (**102**) is unstable in solution at room temperature or as a solid at elevated temperature and loses BnCl to give the *As*-chloroarsole (**55**) (Equation (3)). The 1 : 1 adducts (**103**) of arsoles and *t*-butyl hypochlorite disproportionate at room temperature (Scheme 6) <72AG(E)441a, 83JOM(248)269>. Thermolysis of the As^V dibenzoarsole (**104**) yields spirocyclic arsonium salts (Scheme 18) <64CB769>.



Products derived from the thermolysis of the As^Vspirocycle (**27**) depend on the substituent (R) directly attached to arsenic <64CB769, 68CB121, 71CB1761>. Methyl and phenyl substitution gives the nine-membered arsonin (**105**) as the principal product (Equation (18)), whereas ethyl and butyl

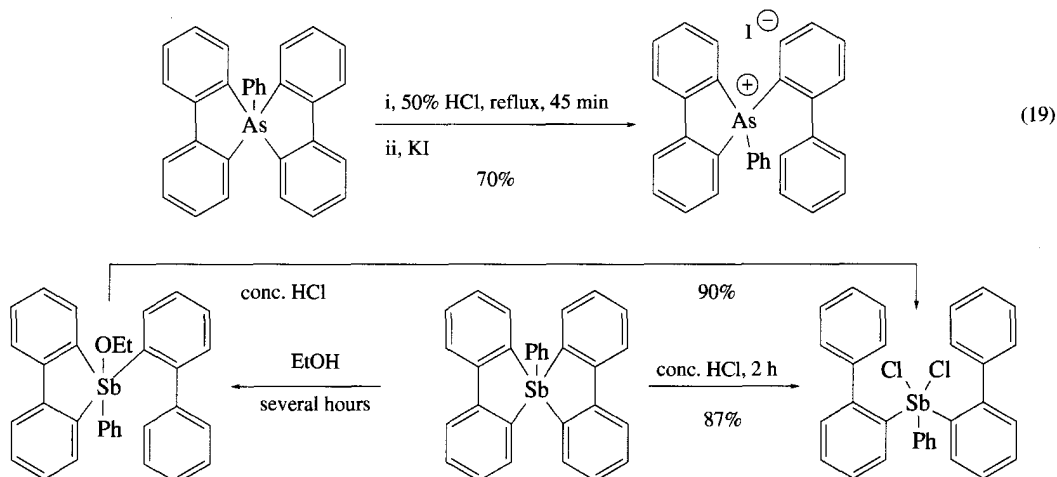


substitution gives via β -elimination the As^{III} derivative (**100**; $\text{R} = \text{Et}$ (83%), Bu^n (92%)). Dimeric products ($\text{R} = \text{H}$) and rearrangements also occur.



2.16.6.5.2 Reactions with electrophilic agents

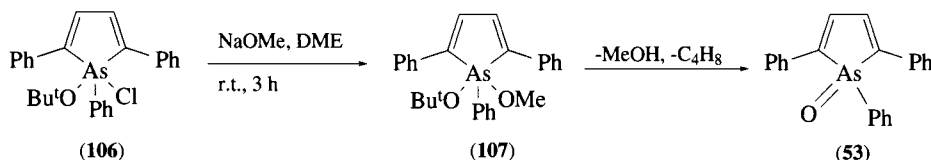
Electrophilic attack on As^{V} derivatives results in ring cleavage and subsequent formation of arsonium salts (Equation (19)) or arsoles (**101**) depending on the R substituent. Different electrophiles (i.e., ROH , MeI , Br_2 , HgCl_2) participate in this reaction <64CB769, 71CB1761>. The corresponding Sb^{V} -spirocyclic heterocycles also undergo this type of ring cleavage but give different products under acid conditions (Scheme 19) <64CB789>.



2.16.6.5.3 Reactions with nucleophilic agents

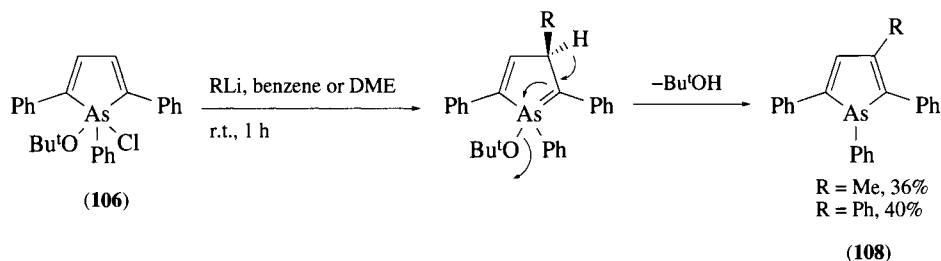
The 1 : 1 adducts formed from arsoles and *t*-butyl hypochlorite, although not isolated because of their hydrolytic and thermal instability, are trapped by nucleophiles to give new arsole derivatives. Reaction of the adduct (**106**) with NaOMe gives the intermediate (**107**) which, after loss of MeOH

and isobutylene, gives the oxide (**53**) (Scheme 20). The *As*-benzyl adduct (**102**) gives the 1-chloro-arsole (**55**) directly in 41% yield. Similar reactivity is seen with 1-substituted-1,1-dichloro-2,5-diphenylarsoles, which are unreactive towards H₂O or alcohols, but react with NaOMe. Arsole-1-oxides are produced under mild conditions in what appears to be a Michaelis–Arbusov reaction where the methoxy group is attacked by displaced chloride followed by loss of MeCl (Scheme 4).



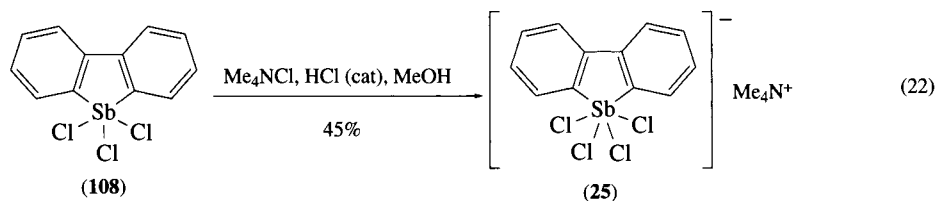
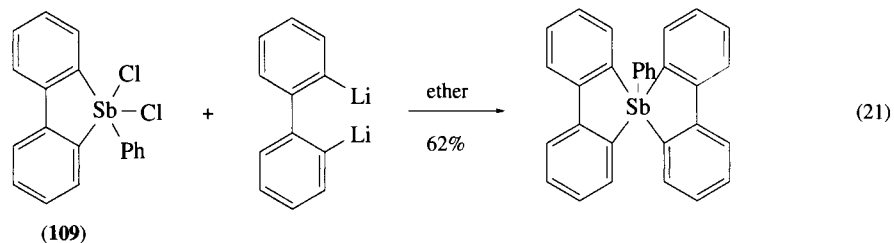
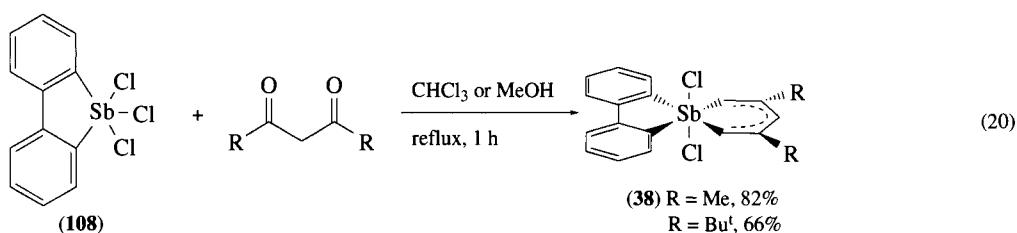
Scheme 20

The adduct (**106**) also undergoes nucleophilic substitution reactions (S_N2') with lithium reagents to form 3-substituted arsoles. Ring attack occurs since substitution at arsenic is precluded by the bulky *t*-butoxy group. Rearomatization forms the arsole (Scheme 21) <72AG(E)441a, 83JOM(248)269>.



Scheme 21

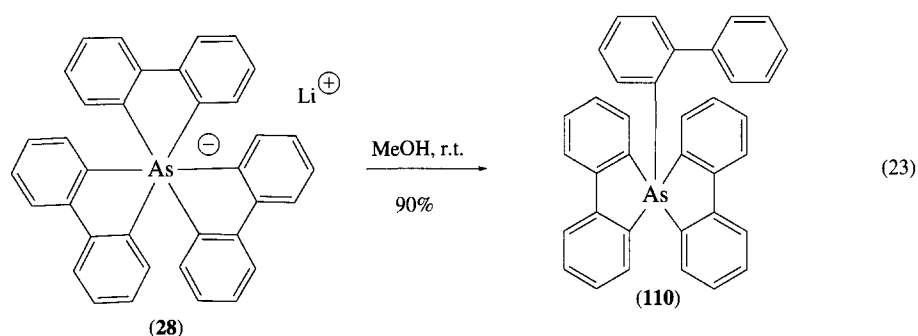
As shown in Equations (20) and (21), the pentavalent 1 : 1 dibenzostiboles-Cl₂ adducts (**108**) and (**109**) react with diaryllithium reagents and pentanedione derivatives to provide spirocyclic antimony heterocycles. This reactivity extends to nucleophilic addition of chloride which yields hexavalent derivatives of stibolanes and their dibenzo analogues (Equation (22)) <77JOM(136)173>.



Nucleophilic substitution at spirocyclic pentacoordinate arsenic heterocycles is covered in Section 2.16.9.1.

2.16.6.6 Derivatives of CN-6

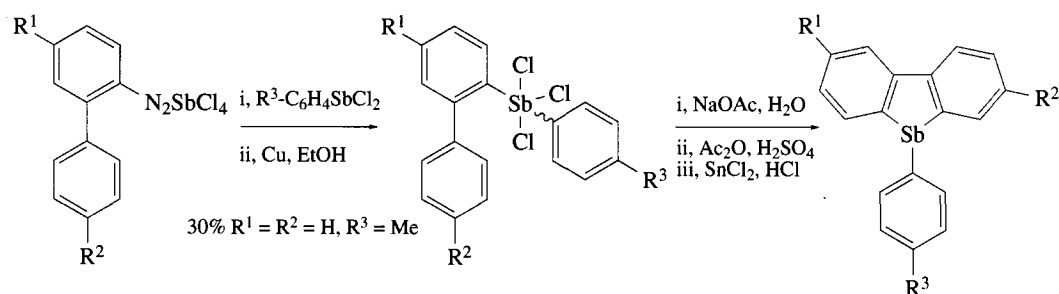
The octahedral hexacoordinate anion (**28**) is readily hydrolyzed by either acids or alcohols to the spirocycle (**110**) (Equation (23)). Chiral systems, which are prepared by fractional crystallization of the methyl Brucinium diastereomers, give optically inactive systems on ring cleavage <67LA(705)66>. Octahedral anionic species similar to (**46**) have been proposed as intermediates in nucleophilic substitution reactions at arsenic <72PS(2)129> (see Section 2.16.9.1).



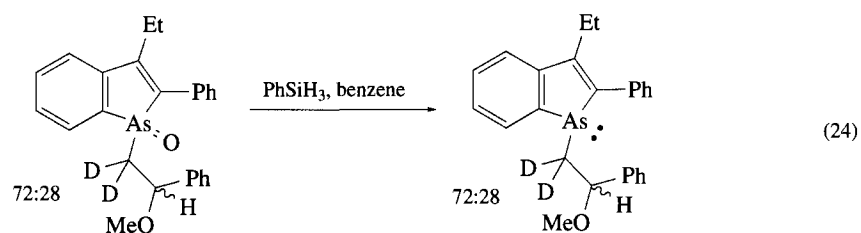
2.16.7 REACTIVITY OF SUBSTITUENTS ATTACHED TO RING HETEROATOMS

Although most chemistry in these ring systems involves reaction at the heteroatom, there are two important reactions involving substituents directly attached to the heteroatom: reduction of oxides and formation of ylides (see Section 2.16.11.1).

Several different reagents are used to reduce *As*-oxides of arsole derivatives. These include SO₃/HCl/KI (Scheme 31), SnCl₂/HCl (Scheme 22), and phenylsilane (Equation (24)). Silane reduction is stereospecific and proceeds with retention of configuration for highly pyramidal arsenic <72JA2861>.



Scheme 22



2.16.8 RING SYNTHESSES CLASSIFIED BY NUMBER OF RING ATOMS

2.16.8.1 Synthesis of Fully Conjugated Systems

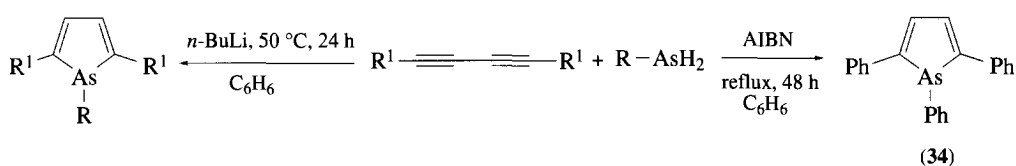
Many methods exist to prepare arsoles, stiboles, and bismoles; while older methodology produces heavily substituted derivatives, newer methods give less-substituted products. The unsubstituted (1; *E*-H) parent systems have yet to be prepared, even as transient intermediates. Preparation of aromatic anion derivatives is covered in Section 2.16.10.1.

2.16.8.1.1 Condensation reactions

(i) 1,3-Diynes

1,2,5-Trisubstituted arsoles are prepared by the base catalyzed cycloaddition of an arsine with a 1,3-butadiyne (Scheme 23). The reaction is catalytic in base and proceeds via addition of the lithium arsenide to give an intermediate vinyl anion. Proton transfer regenerates the lithium arsenide that adds to the alkyne. Both *n*-butyllithium and phenyllithium catalyze the reaction with the alkyl lithium reagent giving slightly higher yields. The reaction works with a variety of diynes and 1° aryl <68TL3257, 83JOM(249)335> and 1° alkyl <74TL203> arsines (Table 7). The arsoles are readily isolated by chromatography on alumina with benzene. Neither stiboles nor bismoles have been prepared in this manner.

Arsoles are also produced in a radical reaction between 1,4-butadiynes and 1° arsines (Scheme 23). A comparative example is shown in Table 7 (entry 9). This reaction also proceeds uncatalyzed, albeit in low yield.



Scheme 23

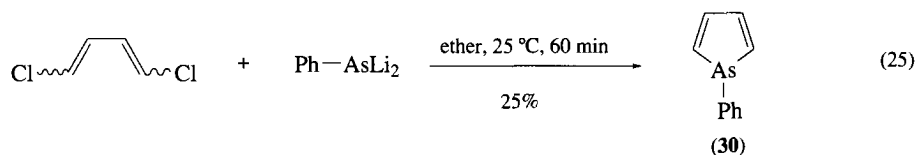
Table 7 Reaction of 1,4-diynes with primary arsines (Scheme 23).

No.	<i>R</i> ¹	<i>R</i>	Method	Yield (%)	Reference
1	Ph	Ph	<i>n</i> -BuLi	61	83JOM(249)335
2	Ph	Ph	PhLi	56	83JOM(249)335
3	<i>p</i> -MeC ₆ H ₄	Ph	<i>n</i> -BuLi	52	83JOM(249)335
4	<i>p</i> -ClC ₆ H ₄	Ph	<i>n</i> -BuLi	53	83JOM(249)335
5	β-Naphthyl	Ph	<i>n</i> -BuLi	66	83JOM(249)335
6	Me	Ph	<i>n</i> -BuLi	37	83JOM(249)335
7	Et	Ph	<i>n</i> -BuLi	18	83JOM(249)335
8	Ph	Bu ¹	<i>n</i> -BuLi	45	74TL203
9	Ph	Ph	AIBN	35	83JOM(249)335
10	Ph	Ph	reflux	28	83JOM(249)335

(ii) 1,4-Dichlorodienes

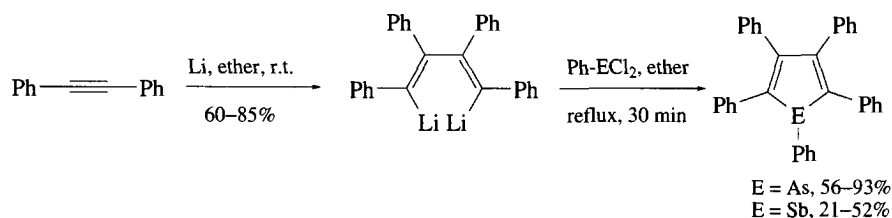
1-Phenylarsole (30) is available by condensation of dilithiophenylarsine with 1,4-dichloro-1,3-butadiene (Equation (25)). The reaction is nonstereospecific as either pure (*Z,Z*)- or (*Z,E*)-isomer

gives the arsole in identical yield. Although the yield is low, this method circumvents the use of explosive 1,4-diiodo-2-butyne <87AG(E)229>.



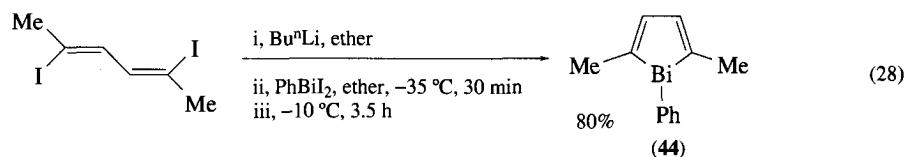
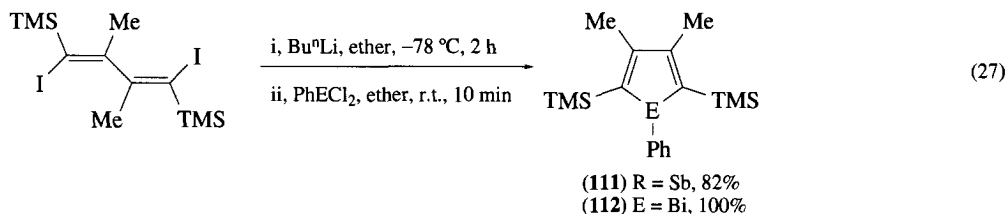
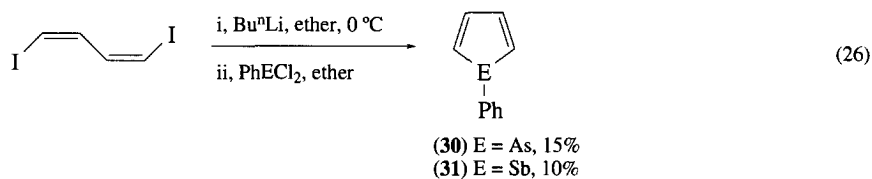
(iii) 1,4-Dilithio systems

Reaction of PhEX_2 with 1,4-dilithium reagents has allowed the preparation of aryl substituted arsoles, stiboles, and bismoles of various substitution patterns. Pentaphenylarsole and -stibole have been made by reacting tolan with lithium and trapping the intermediate dilithium species (Scheme 24). AsCl_3 gives the 1-chloroarsole that may be converted to the *As*-phenyl derivative with either PhLi (35%) or PhMgBr (35%) <60JA5099, 61JA4406>.

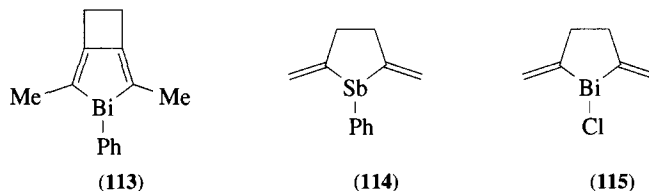


Scheme 24

Less heavily substituted dilithium reagents are prepared from di(vinyl iodides). These are available from zirconium or tin metallacycles or from 1,4-dichloro-2-butyne <85OM1478>. The lithium reagents react readily with PhAsCl_2 , PhSbCl_2 , and PhBiI_2 to give the expected heteroles either as solids or thick Kugelrohr distillable oils. The arsenic derivatives are quite stable while the bismuth systems are labile and are used without much purification. The stibole's stability depends on its substitution pattern with increasing substitution yielding greater stability. Several examples are seen in Equations (26) <85OM1478>, (27) <92OM1491>, and (28) <84OM495, 92JA372, 92OM2743>. This indirect procedure is reported to be superior to the metallacycle method (Section 2.16.8.1(iv)) for the preparation of the stibole (111) in Equation (27).



This chemistry has been extended to make the fused bismole (**113**), a case where its synthesis via the zirconacycle gave poor results <93ACS(47)326>. Similarly, trapping of PhSbCl_2 with its dilithio precursor gives the stibolane (**114**) in 20% yield whereas trapping of the bismolane (**115**; 37%) requires a tin heteroatom exchange reaction <87OM1185>.



Dibenzoarsoles, -stiboles, and -bismoles are readily prepared in this manner (Equation (29)), Table 8). The low yield of chlorodibenzoarsole (**116**) results from side-reactions between itself and the lithium reagent (Equation (30)). The racemic dinaphtharsole (**21**) has also been prepared from the dibromide ($R = \text{Me}$, 57%; $R = \text{Ph}$, 60%) <93JOM(445)71>.

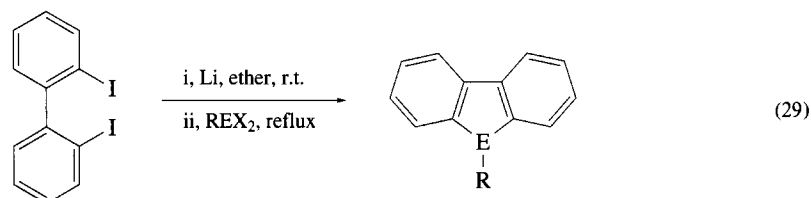
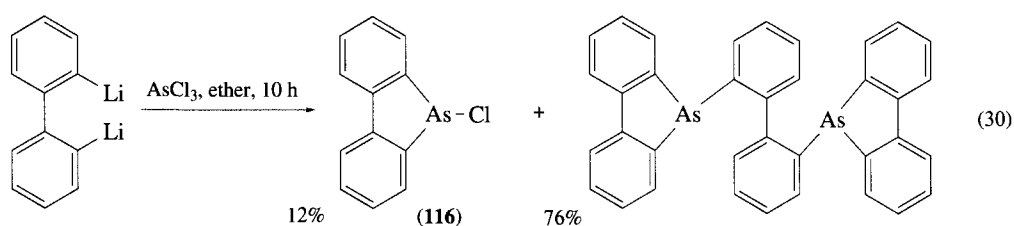


Table 8 Preparation of dibenzoheteroles (Equation (29)).

<i>E</i>	<i>R</i>	<i>X</i>	<i>Solvent</i>	<i>Time</i>	<i>Yield (%)</i>	<i>Reference</i>
As	Ph	Cl	Ether/benzene	60 min	50	63JCS4414
As	Me	I	Ether/benzene	30 min	60	59JCS3101
As	Cl	Cl	Ether	10 h ^a	12	68CB121
Sb	Ph	I	Benzene	60 min	50	59JCS3101
Bi	Ph	I	THF	90 min	72	64CB789

^a – 70°C to 25°C.

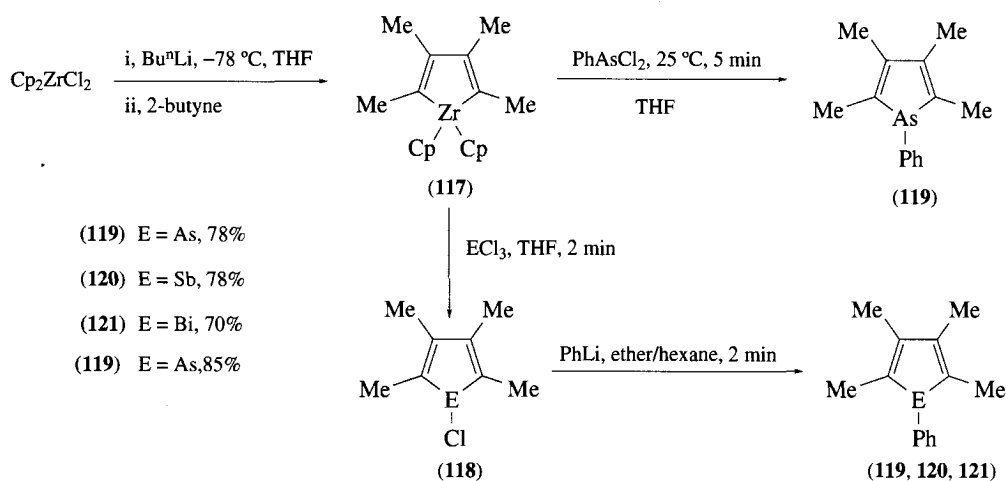


(iv) Metallacycles

The facile preparation of zirconacycles from Cp_2ZrCl_2 and alkynes <86TL2829, 91OM825> has led to the synthesis of heteroles that include low substitution and benzo-systems. Conversion of these metallacycles into arsoles, stiboles, and bismoles occurs readily under mild conditions. The methodology is sometimes referred to as the Fagan–Nugent heterole synthesis. In most reactions, the zirconacycle is stirred with PhECl_2 or ECl_3 (followed by phenyllithium) in a solvent such as ether, THF, benzene, or pentane, the solvent is removed under reduced pressure, the product is then extracted with hexane and filtered to remove any Cp_2ZrCl_2 , and finally isolated by chromatography or sublimation.

As depicted in Scheme 25, reaction of the zirconacyclopentadiene (**117**) is rapid with either PhECl_2 or ECl_3 . A one-pot procedure was used to make the stibole (**120**) and the bismole (**121**) as their

chloro-precursors were too labile to be isolated. The heteroles are isolated either by chromatography on alumina or sublimation <88JA2310, 91CB2453, 94JA1880>. In contrast to the analogous *P*-chlorophosphole, *As*-chloroarsole (**118**) is unstable and has a half-life of about 3 days. It decomposes upon attempted isolation by sublimation and reacts to form the 1,1'-biarsole during chromatography on alumina. It can be isolated by extraction and crystallization from pentane in 80% yield <91CB2453>. In general, this transmetallation reaction can be extended to main group elements that can become hypervalent, three coordinate, or coordinatively unsaturated (B, Si, P, S, Ga, Ge, Se, In, Sn) <94JA1880>.



Scheme 25

Other stibole and bismole derivatives <92OM3492> as well as stibaindoles <90AG(E)771> have been prepared in this way (Figure 2).

Fused stiboles (**123**; R = Me) have also been prepared by this chemistry (Scheme 26). However, on attempted preparation of the bismole (**113**), the tetraene (**51**) is the major product. Bismole (**113**) is only observed by ¹H NMR <93ACS(47)326>. There is a thermodynamic preference for formation of the tetraene since the bismole (**113**) is stable when prepared from the dianion and PhBiX₂. Reaction of the zirconacycle (**122**; R = TMS) with either PhSbCl₂ or PhBiCl₂ provides neither heterole (**113**; R = TMS), (**123**; R = TMS), nor the tetraene, but instead gives the zirconacycle precursor 1,6-bis(trimethylsilyl)hexa-1,5-diyne.

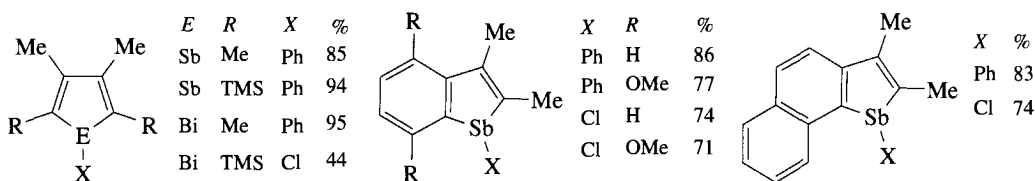
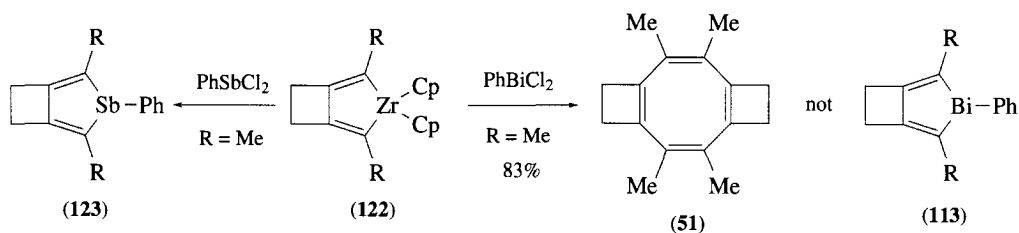


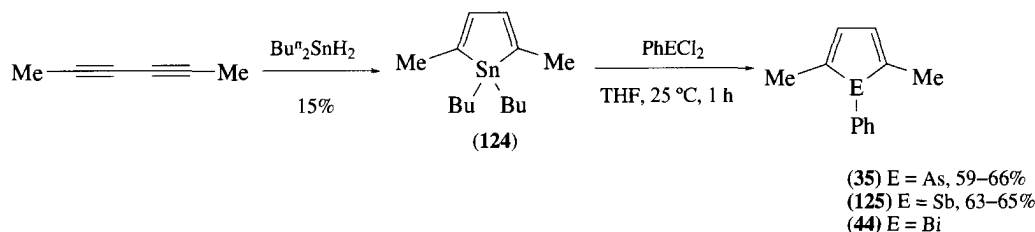
Figure 2 Stibole and bismole derivatives, and stibaindoles prepared by transmetallation.



