# THE PYRIDAZINES

Supplement I

*This is the fifty-seventh volume in the series*

**THE CHEMISTRY OF HETEROCYCLIC COMPOUNDS**

THE CHEMISTRY OF HETEROCYCLIC COMPOUNDS

A SERIES OF MONOGRAPHS

**EDWARD C. TAYLOR AND PETER WIPF,** *Editors*

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# THE PYRIDAZINES

## Supplement I

**D. J. Brown**

Research School of Chemistry
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*To*
*an Octet of Great Pyridazine Chemists*

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# The Chemistry of Heterocyclic Compounds
## Introduction to the Series

The chemistry of heterocyclic compounds is one of the most complex and intriguing branches of organic chemistry, of equal interest for its theoretical implications, for the diversity of its synthetic procedures, and for the physiological and industrial significance of heterocycles.

*The Chemistry of Heterocyclic Compounds*, published since 1950 under the initial editorship of Arnold Weissberger, and later, until Dr. Weissberger's death in 1984, under our joint editorship, has attempted to make the extraordinarily complex and diverse field of heterocyclic chemistry as organized and readily accessible as possible. Each volume has traditionally dealt with syntheses, reactions, properties, structure, physical chemistry, and utility of compounds belonging to a specific ring system or class (e.g., pyridines, thiophenes, pyrimidines, three-membered ring systems). This series has become the basic reference collection for information on heterocyclic compounds.

Many broader aspects of heterocyclic chemistry are recognized as disciplines of general significance that impinge on almost all aspects of modern organic chemistry, medicinal chemistry, and biochemistry, and for this reason we initiated several years ago a parallel series entitled General Heterocyclic Chemistry, which treated such topics as nuclear magnetic resonance, mass spectra, and photochemistry of heterocyclic compounds, the utility of heterocycles in organic synthesis, and the synthesis of heterocycles by means of 1,3-dipolar cycloaddition reactions. These volumes were intended to be of interest of all organic, medicinal, and biochemically oriented chemists, as well as to those whose particular concern is heterocyclic chemistry. It has, however, become increasingly clear that the above distinction between the two series was unnecessary and somewhat confusing, and we have therefore elected to discontinue *General Heterocyclic Chemistry* and to publish all forthcoming volumes in this general area in *The Chemistry of Heterocyclic Compounds* series.

It is a major challenge to keep our coverage of this immense field up to date. One strategy is to publish Supplements or new Parts when merited by the amount of new material, as has been done, *inter alia*, with pyridines, purines, pyrimidines, quinazolines and isoxazoles. This is also the case with *Pyridazines*, which were last covered in this series in 1973. We acknowledge once again the extraordinary contributions of Dr. D. J. Brown, whose previous classics in heterocyclic chemistry in this series: (*The Pyrimidines, The Pyrimidines Supplement I, The Pyrimidines Supplement II, Pteridines, Quinazolines Supplement I*) are now joined by the present exhaustive treatment

of close to the last thirty years of pyridazine chemistry. We extend once again our congratulations and our thanks to Dr. Brown for a further outstanding contribution to the literature of heterocyclic chemistry.

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# Preface

The late Professor Raymond N. Castle and seven coauthors wrote the original volume, *Pyridazines*, that appeared within this series in 1973. Not only did it represent a comprehensive and digestible summary of pyridazine chemistry up to 1971, but it clearly stimulated considerable subsequent research in the area. Accordingly, the need for a supplementary volume to cover nearly three decades of more recent literature has become pressing.

In undertaking this task, the present author has found it necessary to inaugurate two substantial chapters on primary syntheses (covered piecemeal in the original volume) and to reorganize completely the content of remaining chapters in order to better reflect and emphasize current research trends. However, the essential status of the present volume as a *supplement* has been maintained by sectional cross-references (e.g., *H* 24) to pages in the original volume (*Hauptwerk*) where earlier relevant information may be found. Moreover, in view of the vast increase in the number and complexity of individual pyridazines described in recent literature, it has proved necessary to abandon the many classified tables of all known pyridazines in favor of a single alphabetical table of simple pyridazines. To facilitate recovery of any earlier data from tables in the original volume, cross-references (e.g., *H* 555) have been added to appropriate entries in the new table. General chemical nomenclature used in the supplement follows current IUPAC recommendations [*Nomenclature of Organic Chemistry, Sections A–F, H* (eds. J. Rigaudy and S. P. Klesney, Pergamon Press, Oxford, 1970)] with one important exception—in order to keep "pyridazine" as the principal part of each name, those groups that would normally qualify as principal suffixes, but that are not attached directly to the nucleus, are rendered as prefixes. For example, 4-carboxymethyl-3(2*H*)-pyridazinone is used instead of α-(3-oxo-2,3-dihydropyridazine-4-yl)acetic acid. Secondary or tertiary amino groups are rendered as prefixes. Ring systems are named according to Chemical Abstracts Service recommendations [*Ring Systems Handbook* (eds. anon, American Chemical Society, Columbus OH, 1988 and subsequent editions)]. Trivial names for pyridazines are included as appropriate in the table of simple pyridazines and/or in Section 5.7.4. In order to avoid repetition and inevitable confusion, literature references are presented in a single list rather than as separate lists at the end of each chapter. Finally, in preparing this supplement, the massive patent literature on pyridazines has been ignored in the belief that useful factual information therein has appeared subsequently in the regular literature.

I am greatly indebted to my former colleague, Dr G. B. Berlin, for invaluable discussions; to successive Deans of the Research School of Chemistry

(Professors J. W. White and D. J. Evans) for the provision of excellent postretirement accommodation and facilities within the School; to the branch Librarian, Mrs J. Smith, for unfailing help in library matters; and to my wife, Jan, for continual encouragement and her cheerful assistance during indexing and proofreading.

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DES J. BROWN

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# THE PYRIDAZINES

Supplement I

*This is the fifty-seventh volume in the series*

**THE CHEMISTRY OF HETEROCYCLIC COMPOUNDS**

*Chemistry of Heterocyclic Compounds, Volume 57*
D. J. Brown
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CHAPTER 1

# Primary Syntheses from Aliphatic or Carbocyclic Synthons

Primary synthetic routes to pyridazines and hydropyridazines from aliphatic or carbocyclic fragments are so numerous and diverse that it is impossible to devise a system of classification that is wholly satisfactory from all points of view. The pragmatic approach adopted here is based essentially on the ways in which the six ring atoms of pyridazine are supplied by the synthon(s) used in the various methods. Please be guided by the Contents table!

In each subsection, any examples of syntheses leading directly to "aromatic" pyridazines are usually given first, followed by those leading to hydropyridazines. Examples of any pre-1970 syntheses in each category may be found from the the cross-references (e.g., *H* 38) to appropriate pages in Castle's parent volume;[1839] some pre- and post-1970 material on primary synthetic routes has been reviewed elsewhere.[1188, 1575, 1623, 1624, 1626, 1643, 1853, 1916, 2006, 2011]

A brief section at the end of this chapter seeks to assist readers to locate suitable primary syntheses for a range of commonly needed types of pyridazine derivative.

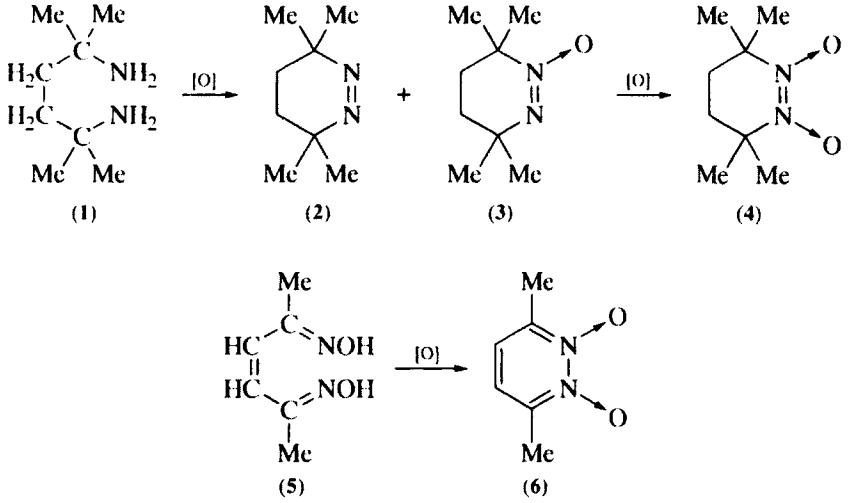
## 1.1 FROM A SINGLE SIX-ATOM SYNTHON

There is a surprisingly large number of such syntheses. They are classified according to the bond (of the final pyridazine) that remains unformed in the acyclic synthon. In most cases, such synthons have been isolated and usually characterized prior to cyclization.

### 1.1.1 By Completion of the N1–N2 Bond

As might be expected, cyclization by completion of the N–N bond is rare. However, *oxidative cyclization of 1,4-alkanediamines* has been used to make hydropyridazines and their *N*-oxides. Thus 2,5-dimethyl-2,5-hexanediamine (**1**) with hydrogen peroxide / sodium tungstate at ~25°* gave a separable 1 : 3 mixture of 3,3,6,6-tetramethyl-3,4,5,6-tetrahydropyridazine (**2**) and its 1-oxide

*All temperature values followed by degree symbol are Celsius (centigrade) unless otherwise indicated.




(**3**), the second of which was oxidized further by *m*-chloroperoxybenzoic acid in dichloromethane to afford the dioxide (**4**) in >70% yield;[307,308] sodium perborate gave an improved yield of the 1-oxide (**3**).[288]

Somewhat similarly, *oxidative cyclization of 1,4-alkenedione dioximes* afforded pyridazine 1,2-dioxides. For example, 2,5-hexenedione dioxime (**5**) with lead tetracetate in dichloromethane at 25° gave 3,6-dimethylpyridazine 1,2-dioxide (**6**) in 33% yield along with other separable products;[190,321] the use of [bis(trifluoroacetoxy)iodo]benzene as a dehydrogenation reagent gave less satisfactory results.[106,190,321,866]

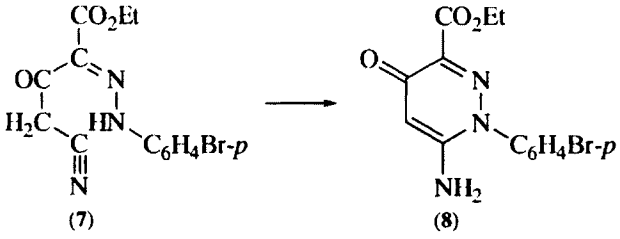
### 1.1.2 By Completion of the N2–C3 Bond

The cyclization of an N–N–C–C–C–C synthon, such as 4-hydrazonobutyric acid, has been used widely to prepare pyridazines and hydropyridazines. The examples are classified according to the substituent (or unsaturation) at the terminal carbon atom; the degree of unsaturation in the synthon naturally determines the degree of aromaticity in the product.

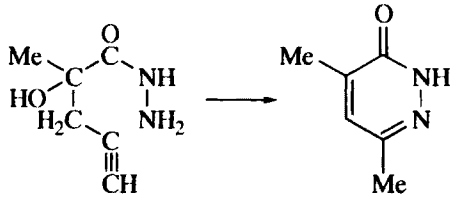
#### *1.1.2.1 From Hydrazonoalkenes or Related Synthons*

Such cyclizations are illustrated in the following examples:

Ethyl 2-*p*-bromophenylhydrazono-4-cyano-3-oxobutyrate (**7**) gave ethyl 6-amino-1-*p*-bromophenyl-4-oxo-1,4-dihydro-3-pyridazinecarboxylate (**8**) (AcOH, reflux, 6 h: 55%).[1304]



(7) (8)



(9) (10)

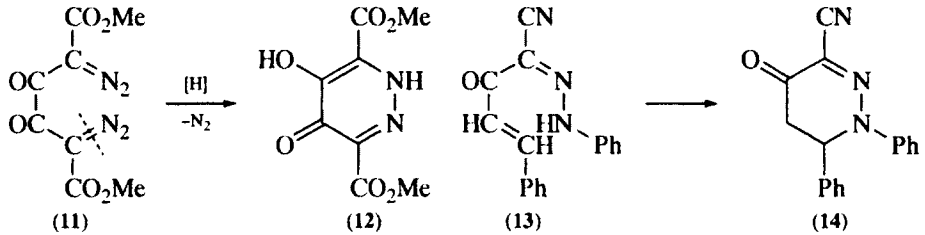
2-Hydroxy-2-methylpent-4-ynohydrazide (**9**) gave 4,6-dimethyl-3(2*H*)-pyridazinone (**10**) (2.5M HCl, reflux, 24 h: 13%, as hydrochloride).[503]

Dimethyl 3,5-bisdiazo-3,4-dioxoadipate (**11**) gave dimethyl 4-hydroxy-5-oxo-2,5-dihydro-3,6-pyridazinedicarboxylate (**12**) ($Pr^iOH$, reflux, 24 h: ~25%).[406]

3-Oxo-5-phenyl-2-phenylhydrazonopent-4-enenitrile (**13**) gave 4-oxo-1,6-diphenyl-1,4,5,6-tetrahydro-3-pyridazinecarbonitrile (**14**) (BuOH, reflux, 90 min: 91%).[1068]

Also other examples.[114, 1304, 1369, 1969, 2028, 2031, 2111]

*Note:* Until 1975 it was thought that simple buta-1,3-dienes coupled with aromatic diazonium salts to afford isolable acyclic intermediates (akin to those used in the foregoing examples), which then underwent cyclization in base. However, it has been shown that such intermediates are simply salts of the final dihydropyridazines, probably formed directly by concerted cycloaddition.[275,583] For example, 2,3-dimethylbuta-1,3-diene with *p*-nitrobenzenediazonium hexafluorophosphate gave 4,5-dimethyl-1-*p*-nitrophenyl-1,2-dihydropyridazine, initially as the salt (MeCN, 25°, 30 min: 79%).[275]



(11) (12) (13) (14)

### *1.1.2.2 From (Hydrazonomethyl)cycloalkanes or Related Synthons*

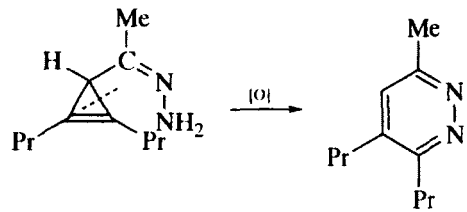
This small category of cyclizations is illustrated by the oxidative conversion of 3-α-hydrazonoethyl-1,2-dipropylcyclopropene (**15**) into 3-methyl-5,6-dipropylpyridazine (**16**) in 58% yield by treatment with yellow mercuric oxide in light petroleum for 24 h;[298] of 6,6-dimethyl-2-oxo-4-(phenylhydrazono)-methyl-1,1,3-cyclohexanetricarbonitrile (**17**) into 5-methyl-3-oxo-2-phenyl-2,3-dihydro-4-pyridazinecarbonitrile (**18**) (60%) by warming in ethanolic piperidine at 40° for 1 h;[1731] and of dimethyl diazo(1,2,3-tri-*t*-butylcycloprop-2-enyl)methanephosphonate into dimethyl 4,5,6-tri-*t*-butyl-3-pyridazinephosphonate (~70%) by heating in benzene for 4 h.[15] Other examples have been reported in the foregoing references and elsewhere.[13,430,1438,1613,1614]

### *1.1.2.3 From Hydrazonoalkanols or Related Synthons*

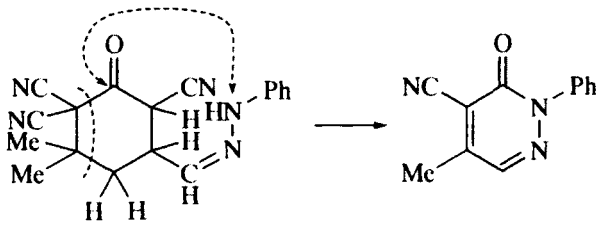
The hydroxy substituent in these synthons acts as a leaving group during cyclization; it may be replaced by a halogeno, amino, or other substituent for the same purpose. Typical examples follow:

1-Phenyl-4-phenylhydrazonobut-2-enol (**19**) gave 2,3-diphenyl-1,2-dihydropyridazine (**20**) (TsOH, PhMe, 25°, until dissolved: 95%).[1]

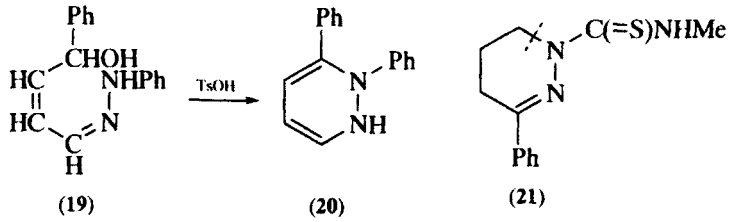
4-Phenyl-4-tosylhydrazonobutanol gave 3-phenylpyridazine (KH, then $MeOCH_2CH_2OMe$, 85°, 24 h: 41%).[282]



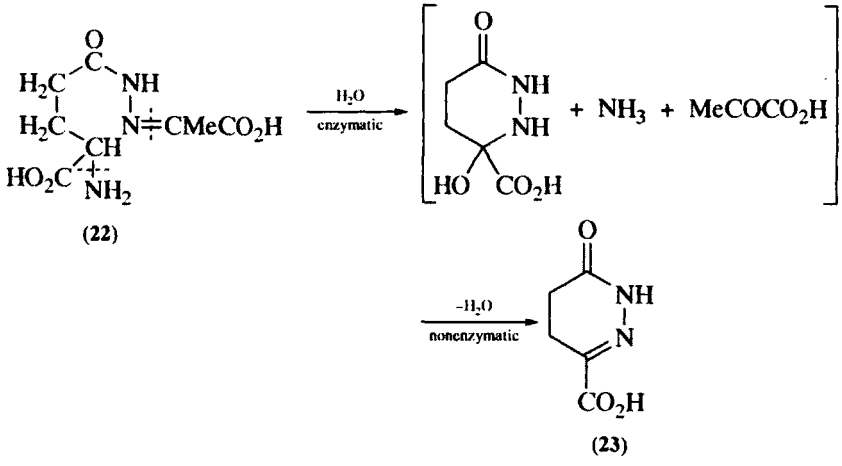

(15) (16)




(17) (18)




(19) (20) (21)




(22) (23)

{4-Chloro-1[4-methyl(thiosemicarbazono)]butyl}benzene gave 1-methyl-(thiocarbamoyl)-3-phenyl-1,4,5,6-tetrahydropyridazine (**21**) (KOH, $PhCH_2Et_3NCl$, $CH_2Cl_2$, 25°, 10 min: 73%).[1121]

Pyruvic acid γ-glutamylhydrazone (**22**) gave 6-oxo-1,4,5,6-tetrahydro-3-pyridazinecarboxylic acid (**23**) with loss of alanine and ammonia (glutamine transaminase from rat liver, pH 7, 37°).[1124]

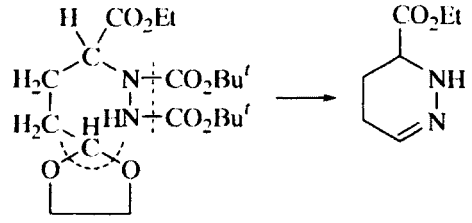
Also other examples in the foregoing references and elsewhere.[1139, 1406, 1898]

### *1.1.2.4 From Hydrazonoalkanals or Related Synthons*

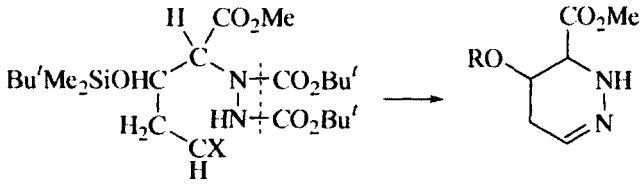
All the available examples in this category employ aldehydes that are liberated from derivatives during or immediately before the cyclization process to give hydropyridazines, as illustrated hereafter.

Ethyl 2-(*N*,*N'*-di-*t*-butoxycarbonylhydrazino)-4-formylbutyrate ethylene acetal (**24**) gave ethyl 2,3,4,5-tetrahydro-3-pyridazinecarboxylate (**25**) ($F_3CCO_2H$, 25°, 15 min: 90%, as trifluoroacetate salt).[1559]

Methyl 2-(*N*,*N'*-di-*t*-butoxycarbonylhydrazino)-4-formylbutyrate dimethyl acetal gave methyl 2,3,4,5-tetrahydro-3-pyridazinecarboxylate ($F_3CCO_2H$, $CH_2Cl_2$, 25°, 30 min: 95%, as base) and thence the hexahydro analogue



(24) (25)



(26) (27)

[$NaBH_3CN$, MeOH, 25°, 12 h: 33% as the 1-(2,4-dinitrophenyl) derivative].[1375, 1648]

Methyl 3-*t*-butyldimethylsiloxy-2-(*N*,*N'*-di-*t*-butoxycarbonylhydrazino)oct-5-enoate (**26**, X = EtCH:) was used to generate a solution of the aldehyde (**26**, X = O) ($O_3$, $CH_2Cl_2$, −78°; then $PPh_3$, $Me_2S$, 40°, 16 h) that afforded methyl 4-*t*-butyldimethylsiloxy-2,3,4,5-tetrahydro-3-pyridazinecarboxylate (**27**, R = $SiMe_2Bu^t$) ($F_3CCO_2H$, reflux, 10 min: 50%) and thence the hydroxypyridazine (**27**, R = H) (or its tautomer, 4-oxohexahydro-3-pyridazinecarboxylic acid) [$Bu_4NF$, THF(tetrahydrofuran), 0°, 30 min: 90%].[1806]

Also other examples.[1563, 1566, 1955]

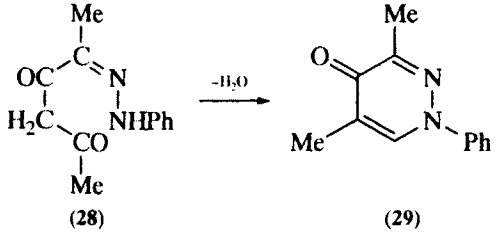
### *1.1.2.5 From Hydrazonoalkanones or Related Synthons*

A variety of such ketones and their derivatives has been used in this cyclization, illustrated in the following examples:

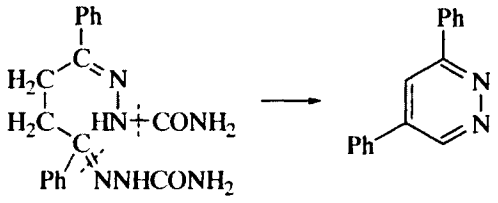
5-Phenylhydrazono-2,4-hexanedione (**28**) gave 3,6-dimethyl-1-phenyl-4(1 *H*)-pyridazinone (**29**) (AcOH, reflux, 2 min: 65%; $NaHCO_3$, EtOH, reflux, 40 min: 57%; $Na_2SO_4$, dry, 190°, 10 min: 76%).[110]

1,4-Diphenyl-1,4-bissemicarbazonobutane (**30**) gave 3,6-diphenylpyridazine (**31**) ($Me_2NCHO$, reflux, 1 h: 97%; mechanism unclear).[58]

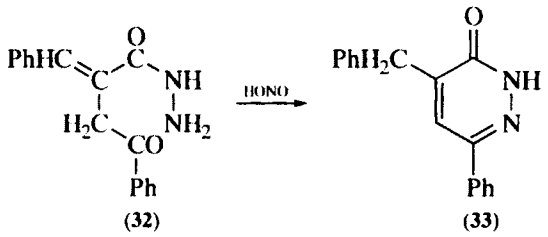
1-Benzoyl-2-benzylidenepropionohydrazide (**32**) or its furfurylidene analogue gave 4-benzyl-6-phenyl-3(2*H*)-pyridazinone (**33**) or its 4-furfuryl analogue, respectively ($HNO_2$, 5°: 80% or 60%, respectively;[367, 1243] also analogous cyclizations in AcOH–HCl, alkali, or AcCl).[1274]



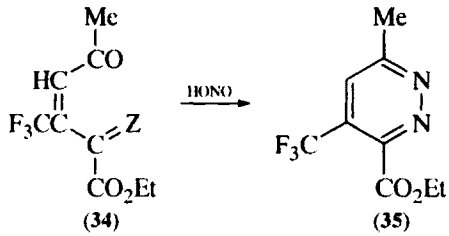
(28) (29)



(30) (31)



(32) (33)



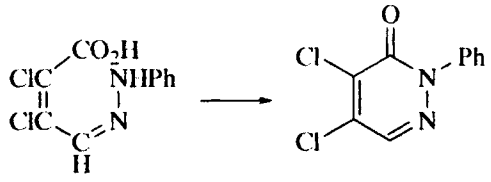
(34) (35)

Ethyl 2-diazo-5-oxo-3-trifluoromethylhex-3-enoate (**34**, $Z = N_2$) gave ethyl 6-methyl-4-trifluoromethyl-3-pyridazinecarboxylate (**35**) via the transient intermediate (**34**, $Z = NN{:}PPh_3$) by loss of $Ph_3PO$ ($Ph_3P$, $Et_2O$, 25°, 12 h: 64%).[1822]

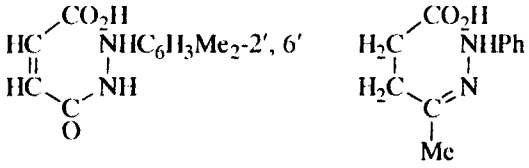
Also other examples in the foregoing references and elsewhere.[203,572,617,1238,1274,1370,1620,1706]

### *1.1.2.6 From Hydrazonoalkanoic Acids or Related Synthons*

The conditions for these cyclizations are illustrated in the following examples.



(36) (37)



(38) (39)

2,3-Dichloro-4-phenylhydrazonocrotonic acid (mucochloric acid phenylhydrazone) (**36**) gave 4,5-dichloro-2-phenyl-3(2*H*)-pyridazinone (**37**) (AcOH, reflux, 15 min: ~60%);[464] similarly, mucochloric acid semicarbazone gave 4,5-dichloro-3(2*H*)-pyridazinone with loss of $CO_2$ and $NH_3$ (AcOH, reflux, 6 h: 70%).[1024]

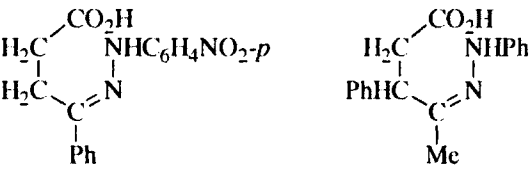
3-[*N'*-(2,6-Dimethylphenyl)hydrazinocarbonyl]acrylic acid (**38**) gave 1-(2,6-dimethylphenyl)-3,6(1*H*,2*H*)-pyridazinedione (AcONa, $Ac_2O$, 60°, 30 min: 82%).[1361]

4-Phenylhydrazonovaleric acid (**39**) gave 6-methyl-2-phenyl-4,5-dihydro-3(2*H*)-pyridazinone (98%, $H_2SO_4$, 25°, 24 h: 70%) and thence 6-methyl-2-phenyl-3(2*H*)-pyridazinone (dichlorodicyanobenzoquinone, dioxane, reflux, 6 h).[1200]

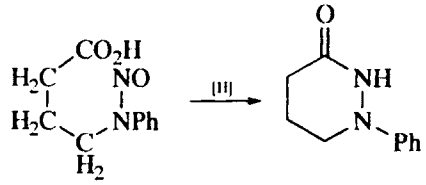
4-*o*-Nitrophenylhydrazono-4-phenylbutyric acid (**40**) gave 2-*o*-nitrophenyl-6-phenyl-4,5-dihydro-3(2*H*)-pyridazinone (TsOH, xylene, reflux, water removal, 18 h: 53%) and thence the didehydro product ($Br_2$, AcOH, reflux, 1 h: 79%).[1069]

3,4-Diphenyl-4-phenylhydrazinobutyric acid (**41**) gave 2,5,6-triphenyl-4,5-dihydro-3(2*H*)-pyridazinone (neat, 170°, 30 min).[391]

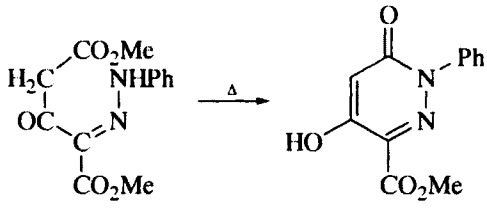
4-(*N*-Nitrosoanilino)butyric acid (**42**) gave 1-phenyl-1,4,5,6-tetrahydro-3(2*H*)-pyridazinone (**43**) by reductive cyclization ($TiCl_4$, Mg, $Et_2O$-



(40) (41)



(42) (43)



(44) (45)

$CH_2Cl_2$, $N_2$ ↓, 20°, 2.5 h; then substrate, 20°, 1 h; then AcONa, $H_2O$, reflux, 6 h: 65%).[1187]

Also other examples.[302, 1059, 1114, 1297, 1298, 1621, 1704, 1776]

### *1.1.2.7 From Hydrazonoalkanoic Esters or Related Synthons*

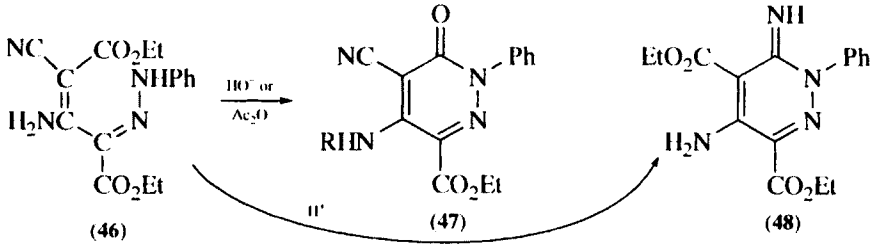
These esters appear to undergo cyclization a little more easily than the corresponding acids (Sections 1.1.2.6), but the choice of synthon usually depends on the ease of preparation / availability. Typical examples follow.

Dimethyl 3-oxo-2-phenylhydrazonoglutarate (**44**) gave methyl 4-hydroxy-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate (**45**) (*o*-dichlorobenzene, reflux, 45 min: 94%).[1090]

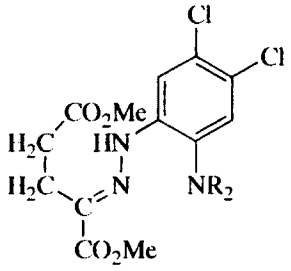
Diethyl 3-amino-2-cyano-4-phenylhydrazonopent-2-ene-1,5-dioate (**46**) gave ethyl 4-amino-5-cyano-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate (**47**, R = H) (NaOH, EtOH, reflux, 30 min: 65%), its 4-acetamido analogue (**47**, R = Ac) ($Ac_2O$, reflux, 10 h: 78%), or diethyl 4-amino-6-imino-1-phenyl-1,6-dihydro-3,5-pyridazinedicarboxylate (**48**) (AcOH–HCl, reflux, 3 h: 50%; the cyano group of the synthon was involved in cyclization under these conditions).[119]

Dimethyl 2-[(2-amino-4,5-dichlorophenyl)hydrazono]glutarate (**49**, R = H), made in situ by catalytic hydrogenation of the nitro analogue (**49**, R = O), gave methyl 1-(2-amino-4,5-dichlorophenyl)-6-oxo-1,4,5,6-tetrahydro-3-pyridazinecarboxylate (NaOH, EtOH–$H_2O$, reflux, 1 min: 75%).[1604]

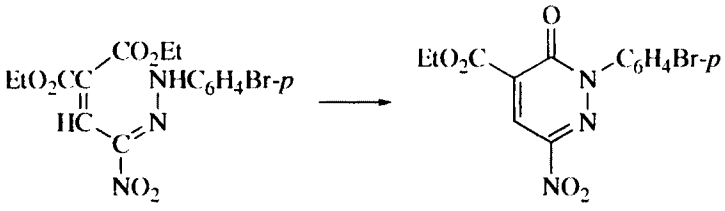
Diethyl 3-*p*-bromophenylhydrazono-3-nitroprop-1-ene-1,1-dicarboxylate (**50**), made in situ from the potassium salt of diethyl 3-nitroprop-1-ene-1,1-dicarboxylate and *p*-bromobenzenediazonium tetrafluoroborate, gave



(46) (47) (48)



(49)



(50) (51)

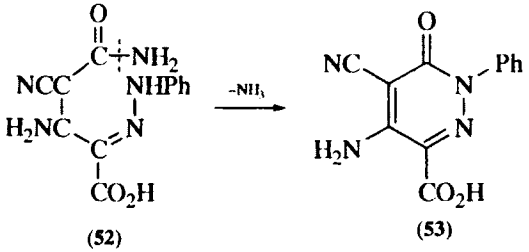
ethyl 2-*p*-bromophenyl-6-nitro-3-oxo-2,3-dihydro-4-pyridazinecarboxylate (**51**) ($H_2O$, 25°, 2.5 h: 90% overall).[1495]

Diethyl 4-diazo-3-trifluoromethylpent-2-ene-1,5-dioate gave ethyl 6-ethoxy-4-trifluoromethyl-3-pyridazinecarboxylate ($Ph_3P$, $Et_2O$, 25°, 12 h: 83%; cf. last example in Section 1.1.2.5).[1822]

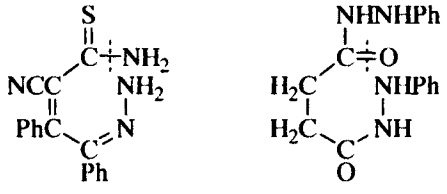
Also other examples.[87,312,376,712,852,898,1212,1216]

### *1.1.2.8 From Hydrazonoalkanamides or Related Synthons*

The limited number of examples in this category suggest that cyclizations between hydrazono and amide (or thioamide) groupings proceed by elimination of ammonia rather than water or hydrogen sulfide; in contrast, cyclization between two carbohydrazide groupings does seem to involve loss of water rather than hydrazine.



(52) (53)



(54) (55)

3-Amino-4-carbamoyl-4-cyano-2-phenylhydrazonobut-3-enoic acid (**52**), prepared in situ from 3-amino-4-carbamoyl-4-cyanobut-3-enoic acid and benzenediazonium chloride, gave 4-amino-5-cyano-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylic acid (**53**) (AcONa, EtOH, $H_2O$, 20°, 15 min: 82%).[129]

2-Cyano-4-hydrazino-3,4-diphenylbut-2-enethioamide (**54**) gave 5,6-diphenyl-3-thioxo-2,3-dihydro-4-pyridazinecarbonitrile (AcOH, reflux, 2 h), characterized as 3-methylthio-5,6-diphenyl-4-pyridazinecarbonitrile (MeI, EtONa, EtOH, reflux, 2 h: 60%).[1390]

$N^2$, $N^{2'}$-Diphenylsuccinohydrazide (**55**) gave 1-phenyl-6-phenylhydrazono-1,4,5,6-tetrahydro-3(2*H*)-pyridazinone (EtOH, reflux, 1 h: 90%; note loss of water rather than hydrazine).[1295]

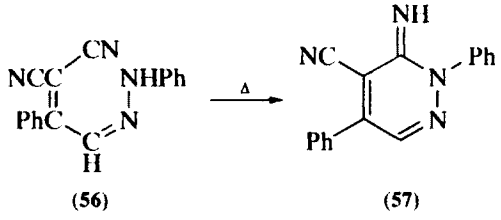
Also other examples.[1214]

### *1.1.2.9 From Hydrazonoalkanenitriles or Related Synthons*

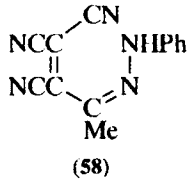
These synthons have undergone cyclization to pyridazinimines under a variety of conditions. However, the use of vigorous alkaline or acidic media has been avoided as a rule in order to obviate hydrolysis to corresponding pyridazinones or other untoward reactions. The following examples illustrate the more important procedures.

#### Thermal Cyclization

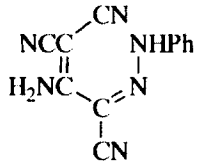
2-Phenyl-3-phenylhydrazonoprop-1-ene-1,1-dicarbonitrile (**56**) gave 3-imino-2,5-diphenyl-2,3-dihydro-4-pyridazinecarbonitrile (**57**) (neat, 160°, 30 min: 80%).[37]



(56) (57)



(58)



(59)

3-Phenylhydrazonobut-1-ene-1,1,2-tricarbonitrile (**58**) gave 3-imino-6-methyl-2-phenyl-2,3-dihydro-4,5-pyridazinedicarbonitrile (MeCN, reflux, 20 min: 60%).[3]

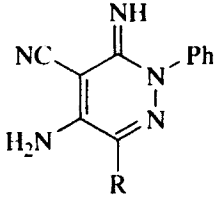
2-Amino-3-phenylhydrazonoprop-1-ene-1,1,3-tricarbonitrile (**59**) gave 4-amino-6-imino-1-phenyl-1,6-dihydro-3,5-pyridazinedicarbonitrile (**60**, R = CN) (dioxane, reflux, 1 h: 90%) or methyl 4-amino-5-cyano-6-imino-1-methyl-1,6-dihydro-3-pyridazinecarboximidate [60, R = C(:NH)OMe] (MeONa, MeOH, reflux, 8 h: 90%).[1365]

## Cyclization in Base

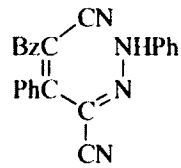
2-Benzoyl-3-phenyl-4-phenylhydrazonopent-2-enedinitrile (**61**) gave 5-benzoyl-6-imino-1,4-diphenyl-1,6-dihydro-3-pyridazinecarbonitrile (<1% NaOH/EtOH, 25°, 12 h; then reflux, 30 min: 80%; note involvement of CN rather than Bz).[49]

3-Oxo-2-phenylhydrazonoglutaronitrile (**62**) gave 4-hydroxy-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarbonitrile (63, X = O), possibly via the imine (**63**, X = NH) (<0.5% NaOH/EtOH, reflux, 5 h: 70%).[1364]

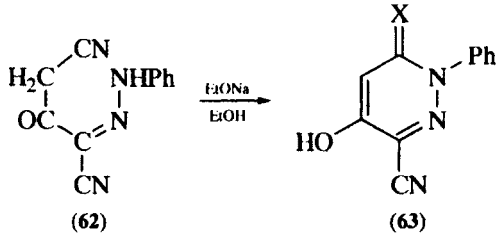
Ethyl 4,4-dicyano-3-methyl-2-phenylhydrazonobut-3-enoate (made in situ) gave ethyl 5-cyano-6-imino-4-methyl-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate (weakly basic, 30 min: 66%).[124]



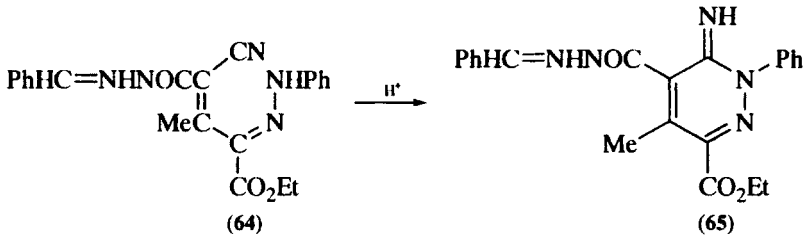
(60)



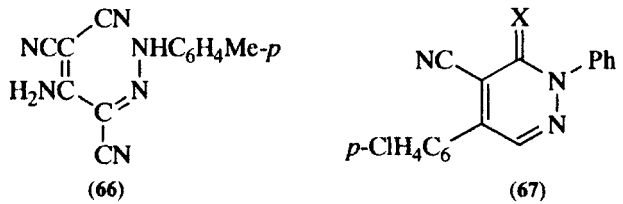
(61)



(62) (63)



(64) (65)



(66) (67)

## Cyclization in Acid

Ethyl 4-benzylidenehydrazinocarbonyl-4-cyano-3-methyl-2-phenyl hydrazonobut-3-enoate (**64**) gave ethyl 5-benzylidenehydrazinocarbonyl-6-imino-4-methyl-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate (**65**) (AcOH, HCl, reflux, 2 h: 75%).[1215]

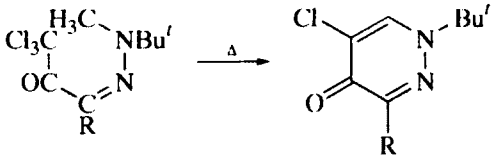
2-Amino-3-*p*-tolylhydrazonoprop-1-ene-1,3-tricarbonitrile (**66**) gave 4-amino-6-imino-1-*p*-tolyl-1,6-dihydro-3,5-pyridazinedicarbonitrile (AcOH, reflux, 3 h: 75%).[55]

2-*p*-Chlorophenyl-3-phenylhydrazonoprop-1-ene-1,1-dicarbonitrile gave 5-*p*-chlorophenyl-3-oxo-2-phenyl-2,3-dihydro-4-pyridazinecarbonitrile (**67**, X = O) (AcOH, $H_2O$, reflux, 2 h: 60%), probably via the imine (**67**, X = NH).[39]

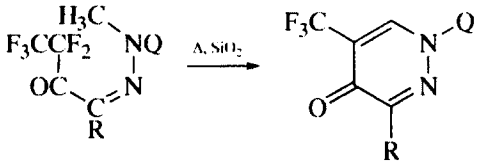
Also other examples in each subcategory.[51,60,119,151,216,219,221,229,234,521,988,1076,1198,1210,1374,1389,1399,1497,1644,1718,1728]

### 1.1.3 By Completion of the C3–C4 Bond

The cyclization of a C–N–N–C–C–C synthon has been achieved recently in three distinct ways—one gave aromatic pyridazines but the others afforded hydropyridazines. None has been developed to its full potential.



(68) (69)

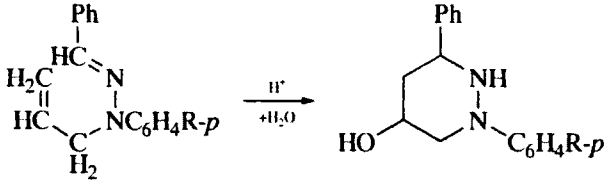


(70) (71)

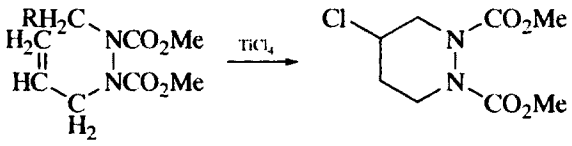
The first involved *thermal cyclization of an α-(N′-alkyl-N′-methylhydrazono)-α-(perhalogenoacyl)toluene (or alkane)* by loss of two hydrohalide molecules from the *N′*-methyl and the dihalogenomethylene part of the acyl grouping. For example, α-(*N′-t*-butyl-*N′*-methylhydrazono)-α-(trichloroacetyl)toluene (**68**, R = Ph) gave 1-*t*-butyl-5-chloro-3-phenyl-4(1*H*)-pyridazinone (**69**, R = Ph) in 56% yield by heating in toluene under reflux for 48 h;[135] the *p*-tolyl (**69**, R = $C_6H_4Me$-*p*) (77%), *p*-nitrophenyl (**69**, R = $C_6H_4NO_2$-*p*) (reflux, 10 days: 63%), and other analogues were made similarly,[135] Likewise, α-dimethylhydrazono-α-(pentafluoropropionyl)toluene (**70**, R = Ph, Q = Me) gave 1-methyl-3-phenyl-5-trifluoromethyl-4(1*H*)-pyridazinone (**71**, R = Ph, Q = Me) in 70% yield by heating with silica gel at 80° under nitrogen for 24 h;[1515] 1-(*N′-t*-butyl-*N′*-methylhydrazono)-1-(pentafluoropropionyl)propane (**70**, R = Et, Q = Bu′) gave 1-*t*-butyl-3-ethyl-5-trifluoromethyl-4(1*H*)-pyridazinone (**71**, R = Et, Q = Bu′) (90°, 6 h: 96%);[1515] and several other analogues were made similarly.[1515]

The second way involved *acid-catalyzed hydrative cyclization of N-allyl-N′-benzylidene-N-phenylhydrazines.* For example, *N*-allyl-*N′*-benzylidene-*N*-*p*-methylsulfonylphenylhydrazine (**72**, R = $SO_2Me$) gave 2-*p*-methylsulfonylphenyl-6-phenylhexahydro-4-pyridazinol (**73**, R = $SO_2Me$) (35%; structure confirmed by X-ray analysis) by stirring in a mixture of 45% sulfuric acid and toluene at 25° for 30 h;[1513] the 2,6-diphenyl analogue (**73**, R = H) could not be made in this way, but the 2-*p*-fluorophenyl-6-phenyl and other analogues were so made.[1513]

The third way involved *Lewis acid–catalyzed cyclization of an N-alkenyl-N′-(substituted-alkyl)hydrazine.* For example, treatment of *N*-allyl-*N,N′*-dimethoxycarbonyl-*N′*-methoxymethylhydrazine (**74**, R = OMe) or *N*-allyl-*N′*-chloromethyl-*N,N′*-dimethoxycarbonylhydrazine (**74**, R = Cl) with titanium (IV) chloride in dichloromethane at −78° → 20° for 3 h gave dimethyl 4-chlorohexahydro-1,2-pyridazinedicarboxylate (**75**) in 32% or 70% yield, respectively;[287] the chloro substituent in the product could be avoided by

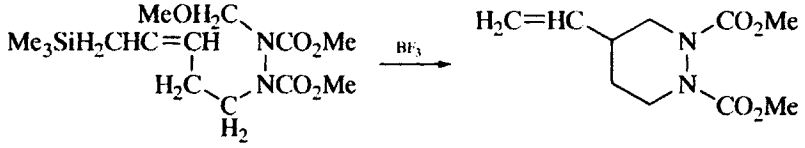


(72) (73)



(74) (75)

using another Lewis acid: thus, cyclization of *N,N'*-dimethoxycarbonyl-*N*-methoxymethyl-*N'*-(5-trimethylsilylpent-5-enyl)hydrazine (**76**) with boron trifluoride etherate afforded dimethyl 4-vinylhexahydro-1,2-pyridazinedicarboxylate (**77**) in 54% yield.[287] Other diverse examples have been reported.[287,1426]



(76) (77)

### 1.1.4 By Completion of the C4–C5 Bond

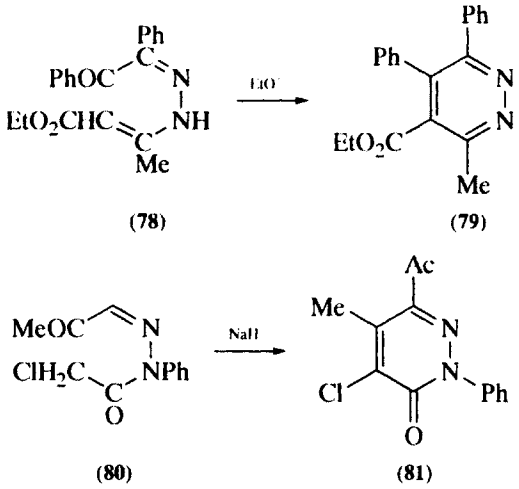
The cyclization of C–C–N–N–C–C synthons has been neglected except for the use of appropriate 1-methylene-6-carbonyl and 1,6-dimethylene entities to afford pyridazines or hydropyridazines. (However, see Ref. 2016.)

The conversion of *1-methylene-6-carbonyl synthons* into pyridazines is represented in the following examples:

Ethyl 3-[*N'*-(α-benzoylbenzylidene)hydrazino]crotonate (**78**) gave ethyl 3-methyl-5,6-diphenyl-4-pyridazinecarboxylate (**79**) (KOH, EtOH, reflux, 10 min: 93%).[314]

3-(*N'*-Chloroacetyl-*N'*-phenylhydrazono)-2,4-pentanedione (**80**) gave 6-acetyl-4-chloro-5-methyl-2-phenyl-3(2*H*)-pyridazinone (**81**) (NaH, PhMe, 25°, 5 h: 60%).[1620]

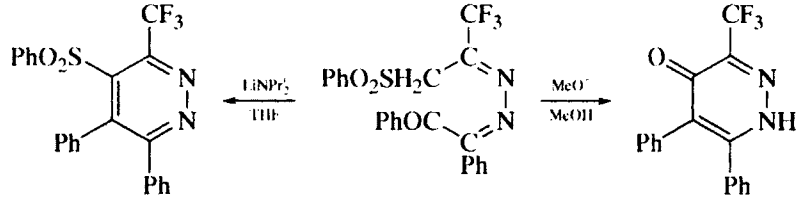
α-{[β-Phenylsulfonyl-α-(trifluoromethyl)ethylidene]hydrazono}deoxybenzoin (**83**) gave 3,4-diphenyl-5-phenylsulfonyl-6-trifluoromethylpyridazine



(78) (79) (80) (81)

(**82**) (LiNPr$^i_2$, THF–$C_6H_{14}$, $N_2$, reflux, 10 h: 15%) or 5,6-diphenyl-3-trifluoromethyl-4(1*H*)-pyridazinone (**84**) (MeONa, MeOH, reflux, 5 h: 58%; note the hydrolytic effect in the protic solvent).[1596]

Also other examples in the foregoing references.



(82) (83) (84)

Cyclization of *1,6-dimethylene synthons* has been used to furnish pyridazines or hydropyridazines according to circumstances, as illustrated in the following examples:

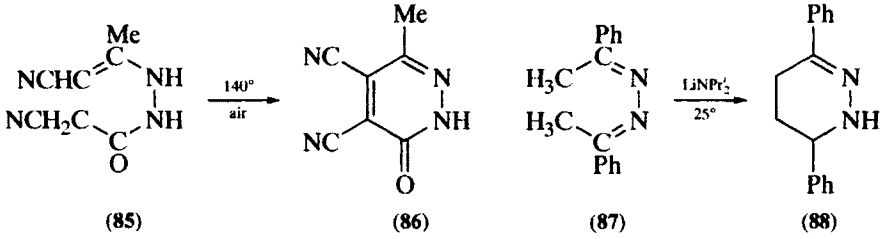
3-*N'*-(Cyanoacetyl)hydrazinocrotononitrile (**85**) gave 3-methyl-6-oxo-1,6-dihydro-4,5-pyridazinedicarbonitrile (**86**) (neat, 140°, air, 5 min: 78%).[1487]

Bis(α-phenylethylidene)hydrazine (**87**) gave 3,6-diphenyl-1,4,5,6-tetrahydropyridazine (**88**) (Pr$^i_2$NLi, THF, 25°, 24 h: 34%).[254,317]

Also other examples in the foregoing references and elsewhere.[568]

## 1.2 FROM TWO SYNTHONS

Apart from those that employ other heterocyclic systems as synthons (Chapter 2), the bulk of pyridazine primary syntheses fall into this category, which is



(85) (86) (87) (88)

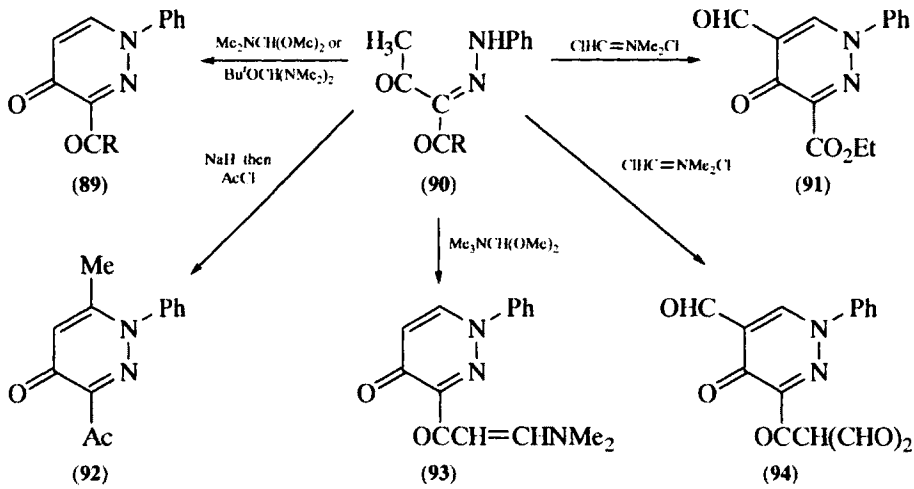
subdivided stepwise according to which of the ring atoms are provided by each of the two synthons.

### 1.2.1 By Using One- and Five-Atom Synthons

Of the three possible categories of this synthesis (N1 + N2–C3–C4–C5–C6, C3 + C4–C5–C6–N1–N2; and C4 + C5–C6–N1–N2–C3), only the second, in which the one-atom synthon supplies C3, has been used from time to time. The following examples illustrate the presently known possibilities:

α-Acetyl-α-phenylhydrazonoacetophenone (**90**, R = Ph) with dimethylformamide dimethyl acetal gave 3-benzoyl-1-phenyl-4(1*H*)-pyridazinone (**89**, R = Ph) (reflux, 30 min: 70%).[974]

Ethyl 3-oxo-2-phenylhydrazonobutyrate (**90**, R = OEt) with dimethylformamide dimethyl acetal gave methyl 4-oxo-1-phenyl-1,4-dihydro-3-pyridazinecarboxylate (**89**, R = OMe) (reflux, 30 min: 70%; note transesterification by the methanol from the acetal)[974] with *t*-butoxybisdimethylaminomethane gave ethyl 4-oxo-1-phenyl-1,4-dihydro-3-pyri- dazinecarboxylate (**89**, R = OEt) ($Me_2NCHO$, 70°, 12 h: 87%),[316] or with the Vilsmeier reagent (from $POCl_3$–$Me_2NCHO$) gave ethyl 5-formyl-4-oxo-1-phenyl-1,4-dihydro-3-pyridazinecarboxylate (**91**) ($Me_2NCHO$, 0° → 60°, 6 h: 59%).[1302]




(89) (90) (91)

(92) (93) (94)

α-Acetyl-α-phenylhydrazonoacetone (**90**, R = Me) with acetyl chloride gave 3-acetyl-6-methyl-1-phenyl-4(1*H*)-pyridazinone (**92**) (NaH, PhMe, < 5°, 40 min, then AcCl, THF, 25°, 3 h: 60%),[1706] with dimethylformamide gave 3-(3-dimethylaminoacryloyl)-1-phenyl–4(1*H*)-pyridazinone (**93**) (reflux, 30 min: 60%),[974] or with the Vilsmeier reagent gave 6-diformylacetyl-5-oxo-2-phenyl-2,5-dihydro-4-pyridazinecarbaldehyde (**94**) ($Me_2NCHO$, 0° → 60°, 6 h: 59%; note triple *C*-formylation of the product).[1302]

Also other examples in the foregoing references and elsewhere.[2015]

### 1.2.2 By Using Two- and Four-Atom Synthons (*H* 25, 230, 355)

This category of synthesis has been used to make pyridazines in so many ways that it must be further divided, initially according to which ring atoms are supplied by the two-atom synthon.

#### *1.2.2.1 Where the Two-Atom Synthon Provides N1 + N2*

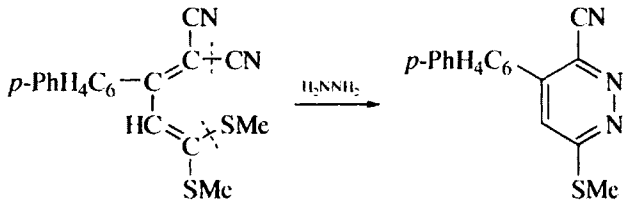
This large division is further divided according to the nature of the N1–N2 synthon: hydrazine, substituted hydrazines, azo compounds (including dialkyl azodicarboxylates), or other such providers.

##### 1.2.2.1.1 Using Hydrazine as the N1–N2 Synthon

Hydrazine or hydrazine hydrate has been used with a great variety of C3–C4–C5–C6 synthons to afford pyridazines and hydropyridazines, as illustrated in the following examples:

**With Appropriate Alkadienes**

2-Biphenyl-4′-yl-4,4-bismethylthiobutadiene-1,1-dicarbonitrile (**95**) gave 4-biphenyl-4′-yl-6-methylthio-3-pyridazinecarbonitrile (**96**) ($Et_3N$, EtOH, 25°, 18 h: ?%).[1366]

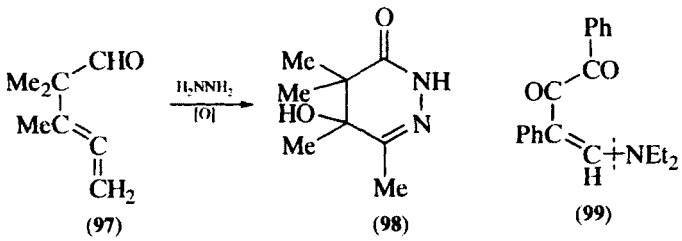



(**95**) (**96**)

**With Appropriate Alkenals**

2,2,3-Trimethylpenta-3,4-dienal (**97**) gave 5-hydroxy-4,4,5,6-tetramethyl-4,5-dihydro-3(2*H*)-pyridazinone (**98**), via aerial oxidation of 3,4,5,5-

tetramethyl-2,5-dihydropyridazine (TsOH, PhH, reflux, water removal: ?%; structure confirmed by X-ray analysis).[1520]

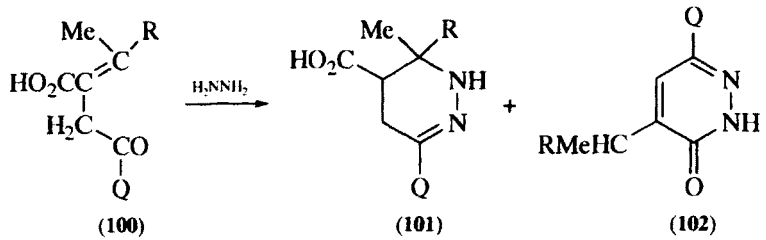


(97) (98) (99)

## With Appropriate Alken- or Alkynones

α-Benzoylformyl-β-diethylaminostyrene (**99**) gave 3,5-diphenyl-4(1*H*)-pyridazinone (anhydrous $H_2NNH_2$, Pr$^i$OH, $Et_2O$, ?h: 60%).[249]

3-(3-Chloro-6-methylphenyl)-2-(*p*-toluoylmethyl)crotonic acid (**100**) gave a separable mixture of 3-(3-chloro-6-methylphenyl)-3-methyl-6-*p*-tolyl-2,3,4,5-tetrahydro-4-pyridazinecarboxylic acid (**101**) and 4-(3-chloro-6,α-dimethylbenzyl)-6-*p*-tolyl-3(2*H*)-pyridazinone (**102**), the latter by reaction of hydrazine with both carbonyl groups of the substrate (**100**) (BuOH, reflux, 16h: 13% and 26%, respectively).[1710]



(100) (101) (102)

[R = 3-Cl-6-MeC$_6$H$_3$; Q = *p*-Me-C$_6$H$_4$]

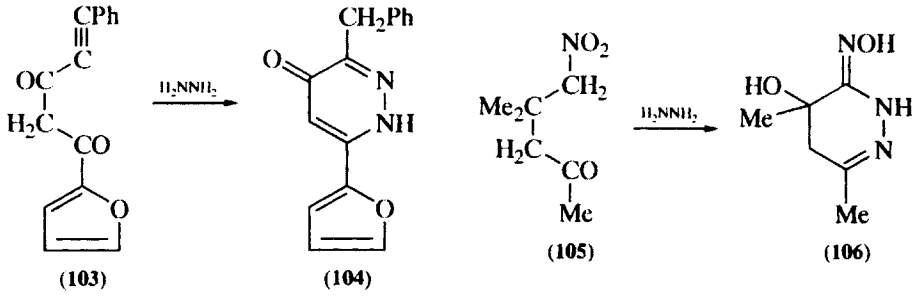
2-(1,3-Dioxo-5-phenylpent-4-ynyl)furan (**103**) gave 3-benzyl-6-furan-2′-yl-4(1H)-pyridazinone (**104**) plus a separable byproduct (EtOH, 25°, 3h: 39%).[1372,1707]

4,4-Dimethyl-5-nitro-2-pentanone (**105**) gave 4,4,6-trimethyl-4,5-dihydro-3(2*H*)-pyridazinone oxime (**106**) ($H_2NNH_2 + H_3PO_4$, pH 7, reflux, 4h: 77%) and thence the free pyridazinone (6M HCl, reflux, 2h: 88%).[4]

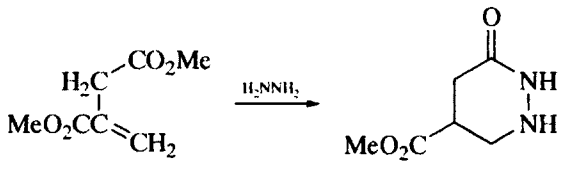
Also other examples.[109,818,1439,1468]

## With Appropriate Alken- or Alkynoic Acids

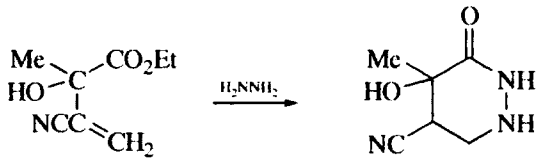
Dimethyl methylenesuccinate (**107**) gave methyl 6-oxohexahydro-4-pyridazinecarboxylate (**108**) (MeOH, 25°, 4h: 80%; structure confirmed by X-ray analysis).[1544]




(103) (104) (105) (106)




(107) (108)

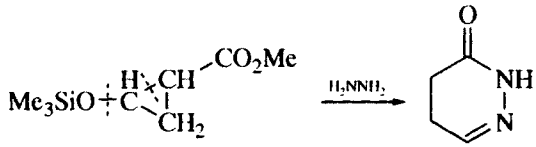



(109) (110)

Ethyl 3-cyano-2-hydroxy-2-methylbut-3-enoate (**109**) gave 5-hydroxy-5-methyl-6-oxohexahydro-4-pyridazinecarbonitrile (**110**) (MeOH, 20°, dark, 5 h: 74%).[1508]

Methyl 2-trimethylsiloxycyclopropane-1-carboxylate (**111**) gave 4,5-dihydro-3(2*H*)-pyridazinone (**112**) (MeOH, 25°, 16 h: 82%).[127]

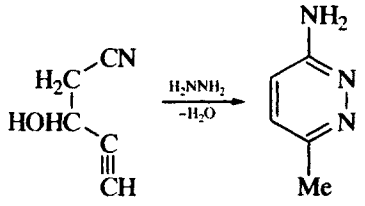
Also other examples in the foregoing references and elsewhere.[109]



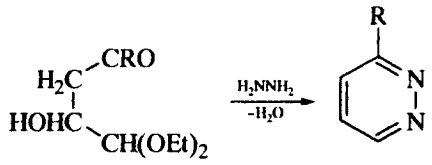

(111) (112)

## With Appropriate Alkene- or Alkynenitriles

3-Hydroxypent-4-ynenitrile (**113**) gave 6-methyl-4-pyridazinamine (**114**) (neat, $H_2NNH_2$, 80°, sealed, 25 h: 66%).[560, 1160]




(113) (114)




(115) (116)

### With Appropriate Dialdehydes

2-Hydroxysuccinaldehyde, as its diethyl acetal (**115**, R = H), gave pyridazine (**116**, R = H) ($H_2NHH_2 \cdot H_2SO_4$, MeOH–$H_2O$, 50°, 18 h: 80%).[414]

2-Methyl-2-phenylsuccinaldehyde gave 4-methyl-4-phenyl-4,5-dihydropyridazine (no details: 86%).[428]

### With Appropriate Aldehydo Ketones

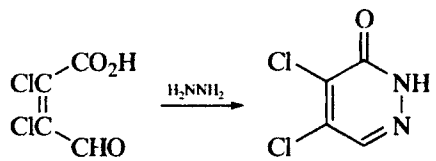
2-Hydroxy-9-oxodecanal diethyl acetal (**115**, R = $C_6H_{13}$) gave 3-hexylpyridazine (**116**, R = $C_6H_{13}$) ($H_2NNH_2 \cdot H_2SO_4$, MeOH–$H_2O$, 45°, 24 h: 85%).[414]

Also other examples.[318]

### With Appropriate Aldehydo Acids or Esters

Mucochloric acid (**117**) gave 4,5-dichloro-3(2*H*)-pyridazinone (**118**) (EtOH, 5°; then reflux, 6 h; 91%).[973]

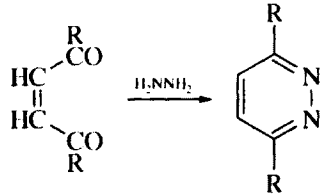
Also other examples.[1794]




(117) (118)

### With Appropriate Aldehydo Nitriles

There are no recent examples in this group.



(119) (120)

### With Appropriate Diketones

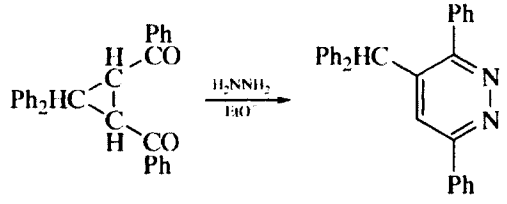
Hex-3-ene-2,5-dione (**119**, R = Me) gave 3,6-dimethylpyridazine (**120**, R = Me) ($H_2O$, reflux, 1 h: 45%).[894]

1,4-Diphenylbut-2-ene-1,4-dione (**119**, R = Ph) gave 3,6-diphenylpyridazine (**120**, R = Ph) (EtOH–$H_2O$, reflux: 84%);[499] the tetracarbonyl iron complex of (**119**, R = Ph) gave the same product (**120**, R = Ph) (AcOH, 50°, 6 h: 70%).[1154]

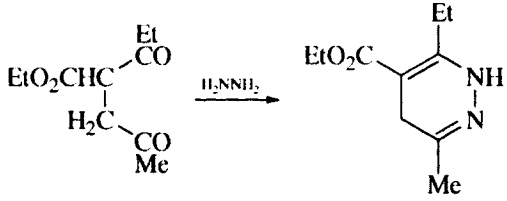
1-*p*-Bromophenyl-1,4-decanedione gave 3-*p*-bromophenyl-6-hexylpyridazine (95% EtOH, reflux, 4 h: 52%; product named incorrectly in original).[1576]

1,2-Dibenzoyl-3-diphenylmethylcyclopropane (**121**) gave 4-diphenyl methyl-3,6-diphenylpyridazine (**122**) (NaOH, EtOH, reflux, 72 h: 65%).[309]

Ethyl 4-oxo-2-propionylvalerate (**123**) gave ethyl 3-ethyl-6-methyl-2,5-dihydro-4-pyridazinecarboxylate (**124**) (EtOH, 25°, 5 h: 55%) and thence the didehydro analogue ($HNO_2$, $H_2O$, 25°, 1 h: 83%).[10]



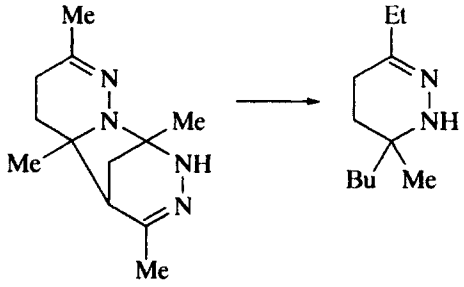
(121) (122)



(123) (124)

Acetonylacetone (2,4-hexanedione) gave a dimer of 3,6-dimethyl-4,5-dihydropyridazine ($H_2O$, 30°, 1 h; then 90°, 20 min; 91%),[1846] shown subsequently to have the structure (**125**).[318, 1199] The dimer afforded

monomeric 3-butyl-3,6-dimethyl-2,3,4,5-tetrahydropyridazine (**126**) (LiBu, $Et_2O$, 30°, 1 h: 50%),[1199,1847] which could be partially dehydrogenated.[1201]



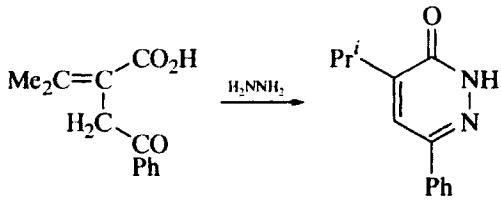
(125) (126)

Also other examples in the foregoing references and elsewhere.[62,186,189,268,328,392,475,577,778,840,1029,1057,1100,1174,1458,1490,2099]

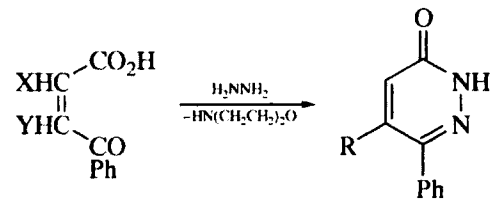
## With Appropriate Keto Acids

2-Benzoylmethyl-3-methylcrotonic acid (**127**) gave 4-isopropyl-6-phenyl-3(2*H*)-pyridazinone (**128**) (MeOH, reflux, 2 h: 81%);[104] also analogous examples.[104,1245]

3-Benzoyl-2-morpholinopropionic acid [**129**, X = N ($CH_2CH_2$)O, Y = H] gave 6-phenyl-3(2*H*)-pyridazinone (**130**, R = H) (BuOH, reflux, 4 h: 80%; oxidation provided by loss of morpholine);[497] somewhat similarly, 3-benzoyl-4-morpholinobutyric acid [**129**, X = H, Y = $CH_2N(CH_2CH_2)_2O$] gave 5-methyl-6-phenyl-3(2*H*)-pyridazinone (**130**, R = Me) ($HOCH_2CH_2OH$, KOH, reflux, 5 h: 65%).[1334]



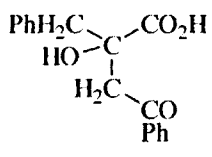
(127) (128)



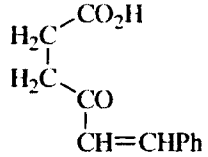
(129) (130)

3-Benzoyl-2-benzyl-2-hydroxypropionic acid (**131**) gave 4-benzyl-6-phenyl-3(2*H*)-pyridazinone (EtOH, reflux, 3 h: 71%; note dehydrative oxidation).[238]

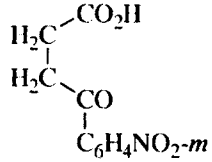
4-Oxo-6-phenylhex-5-enoic acid (**132**) gave 6-styryl-4,5-dihydro-3(2*H*)-pyridazinone (AcOH, reflux, 2 h: 73%).[634]



(131)



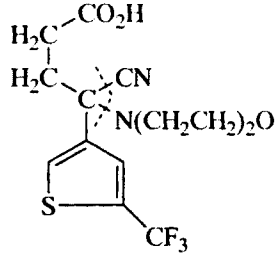
(132)



(133)

3-*m*-Nitrobenzoylpropionic acid (**133**) gave 6-*m*-nitrophenyl-4,5-dihydro-3(2*H*)-pyridazinone (K salt, $H_2O$, reflux, 2 h: 88%) and thence 6-*m*-nitrophenyl-3(2*H*)-pyridazinone ($Br_2$, AcOH, 70°, 2 h: 86%).[919,1848]

4-Cyano-4-morpholino-4-(5-trifluoromethylthien-3-yl)butyric acid (**134**) [≡ 3-(5-trifluoromethylthen-3-oyl)propionic acid] gave 6-(5-trifluoromethylthien-3-yl)-4,5-dihydro-3(2*H*)-pyridazinone (EtOH, reflux, 8 h: 76%) and then the didehydro analogue ($SeO_2$, $(EtOCH_2CH_2)_2O$, reflux, 20 min: 91%).[514]



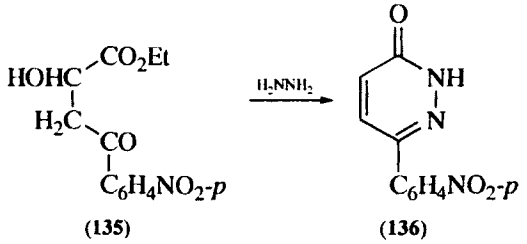
(134)

*p*-Bis(3-carboxy-2-methylpropionyl)benzene gave *p*-bis(4-methyl-6-oxo-1,4,5,6-tetrahydropyridazin-3-yl)benzene ($H_2O$, reflux, 4 h: >30%) and then *p*-bis(4-methyl-6-oxo-1,6-dihydropyridazin-3-yl)benzene (*m*-$O_2NC_6H_4SO_3Na$, NaOH, $H_2O$, reflux, >1 h: 72%).[1325]

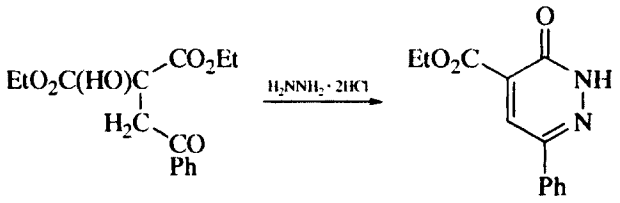
Also many other examples in the foregoing references and elsewhere.[113,152,157,161,164,236,360,369,465,467,504,642,698,737,779,804,870,906,922,1022,1102,1103,1113,1119,1151,1170,1173,1177,1178,1186,1256,1263,1267,1278,1299,1305,1306,1308,1312,1315,1318,1322,1339,1341,1346,1373,1394,1432,1445,1446,1449,1455,1464,1470,1474,1557,1580,1703,1712,1721,1727,1734,1757,1762,1763,1833,1834,1871,1875,1895,1959,1965,2019,2021,2074,2096]

## With Appropriate Keto Esters or Amides

Ethyl 3-*p*-nitrobenzoyllactate (**135**) gave 6-*p*-nitrophenyl-3(2*H*)-pyridazinone (**136**) (BuOH, reflux, 8 h: 88%; note dehydrative oxidation).[1309]




(135) (136)



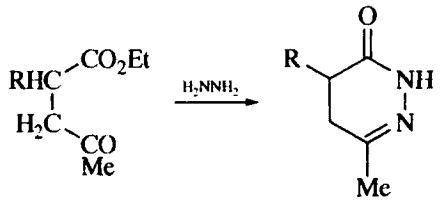

(137) (138)

Diethyl 2-benzoylmethyl-2-hydroxymalonate (**137**) gave ethyl 3-oxo-6-phenyl-2,3-dihydro-4-pyridazinecarboxylate (**138**) ($H_2NNH_2$.2HCl, EtOH, reflux, 18 h: 53; note use of hydrazine salt to avoid hydrazinolysis of the surviving ester grouping).[1767]

Ethyl levulinate (**139**, R = H) gave 6-methyl-4,5-dihydro-3(2*H*)-pyridazinone (**140**, R = H) (EtOH, reflux, 4 h: 90%);[1550] likewise, ethyl 2-hydroxylevulinate (**139**, R = OH) gave 4-hydroxy-6-methyl-4,5-dihydro-3(2*H*)-pyridazinone (**140**, R = OH) (EtOH, 25°, 2 h: 88%), which readily underwent dehydrative oxidation to give 6-methyl-3(2*H*)-pyridazinone (EtOH–HCl, reflux, 2 h: 75%).[1562]

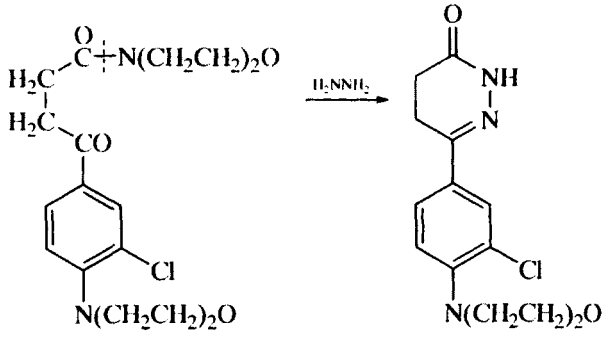
Ethyl 3-(3-thenoyl)propionate gave 6-thien-3′-yl-4,5-dihydro-3(2*H*)-pyridazinone ($H_2O$, reflux, 1 h: 83%) and thence 6-thien-3′-yl-3(2*H*)-pyridazinone ($MeOCH_2CH_2OH$?, $SeO_2$, reflux, 7 h: 84%).[1831]

3′-Chloro-4′-morpholino-3-morpholinocarbonylpropiophenone (**141**) gave 6-(3-chloro-4-morpholinophenyl)-4,5-dihydro-3(2*H*)-pyridazinone (**142**) ($AcOH–H_2O$, reflux, 2 h: 86%).[1759]




(139) (140)

Also other examples.[159,179,752,803,961,1086,1100,1319,1606,1634,1746,1836,2033]



(141) (142)

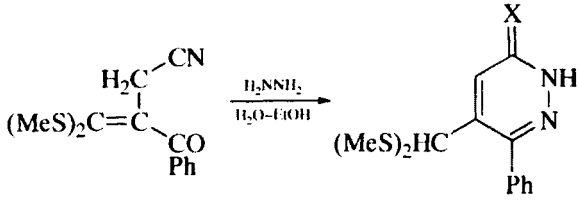
## With Appropriate Keto Nitriles

3-Benzoyl-4,4-bis(methylthio)but-3-enenitrile (**143**) gave 5-bis(methylthio)-methyl-6-phenyl-3(2*H*)-pyridazinone (**144**, X = O) probably via the imine (**144**, X = NH) (EtOH, reflux, 16 h: 68%).[131]

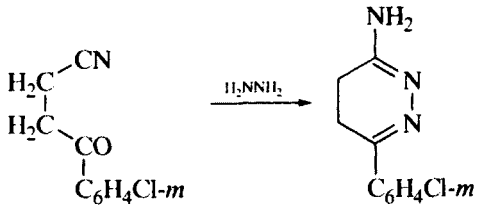
3-*m*-Chlorobenzoylpropiononitrile (**145**) gave 6-*m*-chlorophenyl-4,5-dihydro-3-pyridazinamine (**146**) (AcOH–EtOH, reflux, 2 h: 66%).[322]

7-(3-Cyano-2-methylpropionyl)[1,2,4]triazolo[4,3-*a*]quinoline gave 7-(6-amino-4-methyl-4,5-dihydropyridazin-3-yl)[1,2,4]triazolo[4,3-*a*]quinoline (AcOH–MeOH, reflux, 12 h: 80%).[1758]

Also other examples in the foregoing references and elsewhere.[1694]



(143) (144)

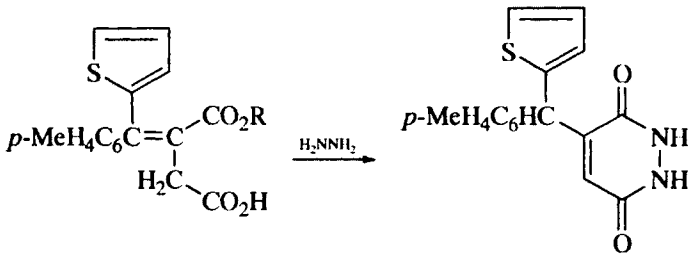


(145) (146)

## With Appropriate Dicarboxylic Acids or Esters

2-(α-Thien-2′-yl-α-*p*-tolylmethylene)succinic acid (**147**, R = H) gave 4-(α-thien-2′-yl-α-*p*-tolylmethyl)-3,6(1*H*,2*H*)-pyridazinedione (**148**) (EtOH,

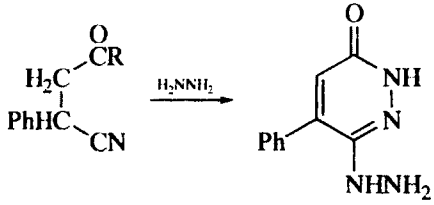
hot, 2 h: 71%); the monomethyl ester (**147**, R = Me) also gave the pyridazinedione (**148**) (EtOH, reflux, 30 min, then 25°, 12 h: 42%).[1000] Also related reactions.[860]



(147) (148)

## With Appropriate Cyano Carboxylic Acids, Esters, or Amides

Ethyl 3-cyano-3-phenylpropionate (**149**, R = OEt) or 3-cyano-3-phenylpropionamide (**149**, R = $NH_2$) gave 4-phenyl-4,5-dihydro-3,6(1*H*,2*H*)-pyridazinedione monohydrazone, probably better formulated as 6-hydrazino-5-phenyl-4,5-dihydro-3(2*H*)-pyridazinone (**150**) (neat, 25°, 1 week: 57% or "satisfactory", respectively);[1353] analogues were made similarly from appropriate esters.[1353]



(149) (150)

## With Appropriate Dinitriles

Succinonitrile gave 3,6-dihydrazino-4,5-dihydropyridazine, possibly via 4,5-dihydro-3,6-pyridazinediamine (excess neat $H_2NNH_2$, 20° 120 h: 50%;[1171] MeOH, 25°, $N_2$, 18 h; 60%).[639]

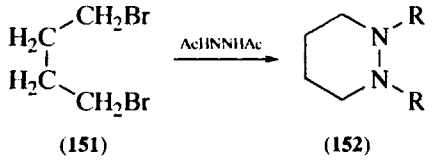
### 1.2.2.1.2 Using Substituted-Hydrazines as N1–N2 Synthons

The use of monosubstituted or unsymmetric disubstituted hydrazines naturally introduces ambiguity into the structure of the product unless the C2–C3–C4–C5 synthon happens to be symmetric. In practice this causes little difficulty. The following examples, divided according to the nature of the cosynthon, illustrate the conditions used and the results to be expected.

### With Appropriate Dihalogeno or Diacyloxyalkanes

1,4-Dibromobutane (**151**) and sodio-$\alpha,\beta$-diacetylhydrazine gave 1,2-diacetylhexahydropyridazine (**152**, R = Ac) and then hexahydropyridazine (piperidazine: **152**, R = H) (27%).[633]

2,3-Bischloromethylbutadiene (**153**) and $\alpha,\beta$-dimethylhydrazine gave 1,2-dimethyl-4,5-dimethylenehexahydropyridazine (**154**) ($Me_2SO$, 60°, 4 h: 27%);[340] also from a complex [made from allene and $PdCl_2 \cdot (PhCN)_2$] (MeHNNHMe, $Ph_3P$, THF–hexane, 25°, 12 h: 29%).[1092] $^1H$ nmr provided some support for the structure.[1082]




(**151**) (**152**)



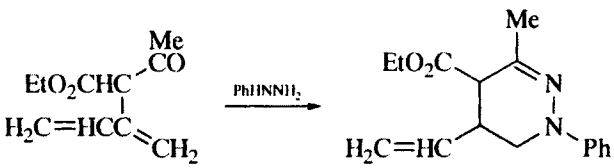

(**153**) (**154**)

1,4-Bis(methanesulfonyloxy)butane and dimethyl $\alpha,\beta$-hydrazinedicarboxylate gave dimethyl hexahydro-1,2-pyridazinedicarboxylate (NaH, $MeOCH_2CH_2OCH_2CH_2OMe$, $N_2$, reflux, 48 h: 30%).[616]

Also other examples.[324,468,1845]

### With Appropriate Alkenones or the Like

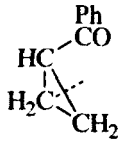
Ethyl 2-acetyl-3-methylenepent-4-enoate (**155**) and phenylhydrazine gave ethyl 3-methyl-1-phenyl-6-vinyl-1,4,5,6-tetrahydro-4-pyridazinecarboxylate (**156**) (45%).[950]



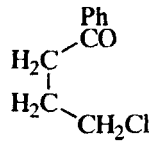

(**155**) (**156**)

Benzoylcyclopropane (**157**) and phenylhydrazine gave 1,3-diphenyl-1,4,5,6-tetrahydropyridazine (HCl gas, EtOH, reflux, 4 h: 26%);[743,1195] 1-benzoyl-3-chloropropane (**158**) with phenylhydrazine gave the same product (AcONa, AcOH–MeOH, reflux, 3 h 75%).[1541,1940]

Also other examples in the foregoing references and elsewhere.[109,1573]



(157)



(158)

### With Appropriate Dialdehydes or Aldehydo Ketones

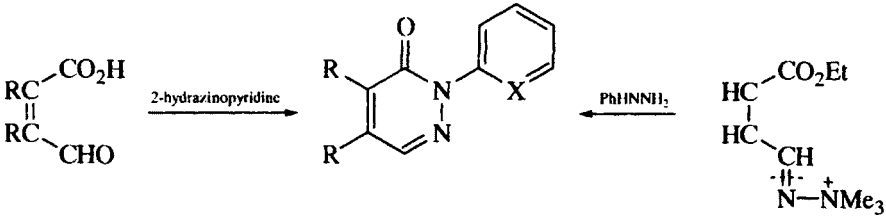
A brief general study has been reported.[561]

### With Appropriate Alkenoic Acids or the Like

Available examples lack relevant details.[1606,1849]

### With Appropriate Aldehydo Acids or Esters

Chloromucic (**159**, R = Cl) or bromomucic acid (**159** R = Br) and 2-hydrazinopyridine gave 4,5-dichloro- (**160**, R = Cl, X = N) or 4,5-dibromo-3-pyridine-2′-yl-3(2*H*)-pyridazinone (**160**, R = Br, X = N) ($H_2O$, 25°, 12 h: 88% or 91%, respectively).[1234]



(159) (160) (161)

Ethyl 4-dimethylhydrazonocrotonate methiodide (**161**) and phenylhydrazine gave 2-phenyl-3(2*H*)-pyridazinone (**160**, R = H, X = CH) (AcOH, then AcONa, $Ac_2O$, reflux: 70%).[290]

Bromomucic acid (**159**, R = Br) and 3-hydrazinopropionitrile gave 4,5-dibromo-2-β-cyanoethyl-3(2*H*)-pyridazinone (AcOH, 38°, 90 min: 96%).[587]

Also other examples.[217,591,892,916,940]

### With Appropriate Diketones

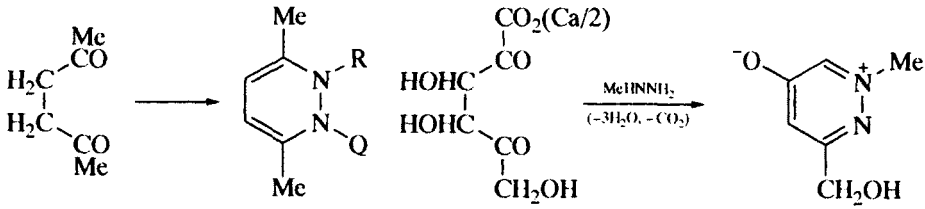
2,5-Hexanedione (**162**) was converted into 1,3,6-trimethyl-1,4-dihydropyridazine or its 1,2-dihydro isomer (**163**, R = Me, Q = H) ($MeHNNH_2$, $C_6H_{14}$, BaO, reflux, $H_2O$ removal, 3 h: 50%),[1158] into 3,6-dimethyl-1-*o*-methylaminobenzoyl-1,2-dihydropyridazine [**163**, R = C(:O)$C_6H_4$NHMe-*o*, Q = H] [$H_2NNHC(:O)C_6H_4NHMe$-*o*, $NEt_3$, reflux, 15 min: 85%],[1301]

or into 1,2,3,6-tetramethyl-1,2-dihydropyridazine (**163**, R = Q = Me) (MeHNNHMe, $ZnCl_2$, $C_6H_{14}$, reflux, 4 h: 36%).[1156]

1,2-Dibenzoylethane and semicarbazide hydrochloride gave 3,6-diphenylpyridazine (MeOH, reflux, 45 min: 50%; oxidation provided by loss of $NH_3$ and $CO_2$).[581]

Calcium 2,5-diketo-D-gluconate (**164**) with methylhydrazine gave 6-hydroxymethyl-2-methylpyridazinium-4-olate (**165**) (pyridine–AcOH–$H_2O$, 90°, 2 h: ~35%).[781]

Also other examples in the foregoing references and elsewhere.[986,1078,2099]

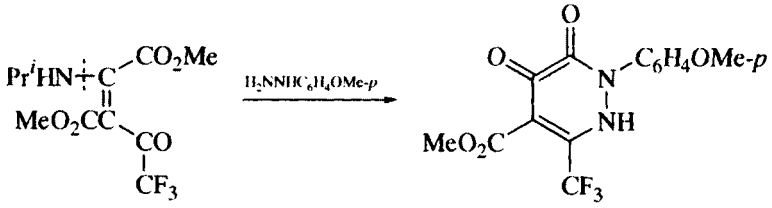



(162) (163) (164) (165)

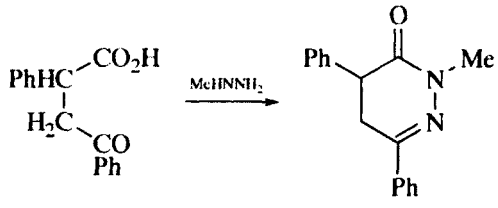
## With Appropriate Keto Acids or Esters

Dimethyl 2-isopropylamino-3-trifluoroacetylbutenedioate (**166**) and *p*-methoxyphenylhydrazine gave methyl 1-*p*-methoxyphenyl-5,6-dioxo-3-trifluoromethyl-1,2,5,6-tetrahydro-4-pyridazinecarboxylate (**167**) ($Et_2O$, 25°, 12 h: 68%, initially as the isopropylamine salt).[1696]

3-Benzoyl-2-phenylpropionic acid (**168**) and methylhydrazine gave 2-methyl-4,6-diphenyl-4,5-dihydro-3(2*H*)-pyridazinone (**169**) (BuOH, reflux, 6 h: 45%) and then its didehydro analogue ($Br_2$, AcOH, 70°, 3 h: 93%).[1082]




(166) (167)




(168) (169)

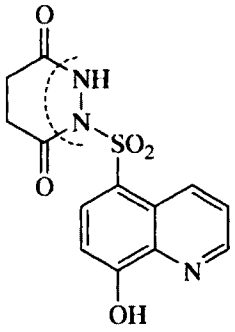
Levulinic acid (4-oxovaleric acid) and phenylhydrazine gave 6-methyl-2-phenyl-4,5-dihydro-3(2*H*)-pyridazinone (neat, reflux or 170°, 6 h: 95% or 44%, respectively).[496,691]

Also other examples in the foregoing references and elsewhere.[361, 535, 566, 590, 592, 613, 661, 735, 771, 800, 906, 915, 926, 928, 934, 951, 978, 979, 983, 991, 995, 1026, 1027, 1041, 1075, 1179, 1257, 1281, 1400, 1416, 1422, 1437, 1450, 1462, 1876]

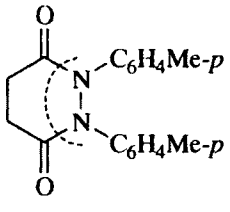
### With Appropriate Dicarboxylic Acids or Derivatives

Dimethyl succinate and 8-hydroxyquinoline-5-sulfonohydrazide gave 5-(3,6-dioxohexahydropyridazin-1-yl)sulfonylquinolin-8-ol (**170**) (AcONa, neat, 200–220°, 25 min, then EtOH, reflux, 3 h: 65%).[1393]