Scheme 26

Stannoles also provide access to arsoles and stiboles by an exchange reaction. Hydrostannation of readily available 2,4-hexadiyne gives the stannole (**124**) in low yield. Reaction of PhECl₂ with

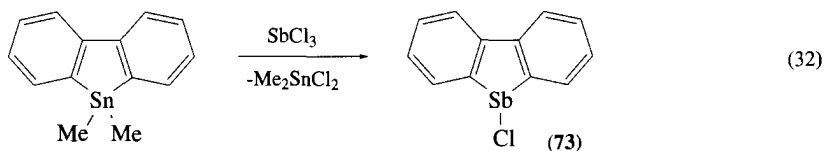
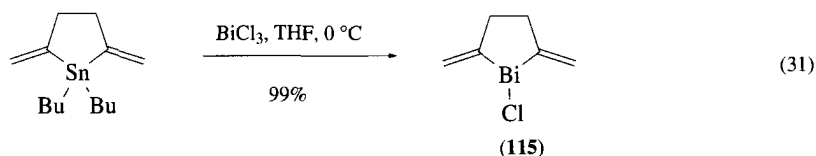
(124) under mild conditions gives the arsole (35) and stibole (125) in moderate yields as distillable light yellow oils (Scheme 27) <80JOM(202)C95, 83OM1005, 91OM2068>.



Scheme 27

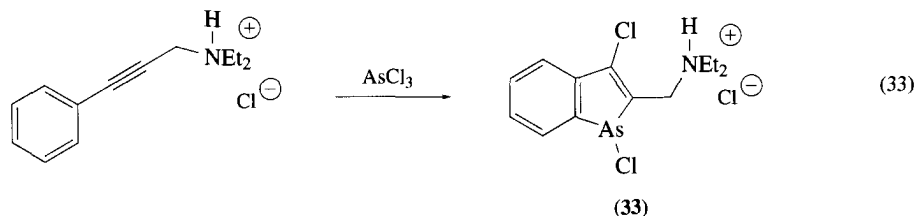
Attempted preparation of the bismole (44) by this method gives only black intractable material <84OM495>, presumably because of the known <82JA5693> acid sensitivity of vinylbismuth compounds. The labile bismole (44) is prepared in 28% yield by converting the stannole to the diiodide with I_2 , followed by lithiation, and trapping with PhBiI_2 . The bis(methylene)bismolane (115), which is a thermally unstable yellow solid, is prepared nearly quantitatively by the exchange reaction shown in Equation (31) <87OM1185>.

Preparation of dibenzostiboles by this methodology is also reported (Equation (32)) <73JOM(55)139, 77JOM(136)173>.



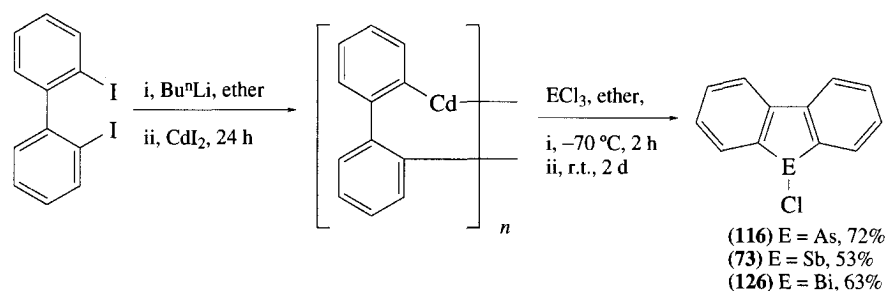
2.16.8.1.2 Miscellaneous

Arsindoles are prepared by cyclization of aryl vinyl arsinous chlorides and aryl vinyl chlorides. Atkinson's review details the generality of these approaches <84CHEC-I(1)539>. In a method of limited scope, the arsindole (33) is prepared by reaction of AsCl_3 with the acetylene salt (Equation (33)) <80JHC1341>.



Chloro dibenzoheteroles (73), (116), and (126) are prepared by transmetalation of biphenylene cadmium oligomer with ECl_3 (Scheme 28). Slightly higher yields are obtained with pure 2,2'-biphenylenecadmium. This one-pot procedure also gives the *E*-iodo derivative directly by hydrolysis of the chloro compound with NaI solution <68CB121, 69JOM(17)389>.

Higher yields of dibenzoheterole result when using this method as compared to condensing the intermediate 2,2'-dilithiobiphenyl directly with ECl_3 (Equation (30)). The organocadmium reaction

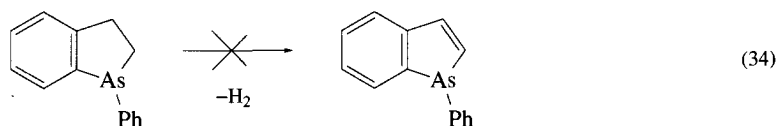


Scheme 28

stops after initial formation of the arsole (**116**), whereas the lithium reagent adds to the arsole to give the oligomer in good yield (68CB121).

A variety of dibenzostiboles have been prepared by the chemistry outlined in Scheme 22 (50JCS3109). Atkinson summarizes information on the properties of these materials (84CHEC-I(1)539). This methodology could also be used to prepare and isolate the *Sb*-oxides.

Attempts to dehydrogenate arsendolines into arsendoles with tetrachloro-*o*-benzoquinone or with Pd/C in ethylene glycol have failed (Equation (34)) (58JCS1719).

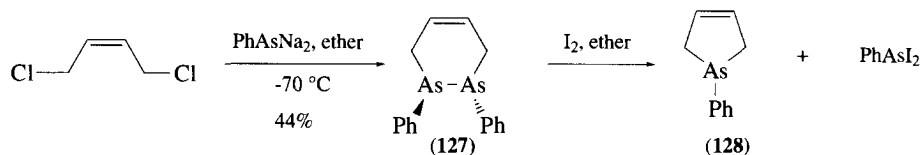


2.16.8.2 Synthesis of Dihydro Derivatives

Several of the approaches to fully conjugated systems are also applicable to the synthesis of arsenic-containing dihydro derivatives. Of these, 3-arsolenes are common, whereas the only 2-arsolene synthesis occurs by isomerization. Benzo derivatives are also known. These methods allow preparation of compounds of different arsenic functionality (e.g., *As*-Cl, *As*-alkyl, *As*-aryl), so different systems may be prepared by interconversion from one type of functionality to another. Stibolenes and bismolenes are still unknown.

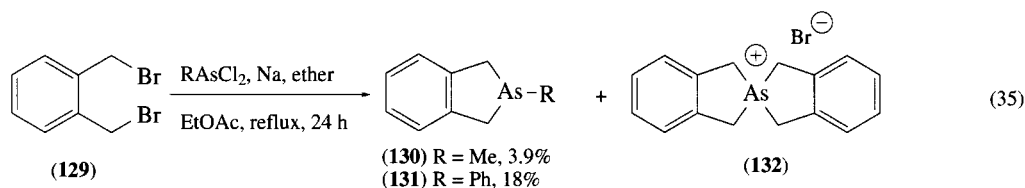
2.16.8.2.1 1,4-Dihalo systems

Direct condensation of 1,4-dichloro-2-butene with PhAsNa_2 does not give the expected arsolene (**128**), but instead gives the diarsine (**127**). Conversion into the arsolene (**128**) by disproportionation with I_2 has been observed by ^1H NMR (Scheme 29) (94JOM(467)57).

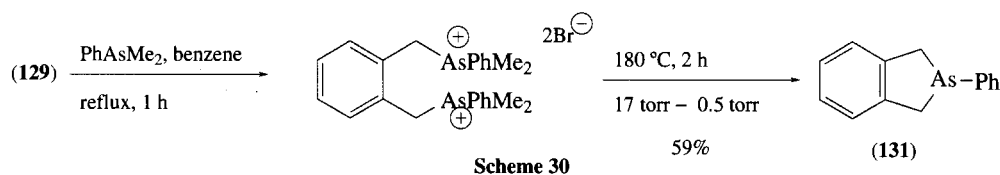


Scheme 29

Isoarsindolines (**130**) and (**131**) are prepared by two routes that both involve reaction with *o*-xylylene dibromide (**129**) (45JCS30, 47JCS662). In the first synthesis, where PhAsNa_2 reacts with the dibromide, EtOAc is crucial and acts as a reaction promoter (Equation (35)). The low yields of isoarsindolines result from secondary reaction of the product arsine with dibromide (**129**) to give the spirocyclic derivative (**132**) after loss of the *As*-substituent. Similar attempts to make the isostibaindoline have failed (45JCS30).

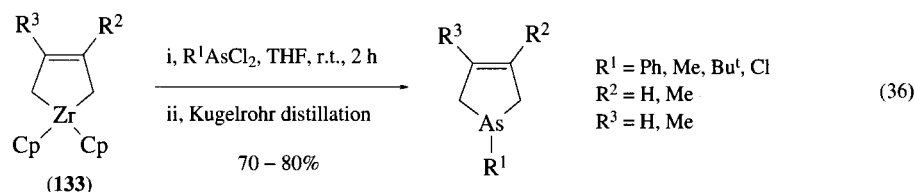


Higher yields are obtained by reaction of the dibromide (129) with PhAsMe_2 (Scheme 30). The synthesis initially involves formation of a bisarsonium salt that at elevated temperatures and reduced pressures loses MeBr and cyclizes to give the isoarsindoline (131), which distills from the reaction mixture <47JCS662>.

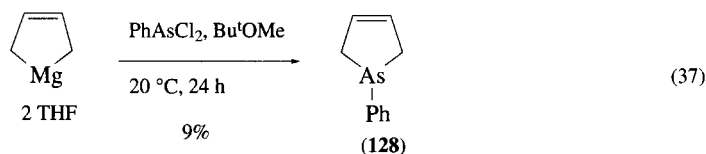


2.16.8.2.2 Metallacycles

As in the synthesis of fully conjugated systems, zirconium metallacycles play an important role in the synthesis of 3-arsolenes. Reaction of zirconocene-diene complexes (133) with alkyl- and aryl-arsinous dichlorides and with AsCl_3 provides good yields of 3-arsolene products (Equation (36)). These products, which are extremely air-sensitive liquids, are easily isolated from the reaction mixture by either chromatography (60–70% yields) or in higher yields by Kugelrohr distillation. Preparation of the *t*-butyl derivatives requires stirring for 24 h. The *As*-chloro arsolene (86; $\text{R}^2 = \text{R}^3 = \text{Me}$) is prepared at -15°C and is isolated in 32% yield <90JOM(396)C8, 94JOM(467)57>.

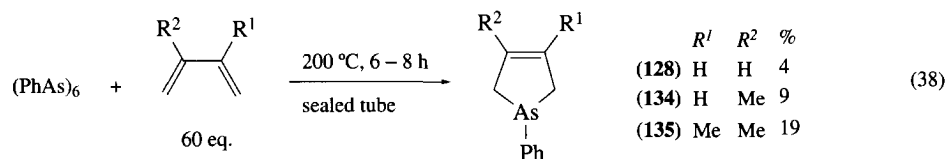


1-Phenyl-3-arsolene (128) has also been synthesized albeit in very poor yield by reaction of PhAsCl_2 with $\text{MgC}_4\text{H}_6 \cdot 2\text{THF}$ (Equation (37)). The order of addition of reagents is important to get any appreciable amount of the product, which is a very air-sensitive oil <90JOM(396)C8, 94JOM(467)57>.



2.16.8.2.3 Cycloaddition reactions

Another synthesis of 3-arsolenes finds analogy to phosphorus chemistry in the synthesis of phospholenes <75AG(E)523>. Thermolysis of cyclopolyarsines in the presence of a large excess of 2,3-disubstituted butadienes provides low yields of 3-arsolenes (Equation (38)) <79TL3157>. Potential mechanistic rationale includes dissociation of the polyarsine into phenylarsinidene followed by [4 + 1]-cycloaddition to the diene <90JOM(396)C8>, and ring-opening of the cyclopolyarsine to give a 1,6-diradical that reacts by 1,4-addition to the diene and subsequent cyclization.

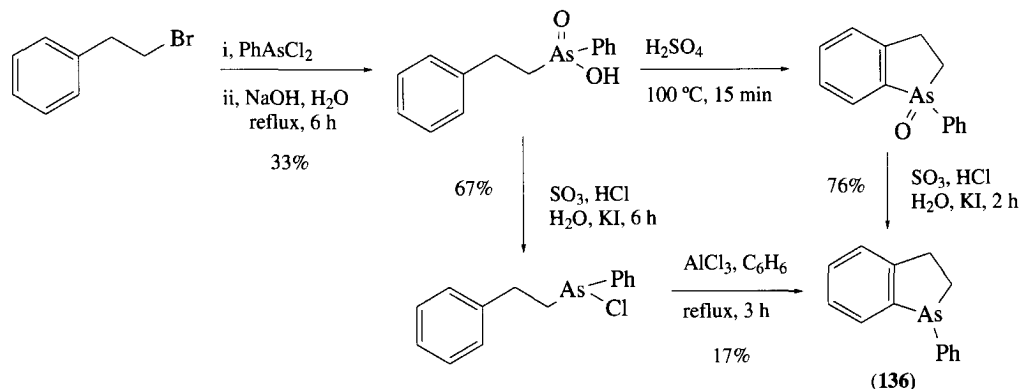


2-Arsolenes are prepared either by isomerization of a 3-arsolene at high temperatures <79TL3157> or by carbene addition to arsoles <74TL203> (Equation (10)). 3-Arsolenes are also prepared by exchange reactions between an arsolene and a different diene (Equation (44)).

Unfortunately, arsenic, antimony, or bismuth chemistry has no reaction analogous to the McCombs reaction for the synthesis of phospholenes and phospholes (see Section 2.15.10.3).

2.16.8.2.4 Miscellaneous

1-Phenylarsindoline (136) has been prepared by cyclization of arsenous chlorides or arsenic acids (Scheme 31) <58JCS1719>. This method can provide other arsindolines.

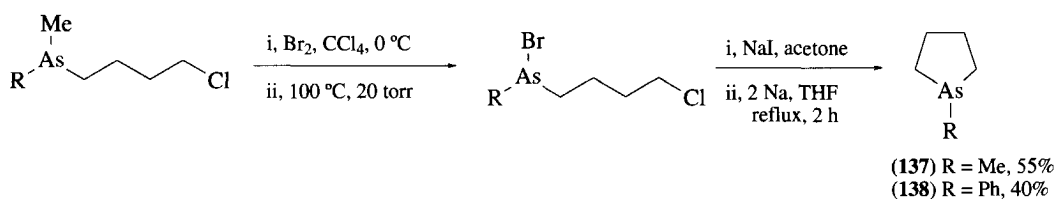


2.16.8.3 Synthesis of Tetrahydro Derivatives

Little has been published on the synthesis of fully saturated derivatives of arsoles, stiboles, or bismoles. There are now bismolane derivatives, and their syntheses will be covered in this section. The synthesis of bis(methylene)stibolanes and -bismolanes is covered in Sections 2.16.8.1.1.3(iv). Atkinson's review also covers general syntheses of arsolanes and stibolanes <84CHEC-I(1)539>. The preparation of anions of the tetrahydro derivatives is covered in Section 2.16.10.1.

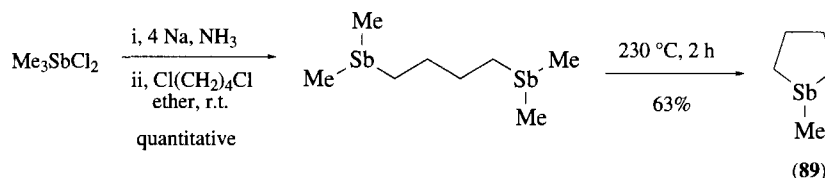
2.16.8.3.1 1,4-Dihalobutanes

1,4-Dihalobutanes are used to prepare all three saturated heteroatom systems. Arsolanes are prepared by reductive cyclization of intermediate iodoarsines (Scheme 32). Arsolane synthesis by this method is reported to be superior over that with the Grignard reagent obtained from 1,4-dibromobutane <77JCS(D)704>. These arsolanes are also prepared by condensation of disodium arsenide with 1,4-dichlorobutane <70ZAAC(377)278>.



Scheme 32

1-Methylstibolane (**89**) is prepared in a more direct manner (Scheme 33). Here, the intermediate distibine cyclizes on thermolysis to give the stibolane in very good yield. Interestingly, condensation of the dimethylstibylsodium with 1,4-dibromobutane gives the stibolane directly in 63% yield without any need for thermolysis. The pathway probably involves quaternization of the initially formed 1:1 adduct to give a cyclic stibonium salt that is subsequently demethylated by another equivalent of dimethylstibylsodium <76JOM(110)183>.

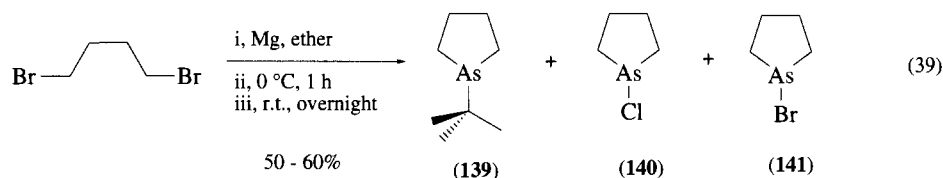


Scheme 33

2.16.8.3.2 1,4-DiGrignard reagents

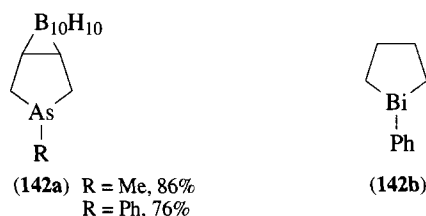
Reaction of the 1,4-diGrignard reagent, which is prepared from 1,4-dibromobutane, with Bu^tAsCl_2 provides the arsolane (**139**) as the main product, with the arsolanes (**140**) and (**141**) being formed in about 10% yield by GC analysis (Equation (39)). The arsolane (**140**) is prepared in low yield by direct reaction of the diGrignard reagent with AsCl_3 <90PS(48)157>. This is a general synthesis and has been applied to the preparation of arsolanes, stibolanes, and bismolanes.

The 3,4-*o*-carboranes (**142a**) <83JGU73, 83ZOB(53)90> and the bismolane (**142b**) (49% yield) <83OM1859> are prepared by reacting the diGrignard reagent with MeAsI_2 , PhAsCl_2 , and PhBiBr_2 .



2.16.8.4 Synthesis of Derivatives with CN-4, -5, and -6

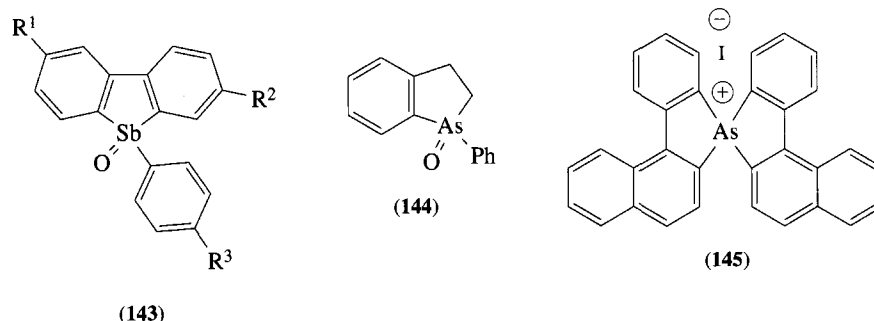
Arsenic, antimony, and bismuth display a wide variety of CN, and their syntheses, where ring formation is the primary process, are included here. Formation of oxides, methiodide salts, and halogen derivatives subsequent to ring formation are included in the appropriate reactivity sections.



2.16.8.4.1 CN-4

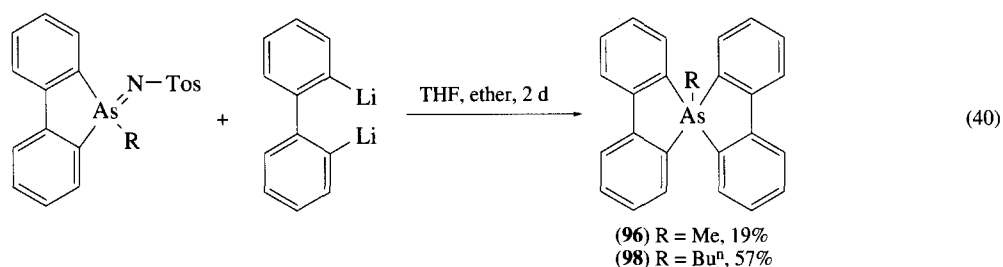
Dibenzostibole oxides (**143**) (Scheme 22) and arsinoline oxides (**144**) (Scheme 31) are synthesized directly in cyclization reactions, but are reduced without isolation during the synthesis of their lower

CN derivatives. Spirocyclic arsonium salts (**95**) are prepared by cyclization of As^{V} chlorides at elevated temperatures (Scheme 18), by acid catalyzed cyclization of arsinic acids to give (**145**) <64CB769>, or by alkylation of isoarsindolines and cyclization (**132**) (see Section 2.16.8.1.2(i)).



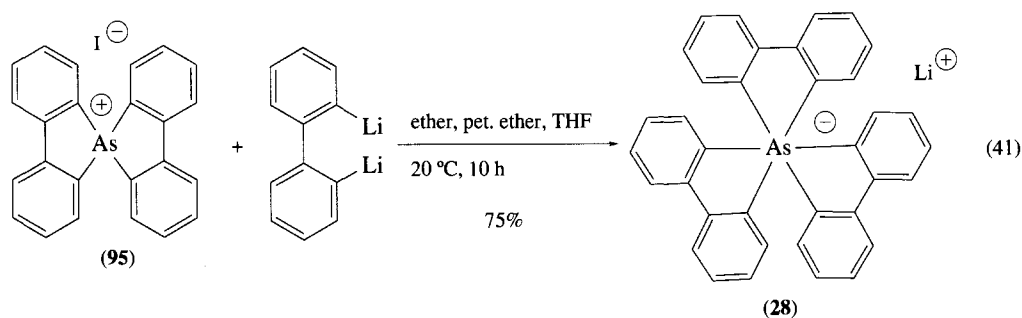
2.16.8.4.2 CN-5

Both arsenic and antimony exist in heterocyclic systems of CN-5, primarily as trigonal bipyramidal species. Condensation of an arsinimine with a dilithium reagent provides ready access to this state in poor to moderate yields (Equation (40)) <64CB769, 68CB121>. Similar antimony derivatives are prepared, but through a Sb^{V} species (**109**), which is prepared by chlorination of the dibenzostibole (Equation (21)) <69JOM(17)389>.



2.16.8.4.3 CN-6

The hexacoordinate arsenate anion (**28**) is prepared by condensing the salt (**95**) with 2,2'-dilithiobiphenyl (Equation (41)). Diastereomeric Brucine salts are prepared that can be crystallized to give optically active derivatives of the anion (**28**) <67LA(705)66>.



Reaction of antimony(V) compounds with β -diketones gives octahedral antimony derivatives of CN-6 (Equation (20)). This method also works to prepare stibolanes of CN-6. Here, the products

are isolated after mixing in CH_2Cl_2 at room temperature for 1 h $\langle 78\text{JOM}(160)435 \rangle$. 1,1,1-Tetrachloro-dibenzostibolanes and -stibolanes are prepared by reaction of the trichloro compound with Me_4NCl (Equation (22)) $\langle 77\text{JOM}(136)173 \rangle$.

2.16.8.5 Miscellaneous Syntheses

Cycloaddition chemistry plays an important role in the synthesis of the arsenic heterocycles below (Figure 3) $\langle 74\text{TL}203, 81\text{TL}4713 \rangle$. As such, their syntheses are detailed in the appropriate reactivity sections (see Sections 2.16.5.6 and 2.16.5.7).

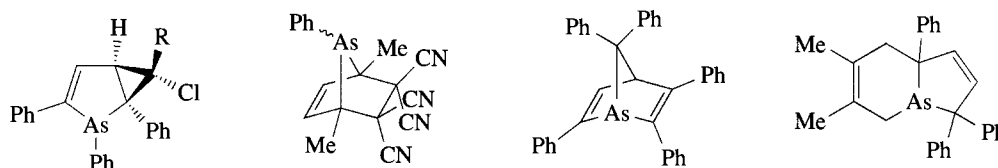


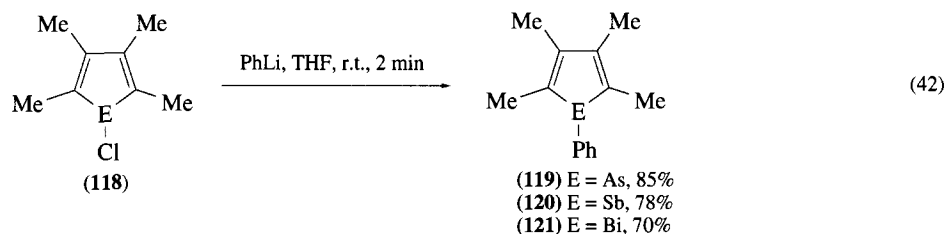
Figure 3 Arsenic heterocycles.

2.16.9 RING SYNTHESIS BY TRANSFORMATION OF ANOTHER RING

Heterocyclic synthesis can also be done by ring interconversion. These processes are treated in more detail in the reactivity sections since they involve reaction at the heteroatom. Here, they are only briefly illustrated to provide examples of the transformations.

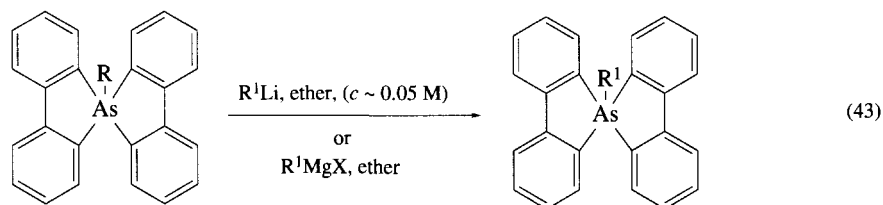
2.16.9.1 Interconversion at the Heteroatom

Nucleophilic attack by phenyllithium on the *As*-chloroheteroles gives the arsole (**119**) $\langle 91\text{CB}2453 \rangle$, the stibole (**120**), and the bismole (**121**) $\langle 94\text{JA}1880 \rangle$ in good yields (Equation (42)). Electron-release appears to increase product formation as 1,2,5-triphenylarsole (**34**) is produced from *As*-chloro-2,5-diphenylarsole (**55**) in 60% yield, under nearly identical reaction conditions in benzene $\langle 83\text{JOM}(248)269 \rangle$.



As-Substituted arsoles are converted into their *As*-chloro arsoles by chlorination and stirring in solution (Equation (3)). Higher yields (75%) are obtainable by warming the unstable As^{V} dichloro intermediate under vacuum at 70°C for 14 h $\langle 72\text{AG}(\text{E})441\text{a}, 83\text{JOM}(248)269 \rangle$. Nucleophilic attack at *As*-alkyl and -aryl arsoles by lithium reagents gives anionic species that can be trapped by electrophiles to give new *As*-derivatives $\langle 72\text{AG}(\text{E})439, 83\text{JOM}(249)335 \rangle$. This process occurs for As^{V} as well as As^{III} compounds (Equation (43)). These interconversions occur by nucleophilic addition to arsenic to form CN-6 intermediate anions (**46**) that eliminate the previously bound R-substituent. Conversion is controlled by stoichiometry, choice of attacking nucleophile, and arsenic substituent (R = alkyl,

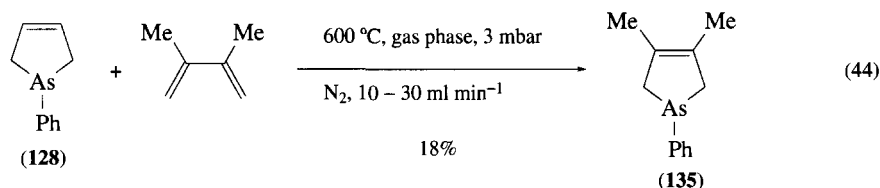
aryl, iodide) <66AG(E)969, 68CB121, 72PS(2)129>. Many other transformations of this type are covered in the reactivity sections.



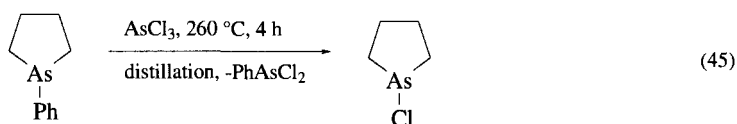
2.16.9.2 Interchange of the Heteroatom

Heterocyclic synthesis can also be done by interchange of the heteroatom. These processes are treated in more detail in the reactivity sections since they involve reaction at the heteroatom. As such, they are briefly illustrated here to provide illustrative examples.

In a gas-phase reaction, the 3-arsolene (**128**) transfers its PhAs moiety to a 77 equivalent excess of 2,3-dimethylbutadiene to give the 3-arsolene (**135**). The reactants are allowed to flow through a quartz hot-tube in an electric oven and collected in a -190°C cold trap. Isolation gives the product in low yield (Equation (44)). This transformation occurs via a presumed [4 + 1] cycloreversion to give phenylarsinidene. Other 3,4-substituted arsolenes react under similar conditions with 1,3-butadiene <93JOM(450)C4>.

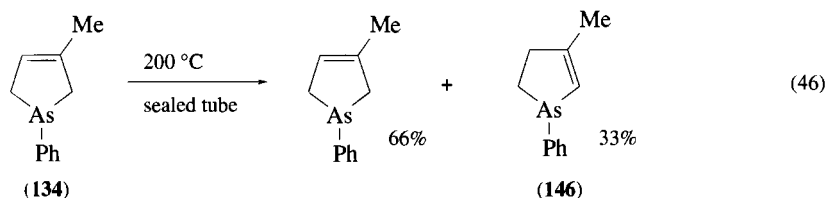


1-Phenylarsolane is converted into the 1-chloroarsolane by heating in a bomb reactor (Equation (45)) <70ZAAC(377)278>. 1,3-Benzoxazole derivatives are prepared from 1,3-benzoxaphospholes and phenylarsine <84PS(20)347>.



2.16.9.3 Isomerization

3-Methyl-1-phenyl-3-arsolene (**134**) partially isomerizes to the 2-arsolene (**146**) on thermolysis in a sealed tube reaction (Equation (46)) <79TL3157>.



2.16.10 SYNTHESIS OF PARTICULAR CLASSES OF COMPOUNDS

The following classes of compounds are covered in detail because of the great interest in their syntheses and properties. Preparation of the initial heterocyclic system, whether fully saturated or unsaturated, was covered in previous sections. The use of the heterocycles in the preparation of specific derivatives will now be discussed. Subsequent sections build upon preceding ones.

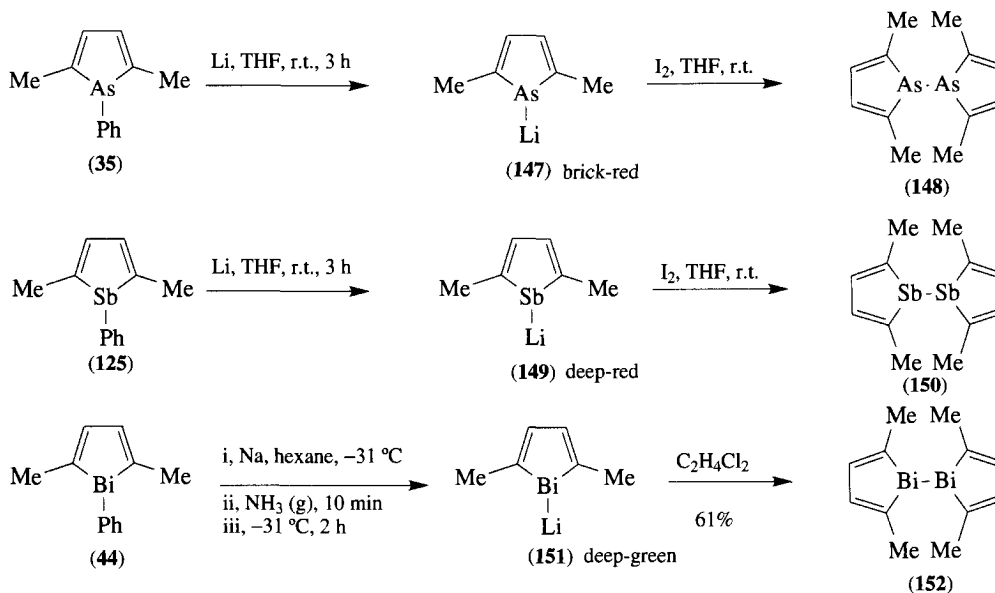
2.16.10.1 Synthesis of Anionic Intermediates

Anions of arsoles, stiboles, bismoles, stibolanes, and bismolanes are known. Although the highly colored species are reactive, they are stable when handled properly. Their lone-pair electrons are fully delocalized and thus are considered to be aromatic. They are used as intermediates in the synthesis of 1,1'-biheteroles, alkylated products, or organometallic derivatives, which may either be σ - or π -bonded.

Alkali metals are useful for cleaving aryl-heteroatom bonds for the group 15 elements (<58JOC1063, 72MI 216-01>). The different combinations of Li/THF and Li/DME, as well as Na/NH₃ (liquid) have been used for this purpose. Aryl substituents are usually cleaved from the ring, but examples of methyl and halogen cleavage are known as well. Generally, it is the exocyclic bond that is cleaved. This is the primary reaction used in the synthesis of heterocyclic anionic species.

Märkl *et al.* first prepared arsolyl anions by this technique. They determined that depending on the number of equivalents of alkali metal (lithium or sodium) used, either radical anions (1 equivalent) or anions (2 equivalents) are produced. Radical anion formation has been confirmed by ESR spectroscopy (<72AG(E)439, 83JOM(248)269, 83JOM(249)335>). The process is step-wise with formation of the persistent blue-green radical anion (36) followed by the violet anion (37). Both nucleophilic species are readily trapped by electrophiles (Scheme 7). Alkylation of the radical anion (36) is presumed to occur through an intermediate λ^5 -arsolyl radical (57) followed by elimination of phenyl radical (Scheme 8) while a normal S_N2 process occurs for the anion (37). Order of addition is important for the latter process (<83JOM(249)335>).

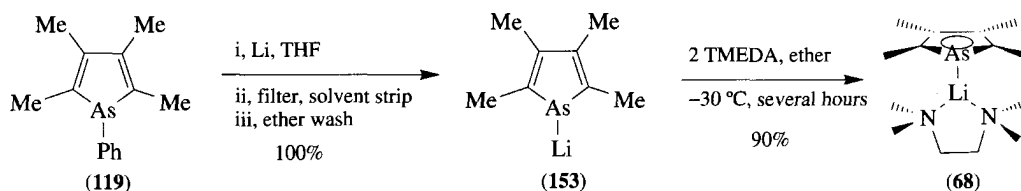
Since the anions are usually generated and then used *in situ*, direct comparison of heteroatom efficiency is difficult because different products have been produced by each heterole or the yields have not been reported. The anions (147), (149) and (151) from phenylarsole (35) (<79ICAL67, 83OM1005>), -stibole (125) (<80JOM(202)C95, 81JA207, 91OM2068>), and -bismole (44) (<84OM495, 92OM2743, 93JOM(447)197>) have all been prepared (Scheme 34). The bismolyl anion (151) has also been prepared by Li/THF cleavage. Phenyllithium, which is a byproduct of this chemistry, has been quenched by addition of 0.33 equivalents of AlCl₃, reaction with NH₃, or acidification with 1.00 equivalents of NH₄Cl.



Scheme 34

Attempts to prepare arsolyl anions by reductive cleavage of *As*-chloro-2,3,4,5-tetramethylarsole or reductive cleavage of the permethylated 1,1'-biarsole result in erratic yields of the anion (153) and are contaminated by elemental arsenic. A superior procedure calls for reduction of the *As*-phenyl derivative (Scheme 35). With this procedure, analytically pure anion (153) is prepared in near quantitative yield by extracting the byproduct PhLi with ether after the reaction has finished. Reaction of this anion with 2.0 equivalents of TMEDA allows the preparation of crystals of its TMEDA adduct (68) that are suitable for x-ray analysis. This shows the nearly planar arsole ring to be η -5 coordinated, the arsenic-carbon bonds to be intermediate between single and double bond

lengths, and the carbon–carbon bond lengths to be very similar within the ring, thus providing further evidence of arsolyl anion aromaticity (91CB2453).



Scheme 35

Heavily substituted stibole and bismole anions (92JA372, 92OM1491, 92OM3492), unsubstituted arsole (87AG(E)229) and arsendole (72JA2861) anions, bis(methylene)stibolane anions (87OM1185), and stibolane (77JOM(136)173) and bismolane (83OM1859) anions are prepared by this chemistry. The bis(methylene)bismolane anion appears to be so reactive that it forms its 1,1'-bibismole immediately under the reaction conditions. Reductive cleavage has also been accomplished with Na(Hg) in THF on 1-chloro-2,3,4,5-tetraphenylarsole and -stibole (Figure 4) (79JCS(D)814).

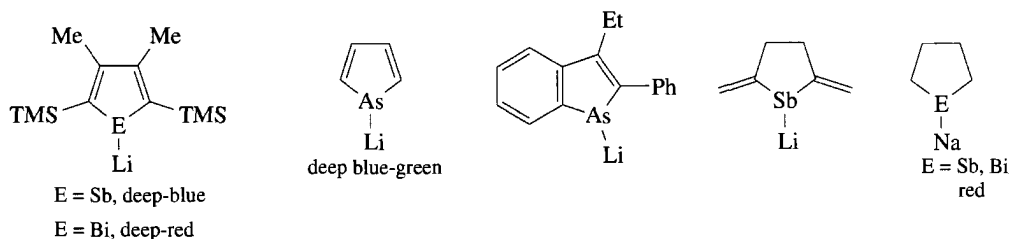
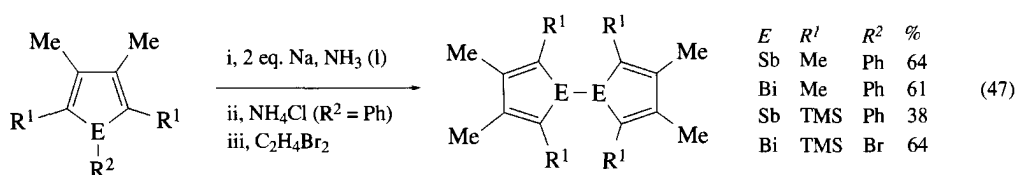


Figure 4 Various anionic structural types.

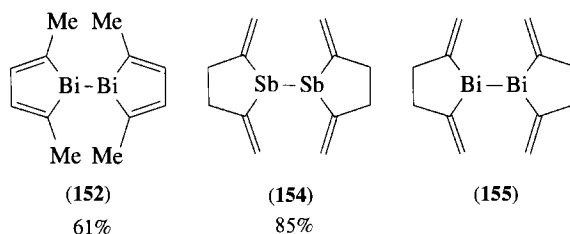
2.16.10.2 Synthesis of 1,1'-Biheterole Derivatives

Preparation of 1,1'-biheterole systems containing arsenic, antimony, and bismuth has been important in understanding thermochromic behavior in terms of structure and bonding. Their interesting solid state structures help explain why some show color transitions, while others do not (see Section 2.16.11.2). The coupled products are sometimes very air-sensitive, but can be isolated by low temperature crystallization for nonpolar solvents or by sublimation. The biarsoles, bistiboles, and bibismoles tend to be thermally stable, while the bibismolanes usually decompose at room temperature. In this section, the different methods by which these compounds may be prepared and isolated are examined.

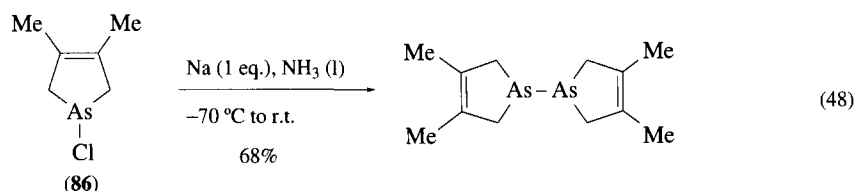
Usually, synthesis of full, partial, or unsaturated 1,1'-biheteroles is done through the anion, which may be coupled by several different methods. As in Scheme 34, anion coupling is done by oxidative dimerization with an I₂/THF solution until the brown I₂ color persists, thus giving the biarsole (148) (83OM1005) and the bistibole (150) (81JA207). 1,1'-Bibismoles are probably not prepared with this reagent because of the extreme reactivity of dibismuthines towards I₂ (82OM1408). Most practitioners couple the anions with 1,2-dichloro- or 1,2-dibromo-ethane after first neutralizing the byproduct PhLi if prepared from the E-Ph precursor. Heavily substituted bistiboles and bibismoles (Equation (47)) have been prepared in this way (92OM3492).



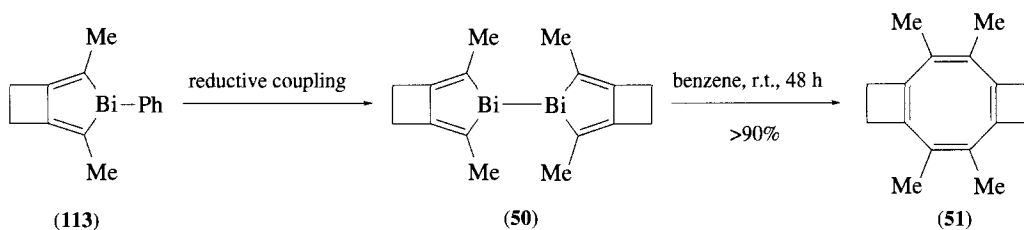
The less substituted bibismole (**152**) <84OM495, 92OM2743>, bibis(methylene)stibolane (**154**) <87OM1185>, bistibolane (**92**) <77JOM(136)173>, and bibismolane (**155**) <83OM1859> are all prepared in this way.



Coupling of the chloroarsolene (**86**) with Na/NH₃ has also been done (Equation (48)). No additional dihalide is necessary <94JOM(467)57>. The 1,1'-bibis(methylene)bismolane (**154**) is prepared in this manner <87OM1185>. Alumina chromatography of the chloroarsole (**118**) produces the permethylated 1,1'-biarsole in 40% yield <91CB2453>. Direct reaction of 2,5-diphenylarsolyl lithium with 1-chloro-2,5-diphenylarsole (**55**) gives the 1,1'-biarsole (**72**) in 79% yield <83JOM(248)269>.



Reductive coupling of the fused bismole (**113**) gives the expected 1,1'-bibismole (**50**) (Scheme 36). This material, which decomposes explosively at ~100 °C, degrades in solution over 48 h to give the tetraene (**51**) and elemental bismuth <93ACS(47)326>.



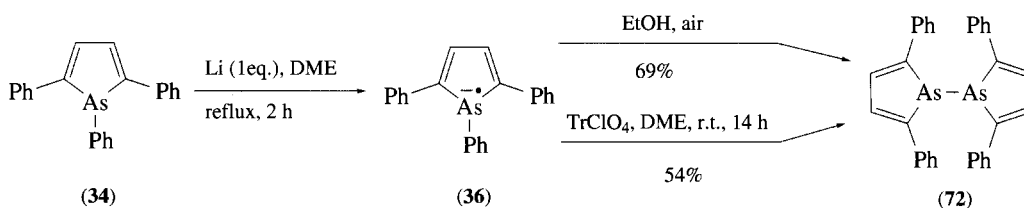
Scheme 36

There is a strong propensity to form these dimers. For example, during the reaction of 2,5-dimethylstibolyl anion with FeCl₂ to make distibaferrocene, the 1,1'-bistibole (**150**) is produced in about 11% yield. Since the ferrocene is stable to Fe²⁺ under the reaction conditions, it is likely that the bistibole is formed by oxidation of the metal <80JOM(202)C95>. It was determined in 1991, that the ferrocene can be prepared free of the bistibole if the reaction is run at -78 °C rather than at 25 °C <91OM2068>.

Many other oxidizing agents lead to the formation of 1,1'-biarsoles as primary products <83JOM(248)269>. The radical anion (**36**), prepared from the arsole (**34**), may be oxidized by air in a protic solvent or by trityl perchlorate to the biarsole (**72**) (Scheme 37). Likewise, 2,5-diphenylarsolyl lithium and 1-methoxymethyl-2,5-diphenylarsole are oxidized to the same biarsole. In an unusual reaction, 1-chloro-2,5-diphenylarsole (**55**) is converted into the biarsole (**72**) on treatment with NaOMe in DME at room temperature. In phosphorus chemistry, the *P*-chlorophosphole gives the *P*-methoxyphosphole under similar conditions.

2.16.10.3 Synthesis of Organometallic Derivatives

Arsoles, stiboles, and bismoles act as soft ligands and have found considerable application in the synthesis of both σ - and π -organometallic complexes. These compounds exhibit interesting conformational properties, by existing as eclipsed and staggered structures for which inversion

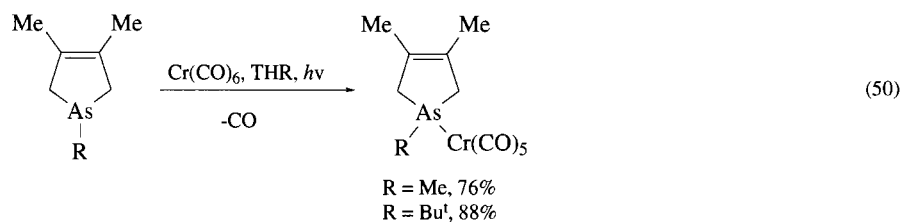
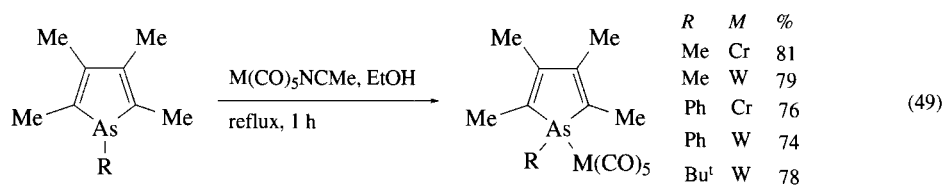


Scheme 37

barriers are known $\langle 92\text{OM}1491 \rangle$ and provide examples of secondary bonding for modeling of thermochromic behavior $\langle 92\text{OM}2743 \rangle$. Brief examples are provided. Detailed coverage is beyond the scope of this chapter, but other reviews of the subject are available $\langle 93\text{CCR}237 \rangle$.

2.16.10.3.1 σ -Complexes

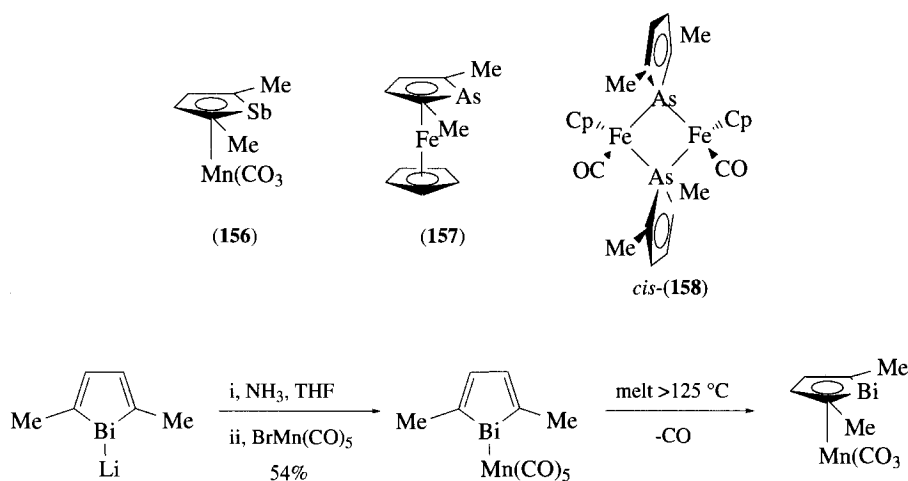
σ -Complexes of all three heteroles and 3-arsolene are known. They are generally air, light, and heat (i.e., below their melting point) stable compounds that are purified by chromatography or crystallization. Synthesis is either by thermal or photochemical means, and depends on the metal and ligands undergoing reaction. Many different arsole $\langle 61\text{JA}4406, 79\text{ICAL}67, 79\text{JCS(D)}814, 79\text{JCS(D)}1552, 83\text{JCU}73, 83\text{ZOB}(53)90 \rangle$ and 3-arsolene $\langle 90\text{JOM}(396)\text{C}8, 94\text{JOM}(467)57 \rangle$ complexes have been prepared with a variety of transition metals and ligands. Illustrative examples are shown in Equations (49) $\langle 94\text{JOM}(467)67 \rangle$ and (50) $\langle 90\text{JOM}(396)\text{C}8 \rangle$. The arsole complexes undergo Diels–Alder reactions $\langle 94\text{JOM}(467)67 \rangle$, and have been prepared as diastereomeric derivatives to study ring inversion processes $\langle 93\text{JOM}(445)71 \rangle$. The heterole complexes can be precursors to their π -complexes.



2.16.10.3.2 π -Complexes

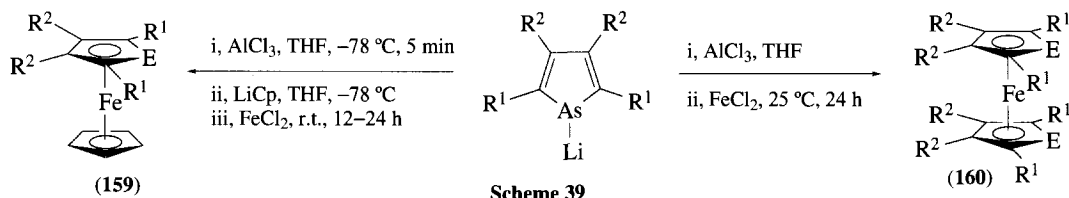
π -Complexes of all three heteroles are known. They too are air, light, and heat stable compounds that are purified by chromatography or crystallization. Several methods of making π -complexes exist. As mentioned above, σ -complexes rearrange to the π -complex (Scheme 38) on thermolysis $\langle 93\text{JOM}(447)197 \rangle$. Many π -complexes, such as $(\eta^5\text{-}2,5\text{-dimethylstibolyl})\text{Mn}(\text{CO})_3$ (**156**; 24%) $\langle 80\text{JOM}(202)\text{C}95 \rangle$, $(\eta^5\text{-}2,3,4,5\text{-tetraphenylarsolyl})\text{Mn}(\text{CO})_3$ $\langle 73\text{CC}258 \rangle$, and 2,5-dimethylarsaferrocene (**157**; 30%) $\langle 79\text{ICAL}67 \rangle$ are prepared in this manner $\langle 79\text{JCS(D)}814 \rangle$. Along with the ferrocene (**157**), a *cis/trans* mixture of the arsolyl complex (**158**) is produced in which the arsole acts as a 3-electron bridging ligand. Depending on arsole substitution and the metal, heating these complexes at 140 °C gives back the η^5 -complex. π -Arsolyl complexes of the type (**156**) are also prepared from *As*-TMS

arsoles and 1,1'-biarsoles <79JCS(D)1552>. No examples of bridging stibolyl or bismolyl derivatives are known <93JOM(447)197>.



Scheme 38

The monostibaferrocene (**159**; E = Sb, R¹ = TMS, R² = Me, 20%) and distibaferrocene (**160**; E = Sb, R¹ = TMS, R² = Me, 30%) are readily prepared from the stibolyl anion (Scheme 39) <92OM1491>. Many examples of diarsaferrocenes (E = As, R¹ = H, Me, R² = H, H) <79ICAL67, 87AG(E)229>, distibaferrocenes (E = Sb; R¹ = Me; R² = H) <80JOM(202)C95, 91OM2068>, dibis-ferrocenes (E = Bi; R¹ = TMS, Me; R² = Me, H) <92OM1491, 92OM2743>, monoarsaferrocenes (E = As, R¹ = Me, R² = H) <79ICAL67> and monobis-ferrocenes (E = Bi, R¹ = TMS, R² = Me) <92JA372, 92OM1491> exist.



Scheme 39

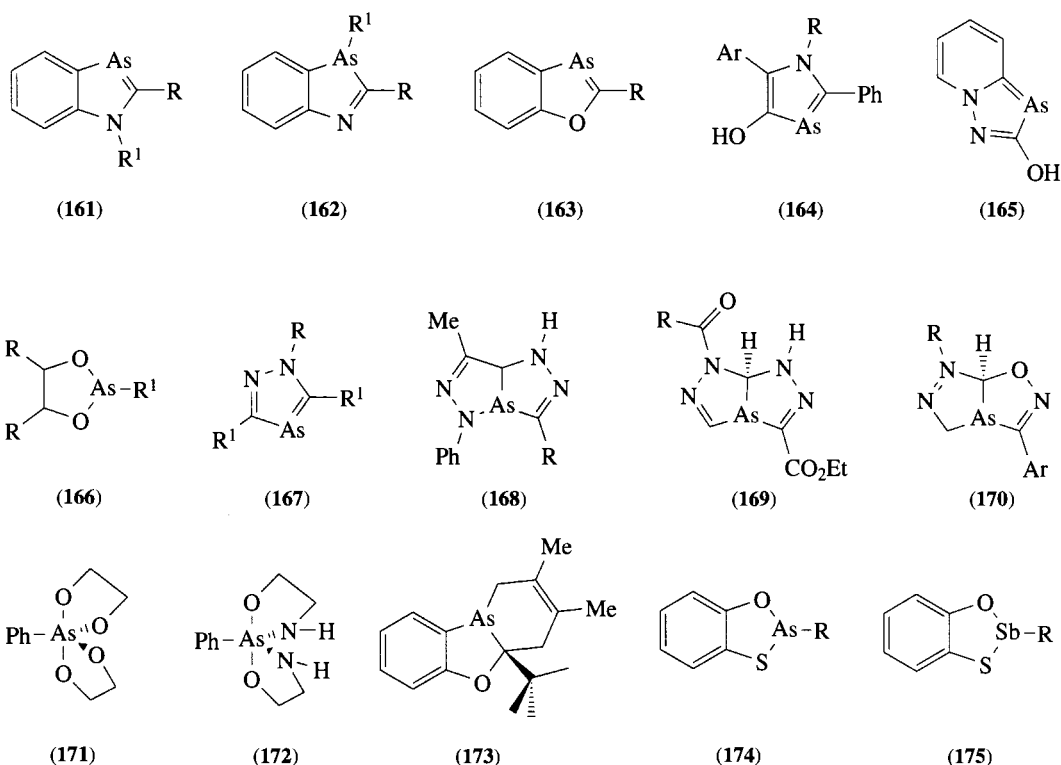
2.16.10.4 Synthesis of Heterocycles Containing Other Heteroatoms

Although not strictly covered in this chapter, there are many reports of heterocycles that contain other heteroatoms in addition to arsenic, antimony, or bismuth. This section provides a pictorial view of ring systems that were found during the literature search for this chapter: (**161**) <78JOM(154)1, 83ZAAC(501)146, 89JOM(373)49>; (**162**) <78JOM(154)1, 89JOM(364)C17, 89JOM(373)57>; (**163**) <80JOM(186)39, 84PS(20)347>; (**164**) <88TL3387>; (**165**) <88TL3387>; (**166**) <82JHC141>; (**167**) <86TL2957, 92HAC151>; (**168**) <92HAC151>; (**169**) <86TL2957>; (**170**) <86TL2957>; (**171**) <85OM708, 85OM714>; (**172**) <85OM708>; (**173**) <83TL5481>; (**174**) and (**175**) <79JHC1439, 82JHC649>.

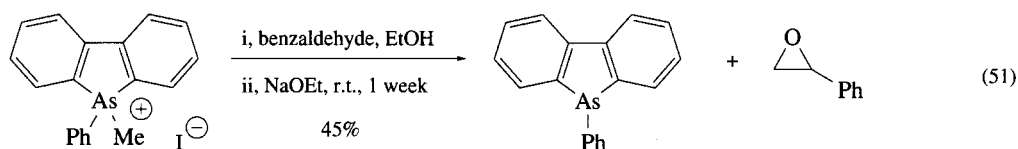
2.16.11 IMPORTANT COMPOUNDS AND APPLICATIONS

2.16.11.1 Application of Arsoles, Stiboles, and Bismoles in Catalysis

As described in Section 2.16.10.3, many different organometallic complexes have been prepared that contain arsoles, stiboles, and bismoles. The structure and properties of these materials have been well-characterized <93CCR237> (also see references in Section 2.16.11.3). Very little has been done to utilize or study these compounds in catalytic processes. When research is undertaken, it is usually to compare phosphorus and arsenic isosteres; for example, <86OM1009>. In contrast to



phosphorus ylides, which give alkenes with aldehydes via Wittig chemistry, arsenic ylides generally yield epoxides under similar conditions. In addition, phosphorus ylides yield the phosphine oxide, whereas with arsenic, the free arsine is generally recovered (Equation (51)) <76JOM(110)315>.



This chemistry has been used to induce optical activity in the product epoxides, albeit with low induction <83OM394> and has important implications for atom economy in potential asymmetric catalytic processes.

2.16.11.2 Thermochromism

Thermochromism refers to the dramatic reversible color change that occurs when a material is melted or dissolved in a solvent. Although this is a phase change rather than a temperature effect, it has been termed thermochromism. Detailed coverage is beyond the scope of this review and Ashe's review of this subject should be consulted for full details <90AOC(30)77>. Many, but not all, acyclic and cyclic distibines and dibismuthines possess this property, but diarsines and diphosphines are nonthermochromic. 1,1'-Bistiboles and 1,1'-bibismoles have played an important role in understanding thermochromic behavior. Crystal structures of thermochromic compounds "show the common structural feature that the pnictogen atoms are aligned in chains with short intramolecular bonds alternating with somewhat longer intermolecular contacts" <93OM343>. Distibine and dibismuthine isosteres exhibit either thermochromism or nonthermochromism, but synthetic examples illustrate that this is not general <92OM3492>. Molecular orbital theory has been used to understand bonding in these substances <82JA3876, 87OM1185, 93OM343>.

2.16.11.3 Chemical Vapor Decomposition

Arsenic heterocycles display interesting elimination pathways on thermolysis and in their mass spectra. As such, there has been interest <88JA2310> in the use of arsoles <91CB2453>, arsolenes <90JOM(396)C8>, and arsolanenes <90PS(48)157> in the preparation of semiconductor films by metal organic chemical vapor deposition (MOCVD).

ACKNOWLEDGMENTS

I would like to thank Lord Corporation for providing resources to help with the literature search performed in assembling this chapter and for allowing me to spend some of my regular work hours to complete it. I would also like to thank Sandra A. Caster for abstracting physical property data, assembling tabular data, and proofreading the chapter.

2.17

Siloles, Germoles, Stannoies, and Plumboies

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2.17.1 INTRODUCTION

The ring systems dealt with in this chapter were reviewed in Chapter 1.20 of the first edition of *Comprehensive Heterocyclic Chemistry* (CHEC-I) as part of a much wider brief. Since 1985, the synthesis, organic chemistry, and physicochemical properties of these metalloles has been extensively surveyed <90CRV215>, as well as their coordination chemistry <90CRV265>. Their chemistry has also been featured in *Comprehensive Organometallic Chemistry*.