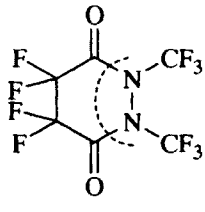
Succinyl dichloride and *p*-hydrazotoluene gave 1,2-di-*p*-tolyl-4,5-dihydro-3,6(1*H*,2*H*)-pyridazinedione (**171**) (PhMe, pyridine 4-$Me_2N$-pyridine, 25°, 6 h: 53%);[27] *p*-azotoluene has been reduced electrolytically in situ for the condensation (70% yield).[468]



(170)



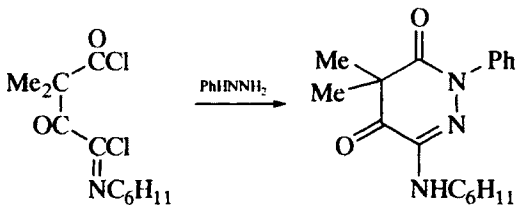
(171)



(172)

Tetrafluorosuccinyl difluoride and tetrafluoroformaldazine ($F_2C{=}NN{=}CF_2$) gave 4,4,5,5-tetrafluoro-1,2-bistrifluoromethyl-4,5-dihydro-3,6(1*H*,2*H*)-pyridazinedione (**172**) (neat, *hν*, 25°, 24 h: 67%).[631]

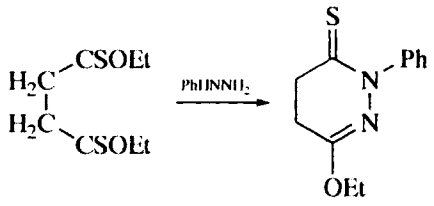
4-Chloro-4-cyclohexylimino-2,2-dimethyl-3-oxobutyryl chloride (**173**) with phenylhydrazine gave 6-cyclohexylamino-4,4-dimethyl-2-phenyl-4,5-dihydro-3,5(2*H*)-pyridazinedione (**174**) ($Et_2O$, 0° → 25°, 15 h: 61%).[50]



(173) (174)

*O,O*-Diethyl thiosuccinate (**175**) and methylhydrazine interestingly gave 6-ethoxy-2-methyl-4,5-dihydro-3(2*H*)-pyridazinethione (**176**).[1015]

Also other examples.[324, 1845, 1882, 2057]




(**175**) (**176**)

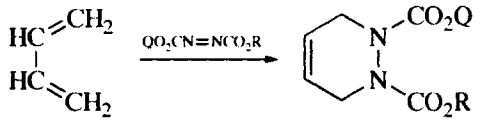
1.2.2.1.3 Using Dialkyl Azodicarboxylates as N1–N2 Synthons

The condensation of dialkyl azodicarboxylates with appropriate alkadienes or related compounds proceeds by a Diels–Alder mechanism, as illustrated in the following example:

**With Simple Dienes**

Buta-1,3-diene (**177**) and dimethyl azodicarboxylate gave dimethyl 1,2,3,6-tetrahydro-1,2-pyridazinedicarboxylate (**178**, Q = R = Me) (PhH, reflux, 3 h: 83%;[1097] PhH, 50°, 2 h, then 25°, 2 days: 88%);[405] diethyl azodicarboxylate gave the diethyl homologue (**178**, Q = R = Et) (PhH, reflux, 24 h: 98%;[1211] PhH, 25°, 2 days: 98%;[622] also less satisfactory variations);[405, 629] and benzyl ethyl azodicarboxylate gave the benzyl ethyl homologue (**178**, Q = $CH_2Ph$, R = Et) (PhH, 25° 24 h: 98%).[741]

1-Phenylbuta-1,3-diene and diethyl azodicarboxylate gave diethyl 3-phenyl-1,2,3,6-tetrahydro-1,2-pyridazinedocarboxylate (PhH, reflux, $N_2$, 4 h: 54%).[1329]

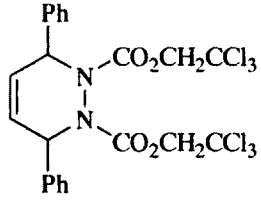



(**177**) (**178**)

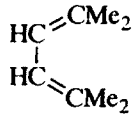
1,4-Diphenylbuta-1,3-diene and bis-β,β,β-trichloroethyl azodicarboxylate gave bis-β,β,β-trichloroethyl 3,6-diphenyl-1,2,3,6-tetrahydro-1,2-pyridazinedicarboxylate (**179**) (PhMe, 85°, 3 h: 91%).[341]

2,5-Dimethylhexa-2,4-diene (**180**) and diethyl azodicarboxylate gave diethyl 3,3,6,6-tetramethyl-1,2,3,6-tetrahydro-1,2-pyridazinedicarboxylate (PhMe, reflux, 7 days: 26%).[7]

Also other examples: [274, 281, 612, 869, 1146, 1424; cf. 246]



(179)



(180)

## With Dienes Bearing Functional Groups

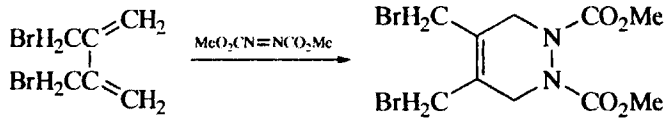
2,3-Bisbromomethylbuta-1,3-diene (**181**) and dimethyl azodicarboxylate gave dimethyl 4,5-bisbromomethyl-1,2,3,6-tetrahydro-1,2-pyridazinedicarboxylate (**182**) (PhH, reflux, 1 h: 63%).[323]

2-Trimethylsiloxybuta-1,3-diene and dimethyl azodicarboxylate gave dimethyl 4-trimethylsiloxy-1,2,3,6-tetrahydro-1,2-pyridazinedicarboxylate ($CHCl_3$, exothermic reaction; then 20°, 60 min: 83%).[1488]

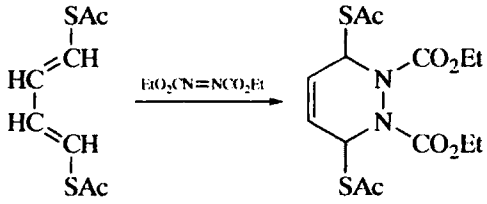
Methyl penta-2,4-dienoate and diethyl azodicarboxylate gave diethyl 3-methoxycarbonyl-1,2,3,6-tetrahydro-1,2-pyridazinedicarboxylate ($Et_2O$, trace hydroquinone, dark, 25°, 12 h; then reflux, 2 h: 84%).[1342]

1,4-Bisacetylthiobuta-1,3-diene (**183**) and diethyl azodicarboxylate gave diethyl 3,6-bisacetylthio-1,2,3,6-tetrahydro-1,2-pyridazinedicarboxylate (**184**) (PhMe, reflux, $N_2$, 10 days: 70%).[108]

Also other examples.[19,20,121,276,301,331,344,349,734,1013,1564,1699,1888,1921,1923]



(181) (182)



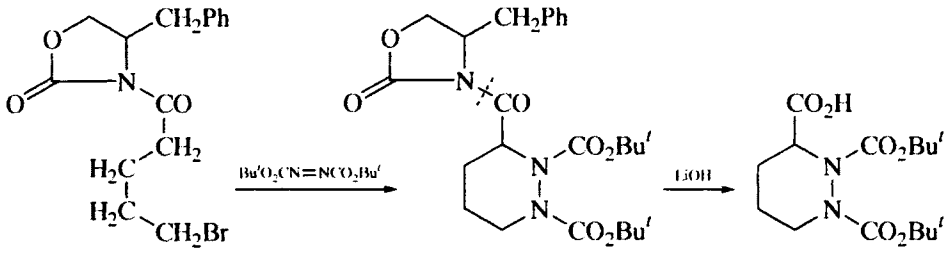
(183) (184)

## With Pseudodienes

*Note*: These reactions resemble, in effect, the foregoing Diels–Alder cyclizations, but their mechanism has been described [1803] as "electrophilic hydrazination–nucleophilic cyclization".

4-Benzyl-3-ε-bromovaleryloxazolidin-2-one (**185**) and di-*t*-butyl azodicarboxylate gave di-*t*-butyl 3-(4-benzyl-2-oxooxazolidin-3-ylcarbonyl)hexahydro-1,2-pyridazinedicarboxylate (**186**) ($Pr^i_2NLi$, THF, −78°, 30 min; then $Bu^tO_2CN{=}NCO_2Bu^t$, $CH_2Cl_2$, −78°, 30 min; then 1,3-dimethyl-3,4,5,6-tetrahydro-2(1*H*)-pyrimidinone, −78°, 50 min: 63%) and then 1,2-di-*t*-butoxycarbonylhexahydro-3-pyridazinecarboxylic acid (**187**) (LiOH, THF-$H_2O$, 0°, 2 h: 89%).[1803]

Also other examples.[1584]

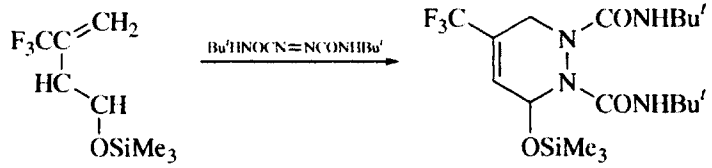



(**185**) (**186**) (**187**)

### 1.2.2.1.4 Using Miscellaneous N1–N2 Synthons

Several types of azo compound akin to the foregoing esters have been used, mainly as dienophilic N1–N2 synthons, to produce hydropyridazines, as illustrated in the following examples:

2-Trifluromethyl-4-trimethylsiloxybuta-1,3-diene (**188**) and *N,N'*-di-*t*-butylazodicarboxamide gave *N,N'*-di-*t*-butyl-4-trifluoromethyl-6-trimethylsiloxy-1,2,3,6-tetrahydro-1,2-pyridazinedicarboxamide (**189**) (PhMe, reflux, 18 h: 33%).[1838]



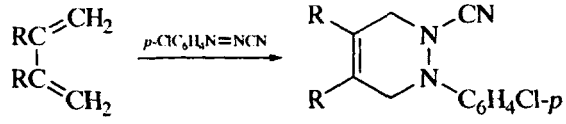

(**188**) (**189**)

Buta-1,3-diene (**190**, R = H) or its 2,3-dimethyl derivative (**190**, R = Me) and *p*-chlorobenzenediazocyanide gave 2-*p*-chlorophenyl-1,2,3,6-tetrahydro-1-pyridazinecarbonitrile (**191**, R = H) (neat, 25°, 5 days: 85%) or its 4,5-dimethyl derivative (**191**, R = Me) (neat, 100°, 3 h: 90%), respectively;[488,621] some kinetic work on such reactions has been reported.[271]

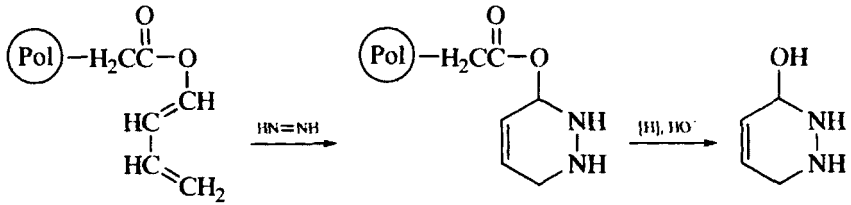
The polymer-attached buta-1,3-dienyl acetate (**192**) even reacted with diimine (HN=NH; prepared in situ from $H_2NNH_2{\cdot}HCl$ and $H_2O_2$ at 25°)

to give the polymer-attached pyridazine (**193**), which was reduced and freed from the polymer by alkaline hydrolysis to afford hexahydro-3-pyridazinol (**194**) (18% overall).[264]

Also other examples.[621,1412,1961,2107] See also Section 2.1.11.



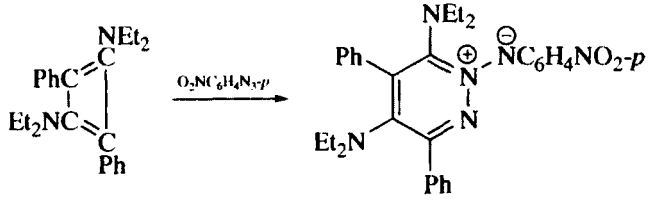
(190) (191)



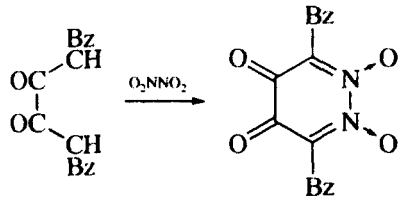
(192) (193) (194)

Aryl and aroyl azides have been used as N1–N2 synthons: for example, 1,3-bisdiethylamino-2,4-diphenylcyclobuta-1,3-diene (**195**) underwent ring fission and addition with *p*-nitrophenyl azide to afford a product formulated as 3,5-bisdiethylamino-4,6-diphenylpyridazine 2-*p*-nitrophenylimide (**196**).[427]

Dinitrogen tetroxide will also act as an N1–N2 synthon with appropriate dimethylene compounds. Thus 1,6-diphenyl-1,3,4,6-hexanetetrone (**197**) in ether was treated with ice-cold liquid $N_2O_4$ to afford, 3,6-dibenzoyl-4,5-dihydro-4,5-pyridazinedione 1,2-dioxide (**198**), an explosively unstable quinone, in 65% yield; several analogues were made similarly.[506]



(195) (196)



(197) (198)

### *1.2.2.2 Where the Two-Atom Synthon Provides N2 + C3*

No examples of such syntheses appear to have been reported in the period under review.

### *1.2.2.3 Where the Two-Atom Synthon Provides C3 + C4*

This large category of pyridazine syntheses is divided initially according to the nature of the C3–C4 synthon the subsequently subdivided (when necessary) according to the nature of the C5–C6–N1–N2 cosynthon.

#### 1.2.2.3.1 Using Ethylene Derivatives as C3–C4 Synthons

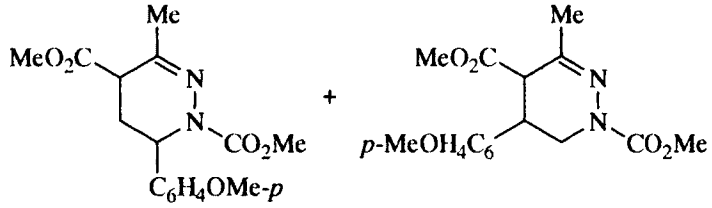
The following classified examples illustrate the scope and usual conditions for such syntheses; some are, in fact, self-condensations between two molecules of the same synthon:

**With Appropriate Azoalkenes**

Methyl 3-(methoxycarbonylazo)crotonate (199) and *p*-methoxystyrene gave a separable 3:2 mixture of dimethyl 6-*p*-methoxyphenyl- (**200**) and dimethyl 5-*p*-methoxyphenyl-3-methyl-1,4,5,6-tetrahydro-1,4-pyridazinedicarboxylate (**201**) ($Et_2O$, 25°, < 14 days: 67% before separation);[256] also analogous examples.[256, 396]



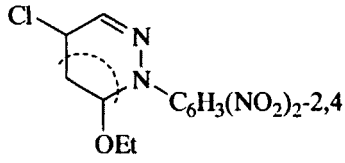

(199)



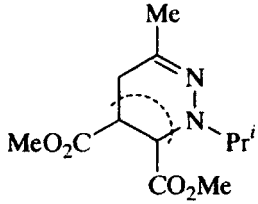

(200) (201)

α-Chloro-β-(2,4-dinitrophenylazo)ethylene and ethoxyethylene gave 4-chloro-1-(2,4-dinitrophenyl)-6-ethoxy-1,4,5,6-tetrahydropyridazine (**202**) (PhMe, 50°, sealed, 24 h: 77%);[739] also analogous cyclizations.[725, 1402]

2-Isopropylazopropene and dimethyl fumarate gave dimethyl 2-isopropyl-6-methyl-2,3,4,5-tetrahydro-3,4-pyridazinedicarboxylate (**203**) ($CH_2Cl_2$, 60°, sealed, 4 days: 60%).[559]



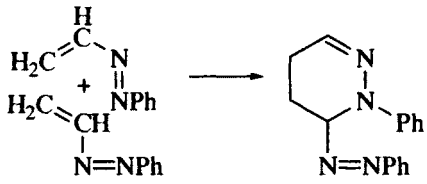
(202)



(203)

Freshly prepared phenylazoethylene (**204**) self-condensed to afford 1-phenyl-6-phenylazo-1,4,5,6-tetrahydropyridazine (**205**) (neat, 25°, 3 days: 59%);[425] such self-condensations have been studied in some detail.[68,70,75,78,80,425,482]

Also other examples.[71,429,1507]



(204) (205)

## With Appropriate Hydrazonoalkyl Halides

β-Bromo-α-semicarbazonoethylbenzene (**206**) and *N*-prop-1-enylmorpholine gave 5-methyl-6-morpholino-3-phenyl-1,4,5,6-tetrahydro-1-pyridazinecarboxamide (**207**) ($Et_3N$, $CHCl_3$, 25°, 48 h: 67%).[394]

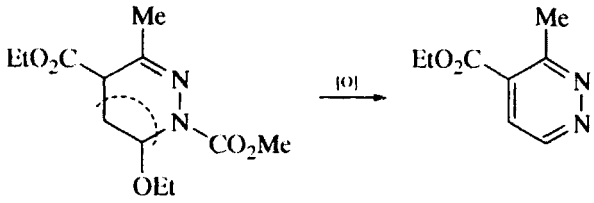


(206) (207)

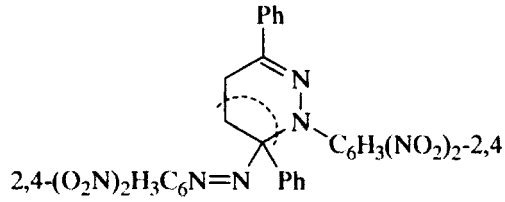
Ethyl 2-chloro-3-methoxycarbonylhydrazonobutyrate and ethoxyethylene gave methyl 6-ethoxy-4-ethoxycarbonyl-3-methyl-1,4,5,6-tetrahydro-1-pyridazinecarboxylate (**208**) ($Pr^i_2O$, $NaHCO_3$, then $EtOCH{=}CH_2$, 25°, 4 days: 84%) and then ethyl 3-methyl-4-pyridazinecarboxylate (**209**) ($Br_2$, AcOH, then $NaHCO_3$: 71%; note oxidative reactions etc.);[1678] also analogous syntheses.[1120,1952]

β-Chloro-α-(2,4-dinitrophenylhydrazono)ethylbenzene underwent self-condensation to afford 1-(2,4-dinitrophenyl)-6-(2,4-dinitrophenylazo)-3,6-

diphenyl-1,4,5,6-tetrahydropyridazine (**210**) ($Na_2CO_3$, THF, 25°, 20 h: 95%).[697]



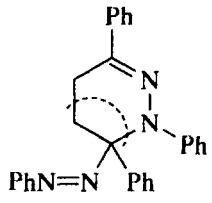
(**208**) (**209**)



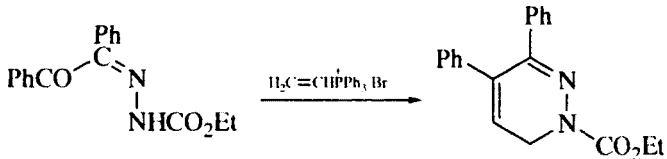
(**210**)

A similar self-condensation of β-(benzotriazol-1/2-yl)-α-(phenylhydrazono)ethylbenzene (in which the usual halogeno has been replaced by benzotriazolyl as a leaving group) afforded 1,3,6-triphenyl-6-phenylazo-1,4,5,6-tetrahydropyridazine (**211**) (NaH, THF, 0°, then reflux, 2 h: 86%).[1637]

Also other examples.[585, 1237, 1545, 1611]



(**211**)



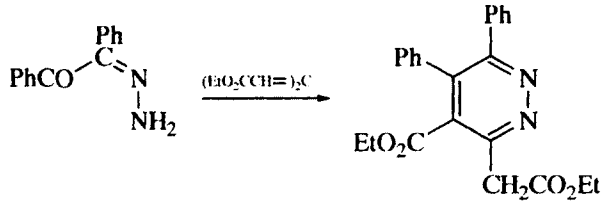
(**212**) (**213**)

## With Appropriate Hydrazonoalkyl Ketones

Benzil mono(ethoxycarbonylhydrazone) (**212**) and triphenylvinylphosphonium bromide gave ethyl 3,4-diphenyl-1,6-dihydro-1-pyridazinecarboxylate (**213**) (NaH, $Me_2NCHO$, 20°; then the bromide, 25°, 1 h: 74%).[293]

Benzil monohydrazone (**214**) and diethyl allene-1,3-dicarboxylate gave ethyl 3-ethoxycarbonylmethyl-5,6-diphenyl-4-pyridazinecarboxylate (**215**) (PhCl, 80°, 1 h; then KOBu$^t$, 0°, 10 min: 20%).[339]

Also other good examples using Wittig-type reactions.[17,269,1376,1822]

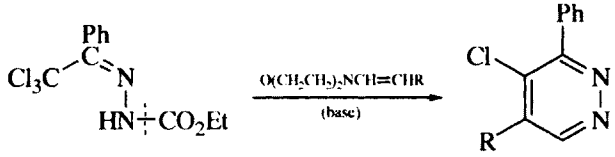


(214) (215)

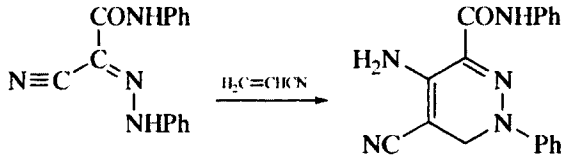
## With Miscellaneous C5–C6–N1–N2 Cosynthons

$\beta,\beta,\beta$-Trichloro-$\alpha$-(ethoxycarbonylhydrazono)ethylbenzene (**216**) and *N*-but-1-enyl- or *N*-(3-methylbut-1-enyl)morpholine gave 4-chloro-5-ethyl-3-phenylpyridazine (**217**, R = Et) or its 5-isopropyl homologue (**217**, R = Pr$^i$) (EtPr$^i_2$N, $CH_2Cl_2$, reflux: 16% or 36%, respectively, in each case after separation from another product).[1802]

2-Cyano-*N*-phenyl-2-phenylhydrazonoacetamide (**218**) and acrylonitrile gave 4-amino-5-cyano-1,*N*-diphenyl-1,6-dihydro-3-pyridazinecarboxamide (**219**).[1002]



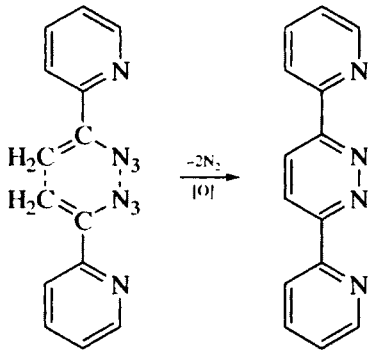
(216) (217)



(218) (219)

Freshly prepared 2-($\alpha$-azidovinyl)pyridine (**220**) underwent spontaneous loss of nitrogen at 25° to afford the corresponding nitrene, which promptly dimerized with subsequent oxidative cyclization in air to afford 3,6-di(pyridin-2-yl)pyridazine (**221**) (3%, after separation from another product).[944]

Also other examples.[2090]



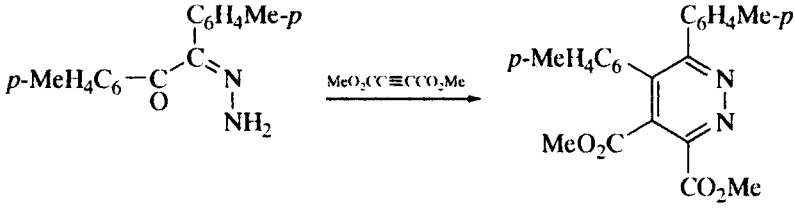
(220) (221)

### 1.2.2.3.2 Using Acetylene Derivatives as C3–C4 Synthons

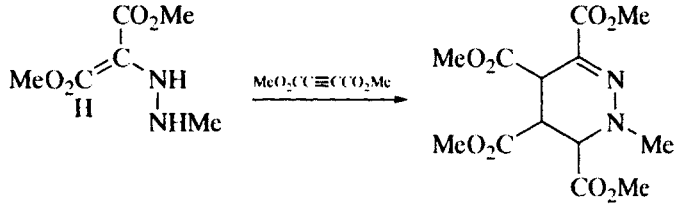
This category of synthesis has not been well developed although it does have considerable potential. Examples follow:

*p,p'*-Dimethylbenzil monohydrazone (**222**) and dimethyl acetylenedicarboxylate gave dimethyl 5,6-di-*p*-tolyl-3,4-pyridazinedicarboxylate (**223**) (neat, 125°, 30 min: 71%, after separation from a byproduct).[1242]

Dimethyl 2-(*β*-methylhydrazino)butenedioate (**224**) and dimethyl acetylenedicarboxylate gave a single stereoisomer (nmr evidence) of tetramethyl 1-methyl-1,4,5,6-tetrahydro-3,4,5,6-pyridazinetetracarboxylate (**225**) (MeOH–$H_2O$, – 8°, 24 h, then 25°, 16 h: 20%), which was subsequently converted into other stereoisomers;[24] the stereochemistry of two higher homologues was checked by X-ray analysis.[24]



(222) (223)



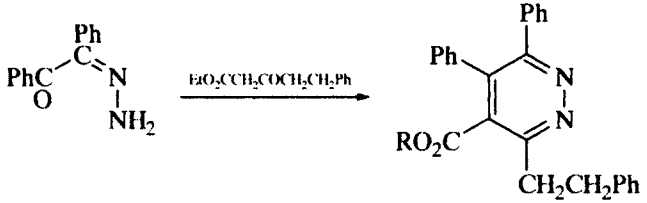
(224) (225)

1.2.2.3.3 Using Aliphatic Aldehydes or Ketones as C3–C4 Synthons

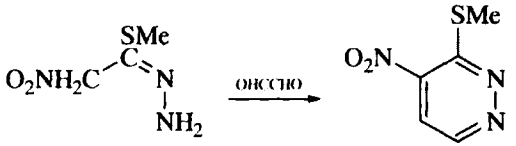
The usual conditions for such cyclizations are indicated in the following examples.

Benzil monohydrazone (**226**) and ethyl 3-oxo-5-phenylvalerate gave the ester (**227**, R = Et), conveniently isolated as 3-phenethyl-5,6-diphenyl-4-pyridazinecarboxylic acid (**227**, R = H) (PhH, trace TsOH, reflux, water removal, 2 h; then KOH, EtOH–$H_2O$, reflux, 12 h: 70%).[330]

Methyl 2-nitrothioacetohydrazonate (**228**) and glyoxal gave 3-methylthio-4-nitropyridazine (**229**) (EtOH–$H_2O$, $Na_2CO_3$, 10°, 1 h: 30%); also analogues similarly.[259]



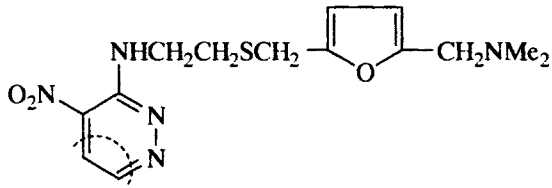
(**226**) (**227**)



(**228**) (**229**)

5-[β-(α-Hydrazino-β-nitrovinylamino)ethyl]thiomethyl-2-dimethylaminomethylfuran and glyoxal gave 3-[β-(5-dimethylaminomethylfuran-2-ylmethylthio)ethyl]amino-4-nitropyridazine (**230**) ($PhCH_2Me_3NOH$, $H_2O$–PhMe, 25°, 18 h: 80%).[1743]

Also other examples.[1822]



(**230**)

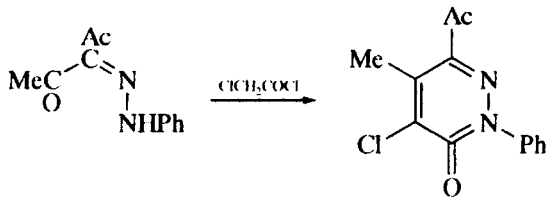
1.2.2.3.4 Using Aliphatic Acids or Acyl Chlorides as C3–C4 Synthons

Aliphatic acids or acyl chlorides have been used sparingly as C3–C4 synthons, although they do react satisfactorily, as illustrated in the following examples:

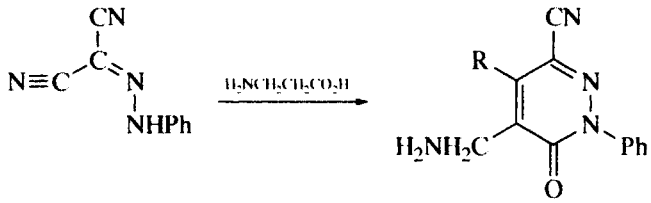
3-Phenylhydrazono-2,4-pentanedione (**231**) and choloroacetyl chloride gave 6-acetyl-4-chloro-5-methyl-2-phenyl-3(2*H*)-pyridazinone (**232**) (NaH, THF, 25°, 40 min; then the chloride, 20°, 6 h: 60%); also analogues likewise.[1620]

α-Phenylhydrazonomalononitrile (**233**) and β-alanine gave 5-aminomethyl-4-hydroxy-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarbonitrile (**234**, R = OH), presumably via hydrolysis of the 4-amino analogue (**234**, $R = NH_2$); also analogues similarly.[1444]

Also other examples.[1303, 2117, 2118]




(231) (232)




(233) (234)

### 1.2.2.3.5 Using Aliphatic Esters as C3–C4 Synthons

In contrast to the foregoing use of acids or acyl chlorides as C3–C4 synthons, aliphatic esters have been used extensively as illustrated in the following examples:

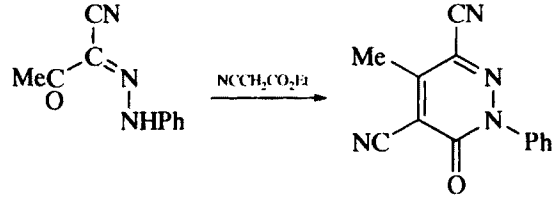
#### With Hydrazonoalkyl Ketones as Cosynthons

3-Oxo-2-phenylhydrazonobutyronitrile (**235**) and ethyl cyanoacetate gave 4-methyl-6-oxo-1-phenyl-1,6-dihydro-3,5-pyridazinedicarbonitrile (**236**) ($AcONH_4$, neat, 160°, 45 min: 80%; $AcONH_4$, AcOH, PhH, reflux, water removal, 20 h: 80%);[1217] analogues.[424, 1719, 1881]

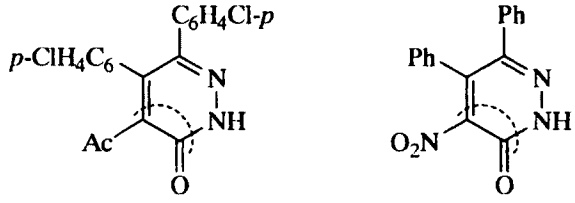
*p*,*p*′-Dichlorobenzil monohydrazone and methyl acetoacetate gave 4-acetyl-5,6-bis-*p*-chlorophenyl-3(2*H*)-pyridazinone (**237**) (xylene, pyridine, reflux, 21 h: ?%).[156]

Benzil monohydrazone and ethyl nitroacetate gave 4-nitro-5,6-diphenyl-3(2*H*)-pyridazinone (**238**)[$(CH_2)_5NH$, 105°: 45%].[795]

Also other examples.[395, 982, 1350, 1351, 1411, 1448, 1618, 2014]



(235) (236)



(237) (238)

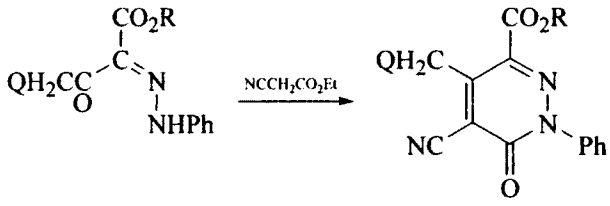
## With Hydrazono Keto Esters as Cosynthons

*Note*: In every case, the keto rather than the ester grouping takes part in the cyclization reaction.

Ethyl 2-phenylhydrazono-3-oxobutyrate (**239**, Q = H, R = Et) and ethyl cyanoacetate gave ethyl 5-cyano-4-methyl-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate (**240**, Q = H, R = Et) ($AcONH_4$, neat, 160°, 30 min: 90%;[52,379,1216] similarly but 140°: 71%).[1240]

Methyl 3-oxo-2-phenylhydrazonovalerate (**239**, Q = R = Me) and ethyl cyanoacetate gave methyl 5-cyano-4-ethyl-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate (**240**, Q = R = Me) ($AcONH_4$, AcOH, PhH, reflux, water removal, 6 h: 85%).[1808] Such reactions are assisted by microwave irradiation under solvent-free conditions.[2043]

Also other examples.[1350,1351,1420,1440]



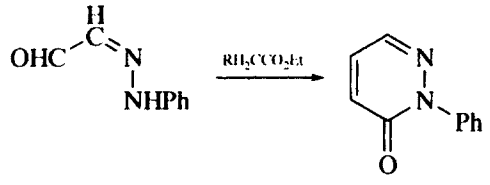
(239) (240)

## With Miscellaneous Cosynthons

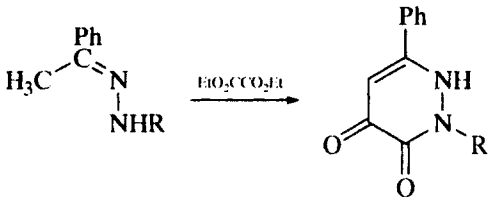
Glyoxal monophenylhydrazone (**241**) and ethyl 2-(diethylphosphono)acetate gave 2-phenyl-3(2*H*)-pyridazinone (**242**) [NaH, THF, 25°, 2 h; then (**241**), 25° → 65°, 10 h: 71%];[1350] replacement of the phosphono reagent by ethyl

2-trimethylsilylacetate gave the same product (**242**) [$(C_6H_{11})_2NLi$, THF, − 78°; then (**241**), − 78° → 25°, 3 h: 74%].[1351] Also related examples.[1877]

α-Hydrazonoethylbenzene (**243**, R = H), as its *N,N,β*-trilithio derivative (made in situ), and diethyl oxalate gave 6-phenyl-3,4(1*H*,2*H*)-pyridazinedione (**244**, R = H) (THF, 0°, 30 min; then HCl, reflux, 1 h: 24%); α-(phenylhydrazono)ethylbenzene (**243**, R = Ph), as its *N,β*-dilithio derivative, likewise gave 2,6-diphenyl-3,4(1*H*,2*H*)-pyridazinedione (**244**, R = Ph) (72%).[942]



(**241**) (**242**)

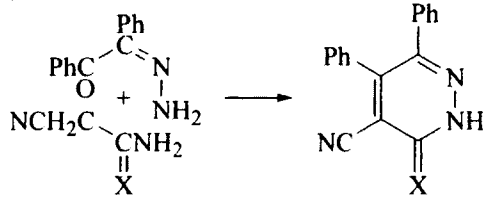


(**243**) (**244**)

### 1.2.2.3.6 Using Aliphatic Amides or Nitriles as C3–C4 Synthons

Substituted acetamides and acetonitriles have been used as C3–C4 synthons, the first sparingly but the second less so. When both functional groups are present, as in cyanoacetamide, the amide grouping appears to take part preferentially in any cyclization. The following examples illustrate conditions and yields:

Benzil monohydrazone (**246**) and cyanoacetamide (**245**, X = O) gave 3-oxo-5,6-diphenyl-2,3-dihydro-4-pyridazinecarbonitrile (**247**, X = O) (pyridine: 70%);[1398, 1726] similar use of cyano (thioacetamide) (**245**, X = S) gave 5,6-diphenyl-3-thioxo-2,3-dihydro-4-pyridazinecarbonitrile (**247**, X = S) in unstated yield.[1390]



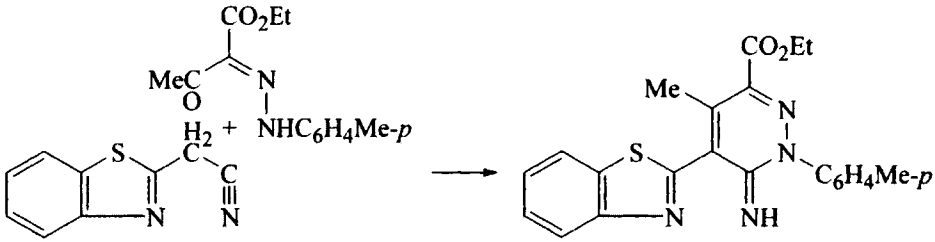
(**245**) (**246**) (**247**)

Ethyl 3-oxo-2-*p*-tolylhydrazonobutyrate (**249**) and 2-cyanomethylbenzothiazole (**248**) gave ethyl 5-benzothiazol-2′-yl-6-imino-4-methyl-1-*p*-tolyl-1,6-dihydro-3-pyridazinecarboxylate (**250**) [$(CH_2)_5NH$, EtOH, reflux, 3 h: 55%].[518]

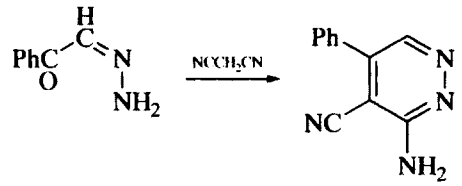
α-Hydrazonoacetophenone (**251**) and malononitrile gave 3-amino-5-phenyl-4-pyridazinecarbonitrile (**252**) [$Me_2SO$, $O(CH_2CH_2)_2NH$, 75°, 15 min: 79%].[364]

2-Phenylhydrazonomalononitrile (**253**) and malononitrile gave 4-amino-6-imino-1-phenyl-1,6-dihydro-3,5-pyridazinedicarbonitrile (**254**) (pyridine, reflux, 10 h: 80%).[1210]

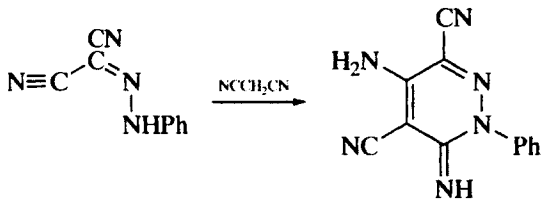
Also other examples in the foregoing references and elsewhere.[713,729,899,911,1663,1665,1702,1829,1862,1864,1960]



(248) (249) (250)



(251) (252)

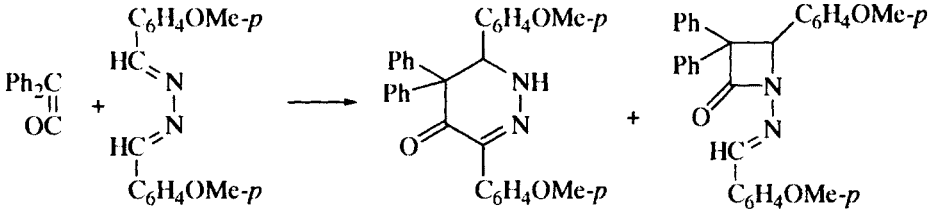


(253) (254)

### *1.2.2.4 Where the Two-Atom Synthon Provides C4 + C5*

This potentially large category of syntheses is represented by very few examples. *p*-Anisaldehyde azine (**256**) reacted with diphenyl ketene (**255**) in refluxing xylene under argon during 6 h to afford two separable products: 3,6-

bis-*p*-methoxyphenyl-5,5-diphenyl-5,6-dihydro-4(1*H*)-pyridazinone (**257**) (20% yield) and the isomeric 1-*p*-methoxybenzylideneamino-4-*p*-methoxyphenyl-3,3-diphenyl-2-azetidinone (**258**) (60%).[1384] The condensation of hexafluoroacetone azine $[(F_3C)_2C{=}N{-}N{=}C(CF_3)_2]$ with 2-methylpropene gave 4,4-dimethyl-3,3,6,6-tetrakistrifluoromethyl-3,4,5,6-tetrahydropyridazine (70°, < 24 h: 91%) and several homologues were also so made.[455]




### 1.2.3 By using two 3-Atom Synthons

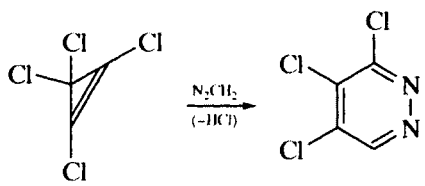
Because of the symmetry of pyridazine, there can be only two possibilities within this category: an N–N–C synthon plus a C–C–C synthon or two N–C–C synthons (which may be the same or different). The first of these combinations has been used fairly frequently, but the second has been employed only rarely.