Stable ring systems relevant to this chapter are shown in Figure 1. As will become evident on reading this chapter, research activity in this area decreases in the sequence siloles > germoles > stannoies > plumboies and examples of many of the species shown in Figure 1 are as yet, only known for silicon. The recommended IUPAC names for the basic ring systems (**1**) are silole, germole, stannoie, and plumbole depending upon the heteroatom. However, some research groups continue to name these compounds as metalla derivatives of the corresponding hydrocarbon namely cyclopentane, cyclopentene, cyclopentadiene, indene, or fluorene.

Simpler derivatives of silole and germole are particularly prone to undergo dimerization (Section 2.17.4.5). The parent silole (**11**) has been especially elusive and has finally been unambiguously generated by vacuum pyrolysis of either (**10**) or (**12**) and trapped in an argon matrix at 12K. This could then be interconverted with the diyl species (**13**), the 2*H*-silole (**14**) and the 3*H*-silole (**15**)

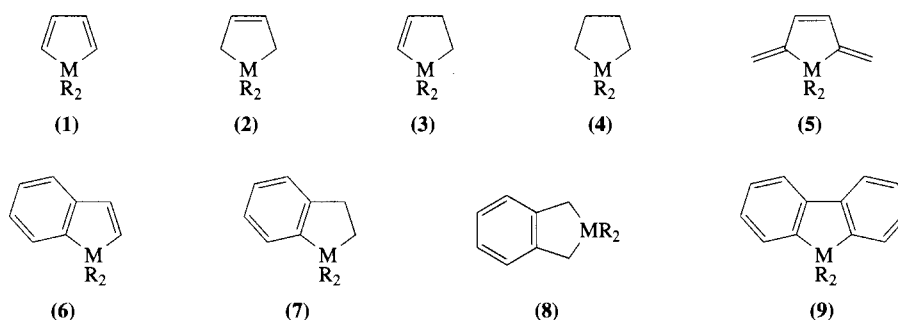
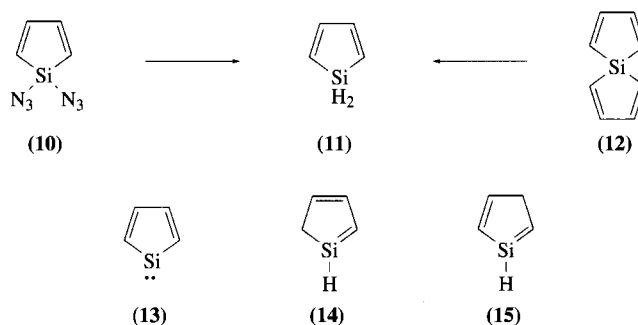


Figure 1 Stable ring systems.

<94JA320>. These observations apart there is only evidence for transitory existence of 2*H*-siloles and their benzo[*c*] homologues (cf. Section 2.17.5.3.2). No corresponding 2*H*- or 3*H*- tautomers have as yet been encountered for germoles, stannoles, or plumboles.

Although an extensive array of complexes are known in which the diene moiety of these metalloles functions as an η^4 ligand <90CRV265> compounds in which the metallocycle anion acts as an η^5 ligand have only been described since the beginning of the 1990s <93AG(E)1744, 94JA8428>.



2.17.2 THEORETICAL METHODS

In the early 1980s, molecular orbital calculations at the 3-21G/STO-2G level of sophistication for the planar (C_{2v}) silolyl anion, $C_4H_4Si^-$, indicated that it only had about 25% of the aromaticity of the cyclopentadienyl anion <83JA4972>. Subsequent calculations at the 6-31G* level indicate that the ground state geometry of the silolyl anion is pyramidal at silicon (C_{2s}) and that the planar (C_{2v}) species is the transition state for pyramidal inversion for which an energy barrier of 16.2 K cal mol⁻¹ is estimated <86JOC5028>. However, NMR studies of the 1-*t*-butyl-2,3,4,5-tetraphenylsilolyl anion indicate considerable delocalisation of the negative charge <93JA5883>. MINDO calculations have also been reported for the C_4H_4M silylene, germylene, and stannylene species <91MI 217-01>. Extensive *ab initio* calculations have been employed to aid in the assignment of the UV and IR spectra of the species (11), (13), (14), and (15) <94JA320>.

2.17.3 EXPERIMENTAL STRUCTURAL METHODS

A full range of spectral data is routinely reported for each new compound isolated. However, this normally shows only the expected variations from data published which is already conveniently tabulated and discussed elsewhere <90CRV215>. Apart from the obligatory ¹H and ¹³C NMR data, corresponding information on the heteroatoms tends to be restricted to ²⁹Si, though ¹¹⁹Sn <92CB643, 92CB1597, 93CB1361, 94ICA161> and ²⁰⁷Pb <90JOM(399)1> shifts have been recorded for some fully substituted metalloles. Although of less general diagnostic applicability, the assignment of their vibrational spectra played a major role in characterising (11), (13), (14), and (15) <94JA320>. The far-infrared spectra of 2,5-dihydrosilole <84JCP(80)5475> and 2,3-dihydrosilole <88JCP(92)4056> have been reported and confirm the planarity of the ring system in both cases.

The structures of several metalloles have been established by x-ray crystallography and these are conveniently summarized in Figure 2. An unexpected feature is the folded conformation of the germole ring reported for 1,1-dimethyl-2,3,4,5-tetraphenylgermole <93JOM(453)33>.

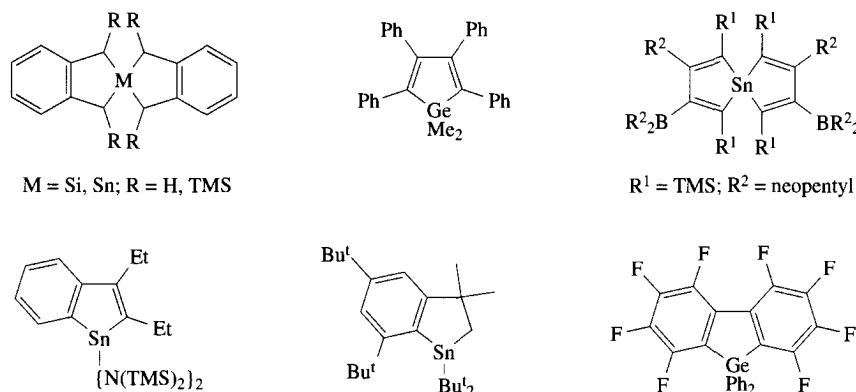


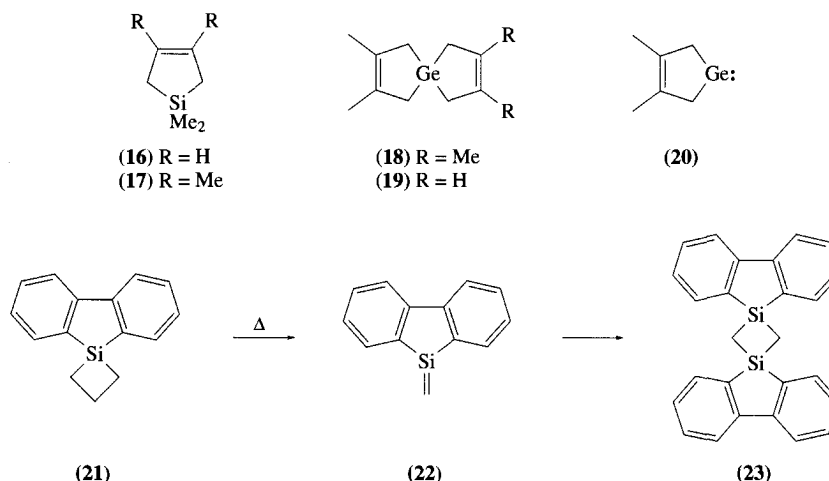
Figure 2 Structure of metalloles established by x-ray crystallography <86JOM(302)343, 90JOM(381)173, 92CB643, 92JCS(4)775, 93JOM(453)33, 94BSF715>.

2.17.4 REACTIVITY

2.17.4.1 Thermal Reactions

The synthesis of 2,5-dihydrosiloles or -germole by addition of a silylene or germylene to a 1,3-diene is in principle a reversible process. Thus at 600–650°C the dihydrosilole (**16**) dissociates to butadiene and dimethylsilylene. The latter species can be intercepted as the dihydrosilole (**17**) if the pyrolysis is conducted in the presence of 2,3-dimethylbutadiene <88OM2076>. Analogous behaviour has been observed with 2,5-dihydrogermole <91POL1221>. In the case of the spirogermole (**18**), the dissociation to two moles of diene and germanium occurs in a stepwise fashion and the intermediary germylene (**20**) has been trapped in an argon matrix at 12K, as well as being intercepted with butadiene as the adduct (**19**) <89IZV1428>.

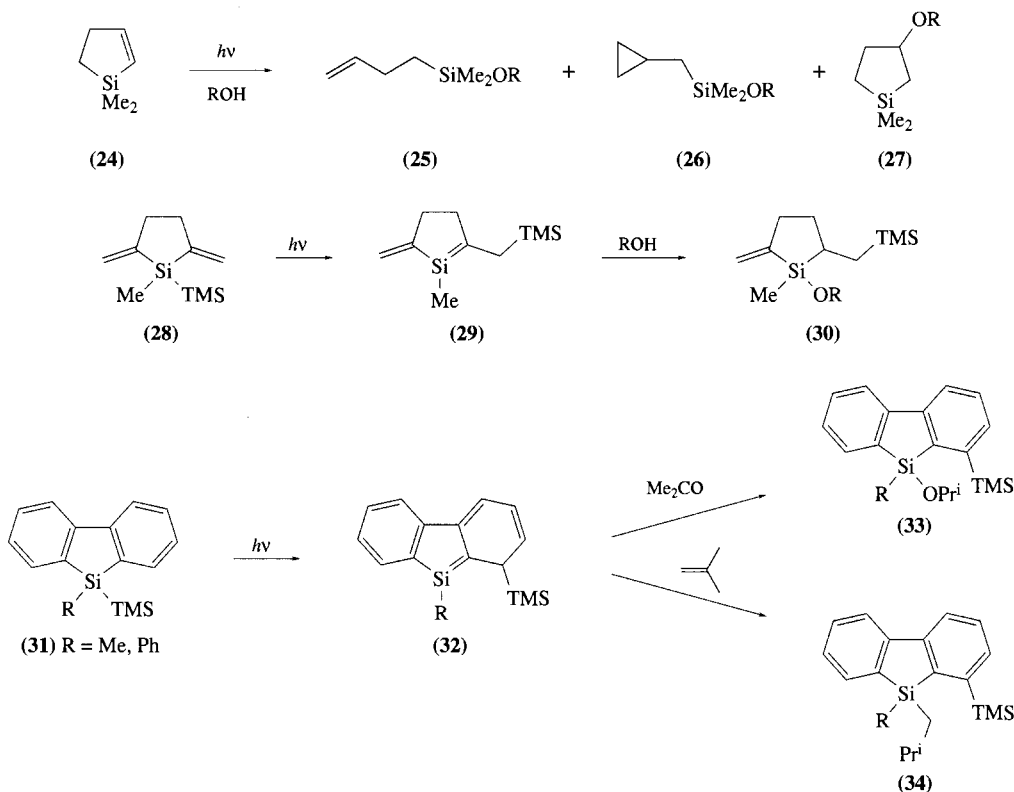
The pyrolysis of the dibenzosilole (**21**) at 650°C generates ethene and the unstable silene (**22**) which dimerises to (**23**) <87BCJ2263>.



2.17.4.2 Photochemical Reactions

Photolysis of solutions of the 2,3-dihydrosilole (**24**) in alcohols provides a mixture of (**25**), (**26**), and (**27**) <92JOC3107>. Irradiation of (**28**) in various alcohols apparently proceeds via a 1,3-silyl shift

to the silene (**29**) which then undergoes addition of the alcohol forming a mixture of the *cis* and *trans* adducts (**30**) <91JA3986>. An analogous 1,3-silyl shift appears to be involved in the photochemical transformations of the dibenzosilole (**31**). The putative intermediate (**32**) provides a ready explanation for the formation of (**33**) by photolysis in acetone, and (**34**) in the presence of isobutene <84JOM(264)79>.

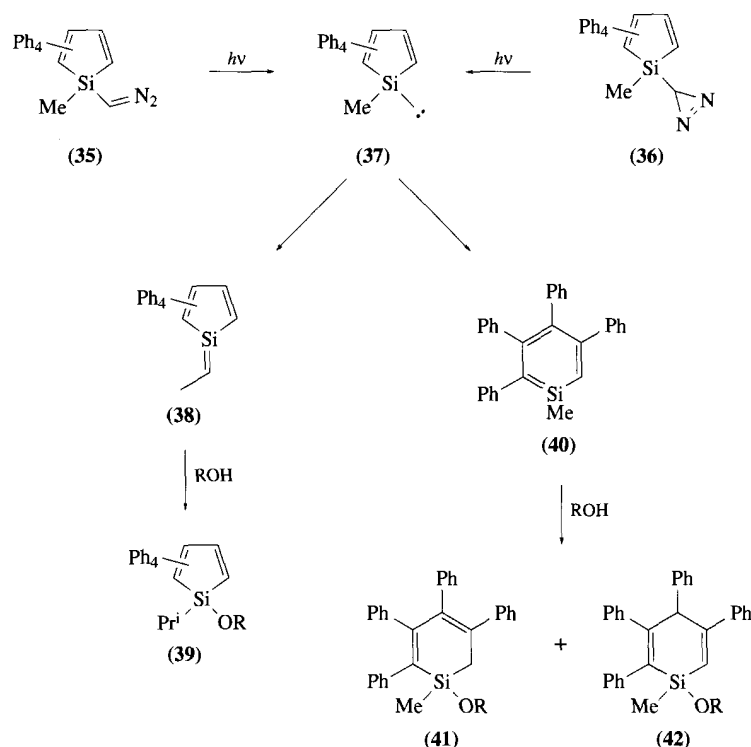


An interesting collection of reactive intermediates are involved in the decomposition of the diazo compound (**35**) or the diaziridine (**36**). The resulting carbene (**37**) in part rearranges to the silene (**38**) which is trapped as the silole (**39**), and in part rearranges to the silabenzene (**40**) which gives rise to the adducts (**41**) and (**42**) <83TL4245, 85OM584>.

2.17.4.3 Reactions with Organolithium Reagents

In the majority of cases the initial site of attack of organolithium reagents on these metalloles (**43**) is the heteroatom leading to the anion (**44**) <85TL1315, 89OM1799>. Subsequent dissociation of (**44**) to (**45**) results in the overall replacement of one of the original heteroatom substituents. From the examples quoted, the ease of replacement is hydrogen > phenyl > alkyl. In the case of (**43**)/(**45**) an accompanying reaction is deprotonation of a C-methyl group leading to the anion (**46**). Protonation of this yields the *trans* dienoid (**47**), while reaction with other electrophiles occurs at the exocyclic carbon forming, for example, (**48**). A somewhat different behaviour has been observed with the silole (**49**) <83JOM(250)109>. Here the intermediary anion (**50**) not only eliminates trimethylsilyllithium giving the silole (**51**), but also undergoes competing silyl migration to (**52**) resulting in the formation of the dihydrosilole (**53**). No comparable chemistry has been reported for stannoles though treatment of 1,1-dibutyl-benzo[*b*]stannole with butyllithium generates the deep violet ate-complex (**54**) <92AG(E)1339>.

Much fruitless effort has been expended on attempts to generate metalloyl anions, $\text{C}_4\text{H}_4\text{MR}^-$, analogous to the cyclopentadienyl anion. So far success has only been achieved in the germole series. The anion (**55**) has been obtained by reaction of 1-phenyl-2,3,4,5-tetramethylgermole with butyllithium at -70°C . A ^{13}C NMR study suggests that the negative charge is strongly localised on the germanium atom which is the site of electrophilic silylation and stannylation <90OM3001>. The anion (**56**) has been obtained in similar fashion and reacted with $[(\eta^5\text{-Me}_5\text{C}_5)\text{Ru}(\mu\text{-Cl})_4]$ to give



the ruthenocene analogue (**57**) $\langle 93\text{AG(E)1744} \rangle$. It is notable that the corresponding silole analogue of (**56**) could not be generated in the same fashion although the silole counterpart of (**57**) was eventually prepared by a roundabout route $\langle 94\text{JA8428} \rangle$.

Silicon is also the preferred site of attack by organolithium reagents on 2,5-dihydrosiloles (e.g. (**58**)) and leads to formation of ring opened polymers (**59**) $\langle 87\text{JOC4489} \rangle$. However, if the silicon substituents are sufficiently bulky as in (**60**) then deprotonation to the anion (**61**) predominates. Subsequent reaction of (**61**) with electrophiles (E) can occur either at position two (**62**) or at position four (**63**). In the case of protonation the 2,5-dihydrosilole (**62**; E = H) is virtually the sole product, but with larger electrophiles rather more of (**63**) than of (**62**) is formed.

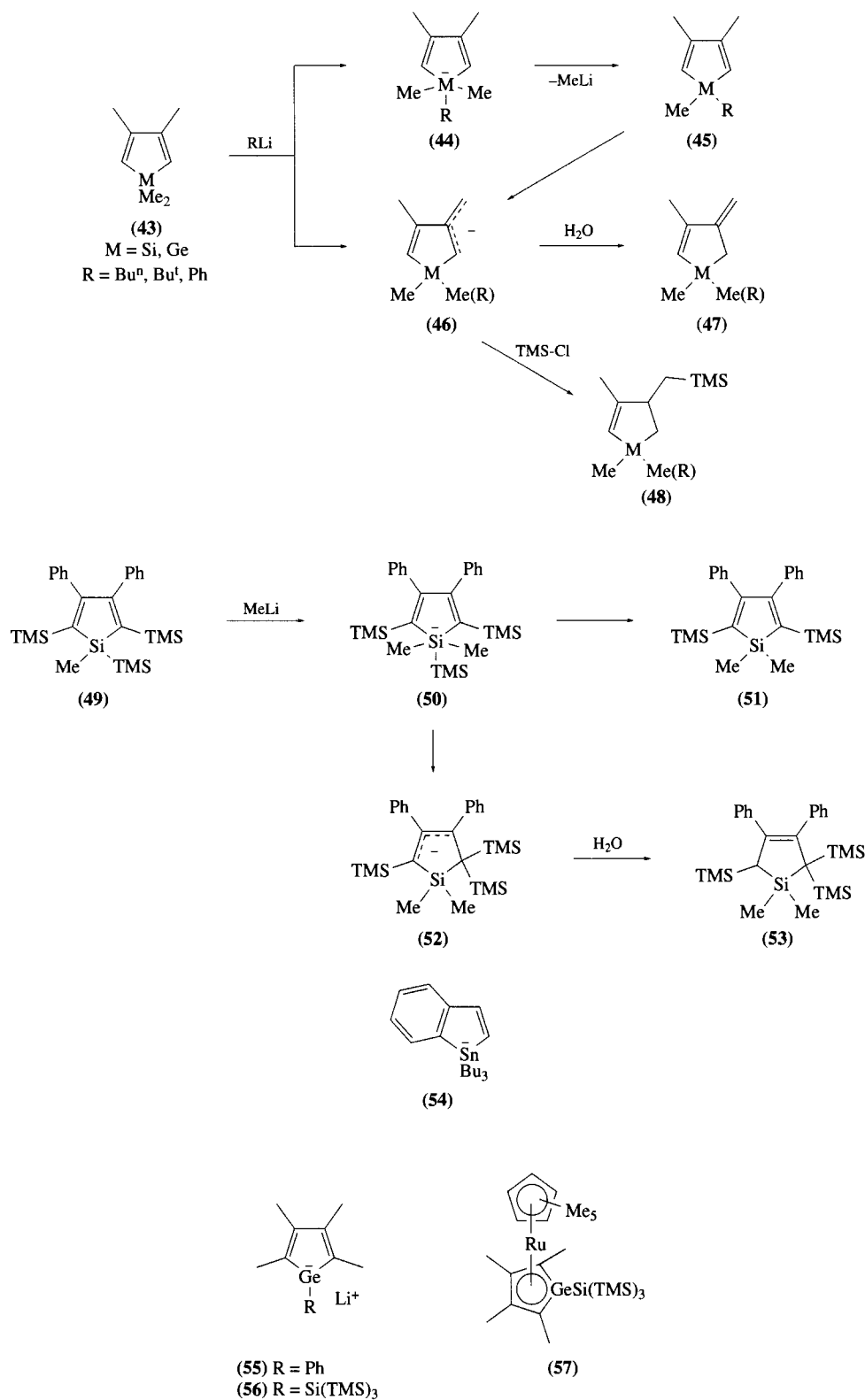
The tetrahydrosilole epoxide (**65**) is also attacked at silicon by methyl or *t*-butyllithium leading to the acyclic product (**64**) $\langle 83\text{OM391} \rangle$. However LDA preferentially removes the C(2) proton affording (**66**). It is noteworthy that the corresponding amino Grignard, R_2NMgBr , effects simple epoxide ring opening instead $\langle 94\text{JOM(484)119} \rangle$. Introduction of an additional anion stabilising substituent such as trimethylsilyl or phenylthio at C(2) of the tetrahydrosilole ring as in (**67**) facilitates deprotonation to (**68**) $\langle 92\text{CL2139} \rangle$. Reaction with alkyl halides then provides (**69**), which can be converted by hydrogen peroxide and potassium fluoride to the corresponding 1,4-butanediol.

2.17.4.4 Heteroatom Reactions

Once more most of the pertinent chemistry reported centres upon siloles and parallels that are generally associated with silanes. Apart from those encountered in Section 2.17.4.3 the more common conversions encountered are summarized in Table 1 along with appropriate references.

2.17.4.5 Cycloaddition Reactions

As mentioned in the introduction to this chapter, some of the simplest metalloles only enjoy a fleeting monomeric lifetime prior to dimerization. Thus the parent silole is only known as the monomer in an argon matrix $\langle 94\text{JA320, 92OM559} \rangle$, and its 1,1-dimethyl derivative dimerises to (**70**) over a couple of hours at room temperature $\langle 83\text{CL213} \rangle$. This propensity is reduced by introducing bulky substituents such as mesityl groups onto the ring silicon $\langle 93\text{JOM(461)173} \rangle$. 1,1-Dimethylgermole



dimerizes more slowly and 1,1-dibutylstannole is perfectly stable as the monomer <88OM1878>. The presence of *C*-substituents greatly reduces dimerizing tendencies, for example, the introduction of methyl groups at positions three and four of 1,1-dimethylgermole leads to a perfectly stable monomer <83TL3521>. Interception of siloles <82NJC67, 88JOM(350)3, 89OM1292, 92JOM(426)C1, 93JOM(461)173,

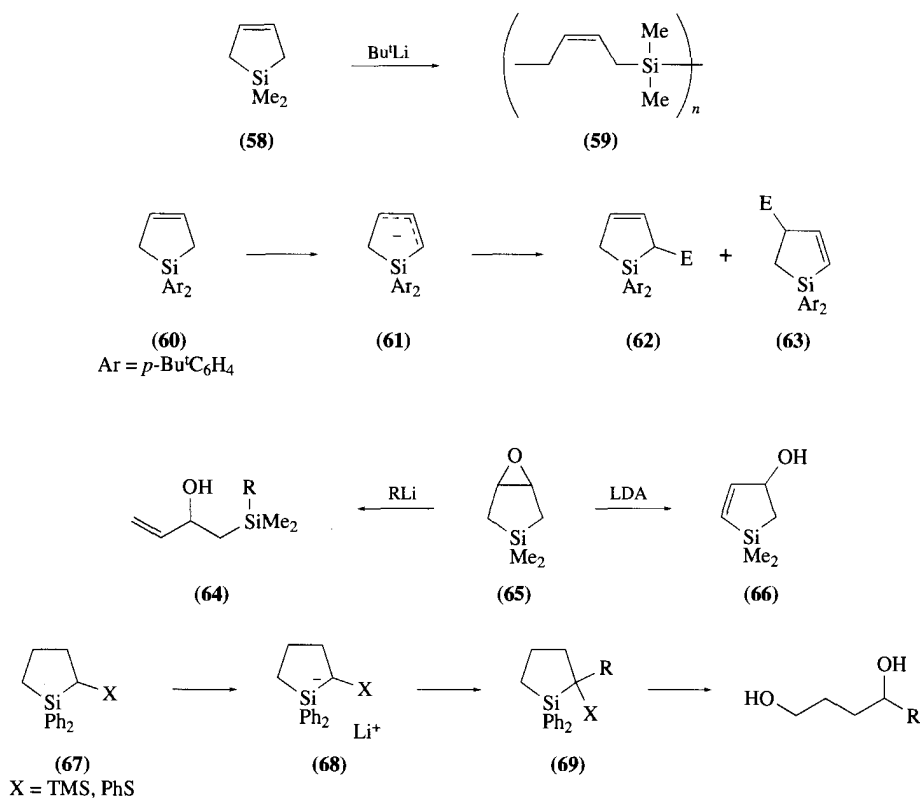
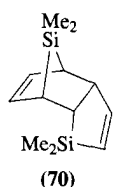


Table 1 Typical conversion reactions of siloles.

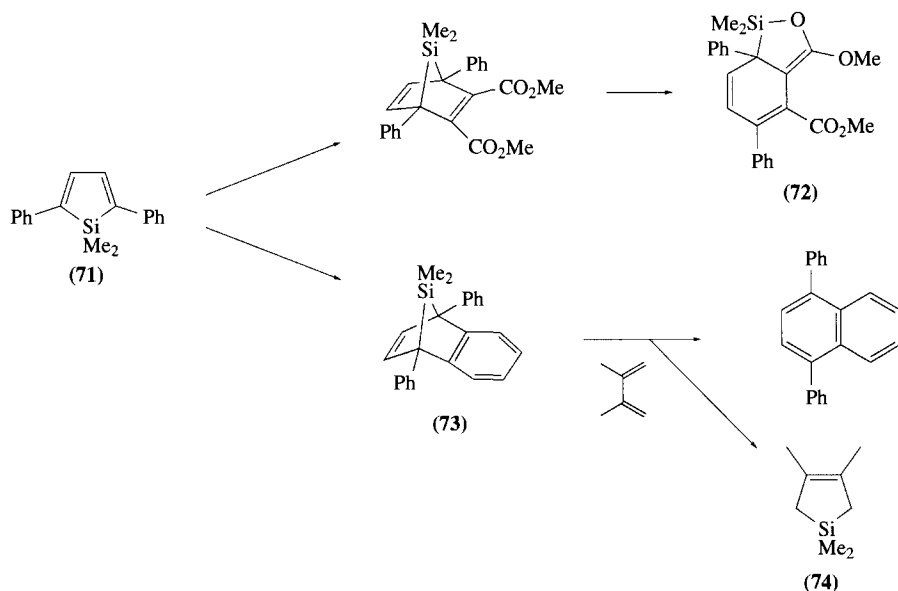
Transformation	Reagents	Ref.
Si-OMe → Si-H	LiAlH ₄	86OM910
Si-F	BF ₃	89OM1799
Si-H → Si-F	Ph ₃ C ⁺ BF ₄ ⁻	89OM1799
Si-Cl	SO ₂ Cl ₂ or PCl ₅	86JOM(304)93, 86OM910
Si-Br	NBS	86JOM(304)93
Si-OR	ROH/(Ph ₃ P) ₃ RhCl	86JOM(304)93
Si-OR	<i>n</i> -Bu ₃ SnOR	89OM1799
SiNEt ₂	Et ₂ NLi	89OM1799
Si-allyl	AllylMgBr/(Ph ₃ P) ₂ NiBr ₂	86OM910

94BCJ2763> or germoles <82NJC67, 90OM869, 93CB1385, 91JOM(419)57> as their maleic anhydride or triazolinedione adducts has been widely used as a means of characterisation. The resulting adducts all appear to have the anticipated endo stereochemistry.

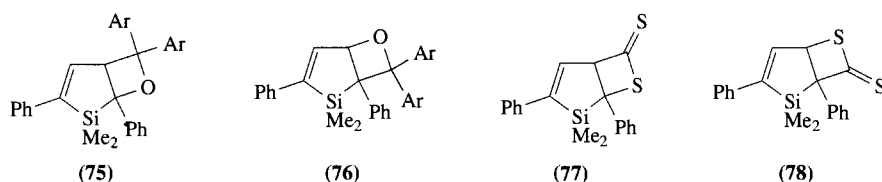


Adducts with acetylenic dienophiles tend to undergo subsequent rearrangement thus 1,1-dimethyl-2,5-diphenylsilole (71) with DMAD yields (72) as the only isolable product <82JOM(225)87>. Similar behaviour has been observed with butynone and methyl propiolate <85JOM(291)9>. Addition of benzyne, generated from 1-aminobenzotriazole and lead tetraacetate, to (71) yields the silabenzonornbornadiene adduct (73) <82JA6156>. Such adducts are of particular interest as on thermolysis

they eliminate for example dimethylsilene which can be trapped with 2,3-dimethylbutadiene as the dihydrosilole (**74**).



The [2 + 2] cycloaddition reactions have received little attention up to 1995. The photochemically promoted addition of benzophenones to 1,1-dimethyl-2,5-diphenylsilole provides the adducts (**75**) and (**76**) as the major and minor products respectively <90CC1718>. Similarly the addition of carbon disulfide to the same substrate yields the β -thiolactones (**77**) and (**78**) <92TL4013>.