#### *1.2.3.1 Where the Synthons Provide N1–N2–C3 and C4–C5–C6*

Most such reported syntheses have involved the condensation of diazoalkanes or derivatives thereof with cyclopropene derivatives, but a few have involved hydrazine derivatives with acyclic C–C–C synthons. The following examples illustrate the present state of development:

**With Diazoalkanes as N–N–C Synthons**

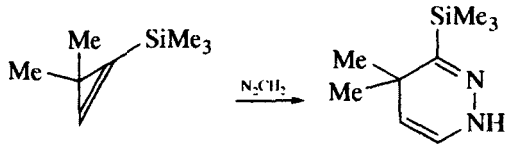
Tetrachlorocyclopropene (**259**) gave 3,4,5-trichloropyridazine (**260**) [$CH_2N_2$, $Et_2O$, $Pd(OAc)_2$, 0° → 20°, until $N_2\uparrow$ ceased: 70%].[573,727]



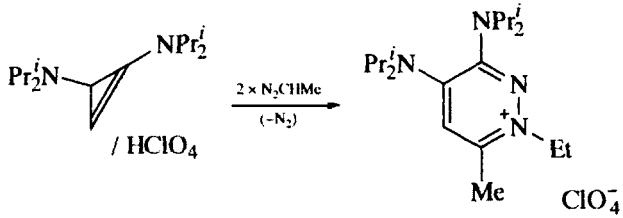

(**259**) (**260**)

3,3-Dimethyl-1-trimethylsilylcyclopropene (**261**) gave 4,4-dimethyl-3-trimethylsilyl-1,4-dihydropyridazine (**262**) ($CH_2N_2$, $Et_2O$, 0° → 20°, 1 h: 91%).[418] Also a somewhat analogous example.[311]

Bis(diisopropylamino)cyclopropene, as its perchlorate salt (**263**), gave 3,4-bis(diisopropylamino)-1-ethyl-6-methylpyridazinium perchlorate (**264**) (2 × $MeCHN_2$, $Et_2O$, 20°, 3 h: ~75%?).[31]



(**261**) (**262**)



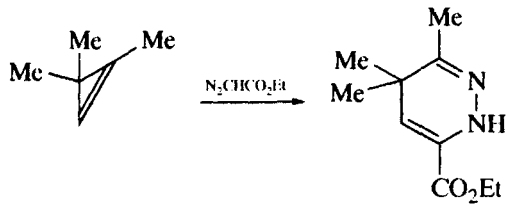
(**263**) (**264**)

### With Alkyl Diazoacetates as N–N–C Synthons

1,3,3-trimethylcyclopropene (**265**) gave ethyl 5,5,6-trimethyl-2,5-dihydro-3-pyridazinecarboxylate (**266**) ($N_2CHCO_2Et$, neat, 65°, 36 h: 59% crude, 10% purified);[562,657] 3,3-dimethylcyclopropene gave methyl 5,5-dimethyl-2,5-dihydro-3-pyridazinecarboxylate ($N_2CHCO_2Me$, neat, 25°, 2 weeks: < 30%).[320]

Trisdimethylaminocyclopropene hydrochloride (**267**) gave ethyl 4,5,6-trisdimethylamino-3-pyridazinecarboxylate (**268**) ($N_2AgCCO_2Et$, $CHCl_3$–$Et_2O$, 0°, 1 h: 60%).[8]

Also other examples.[9,440]

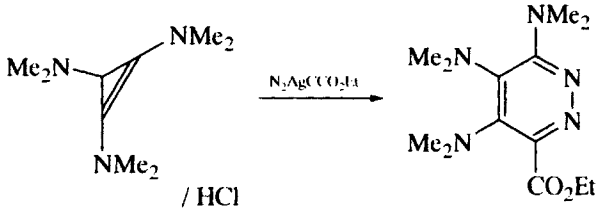


(**265**) (**266**)

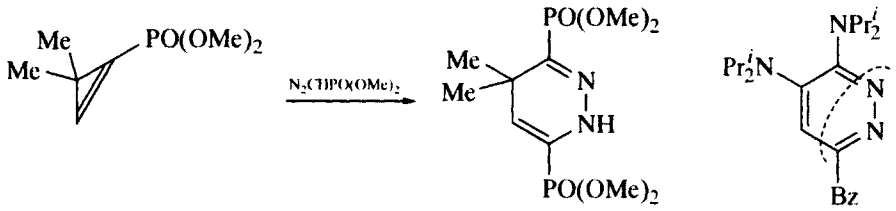
### With Other Substituted Diazoalkanes as N–N–C synthons

Dimethyl 3,3-dimethyl-1-cyclopentenephosphonate (**269**) and dimethyl diazomethanephosphonate gave tetramethyl 4,4-dimethyl-1,4-dihydro-3,6-pyridazinediphosphonate (**270**) ($Et_2O$, 20°, 4 h: 32%).[9]

Bis(diisopropylamino)cyclopropene perchlorate salt (**263**) with α-diazoacetophenone ($BzCHN_2$) gave 3-benzoyl-5,6-bisdiisopropylaminopyridazine (**271**) ($CH_2Cl_2$, 1,5-diazabicyclo[4,3,0]non-5-ene as base, 25°, 2 days: 47%);[1070] also analogues.[1070, 1123]



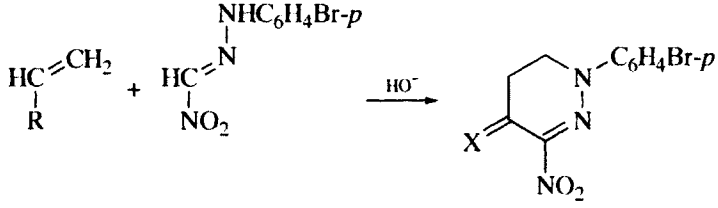
(267) (268)



(269) (270) (271)

## With Hydrazine Derivatives as N–N–C Synthons

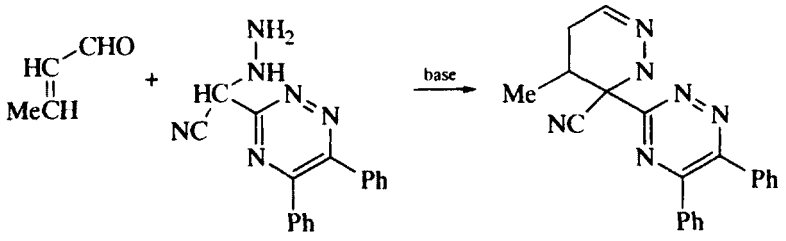
Acrylonitrile (**272**, R = CN) or acrylamide (**272**, $R = CONH_2$) with *N*-*p*-bromophenyl-*N'*-nitromethylenehydrazine (**273**) gave 1-*p*-bromophenyl-3-nitro-5,6-dihydro-4(1*H*)-pyridazinimine (**274**, X = NH) (0.6M NaOH, 25°, 5 days: 57%) or the corresponding pyridazinone (**274**, X = O) (0.6M NaOH, 25°, 10 days: 47%), respectively: also analogues,[565]



(272) (273) (274)

Crotonaldehyde (**275**) with 3-[*C*-cyano(hydrazinomethyl)]-5,6-diphenyl-1,2,4-triazine (**276**) gave 3-(5,6-diphenyl-1,2,4-triazin-3-yl)-4-methyl-2,3,4,5-tetrahydro-3-pyridazinecarbonitrile (**277**) [$(CH_2)_5NH$, EtOH, reflux, 8 h: 70%].[1708]

Also other examples.[1799]



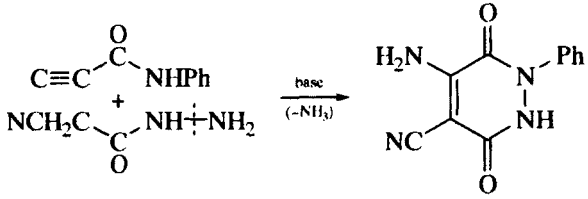
(275) (276) (277)

### *1.2.3.2 Where the Synthons Provide N2 + C3 + C4 and C5 + C6 + N1*

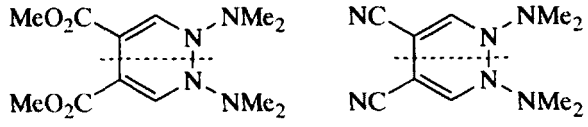
This underdeveloped category is represented by the few reported examples that are summarized below:

1-Cyanoformanilide (**278**) and 2-cyanoacetohydrazide (**279**) gave 5-amino-3,6-dioxo-1-phenyl-1,2,3,6-tetrahydro-4-pyridazinecarbonitrile (**280**) [$(CH_2)_5NH$, EtOH, warm, then 25°, 15 h: 68%].[232]

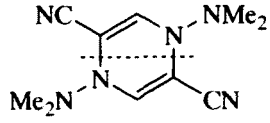
Methyl 3-(β,β-dimethylhydrazino)propionate underwent oxidative self-condensation to give only dimethyl 1,2-bisdimethylamino-1,2-dihydro-4,5-pyridazinedicarboxylate (**281**) (yellow HgO, $Na_2SO_4$, dioxane, reflux, 12 h: 72%);[1175] in contrast, 3-(β,β-dimethylhydrazino)propionitrile gave 1,2-bisdimethylamino-1,2-dihydro-4,5-pyridazinedicarbonitrile (**282**) (< 10%) accompanied by the isomeric pyrazine (**283**) (83%).[1175]



(278 / 279) (280)



(281) (282)



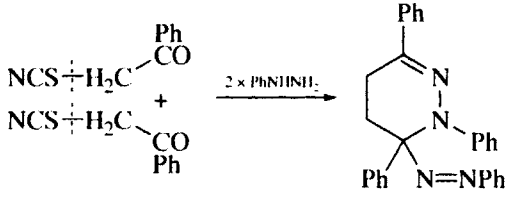
(283)

## 1.3 FROM THREE OR MORE SYNTHONS (*H* 30)

Of all the ways in which three synthons could provide the ring atoms of pyridazine, only two combinations are represented meagerly in the post-1970 literature. Syntheses employing more than three synthons are completely unrepresented.

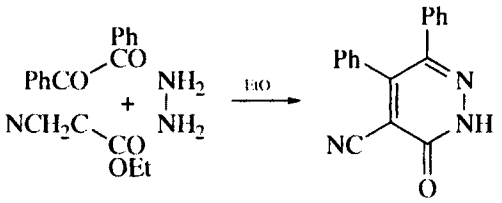
### 1.3.1 Where Three Synthons Provide N1 + N2, C3 + C4, and C5 + C6

The established reaction of α-bromoacetophenone with equimolecular phenylhydrazine in ethanolic sodium acetate to give 60% of 1,3,6-triphenyl-6-phenylazo-1,4,5,6-tetrahydropyridazine (**285**)[185] has been modified. Thus α-thiocyanatoacetophenone (**284**) and phenylhydrazine in ethanolic acetic acid at 25° for 30 min gave the same product (**285**) in 76% yield;[1396] the mechanism remains doubtful.



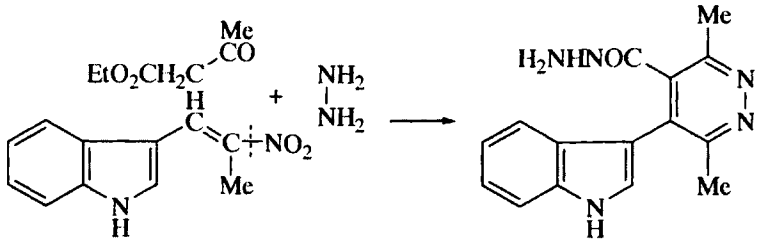

(**284**) (**285**)

Likewise, the three-component condensation, typified by that of ethyl cyanoacetate, benzil, and hydrazine hydrate in refluxing ethanolic ethoxide for 2 h to give 3-oxo-5,6-diphenyl-2,3-dihydro-4-pyridazinecarbonitrile (**286**),[1851] has been reinvestigated in respect of its mechanism.[314]




(**286**)

The condensation of 3-(2-nitroprop-1-enyl)indole (**287**), ethyl acetoacetate, and hydrazine hydrate in refluxing ethanol for 5 h has been used to make 5-indol-3′-yl-3,6-dimethyl-4-pyridazinecarbohydrazide (**288**) in 78% yield.[1709] The mechanism probably involved a Nef conversion[1852] of the nitropropenyl group into the corresponding ketone prior to condensation, as well as hydrazinolysis of the ester grouping; several analogues were made similarly.[1709] Somewhat

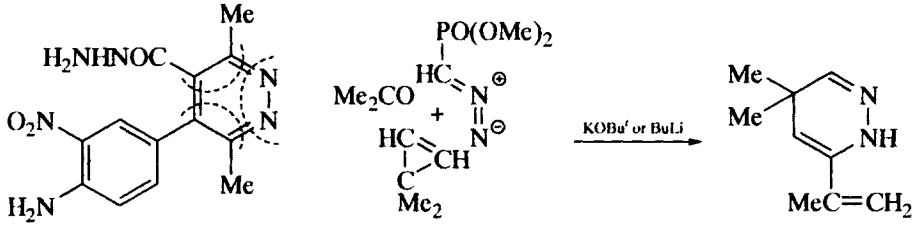


(287) (288)

similarly, 2-nitro-4-(2-nitroprop-1-enyl)aniline, methyl acetoacetate, and hydrazine in refluxing ethanol for 3 h gave 5-(4-amino-3-nitrophenyl)-3,6-dimethyl-4-pyridazinecarbohydrazide (**289**) in 70% yield.[1074]

### 1.3.2 Where Three Synthons Provide N1 + N2 + C3, C4 and C5 + C6

The only example in this category clearly involved the condensation of dimethyl diazomethanephosphonate (**291**), 3,3-dimethylcyclopropene (**290**),



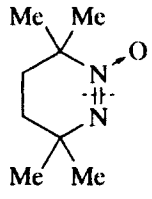
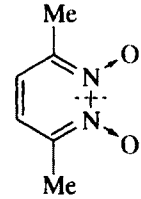
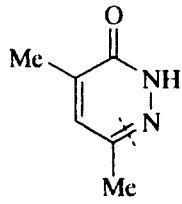
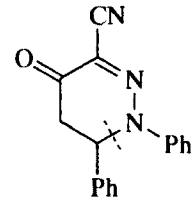
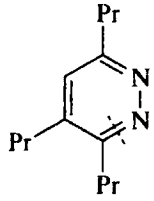
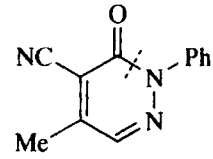
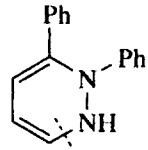
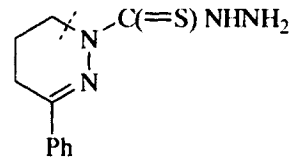
(289) (290) (291) (292)

and acetone (in the presence of potassium *t*-butoxide or butyllithium) to afford 3-isopropenyl-5,5-dimethyl-2,5-dihydropyridazine (**292**) in 5–10% yield, but no other experimental details were given.[619] However, several steps in the mechanism were deduced.[619]

## 1.4 APPENDIX: GLANCE INDEX TO TYPICAL PYRIDAZINES AVAILABLE FROM ALIPHATIC OR CARBOCYLIC SYNTHONS

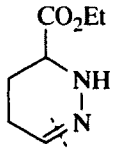
The following glance index has been added to assist in the choice of a primary synthesis that may afford a required type of pyridazine derivative from aliphatic or carbocyclic synthons. In using the index, it should be borne in mind that products analogous to those formulated can often be produced by minor changes in the synthon(s) involved: for example, by change of the halogeno substituent(s): by change, addition, or deletion of alkyl or aryl groups; by change or interchange of acid, ester, amide, nitrile, and such like groups; by

interchange of oxo, thioxo, and imino substituents; by interchange of alkoxy, aryloxy, alkylthio, and arylthio groups; and so on.

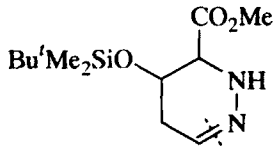
| Section | Typical Products |
|---|---|
| 1.1.1 |  85%  33% |
| 1.1.2.1 |  13%  91% |
| 1.1.2.2 |  58%  60% |
| 1.1.2.3 |  95%  73% |

| Section | Typical Products |
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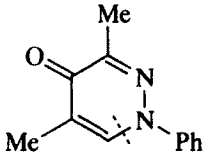



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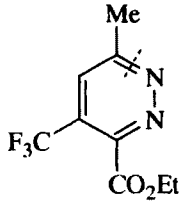



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1.1.2.5

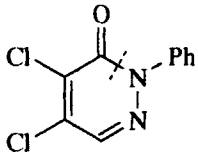



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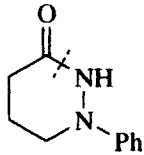



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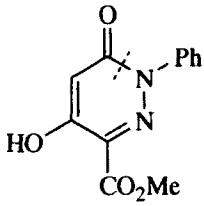



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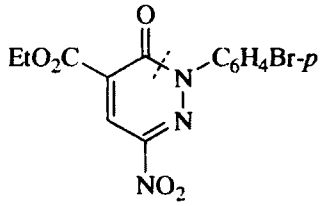



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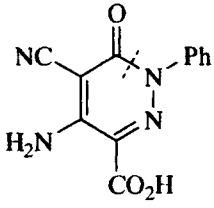



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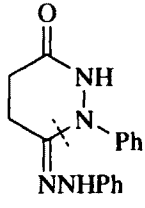



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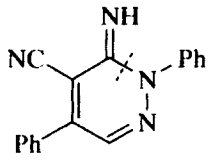

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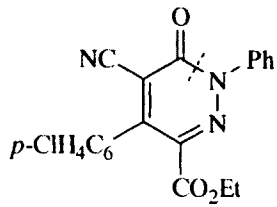

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| Section | Typical Products |
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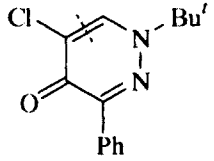


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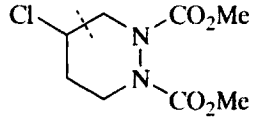


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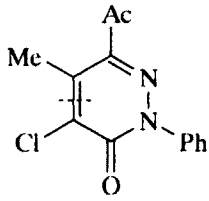


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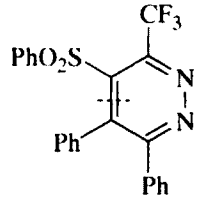


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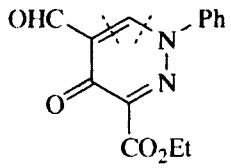


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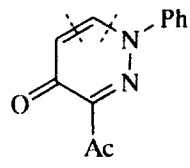


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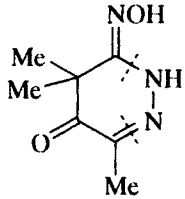


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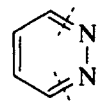


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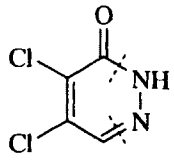
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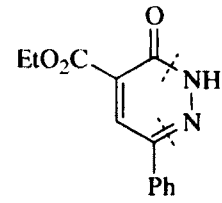
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| Section | Typical Products |
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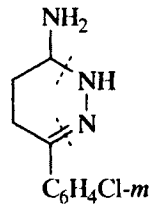
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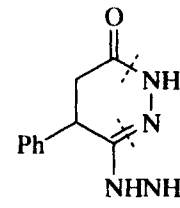
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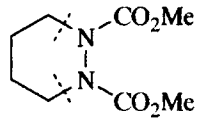


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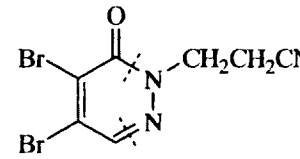


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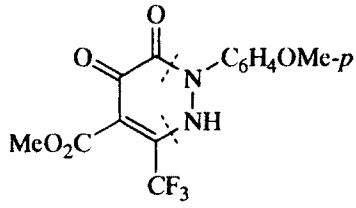
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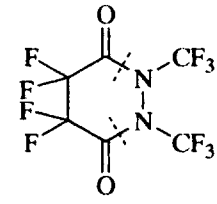
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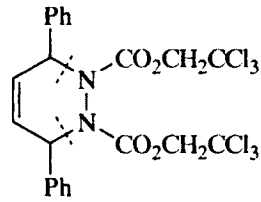


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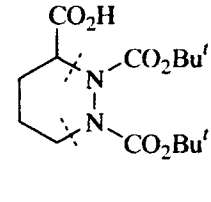


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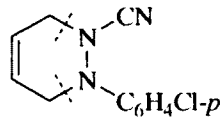
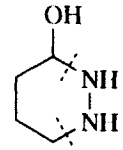
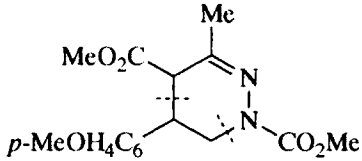
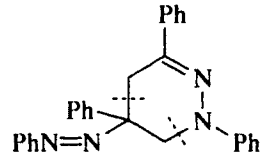
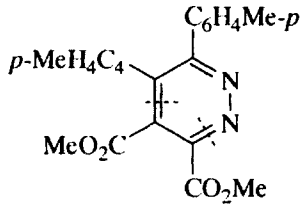
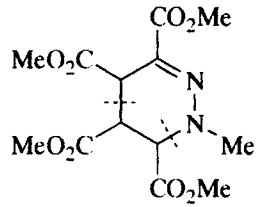
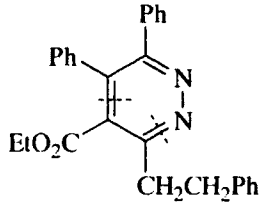
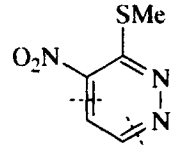
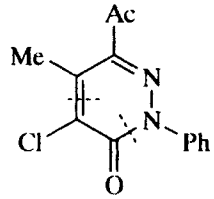
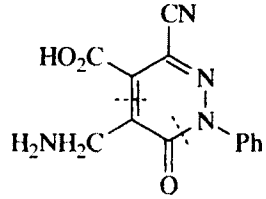
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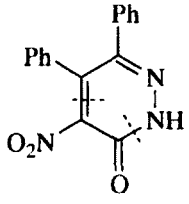
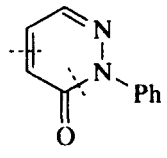
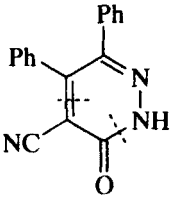
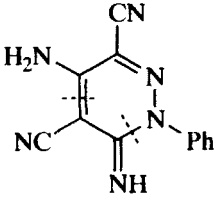
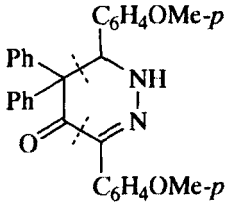
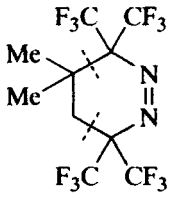
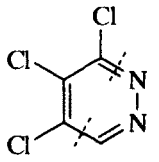
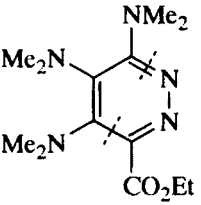
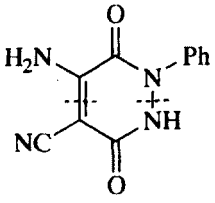
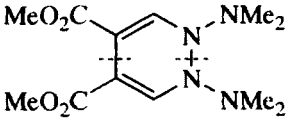


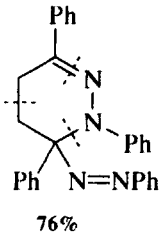
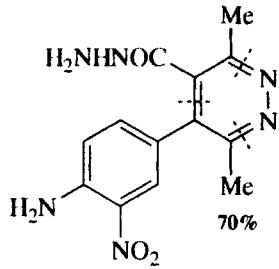
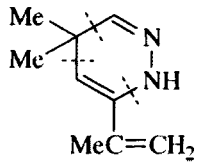
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D. J. Brown
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CHAPTER 2

# Primary Syntheses from Other Heterocyclic Systems

The primary synthesis of pyridazines from other heterocyclic systems has a large but diffuse literature that has never been reviewed as such in depth. However, many examples can be found in Castle's original book[1839] and in some subsequent reviews.[1188,1575,1624,1643,1844,1853,1916,2006,2011]

For pragmatic reasons, the present treatment of such post-1970 primary syntheses is divided according to the fundamental heterocyclic substrate, initially by ring-size and then in alphabetical order. It should be noted that cyclic anhydrides, imides, lactones, and other compounds are considered here as heterocycles for the purpose of classification: for example, syntheses involving maleic anhydride as a substrate will be found in the furan subsection. As in Chapter 1, a glance index is appended (Section 2.9).

## 2.1 PYRIDAZINES FROM THREE-, FOUR-, OR FIVE-MEMBERED HETEROCYCLIC SYSTEMS (*H* 32, 360, 408)

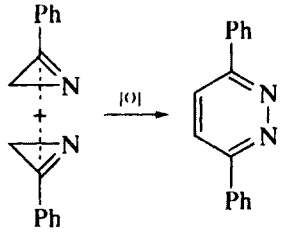
Very few pyridazines have been made from three- or four-membered heterocyclic substrates but five membered substrates have been used widely.

### 2.1.1 Azirines as Substrates

2-Phenylazirine (**1**) underwent oxidative self-condensation to afford 3,6-diphenylpyridazine (**2**) and other separable products ($TiCl_4$, $C_6H_4$, $-78°$, 1 h: 22%;[607] *N*-bromosuccinimide, TsOH, $-23° \rightarrow 20°$, $>1$ h: 13%).[409] Some simple derivatives were made similarly but in even lower yield.[607]

### 2.1.2 Furans as Substrates

The replacement of –O– in a furan by –N–N– to give a pyridazine usually occurs quite readily on treatment with hydrazine or the like, but intermediates are seldom observed or isolated. Most of the furans used to make pyridazines

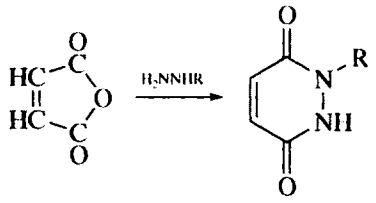


(1) (2)

are derivatives of maleic anhydride, succinic anhydride, or γ-lactones, as will be evident from the following typical examples:

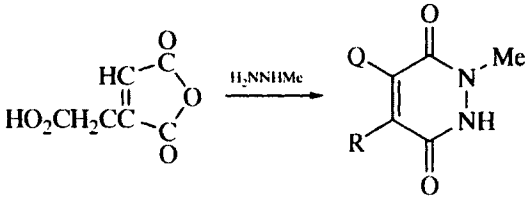
### Using Dihydro-2,5-furandiones (Maleic Anhydrides) as Substrates

Maleic anhydride (**3**) and hydrazine gave 3,6(1*H*,2*H*)-pyridazinedione (**4**, R = H) ($H_2NNH_2 \cdot HCl$, $H_2O$, reflux, 4–5 h: 95%;[1345] $H_2NNH_2 \cdot H_2SO_4$, similarly: 76–78%);[99,153,1098] likewise, ethylhydrazine gave 1-ethyl-3.6 (1*H*,2*H*)-pyridazinedione (4, R = Et) (AcOH, < 30°, 20 min: ~ 35%),[292] (β-hydroxyethyl)hydrazine gave 1-β-hydroxyethyl-3,6(1*H*,2*H*)-pyridazinedione (4, $R = CH_2CH_2OH$) ($H_2O$, 5°, ?h: 64%),[569] and phenylhydrazine gave 1-phenyl-3,6(1*H*,2*H*)-pyridazinedione (**4**, R = Ph) (AcOH, reflux, 1 h: 47%).[313]



(3) (4)

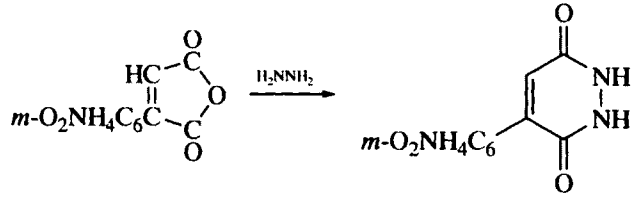
2-Carboxymethylmaleic anhydride (*cis*-aconitic anhydride) (**5**) and methylhydrazine gave a separable mixture of two isomers, 4-carboxy-methyl-1-methyl-(**6**, Q = H, $R = CH_2CO_2H$) and 4-carboxymethyl-2-methyl-3,6(1*H*,2*H*)-pyridazinedione (**6**, $Q = CH_2CO_2H$, R = H) ($H_2N$ NHMe, $MeOCH_2CH_2OMe$, reflux, 2 h: 44% and 19%, respectively; $H_2NNHMe$. $H_2SO_4$, $H_2O$, reflux, 20 min: 64% and 0%, respectively).[207]



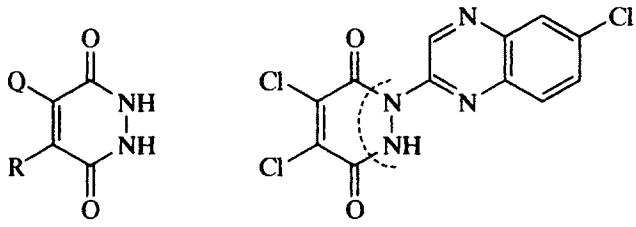
(5) (6)

2-*m*-Nitrophenylmaleic anhydride (**7**) and hydrazine gave 4-*m*-nitrophenyl-3,6(1*H*,2*H*)-pyridazinedione (**8**) ($H_2O$-$MeOCH_2CH_2OMe$, reflux, 3 h: 60%);[1186] the *o*-nitro isomer was made similarly (HCl, reflux?: 82%).[525]

2,3-Dichloromaleic anhydride and hydrazine hydrate gave 4,5-dichloro-3.6(1*H*,2*H*)-pyridazinedione (**9**, Q = R = Cl) (AcOH, 80°, 10 h: 92%);[802] 4-chloro-(**9**, Q = Cl, R = H),[802] 4-fluoro-(**9**, Q = F, R = H),[917] and 4-bromo-3,6(1*H*,2*H*)-pyridazinedione (**9**, Q = Br, R = H)[614,654] were made from appropriate 2-halogenomaleic anhydrides.




(**7**) (**8**)




(**9**) (**10**)

2,3-Dichloromaleic anhydride and 6-chloro-2-hydrazinoquinoxaline gave 4,5-dichloro-1-(6-chloroquinoxalin-2-yl)-3,6(1*H*,2*H*)-pyridazinedione (**10**) (AcOH, 100°, 3 h: 87%);[217] the 4,5-dibromo-1-*p*-chlorophenyl analogue was made in a broadly similar way (64% $H_2SO_4$: ~50%).[547]

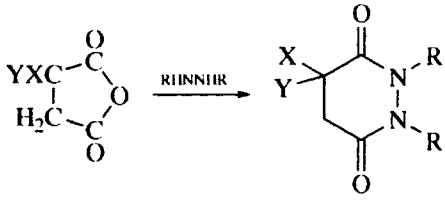
Also other examples.[461,670,726,871,917,943,957,987,1218,1397,1460,1776]

## Using Tetrahydro-2,5-furanediones (Succinic Anhydrides) as Substrates

2,2-Dimethylsuccinic anhydride (**11**, X = Y = Me) and *N,N'*-dimethylhydrazine gave 1,2,4,4-tetramethyl-4,5-dihydro-3,6(1*H*,2*H*)-pyridazinedione (**12**, R = X = Y = Me) (MeHNNHMe·2HCl, AcOH, reflux, 12 h: 22%;[93,cf.1854] similarly but then evaporate, xylene, reflux, 6 h, water removal: 64%).[93,cf.1855]

2-Phenylsuccinic anhydride (**11**, X = Ph, Y = H) and *N,N'*-diethylhydrazine gave 1,2-diethyl-4-phenyl-4,5-dihydro-3,6(1*H*,2*H*)-pyridazinedione (**12**, R = Et, X = Ph, Y = H) (THF, 20°, 12 h; then reflux, 6 h: 46%).[1328]

Succinic anhydride (**11**, X = Y = H) and phenylhydrazine gave 1-phenyl-4,5-dihydro-3,6(1*H*,2*H*)-pyridazinedione ($H_2NNH_2$, AcOH, reflux 4 h: 85%;



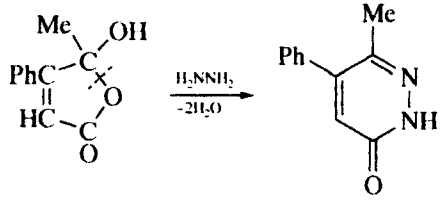
(11) (12)

$H_2NNH_2$, AcOH, microwave irradiation, 4 min: 87%!); such irradiation was also effective for analogous reactions.[1887]

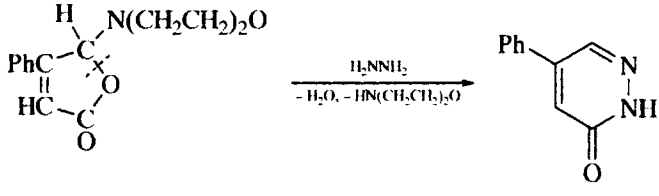
Also other examples.[1295, 1554]

### Using Di- or Tetrahydro-2-furanones (γ-Lactones) as Substrates

2-Hydroxy-2-methyl-3-phenyl-2,5-dihydro-5-furanone (**13**) gave 6-methyl-5-phenyl-3(2*H*)-pyridazinone (**14**) ($H_2NNH_2.H_2O$, BuOH, reflux, water removal, 12 h: 89%).[1309]



(13) (14)



(15) (16)

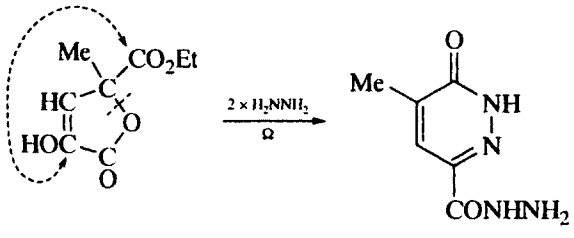
5-Morpholino-4-phenyl-2,5-dihydro-2-furanone (**15**) gave 5-phenyl-3(2*H*)-pyridazinone (**16**) ($H_2NNH_2$, MeOH, reflux, 12 h: 75%).[327]

Ethyl 4-hydroxy-2-methyl-5-oxo-2,5-dihydro-2-furancarboxylate (**17**) gave 5-methyl-6-oxo-1,6-dihydro-3-pyridazinecarbohydrazide (**18**) (2 × $H_2NNH_2 \cdot H_2O$, neat, reflux, 6 h: 54%; note involvement of the ester grouping in recyclization).[503]

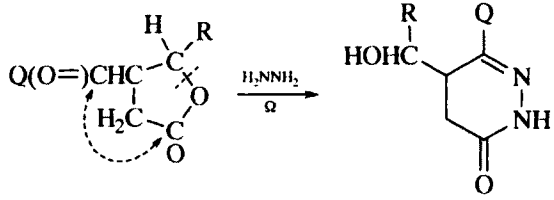
4-Acetyl-5-phenyltetrahydro-2-furanone (**19**, Q = Me, R = Ph) gave 5-α-hydroxybenzyl-6-methyl-4,5-dihydro-3(2*H*)-pyridazinone (**20**, Q = Me, R = Ph) ($H_2NNH_2 \cdot H_2O$, EtOH, reflux, 2 h: 74%);[1045] likewise, 4-benzoyl-5-methyltetrahydro-2-furanone (**19**, Q = Ph, R = Me) gave 5-α-hydroxyethyl-6-phenyl-4,5-dihydro-3(2*H*)-pyridazinone (**20**, Q = Ph,

R = Me) (88%) and then the didehydro analogue ($SeO_2$, EtOH, reflux, 8 h: 93%).[370] The 5-(*o*-chloro-α-hydroxybenzyl) analogue (**20**, Q = Me, R = $C_6H_4Cl$-*o*) was confirmed in structure by X-ray analysis.[1045]

Also other examples.[85,302,354,611,717,738,845,848,941,984,1106,1252,1274,1286,1496,1588 1742,2061,2114]



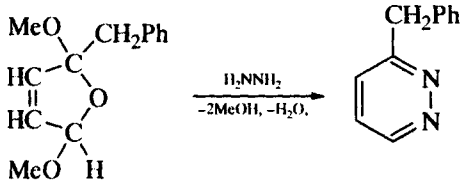

(17) (18)




(19) (20)

## Using Other Furans as Substrates

2-Benzyl-2,5-dimethoxy-2,5-dihydrofuran (**21**) and hydrazine gave a separable mixture of 3-benzylpyridazine (**22**) and 6-benzyl-4,5-dihydro-3(2*H*)-pyridazinone as a byproduct ($H_2NNH_2 \cdot 2HCl$, PrOH, pH 6.5 buffer, reflux, 10 h: 40% and 35%, respectively).[1108]

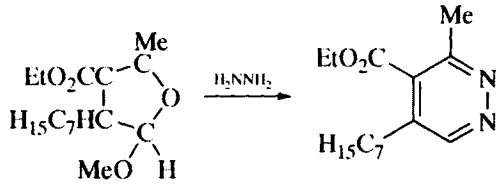



(21) (22)

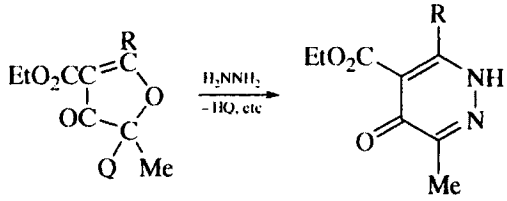
Ethyl 4-heptyl-5-methoxy-2-methyl-4,5-dihydro-3-furancarboxylate (**23**) and hydrazine gave ethyl 5-heptyl-3-methyl-4-pyridazinecarboxylate (**24**) ($H_2NNH_2 \cdot H_2SO_4$, $H_2SO_4$, $H_2O$–EtOH, reflux, 3 h: 82%; aerial oxidation involved?).[1494]

Ethyl 5-ethoxy-5-methyl-4-oxo-4,5-dihydro-3-furancarboxylate (**25**, Q = OEt, R = H) and hydrazine gave ethyl 6-methyl-5-oxo-2,5-dihydro-

4-pyridazinecarboxylate (**26**, R = H) ($H_2NNH_2 \cdot H_2O$, EtOH, reflux, ? h: 50%);[500] likewise ethyl 5-acetoxy-5-methyl-4-oxo-2-phenyl-4,5–dihydro-3-furancarboxylate (**25**, Q = OAc, R = Ph) gave ethyl 6-methyl-5-oxo-3-phenyl-2,5-dihydro-4-pyridazinecarboxylate (**26**, R = Ph) ($H_2NNH_2$, EtOH, 20°, 24 h: 85%).[948]



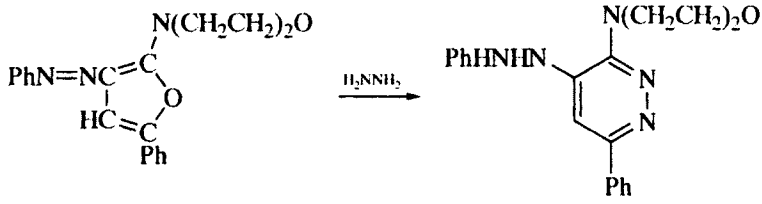

(23) (24)




(25) (26)

2-Morpholino-5-phenyl-3-phenylazofuran (**27**), as fluoroborate salt, with hydrazine hydrate gave 3-morpholino-6-phenyl-4-$\beta$-phenylhydrazinopyridazine (**28**) (EtOH, warmed until homogeneous: 78%).[651]

Also other examples in the foregoing references and elsewhere.[150,318,333,662,847,925,1163,1164,1405,1486,1489,1493,1939,1957]

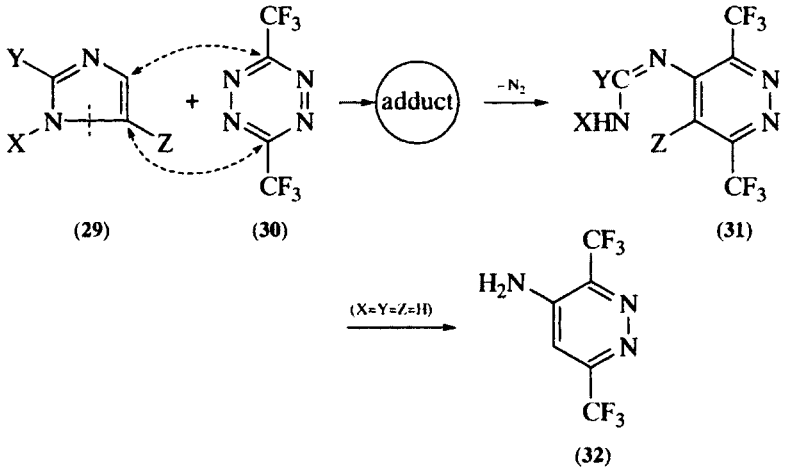



(27) (28)

## 2.1.3 Imidazoles as Substrates

Simple imidazoles have been used as dienophiles for Diels–Alder cycloaddition with 3,6-bis(trifluoromethyl)-1,2,4,5-tetrazine (**30**) with subsequent degradation of the adducts to afford simple pyridazines (cf. Section 2.2.7). Thus imidazole (**29**, X = Y = Z = H) and the tetrazine (**30**) in refluxing toluene for 1 h gave 3,6-bistrifluoromethyl-4-pyridazinamine (**32**) in 30%

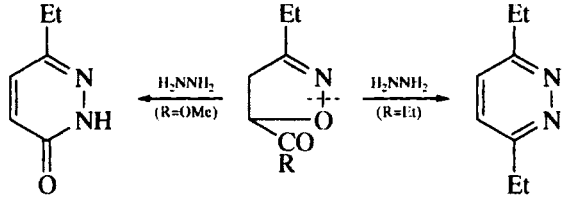
yield.[520] Similarly, 1-methylimidazole (**29**, X = Me, Y = Z = H) gave 4-methylaminomethyleneamino-3,6-bistrifluoromethylpyridazine (**31**, X = Me, Y = Z = H) (58%), which was not degraded further to the amine (**32**); 2-methylimidazole (**29**, X = Z = H, Y = Me) gave 4-(α-aminoethylideneamino)-3,6-bistrifluoromethylpyridazine (**31**, X = Z = H, Y = Me) (10%); and 4-methylimidazole (**29**, X = Y = H, Z = Me) gave 4-aminomethyleneamino-5-methyl-2,6-bistrifluoromethylpyridazine (**31**, X = Y = H, Z = Me) (72%).[520] Other such reactions have been reported.[662]




### 2.1.4 Isoxazoles as Substrates

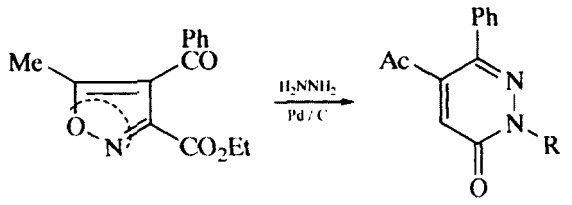
There are several ways to convert isoxazoles into pyridazines, as illustrated in the following examples:

Methyl 3-ethyl-4,5-dihydro-5-isoxazolecarboxylate (**34**, R = OMe) gave 6-ethyl-3(2*H*)-pyridazinone (**33**) ($H_2NNH_2$, PhMe, reflux, 24 h: 21%; note elimination of MeOH, $NH_3$, and $H_2O$);[186] somewhat similarly, 3-ethyl-5-propionyl-4,5-dihydroisoxazole (**34**, R = Et) gave 3,6-diethylpyridazine (**35**) ($H_2NNH_2$, TsOH, MeOH, reflux, 3 h: 48%; note elimination of $2 \times H_2O$ and $NH_3$).[186]

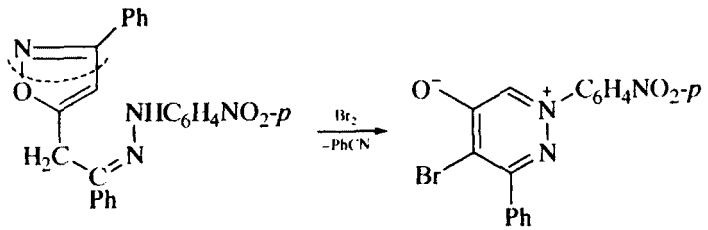


Ethyl 4-benzoyl-5-methyl-3-isoxazolecarboxylate (**36**) gave 5-acetyl-4-amino-6-phenyl-3(2*H*)-pyridazinone (**37**, R = H) ($H_2NNH_2$, EtOH, Pd/C, reflux, 20 min: 90%);[1590] likewise, methylhydrazine gave the 2-methyl derivative (**37**, R = Me) (89%).[1590]

5-[β-(*p*-Nitrophenylhydrazono)phenethyl]-3-phenylisoxazole (**38**) gave 5-bromo-2-*p*-nitrophenyl-6-phenylpyridazinium-4-olate (**39**) ($2 \times Br_2$, $CHCl_3$, reflux, ? min: 60%, initially as the hydrochloride; the eliminated benzonitrile was isolated, a rational mechanism was proposed, and analogues were made similarly);[273] the structure (**39**) was subsequently confirmed by X-ray analysis.[167]



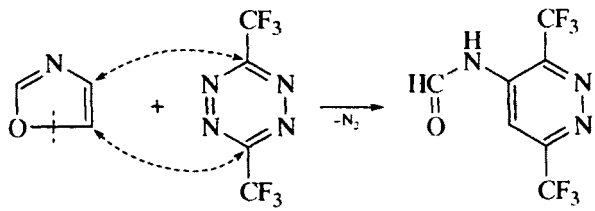

(36) (37)




(38) (39)

## 2.1.5 Oxazoles as Substrates

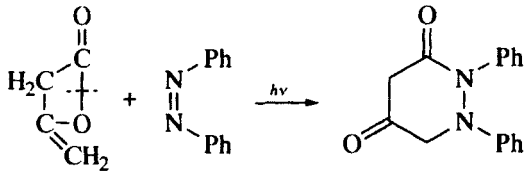
Like imidazoles (Section 2.1.3), oxazole (**40**) has been used as a dienophile with 3,6-bistrifluoromethyl-1,2,4,5-tetrazine (**41**) to furnish eventually 4-formamido-3,6-bistrifluoromethylpyridazine (**42**); the transformation occurred in refluxing toluene during 55 h to give an 80% yield.[520] A few analogues have been made similarly,[1031] but the method has not been developed fully.




(40) (41) (42)

### 2.1.6 Oxetes as Substrates

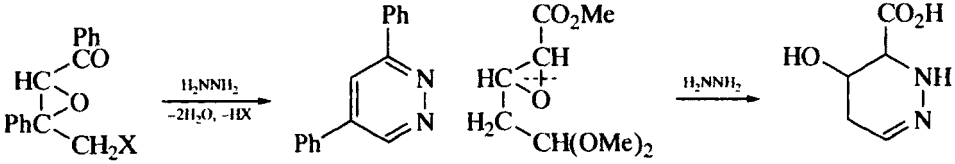
Irradiation of 4-methylene-2-oxetanone (diketene: **43**) with azobenzene (**44**) in chloroform solution for 48 h afforded 5-oxo-1,2-diphenyl-1,4,5,6-tetrahydro-3(2*H*)-pyridazinone (**45**) in 62% yield.[305] Each symmetrically substituted azobenzene naturally gave a single product, for example, 1,2-bis-*p*-chlorophenyl-5-oxo-1,4,5,6-tetrahydro-3(2*H*)-pyridazinone (72%); each unsymmetrically substituted azobenzene also appeared to give (stereoselectively) a single product, for example, 1-*p*-methoxyphenyl-5-oxo-2-phenyl-1,4,5,6-tetrahydro-3(2*H*)-pyridazinone (62%).[305] Diketene also formed unstable adducts with 1,2,4,5-tetrazines which decomposed to give, for example, 4-methyl-3,6-bistrifluoromethylpyridazine in low yield.[875]



(43) (44) (45)

### 2.1.7 Oxirenes as Substrates

Several appropriately substituted oxiranes have been used as substrates for making pyridazines or hydropyridazines. Thus 2-benzyl-3-bromomethyl-3-phenyloxirane (diphenacyl bromide: **46**, X = Br) or the corresponding chloride (**46**, X = Cl) reacted with hydrazine hydrate in refluxing ethanol during 30 min to give 3,5-diphenylpyridazine (**47**) in 85% or 65% yield, respectively.[1795] In contrast, methyl 3-$\beta$,$\beta$-dimethoxyethyl-2-oxiranecarboxylate (**48**) was converted into 4-hydroxy-2.3.4.5-tetrahydro-3-pyridazinecarboxylic acid (**49**) by a one-pot saponification, hydrazination, and cyclization process — both *cis* and *trans* forms were so made from the appropriate substrates.[355]



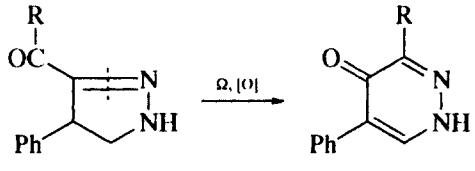
(46) (47) (48) (49)

### 2.1.8 Pyrazoles as Substrates

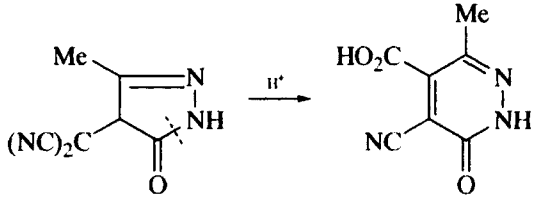
Several undeveloped reaction pathways from pyrazoles to pyridazines have been reported. They are illustrated briefly in the following examples:

3-Acetyl-4-phenyl-2-pyrazoline (**50**, R = Me) gave 3-methyl-5-phenyl-4(1*H*)-pyridazinone (**51**, R = Me) (PhH, 25°, 30 days: 40%);[1713] likewise the 3-benzoyl substrate (**50**, R = Ph) gave 3,5-diphenyl-4(1*H*)-pyridazinone (**51**, R = Ph) (PhH or dioxane, reflux, 12 h: 12%).[208]

4-Dicyanomethylene-3-methyl-2-pyrazolin-5-one (**52**) gave 5-cyano-3-methyl-6-oxo-1,6-dihydro-4-pyridazinecarboxylic acid (**53**) (20% $H_2SO_4$, reflux, 10 h: 69%).[128]




(**50**) (**51**)




(**52**) (**53**)

3,4,5-Trimethylpyrazole gave 4-chloro-3,5,6-trimethylpyridazine [NaOH, benzyldimethyloctadecylammonium chloride (or the like), $H_2O$–PhH–$CHCl_3$, 50°, 30 min: 31%, after separation from other products].[105]

Also other examples.[391,404,582]

### 2.1.9 Pyrroles as Substrates

Most transformations of pyrroles into pyridazines involve the simple rearrangement of *N*-aminopyrroles, but other processes are also represented. The following examples illustrate the presently known synthetic possibilities:

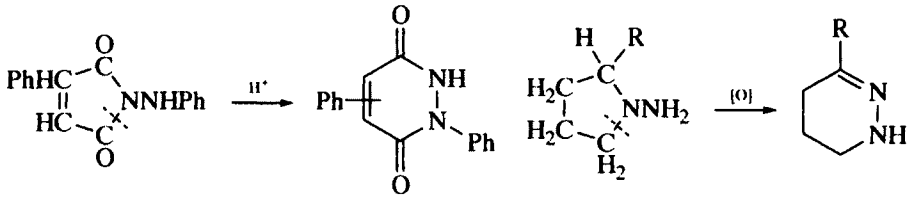
1-Anilino-3-phenyl-3-pyrroline-2,5-dione (*N*-anilino-2-phenylmaleimide: **54**) rearranged into 1,4/1,5-diphenyl-3,6(1*H*,2*H*)-pyridazinedione (**55**) (50% $H_2SO_4$–AcOH, reflux, 3 h: 82%; the position of the *C*-phenyl group was not determined);[462] several analogues (some of unambiguous structure) have been made somewhat similarly.[462,586,648,685,799,1457]

1-Amino-5-hydroxy-5-(2,3,5-tri-*O*-benzoyl-β-D-ribofuranosyl)-3-pyrrolin-2-one gave 6-(2,3,5-tri-*O*-benzoyl-β-D-ribofuranosyl)-3(2*H*)-pyridazinone with loss of water (PhH, reflux, 2 h: 95%).[351]

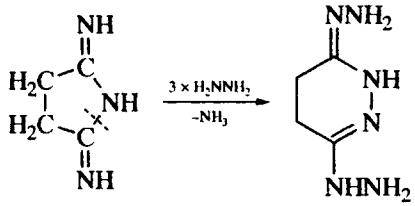
1-Pyrrolidinamine (**56**, R = H) or its 2-phenyl derivative (**56**, R = Ph) gave 1,4,5,6-tetrahydropyridazine (**57**, R = H) or its 3-phenyl derivative (**57**,

R = Ph), respectively (silica gel, $CHCl_3$–MeOH, 20°, 12 h: > 40%; note oxidation, assumed to be by $CHCl_3$ rather than air).[267]

2,5-Pyrrolediamine (formulated as 2,5-pyrrolidinediimine: **58**) gave 3,6-dihydrazino-4,5-dihydropyridazine, as the tautomeric 3-hydrazino-6-hydrazono-1,4,5,6-tetrahydropyridazine (**59**) on nmr evidence ($3 \times H_2NNH_2 \cdot H_2O$, MeOH, 25°, $N_2$, $NH_3\uparrow$, > 3 h: 85%; note transamination);[639] analogous reactions were also reported.[639]



(54) (55) (56) (57)

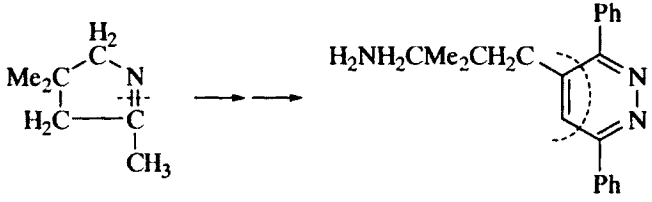


(58) (59)

2,4,4-Trimethyl-1-pyrroline (**60**) and 3,6-diphenyl-1,2,4,5-tetrazine gave a Diels–Alder adduct that lost nitrogen to give 4-(γ-amino-β,β-dimethylpropyl)-3,6-diphenylpyridazine (**61**) (neat, ~160°, until color faded: 78%).[512]

2-Methyl-5-morpholino-4-phenyl-1-pyrrolin-4-ol 1-oxide gave only 3-methyl-5-phenylpyridazine ($H_2NNH_2$, AcOH–$H_2O$, reflux, 15 min: 91%).[1982]

Also other examples.[442, 662, 824, 1554]

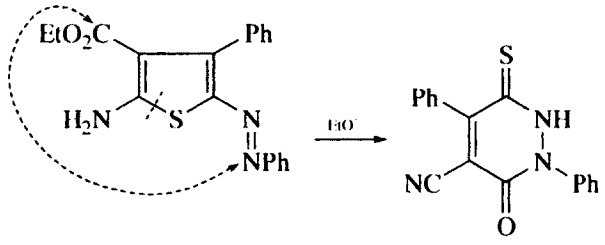


(60) (61)

## 2.1.10 Thiophenes as Substrates

Several routes from thiophenes to pyridazines are possible, but none has been used much. Some examples follow:

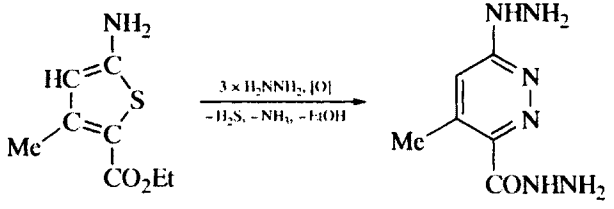
Ethyl 2-amino-4-phenyl-5-phenylazo-3-thiophenecarboxylate (**62**) rearranged into 3-oxo-2,5-diphenyl-6-thioxo-1,2,3,6-tetrahydro-4-pyridazinecarbonitrile (**63**) with loss of ethanol (KOH, EtOH, reflux, 40 min: 62%).[88]



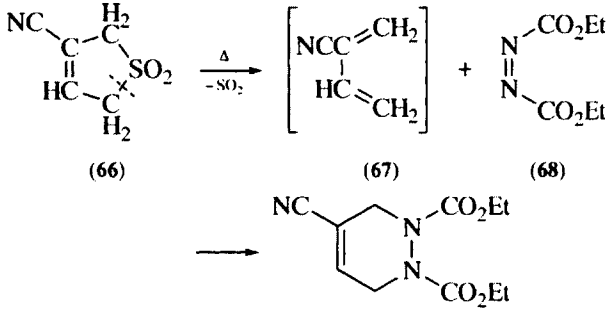
(62) (63)

Ethyl 5-amino-3-methyl-2-thiophenecarboxylate (**64**) and hydrazine gave 6-hydrazino-4-methyl-3-pyridazinecarbohydrazide (**65**) (excess $H_2NNH_2 \cdot H_2O$, neat, reflux, 2 h: 60%);[21] an contrast, 5-phenyl-2-thiophenamine under similar conditions (for 6 h) gave *N,N'*-bis(6-phenyl-4,5-dihydropyridazin-3-yl)hydrazine (55%).[21]

2,5-Dihydro-3-thiophenecarbonitrile 1,1-dioxide (**66**), a stable precursor of 3-cyano-1,3-butadiene (**67**), reacted with the dienophilic diethyl azodicarboxylate (**68**) to give diethyl 4-cyano-1,2,3,6-tetrahydro-1,2-pyridazinedicarboxylate (**69**) by a Diels–Alder mechanism (PhMe, reflux, 12 h: 95%);[423] such dioxides have been used as cosynthones with the heterocyclic dienophile, 4-phenyl-1,2,4-triazoline-3,5-dione;[1391,1518] see next subsection.



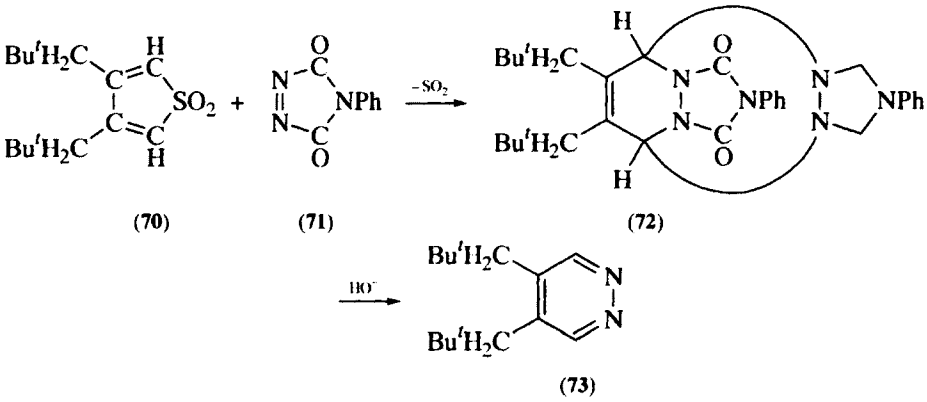
(64) (65)



(66) (67) (68) (69)

### 2.1.11 1,2,4-Triazoles as Substrates

The dienophile, 4-phenyl-1,2,4-triazoline-3,5-dione (**71**), reacted with 3,4-dineopentylthiophene 1,1-dioxide (**70**) in refluxing benzene to afford the bis adduct (**72**) in 77% yield with loss of sulfur dioxide; treatment of the adduct with methanolic alkali at 25° then gave 4,5-dineopentylpyridazine (**73**) in 74% yield.[1518] 4,5-Diadamant-1′-ylpyridazine was made similarly in 82% yield (based on the adduct).[1391]




## 2.2 PYRIDAZINES FROM OTHER SIX-MEMBERED HETEROCYCLIC SYSTEMS

Although many such transformations are represented in the recent literature, only 1,2,4,5-tetrazines have been used extensively to produce pyridazines (mainly via Diels–Alder-type adducts).

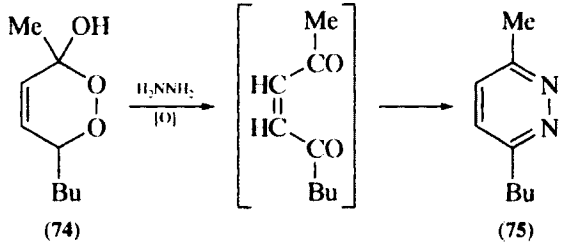
### 2.2.1 1,2-Dioxins or 1,2-Dithiins as Substrates

This rare synthesis is illustrated by the conversion of 6-butyl-3-methyl-3,6-dihydro-1,2-dioxin-3-ol (**74**) into 3-butyl-6-methylpyridazine (**75**) (65% yield) by passing oxygen through a solution of the substrate, hydrazine, and copper sulfate in methanolic acetic acid at 25° for 10 h; the postulated intermediate, non-3-ene-2,5-dione, was not isolated or detected.[1659] Somewhat similarly, 3,6-diphenyl-1,2-dithiin and hydrazine in refluxing ethanolic pyridine for 2 h gave 3,6-diphenylpyridazine in 43% yield; analogues were made likewise.[1883]

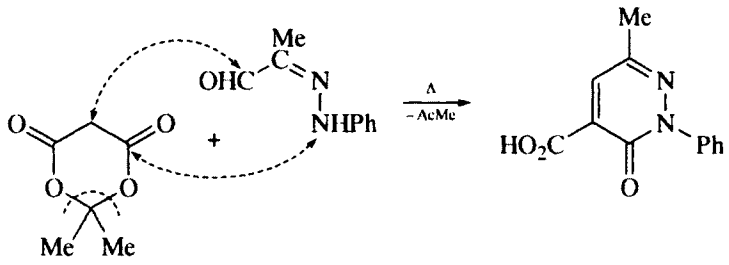
### 2.2.2 1,3-Dioxins as Substrates

Pyridazines have been made from 1,3-dioxins in two related ways. The first is illustrated by the condensation of 2,2-dimethyl-1,3-dioxane-4,6-dione (**76**)

with 2-phenylhydrazonopropionaldehyde (**77**) to afford 6-methyl-3-oxo-2-phenyl-2,3-dihydro-4-pyridazinecarboxylic acid (**78**) in 56% yield, by refluxing in benzene containing a little piperidine acetate for 2.5 h.[715]

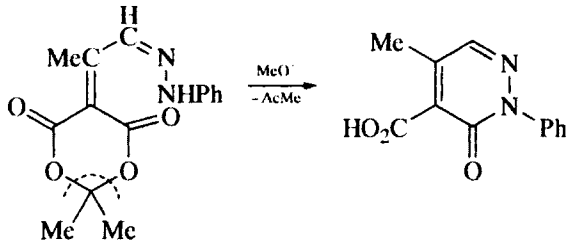



(74) (75)




(76) (77) (78)

The second method appears to be more versatile. For example, 2,2-dimethyl-4-$\alpha$-(phenylhydrazonomethyl)ethylidene-1,3-dioxane-4,6-dione (**79**) underwent transformation in refluxing methanolic sodium methoxide during 20 min to afford 5-methyl-3-oxo-2-phenyl-2,3-dihydro-4-pyridazinecarboxylic acid (**80**) in 70% yield:[715] analogues were made similarly.[715, 2024]



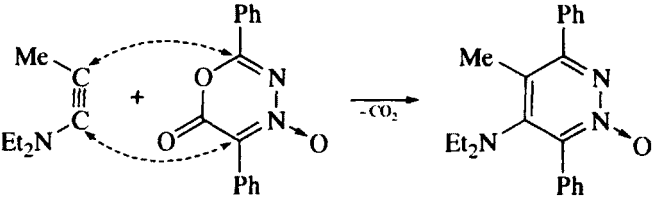

(79) (80)

### 2.2.3 1,3,4-Oxadiazines as Substrates

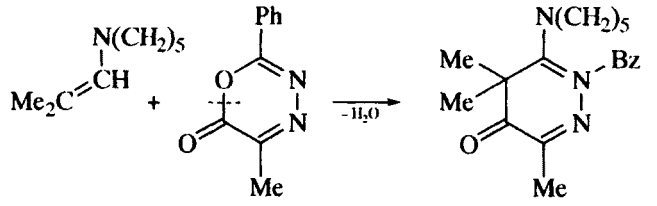
Several derivatives of 1,3,4-oxadiazin-6-one have been used to make pyridazines by Diels–Alder-type cycloadditions, as illustrated in the following examples:

2,5-Diphenyl-1,3,4-oxadiazin-6-one 4-oxide (**82**) and 1-diethylaminopropyne (**81**) regioselectively gave 4-diethylamino-5-methyl-3,6-diphenylpyridazine 2-oxide (**83**) with loss of carbon dioxide ($CH_2Cl_2$, exothermic, then 25°, 30 min: 94%; no isomeric product detected);[311] several analogues were made similarly but most needed more vigorous conditions: for example, 3-methyl-4-(*N*-methylanilino)-6-phenylpyridazine 2-oxide (reflux, 20 h: 95%).[311]

5-Methyl-2-phenyl-1,3,4-oxadiazin-6-one (**85**) and *N*-(2-methylprop-1-enyl)pyrrolidine (**84**) gave 1-benzoyl-3,5,5-trimethyl-6-pyrrolidin-1′-yl-5,6-dihydro-4(1*H*)-pyridazinone (**86**) (PhH, reflux, $N_2$, 4 h: <36% after separation from other products).[280,415]




(**81**) (**82**) (**83**)




(**84**) (**85**) (**86**)

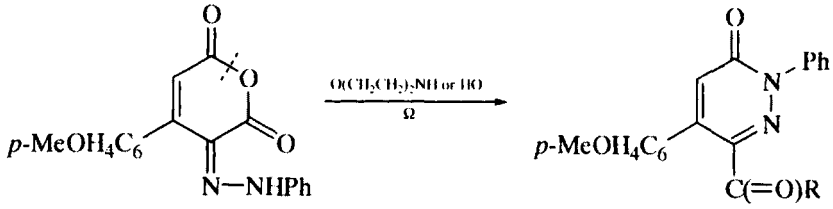
## 2.2.4 Pyrans as Substrates

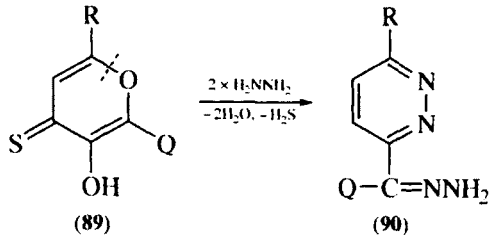
There are at least three ways in which pyrans have been converted into pyridazines directly. They are illustrated in the following examples:

4-*p*-Methoxyphenyl-3-phenylhydrazono-3,6-dihydro-2*H*-pyran-2,6-dione (**87**) rearranged into 4-*p*-methoxyphenyl-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylic acid (**88**, R = OH) (2M NaOH, reflux, 30 min: 82%) or (with subsequent aminolysis) into 5-*p*-methoxyphenyl-6-morpholinocarbonyl-2-phenyl-3(2*H*)-pyridazinone [**88**, $R = N(CH_2CH_2)_2O$] [neat $HN(CH_2CH_2)_2O$, 70°, 30 min: 69%];[1294] analogues were made similarly.[1036,1294]

3-Hydroxy-6-methyl-4-pyranthione (**89**, Q = H, R = Me) gave 6-methyl-3-pyridazinecarbaldehyde, as its hydrazone (**90**, Q = H, R = Me)

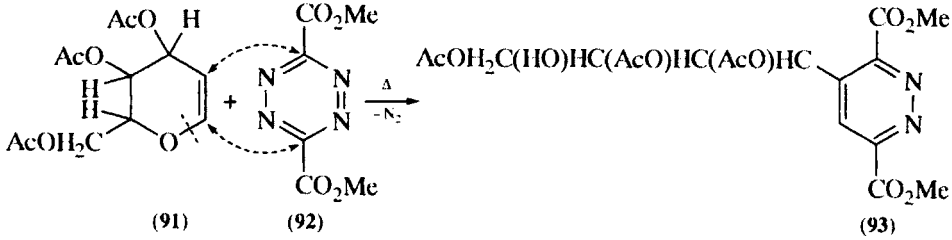
($2 \times H_2NNH_2$, MeOH, reflux, 3 min: 44%); 3-hydroxy-2-methyl-4-pyranthione (**89**, Q = Me, R = H) likewise gave 3-acetylpyridazine, as its hydrazone (**90**, Q = Me, R = H) (reflux, 90 min: 35% after separation from a byproduct).[35]




3,4-Diacetoxy-2-acetoxymethyl-3,4-dihydro-2*H*-pyran [3,4,6-tri-*O*-acetyl-1,5-dihydro-2-desoxy-D-*arabino* (or *lyxo*)-hex-1-enitol: **91**, stereochemistry ignored for simplicity in the present context] and dimethyl 1,2,4,5-tetrazine-3,6-dicarboxylate (**92**) formed a Diels–Alder-type complex that lost nitrogen to afford dimethyl 4-(α,β,δ-triacetoxy-γ-hydroxybutyl)-3,6-pyridazinedicarboxylate (**93**) (dioxane, reflux, 20 h: ~75%).[47]

Also other examples.[1485]

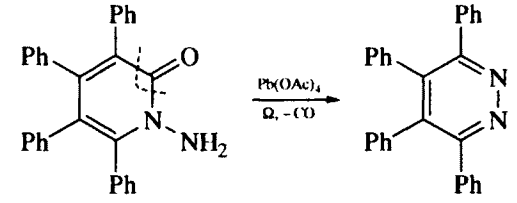



## 2.2.5 Pyridines as Substrates

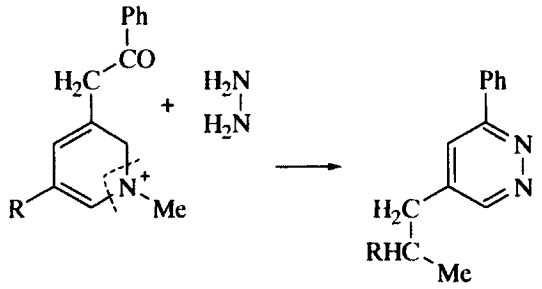
Oxidation of 1-amino-3,4,5,6-tetraphenyl-2(1*H*)-pyridinone (**94**) by lead tetracetate in dichloromethane at 20° during 1 h resulted in rearrangement to 3,4,5,6-tetraphenylpyridazine (**95**) (58%) with loss of carbon monoxide:[632]

somewhat similarly, 1-amino-3,4,5-triphenyl-2(1*H*)-pyridinone gave 3,4,5-triphenylpyridazine but in only 7% yield.[632]

In a different way, treatment of 3-phenacylpyridine methiodide (**96**, R = H) with hydrazine hydrate in di(β-hydroxyethyl) ether under reflux for 2 h, followed by the addition of solid potassium hydroxide and further heating during 2.5 h, afforded 3-phenyl-5-propylpyridazine (**97**, R = H) in ~50% yield;[1797] likewise, 3-methyl-5-phenacylpyridine methiodide (**96**, R = Me) gave 4-isobutyl-6-phenylpyridazine (**97**, R = Me) (~50%).[1797]




(94) (95)



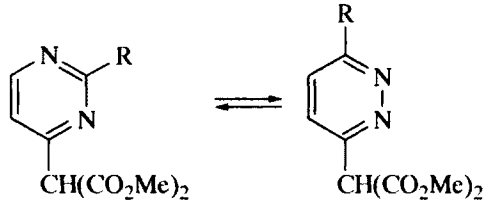

(96) (97)

### 2.2.6 Pyrimidines as Substrates

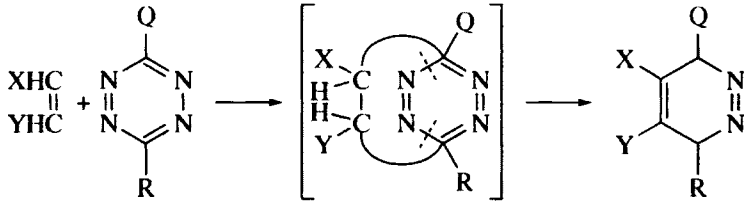
Brief kinetic studies of the reversible (photochemical or thermal?) isomerizations of 4-dimethoxycarbonylmethylpyrimidine (**98**, R = H) into 3-dimethoxycarbonylmethylpyridazine (**99**, R = H) and of 2-chloro-4-dimethoxycarbonylmethylpyrimidine (**98**, R = Cl) into 3-chloro-6-dimethoxycarbonylmethylpyridazine (**99**, R = Cl) have been reported.[872]

### 2.2.7 1,2,4,5-Tetrazines as Substrates

1,2,4,5-Tetrazine derivatives (**101**) have been used extensively to make dihydropyridazines (**103**; or tautomers)[1499] by "inverse electron-demand Diels–Alder reactions"[1844] with dienophiles, such as **100**, followed by loss of nitrogen from the usually unstable complexes (**102**); if the dienophile has a triple or potentially triple bond, the resulting pyridazine will be formally aromatic.



(98) (99)



(100) (101) (102) (103)

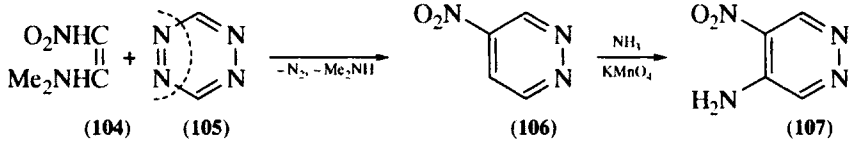
Special cases, in which another heterocyclic system is employed as the dienophile, are exemplified elsewhere in this chapter under the appropriate system: 5,8-ethanophthalazines, Section 2.7; furans, Section 2.1.2; imidazoles, Section 2.1.3; oxazoles, Section 2.1.5; oxetes, Section 2.1.6; pyrroles, Section 2.1.9; pyrans, Section 2.2.4; and thiepins, Section 2.3.3.

'Normal' cases, in which alkenes or alkynes are employed as the dienophiles, are illustrated in the following examples, classified according to the type of tetrazine derivative used in the reaction:

### 1,2,4,5-Tetrazine with Dienophiles

α-Dimethylamino-β-nitroethylene (**104**) and tetrazine (**105**) gave 4-nitropyridazine (**106**) ($CHCl_3$, reflux, $N_2$, 4 days; oxidation provided by elimination of $Me_2NH$), characterized as 5-nitro-4-pyridazinamine (**107**) (18%) after amination.[1080]

Also other examples.[2046]



(104) (105) (106) (107)

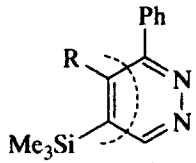
### Monoaryl-1,2,4,5-tetrazines with Dienophiles

*Note*: Such condensations with unsymmetric dienophiles are essentially ambiguous but often so regioselective that only one product is formed. However, solvent polarity can affect the identity of the product.[277]

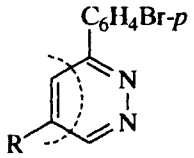
Trimethylsilylacetylene and 3-phenyl-1,2,4,5-tetrazine gave 3-phenyl-5-trimethylsilylpyridazine (**108**, R = H) (PhMe, 75°, 24 h: 90%); 1-trimethylsilylhex-1-yne similarly gave 4-butyl-3-phenyl-5-trimethylsilylpyridazine (**108**, R = Bu) (63%); and other analogues were so made.[12]

3-Morpholinoacrylonitrile and 3-*p*-bromophenyl-1,2,4,5-tetrazine gave 6-*p*-bromophenyl-4-pyridazinecarbonitrile (**109**, R = CN) with loss of morpholine (dioxane, reflux until colorless, 1–3 days: 48%); α-dimethylamino-β-nitroethylene similarly gave 3-*p*-bromophenyl-5-nitropyridazine (**109**, $R = NO_2$) (46%); and other analogues were so made.[215]

Also other examples.[248,473,2093]




(**108**)

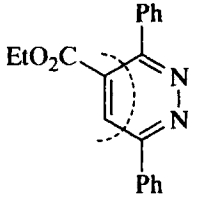



(**109**)

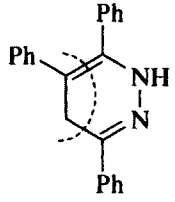
## Diaryl-1,2,4,5-tetrazines with Dienophiles

Ethyl propiolate and 3,6-diphenyl-1,2,4,5-tetrazine gave ethyl 3,6-diphenyl-4-pyridazinecarboxylate (**110**) (xylene, reflux, 70 h: 63%);[40] methylpropiolate likewise gave methyl 3,6-diphenyl-4-pyridazinecarboxylate (dioxane, reflux, 4 h: 87%).[41]

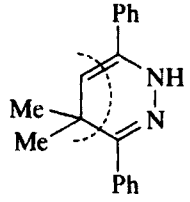
Styrene and 3,6-diphenyl-1,2,4,5-tetrazine gave 3,4,6-triphenyl-2,5-dihydropyridazine (**111**) (PhH, $N_2$, 80°, sealed, 12 h: 20%); 2-methylpropene similarly gave 4,4-dimethyl-3,6-diphenyl-1,4-dihydropyridazine (**112**).[1029] (For microwave assistance, see Ref. 1922.)




(**110**)




(**111**)

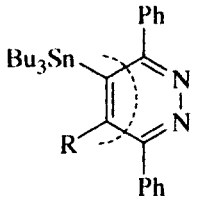



(**112**)

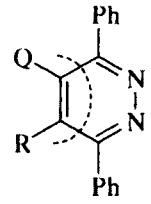
Tributylstannylacetylene and 3,6-diphenyl-1,2,4,5-tetrazine gave 3,6-diphenyl-4-tributylstannylpyridazine (**113**, R = H) (PhMe, reflux, 60 h: 85%); 1-tributylstannylhex-1-yne likewise gave 4-butyl-3,6-diphenyl-5-tributylstannylpyridazine (**113**, R = Bu) (38%); and other analogues were so made.[1591]

Acetaldehyde and 3,6-diphenyl-1,2,4,5-tetrazine gave 3,6-diphenylpyridazine (**114**, Q = R = H) with loss of water and nitrogen (THF, KOH–

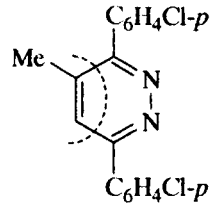
MeOH, 25°, 8 min: 88%); acetone likewise gave 4-methyl-3,6-diphenylpyridazine (**114**, Q = Me, R = H) (5 min: 72%); ethyl methyl ketone gave 4,5-dimethyl-3,6-diphenylpyridazine (**114**, Q = R = Me) (25 min: 52%); and analogues were made similarly.[319] The product (**114**, Q = Me, R = H) was also made by a modified process ($Me_2CO$, $Me_2NH$, 20°, 16 h: 69%).[43]




(113)




(114)




(115)

Lithium diisopropylamide [possibly existing as $Li^+$ $Pr^i$–N–C(=$CH_2$)Me] and 3,6-bis(*p*-chlorophenyl)-1,2,4,5-tetrazine gave 3,6-bis-*p*-chlorophenyl-4-methylpyridazine (**115**) with loss of $Pr^iNH_2$ (THF, −70° → 20°, 2 h; then $NH_4Cl$–$H_2O$: ~ 15%).[722] Somewhat analogous reactions have been reported.[56,278,2073]

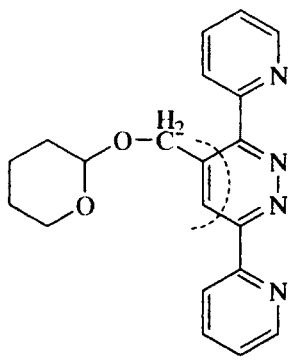
Also other examples.[226,451,1922]

## Mono- or Dipyridinyl-1,2,4,5-tetrazines with Dienophiles

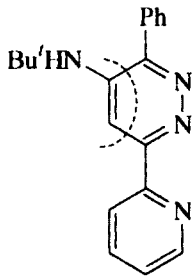
2-(Prop-2-ynyloxy)tetrahydropyran and 3,6-dipyridin-2′-yl-1,2,4,5-tetrazine gave 3,6-dipyridin-2′-yl-4-(tetrahydropyran-2-yloxymethyl)pyridazine (**116**) (PhMe, reflux, 2 days: 92%).[1096]

$N^2$-*t*-Butyl-$N^1$,$N^1$-pentamethyleneacetamidine [MeC(=N$Bu^t$)N$(CH_2)_5$] and 3-phenyl-6-pyridin-2′-yl-1,2,4,5-tetrazine gave a separable mixture of 4-*t*-butylamino-3-phenyl-6-pyridin-2′-yl- (**117**) and 4-*t*-butylamino-6-phenyl-3-pyridin-2′-ylpyridazine (**118**) with loss of piperidine and nitrogen (PhMe, reflux, 18 h: 48% and 35%, respectively).[56]

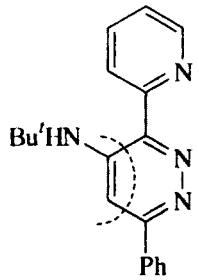
Also other examples.[215,470,1889]




(116)



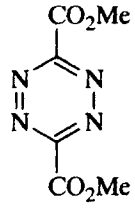

(117)

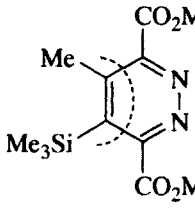


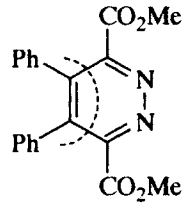

(118)

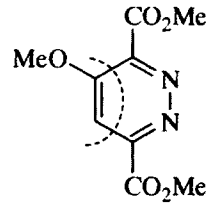
## Dimethyl 1,2,4,5-Tetrazinedicarboxylate with Dienophiles

1-Trimethylsilylprop-1-yne and dimethyl 1,2,4,5-tetrazine-3,6-dicarboxylate (**119**) gave dimethyl 4-methyl-5-trimethylsilyl-3,6-pyridazinedicarboxylate (**120**) ($CH_2Br_2$, reflux, 6 h; then 75°, 24 h: 69%);[11] several homologues were made similarly.[11,1208]


(119)


(120)


(121)


(122)

Diphenylacetylene and ester (**119**) gave dimethyl 4,5-diphenyl-3,6-pyridazinedicarboxylate (**121**) (xylene, reflux, 30 h: 75%).[41]

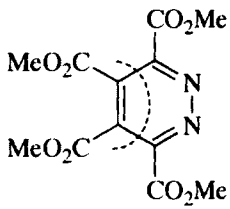
Ketene dimethylacetal ($\alpha,\alpha$-dimethoxyethylene) and the ester (**119**) gave dimethyl 4-methoxy-3,6-pyridazinedicarboxylate (**122**) with loss of methanol and nitrogen (dioxane, 25°, $N_2$, 30 min: 65%);[337,cf.1509] also several analogues.[337]

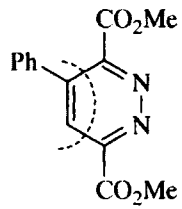
Dimethyl acetylenedicarboxylate and the same ester (**119**) gave tetramethyl 3,4,5,6-pyridazinetetracarboxylate (**123**) (*o*-xylene, reflux, A, 24 h: 60%;[357] xylene, reflux, 15 h: 53%).[40]

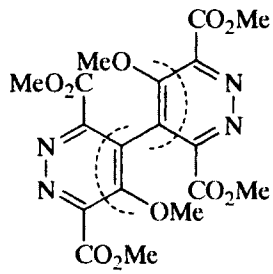
$\alpha$-Trimethylsiloxystyrene and the ester (**119**) gave dimethyl 4-phenyl-3,6-pyridazinedicarboxylate (**124**) with loss of $HOSiMe_3$ and nitrogen (dioxane, 25°, $N_2$, 8 h: ~95%).[1568]

$\alpha,\alpha,\delta,\delta$-Tetramethoxybutadiene and 2 × ester (**119**) gave tetramethyl 5,5′-dimethoxy-4,4′-bipyridazine-3,3′,6,6′-tetracarboxylate (**125**) (PhH, 22° → 80°, A, 72 h; then AcOH, 80°, A, 60 h: 56%; or $CHCl_3$. 4Å molecular sieves, 22°, A, 25 min; then reflux, A, 5 days: 65%).[1662]

Also other examples.[2,283,286,343,352,445,507,508,510,624,841,1388,1591,1735,1738,1863,2106]

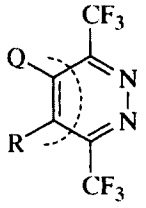

(123)


(124)

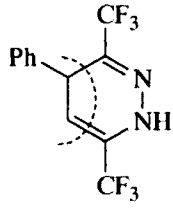

(125)

### Bistrifluoromethyl-1,2,4,5-tetrazine with Dienophiles

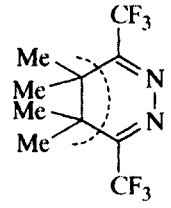
Acetylene and 3,6-bistrifluoromethyl-1,2,4,5-tetrazine gave 3,6-bistrifluoromethylpyridazine (**126**, Q = R = H) (neat, 25°, 48 h: 79%); propyne likewise gave 4-methyl-3,6-bistrifluoromethylpyridazine (**126**, Q = Me, R = H) (25°, 10 days: 91%); and bistrimethylstannylacetylene gave 3,6-bistrifluoromethyl-4,5-bistrimethylstannylpyridazine (**126**, Q = R = $SnMe_3$) ($Et_2O$, 0°, 2 days: 78%).[696] In contrast, styrene gave 4-phenyl-3,6-bistrifluoromethyl-1,4-dihydropyridazine (**127**) (neat, 0°, 1 h: 95%; nmr evidence for structure); and 2,3-dimethylbut-2-ene gave 4,4,5,5-tetramethyl-3,6-bistrifluoromethyl-4,5-dihydropyridazine (**128**) (neat, 25°, 2 weeks: 91%).[696]



(126)



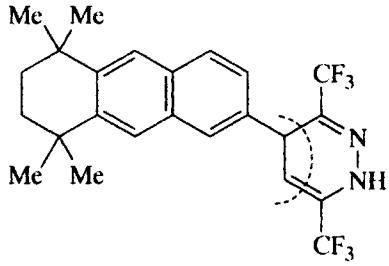
(127)



(128)

1,1,4,4-Tetramethyl-6-vinyl-1,2,3,4-tetrahydroanthracene and 3,6-bistrifluoromethyl-1,2,4,5-tetrazine gave 4-(5,5,8,8-tetramethyl-5,6,7,8-tetrahydroanthracen-2-yl)-3,6-bistrifluoromethyl-1,4-dihydropyridazine (**129**) (PhMe, A, 25° → 80°, 3.5 h: 48%).[1360]

Also other examples.[1038,2104]

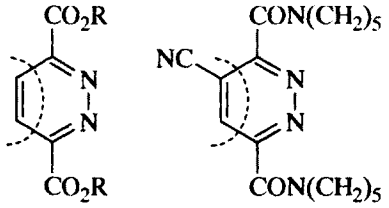


(129)

### Miscellaneous 1,2,4,5-Tetrazines with Dienophiles

Ethoxyethylene and disodium 1,2,4,5-tetrazine-3,6-dicarboxylate gave disodium 3,6-pyridazinedicarboxylate (**130**, R = Na) with loss of ethanol ($H_2O$, 25°, until colorless: 90%) and thence the free dicarboxylic acid (**130**, R = H) ($H_2O$, Amberlite IRN 77, 30 min: 75%).[1077]

3-Morpholinoacrylonitrile and 3,6-bispiperidinocarbonyl-1,2,4,5-tetrazine gave 3,6-bispiperidinocarbonyl-4-pyridazinecarbonitrile (**131**) with loss of morpholine (dioxane, reflux, 1 h: 55%).[215]

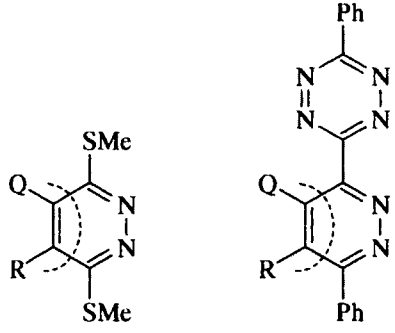


(130) (131)

*N,N*-Diethylprop-1-ynylamine and 3,6-bismethylthio-1,2,4,5-tetrazine gave 4-diethylamino-5-methyl-3,6-bismethylthiopyridazine (**132**, Q = $NEt_2$, R = Me) (dioxane, 25°, $N_2$, 1 h: 96%); $\alpha,\alpha$-dimethoxyethylene likewise gave 4-methoxy-3,6-bismethylthiopyridazine (**132**, Q = OMe, R = H) (dioxane, 80°, 13 h: 86%); *N*-vinylpyrrolidin-2-one gave 3,6-bismethylthiopyridazine (**132**, Q = R = H) with loss of pyrollidinone (dioxane, reflux, 22 h: 79%); and other analogues similarly.[353]

*N,N*-Dimethylprop-1-ynylamine and (equimolar?) 6,6′-diphenyl-3,3′-bi(1,2,4,5-tetrazine) gave a 3:2 mixture of 3-(4-dimethylamino-5-methyl-6-phenylpyridazin-3-yl)- (**133**, Q = $NMe_2$, R = Me) and 3-(5-dimethylamino-4-methyl-6-phenylpyridazin-3-yl)-6-phenyl-1,2,4,5-tetrazine (**133**, Q = Me, R = $NMe_2$) (81% of mixture).[1522]

Also other examples.[1943, 2024, 2026]

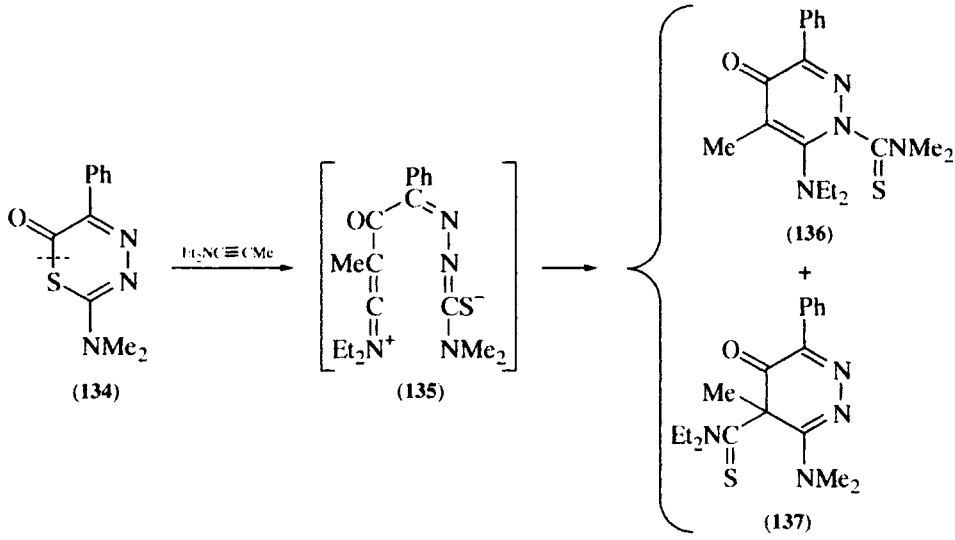


(132) (133)

## 2.2.8 1,3,4-Thiadiazines as Substrates

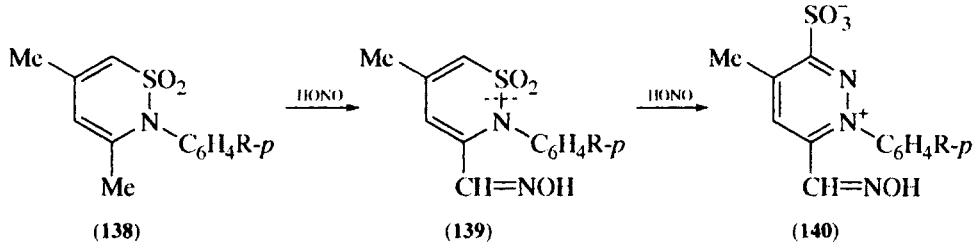
A derivative of this unusual system, 2-dimethylamino-5-phenyl-1,3,4-thiadiazin-6-one (**134**), underwent addition of *N,N*-diethylprop-1-ynylamine and subsequent reactions (under unspecified conditions) to afford two isomeric products, 6-diethylamino-5,*N,N*-trimethyl-4-oxo-3-phenyl-1,4-dihydro-1-pyridazinecarbothioamide (**136**) (2%), and 3-dimethylamino-*N,N*-diethyl-4-methyl-5-oxo-6-phenyl-4,5-dihydro-4-pyridazinecarbothioamide (**137**) (72%),

probably via a common intermediate (**135**).[489] The structures **136** and **137** were confirmed subsequently by X-ray analysis.[169]




## 2.2.9 1,2-Thiazines as Substrates

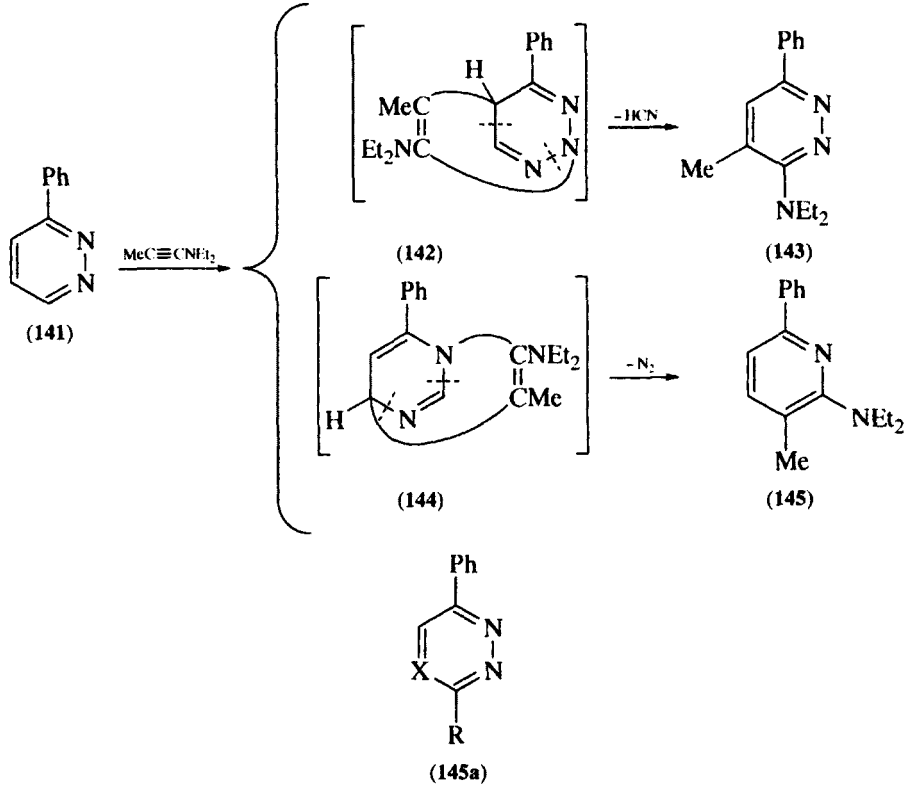
3,5-Dimethyl-2-phenyl-1,2-thiazine 1,1-dioxide (**138**, R = H) underwent a peculiar and interesting reaction on treatment with sodium nitrite and hydrochloric acid in acetonitrile at 0° for 30 min, to give 6-hydroxyimino-4-methyl-1-phenylpyridazinium-3-sulfonate (**140**, R = H) (26%), via the isolable intermediate oxime (**139**); several analogues (**140**, R = OMe, Me, Cl, or Br) were made similarly in mediocre yields.[1785] The structure (**140**, R = H) was confirmed by X-ray analysis.[1536]




## 2.2.10 1,2,3-Triazines as Substrates

1,2,3-Triazines can react with an appropriate electron-rich dienophile to give Diels–Alder adducts that can lose hydrogen cyanide to give pyridazines or nitrogen to give pyridines. Thus 4-phenyl-1,2,3-triazine (**141**) and *N,N*-

diethylprop-1-ynylamine in refluxing chloroform for 92 h gave a separable mixture of 3-diethylamino-4-methyl-6-phenylpyridazine (**143**) (24% yield) and 2-diethylamino-3-methyl-6-phenylpyridine (**145**) (21% yield), via the respective unisolated adducts **142** and **144**; analogues were made similarly.[1442]




### 2.2.11 1,2,4-Triazines as Substrates

3-Chloro-6-phenyl-1,2,4-triazine (**145a**, R = Cl, X = N) reacted with phenylacetonitrile in dimethylformamide containing an excess of potassiun *t*-butoxide at 0° during 1 h to give an unstable acyclic intermediate that readily underwent recyclization in aqueous–acetonic ammonia to afford 4,6-diphenyl-3-pyridazinamine (**145a**, R = $NH_2$, X = CPh) in 86% overall yield; several analogues were made similarly.[1885]

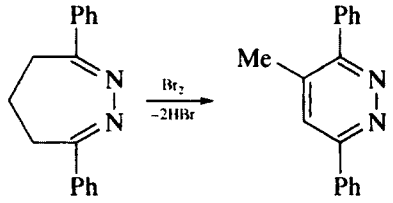
## 2.3 PYRIDAZINES FROM SEVEN-MEMBERED HETEROMONOCYCLIC SYSTEMS (*H* 363)

Few such substrates have been used in the recent literature to make pyridazines. No eight-membered or higher heteromonocyclic systems have been so employed.