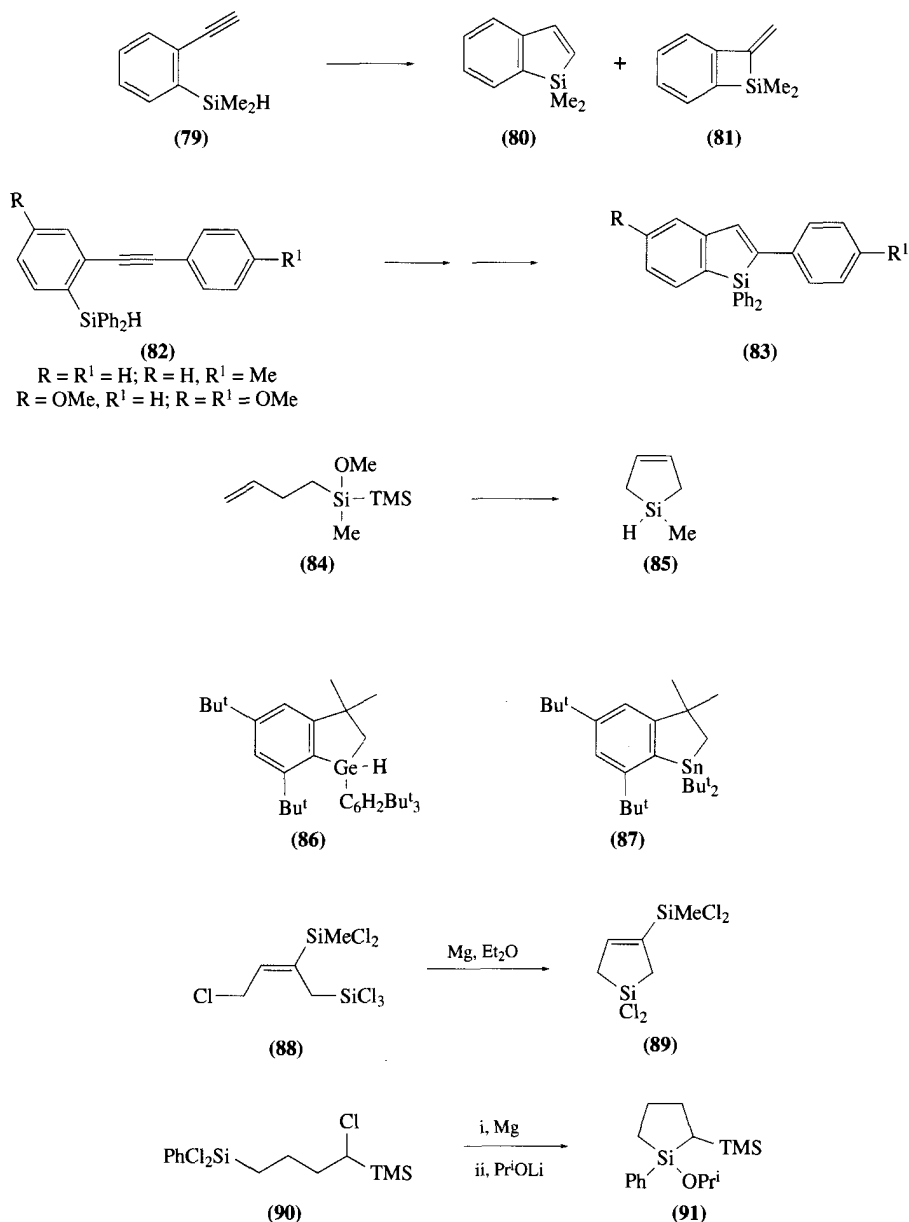
2.17.5 RING SYNTHESIS

2.17.5.1 Formation of One Bond

2.17.5.1.1 Adjacent to the heteroatom

Flash vapour pyrolysis of *o*-dimethylsilylphenylacetylene (**79**) at 800°C provides the benzo[*b*]silole (**80**) in good yield <85OM575>. At lower pyrolysis temperatures the isomeric silabenzocyclobutene (**81**) is also obtained. Diarylacetylenes (**82**) are converted by chloroplatinic acid to an intermolecular polymer, which on heating to 350°C generates the corresponding benzo[*b*]siloles (**83**) <92TL1601>. Pyrolysis of the butenylsilane (**84**) proceeds via the alkenylsilene to the dihydrosilole (**85**) <83OM1>.

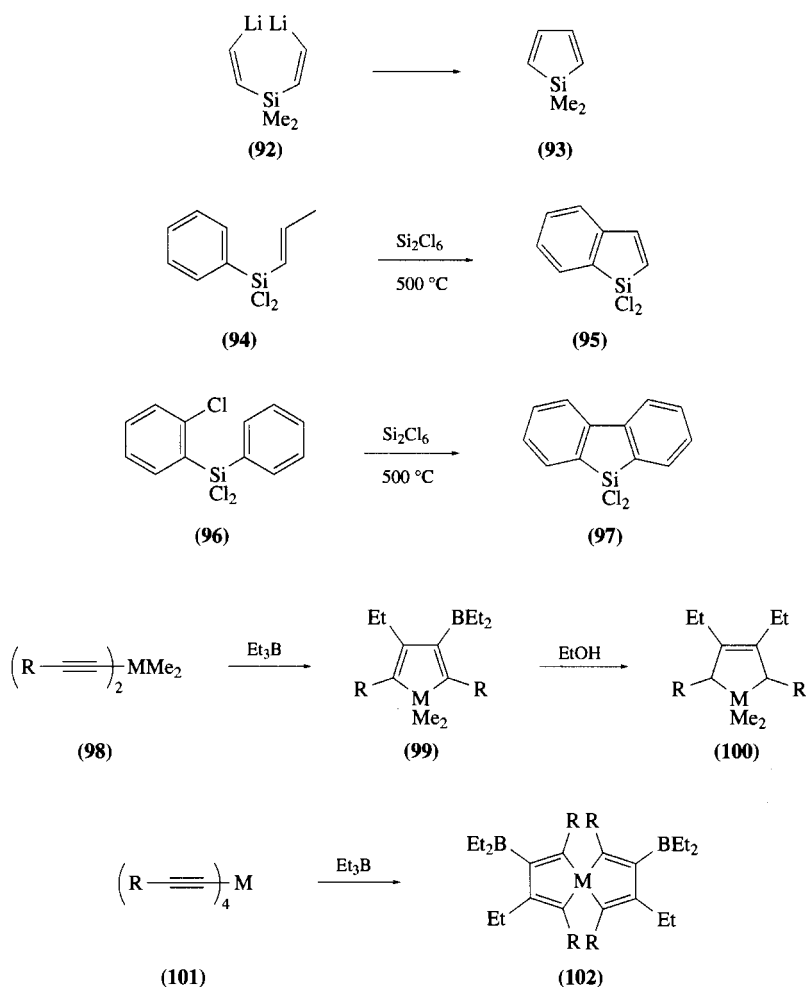
The reaction of 2,4,6-tri-*t*-butylphenyllithium with germanium dichloride at -10°C generates bis(2,4,6-tri-*t*-butylphenyl)germylene which, at room temperature, undergoes intramolecular C—H insertion forming the dihydrobenzogermylene (**86**) <87JOM(329)C17>. An analogous benzostannole (**87**) has been obtained by treatment of di-*t*-butylchloro(2,4,6-tri-*t*-butylphenyl)silane with *t*-butyllithium <90JOM(381)173>. The cyclization is presumably initiated by deprotonation of an adjacent *t*-butyl methyl group followed by attack of the resulting carbanion on tin. A mechanistically analogous process is involved in the cyclization of (**88**) to (**89**) on treatment with magnesium <93JOM(444)37>, and in the cyclization of (**90**) to (**91**) <94BCJ1694>.



2.17.5.1.2 γ to the Heteroatom

The copper iodide mediated coupling of bis(2-lithiovinyl)dimethylsilane (92) provided 1,1-dimethylsilole (93), which was isolated as the dimer $\langle 83\text{CL}213 \rangle$. Thermolysis of the phenylvinylsilane (94) with hexachlorodisilane yields the benzo[*b*]silole (95) $\langle 84\text{JOM}(271)129 \rangle$. Analogously, pyrolysis of (*o*-chlorophenyl)phenyldichlorosilane (96) with hexachlorosilane provides high yields of the dichlorodibenzosilole (97) $\langle 84\text{JOM}(271)129, 85\text{ZOB}2317 \rangle$.

By far the most extensively studied synthesis of this class is the cyclization of a bisalkynylmetal derivative (98) by reaction with a trialkylborane to form the metallole (99). This reaction has been applied to the production of siloles $\langle 86\text{JOM}(310)151, 93\text{CB}1107, 93\text{CB}2221 \rangle$, germoles $\langle 86\text{JOM}(310)151 \rangle$, stannoles $\langle 94\text{ICA}161, 93\text{CB}1361 \rangle$, and the elusive plumboles $\langle 90\text{JOM}(399)1 \rangle$. Subsequent reaction of (99; M = Si, Ge, R = SnMe₃) with ethanol provides the 2,5-dihydrometallole (100) $\langle 86\text{JOM}(310)151 \rangle$. Various putative intermediates have been characterized by NMR and x-ray crystallography. Extension to tetraalkynylmetal derivatives (101) has provided access to the spiroiloles (102; M = Si), spirogermoles (102; M = Ge) $\langle 93\text{CB}1385 \rangle$, and spirostannoles (102; M = Sn) $\langle 92\text{CB}1597 \rangle$.



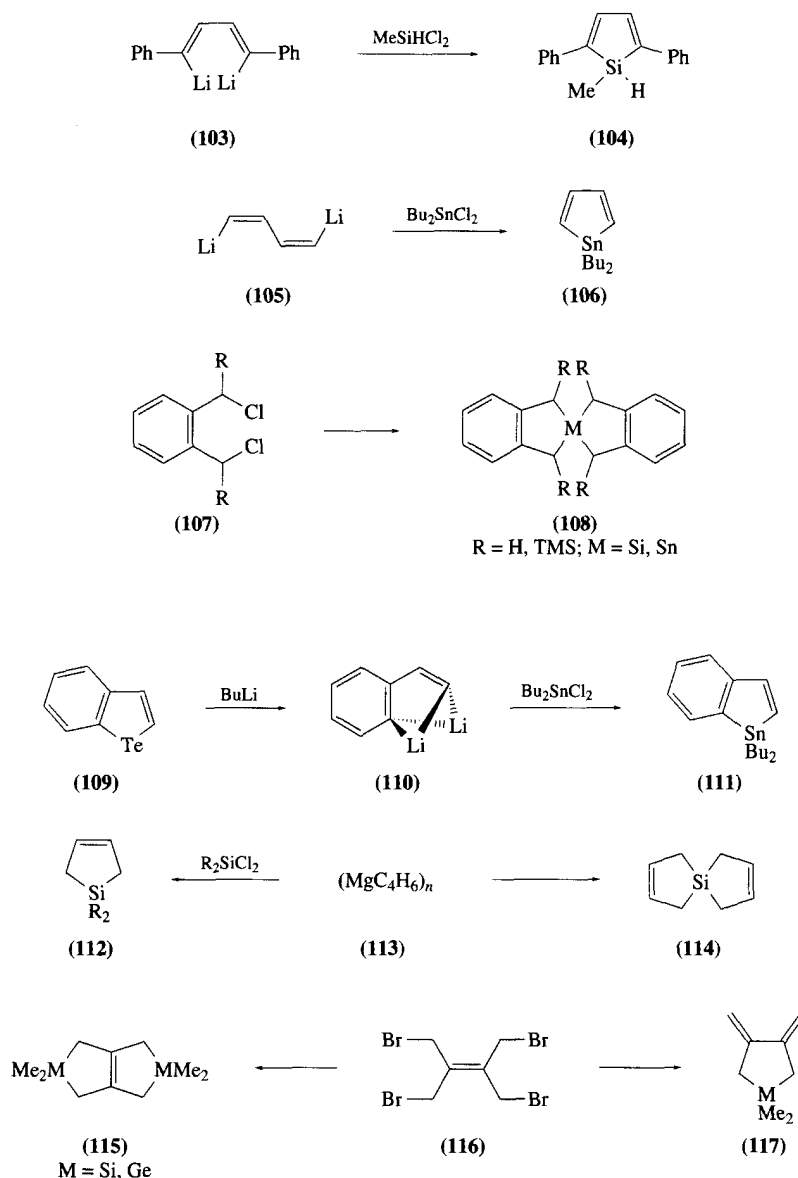
2.17.5.2 Formation of Two Bonds

The classic ring synthesis of this type entails the reaction of a 1,4-dilithio (or magnesio) compound with an appropriate metal dihalide as in the synthesis from 2,2-dilithiobiphenyls of dibenzosiloles <86JOM(307)7>, dibenzostannoles <91JOM(402)171>, and octafluoro-dibenzogermoles <86JOM(302)343>. The 2,5-diphenylsilole (104) has been prepared from the dilithiodiene (103) <86OM910>, and the dibutylstannole (106) from 1,4-dilithiobutadiene (105) <88OM1878>. The dilithio- or Grignard reagents derived from the *o*-xylyl chlorides (107) have been used to prepare the spiro compounds (108) <92JCS(D)775>.

The cleavage of benzotellurophene (109) with butyllithium provides a dilithio species formulated as (110) which has been converted to the benzo[b]stannole (111) by reaction with dibutyltin dichloride <92AG(E)1339>. The magnesio species derived from 1,3-butadienes (113) is a useful precursor of dihydrosiloles (112) and (114) <82S1102, 85JOM(289)45, 91JOC3109>. The tetrabromodimethylbutene (116) reacts with dimethylsilicon or germanium dichloride in the presence of lithium, sodium, or potassium providing the bicyclic metalloles (115), whereas in the presence of zinc the reaction gives the monocycles (117) instead <91JOM(406)119>.

An alternate approach to this mode of ring construction entails the addition of a divalent metal species to a 1,3-diene. Such reactions are only exemplified for silicon and germanium. Thus addition of germanium diiodide to 2,3-dimethylbutadiene yields the dihydrogermole (118) <85BCJ974>. Similar additions to dienes such as (119), followed by reaction with methylmagnesium iodide provide (120). In those cases where $\text{X} = \text{S}$ or Se the chalcogen ring can be dehydrogenated with DDQ to give (121). When $\text{X} = \text{S}$ the bicycle (120) can be oxidised to the sulfone (122) which eliminates sulfur dioxide, forming the germole (123) <89JOM(366)57, 91JOM(402)35, 92JOM(439)115>.

In the silicon series there are no suitable divalent compounds available off the shelf and appropriate silenes must be generated *in situ*. Reduction of dialkoxydichlorosilanes with magnesium in the

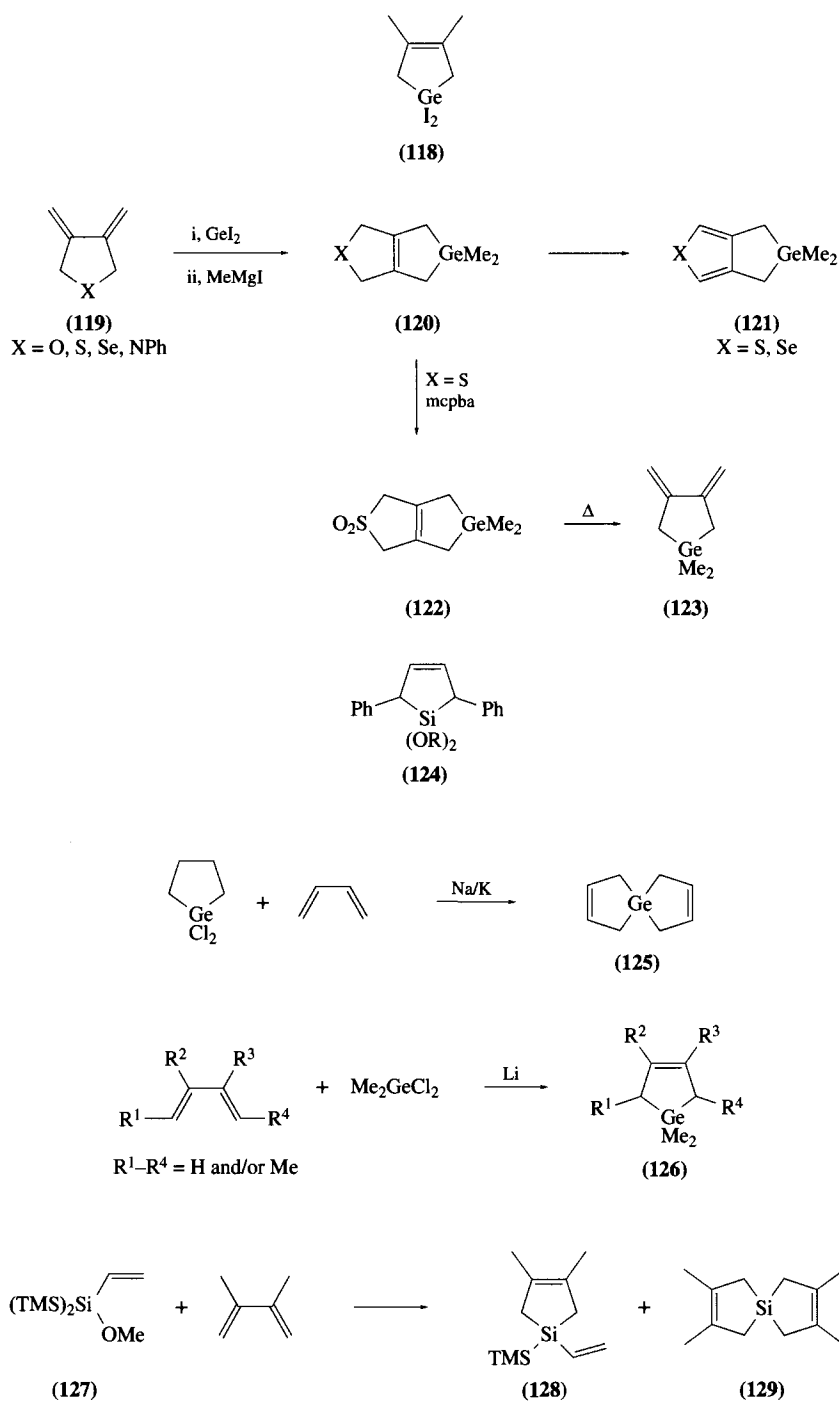


presence of 1,4-diphenylbutadiene has been shown to yield a mixture of the *trans*- and *cis*-dihydrosiloles (124) <93BCJ2294>. Similar approaches have been adopted for the syntheses of the germoles (125) <86JOM(301)153> and (126) <91POL1221>.

An alternative source of silene intermediates is the pyrolysis of suitable silanes <94JOM(474)71>. Thus pyrolysis of hexafluorodisilane at 670–720 °C in the presence of 1,3-butadiene provides a good yield of 1,1-difluoro-2,5-dihydrosilole <86JOM(307)151>, and (127) at 500 °C in the presence of 2,3-dimethyl-1,3-butadiene gives mainly (128) accompanied by a little of the spiro compound (129) <83TL159>. These reactions appear to proceed through initial formation of a vinylsilacyclopropane followed by a 1,3-silyl shift <88CC241, 88CC1139>.

2.17.5.3 Formation of Three Bonds

The reaction of disubstituted acetylenes with 1,1,2,2-tetramethyldisilane catalyzed by bis(triethylphosphine)nickel(II) or palladium(II) dichloride provides a convenient source of siloles

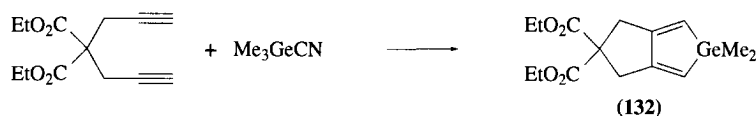
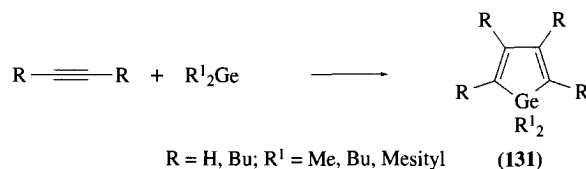
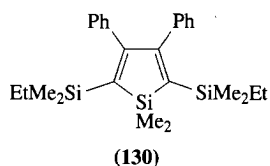


(130) <81JOM(217)43, 83OM974, 88MI 217-01>. Corresponding palladium mediated reactions have also been reported for the synthesis of the germoles **(131)** <90OM869, 91SL431> and **(132)** <94JOM(473)335>.

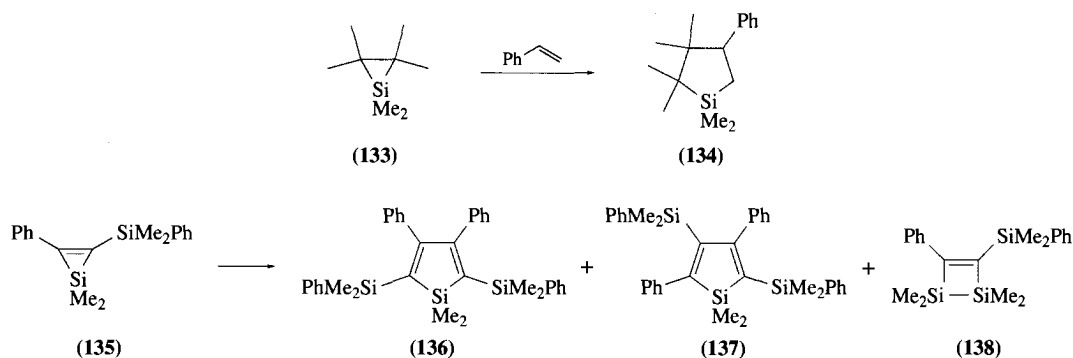
The reaction of styrene with dimethyldichlorosilane in the presence of lithium gives a high yield of 2,5-diphenyltetrahydrosilole <93BCJ2294>.

2.17.5.4 Transformation of Existing Heterocycles

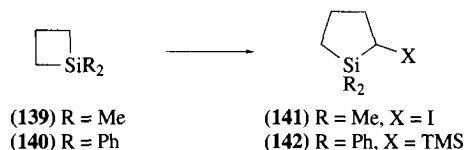
Hexamethylsilirane **(133)** undergoes insertion of styrene at room temperature forming the tetrahydrosilole **(134)**. Analogous insertions have been observed with 1,3-dienes, acetylenes and benzyne



⟨84OM574⟩. Thermolysis of the silirene (135) at 250°C provides a mixture of the siloles (136) and (137) together with some (138) ⟨83OM974⟩.



Although the gas-phase reaction of singlet methylene with 1,1-dimethylsilacyclobutane to form 1,1-dimethyltetrahydrosilole is probably of limited synthetic importance ⟨89CC284⟩, ring expansion of (139) with diiodomethane and LDA to (141) ⟨93T8487⟩, and of (140) with ICH(Li)SiMe₃ to (142) ⟨94BCJ1694⟩ provides convenient access to functionally substituted compounds.

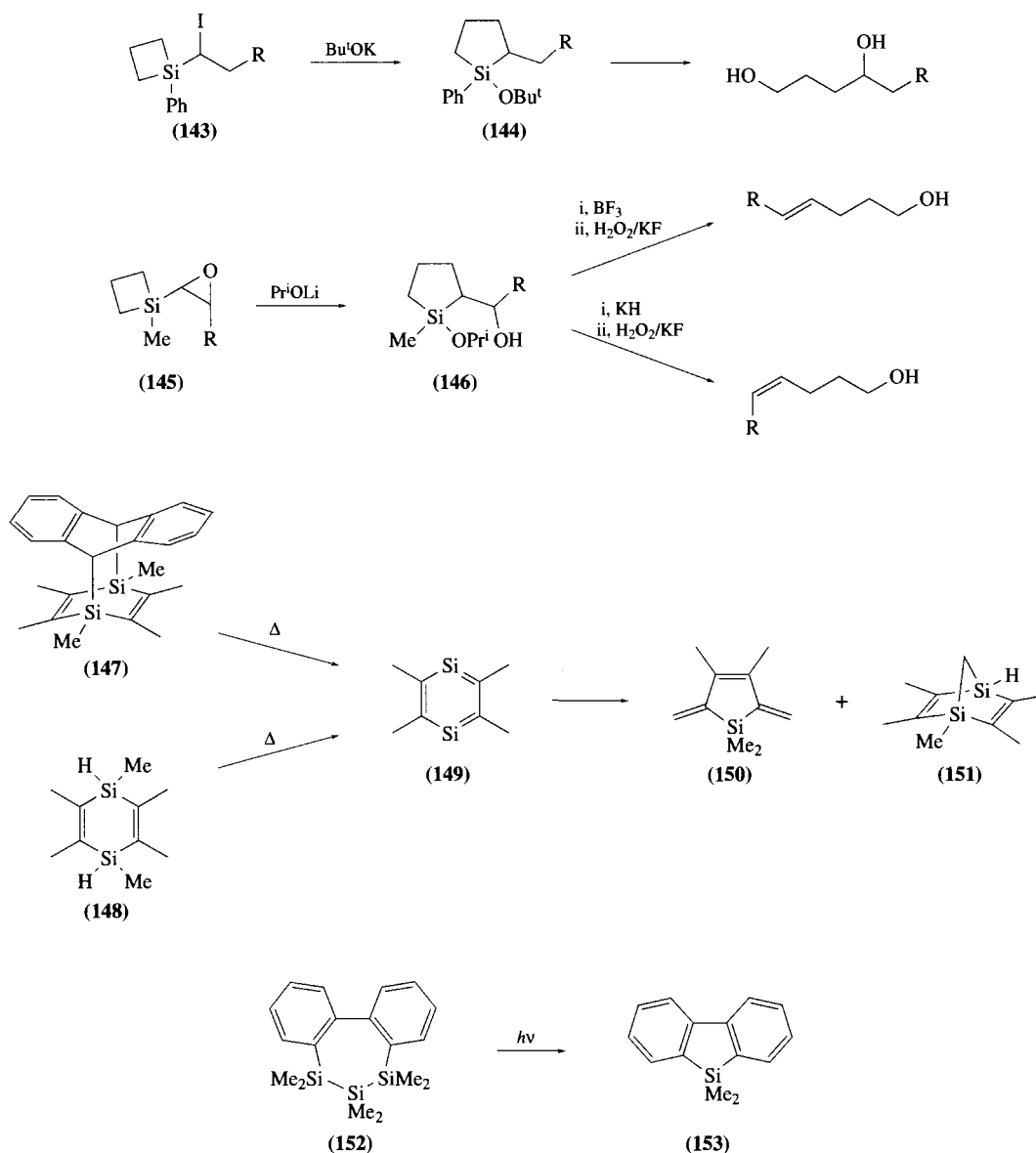


Examples of ring expansions involving substituents already bonded to silicon are provided by the conversion of (143) to (144) ⟨91TL6383⟩, and of (145) to (146) ⟨91TL4545⟩. The products of ring cleavage of (144) and (146) point to wider synthetic applicability of these reactions.

Flash vapour pyrolysis of the 1,4-disilabenzene (149), generated from the anthracene adduct (147) or the dihydro derivative (148), provides the unusual silole (150) accompanied by minor amounts of (151) ⟨87JOM(325)105⟩.

Photolysis of (152) yields the dibenzosilole (153) and the expelled dimethylsilene species can be trapped as the dihydrosilole with 2,3-dimethylbutadiene ⟨84CL1379⟩. However, photolysis of the germanium analogue of (152) only proceeds as far as the expulsion of one dimethylgermene unit.

Since the early 1990s zirconacyclopentanes and zirconacyclopentadienes have become very readily available (cf. Chapter 2.19) so that replacement of the zirconium by reaction with a suitable dihalo derivative of silicon, germanium, or tin provides a very attractive route to siloles, germoles, or



stannoles. A variety of examples is shown in Figure 3 <88JA2310, 90JOM(384)61, 93JOM(453)33, 93JOM(454)51, 94BSF715, 94JA1880>. In most cases it is not necessary to isolate the intermediary zirconacycle.

2.17.5.5 Interconversion of Metalloles and Their Reduced Derivatives

Some of the most convenient methods of ring synthesis provide the 2,5-dihydroheterocycle rather than the fully saturated or unsaturated one, hence much effort has been devoted to establishing appropriate methods for interconversion. A successful strategy in the silole series is to prepare the 1-allyl-2,5-dihydro compound as in (154). Subsequent flash vapour pyrolysis results in elimination of propene and formation of the 2*H*-silole (155), which then rearranges to the more stable 1*H*-silole (156) <81JOM(209)C25, 89OM1292, 86OM1742, 92JOM(426)C1>. When R^2 is a methyl group, a minor amount of the exocyclic diene (157) is also obtained <89OM1292>. The reaction fails in the germole series as the dihydrogermole preferentially dissociates to the diene <86OM1742>. Although pyrolysis of the dihydrobenzo[*c*]silole (158) has been shown to generate the anticipated 2*H*-benzosilole (159), the isolated products are the benzo[*b*]silole (160), indene (161), and the silanaphthalene (162) <82OM1455>.