### 2.3.1 1,2-Diazepines as Substrates

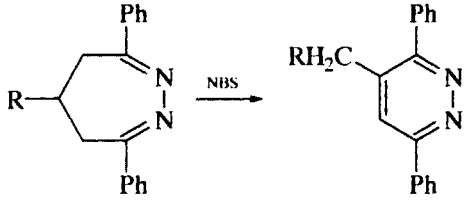
Several 3,7-diphenyl-5,6-dihydro-4*H*-diazepines have been converted into pyridazines or dihydropyridazines according to the reagent employed, as illustrated in the following examples:

3,7-Diphenyl-5,6-dihydro-4*H*-diazepine (**146**) gave 4-methyl-3,6-diphenylpyridazine (**147**) by a rational oxidative ring contraction [$Br_2$, $CH_2Cl_2$, reflux, 1 h: 8%; *N*-bromosuccinimide (NBS), $CCl_4$, reflux; 1 h: 5%].[28]



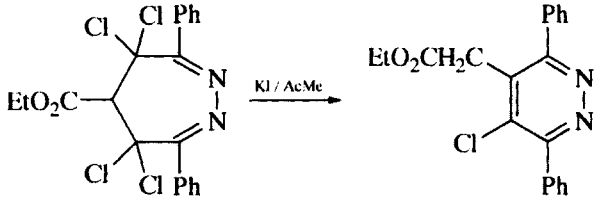

(**146**) (**147**)

3,7-Diphenyl-5,6-dihydro-4*H*-diazepine-5-carbonitrile (**148**, R = CN) gave 4-cyanomethyl-3,6-diphenylpyridazine (**149**, R = CN) (NBS, $CCl_4$, reflux: 73%); ethyl 3,7-diphenyl-5,6-dihydro-4*H*-diazepine-5-carboxylate (**148**, R = $CO_2Et$) likewise gave 4-ethoxycarbonylmethyl-3,6-diphenylpyridazine (**149**, R = $CO_2Et$) (32%).[222]




(**148**) (**149**)

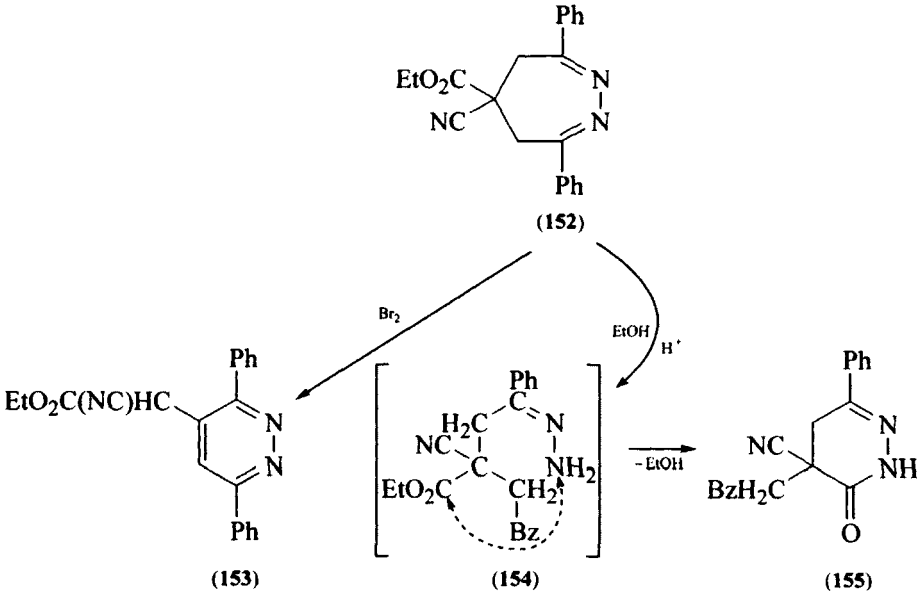
Ethyl 4,4,6,6-tetrachloro-3,7-diphenyl-5,6-dihydro-4*H*-diazepine-5-carboxylate (**150**) gave 4-chloro-5-ethoxycarbonylmethyl-3,6-diphenylpyridazine (**151**) (NaI, AcMe, reflux, 2 h: 82%).[222]




(**150**) (**151**)

Ethyl 5-cyano-3,7-diphenyl-5,6-dihydro-4*H*-diazepine-5-carboxylate (**152**) gave 4-(α-cyano-α-ethoxycarbonylmethyl)-3,6-diphenylpyridazine (**153**)

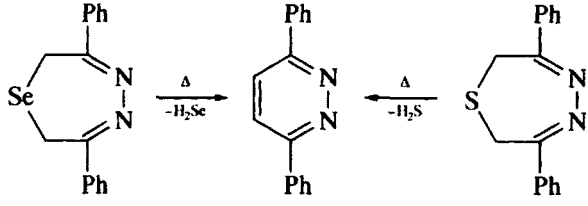
and 3,6-diphenylpyridazine ($Br_2$, $CH_2Cl_2$, 25°, 30 min: 55% and 35%, respectively) or 3-oxo-4-phenacyl-6-phenyl-2,3,4,5-tetrahydro-4-pyridazinecarbonitrile (**155**) via the unisolated intermediate (**154**) (HCl, EtOH, reflux, 15 min: 87%).[1071,2052]




### 2.3.2 1,4,5-Selena- or 1,4,5-Thiadiazepines as Substrates

The synthesis of pyridazines from dihydro derivatives of these rare systems is illustrated in the following examples:

3,6-Diphenyl-2,7-dihydro-1,4,5-selenadiazepine (**156**) underwent thermal ring contraction to 3,6-diphenylpyridazine (**157**) with loss of hydrogen selenide ($HOCH_2CH_2OH$, reflux, 3 h: "high yield"); 3,6-bis-*p*-chlorophenylpyridazine was made similarly.[896]

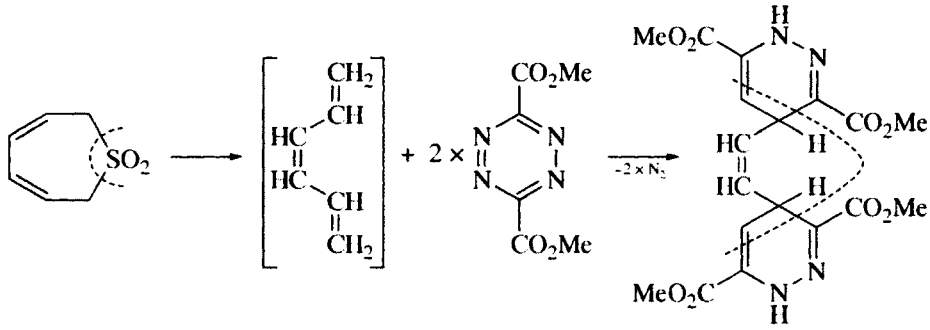



3,6-Diphenyl-2,7-dihydro-1,4,5-thiadiazepine (**158**) likewise gave 3,6-diphenylpyridazine (**157**) with loss of hydrogen sulfide (2 h: 91%).[38]

3,6-Dithien-2′-yl-2,7-dihydro-1,4,5-thiadiazepine similarly gave 3,6-dithien-2′-ylpyridazine (3 h: 70%) and several analogues were so made satisfactorily.[38]

### 2.3.3 Thiepines as Substrates

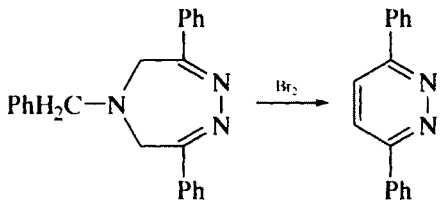
When 2,7-dihydrothiepine 1,1-dioxide (**159**) reacted with dimethyl 1,2,4,5-tetrazine-3,6-dicarboxylate (**161**), the major product was α,β-bis(3,6-dimethoxycarbonyl-1,4-dihydropyridazin-4-yl)ethylene (**162**) (PhCl, 100°, 3 h: 38% after purification), presumably formed via a Diels–Alder adduct with the transient triene (**160**).[501] The initial product (**162**) was subsequently dehydrogenated to α,β-bis(3,6-dimethoxycarbonylpyridazin-4-yl)ethylene (tetrachlorobenzoquinone, $CCl_4$, reflux, 36 h: 8%).[501]



(159) (160) (161) (162)

### 2.3.4 1,2,5-Triazepines as Substrates

5-Benzyl-3,7-diphenyl-5,6-dihydro-4*H*-triazepine (**163**) underwent oxidative ring contraction on refluxing with bromine in carbon tetrachloride for 1 h to afford 3,6-diphenylpyridazine (**164**) (73%) accompanied by some benzoic acid; 3,6-di-*p*-tolyppyridazine was made similarly (62%).[29]



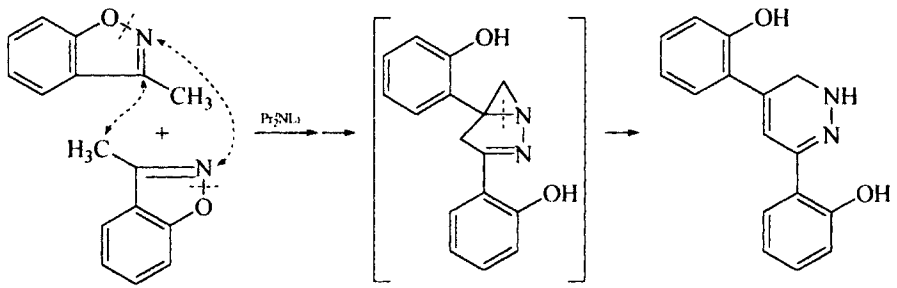
(163) (164)

## 2.4 PYRIDAZINES FROM HETEROBICYCLIC SYSTEMS

Many heterobicyclic systems have been used to make pyridazines. Such syntheses are covered briefly in the following subsections, which are arranged in alphabetical order according to the substrate system.

### 2.4.1 1,2-Benzisoxazoles as Substrates

Treatment of 3-methyl-1,2-benzisoxazole (**165**) with lithium diisopropylamide in tetrahydrofuran at − 75° for 7 min induced self-condensation, probably by a multistep process involving the key intermediate (**166**), to afford 3,5-bis-*o*-hydroxyphenyl-1,6-dihydropyridazine (**167**) which underwent oxidation by manganese dioxide in tetrahydrofuran at 25° during 1 h to give 3,5-bis-*o*-hydroxyphenylpyridazine (61% overall).[356] 3,5-Bis[2-(*t*-butyldimethylsiloxy)-6-hydroxyphenyl]pyridazine (30%) was made by a similar process,[356] but the synthesis has not been developed further.




(165) (166) (167)

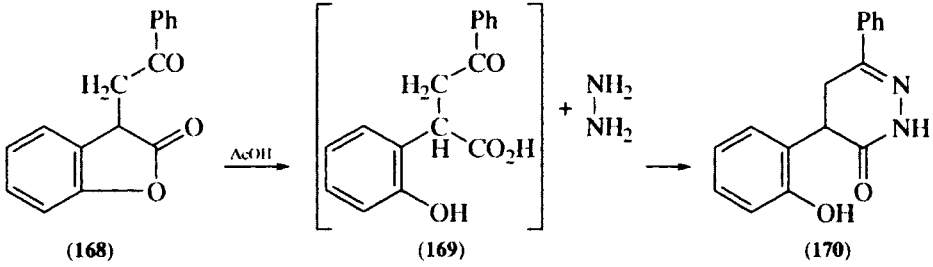
### 2.4.2 Benzofurans as Substrates

This synthesis is exemplified by the conversion of 3-phenacyl-2,3-dihydro-2-benzofuranone (**168**), which may be considered as the lactone of 3-benzoyl-2-*o*-hydroxyphenylpropionic acid (**169**), into 4-*o*-hydroxyphenyl-6-phenyl-4,5-dihydro-3(2*H*)-pyridazinone (**170**) (86%) by heating under reflux with hydrazine hydrate in acetic acid.[526]

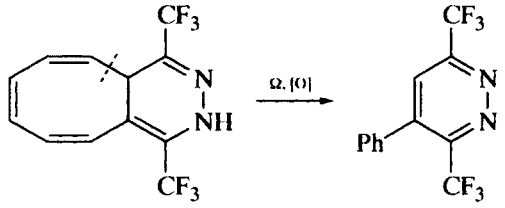
### 2.4.3 Cycloocta[*d*]pyridazines as Substrates

The sole example of this synthesis involves the (aerial?) oxidative rearrangement of 1,4-bistrifluoromethyl-2,4a-dihydrocycloocta[*d*]pyridazine

(**171**) in nitrobenzene (containing cuprous iodide) under reflux for 24 h: the product, 4-phenyl-3,6-bistrifluoromethylpyridazine (**172**) was obtained in 25% yield.[1615,cf.1503]




(**168**) (**169**) (**170**)



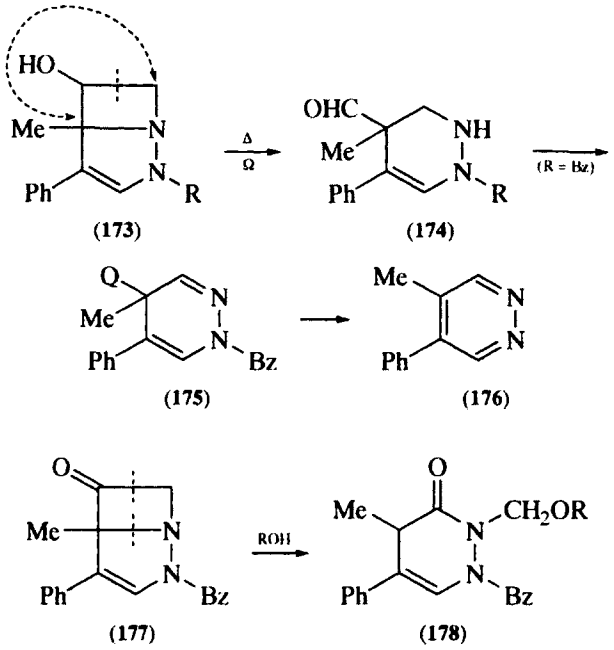

(**171**) (**172**)

### 2.4.4 1,2-Diazabicyclo[3.2.0]heptanes as Substrates

This transformation may be induced in two distinct ways, as illustrated in the following examples:

2-Acetyl- (**173**, R = Ac) or 2-benzoyl-5-methyl-4-phenyl-1,2-diaza[3.2.0]-hept-3-en-6-ol (**173**, R = Bz) underwent isomerization to 1-acetyl- (**174**, R = Ac) or 1-benzoyl-4-methyl-5-phenyl-1,2,3,4-tetrahydro-4-pyridazinecarbaldehyde (**174**, R = Bz) (PhMe, reflux, 4 h: 84% or 90%, respectively).[297] The latter product (**174**, R = Bz) was converted easily into 1-benzoyl-4-methyl-5-phenyl-1,4-dihydro-4-pyridazinecarbaldehyde (**175**, Q = CHO) (oxidation; NBS, $CH_2Cl_2$–pyridine, 25°, 6 h: 76%) and thence into either 1-benzoyl-4-methyl-5-phenyl-1,4-dihydropyridazine (**175**, Q = H) (*C*-deformylation; 1% KOH in $H_2O$–MeOH, 25°, 20 min: 58%) or 4-methyl-5-phenylpyridazine (**176**) (*C*-deformylation, *N*-debenzoylation, and oxidation; KOH, $H_2O$–MeOH, 25°, 1 h, then $O_2\downarrow$, 60 min: 45%).[297]

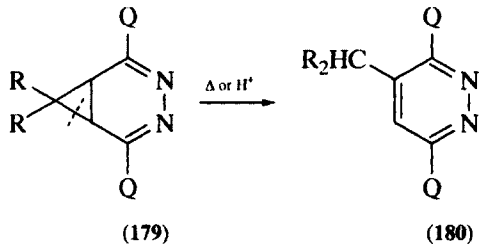
2-Benzoyl-5-methyl-4-phenyl-1,2-diazobicyclo[3.2.0]hept-3-en-6-one (**177**) afforded 1-benzoyl-2-ethoxymethyl-4-methyl-5-phenyl-1,4-dihydro-3(2*H*)-pyridazinone (**178**, R = Et) (EtOH, reflux, 8 h: ~45%, structure confirmed by X-ray analysis);[310] the isopropoxymethyl homologue (**178**, R = Pr$^i$) was made from the same substrate (Pr$^i$OH, 50°, 4 days: ~35%).[310]




### 2.4.5 3,4-Diazabicyclo[4.1.0]heptanes as Substrates

This synthesis usually involves isomerization, sometimes accompanied by subsequent changes induced by the reagents or conditions employed. Examples follow:

2,5,7,7-Tetraphenyl-3,4-diazabicyclo[4.1.0]hepta-2,4-diene (**179**, Q = R = Ph) gave 4-diphenylmethyl-3,6-diphenylpyridazine (**180**, Q = R = Ph) (HCl, dioxane, reflux, 3 h: 46%).[309]

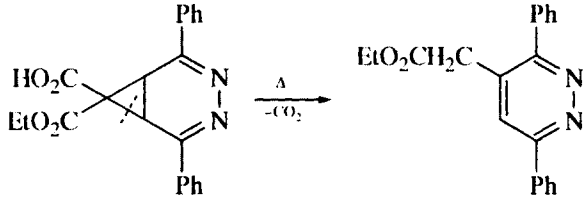



2,5-Diphenyl-7,7-bistrifluoromethyl-3,4-diazabicyclo[4.1.0] hepta-2,4-diene (**179**, Q = Ph, R = $CF_3$) gave 4-(bistrifluoromethyl) methyl-3, 6-diphenyl-pyridazine (**180**, Q = Ph, R = $CF_3$) (neat, 210°, 1 h, 90%;[45] ~ 180°: ?%).[255]

7,7-Dimethyl-2,5-diphenyl-3,4-diazabicyclo[4.1.0]hepta-2,4-diene (**179**, Q = Ph, R = Me) gave 4-isopropyl-3,6-diphenylpyridazine (**180**, Q = Ph,

R = Me) ($Me_2SO$, 160°, 30 min: 72%;[438] HCl, AcOH, reflux, 90 min: 81%).[407]

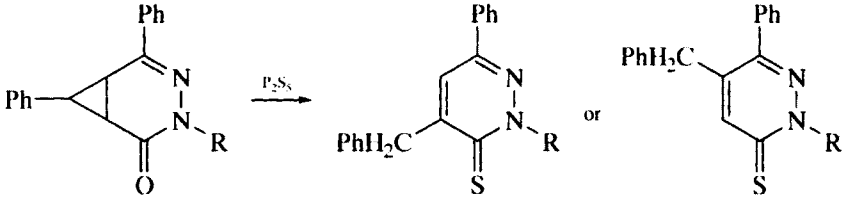
7-Ethoxycarbonyl-2,5-diphenyl-3,4-diazabicyclo[4.1.0]hepta-2,4-diene-7-carboxylic acid (**181**) gave 4-ethoxycarbonylmethyl-3,6-diphenylpyridazine (**182**) with loss of $CO_2$ (neat, 135°, 5 min: 63%).[34]



(**181**) (**182**)

5,7-Diphenyl-2,3-dihydro-3,4-diazarbicyclo[4.1.0]hept-4-en-2-one (**183**, R = H) gave either 4- (**184**, R = H) or 5-benzyl-6-phenyl-3(2*H*)-pyridazinethione (**185**, R = H) ($P_2S_5$, PhH, reflux, 7 h; 65%);[968] likewise the 3,5,7-triphenyl substrate (**183**, R = Ph) gave the 2,6-diphenyl product (**184**, R = Ph) or (**185**, R = Ph).[968]

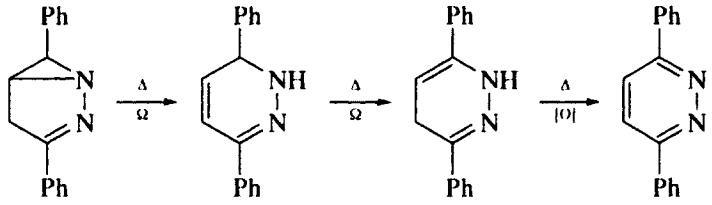
Also other examples.[28]



(**183**) (**184**) (**185**)

### 2.4.6 1,2-Diazabicyclo[3.1.0]hexanes as Substrates

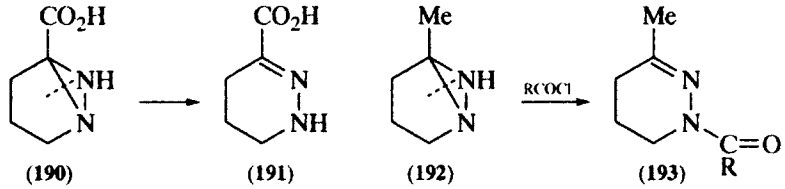
One interesting example has been reported. 3,6-Diphenyl-1,2-diazabicyclo[3.1.0]hex-2-ene (**186**) underwent isomerization in refluxing benzene during 12 h to give 3,6-diphenyl-1,6-dihydropyridazine (**187**), which, on prolonged heating, gradually afforded the more stable 1,4-dihydro isomer (**188**) and thence 3,6-diphenylpyridazine (**189**) on oxygenation.[268]



(**186**) (**187**) (**188**) (**189**)

### 2.4.7 1,6-Diazabicyclo[3.1.0]hexanes as Substrates

Only fully saturated substrates have been used in this synthesis. Thus dissolution of 1,6-diazabicyclo[3.1.0]hexane-5-carboxylic acid (**190**) in chloroform at 20° for 1 month gave the isomeric 1,4,5,6-tetrahydro-3-pyridazinecarboxylic acid (**191**) in 62% yield.[1137] In a different way, acetylation of 5-methyl-1,6-diazabicyclo[3.1.0]hexane (**192**) with acetyl chloride in dichloromethane containing triethylamine, initially at −70° and subsequently at 0° for 10 h, afforded 1-acetyl-5-methyl-1,4,5,6-tetrahydropyridazine (**193**, R = Me) in 79% yield;[1141] the same substrate (**192**) also underwent acylation by *N*-tosylprolyl chloride (−70°, 5 days) to afford 84% of 3-methyl-1-*N*-tosylprolyl-1,4,5,6-tetrahydropyridazine (**193**, R = *N*-tosylpyrrolidin-2-yl) with the structure confirmed by X-ray analysis.[1825]

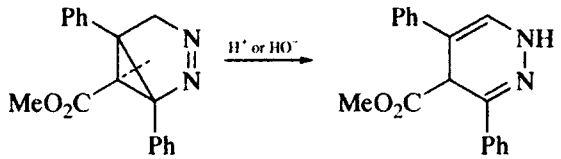



(**190**) (**191**) (**192**) (**193**)

### 2.4.8 2,3-Diazabicyclo[3.1.0]hexanes as Substrates

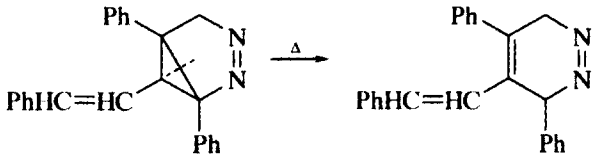
The mono unsaturated substrates have been used in this synthesis to provide a variety of hydropyridazines, as illustrated by the following examples:

Methyl 1,5-diphenyl-2,3-diazabicyclo[3.1.0]hex-2-ene-6-carboxylate (**194**) gave methyl 3,5-diphenyl-1,4-dihydro-4-pyridazinecarboxylate (**195**) ($Et_2O$, trace HCl gas, 25°, 5 min: >90%); $Et_2O$, trace KOH in EtOH, 25°, 5 min: >90%), which underwent saponification, decarboxylation, and oxidation to 3,5-diphenylpyridazine (10% NaOH–EtOH, reflux, 3 h: 82%).[563]



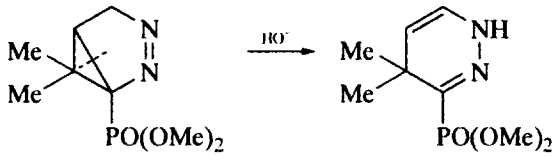

(**194**) (**195**)

1,5-Diphenyl-6-styryl-2,3-diazabicyclo[3.1.0]hex-2-ene (**196**) gave 3,5-diphenyl-4-styryl-3,6-dihydropyridazine (**197**) (xylene, reflux, A, until tlc showed to substrate: 75%).[567]



(196) (197)

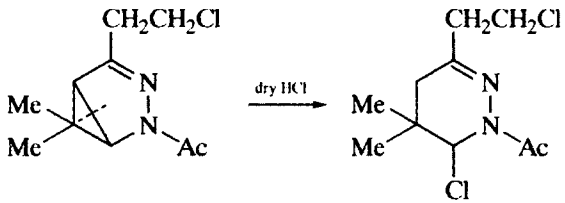
Dimethyl 6,6-dimethyl-2,3-diazabicyclo[3.1.0]hex-2-ene-1-phosphonate (**198**) gave dimethyl 4,4-dimethyl-1,4-dihydro-3-pyridazinephosphonate (**199**) ($Et_2O$, KOH–EtOH, 5°, 48 h: 91%; or silica gel/EtOAc chromatography: 57%);[9] also several analogues.[9]



(198) (199)

2-Acetyl-4-β-chloroethyl-6,6-dimethyl-2,3-diazabicyclo[3.1.0]hex-3-ene (**200**) gave 1-acetyl-6-chloro-3-β-chloroethyl-5,5-dimethyl-1,4,5,6-tetrahydropyridazine (**201**) by addition of HCl (HCl gas, $CHCl_3$, 5°, 10 min, then A↓, 5 min: >85% crude).[1824]

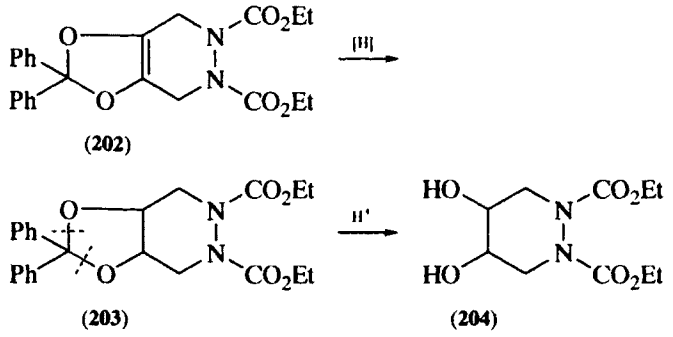
Also other examples.[14,44,1232]



(200) (201)

## 2.4.9 1,3-Dioxolo[4,5-*d*]pyridazines as Substrates

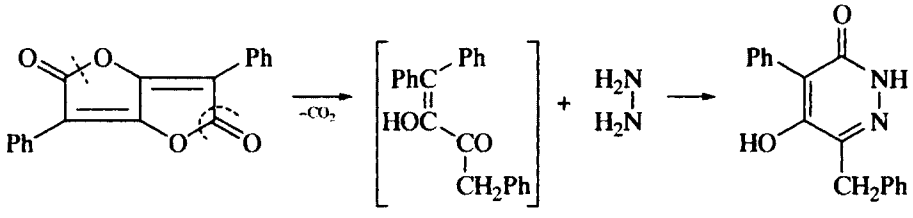
There appears to be only one reported example in this class of synthesis. Diethyl 2,2-diphenyl-4,5,6,7-tetrahydro-1,3-dioxolo[4,5-*d*]pyridazine-5,6-dicarboxylate (**202**), easily made from 4,5-dimethylene-2,2-diphenyl-1,3-dioxolane and diethyl azodicarboxylate, underwent catalytic hydrogenation over palladium/carbon in ethanol at 25° during 18 h to furnish its hexahydro analogue (**203**). This cyclic ketal afforded diethyl 4,5-dihydroxyhexahydro-1,2-pyridazinedicarboxylate (**204**) (77%) on warming in dioxane containing a little hydrochloric acid at 85° for 24 h.[901]



(202) (203) (204)

### 2.4.10 Furo[3,2-*b*]furans as Substrates

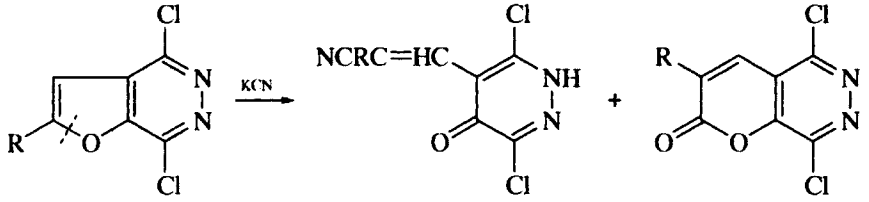
3,6-Diphenyl-2,5-dihydrofuro[3,2-*b*]furan (**205**) (the dilactone of pulvinic acid) reacted with hydrazine in refluxing glacial acetic acid during 6 h to give 6-benzyl-5-hydroxy-4-phenyl-3(2*H*)-pyridazinone (**207**) in 75% yield, possibly via the keto acid (**206**).[1296] No other examples have been reported.



(205) (206) (207)

### 2.4.11 Furo[2,3-*d*]pyridazines as Substrates

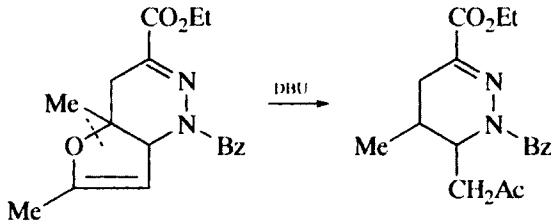
This minor synthesis is exemplified in the reaction of 4,7-dichlorofuro[2,3-*d*]pyridazine (**208**, R = H) with potassium cyanide in dimethyl sulfoxide at 25° for 4 days to afford a separable mixture of 3,6-dichloro-5-β-chlorovinyl-4(1*H*)-pyridazinone (**209**, R = H) (35%) and 5,8-dichloro-2*H*-pyrano[2,3-*d*]pyridazin-2-one (**210**, R = H) (9%).[963] However, similar treatment of even the closely analogous 2-methyl substrate (**208**, R = Me) apparently gave no pyridazine (**209**, R = Me) but only the pyranopyridazinone (**210**, R = Me).[963]



(208) (209) (210)

### 2.4.12 Furo[3,2-*c*]pyridazines as Substrates

This undeveloped synthesis is illustrated in ring fission of the easily made substrate, ethyl 1-benzoyl-4a,6-dimethyl-1,4,4a,7a-tetrahydrofuro[3,2-*c*]pyridazine (**211**), by 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU) in dichloromethane at 25° during 48 h to furnish ethyl 6-acetonyl-1-benzoyl-5-methyl-1,6-dihydro-3-pyridazinecarboxylate (**212**) of nmr-confirmed structure in 58% yield.[744]

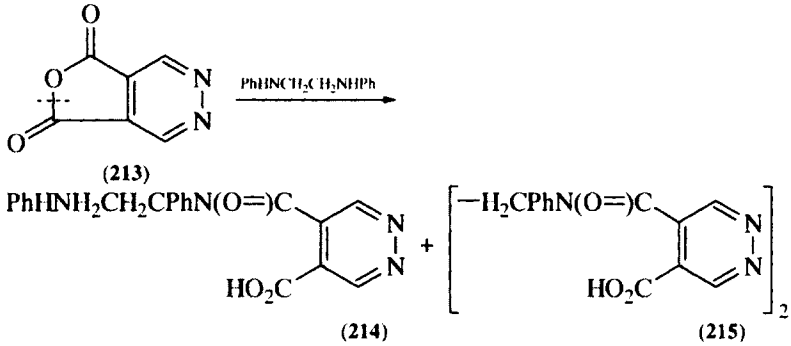



(211) (212)

### 2.4.13 Furo[3,4-*d*]pyridazines as Substrates

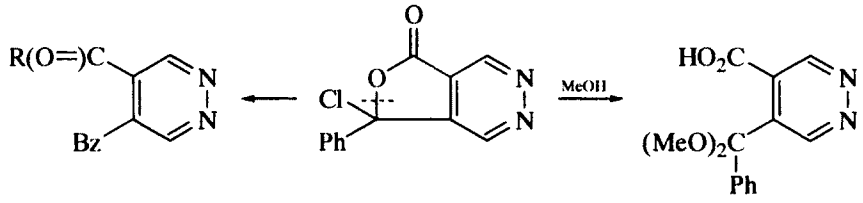
Two distinct types of substrate have been used within this class, as illustrated by the following examples:

5,7-Dihydrofuro[3,4-*d*]pyridazine-5,7-dione (**213**), namely, pyridazine-4,5-dicarboxylic anhydride, with equimolar *N,N'*-diphenylethylenediamine gave a separable mixture of 5-[*N*-(*β*-anilinoethyl)-*N*-phenylcarbamoyl]-4-pyridazinecarboxylic acid (**214**) and *N,N'*-bis (5-carboxypyridazin-4-ylcarbonyl)-*N,N'*-diphenylethylenediamine (**215**) (THF, 25°, ~24 h: ~40% each);[393] the same substrate (**213**) with ammonia in THF at 25° gave the simple analogue, 5-carbamoyl-4-pyridazinecarboxylic acid (90%);[958] and the same substrate (**213**) with a freshly made solution of 1,4-difluoro-2-sodiobenzene in THF gave 5-(2,5-difluorobenzoyl)-4-pyridazinecarboxylic acid (THF, $N_2$, −80°→20°, 12 h: 59%).[1697] Other such ring fissions have been reported.[656]




(213) (214) (215)

7-Chloro-7-phenyl-5,7-dihydrofuro[3,4-*d*]pyridazin-5-one (**217**) gave methyl 5-benzoyl-4-pyridazinecarboxylate (**216**, R = OMe) (MeOH, pyridine, 25°, 24 h: 77%), 5-benzoyl-4-pyridazinecarboxylic acid dimethyl acetal (**218**) (MeOH, 60°, 2 h; then $NH_3\downarrow$: 70%, initially as the ammonium salt), 5-benzoyl-*N*,*N*-diethyl-4-pyridazinecarboxamide (**216**, $R = NEt_2$) ($Et_2NH$, $H_2O$, 25°, 2 h: 72%; or $Et_2NH$, MeAc, 0° → 25°, 2 h: 75%), or other related products.[46]

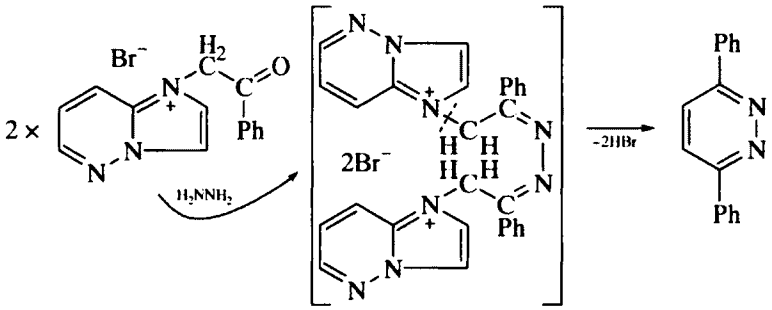



(216) (217) (218)

## 2.4.14 Imidazo[1,2-*b*]pyridazines as Substrates

Several such substrates have been used to afford pyridazines, as illustrated in the following examples:

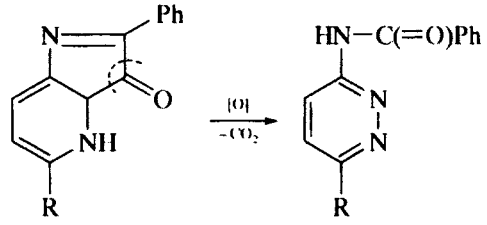
6-Chloro-1-phenacylimidazo[1,2-*b*]pyridazinium bromide (**219**) and hydrazine hydrate gave 3,6-diphenylpyridazine (**221**), perhaps via the intermediate azine (**220**) (neat, reflux, 30 min: 78%; note that only the side chain of the substrate is involved directly in forming the pyridazine!).[918]



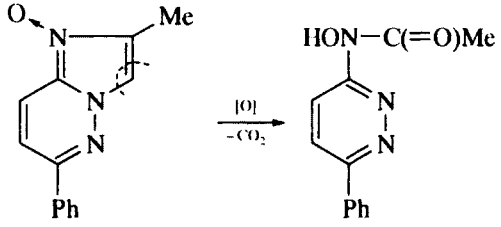

(219) (220) (221)

6-Chloro-2-phenylimidazo[1,2-*b*]pyridazin-3(5*H*)-one (**222**, R = Cl) gave 3-benzamindo-6-chloropyridazine (**223**, R = Cl) with loss of $CO_2$($H_2O_2$, 1M NaOH, warmed until colorless: ?%); the dechloro substrate (**222**, R = H) likewise gave 3-benzamidopyridazine (**223**, R = H).[1140]

2-Methyl-6-phenylimidazo[1,2-*b*]pyridazine or its 1-oxide (**224**) gave 3-*N*-acetyl(hydroxyamino)-6-phenylpyridazine (**225**) with loss of $CO_2$ (*m*-$ClC_6H_4CO_3H$,? conditions: 38%).[193]



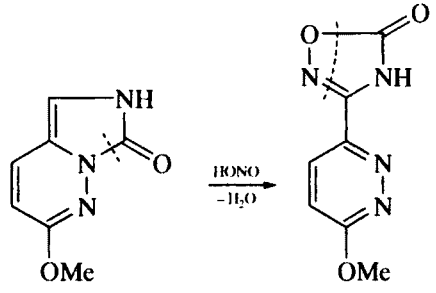

(222) (223)




(224) (225)

## 2.4.15 Imidazo[1,5-*b*]pyridazines as Substrates

When 2-methoxyimidazo[1,5-*b*]pyridazin-7(6*H*)-one (**226**) in aqueous acetic acid was treated with sodium nitrite at $< 5°$ for 1 h, ring fission and recyclization occurred to give 3-methoxy-6-(5-oxo-1,2,4-oxadiazolin-3-yl)pyridazine (**227**) in 82% yield.[964] This route to pyridazines appears to have little potential for development.

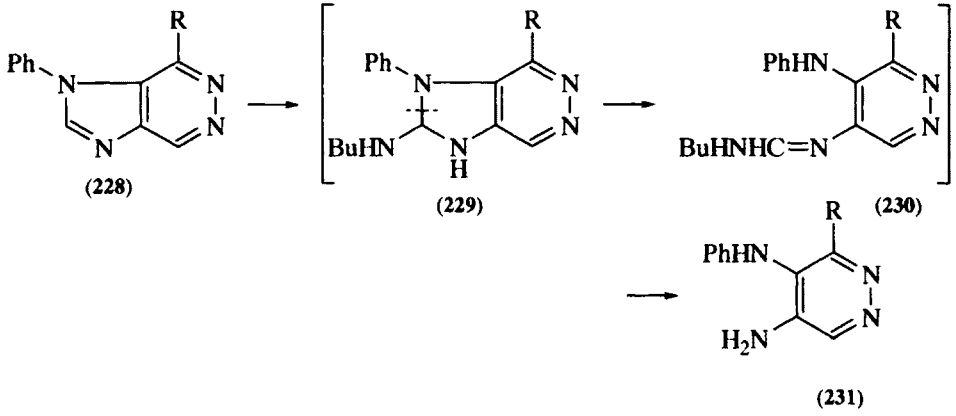



(226) (227)

## 2.4.16 Imidazo[4,5-*d*]pyridazines as Substrates

Although both 7-chloro-(**228**, R = Cl) and 7-methylsulfonyl-1-phenyl-1*H*-imidazo[4,5-*d*]pyridazine (**228**, R = $SO_2Me$) undergo the expected displace-

ments of their leaving groups with reagents such as carbanions or methoxide ion with primary amines, they behave differently. Thus the chloro substrate (**228**, R = Cl) and neat butylamine in a sealed tube at 180° for 6 h afforded 5-anilino-6-chloro-4-pyridazinamine (**231**, R = Cl) in 65% yield, presumably by addition of the amine, ring fission, and loss of *N*-butylformamide as indicated in formulas **228–231** (R = Cl); the sulfone (**228**, R = $SO_2Me$) likewise gave 5-anilino-6-methylsulfonyl-4-pyridazinamine (**231**, R = $SO_2Me$) ($BuNH_2$, 150°, 4 h: 53%).[887]



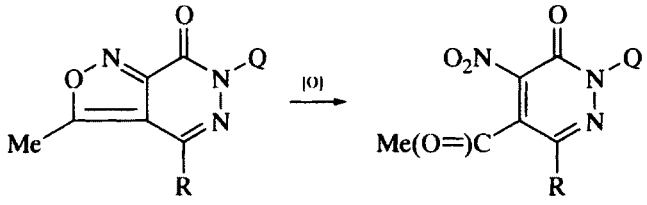

(228) (229) (230) (231)

## 2.4.17 Isoxazolo[3,4-*d*]pyridazines as Substrates

These substrates have been used quite extensively to make pyridazines by oxidative or reductive procedures, as illustrated in the following examples:

### Oxidative Routes

3,4,6-Trimethylisoxazolo[3,4-*d*]pyridazin-7(6*H*)-one (**232**, Q = R = Me) gave 5-acetyl-2,6-dimethyl-4-nitro-3(2*H*)-pyridazinone (**233**, Q = R = Me) [$Ce(NH_4)_2(NO_3)_6$, $HNO_3$, AcOH, $H_2O$, 60°, 15 min: 42%); also homologues.[132,1997]




(232) (233)

6-β-Cyanoethyl-3-methyl-4-phenylisoxazolo[3,4-*d*]pyridazin-7(6*H*)-one (**232**, Q = $CH_2CH_2CN$, R = Ph) gave 5-acetyl-2-β-cyanoethyl-4-nitro-6-

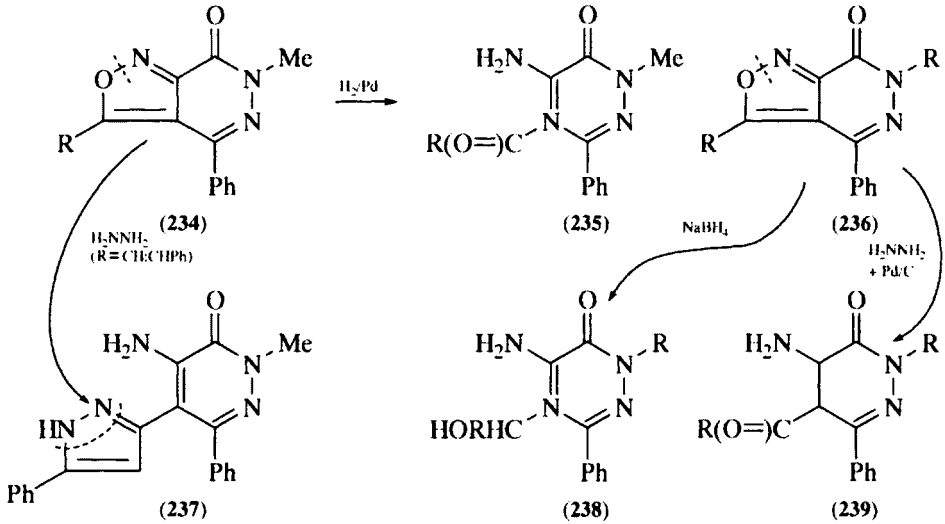
phenyl-3(2*H*)-pyridazinone (**232**, Q = $CH_2CH_2CN$, R = Ph) [$Ce(NH_4)_2(NO_3)_6$, $HNO_3$, AcOH, $H_2O$, 60°, 1 h: 38%]; also analogues.[1769]

### Reductive Routes

3,6-Dimethyl-4-phenylisoxazolo[3,4-*d*]pyridazin-7(6*H*)-one (**234**, R = Me) gave 5-acetyl-4-amino-2-methyl-6-phenyl-3(2*H*)-pyridazinone (**235**, R = Me) (EtOH, Pd/C, $H_2$, 2–3 atm, 25°, 15 h: 91%);[223] 6-methyl-4-phenylisoxazolo[3,4-*d*]pyridazin-7(6*H*)-one (**234**, R = H) likewise gave 5-amino-1-methyl-6-oxo-3-phenyl-1,6-dihydro-4-pyridazinecarbaldehyde (**235**, R = H) (20 min: 78%);[1501] and other such examples have been reported.[1769,1832]

4-Phenylisoxazolo[3,4-*d*]pyridazin-7(6*H*)-one (**236**, R = H) gave 4-amino-5-hydroxymethyl-6-phenyl-3(2*H*)-pyridazinone (**238**, R = H) ($NaBH_4$, $Me_2SO$–$H_2O$, 25°, 4 h: 47%); 3,6-dimethyl-4-phenylisoxazolo[3,4-*d*]-pyridazin-7(6*H*)-one (**236**, R = Me) likewise gave 4-amino-5-α-hydroxyethyl-2-methyl-6-phenyl-3(2*H*)-pyridazinone (**238**, R = Me) (78%); and other analogues were so made.[1590]

In contrast, 4-phenylisoxazolo[3,4-*d*]pyridazin-7(6*H*)-one (**236**, R = H) gave 5-amino-6-oxo-3-phenyl-1,6-dihydro-4-pyridazinecarbaldehyde (**239**, R = H) ($H_2NNHPh$, Pd/C, EtOH, reflux, 10 min: 40%); and 3,6-dimethyl-4-phenylisoxazolo[3,4-*d*]pyridazin-7(6*H*)-one (**236**, R = Me) gave 5-acetyl-4-amino-2-methyl-6-phenyl-3(2*H*)-pyridazinone (**239**, R = Me) ($H_2NNH_2.H_2O$, Pd/C, EtOH, reflux, 15 min: 94%).[1590]

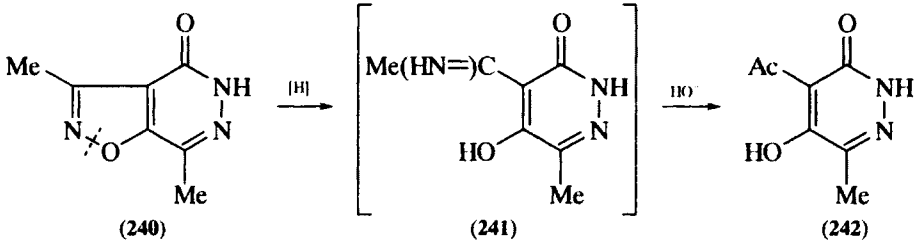



3,6-Dimethyl-4-phenylisoxazolo[3,4-*d*]pyridazin-7(6*H*)-one (**234**, R = Me) gave 5-acetyl-4-amino-2-methyl-6-phenyl-3(2*H*)-pyridazinone (**235**, R = Me) ($H_2NNH_2.H_2O$, EtOH, reflux, 10 h: 60%).[241]

6-Methyl-4-phenyl-3-styrylisoxazolo[3,4-*d*]pyridazin-7(6*H*)-one (**234**, R = CH:CHPh) gave 4-amino-2-methyl-6-phenyl-5-(5-phenylpyrazol-3-yl)-3(2*H*)-pyridazinone (**237**) (excess $H_2NNH_2.H_2O$, EtOH, reflux, 15 min: 59%);[223] also other such examples.[223,1769]

### 2.4.18 Isoxazolo[4,5-*d*]pyridazines as Substrates

Although this system is somewhat rare, some derivatives are not difficult to make and may be converted into acylpyridazines by reduction and subsequent hydrolysis. Thus hydrogenation of 3,7-dimethylisoxazolo[4,5-*d*]pyridazin-4(5*H*)-one (**240**) over Pd/C in ethyl acetate induces ring-fission to the unisolated imine (**241**), which underwent hydrolysis in refluxing 0.5M alkali during 10 min to afford 4-acetyl-5-hydroxy-6-methyl-3(2*H*)-pyridazinone (**242**) in 69% yield; several homologues were made similarly.[1122]




(240) (241) (242)

### 2.4.19 7-Oxa-3,4-diazabicyclo[4.1.0]heptanes as Substrates

The conversion of such reduced 4,5-epoxypyridazines into pyridazines is covered in Section 5.4.2.4.

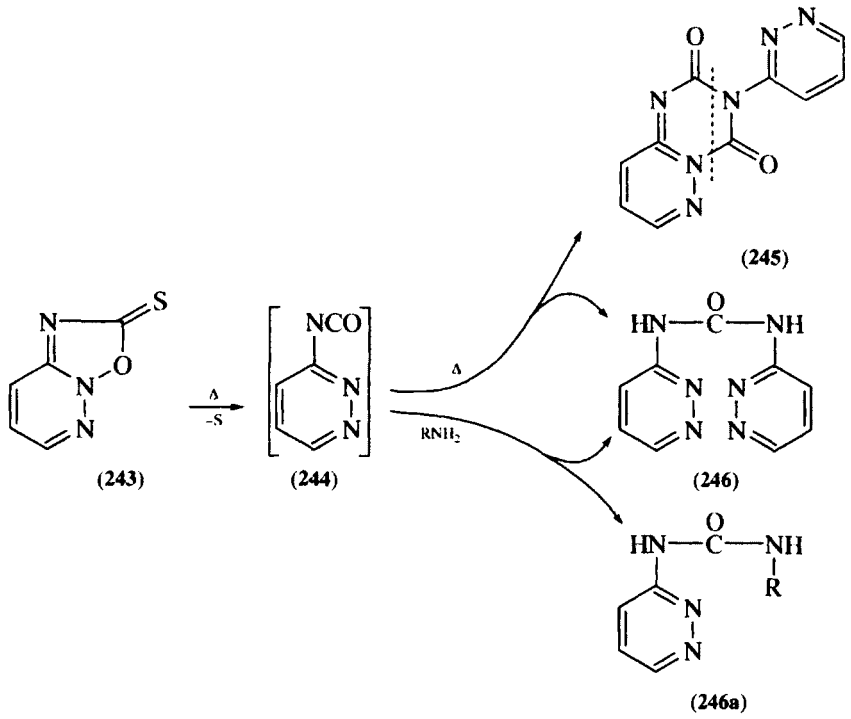
### 2.4.20 [1,2,4]Oxadiazolo[2,3-*b*]pyridazines as Substrates

Although more of interest than utility, 2*H*-[1,2,4]oxadiazolo[2,3-*b*]pyridazine-2-thione (**243**) can afford a variety of ureidopyridazines in two ways.

Thermolysis of the substrates (**243**) in dimethyl sulfoxide at 80° for 1 h gave two products: 3-(pyridazin-3-yl)-2*H*-pyridazino[1,6-*a*]-1,3,5-triazine-2,4(3*H*)-dione (**245**) [78%; a dimer of the presumed intermediate isocyanate (**244**)] and *N*,*N*′-bispyridazin-3-ylurea (**246**) (20%); other solvents produced different yields.[835,1856]

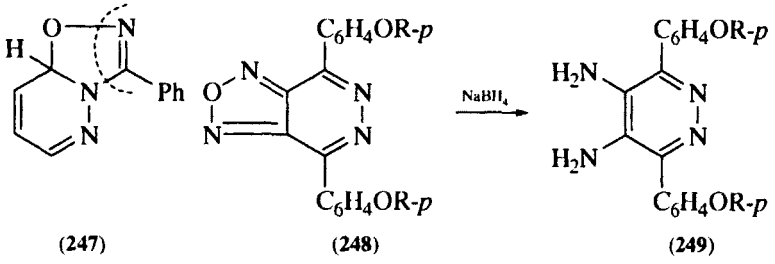
More satisfactory was the reaction of substrate (**243**) with a variety of primary or secondary amines, also in dimethyl sulfoxide at ~80% for 1–2 h. Thus 3-pyridazinamine gave *N*,*N*′-bispyridazin-3-ylurea (**246**) in 82% yield, aniline gave 3-(*N*′-phenylureido)pyridazine (**246a**, R = Ph) (41–74%), 2-

pyrimidinamine gave 3-(*N'*-pyrimidin-2-ylureido)pyridazine (78%), piperidine gave 3-(*C*-piperidinoformamido)pyridazine [**246a**, NHR=N$(CH_2)_5$] (59%), and other such products were similarly made.[835,1856]




## 2.4.21 1,2,4-Oxadiazolo[4,5-*b*]pyridazines as Substrates

This undeveloped route to pyridazines is represented by a single observation. Pyridazine reacted with benzonitrile oxide (prepared in situ) to give 3-phenyl-8a*H*-1,2,4-oxadiazolo[4,5-*b*]pyridazine (**247**). This proved stable as a solid, but in benzene solution it underwent aerial oxidation in daylight during 2 weeks to afford 3(2*H*)-pyridazinone in 90% yield.[1878]



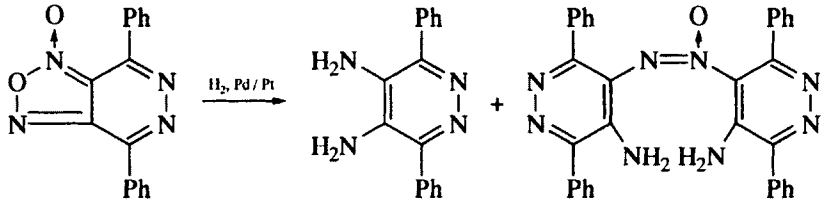
### 2.4.22 [1,2,5]Oxadiazolo[3,4-*d*]pyridazines as Substrates

These substrates may be reduced to furnish mainly pyridazinediamines as illustrated in the following examples:

4,7-Bis(*p*-butoxyphenyl)[1,2,5]oxadiazolo[3,4-*d*]pyridazine (**248**, R = Bu) gave 3,6-bis(*p*-butoxyphenyl)-4,5-pyridazinediamine (**249**, R = Bu) ($NaBH_4$, EtOH, reflux, 15 min: 53%); the 3,6-bis(*p*-octyloxyphenyl)homologue (**249**, $R = C_8H_{17}$) was made similarly (25 min: 65%).[1683]

4,7-Diphenyl[1,2,5]oxadiazolo[3,4-*d*]pyridazine 1-oxide (**250**) gave a separable mixture of 3,6-diphenyl-4,5-pyridazinediamine (**251**) and 5-(5-amino-3,6-diphenylpyridazin-4-ylazoxy)-3,6-diphenyl-4-pyridazinamine (**252**) ($Me_2NCHO$ or AcOH, Pd/C or $PtO_2$, $H_2$, 4 atm, 50–60°, 3 h: 52% and 28%, respectively; limited $H_2$ at 30° favored the azoxy product);[1358] several tolyl or other analogues were made similarly.[1358]

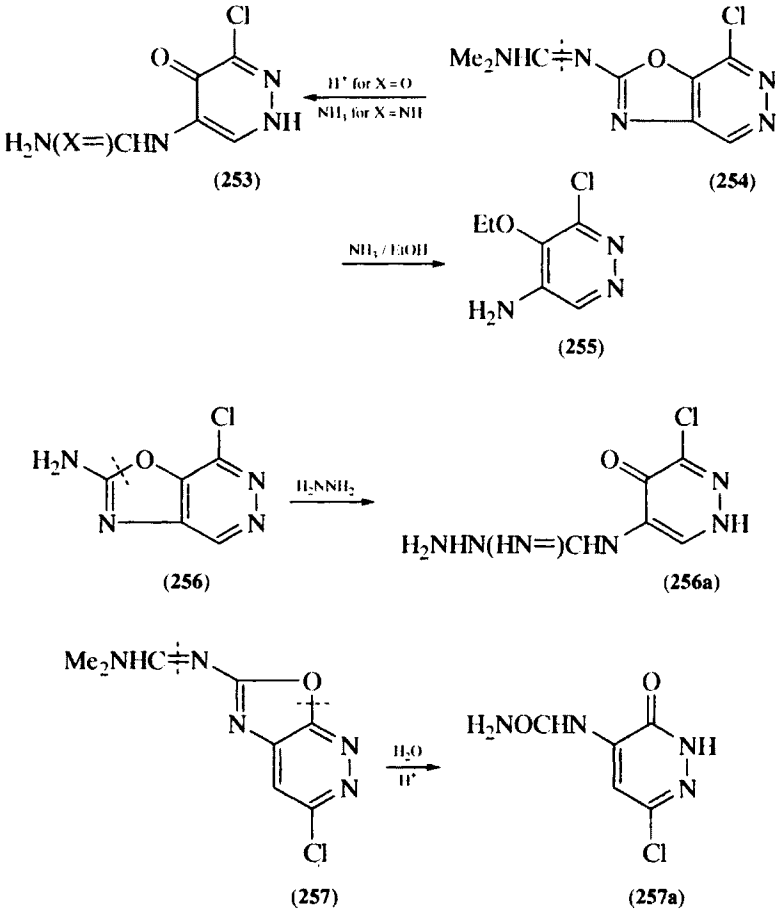
Also other examples.[601,650]




(250) (251) (252)

### 2.4.23 Oxazolo[4,5-*d*]- and Oxazolo[5,4-*c*]pyridazines as Substrates

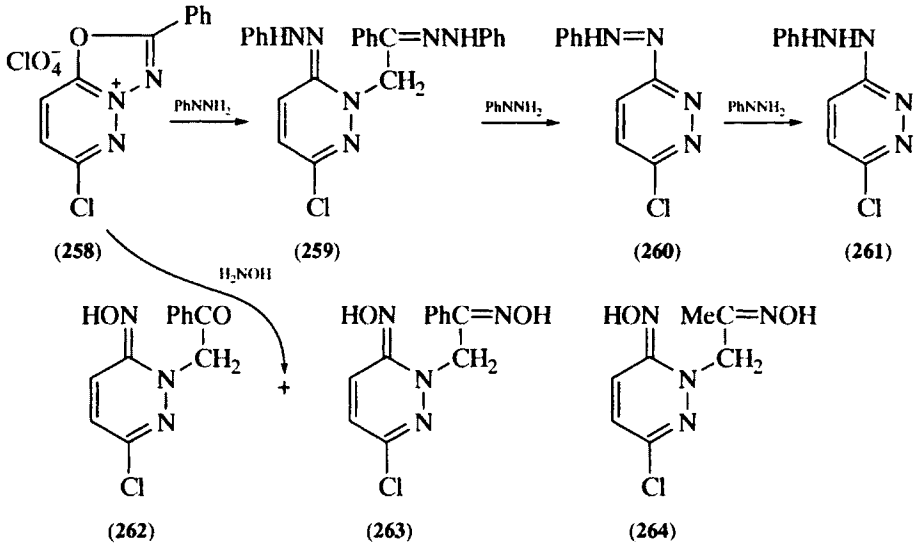
Available examples of these conversions are somewhat specialized but of some practical use. Thus 7-chloro-2-(dimethylaminomethyleneamino)oxazolo[4,5-*d*]pyridazine (**254**) has been converted into 3-chloro-5-ureido-4(1*H*)-pyridazinone (**253**, X = O) (10M HCl, reflux, 2 h: 70%), 3-chloro-5-guanidino-4(1*H*)-pyridazinone (**253**, X = NH) ($NH_3$, EtOH, 100°, sealed, 24 h: 64%), and 6-chloro-5-ethoxy-4-pyridazinamine (**255**) ($NH_3$, EtOH, 80°, sealed, 24 h: 54%);[83] in addition, 7-chlorooxazolo[4,5-*d*]pyridazin-2-amine (**256**) afforded 5-aminoguanidino-3-chloro-4(1*H*)-pyridazinone (**256a**) (49%) by heating with ethanolic hydrazine hydrate under reflux for 4 h.[83] In a broadly similar way, 3-chloro-6-dimethylaminomethyleneamino-oxazolo[5,4-*c*]pyridazine (**257**) gave 6-chloro-4-ureido-3(2*H*)-pyridazinone (**257a**) in 73% yield on hydrolysis in refluxing dilute hydrochloric acid for 2 h.[84]




### 2.4.24 Oxazolo[3,2-*b*]pyridazin-4-iums as Substrates

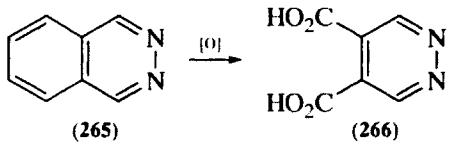
Several related oxazolo[3,2-*b*]pyridazinium perchlorates have been converted into somewhat esoteric pyridazines by treatment with phenylhydrazine or hydroxylamine. For example, 6-chloro-2-phenyloxazolo[3,2-*b*]pyridazin-4-ium perchlorate (**258**) with phenylhydrazine in dichloromethane at 20° for 24 h gave 6-chloro-2-phenacyl-3(2*H*)-pyridazinone bisphenylhydrazone (**259**) in 90% yield.[854] However, when a similar reaction mixture was heated under reflux for 24 h, the initial product (**259**) was degraded to a separable mixture of 6-chloro-3-phenylazopyridazine (**260**) (5%), 6-chloro-3-*β*-phenylhydrazinopyridazine (**261**) (37%), and phenylglyoxal bisphenylhydrazone.[854] Several analogous pyridazines were made likewise.[854] Somewhat similarly, treatment of the same substrate (**258**) with hydroxylamine in dimethylformamide under reflux for 20 h afforded a separable mixture of the monooxime, 3-chloro-6-hydroxyimino-1,6-dihydro-1-phenacylpyridazine (**262**) (25%), and 6-chloro-2-

phenacyl-3(2*H*)-pyridazinone dioxime (**263**) (36%).[398,859] Appropriate substrates gave several analogous pyridazines, such as 2-acetonyl-6-phenyl-3(2*H*)-pyridazinone dioxime (**264**) (61%).[398,859]




## 2.4.25 Phthalazines as Substrates

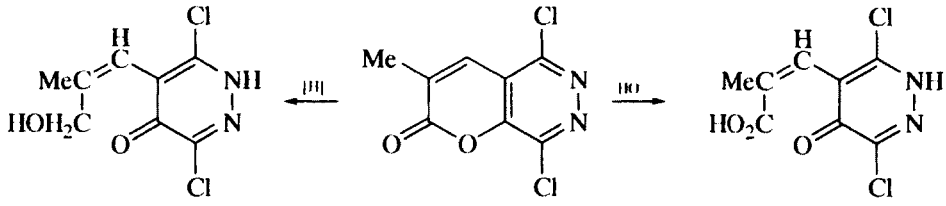
A detailed procedure, based on that of S. Gabriel[1857] for the oxidation of phthalazine (**265**) to 4,5-pyridazinedicarboxylic acid (**266**), has been reported. After slow addition of aqueous sodium permanganate to the substrate in dilute alkali at 90° with subsequent heating under reflux for 12 h, the product was purified via its barium salt and isolated in 66% yield.[1697] No other example has appeared recently.




## 2.4.26 Pyrano[2,3-*d*]pyridazines as Substrates

5,8-Dichloro-3-methyl-2*H*-pyrano[2,3-*d*]pyridazin-2-one (**268**) has been converted into two interesting pyridazines by reduction or hydrolysis, respectively. Thus treatment of the substrate (**268**) with lithium aluminum hydride in THF at 0°→20° during 1 h gave 3,6-dichloro-5-(3-hydroxy-2-methylprop-1-enyl)-4(1*H*)-pyridazinone (**267**) in 32% yield;[963] in contrast,

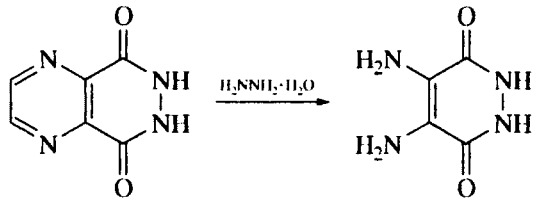
treatment of the same substrate (**268**) in 2.5M sodium hydroxide at 25° for 1 h afforded 5-(2-carboxypropl-1-enyl)-3,6-dichloro-4(1*H*)-pyridazinone (**269**) in 95% yield.[963]



(267) (268) (269)

## 2.4.27 Pyrazino[2,3-*d*]pyridazines as Substrates

Pyrazino[2,3-*d*]pyridazine-5,8(6*H*,7*H*)-dione (**270**) and several derivatives or analogues have been converted into 4,5-diamino-3,6(1*H*,2*H*)-pyridazinedione (**271**) or the like by cleavage of the pyrazine ring with hydrazine hydrate.[1401]



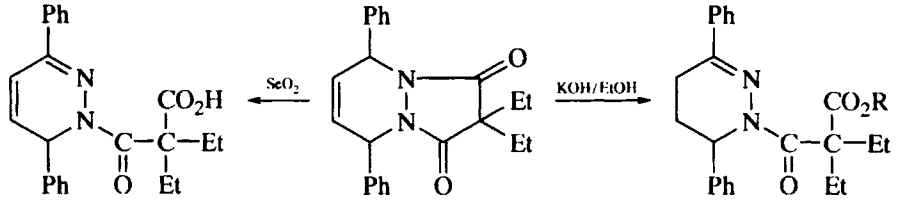
(270) (271)

## 2.4.28 Pyrazolo[1,2-*a*]pyridazines as Substrates

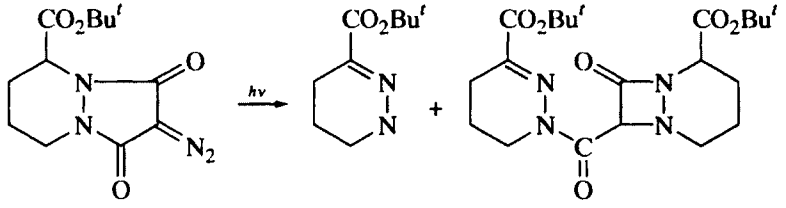
Only hydro derivatives of these substrates have been converted into hydropyridazines. Thus 2,2-diethyl-5,8-diphenyl-2,3,5,8-tetrahydro-1*H*-pyrazolo[1,2-*a*]pyridazine-1,3-dione (**273**) underwent oxidative ring fission on treatment with selenium dioxide in refluxing acetic acid during 4 h to give 1-(2-carboxy-2-ethylbutyryl)-3,6-diphenyl-1,6-dihydropyridazine (**272**) in 72% yield;[347] the same substrate (**273**) underwent hydrolytic ring fission in aqueous ethanolic alkali under reflux for 6 h to furnish a separable mixture of 1-(2-ethoxycarbonyl-2-ethylbutyryl)- (**274**, R = Et) and 1-(2-carboxy-2-ethylbutyryl)-3,6-diphenyl-1,4,5,6-tetrahydropyridazine (**274**, R = H) in 33% and 62% yields, respectively.[890]

Irradiation of *t*-butyl 2-diazo-1,3-dioxohexahydro-1*H*-pyrazolo[1,2-*a*]pyridazine-5-carboxylate (**275**) in ether gave a separable mixture of *t*-butyl 1,4,5,6-tetrahydro-3-pyridazinecarboxylate (**276**) (51%) and *t*-butyl 7-(3-*t*-butoxycarbonyl-1,4,5,6-tetrahydropyridazin-1-yl)carbonyl-8-oxo-1,6-diazabiclo[4.2.0]

octane-2-carboxylate (**277**) (11%); the latter was confirmed in structure by X-ray analysis.[742]



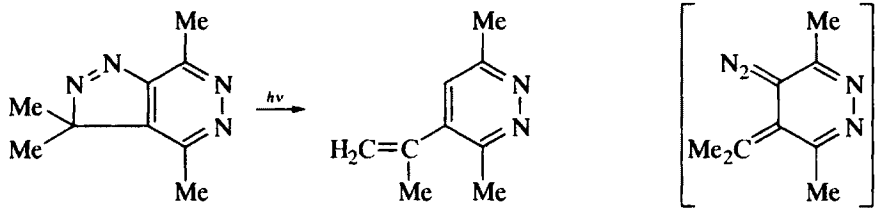
(**272**) (**273**) (**274**)



(**275**) (**276**) (**277**)

## 2.4.29 Pyrazolo[3,4-*d*]pyridazines as Substrates

Irradiation of 3,3,4,7-tetramethyl-3*H*-pyrazolo[3,4-*d*]pyridazine (**278**) in pentane–dichloromethane at 5° with 365 nm light on a small preparative scale gave mainly 4-isopropenyl-3,6-dimethylpyridazine (**279**);[1567] irradiation in $N_2$ or A at −263° with 350 nm light on a nonpreparative scale gave (on spectroscopic evidence) the same product (**279**) probably via 4-diazo-5-isopropylidene-3,6-dimethyl-4,5-dihydropyridazine (**280**).[1567]

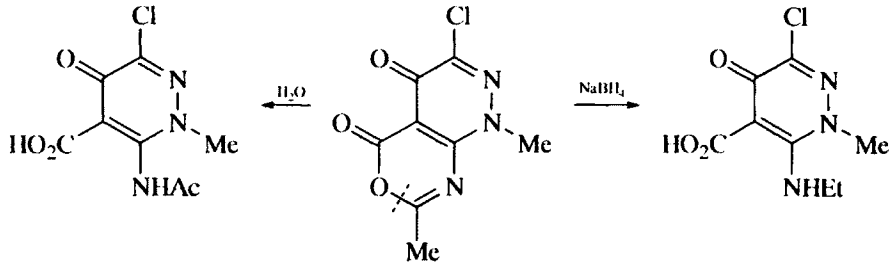


(**278**) (**279**) (**280**)

## 2.4.30 Pyridazino[3,4-*d*][1,3]oxazines as Substrates

3-Chloro-1,7-dimethyl-4*H*-pyridazino[3,4-*d*][1,3]oxazine-4,5(1*H*)-dion (**282**) has been converted into 3-acetamido-6-chloro-2-methyl-5-oxo-2,5-dihydro-4-pyridazinecarboxylic acid (**281**) in 75% yield by hydrolysis in boiling water for 30 min;[967] the same substrate has been converted also into 6-chloro-3-ethyl-

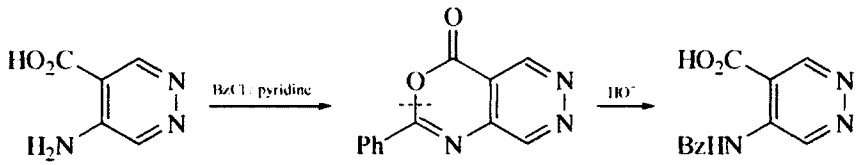
amino-2-methyl-5-oxo-2,5-dihydro-4-pyridazinecarboxylic acid (**283**) in 95% yield by reductive cleavage with sodium borohydride in ethanol at 25° for 3 h.[967]




(**281**) (**282**) (**283**)

## 2.4.31 Pyridazino[4,5-*d*][1,3]oxazines as Substrates

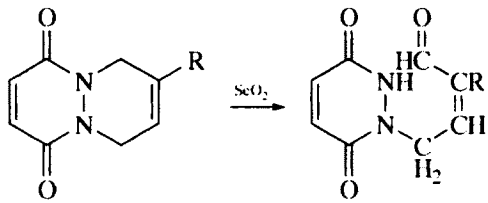
In an attempt to *N*-benzoylate 5-amino-4-pyridazinecarboxylic acid (**284**) with benzoyl chloride in pyridine, cyclization was effected to give 3-phenyl-4*H*-pyridazino[4,5-*d*][1,3]oxazin-4-one (**285**) in 68% yield; this readily underwent alkaline hydrolysis at 25° during 24 h to afford 5-benzamido-4-pyridazinecarboxylic acid (**286**) ( ~ 90%).[958] Apparently no other such syntheses have been reported.




(**284**) (**285**) (**286**)

## 2.4.32 Pyridazino[1,2-*a*]pyridazines as Substrates

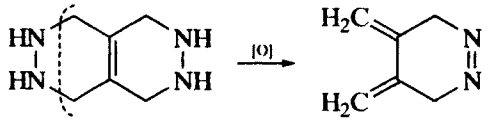
Oxidation of 1,4,6,9-tetrahydropyridazino[1,2-*a*]pyridazine (**287**, R = H) or its 7-methyl derivative (**287**, R = Me) with selenium dioxide furnished 1-(γ-formylallyl)- (**288**, R = H) or 1-(3-formylbut-2-enyl)-3,6(1*H*,2*H*)-pyridazinedione (**288**, R = Me).[664]




(**287**) (**288**)

### 2.4.33 Pyridazino[4,5-*d*]pyridazines as Substrates

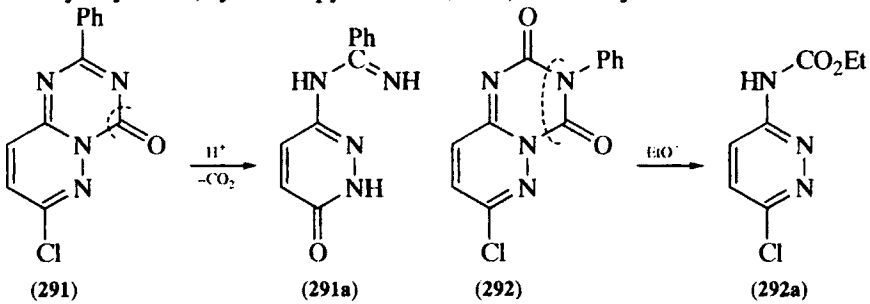
The oxygen-sensitive 1,2,3,4,5,6,7,8-octahydropyridazino[4,5-*d*]-pyridazine (**289**) underwent oxidation by nickel peroxide in carbon tetrachloride–chloroform at −78° to afford an isomer of 4,5-dimethylpyridazine, identified in cold solution as 4,5-dimethylene-3,4,5,6-tetrahydropyridazine (**290**) by its nmr spectrum; it could not be isolated without decomposition.[623]



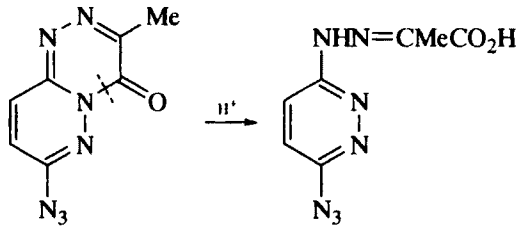
(289) (290)

### 2.4.34 Pyridazino[1,6-*a*]-1,3,5- and Pyridazino[6.1-*c*][1,2,4]triazines as Substrates

At least three examples of the conversion of these substrates into pyridazines have been reported. 7-Chloro-2-phenyl-4*H*-pyridazino[1,6-*a*]-1,3,5-triazin-4-one (**291**) suffered hydrolysis (with loss of $CO_2$) in refluxing 2.5M hydrochloric acid during 2 h to afford 6-(α-iminobenzylamino)-3(2*H*)-pyridazinone (**291a**) in 72% yield;[295] in contrast, 7-chloro-3-phenyl-2*H*-pyridazino[1,6-*a*]-1,3,5-triazine-2,4(3*H*)-dione (**292**) in refluxing ethanolic sodium ethoxide for 15 min retained its chloro substituent but underwent more profound degradation of the triazine ring to furnish 3-chloro-6-ethoxycarbonylaminopyridazine (**392a**) in 67% yield.[295] 7-Azido-3-methyl-4*H*-pyridazino[6,1-*c*][1,2,4]triazin-4-one (**293**) in boiling 6M hydrochloric acid for 2 min afforded 3-azido-6-(α-carboxyethylidene)hydrazinopyridazine (**293a**) in 93% yield.[1008]



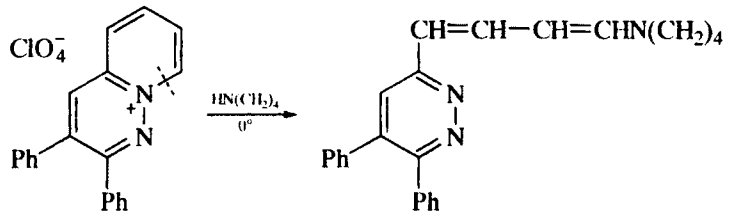
(291) (291a) (292) (292a)



(293) (293a)

### 2.4.35 Pyrido[1,2-*b*]pyridazin-9-iums as Substrates

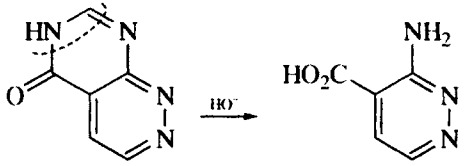
An interesting cleavage of the easily made 2,3-diphenylpyrido[1,2-*b*]pyridazin-9-ium perchlorate (**294**) occurred on treatment with pyrrolidine at 0° for 1 h; the product, 3,4-diphenyl-6-[4-(pyrrolidin-1-yl)buta-1,3-dienyl]pyridazine (**294a**) (70–80%), had a 1-*cis*-3-*trans* or a 1-*trans*-3-*trans* side chain when the reaction was carried out in acetonitrile or aqueous ethanol, respectively.[1892]



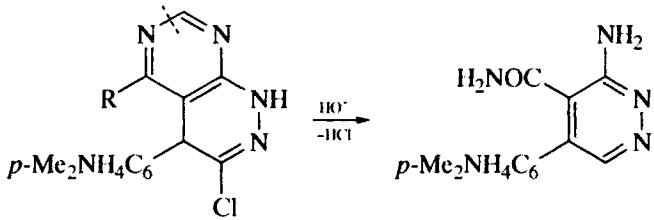

(294) (294a)

### 2.4.36 Pyrimido[4,5-*c*]pyridazines as Substrates

On meager existing evidence, this system undergoes preferential degradation of its pyrimidine ring on hydrolysis or alcoholysis to give pyridazines. Thus pyrimido[4,5-*c*]pyridazin-5(6*H*)-one (**295**) in 1M alkali under reflux for 90 min gave 3-amino-4-pyridazinecarboxylic acid (**296**) in 94% yield;[338] 3,5-dichloro-4-*p*-dimethylaminophenyl-1,4-dihydropyrimido[4,5-*c*]pyridazine (**297**, R = Cl) or 3-chloro-4-*p*-dimethylaminophenyl-1,4-dihydropyrimido[4,5-*c*]pyridazin-5-amine (**297**, R = $NH_2$) in refluxing aqueous–ethanolic alkali for 1–2 h gave 3-amino-5-*p*-dimethylaminophenyl-4-pyridazinecarboxamide (**298**) in 50–75% yield, with dehydrogenation provided by elimination of HCl in each case.[1099]



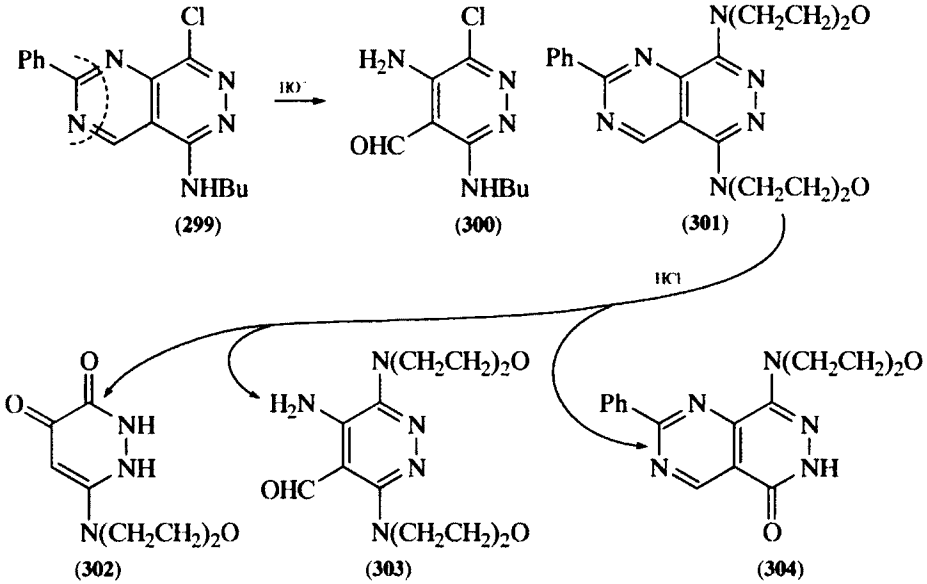

(295) (296)




(297) (298)

### 2.4.37 Pyrimido[4,5-*d*]pyridazines as Substrates

This system is prone to degradation under hydrolytic conditions. For example, 5-butylamino-8-chloro-2-phenylpyrimido[4,5-*d*]pyridazine (**299**) was digested in aqueous–ethanolic alkali to give 5-amino-3-butylamino-6-chloro-4-pyridazinecarbaldehyde (**300**) in 25% yield;[524] in contrast, hydrolysis of the related substrate, 5,8-dimorpholino-2-phenylpyrimido[4,5-*d*]pyridazine (**301**), in 10% hydrochloric acid gave a separable mixture of 8-morpholino-2-phenylpyrimido[4,5-*d*]pyridazin-5(6*H*)-one (**304**) (50%), 5-amino-3,6-dimorpholino-4-pyridazinecarbaldehyde (**303**) (2%), and 6-morpholino-3,4(1*H*,2*H*)-pyridazinedione (**302**) (46%), arising from at least two parallel pathways.[524]



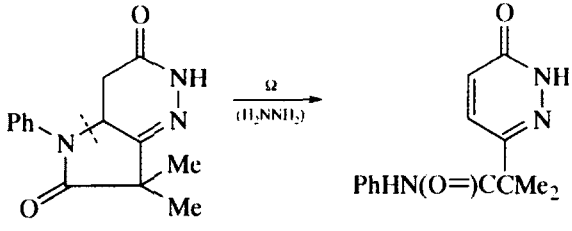

### 2.4.38 Pyrrolo[3,2-*c*]pyridazines as Substrates

7,7-Dimethyl-5-phenyl-4,4a-dihydro-7*H*-pyrrolo[3,2-*c*]pyridazine-3,6(2*H*,5*H*)-dione (**305**) isomerized in ethanolic hydrazine hydrate under reflux during 8 h to give 6-[α-methyl-α-(phenylcarbamoyl)ethyl]-3(2*H*)-pyridazinone (**306**) in 80% yield.[442]

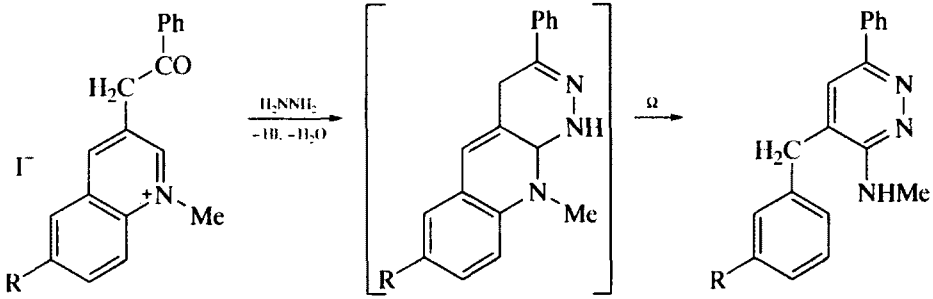
### 2.4.39 Quinolines as Substrates

This unlikely transformation is represented by two examples. Brief treatment of 3-phenacyl- (**307**, R = H) or 6-methyl-3-phenacylquinoline methiodide (**307**,

R = Me) with hydrazine hydrate in hot diethylene glycol gave ~40% of 4-*o*-methylaminobenzyl- (**309**, R = H) or 4-(3-methyl-6-methylaminobenzyl)-6-phenylpyridazine (**309**, R = Me), respectively, possibly by isomerization of the tricyclic intermediate (**308**, R = H or Me).[1797]



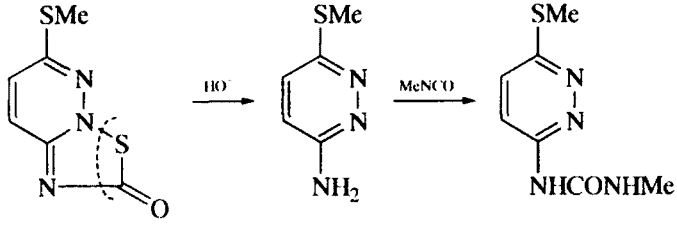

(305) (306)




(307) (308) (309)

## 2.4.40 [1,2,4]Thiadiazolo[2,3-*b*]pyridazines as Substrates

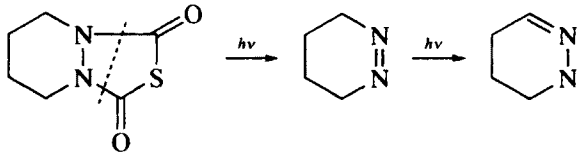
6-Methylthio-2*H*-[1,2,4]thiadiazolo[2,3-*b*]pyridazin-2-one (**310**) was converted into crude 6-methylthio-3-pyridazinamine (**311**) (by warming with 10% alkali for 30 min) and then into 3-methylthio-6-*N'*-methylureidopyridazine (**312**) by treatment with methyl isocyanate in dimethylformamide at 25° for 24 h; the overall yield was only 20%.[300] The route has not been developed further.




(310) (311) (312)

### 2.4.41 [1,3,4]Thiadiazolo[3,4-*a*]pyridazines as Substrates

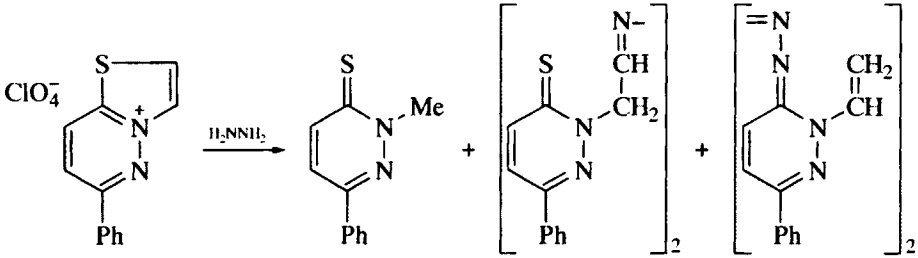
Irradiation of 4,5,6,7-tetrahydro-1*H*,3*H*-[1,3,4]thiadiazolo[3,4-*a*]pyridazin-1,3-dione (**313**) in acetonitrile with 254-nm light for 20 min caused complete conversion into 3,4,5,6-tetrahydropyridazine (**314**), isolated in 90% yield; further irradiation or prolonged standing at 25° induced isomerization to 1,4,5,6-tetrahydropyridazine (**315**).[1383]




(313) (314) (315)

### 2.4.42 Thiazolo[3,2-*b*]pyridazin-4-iums as Substrates

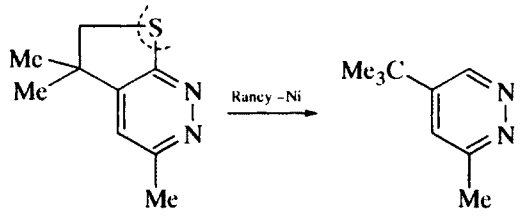
The reactions of several blandly substituted substrates of this type with hydrazine hydrate have been studied in some detail.[400,538] The number and types of products vary according to the substituent(s) and to the conditions; among the 10 types of product reported, four are pyridazines. A typical examples is the treatment of 6-phenylthiazolo[3,2-*b*]pyridazin-4-ium perchlorate (**316**) with a 10-fold molar quantity of hydrazine hydrate (in acetonitrile under reflux for 4 h) to give a separable mixture of 2-methyl-6-phenyl-3(2*H*)-pyridazinethione (**317**) (50%), β,β-azinobis[α-(3-phenyl-6-thioxo-1,6-dihydropyridazin-1-yl)ethane] (**318**) (28%), and 1,4-bis(6-phenyl-2-vinylpyridazin-3-ylidene)tetraz-2-ene (**319**) (28%).[538] Such reactions deserve development into practical pyridazine syntheses.