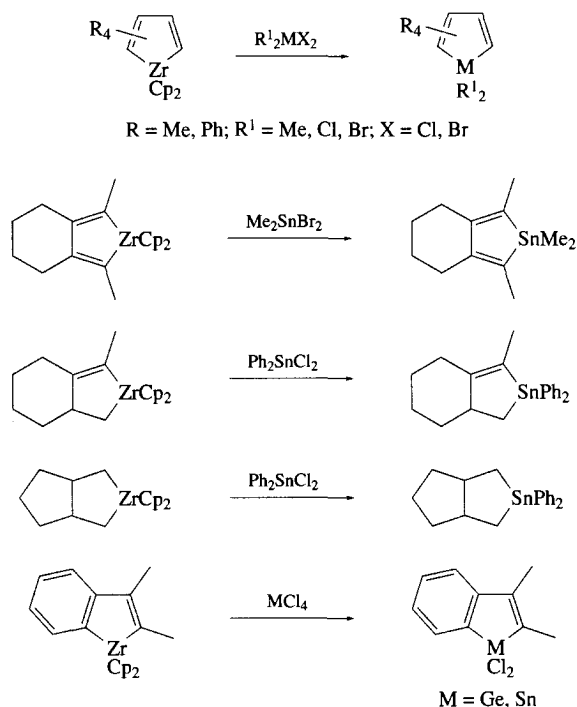
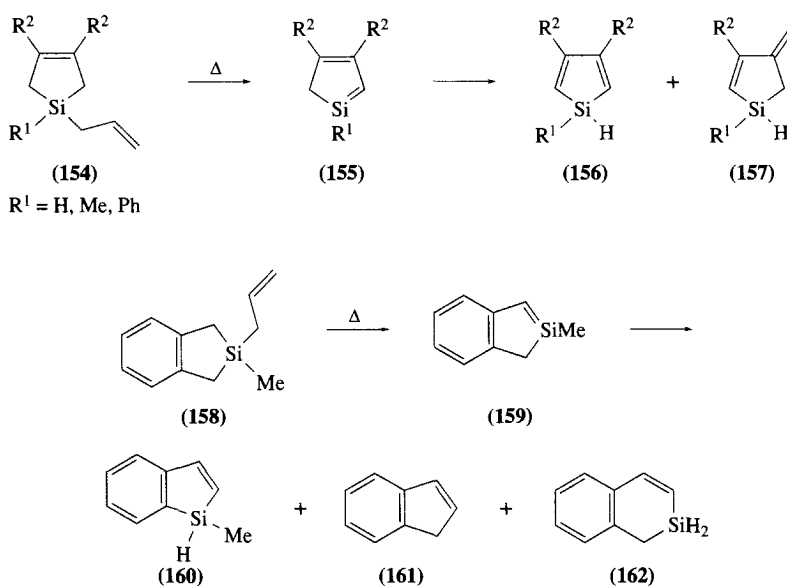
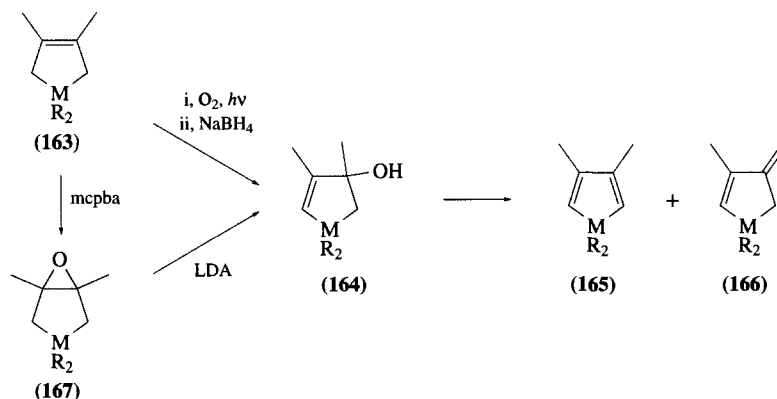


Figure 3 Examples of zirconacyclopentanes and zirconacyclopentadienes <88JA2310, 90JOM(384)61, 93JOM(453)33, 93JOM(454)51, 94BSF715, 94JA1880>.

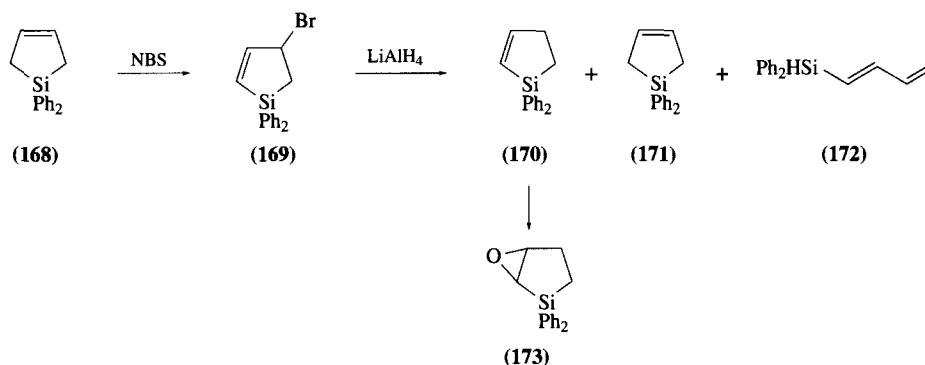


Early attempts to apply bromination–dehydrobromination methods to this area proved problematical. However, this approach has been successfully applied to the dehydrogenation of 1,1,3,4-tetramethyl-2,5-dihydrosilole <85MI 217-01>. 1,1,3-Trimethyl-2,5-diphenyltetrahydrosilole has also been converted to the silole by successive bromination with NBS and dehydrobromination with potassium acetate <92TL4013>. In view of the problems originally encountered with this approach, an alternative sequence has been established. This commences with the photo-oxygenation of the dihydroheterocycle (163) followed by borohydride reduction of the resulting hydroperoxide to the alcohol (164). The product may either be dehydrated by heating with alumina at ca. 300 °C, or by thermolysis of the xanthate or phenylcarbamate. Again, the presence of a methyl group at position three results in formation of some of the diene (166) <82NJC67, 83JOM(244)C12, 83TL3521, 85OM636,

93JOM(461)173>. A more selective route to the alcohol (**164**) entails epoxidation of (**163**) to (**167**) with MCPBA, followed by isomerization to (**164**) with LDA <83OM391, 94JOM(484)119>. These routes have been successfully applied to both siloles and germoles.



At best, most synthetic routes produce only minor yields of 2,3-dihydroheterocycles. Free radical bromination of the dihydrosilole (**168**) with NBS and benzoyl peroxide provides the bromide (**169**) which is converted by lithium aluminum hydride to a mixture of (**170**) 70%, (**171**) 10%, and (**172**) 20% <93JOM(447)167>. Compound (**170**) is readily epoxidised to (**173**) and this, like (**167**), undergoes ring opening with a range of nucleophiles providing access to a variety of tetrahydrosiloles.



The reduction of these heterocycles has so far received much less attention. 1-Phenyl-1,3,4-trimethylsilole has been reduced to the 2,5-dihydro derivative with lithium in THF <86OM1742>, and the palladium on carbon catalyzed hydrogenation of 1,1-dialkoxy-2,5-dihydro-2,5-diphenylsiloles to the corresponding tetrahydrosiloles has been reported <93BCJ2294>.

2.18

Boroles

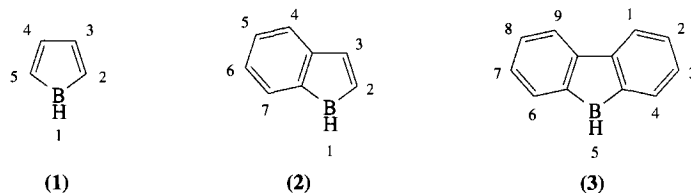
GEORGE VARVOUNIS
University of Ioannina, Greece

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2.18.1 INTRODUCTION

Free boroles are highly reactive four π -electron antiaromatic systems, and only a few sterically protected derivatives have been isolated up to 1995. The only known isolable monomeric boroles without annulated aromatic groups are derivatives of 1,2,3,4,5-pentaphenylborole (**1**), first described in 1961. The antiaromaticity is evident by the rapidity of reactions that remove the empty $2p_z$ -orbital on boron from conjugation (Lewis complexation) or disrupt the borole ring system altogether (e.g., Diels–Alder addition). An earlier review in the first edition of *Comprehensive Heterocyclic Chemistry* (CHEC-I) has only dealt with the borole ring <84CHEC-I(1)629>.

The numbering system used for borole (**1**), benzo[*b*]borole (**2**), and dibenzoborole (**3**) is shown. An alternative name for dibenzoborole is 9-boraffluorene.



2.18.2 THEORETICAL METHODS

An SCF- π method including variable π -electronegativity and σ -polarization was applied to the calculation of electronic transitions and ionization potentials of several heteroatomic molecules including dibenzoborole. The agreement between calculated and observed transition energies, oscillator strengths, and ionization potentials was very good <70TCA239>.

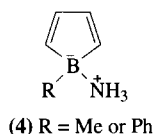
The variable electronegativity self-consistent field (VESCF) method has been used to calculate the electronic spectra of 1-ethylborole and 5-ethylidibenzoborole. Experimental and calculated values were in fair agreement <75JA752>.

The relative energies of the π -electron system of borole have been calculated by the Hückel MO approximation <81JOC315>.

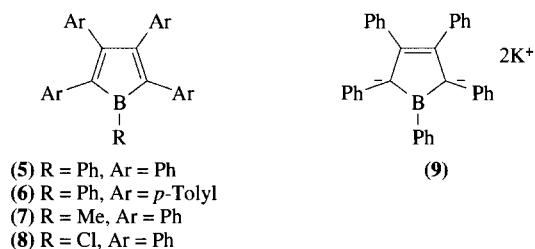
The static electronic polarizabilities and hyperpolarizabilities were determined for some aromatic and antiaromatic heterocycles such as pyrrole and borole, in ground and excited first singlet and triplet electronic states. It was found that the singlet excitation state of borole is connected to a significant decrease of the polarizabilities and hyperpolarizabilities <93IJQ332>.

2.18.3 EXPERIMENTAL STRUCTURAL METHODS

The instability of borole species has limited the in-depth study of the chemistry of boroles and as a consequence their spectroscopic properties. The simplest ^1H -borole derivatives prepared are the ammonia adducts (4); however, the ^1H NMR signals from these compounds dissolved in ammonia where they are stable, are broad, possibly owing to the presence of traces of paramagnetic material in their solutions. The alkenic protons are found in the normal spectral region at δ 6.43 for the α -protons and δ 6.28 for the β -protons, but the expected AA' BB' pattern cannot be resolved. Two NH_3 proton signals are found, a singlet for dissolved ammonia at δ 0.39 and a broad signal for the boron-bonded NH_3 at much lower field. NMR experiments with these compounds in THF demonstrates exchange at room temperature of the stabilizing Lewis base <87JOM(336)29>.



The spectral properties of pentaarylboroles (5)–(8), and their adducts with benzonitrile, potassium, pyridine, and tetrahydrofuran are available <69JA4575, 86JA379>. The ^1H , ^{13}C , and ^{11}B NMR spectra of the adducts exhibit absorptions useful in characterizing the borole nucleus and the π -electron density on boron. For example, in the ^1H NMR spectrum of the THF adduct of borole (5) the protons of the B-phenyl group occur at the lowest field, δ 7.3–7.5, well separated from the C-phenyl protons. In the ^{13}C NMR spectrum of the same compound, however, all the phenyl carbons fall between 125 and 131 ppm. On the other hand, in the ^{13}C NMR spectrum of the dipotassium adduct (9), the carbons of the B-phenyl group are clearly shifted upfield 117–121 ppm, while the remaining C-phenyl carbons still resonate between 127 and 136 ppm. An interesting shift is discerned in the ^{11}B NMR spectra of borole (5) and its pyridine adduct. The ^{11}B resonance of (5) at 55 ppm undergoes an upfield shift to 5 ppm upon complexation with pyridine. This shift of 50 ppm is much greater than the shift of 26 ppm reported for the dipotassium adduct (9).



The most striking spectral characteristic of these compounds is the blue to blue-violet colour of the uncomplexed borole ($\lambda_{\text{max}} = 540\text{--}605\text{ nm}$) and the pale yellow colour of the neutral borole

complexes ($\lambda_{\max} = 330$ nm). The red colour of the dianion (**9**) represents an intermediate electronic spectral shift.

The IR spectra of the boroles (**5**)–(**8**) and their adducts display a sharp band of medium to strong intensity in the 1575 – 1595 cm^{-1} region due to the stretching of the ring alkenic bonds conjugated with the phenyl groups. Upon complexation of benzonitrile with borole (**5**) a shift in the nitrile stretching frequency from 2300 to 2370 cm^{-1} is observed. This shift exceeds by 11 cm^{-1} the corresponding shift observed when benzonitrile complexes with AlCl_3 . Since the magnitudes of such shifts have been correlated with Lewis acid strengths, boroles can be considered as being among the stronger Lewis acids.

Benzo[*b*]boroles (**2**) are also a very reactive species. An attempted synthesis in 1987 of 1-chloro-benzoborole gave the dimer of this compound and ^1H NMR spectroscopic identification of the monomer could not even be achieved at -90°C [87AG(E)665].

Although dibenzoborole (**3**) is a much more stable ring system, the difficulty in synthesizing these compounds has remained an obstacle in the study of their chemistry. In a report the ^1H NMR and ^{11}B NMR spectra of various 5-substituted derivatives such as bromo, iodo, methylthio, methoxy, and diethylamino were recorded [85JOM(281)131]. The aromatic proton signals resonate in the range δ 6.60–7.88 ppm, and the ^{11}B signal appears between δ 46.0 and 65.8 ppm.

2.18.4 THERMODYNAMIC METHODS

The empty $2p_z$ -orbital on boron interacting with the π -electrons of the butadienyldiene system in borole (**5**) is proved by the pronounced bathochromic shift in the visible spectrum compared with that of its pyridine adduct. This π -electron delocalization in boroles is destabilizing and produces a 4π -electron antiaromatic system. The antiaromaticity is further evidenced by the rapidity of reactions that remove the $2p_z$ -orbital on boron from conjugation. These reactions include Lewis complexation, transition metal complexation, or borole ring opening reactions such as Diels–Alder addition, oxidation, and protodeboration. This removal of the antiaromaticity from borole results in transforming the compound to a typical, low reacting, sterically hindered arylborane.

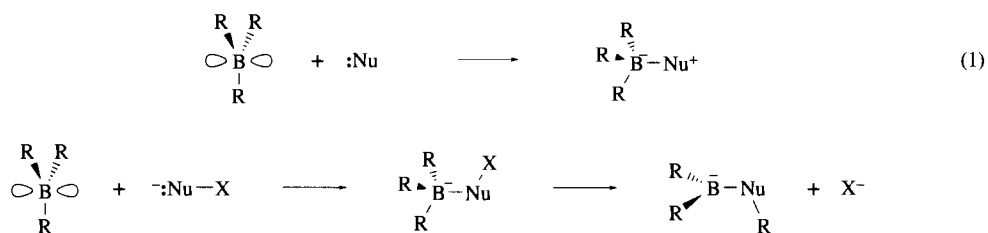
The antiaromaticity of benzo[*b*]boroles is exhibited by their 8π -electron systems and the inability to isolate and characterize these compounds due to their high reactivity.

Dibenzoboroles, on the other hand, are an example of the incorporation of a boron atom into an aromatic π -electron system. Stable trivalent compounds are formed since the aromatic 6π -electrons of each benzene ring do not interact with the empty $2p_z$ -orbital on boron.

2.18.5 REACTIVITY OF FULLY CONJUGATED RINGS

2.18.5.1 General Survey of Reactivity

The empty p -orbital on the sp^2 boron atom of triorganoboranes renders them electrophilic (Lewis acidic) and highly susceptible to attack by electron-rich species (Equation (1)). If a charged nucleophile bearing a leaving group attacks, 1,2-migrations occur very easily (Scheme 1). Corresponding reactions with boroles are, in principle, analogous although the interaction of the dienyldiene system and the presence of a cyclic structure usually changes the nature of the products. As four-electron ligands, boroles are able to form transition metal complexes with strong metal-to-ligand back-bonding.

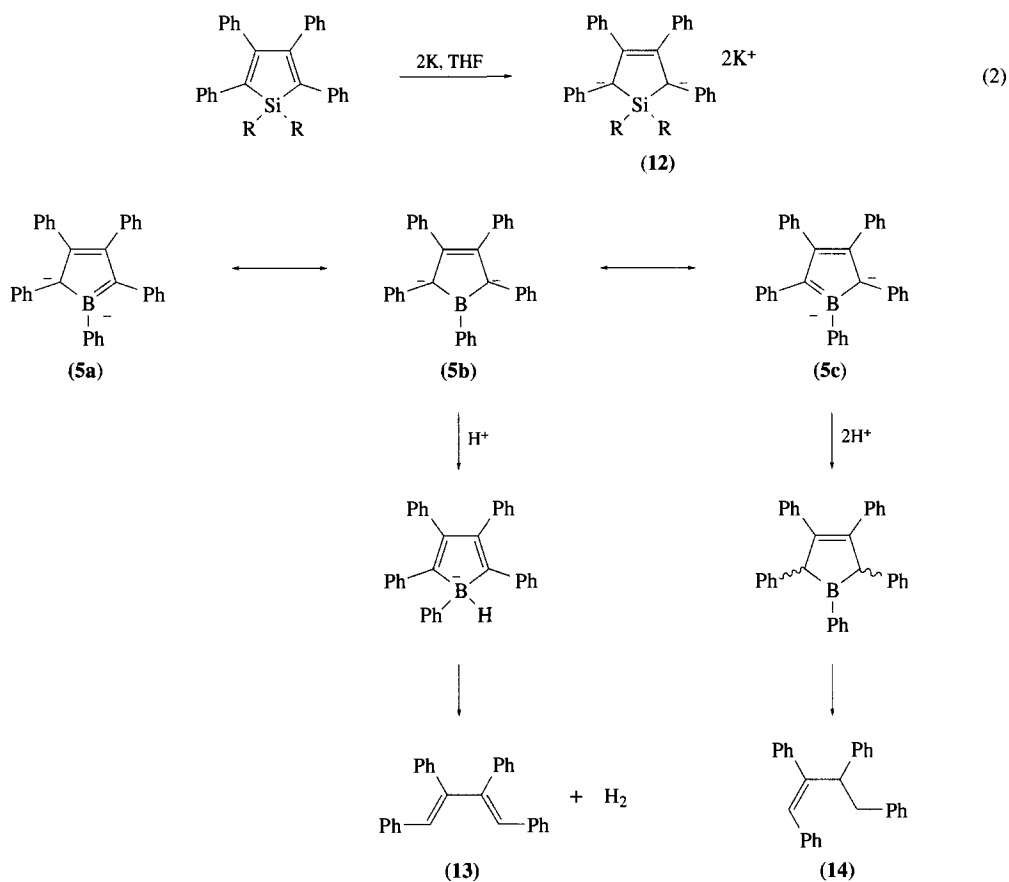


Scheme 1

2.18.5.1.1 Complexation

Pentaphenylborole (**5**) is a deeply colored solid, highly reactive both to air and toward alcohols. It forms colorless to pale yellow adducts with THF, pyridine, or benzonitrile. The ^1H and ^{13}C NMR spectra of the 1 : 1 borole–tetrahydrofuran adduct in CCl_4 , the IR and UV spectra of the borole–pyridine adduct, and the IR spectrum of the borole–benzonitrile adduct have been recorded.

The dipotassium adduct (**9**) was prepared from potassium metal and pentaphenylborole (**5**) in THF solution, as a dark red solid in 91% yield. Since siloles (**12**) also form dimetallo adducts readily (Equation (2)), the independent reaction of pyridine and 4-(dimethylamino)pyridine complexes of (**5**) with potassium metal to give the dipotassium adduct (**9**) confirms the participation of the boron $2p_z$ -orbital in the reaction (Scheme 2). The absence of pyridine from the dipotassium adduct (**9**) proves that the anionic charge in the borole displaces the coordinated pyridine in order to utilize the $2p_z$ -orbital on boron.



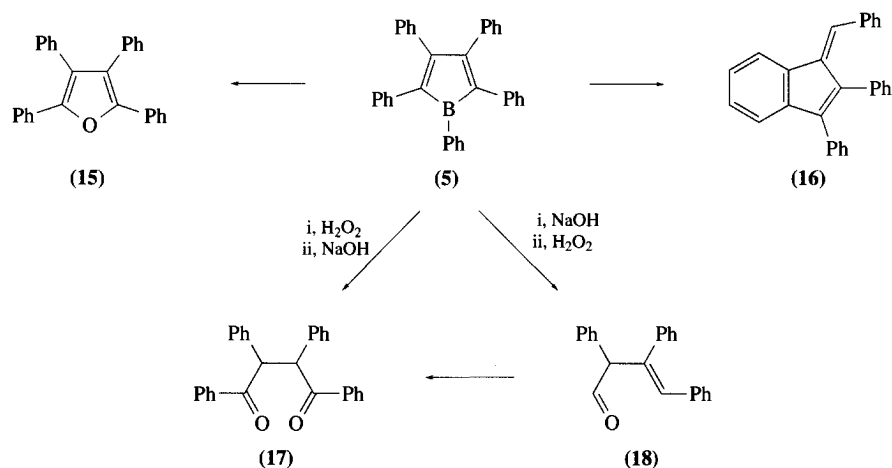
Scheme 2

The dipotassium adduct (**9**) was cleaved by heating in a mixture of glacial acetic acid and 6M HCl to give small amounts of (*E,E*)-1,2,3,4-tetraphenyl-1,3-butadiene (**13**), its (*E,Z*)- and (*Z,Z*)-isomers and (*E*)-1,2,3,4-tetraphenyl-1-butene (**14**) as the major product. The final ring disruption seems to occur with allylic rearrangement <86JA379>.

2.18.5.1.2 Oxidation

Air oxidation of pentaphenylborole (**5**) followed by hydrolytic work-up led to the isolation of tetraphenylfuran (**15**) and 3-benzylidene-1,2-diphenylindene (**16**). Similar treatment of boroles (**6**)–(**8**) yielded 40–80% of the tetraphenylfuran (**15**) (Scheme 3).

The oxidizing action of 30% H_2O_2 in aqueous NaOH solution on borole (**5**) depends upon which reagent is added first. When H_2O_2 was added to (**5**) before introducing the base, oxidation of both

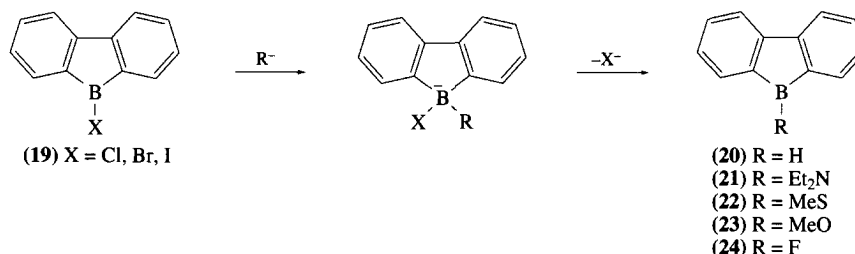


Scheme 3

ring bonds occurred to give 1,4-diketone (17). When aqueous NaOH was added to (5) followed by H_2O_2 , (E)-2,3,4-triphenyl-3-butenophenone (18) was obtained [86JA379]. The initial step of the mechanism for these oxidations must be interaction between the empty *p*-orbital on boron and an electron pair in one of the oxygen's two filled *p*-orbitals as depicted in Scheme 1.

2.18.5.1.3 Nucleophilic substitution on boron

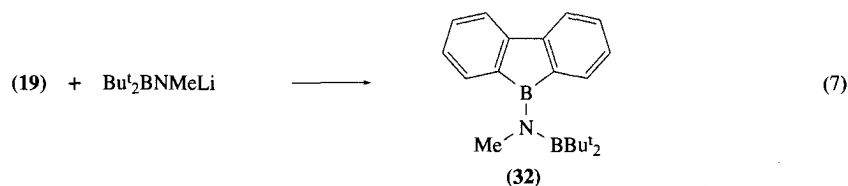
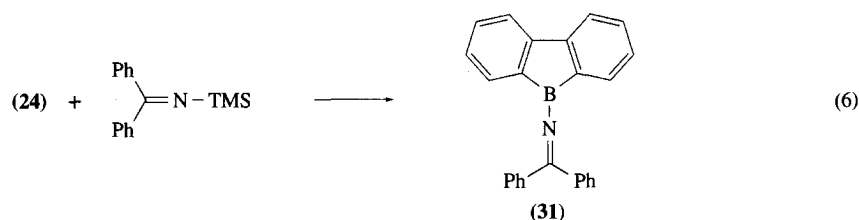
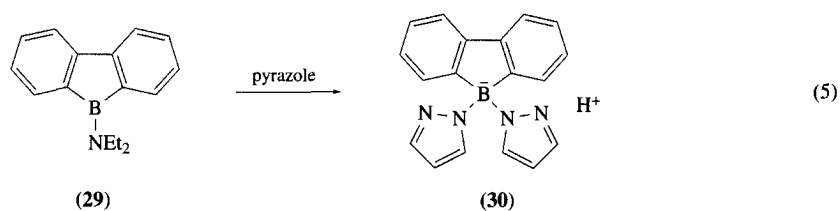
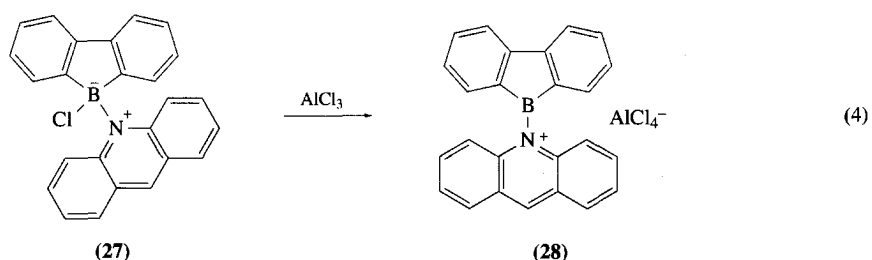
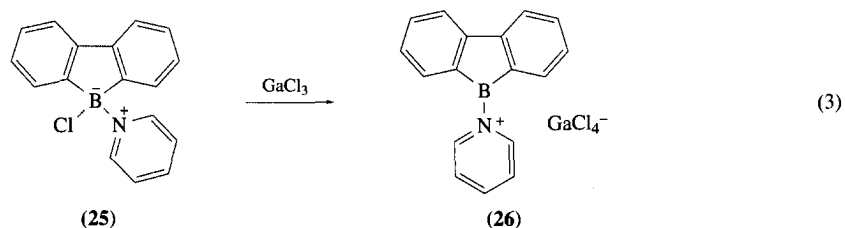
The halogen groups of 5-halodibenzoboroles (19) have been displaced by a variety of nucleophiles. The reaction involves initial nucleophilic addition to the empty *p*-orbital on boron followed by elimination of halogen.



Scheme 4

The hydride anion from sodium triethylborohydride displaced the chlorine atom from 5-chlorodibenzoborole (19) in boiling hexane, to give the parent compound (20) [63AG419]. 5-Chlorodibenzoborole (19) was treated with diethylamine in hexane at -78°C and then brought to room temperature and refluxed to give the diethylamino derivative (21). 5-Bromodibenzoborole (19) with bis(methylthio)lead gave the 5-methylthio compound (22) whereas the same starting material was converted to 5-methoxydibenzoborole (23) via methanolysis of the 2,6-lutidine adduct [85JOM(281)131]. The pyridine adduct (25) was obtained by the reaction of chloro compound (19) with pyridine, which was converted to the salt (26) by the addition of trichlorogallium, a strong chloride ion acceptor (Equation (3)). The acridine adduct (27) with AlCl_3 gave the salt (28) whose structure was proved by x-ray crystallography (Equation (4)) [85IC2532]. A transhalogenation reaction converts 5-bromodibenzoborole (19) to the corresponding 5-fluoro derivative (24) by treatment with potassium fluoride in acetonitrile [88JOM(340)267]. 5-Diethylaminodibenzoborole (29) reacted with two equivalents of pyrazole at room temperature by simultaneous displacement of diethylamine and complexation to form the complex (30) (Equation (5)) [90IC3845]. Benzylideneamino derivatives (31) have been prepared from diphenylmethyleneamino-trimethylsilane and (24) (Equation (6)) [70JCS(A)2010]. Dibenzoborol-5-yl(di-*t*-butylboryl)methyl-

amine (32) was obtained by reacting 5-chlorodibenzoborole (19) with $\text{Bu}^t_2\text{BNMeLi}$ in pentane at -69°C (Equation (7)) <86JOM(310)1>.

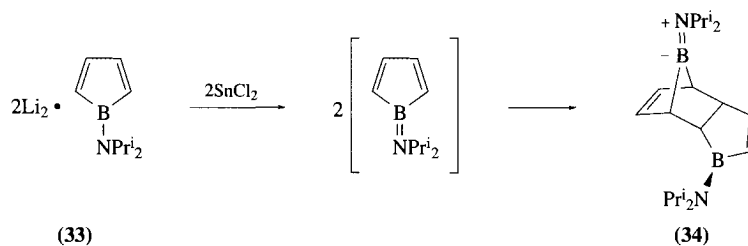


Compounds containing stable covalent metal-boron bonds have also been synthesized <66ZAAC(345)69>.

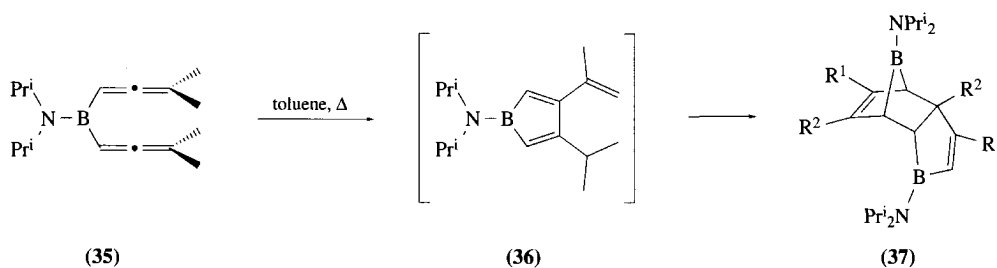
2.18.5.2 Cyclic Transition State Reactions

Dilithium 1-(diisopropylamino)borolenediide (33) upon oxidation with SnCl_2 forms the Diels-Alder dimer (34) (Scheme 5) <85CB4303>. The Diels-Alder reaction between pentaphenylborole (5) and diphenylalkyne to give the borepin ring system, described in CHEC-I, was further investigated <90OM2342>. Another Diels-Alder product (37) was obtained by heating in toluene (diisopropylamino)bis(3,3-dimethylallenyl)borane (35). The reaction proceeds via the nonisolable borole (36) which dimerizes (Scheme 6) <92CB1981>.

The high reactivity of 1-chlorobenzo[*b*]borole (39) did not allow its isolation when 1-chloro-1-boraindane (38) was dehydrogenated by flash vacuum pyrolysis at $900^\circ\text{C}/10^{-3}$ torr. Instead, the

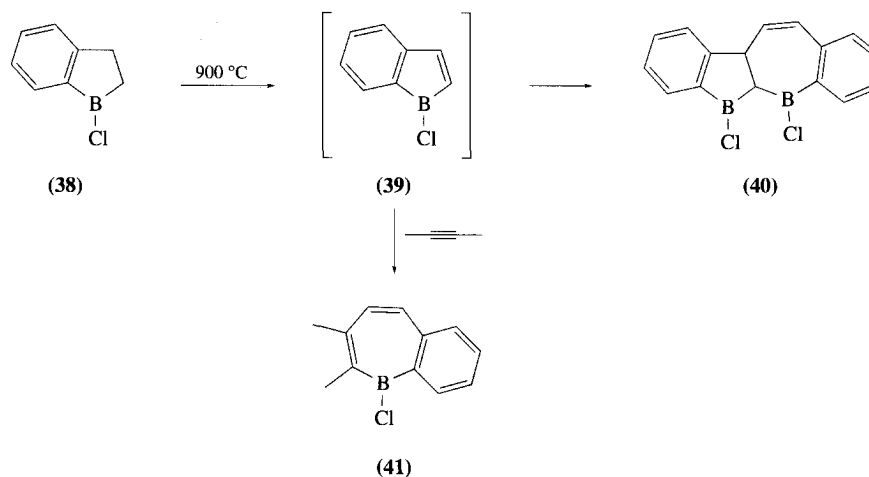


Scheme 5



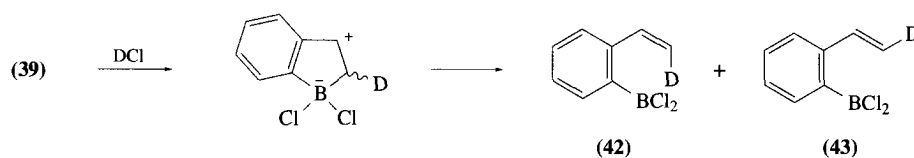
Scheme 6

benzoborole dimer, *cis*-11,12-dichloro-4*b*,11,11*a*,12-tetrahydro-11,12-diboradibenzo[*a,f*]azulene (40) was obtained in 56% yield. Formation of (40) can be explained in terms of a regioselective *syn*-vinylboration of a borole double bond with the $\text{B}-\text{C}_{\text{vinyl}}$ bond of a second molecule of (39) (Scheme 7).



Scheme 7

The existence of benzoborole (39) was further established by trapping with 2-butyne to give 1-chloro-2,3-dimethyl-1-benzoborepin (41) in 10% yield. Furthermore, trapping with electrophilic reagents such as DCl led to exclusive $\text{B}-\text{C}_{\text{vinyl}}$ bond cleavage and formation of a 1:1 mixture of *Z*- and *E*-(2-(β -deuteriovinyl)phenylboranes (42) and (43), respectively (Scheme 8) <87AG(E)665>.



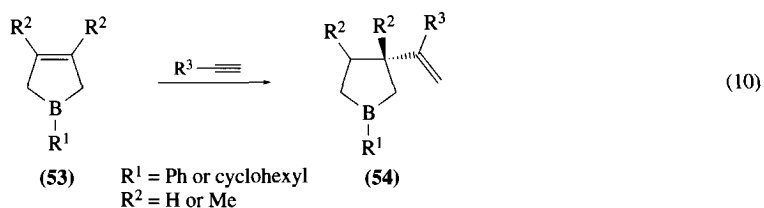
Scheme 8

Dilithium 1-(dialkylamino)dihydroborole diides (**44**) reacted with $\text{NiCl}_2 \cdot \text{DME}$ to give bis(borole) nickel complexes (**45**) (Equation (8)) $\langle 91\text{CB}17 \rangle$. 5-Chlorodibenzoborole (**19**) has been used to form complexes with $(\text{diphos})_2\text{CoH}$ ($\text{diphos} = (\text{Ph})_2\text{PCH}_2\text{CH}_2\text{P}(\text{Ph})_2$) and with $\text{Ph}(\text{P}(\text{Me})_2)_2$ $\langle 67\text{CB}2899 \rangle$. A triphenylphosphine cobalt(III) complex of dibenzoborole has also been reported $\langle 68\text{CB}1205 \rangle$.

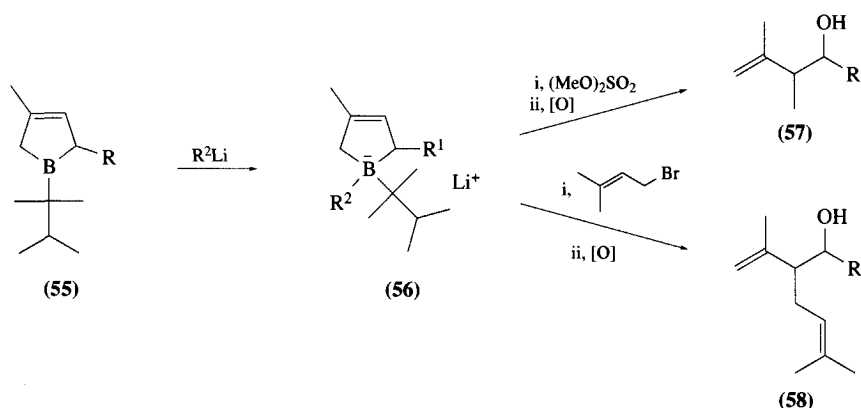


1-Substituted 2,3-dihydroboroles (2-borolenes) and 2,5-dihydroboroles (3-borolenes) react with a wide variety of transition metal complexes by dehydrogenating complexation leading to the formation of simple (η^5 -borole)metal complexes (83AG1024, 83JOM(246)141, 86JOM(304)271, 86JOM(308)153, 87JOM(319)311, 88JOM(350)81, 89JOM(362)259, 89AG328, 92CB2351, 94OM619). For example, 1-phenyl-3-borolene (**46**) reacts with $\text{Ru}_3(\text{CO})_{12}$ on heating to give the (borole)tricarbonylruthenium complex (**47**) in 35% yield. Reaction with $\text{Ru}(\text{C}_6\text{H}_6)(\text{C}_6\text{H}_8)$ in liquid benzene at 160°C leads to formation of complex (**48**) as the main product. Compound (**46**) also reacts with $\text{Mn}_2(\text{CO})_{10}$ in boiling mesitylene to afford the triple-decked complex (**49**) in 15% yield. Dehydrogenating complexation with the catalytically highly active $((\text{C}_2\text{H}_4)_2\text{RhCl})_2$ furnishes the triple decked complex (**50**) (Scheme 9).


$$\begin{array}{ccc}
 \begin{array}{c} \text{Cyclopentadienyl ring} \\ | \\ \text{B} \\ | \\ \text{R} \end{array} & \xrightarrow{[(\text{C}_2\text{H}_4)_2\text{RhCl}]_2} & \begin{array}{c} \text{Cyclopentadienyl ring} \\ | \\ \text{B} \\ | \\ \text{R} \end{array} \\
 \text{(51)} & \text{R = Ph, NMe}_2, \text{NEt}_2, \text{NPr}^i_2 & \text{(52)}
 \end{array} \quad (9)$$

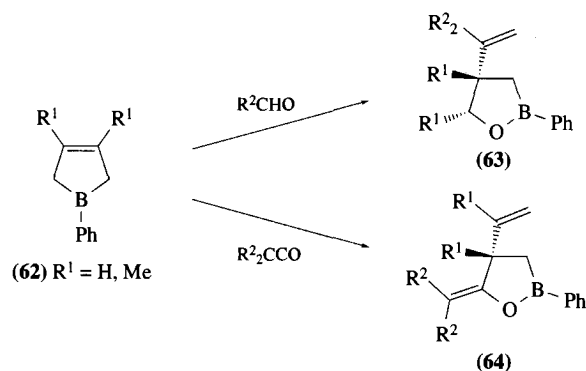
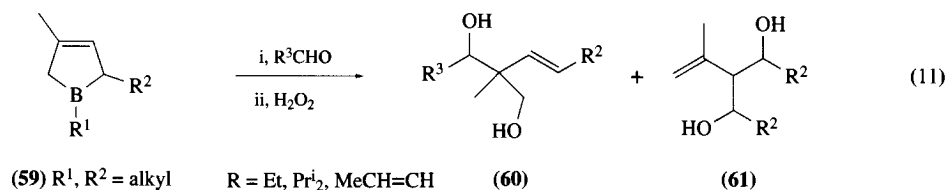


The 3-borolene *ate* complexes (56) are derived from 3-borolene (55) and an alkyllithium compound. They react with dimethyl sulfate or 1-bromo-3-methyl-2-butene in a regio- and diastereo-selective manner to furnish, after oxidative work-up, the corresponding substituted homoallylic alcohols (57) and (58) (Scheme 10) <87JOC5484>.



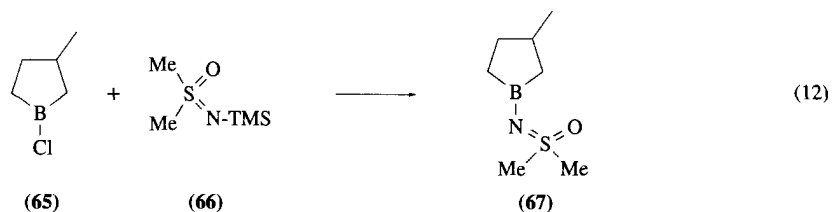
Scheme 10

1-Alkyl-3-borolenes (59) when treated with aldehydes followed by oxidation of the resultant organoboron intermediate with alkaline hydrogen peroxide, provided two regioisomeric diols (60) and (61) (Equation (11)). The ratio of the products depends on the substituents on the borolene and the type of aldehyde used <88JA5578>. Without the oxidation step 1-phenyl-3-borolenes (62) react with aldehydes to give *cis*-1,2-oxaborolanes (63) with stereoselectivity between 97 and 100%. If, instead of aldehydes, ketenes are used in the previous reaction, 5-alkylidene-4-vinyl-1,2-oxaborolanes (64) are formed, which tend to undergo double-bond migration (Scheme 11) <93CB297>.

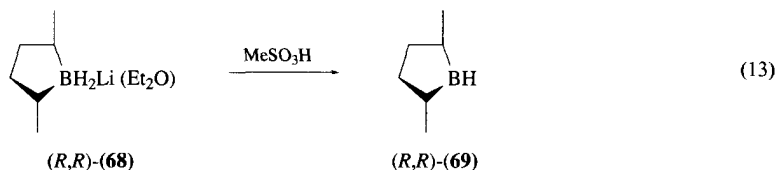


Scheme 11

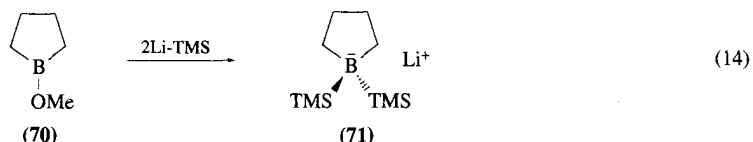
1-Chloro-3-methylborolane (65) has been used in the borylation of sulfonimine (66) to give monoborylated sulfonimine (67) (Equation (12)) <89ZN47>.



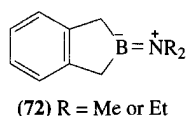
(*R,R*)-2,5-Dimethylborolane (**69**) generated *in situ* by treating dihydroborate (**68**) with methanesulfonic acid asymmetrically reduced ketones to alcohols (Equation (13)) <86JA7402, 86JA7404>.



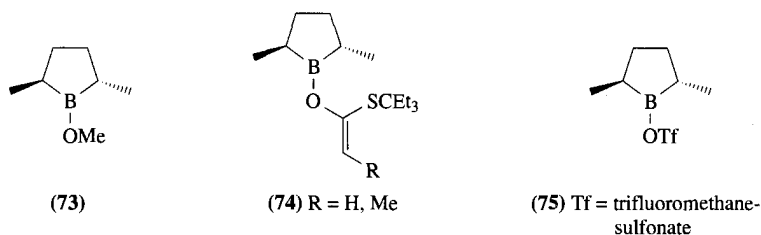
Reaction of 1-methoxyborolane (**70**) with trimethylsilyllithium in hexane provides the lithium bis(trimethylsilyl)borate (**71**) (Equation (14)) <82CB934>.



2-Dialkylamino-1,3-dihydro-2-benzoboroles (**72**) upon treatment with LiTMP/TMEDA in THF yielded the dilithio derivatives $(\text{Li}(\text{TMEDA})_2 \cdot \text{C}_6\text{H}_4(\text{CH}_2)\text{BNR}_2)$ in 40% yield which showed triple-decked structures consisting of an aromatic 10 π -electron 2-benzoborole dianion and two Li (TMEDA)⁺ units facially bound to the borole ring <93CB1397>.



Chiral boron enolate reagents (only *R,R*-configuration shown) such as (**73**) <87JOC4831> (**74**) <89JOC2817, 89TL7357, 92T4183>, and (**75**) <86JA8279, 87TL2841, 90TL3863> are capable of achieving high diastereoselection as well as enantioselection in their reactions with chiral aldehydes.



2.18.7 RING SYNTHESIS

2.18.7.1 Introduction

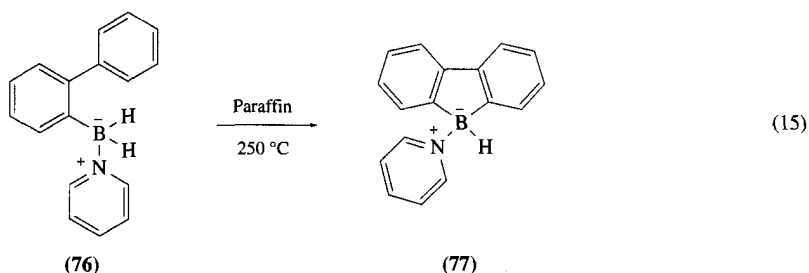
The high reactivity of boroles has made their synthesis very difficult and, as a consequence, the study of their chemical and physical properties. The 4 π -electrons of borole interact with the empty 2 p_z -orbital on boron causing destabilization. Benzo[*b*]boroles are relatively unknown, the alkenyl

2π -electrons also interact with the empty $2p_z$ -orbital on boron causing destabilization. In dibenzoboroles the 12π -electron system of the two benzene rings does not interact with the empty boron $2p_z$ -orbital and these compounds are quite stable with high melting points.

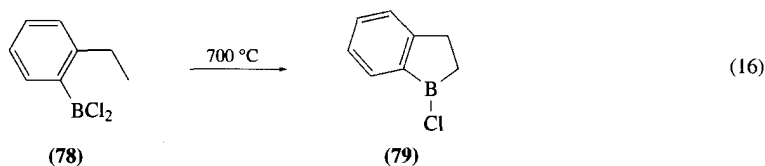
2.18.7.2 Formation of One Bond

1-Phenyl-3-borolene (**62**; $R^1 = H$) can be synthesized in 58% yield by reacting butendiylbis(tetrahydrofuran)magnesium and dichlorophenylborane at -80°C in diethylether <82JOM(233)35>.

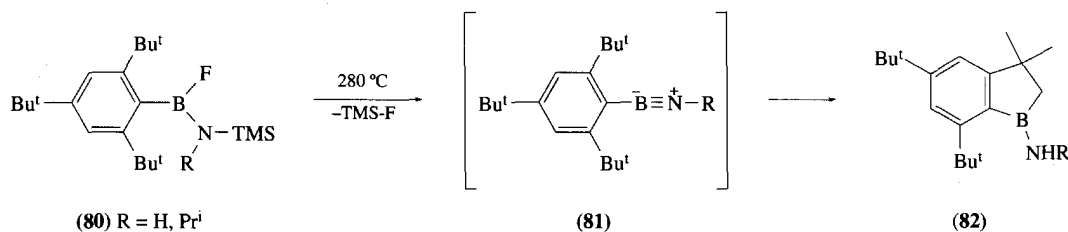
The synthesis of the first simple dibenzoborole was as the pyridine adduct (**77**), obtained by the pyrolysis of pyridine-2-biphenylborane (**76**) in paraffin at 250°C (Equation (15)) <73JOM(47)33>.



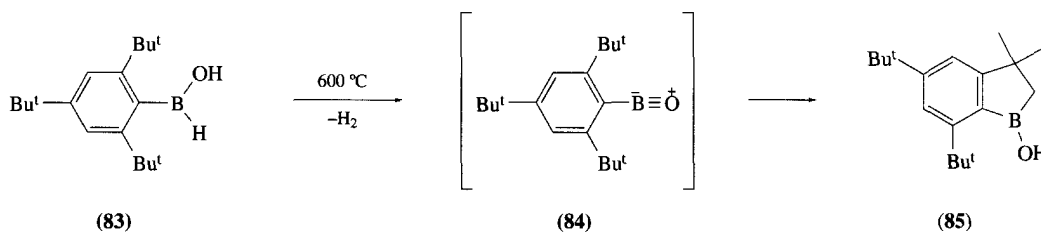
Simple benzo[*b*]borolanes (or boraindanes) such as the 1-chloro derivative (**79**) have been synthesised by flash vacuum pyrolysis of dichloro(2-ethylphenyl)borane (**78**) (Equation (16)) <87AG(E)665, 87CB1331>.



Thermal elimination of fluorotrimethylsilane from the 2,4,6-tri-*t*-butylphenyl fluoro-[(trimethylsilyl)amino]boranes (**80**) gives intermediates (**81**) which rearrange to the substituted benzo[*b*]borolanes (**82**) (Scheme 12) <90JOM(381)299, 93ZAAC(619)1474>. A similar type of intermediate (**84**) is formed when borane (**83**) is dehydrogenated to the 1-hydroxy derivative (**85**) in the gas phase at 600°C (Scheme 13) <88CB809>.



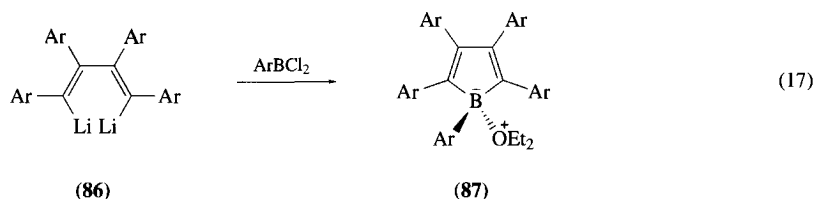
Scheme 12



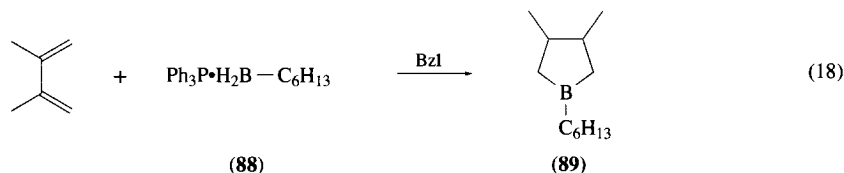
Scheme 13

2.18.7.3 Formation of Two Bonds

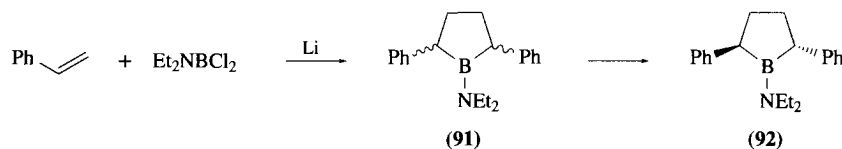
The only practical synthesis of the highly reactive borole ring is based upon (*E,E*)-(1,2,3,4-tetraaryl-1,3-butadiene-1,4-ylidene)lithium (**86**) which is treated with an aryl (dihalo)borane in ether to form the relatively stable pentaarylboroles (**87**) (Equation (17)) <86JA379>.



One approach to the borolane ring is by hydroboration of an appropriate butadiene. The triphenylphosphine-*n*-hexylborane adduct (**88**) reacted with 2,3-dimethylbutadiene in the presence of benzyl iodide to give the borolane derivative (**89**) (Equation (18)) <81AG1098>. Hydroboration of 1-(trimethylsilyl)-1,3-butadiene with $\text{BH}_3 \cdot \text{SMe}_2$ in ether, methanolysis, and thermal isomerization, in one pot, provided racemic B-methoxy-2-(trimethylsilyl)borolane (**90**) (Equation (19)) <89JA1892>.

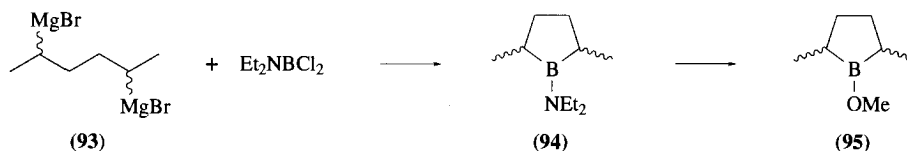


Reductive dimerization of styrene in the presence of (diethylamino)dichloroborane gave a mixture of *cis* and *trans* borolane (**91**). Crystallization afforded a 45% yield of (**92**) in >99% purity (one enantiomer shown) (Scheme 14) <86TL4721>.



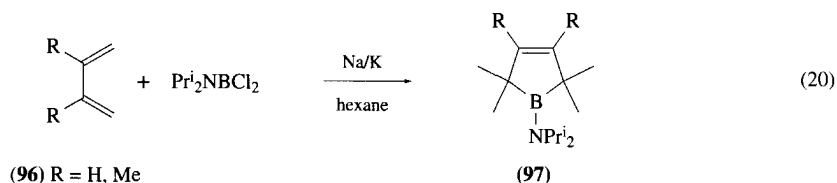
Scheme 14

Reaction of (diethylamino)dichloroborane with the Grignard reagent (**93**) gave a mixture of *cis* and *trans* 2,5-dimethylborolanes (**94**) which were converted with ethereal HCl and methanol into the corresponding methoxyl derivatives (**95**) in 82–86% yield (*cis/trans* ratio 47:53) (Scheme 15) <85JA4549, 87USP4644075>.

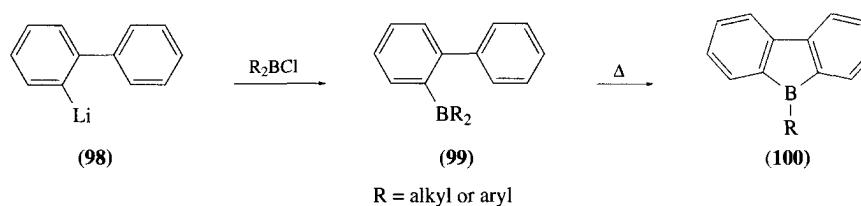


Scheme 15

The 3-borolenes (**97**) were among the several products obtained by the reaction of dichloro-(diisopropylamino)borane with the corresponding 1,3-dienes (**96**) in the presence of Na/K alloy (Equation (20)) <90JOM(386)1>. Reaction of alkenylboranes with alkynyllead compounds is another approach <91ZN1207>.



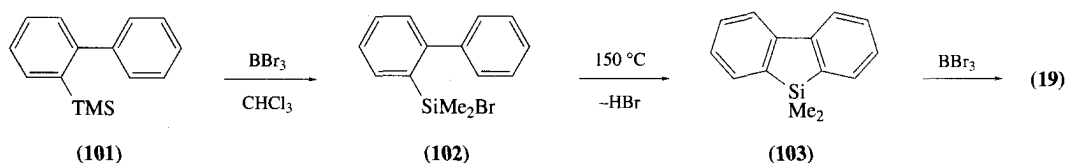
The general approach for the synthesis of dibenzoboroles is ring closure of appropriate biphenyls with boron compounds. The earliest practical syntheses of 5-alkyl or aryl dibenzoboroles (**100**) required reaction of *o*-lithiumbiphenyl (**98**) with a dialkyl or diarylchloroborane to give 2-biphenyl-yl(disubstituted)boranes (**99**). The latter were then thermally cyclized to the 5-substituted dibenzoboroles (**100**) (Scheme 16) <63AG1079, 67LA(702)197>.



Scheme 16

A one step synthesis of 5-halodibenzoboroles (**19**) involves reaction of 2,2'-biphenylmercury with the corresponding boron trihalides <85JOM(281)131>.

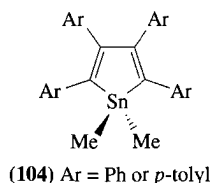
In 1987 silylated biphenyl (**101**) was reacted with tribromoborane to suffer methyl abstraction and give the bromodimethylsilyl derivative (**102**) <87CB991>. Heating (**102**) at 150 °C causes cyclization to 5-dimethylsiladibenzoborole (**103**) (Scheme 17). Further reaction of (**103**) with tribromoborane yields 5-bromodibenzoborole (**19**).



Scheme 17

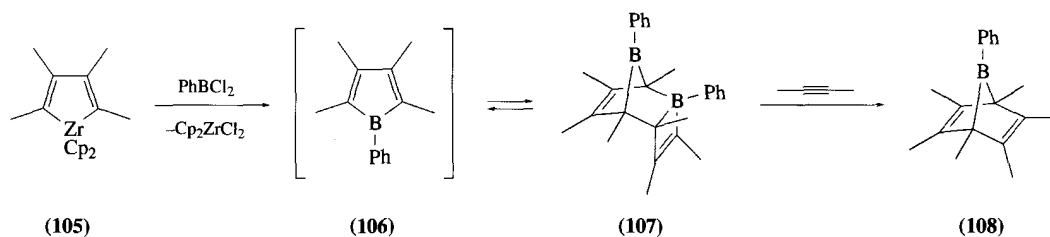
2.18.7.4 Transformation of Existing Heterocycles

The replacement of tin by boron in 1,1-dimethyl-2,3,4,5-tetraarylstannoles (**104**) by reaction with aryl(dichloro)boranes provides pentaarylboroles (**5**) or (**6**). Stannoles (**104**) are obtained from the lithium reagent (**86**) by the addition of stoichiometric amount of dichloro(dimethyl)tin <86JA379>.



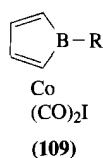
In an analogous reaction, the zirconium metallacycle (**105**) reacts with dichlorophenylborane to give the intermediate borole (**106**) which dimerizes to the stable borole dimer (**107**). Although direct detection of (**106**) was not possible, heating the dimer (**107**) at 60 °C with 2-butyne in toluene

produced the Diels–Alder adduct (**108**) in 76% yield (Scheme 18). This proves that borole (**106**) exists as an intermediate in these transformations <88JA2979>.

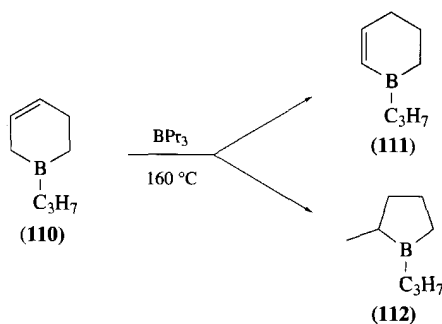


Scheme 18

Borole ammonia adducts (**4**) have been prepared by ammonia degradation of the (η^5 -borole)carbonyliodocobalt complexes (**109**) <87JOM(336)29>.



Heating 1-propyl-3-borinene (**110**) with tripropylborane in a sealed tube at 160 °C resulted in an equimolar mixture of 1-propyl-2-borinene (**111**) and 1-propyl-2-methyl-2-borolene (**112**). These compounds were not isolated but detected in solution by NMR spectroscopy (Scheme 19) <92OM4056>.



Scheme 19

References

EXPLANATION OF THE REFERENCE SYSTEM

Throughout this work, references are designated by a number–lettering coding of which the first two numbers denote tens and units of the year of publication, the next one to three letters denote the journal, and the final numbers denote the page. This code appears in the text each time a reference is quoted; the advantages of this system are outlined in the Introduction. The system has been used previously in the first edition of *Comprehensive Heterocyclic Chemistry*, eds A. R. Katritzky and C. W. Rees, Pergamon, Oxford, 1984 and is based on that used in the following two monographs: (a) A. R. Katritzky and J. M. Lagowski, "Chemistry of the Heterocyclic N-Oxides," Academic Press, New York, 1971; (b) J. Elguero, C. Marzin, A. R. Katritzky and P. Linda, "The Tautomerism of Heterocycles," in "Advances in Heterocyclic Chemistry," Supplement 1, Academic Press, New York, 1976.

The following additional notes apply:

1. A list of journal codes in alphabetical order, together with the journals to which they refer, is given immediately following these notes. Journal names are abbreviated throughout using the CASSI (Chemical Abstracts Service Source Index) system.
2. Each volume contains all the references cited *in that volume*; no separate lists are given for individual chapters.
3. The list of references is arranged in order of (a) year, (b) journal in alphabetical order of journal code, (c) part letter or number if relevant, (d) volume number if relevant, (e) page number.
4. In the reference list the code is followed by (a) the complete literature citation in the conventional manner and (b) the number(s) of the page(s) on which the reference appears, whether in the text or in tables, schemes, etc.
5. For nontwentieth-century references the year is given in full in the code.
6. For journals which are published in separate parts, the part letter or number is given (when necessary) in parentheses immediately after the journal code letters.
7. Journal volume numbers are *not* included in the code numbers unless more than one volume was published in the year in question, in which case the volume number is included in parentheses immediately after the journal code letters.
8. Patents are assigned appropriate three-letter codes.
9. Frequently cited books are assigned codes.
10. Less common journals and books are given the code "MI" for miscellaneous with the whole code for books prefixed by the letter "B-".
11. Where journals have changed names, the same code is used throughout, e.g. CB refers to both *Chem. Ber.* and to *Ber. Dtsch. Chem. Ges.*

Journal Codes

AAC	Antimicrob. Agents Chemother.
ABC	Agric. Biol. Chem.
AC	Appl. Catal.
AC(P)	Ann. Chim. (Paris)
AC(R)	Ann. Chim. (Rome)
ACH	Acta Chim. Acad. Sci. Hung.

ACR	Acc. Chem. Res.
ACS	Acta Chem. Scand.
ACS(A)	Acta Chem. Scand., Ser. A
ACS(B)	Acta Chem. Scand., Ser. B
AF	Arzneim.-Forsch.
AFC	Adv. Fluorine Chem.
AG	Angew. Chem.
AG(E)	Angew. Chem., Int. Ed. Engl.
AHC	Adv. Heterocycl. Chem.
AHCS	Adv. Heterocycl. Chem. Supplement
AI	Anal. Instrum.
AJC	Aust. J. Chem.
AK	Ark. Kemi
AKZ	Arm. Khim. Zh.
AM	Adv. Mater. (Weinheim, Ger.)
AMLS	Adv. Mol. Spectrosc.
AMS	Adv. Mass. Spectrom.
ANC	Anal. Chem.
ANL	Accad. Naz. Lincei
ANY	Ann. N. Y. Acad. Sci.
AOC	Adv. Organomet. Chem.
AP	Arch. Pharm. (Weinheim, Ger.)
APO	Adv. Phys. Org. Chem.
AQ	An. Quim.
AR	Annu. Rep. Prog. Chem.
AR(A)	Annu. Rep. Prog. Chem., Sect. A
AR(B)	Annu. Rep. Prog. Chem., Sect. B
ARP	Annu. Rev. Phys. Chem.
ASI	Acta Chim. Sin. Engl. Ed.
ASIN	Acta Chim. Sin.
AX	Acta Crystallogr.
AX(A)	Acta Crystallogr., Part A
AX(B)	Acta Crystallogr., Part B
B	Biochemistry
BAP	Bull. Acad. Pol. Sci., Ser. Sci. Chim.
BAU	Bull. Acad. Sci. USSR, Div. Chim. Sci.
BBA	Biochim. Biophys. Acta
BBR	Biochim. Biophys. Res. Commun.
BCJ	Bull. Chem. Soc. Jpn.
BEP	Belg. Pat.
BJ	Biochem. J.
BJP	Br. J. Pharmacol.
BMC	Bioorg. Med. Chem. Lett.
BP	Biochem. Biopharmacol.
BPJ	Br. Polym. J.
BRP	Br. Pat.
BSB	Bull. Soc. Chim. Belg.
BSF	Bull. Soc. Chim. Fr.
BSF(2)	Bull. Soc. Chim. Fr., Part 2
C	Chimia
CA	Chem. Abstr.
CAN	Cancer
CAR	Carbohydr. Res.
CAT	Chim. Acta Turc.
CB	Chem. Ber.

CBR	Chem. Br.
CC	J. Chem. Soc., Chem. Commun.
CCA	Croat. Chem. Acta
CCC	Collect. Czech. Chem. Commun.
CCR	Coord. Chem. Rev.
CE	Chem. Express
CEN	Chem. Eng. News
CHE	Chem. Heterocycl. Compd. (Engl. Transl.)
CHEC-I	Comp. Heterocycl. Chem., 1st edn.
CI(L)	Chem. Ind. (London)
CI(M)	Chem. Ind. (Milan)
CJC	Can. J. Chem.
CJS	Can. J. Spectrosc.
CL	Chem. Lett.
CLY	Chem. Listy
CM	Chem. Mater.
CMC	Comp. Med. Chem.
COC	Comp. Org. Chem.
COMC-I	Comp. Organomet. Chem., 1st edn.
COS	Comp. Org. Synth.
CP	Can. Pat.
CPB	Chem. Pharm. Bull.
CPH	Chem. Phys.
CPL	Chem. Phys. Lett.
CR	C. R. Hebd. Seances Acad. Sci.
CR(A)	C. R. Hebd. Seances Acad. Sci., Ser. A
CR(B)	C. R. Hebd. Seances Acad. Sci., Ser. B
CR(C)	C. R. Hebd. Seances Acad. Sci., Ser. C
CRAC	Crit. Rev. Anal. Chem.
CRV	Chem. Rev.
CS	Chem. Scr.
CSC	Cryst. Struct. Commun.
CSR	Chem. Soc. Rev.
CT	Chem. Tech.
CZ	Chem.-Ztg.
CZP	Czech. Pat.
DIS	Diss. Abstr.
DIS(B)	Diss. Abstr. Int. B.
DOK	Dokl. Akad. Nauk SSSR
DP	Dyes Pigm.
E	Experientia
EC	Educ. Chem.
EF	Energy Fuels
EGP	Ger. (East) Pat.
EJM	Eur. J. Med. Chem.
EUP	Eur. Pat.
FCF	Forstchr. Chem. Forsch.
FCR	Fluorine Chem. Rev.
FES	Farmaco Ed. Sci.
FOR	Fortschr. Chem. Org. Naturst.
FRP	Fr. Pat.
G	Gazz. Chim. Ital.
GAK	Gummi Asbest Kunstst.
GEP	Ger. Pat.
GEP(O)	Ger. Pat. Offen.

GSM	Gen. Synth. Methods
H	Heterocycles
HAC	Heteroatom Chem.
HC	Chem. Heterocycl. Compd.
HCA	Helv. Chim. Acta
HOU	Methoden Org. Chem. (Houben-Weyl)
HP	Hydrocarbon Process
IC	Inorg. Chem.
ICA	Inorg. Chim. Acta
IEC	Ind. Eng. Chem. Res.
IJ	Isr. J. Chem.
IJC	Indian J. Chem.
IJC(A)	Indian J. Chem., Sect. A
IJC(B)	Indian J. Chem., Sect. B
IJM	Int. J. Mass Spectrom. Ion Phys.
IJQ	Int. J. Quantum Chem.
IJS	Int. J. Sulfur Chem.
IJS(A)	Int. J. Sulfur Chem., Part A
IJS(B)	Int. J. Sulfur Chem., Part B
IS	Inorg. Synth.
IZV	Izv. Akad. Nauk SSSR, Ser. Khim.
JA	J. Am. Chem. Soc.
JAN	J. Antibiot.
JAP	Jpn. Pat.
JAP(K)	Jpn. Kokai
JBC	J. Biol. Chem.
JC	J. Chromatogr.
JCC	J. Coord. Chem.
JCE	J. Chem. Ed.
JCED	J. Chem. Eng. Data
JCI	J. Chem. Inf. Comput. Sci.
JCP	J. Chem. Phys.
JCPB	J. Chim. Phys. Physico-Chim. Biol.
JCR(M)	J. Chem. Res. (M)
JCR(S)	J. Chem. Res. (S)
JCS	J. Chem. Soc.
JCS(A)	J. Chem. Soc. (A)
JCS(B)	J. Chem. Soc. (B)
JCS(C)	J. Chem. Soc. (C)
JCS(D)	J. Chem. Soc., Dalton Trans.
JCS(F1)	J. Chem. Soc., Faraday Trans. 1
JCS(F2)	J. Chem. Soc., Faraday Trans. 2
JCS(P1)	J. Chem. Soc., Perkin Trans. 1
JCS(P2)	J. Chem. Soc., Perkin Trans. 2
JCS(S2)	J. Chem. Soc. (Suppl. 2)
JEC	J. Electroanal. Chem. Interfacial Electrochem.
JEM	J. Energ. Mater.
JES	J. Electron. Spectrosc.
JFA	J. Sci. Food. Agri.
JFC	J. Fluorine Chem.
JGU	J. Gen. Chem. USSR (Engl. Transl.)
JHC	J. Heterocycl. Chem.
JIC	J. Indian Chem. Soc.
JINC	J. Inorg. Nucl. Chem.
JLC	J. Liq. Chromatogr.

JMAS	J. Mat. Sci.
JMC	J. Med. Chem.
JMOC	J. Mol. Catal.
JMR	J. Magn. Reson.
JMS	J. Mol. Sci.
JOC	J. Org. Chem.
JOM	J. Organomet. Chem.
JOU	J. Org. Chem. USSR (Engl. Transl.)
JPC	J. Phys. Chem.
JPJ	J. Pharm. Soc. Jpn.
JPO	J. Phys. Org. Chem.
JPP	J. Pharm. Pharmacol.
JPR	J. Prakt. Chem.
JPS	J. Pharm. Sci.
JPS(A)	J. Polym. Sci., Polym. Chem., Part A
JPU	J. Phys. Chem. USSR (Engl. Transl.)
JSC	J. Serbochem. Soc.
JSP	J. Mol. Spectrosc.
JST	J. Mol. Struct.
K	Kristallografiya
KFZ	Khim. Farm. Zh.
KGS	Khim. Geterotsikl. Soedin.
KO	Kirk-Othmer Encyc.
KPS	Khim. Prir. Soedin.
L	Langmuir
LA	Liebigs Ann. Chem.
LC	Liq. Cryst.
LS	Life Sci.
M	Monatsh. Chem.
MAC	Macromol. Chem.
MC	Mendeleev Chem. J. (Engl. Transl.)
MCLC	Mol. Cryst. Liq. Cryst.
MI	Miscellaneous [book/journal]
MIP	Miscellaneous Pat.
MM	Macromolecules
MP	Mol. Phys.
MRC	Magn. Reson. Chem.
N	Naturwissenschaften
NAT	Nature
NEP	Neth. Pat.
NJC	Nouv. J. Chim.
NKK	Nippon Kagaku Kaishi (J. Chem. Soc. Jpn.)
NKZ	Nippon Kagaku Zasshi
NZJ	N. Z. J. Sci. Technol.
OCS	Organomet. Synth.
OM	Organometallics
OMR	Org. Magn. Reson.
OMS	Org. Mass Spectrom.
OPP	Org. Prep. Proced. Int.
OR	Org. React.
OS	Org. Synth.
OSC	Org. Synth., Coll. Vol.
P	Phytochemistry
PA	Polym. Age
PAC	Pure Appl. Chem.

PAS	Pol. Acad. Sci.
PB	Polym. Bull.
PC	Personal Communication
PCS	Proc. Chem. Soc.
PHA	Pharmazi
PHC	Prog. Heterocycl. Chem.
PIA	Proc. Indian Acad. Sci.
PIA(A)	Proc. Indian Acad. Sci., Sect. A
PJC	Pol. J. Chem.
PJS	Pak. J. Sci. Ind. Res.
PMH	Phys. Methods Heterocycl. Chem.
PNA	Proc. Natl. Acad. Sci. USA
POL	Polyhedron
PP	Polym. Prepr.
PRS	Proceed. Roy. Soc.
PS	Phosphorus Sulfur
QR	Q. Rev., Chem. Soc.
QRS	Quart. Rep. Sulfur. Chem.
QSAR	Quant. Struct. Act. Relat. Pharmacol. Chem. Biol.
RC	Rubber Chem. Technol.
RCM	Rapid Commun. Mass Spectrom.
RCP	Rec. Chem. Prog.
RCR	Russ. Chem. Rev. (Engl. Transl.)
RHA	Rev. Heteroatom Chem.
RJ	Rubber J.
RP	Rev. Polarogr.
RRC	Rev. Roum. Chim.
RS	Ric. Sci.
RTC	Recl. Trav. Chim. Pays-Bas
RZC	Rocz. Chem.
S	Synthesis
SA	Spectrochim. Acta
SA(A)	Spectrochim. Acta, Part A
SAP	S. Afr. Pat.
SC	Synth. Commun.
SCI	Science
SL	Synlett
SM	Synth. Met.
SR	Sulfur Reports
SRI	Synth. React. Inorg. Metal-Org. Chem.
SS	Sch. Sci. Rev.
SST	Org. Compd. Sulphur, Selenium, Tellurium [R. Soc. Chem. series]
SUL	Sulfur Letters
SZP	Swiss Pat.
T	Tetrahedron
T(S)	Tetrahedron, Suppl.
TA	Tetrahedron Asymmetry
TAL	Talanta
TCA	Theor. Chim. Acta
TCC	Top. Curr. Chem.
TCM	Tetrahedron, Comp. Method.
TFS	Trans. Faraday Soc.
TH	Thesis
TL	Tetrahedron Lett.
TS	Top. Stereochem.

UK	Usp. Khim.
UKZ	Ukr. Khim. Zh. (Russ. Ed.)
UP	Unpublished Results
URP	USSR Pat.
USP	US Pat.
WCH	Wiadom. Chem.
YGK	Yuki Gosei Kagaku Kyokaishi
YZ	Yakugaku Zasshi (J. Pharm. Soc. Jpn.)
ZAAC	Z. Anorg. Allg. Chem.
ZAK	Zh. Anal. Khim.
ZC	Z. Chem.
ZN	Z. Naturforsch.
ZN(A)	Z. Naturforsch., Teil A
ZN(B)	Z. Naturforsch., Teil B
ZOB	Zh. Obshch. Khim.
ZOR	Zh. Org. Khim.
ZPC	Hoppe-Seyler's Z. Physiol. Chem.
ZPK	Zh. Prikl. Khim.

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EXPLANATION OF THE REFERENCE SYSTEM

Throughout this work, references are designated by a number–lettering coding of which the first two numbers denote tens and units of the year of publication, the next one to three letters denote the journal, and the final numbers denote the page. This code appears in the text each time a reference is quoted; the advantages of this system are outlined in the Introduction. The system has been used previously in the first edition of *Comprehensive Heterocyclic Chemistry*, eds A. R. Katritzky and C. W. Rees, Pergamon, Oxford, 1984 and is based on that used in the following two monographs: (a) A. R. Katritzky and J. M. Lagowski, "Chemistry of the Heterocyclic N-Oxides," Academic Press, New York, 1971; (b) J. Elguero, C. Marzin, A. R. Katritzky and P. Linda, "The Tautomerism of Heterocycles," in "Advances in Heterocyclic Chemistry," Supplement 1, Academic Press, New York, 1976.

The following additional notes apply:

1. A list of journal codes in alphabetical order, together with the journals to which they refer, is given immediately following these notes. Journal names are abbreviated throughout using the CASSI (Chemical Abstracts Service Source Index) system.
2. Each volume contains all the references cited *in that volume*; no separate lists are given for individual chapters.
3. The list of references is arranged in order of (a) year, (b) journal in alphabetical order of journal code, (c) part letter or number if relevant, (d) volume number if relevant, (e) page number.
4. In the reference list the code is followed by (a) the complete literature citation in the conventional manner and (b) the number(s) of the page(s) on which the reference appears, whether in the text or in tables, schemes, etc.
5. For nontwentieth-century references the year is given in full in the code.
6. For journals which are published in separate parts, the part letter or number is given (when necessary) in parentheses immediately after the journal code letters.
7. Journal volume numbers are *not* included in the code numbers unless more than one volume was published in the year in question, in which case the volume number is included in parentheses immediately after the journal code letters.
8. Patents are assigned appropriate three-letter codes.
9. Frequently cited books are assigned codes.
10. Less common journals and books are given the code "MI" for miscellaneous with the whole code for books prefixed by the letter "B-".
11. Where journals have changed names, the same code is used throughout, e.g. CB refers to both *Chem. Ber.* and to *Ber. Dtsch. Chem. Ges.*

Journal Codes

AAC	Antimicrob. Agents Chemother.
ABC	Agric. Biol. Chem.
AC	Appl. Catal.
AC(P)	Ann. Chim. (Paris)
AC(R)	Ann. Chim. (Rome)
ACH	Acta Chim. Acad. Sci. Hung.

ACR	Acc. Chem. Res.
ACS	Acta Chem. Scand.
ACS(A)	Acta Chem. Scand., Ser. A
ACS(B)	Acta Chem. Scand., Ser. B
AF	Arzneim.-Forsch.
AFC	Adv. Fluorine Chem.
AG	Angew. Chem.
AG(E)	Angew. Chem., Int. Ed. Engl.
AHC	Adv. Heterocycl. Chem.
AHCS	Adv. Heterocycl. Chem. Supplement
AI	Anal. Instrum.
AJC	Aust. J. Chem.
AK	Ark. Kemi
AKZ	Arm. Khim. Zh.
AM	Adv. Mater. (Weinheim, Ger.)
AMLS	Adv. Mol. Spectrosc.
AMS	Adv. Mass. Spectrom.
ANC	Anal. Chem.
ANL	Accad. Naz. Lincei
ANY	Ann. N. Y. Acad. Sci.
AOC	Adv. Organomet. Chem.
AP	Arch. Pharm. (Weinheim, Ger.)
APO	Adv. Phys. Org. Chem.
AQ	An. Quim.
AR	Annu. Rep. Prog. Chem.
AR(A)	Annu. Rep. Prog. Chem., Sect. A
AR(B)	Annu. Rep. Prog. Chem., Sect. B
ARP	Annu. Rev. Phys. Chem.
ASI	Acta Chim. Sin. Engl. Ed.
ASIN	Acta Chim. Sin.
AX	Acta Crystallogr.
AX(A)	Acta Crystallogr., Part A
AX(B)	Acta Crystallogr., Part B
B	Biochemistry
BAP	Bull. Acad. Pol. Sci., Ser. Sci. Chim.
BAU	Bull. Acad. Sci. USSR, Div. Chim. Sci.
BBA	Biochim. Biophys. Acta
BBR	Biochim. Biophys. Res. Commun.
BCJ	Bull. Chem. Soc. Jpn.
BEP	Belg. Pat.
BJ	Biochem. J.
BJP	Br. J. Pharmacol.
BMC	Bioorg. Med. Chem. Lett.
BP	Biochem. Biopharmacol.
BPJ	Br. Polym. J.
BRP	Br. Pat.
BSB	Bull. Soc. Chim. Belg.
BSF	Bull. Soc. Chim. Fr.
BSF(2)	Bull. Soc. Chim. Fr., Part 2
C	Chimia
CA	Chem. Abstr.
CAN	Cancer
CAR	Carbohydr. Res.
CAT	Chim. Acta Turc.
CB	Chem. Ber.

CBR	Chem. Br.
CC	J. Chem. Soc., Chem. Commun.
CCA	Croat. Chem. Acta
CCC	Collect. Czech. Chem. Commun.
CCR	Coord. Chem. Rev.
CE	Chem. Express
CEN	Chem. Eng. News
CHE	Chem. Heterocycl. Compd. (Engl. Transl.)
CHEC-I	Comp. Heterocycl. Chem., 1st edn.
CI(L)	Chem. Ind. (London)
CI(M)	Chem. Ind. (Milan)
CJC	Can. J. Chem.
CJS	Can. J. Spectrosc.
CL	Chem. Lett.
CLY	Chem. Listy
CM	Chem. Mater.
CMC	Comp. Med. Chem.
COC	Comp. Org. Chem.
COMC-I	Comp. Organomet. Chem., 1st edn.
COS	Comp. Org. Synth.
CP	Can. Pat.
CPB	Chem. Pharm. Bull.
CPH	Chem. Phys.
CPL	Chem. Phys. Lett.
CR	C. R. Hebd. Seances Acad. Sci.
CR(A)	C. R. Hebd. Seances Acad. Sci., Ser. A
CR(B)	C. R. Hebd. Seances Acad. Sci., Ser. B
CR(C)	C. R. Hebd. Seances Acad. Sci., Ser. C
CRAC	Crit. Rev. Anal. Chem.
CRV	Chem. Rev.
CS	Chem. Scr.
CSC	Cryst. Struct. Commun.
CSR	Chem. Soc. Rev.
CT	Chem. Tech.
CZ	Chem.-Ztg.
CZP	Czech. Pat.
DIS	Diss. Abstr.
DIS(B)	Diss. Abstr. Int. B.
DOK	Dokl. Akad. Nauk SSSR
DP	Dyes Pigm.
E	Experientia
EC	Educ. Chem.
EF	Energy Fuels
EGP	Ger. (East) Pat.
EJM	Eur. J. Med. Chem.
EUP	Eur. Pat.
FCF	Forstchr. Chem. Forsch.
FCR	Fluorine Chem. Rev.
FES	Farmaco Ed. Sci.
FOR	Fortschr. Chem. Org. Naturst.
FRP	Fr. Pat.
G	Gazz. Chim. Ital.
GAK	Gummi Asbest Kunstst.
GEP	Ger. Pat.
GEP(O)	Ger. Pat. Offen.

GSM	Gen. Synth. Methods
H	Heterocycles
HAC	Heteroatom Chem.
HC	Chem. Heterocycl. Compd.
HCA	Helv. Chim. Acta
HOU	Methoden Org. Chem. (Houben-Weyl)
HP	Hydrocarbon Process
IC	Inorg. Chem.
ICA	Inorg. Chim. Acta
IEC	Ind. Eng. Chem. Res.
IJ	Isr. J. Chem.
IJC	Indian J. Chem.
IJC(A)	Indian J. Chem., Sect. A
IJC(B)	Indian J. Chem., Sect. B
IJM	Int. J. Mass Spectrom. Ion Phys.
IJQ	Int. J. Quantum Chem.
IJS	Int. J. Sulfur Chem.
IJS(A)	Int. J. Sulfur Chem., Part A
IJS(B)	Int. J. Sulfur Chem., Part B
IS	Inorg. Synth.
IZV	Izv. Akad. Nauk SSSR, Ser. Khim.
JA	J. Am. Chem. Soc.
JAN	J. Antibiot.
JAP	Jpn. Pat.
JAP(K)	Jpn. Kokai
JBC	J. Biol. Chem.
JC	J. Chromatogr.
JCC	J. Coord. Chem.
JCE	J. Chem. Ed.
JCED	J. Chem. Eng. Data
JCI	J. Chem. Inf. Comput. Sci.
JCP	J. Chem. Phys.
JCPB	J. Chim. Phys. Physico-Chim. Biol.
JCR(M)	J. Chem. Res. (M)
JCR(S)	J. Chem. Res. (S)
JCS	J. Chem. Soc.
JCS(A)	J. Chem. Soc. (A)
JCS(B)	J. Chem. Soc. (B)
JCS(C)	J. Chem. Soc. (C)
JCS(D)	J. Chem. Soc., Dalton Trans.
JCS(F1)	J. Chem. Soc., Faraday Trans. 1
JCS(F2)	J. Chem. Soc., Faraday Trans. 2
JCS(P1)	J. Chem. Soc., Perkin Trans. 1
JCS(P2)	J. Chem. Soc., Perkin Trans. 2
JCS(S2)	J. Chem. Soc. (Suppl. 2)
JEC	J. Electroanal. Chem. Interfacial Electrochem.
JEM	J. Energ. Mater.
JES	J. Electron. Spectrosc.
JFA	J. Sci. Food. Agri.
JFC	J. Fluorine Chem.
JGU	J. Gen. Chem. USSR (Engl. Transl.)
JHC	J. Heterocycl. Chem.
JIC	J. Indian Chem. Soc.
JINC	J. Inorg. Nucl. Chem.
JLC	J. Liq. Chromatogr.

JMAS	J. Mat. Sci.
JMC	J. Med. Chem.
JMOC	J. Mol. Catal.
JMR	J. Magn. Reson.
JMS	J. Mol. Sci.
JOC	J. Org. Chem.
JOM	J. Organomet. Chem.
JOU	J. Org. Chem. USSR (Engl. Transl.)
JPC	J. Phys. Chem.
JPJ	J. Pharm. Soc. Jpn.
JPO	J. Phys. Org. Chem.
JPP	J. Pharm. Pharmacol.
JPR	J. Prakt. Chem.
JPS	J. Pharm. Sci.
JPS(A)	J. Polym. Sci., Polym. Chem., Part A
JPU	J. Phys. Chem. USSR (Engl. Transl.)
JSC	J. Serbochem. Soc.
JSP	J. Mol. Spectrosc.
JST	J. Mol. Struct.
K	Kristallografiya
KFZ	Khim. Farm. Zh.
KGS	Khim. Geterotsikl. Soedin.
KO	Kirk-Othmer Encyc.
KPS	Khim. Prir. Soedin.
L	Langmuir
LA	Liebigs Ann. Chem.
LC	Liq. Cryst.
LS	Life Sci.
M	Monatsh. Chem.
MAC	Macromol. Chem.
MC	Mendeleev Chem. J. (Engl. Transl.)
MCLC	Mol. Cryst. Liq. Cryst.
MI	Miscellaneous [book/journal]
MIP	Miscellaneous Pat.
MM	Macromolecules
MP	Mol. Phys.
MRC	Magn. Reson. Chem.
N	Naturwissenschaften
NAT	Nature
NEP	Neth. Pat.
NJC	Nouv. J. Chim.
NKK	Nippon Kagaku Kaishi (J. Chem. Soc. Jpn.)
NKZ	Nippon Kagaku Zasshi
NZJ	N. Z. J. Sci. Technol.
OCS	Organomet. Synth.
OM	Organometallics
OMR	Org. Magn. Reson.
OMS	Org. Mass Spectrom.
OPP	Org. Prep. Proced. Int.
OR	Org. React.
OS	Org. Synth.
OSC	Org. Synth., Coll. Vol.
P	Phytochemistry
PA	Polym. Age
PAC	Pure Appl. Chem.

PAS	Pol. Acad. Sci.
PB	Polym. Bull.
PC	Personal Communication
PCS	Proc. Chem. Soc.
PHA	Pharmazi
PHC	Prog. Heterocycl. Chem.
PIA	Proc. Indian Acad. Sci.
PIA(A)	Proc. Indian Acad. Sci., Sect. A
PJC	Pol. J. Chem.
PJS	Pak. J. Sci. Ind. Res.
PMH	Phys. Methods Heterocycl. Chem.
PNA	Proc. Natl. Acad. Sci. USA
POL	Polyhedron
PP	Polym. Prepr.
PRS	Proceed. Roy. Soc.
PS	Phosphorus Sulfur
QR	Q. Rev., Chem. Soc.
QRS	Quart. Rep. Sulfur. Chem.
QSAR	Quant. Struct. Act. Relat. Pharmacol. Chem. Biol.
RC	Rubber Chem. Technol.
RCM	Rapid Commun. Mass Spectrom.
RCP	Rec. Chem. Prog.
RCR	Russ. Chem. Rev. (Engl. Transl.)
RHA	Rev. Heteroatom Chem.
RJ	Rubber J.
RP	Rev. Polarogr.
RRC	Rev. Roum. Chim.
RS	Ric. Sci.
RTC	Recl. Trav. Chim. Pays-Bas
RZC	Rocz. Chem.
S	Synthesis
SA	Spectrochim. Acta
SA(A)	Spectrochim. Acta, Part A
SAP	S. Afr. Pat.
SC	Synth. Commun.
SCI	Science
SL	Synlett
SM	Synth. Met.
SR	Sulfur Reports
SRI	Synth. React. Inorg. Metal-Org. Chem.
SS	Sch. Sci. Rev.
SST	Org. Compd. Sulphur, Selenium, Tellurium [R. Soc. Chem. series]
SUL	Sulfur Letters
SZP	Swiss Pat.
T	Tetrahedron
T(S)	Tetrahedron, Suppl.
TA	Tetrahedron Asymmetry
TAL	Talanta
TCA	Theor. Chim. Acta
TCC	Top. Curr. Chem.
TCM	Tetrahedron, Comp. Method.
TFS	Trans. Faraday Soc.
TH	Thesis
TL	Tetrahedron Lett.
TS	Top. Stereochem.

UK	Usp. Khim.
UKZ	Ukr. Khim. Zh. (Russ. Ed.)
UP	Unpublished Results
URP	USSR Pat.
USP	US Pat.
WCH	Wiadom. Chem.
YGK	Yuki Gosei Kagaku Kyokaishi
YZ	Yakugaku Zasshi (J. Pharm. Soc. Jpn.)
ZAAC	Z. Anorg. Allg. Chem.
ZAK	Zh. Anal. Khim.
ZC	Z. Chem.
ZN	Z. Naturforsch.
ZN(A)	Z. Naturforsch., Teil A
ZN(B)	Z. Naturforsch., Teil B
ZOB	Zh. Obshch. Khim.
ZOR	Zh. Org. Khim.
ZPC	Hoppe-Seyler's Z. Physiol. Chem.
ZPK	Zh. Prikl. Khim.

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