(316) (317) (318) (319)

### 2.4.43 Thieno[2,3-*c*]pyridazines as Substrates

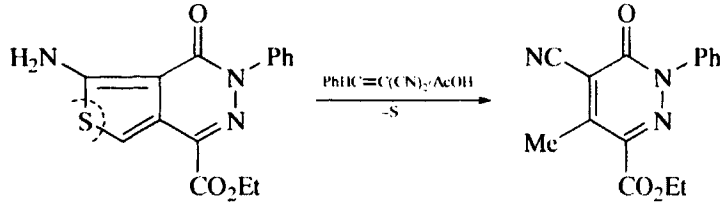
Only one briefly described example of this synthesis has been reported. 3,5,5-Trimethyl-5,6-dihydrothieno[2,3-*c*]pyridazine (**320**) underwent desulfurization by Raney nickel to give 4-*t*-butyl-6-methylpyridazine (**321**).[880]




(320) (321)

### 2.4.44 Thieno[3,4-*d*]pyridazines as Substrates

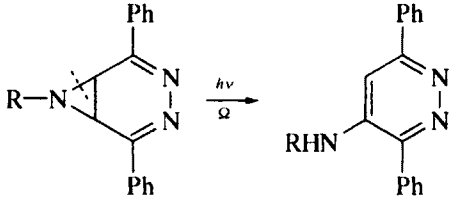
Treatment of ethyl 5-amino-4-oxo-3-phenyl-3,4-dihydrothieno[3,4-*d*]pyridazine-1-carboxylate (**322**) with α-benzylidenemalononitrile in refluxing acetic acid for 5 h afforded ethyl 5-cyano-4-methyl-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate (**323**) (36%), probably via some undetected adduct;[54] the 1-*p*-methoxyphenyl analogue (42%) was made similarly.[54]




(322) (323)

### 2.4.45 3,4,7-Triazabicyclo[4.1.0]heptanes as Substrates

This transformation has not been developed into a preparative procedure. However, irradiation of 7-benzyl-2,5-diphenyl-3,4,7-triazabicyclo[4.1.0]hepta-2,4-diene (**324**, R = $CH_2Ph$) in benzene gave the isomeric 4-benzylamino-3,6-diphenylpyridazine (**325**, R = $CH_2Ph$) as a separable minor product (6%); the 7-cyclohexyl substrate (**324**, R = $C_6H_{11}$) likewise gave 4-cyclohexylamino-3,6-diphenylpyridazine (**325**, R = $C_6H_{11}$) (8%).[252]




(324) (325)

### 2.4.46 [1,2,4]Triazolo[1,2-*a*]pyridazines as Substrates

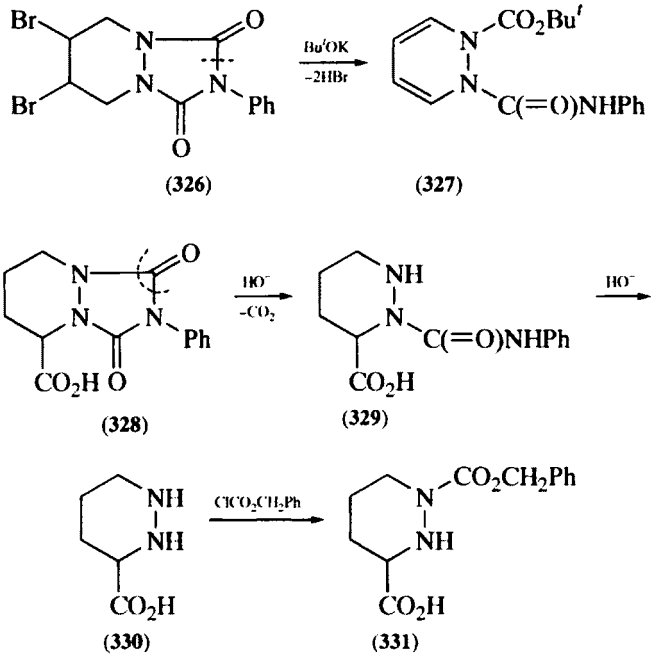
These substrates have been used to make only hydropyridazines, as illustrates in the following examples.

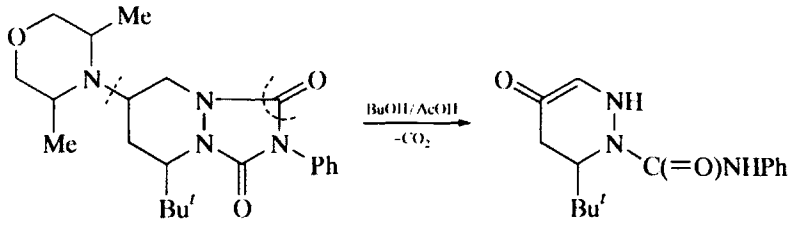
6,7-Dibromo-2-phenyl-5,6,7,8-tetrahydro-1*H*-[1,2,4]triazolo[1,2-*a*]pyridazine-1,3(2*H*)-dione (**326**) gave *t*-butyl 2-*N*-phenylcarbamoyl-1,2-dihydro-1-pyridazinecarboxylate (**327**) ($Bu^tOK$, $Me_2NCHO$, $>-55°$, 3 h: 65%; didehydrobromination but no ring fission occurred at $<-55°$).[342]

1,3-Dioxo-2-phenyl-2,3,5,6,7,8-hexahydro-1*H*-[1,2,4]triazolo[1,2-*a*]pyridazine-5-carboxylic acid (**328**) gave a separable mixture of 2-*N*-phenylcarbamoylhexahydro-3-pyridazinecarboxylic acid (**329**) and hexahydro-3-pyridazinecarboxylic acid (**330**; piperazic acid) (4M NaOH, reflux, 4 h: 65%) and "low yield", respectively;[683] alternatively, the same substrate (**328**) gave only piperazic acid (**330**), isolated as 1-benzyloxycarbonylhexahydro-3-pyridazinecarboxylic acid (**331**) (KOH, $H_2O$–BuOH, reflux, 18 h; then $ClCO_2CH_2Ph$, PhMe–$H_2O$, pH 11, 10°, 2 h: 89%).[63]

8-*t*-Butyl-6-(3,5-dimethylmorpholino)-2-phenyl-7,8-dihydro-1*H*-[1,2,4]triazolo[1,2-*a*]pyridazine-1,3(2*H*)-dione (**332**) gave 6-*t*-butyl-4-oxo-*N*-phenylhexahydro-1-pyridazinecarboxamide (**333**) ($Bu^tOK$, THF, 25°, 12 h; then AcOH–$H_2O$, 25°, 30 min: 72%).[1560]

Also other examples.[2029,2050]





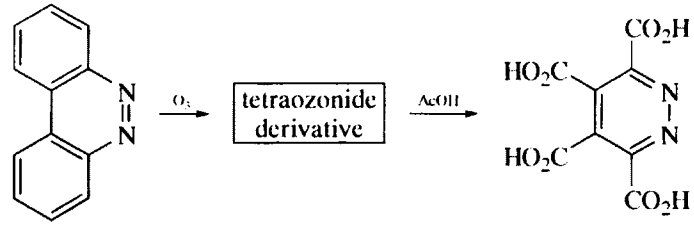

(332) (333)

## 2.5 PYRIDAZINES FROM HETEROTRICYCLIC SYSTEMS

Synthetic routes from heterotricyclic systems to pyridazines are few and offer little for development into useful general methods. Presently available procedures are illustrated by the following examples, arranged alphabetically according to the substrate system:

### From Benzo[*c*]cinnolines

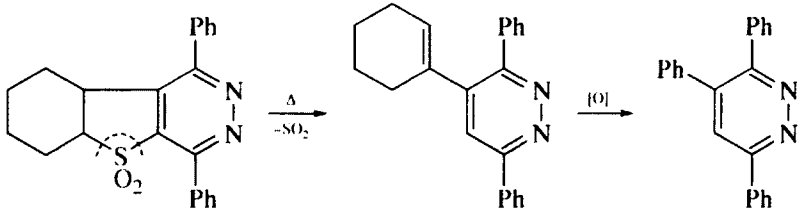
Benzo[*c*]cinnoline (**334**) gave 3,4,5,6-pyridazinetetracarboxylic acid (**335**) via a tetraozonide ($O_3$; then AcOH, 20°: 5%).[680]




(334) (335)

### From [1]Benzothieno[2,3-*d*]pyridazines

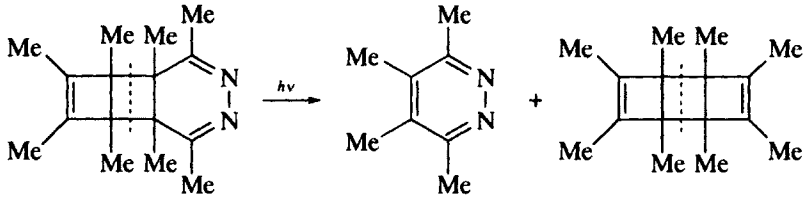
1,4-Diphenyl-5a,6,7,8,9,9a-hexahydro[1]benzothieno[2,3-*d*]pyridazine 5,5-dioxide (**336**) gave 4-cyclohex-1′-enyl-3,6-diphenylpyridazine (**337**) (neat, 210°, 10 min: 33%) and thence 3,4,6-triphenylpyridazine (**338**) (*N*-bromosuccinimide, trace $Bz_2O_2$, $CCl_4$, reflux, 20 h: 15%).[33]




(336) (337) (338)

### From 8,9-Diazatricyclo[4.4.0.0$^{2,5}$]decanes

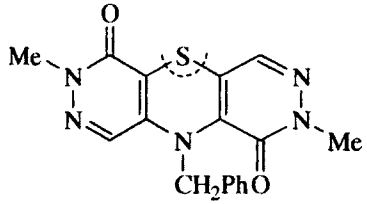
1,2,3,4,5,6,7,10-Octamethyl-8,9-diazabicyclo[4.4.0.0$^{2,5}$]deca-3,7,9-triene (**339**) gave 3,4,5,6-tetramethylpyridazine (**340**) as a major product ($CH_2Cl_2$, $h\nu$); the remainder of the substrate dimerized to the *syn*-tricyclic hydrocarbon (**340a**).[1088]



(339) (340) (340a)

### From Dipyridazino[4,5-*b*: 4′,5′-e][1,4]thiazines

10-Benzyl-2,7-dimethyl-10*H*-dipyridazino[4,5-*b*:4′,5′-*e*][1,4]thiazine-1,6(2*H*,7*H*)-dione (**341**) gave *N*-benzyl-*N*-(1-methyl-6-oxo-1,6-dihydropyridazin-4-yl)-*N*-(2-methyl-3-oxo-2,3-dihydropyridazin-4-yl)amine (Raney Ni, $Me_2NCHO$, reflux, 5 h: 74%).[867]



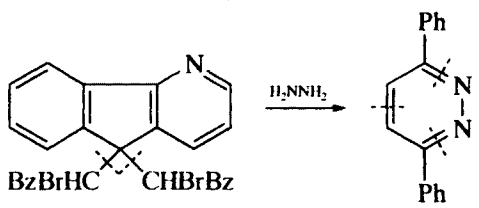
(341)

### From 6H-Furo[2′,3′: 4,5]oxazolo[3,2-*b*]pyridazines

The ring fission of one such substrate to afford a pyridazine is covered in Section 5.4.2.1.

### From Indeno[1,2-*b*]pyridines

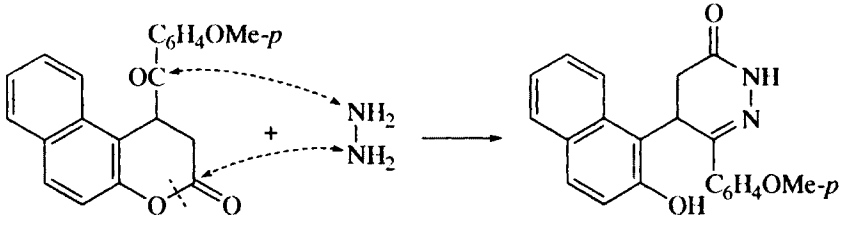
5,5-Bis(α-bromophenacyl)-5*H*-indeno[1,2-*b*]pyridine (**342**) gave 3,6-diphenylpyridazine (**343**) ($H_2NNH_2$, EtOH, 55°, 10 h: 48%; the mechanism was postulated to involve bromophenacyl radicals).[1492]



(342) (343)

## From Naphtho[2,1-*b*]pyrans

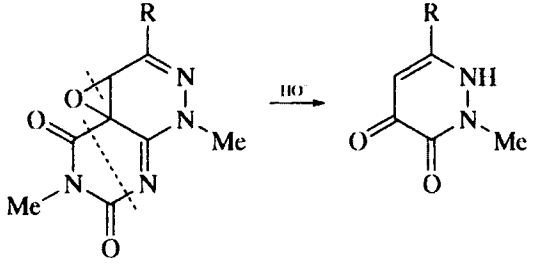
1-*p*-Anisoyl-2,3-dihydro-1*H*-naphtho[2,1-*b*]pyran-3-one (**344**) gave 5-(2-hydroxynaphthalen-1-yl)-6-*p*-methoxyphenyl-4,5-dihydro-3(2*H*)-pyridazinone (**345**) ($H_2NNH_2 \cdot H_2O$, PhH, reflux, 5 h: ?%).[1705]




(344) (345)

## From Oxireno[*d*]pyrimido [4,5-*c*]pyridazines

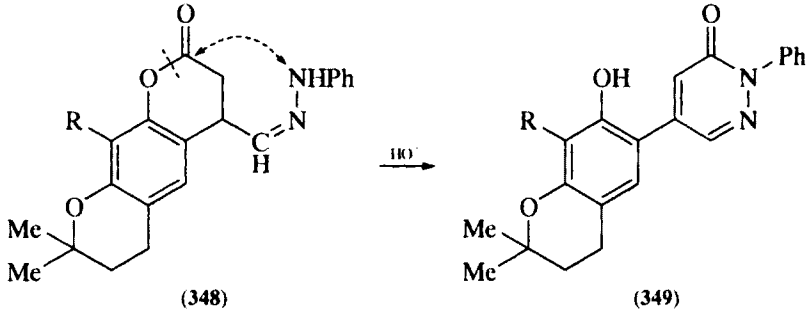
4,7-Dimethyl-2-phenyl-1a,4-dihydro-6*H*-oxireno[*d*]pyrimido[4,5-*c*]pyridazine-6,8(7*H*)-dione (**346**, R = Ph) gave 2-methyl-6-phenyl-3,4(1*H*,2*H*)-pyridazinedione (**347**, R = Ph) by a proposed multistep mechanism (2.5M NaOH, 25°, 6 h: 35%); also 6-*p*-bromophenyl- (**347**, R = $C_6H_4Br$-*p*) and 6-*p*-chlorophenyl-2-methyl-3,4(1*H*,2*H*)-pyridazinedione (**347**, R = $C_6H_4Cl$-*p*) (similarly: 43% and 50%, respectively).[842]




(346) (347)

## From Pyrano[3,2-*g*][1]benzopyrans

8,8-Dimethyl-2-oxo-6,7-dihydro-2*H*,8*H*-pyrano[3,2-*g*][1]benzopyran-4-carbaldehyde phenylhydrazone (**348**, R = H) gave 5-(7-hydroxy-2,2-

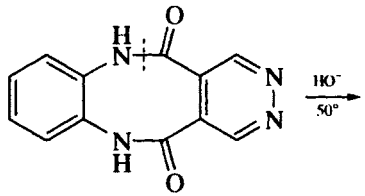



(348) (349)

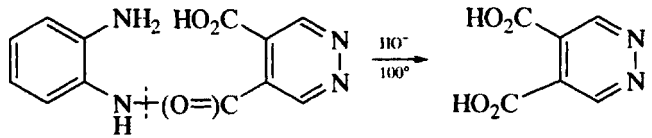
dimethylchroman-6-yl)-2-phenyl-3(2*H*)-pyridazinone (**349**, R = H) (KOH, EtOH–$H_2O$, reflux, 1 h: ~50%); likewise the 2,2,8-trimethyl analogue (**349**, R = Me).[1280]

## From Pyridazino[4,5-*c*][1,6]benzodiazocines

Pyridazino[4,5-*c*][1,6]benzodiazocine-5,12(6*H*,11H)-dione (**350**) gave either 5-*o*-aminophenylcarbamoyl-4-pyridazinecarboxylic acid (**351**) (2.5M NaOH, 50°, 5 h: ~75%) or 4,5-pyridazinedicarboxylic acid (**352**) (2.5M NaOH, reflux, 8 h: ~60%).[656]



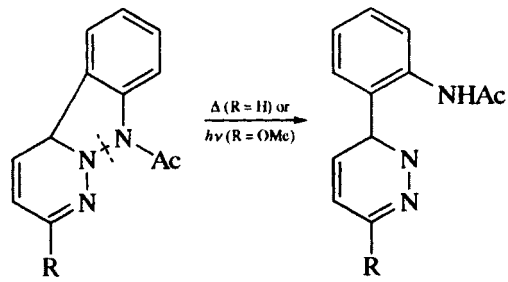
(**350**)



(**351**) (**352**)

## From Pyridazino[1,6-*b*]indazoles

9-Acetyl-4a,9-dihydropyridazino[1,6-*b*]indazole (**353**, R = H) gave 3-*o*-acetamidophenylpyridazine (**354**, R = H) (xylene, reflux 2 days: 55% after separation from another product).[797]



(**353**) (**354**)

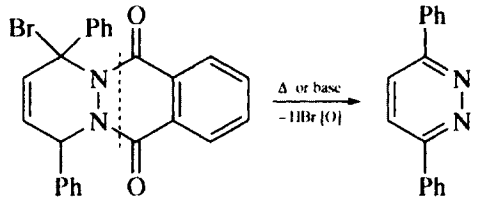
9-Acetyl-2-methoxy-4a,9-dihydropyridazino[1,6-*b*]indazole (**353**, R = OMe) gave, among other products, 3-*o*-acetamidophenyl-6-methoxypyridazine

(**354**, R = OMe) ($h\nu$, ? conditions: 27%); likewise the 6-ethoxy analogue (**354**, R = OEt) (37%).[817]

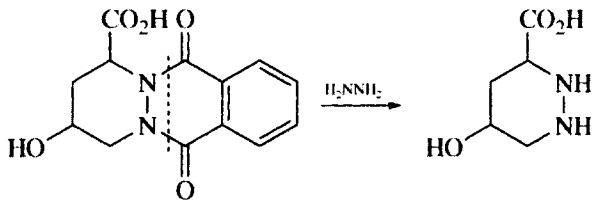
### From Pyridazino[1,2-*b*]phthalazines

1-Bromo-1,4-diphenyl-1,4,6,11-tetrahydropyridazino[1,2-*b*]phthalazine-6,11-dione (**355**) gave 3,6-diphenylpyridazine (**356**) [neat, 110°, 1 mmHg, 40 min: <35%; base: no details; aromatization by loss of HBr and additional (aerial?) oxidation].[345]

3-Hydroxy-6,11-dioxo-1,2,3,4,6,11-hexahydropyridazino[1,2-*b*]phthalazine-1-carboxylic acid (**357**) gave 5-hydroxyhexahydro-3-pyridazinecarboxylic acid (**358**) ($H_2NNH_2$, $H_2O$, 100°, 2 h: isolated as its *N*-(2,4-dinitrophenyl) derivative (no details).[32]




(**355**) (**356**)



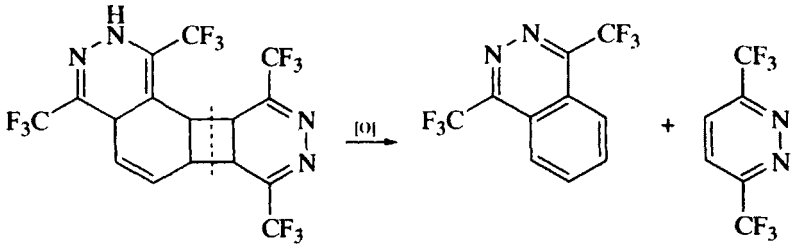

(**357**) (**358**)

## 2.6 PYRIDAZINES FROM HETEROTETRACYCLIC SYSTEMS

The only available synthetic routes in this category are illustrated in the following examples:

### From Pyridazino[4′,5′: 3,4]cyclobuta[1,2-*f*]phthalazines

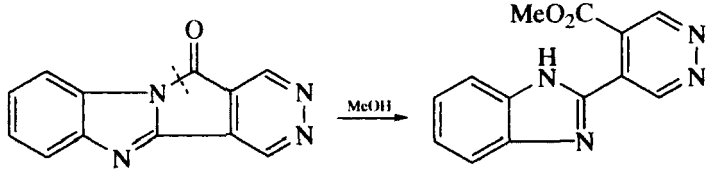
1,4,7,10-Tetrakistrifluoromethyl-2,4a,6a,6b,10a,10b-hexahydropyridazino[4′,5′:3,4]cyclobuta[1,2-*f*]phthalazine (**359**) suffered oxidative fission to a separable mixture of 3,6-bistrifluoromethylpyridazine (**361**) and 1,4-bistrifluoromethylphthalazine (**360**) ($Ag_2O$, $CH_2Cl_2$, reflux, 4 days: 61% and 64%, respectively).[1615]



(359) (360) (361)

**From Pyridazino[4′,5′:3,4]pyrrolo[1,2-*b*]benzimidazoles**

11*H*-Pyridazino[4′,5′:3,4]pyrrolo[1,2-*a*]benzimidazol-11-one (**362**) gave 2-(5-methoxycarbonylpyridazin-4-yl)benzimidazole (**363**) (MeOH, reflux, 30 min: 95%).[656]



(362) (363)

## 2.7 PYRIDAZINES FROM BRIDGED HETEROCYCLIC SYSTEMS

A few miscellaneous bridged heterocyclic systems have been used as substrates for the synthesis of pyridazines, but most such routes appear to be more of interest than practical importance. The following examples illustrate the present limited scope of these reactions and may suggest further uses for them:

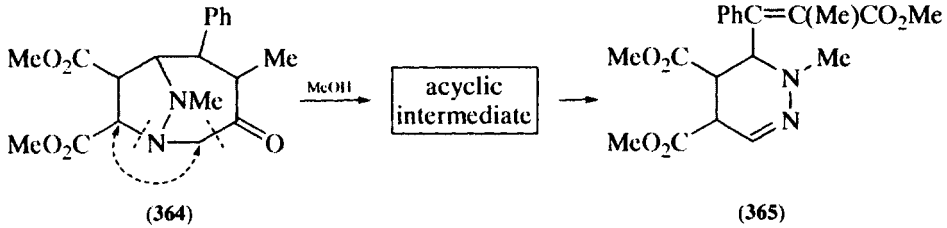
**From 1,9-Diazabicyclo[4.2.1]nonanes**

Dimethyl 4,9-dimethyl-3-oxo-5-phenyl-1,9-diazabicyclo[4.2.1]nona-4,7-diene-7,8-dicarboxylate (**364**) gave dimethyl 3-(2-methoxycarbonyl-1-phenylprop-1-enyl)-2-methyl-2,3,4,5-tetrahydro-4,5-pyridazinedicarboxylate (**365**), probably by fission of the 2,3 and 1,8 bonds of the substrate and recyclization (MeONa, MeOH, $N_2$, 25°, 2 h: ~20% after separation from another product);[754] the structure (**365**) was confirmed by X-ray analysis.[754]

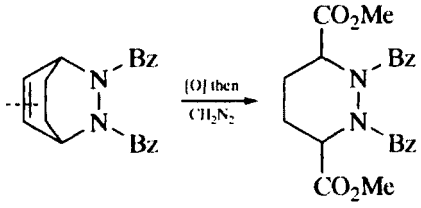
**From 2,3-Diazabicyclo[2.2.2]octanes**

The Diels–Alder adduct, 2,3-dibenzoyl-2,3-diazabicyclo[2.2.2]oct-5-ene (**366**), gave dimethyl 1,2-dibenzoylhexahydro-3,6-pyridazinedicarboxy-

late (**367**) by a one-pot oxidation and esterification ($NaIO_4$, $RuO_2$, AcOEt, 0°; then $CH_2N_2$: 95%).[1812]



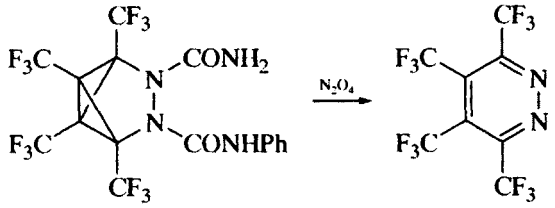

(**364**) (**365**)




(**366**) (**367**)

## From 3,4-Diazatricyclo[3.1.0.0$^{2,6}$]hexanes

4-Phenylcarbamoyl-1,2,5,6-tetrakistrifluoromethyl-3,4-diazatricyclo[3.1.0.0$^{2,6}$]hexane-3-carboxamide (**368**) gave 3,4,5,6-tetrakistrifluoromethylpyridazine (**369**) ($CH_2Cl_2$, $N_2O_4\downarrow$, ? conditions: 14%).[270]




(**368**) (**369**)

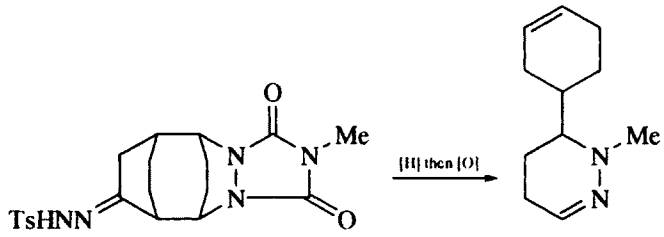
## From 5,10:6,9-Diethano[1,2,4]triazolo[1,2-*a*][1,2]diazocines

2-Methyl-7-*p*-toluenesulfonylhydrazono-5,6,7,8,9,10-hexahydro-1*H*-5,10:6,9-diethano[1,2,4]triazolo[1,2-*a*][1,2]diazocine-1,3(2*H*)-dione (**370**) gave 3-cyclohex-3′-enyl-2-methyl-2,3,4,5-tetrahydropyridazine (**371**) as the major product ($LiAlH_4$, THF, $N_2$, 10° → 55°, 12 h; then crude product, $MnO_2$, $CH_2Cl_2$, 0°, 4 h: 43%).[444]

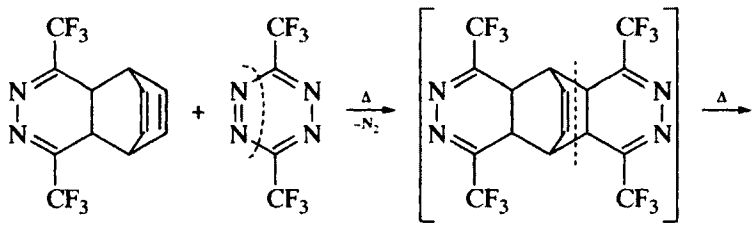
## From 5,8-Ethenophthalazines

1,4-Bistrifluoromethyl-5,8-dihydro-5,8-ethenophthalazine (**372**) with 2,6-bistrifluoromethyl-1,2,4,5-tetrazine (**373**) gave the unstable adduct **374**,

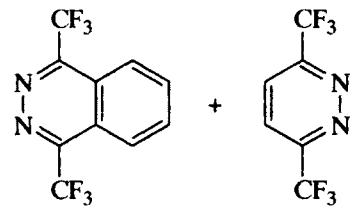
which suffered spontaneous reversion to 1,3-bistrifluoromethylphthalazine (**375**) and 3,6-bistrifluoromethylpyridazine (**376**) (PhMe, reflux, 15 h: 82% and 78%, respectively).[1503,1615]




(370) (371)



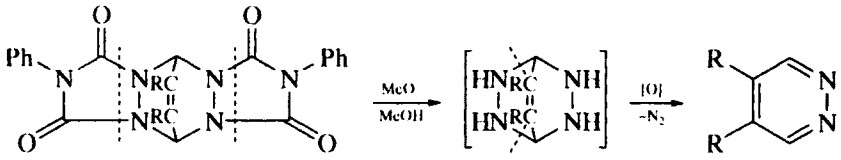

(372) (373) (374)




(375) (376)

## From 5,11-Etheno[1,2,4]triazolo[1′,2′:4,5][1,2,4,5]tetrazino-[1,2-*a*][1,2,4]triazoles

12,13-Di-*t*-butyl-2,8-diphenyl-1*H*,9*H*-5,11-etheno[1,2,4]triazolo-[1′,2′-4,5][1,2,4,5]tetrazino[1,2-*a*][1,2,4]triazole-1,3,7,9(2*H*,8*H*)-tetrone (**377**, R = Bu$^t$) gave 4,5-di-*t*-butylpyridazine (**379**, R = Bu$^t$) probably by loss of nitrogen from the intermediate (**378**) (KOH, MeOH, 0° → 25°, 2 h; then crude product, $Et_2O$, 2 h: 80%);[239,628] 4,5-dineopentyl-(**379**, R = $CH_2Bu^t$)[1518] and 4,5-diadamant-1′-ylpyridazine (**379**, R = adamantyl)[627,1391] were made likewise in comparable yields.



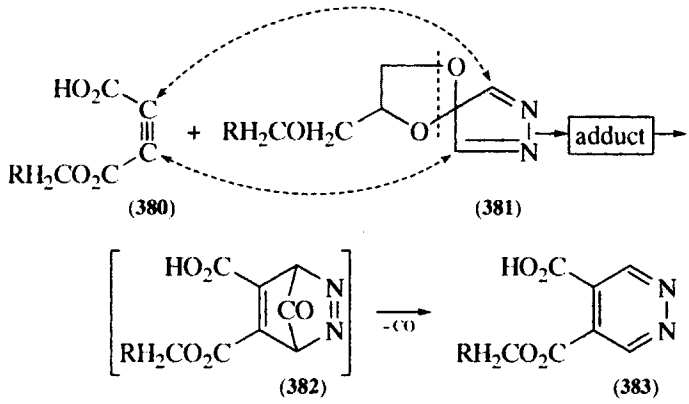
(377) (378) (379)

## 2.8 PYRIDAZINES FROM SPIRO HETEROCYCLIC SYSTEMS

This group of syntheses remains almost undeveloped, mainly because the substrates are seldom readily available. Examples follow:

### From 1,4-Dioxa-7,8-Diazaspiro[4.4]nonanes

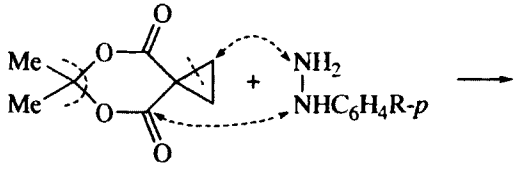
Polymer-bound 2-methoxymethyl-1,4-dioxa-7,8-diazaspiro[4.4]nona-6,8-diene (**381**, R = polymer) and polymer-bound 3-methoxycarbonylpropiolic acid (**380**, R = polymer) gave an adduct that underwent spontaneous degradation, probably via the bridged intermediate (**382**), to afford polymer-bound 5-methoxycarbonyl-4-pyridazinecarboxylic acid (**383**, R = polymer) (dioxane–$F_3CCO_2H$, 25°, 24 h) and then 4,5-pyridazinedicarboxylic acid (KOH, dioxane–$H_2O$: 42% overall).[1378]



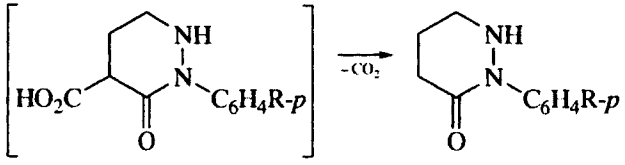
(380) (381) (382) (383)

### From 5,7-Dioxaspiro[2.5]octanes

6,6-Dimethyl-5,7-dioxaspiro[2.5]octane-4,8-dione (**384**) and phenylhydrazine (**385**, R = H) gave 2-phenyl-1,4,5,6-tetrahydro-3(2*H*)-pyridazinone (**387**, R = H) via the intermediate carboxylic acid (**386**) (MeCN, reflux, 5 h: 58%); the 2-*p*-tolyl (**387**, R = Me) (54%) 2-*p*-nitrophenyl (**387**, R = $NO_2$) (38%), and other such analogues were made similarly.[1599]



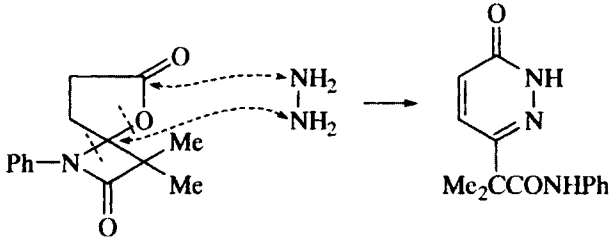
(384) (385)



(386) (387)

## From 5-Oxa-1-azaspiro[3.4]octanes

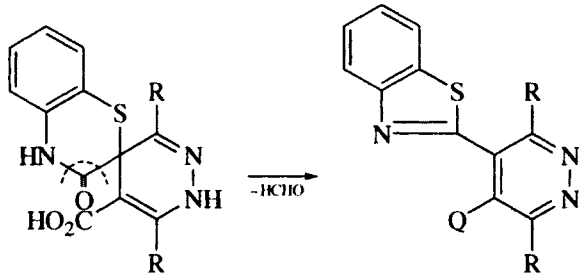
3,3-Dimethyl-1-phenyl-5-oxa-1-azaspiro[3.4]octane-2,6-dione (**388**) and hydrazine hydrate gave 6-[α-methyl-α-(phenylcarbamoyl)ethyl]-3(2*H*)-pyridazinone (**389**) (EtOH, exothermic, then 40°, 3 h: 60%).[442]



(388) (389)

## From Spiro[2H-1,4-benzothiazine-2,4′-(1′H)-pyridazine]s

3-Oxo-3,4-dihydrospiro[2*H*-1,4-benzothiazine-2,4′-(1′*H*)-pyridazine]-5′-carboxylic acid (**390**, R = H) gave a separable mixture of 5-benzothiazol-2′-yl-4-pyridazinecarboxylic acid (**391**, Q = $CO_2H$, R = H) and 2-pyridazin-4′-ylbenzothiazole (**391**, Q = R = H) (AcOH, reflux, 6 h:

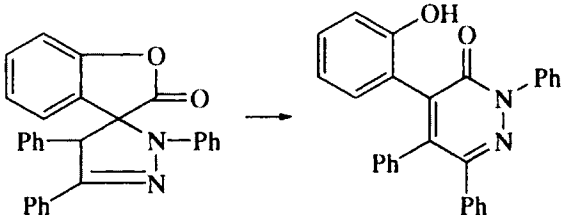


(390) (391)

~25% and ~60%, respectively; when heating was continued for 12 h, only the latter was isolated: 90%); in contrast, the 3′,6′-dimethylated substrate (**390**, R = Me) gave only 2-(3,6-dimethylpyridazin-4-yl)benzothiazole (**391**, Q = H, R = Me) even under more gentle conditions (AcOH, 50°, 2 h: 94%).[393]

### From Spiro[benzofuran-3(2H),3′-[3H]pyrazole]s

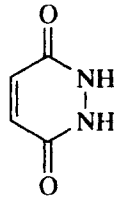
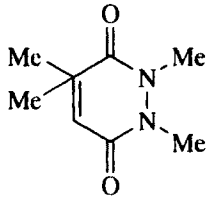
2′,4′,5′-Triphenyl-2′,4′-dihydrospiro[benzofuran-3(2H),3′-[3*H*]pyrazole]-2-one (**392**), easily made from 3-benzylidenebenzofuran-2(3*H*)-one, gave 4-*o*-hydroxyphenyl-2,5,6-triphenyl-3(2*H*)-pyridazinone (**393**) (neat $F_3CCO_2H$, reflux, 3 h: >95%); several analogues similarly.[2105]

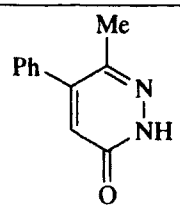
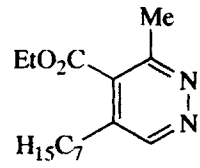
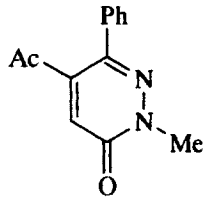
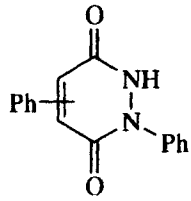
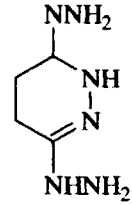
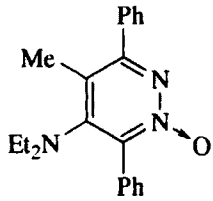
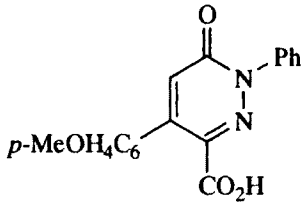


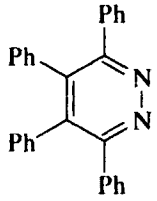
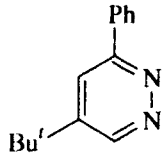
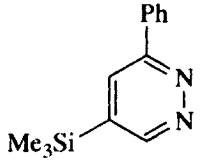
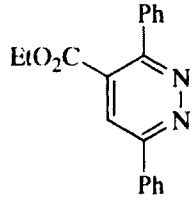
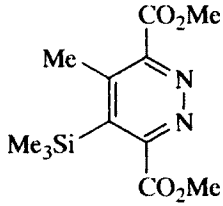
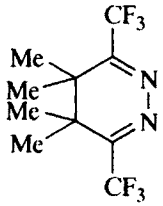
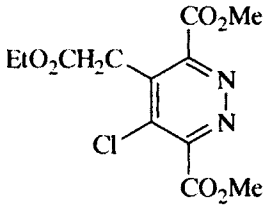
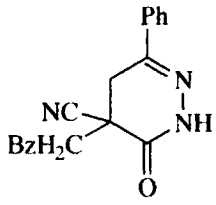
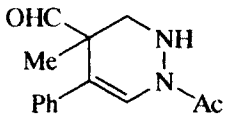
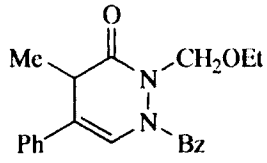

(392) (393)

## 2.9 APPENDIX: GLANCE INDEX TO TYPICAL PYRIDAZINES READILY AVAILABLE FROM OTHER HETEROCYCLIC SYSTEMS

This glance index has been added to assist in the choice of a primary synthesis that may afford a required type of pyridazine derivative from another heterocyclic system. Procedures that employ heterocyclic substrates difficult of access, those that afford very poor yields, and those that do not appear to be of general applicability in their present state of development have been omitted; nevertheless, such syntheses are often of great interest and may prove invaluable in the right context.

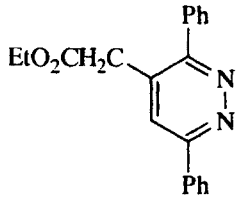
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|---|---|---|
| 2.1.2 |  |  |
| | 95% | 64% |

| Section | Typical Products |
|---|---|
| 2.1.2 |  89%  82% |
| 2.1.4 |  89% |
| 2.1.9 |  82%  85% |
| 2.2.3 |  94% |
| 2.2.4 |  82% |

| Section | Typical Products |
|---|---|
| 2.2.5 |  58%  50% |
| 2.2.7 |  95%  63%  69%  91% |
| 2.3.1 |  82%  87% |
| 2.4.4 |  84%  45% |

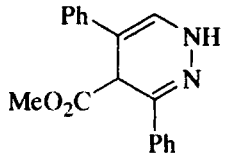
| Section | Typical Products |
|---|---|

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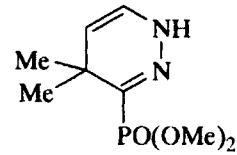


63%

2.4.8

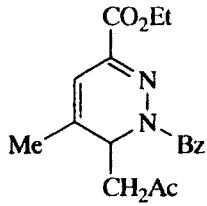


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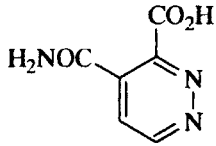
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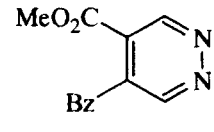


58%

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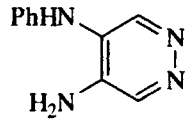


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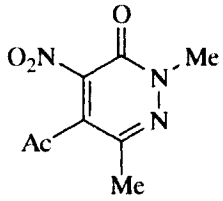
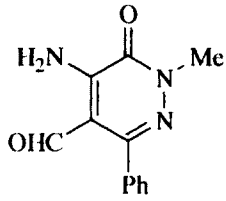
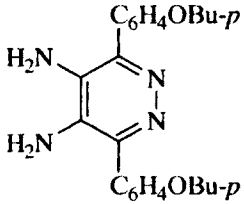
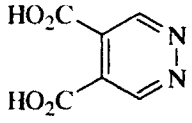
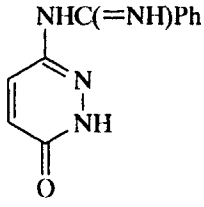
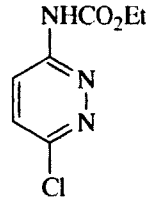
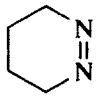
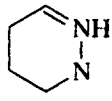


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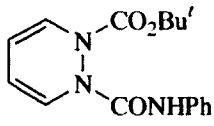


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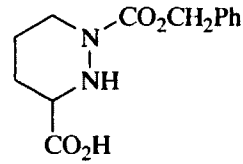
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| 2.4.22 |  53% |
| 2.4.25 |  66% |
| 2.4.34 |  72%  67% |
| 2.4.41 |  95%  ?% |

| Section | Typical Products |
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2.4.46

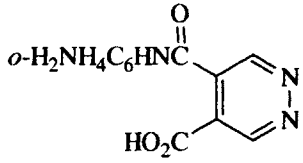


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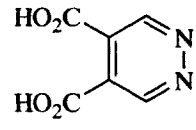


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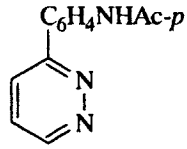
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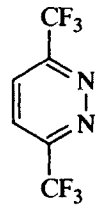


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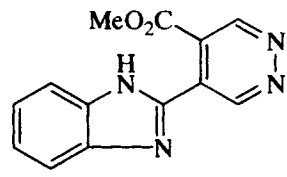


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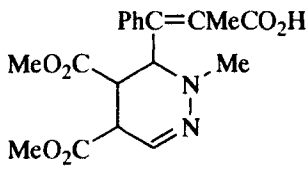


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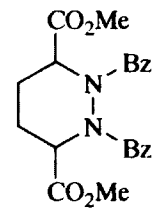


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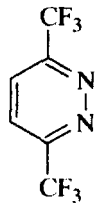
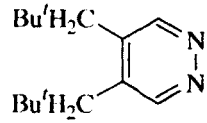
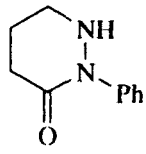
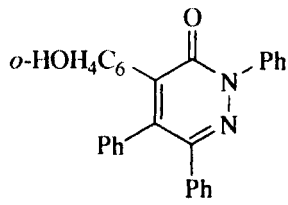
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| 2.8 |  58% |  95% |

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D. J. Brown
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CHAPTER 3

# Pyridazine, Alkylpyridazines, and Arylpyridazines

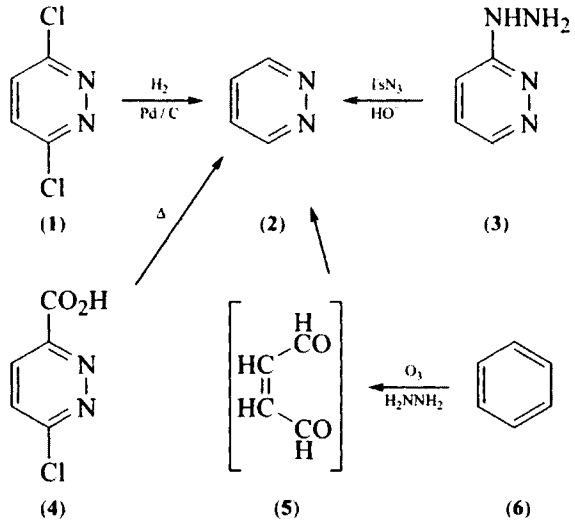
This chapter covers the preparations, physical properties, and reaction of pyridazine and its *C*-alkyl, *N*-alkyl, or *C*-aryl derivatives as well as their di-, tetra-, or hexahydro derivatives. In addition, it includes methods for introducing *C*-alkyl or *C*-aryl groups (substituted or otherwise) into pyridazines already bearing functional or other substituents and the reactions specific to the alkyl or aryl group(s) in such products. For simplicity, the term *alkylpyridazine* in this chapter is intended to include alkyl-, alkenyl-, alkynyl- cycloalkyl-, and aralkylpyridazines; likewise, the term *arylpyridazine* includes aryl- and heteroarylpyridazines.

## 3.1 PYRIDAZINE (*H* 1)

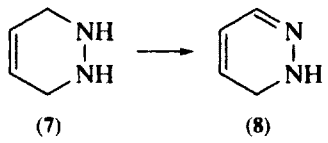
First prepared in the late nineteenth century,[1924] pyridazine is now commercially available in quantity. In recent years it has been investigated extensively, especially in respect of its reactions.

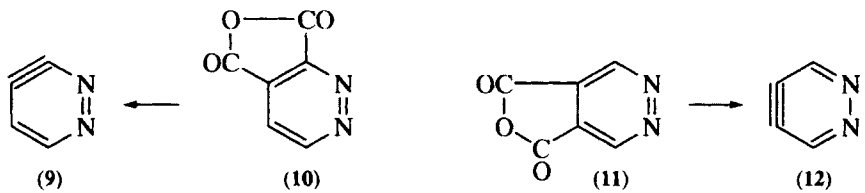
### 3.1.1 Preparation of Pyridazine

Of the recently reported routes to pyridazine, two are of practical significance. Hydrogenolysis of 3,6-dichloropyridazine (**1**) over palladium in ethanolic ammonium hydroxide at 25° and subsequent purification of the product through a silica-gel column gave pyridazine (**2**) in 85% yield[1098] (cf. 27–61% by earlier analogous procedures[1925]). Treatment of 3-hydrazinopyridazine (**3**) with *p*-toluenesulfonyl azide in a mixture of aqueous alkali and benzene containing a trace of tetraethylammonium bromide under reflux for 8 h afforded pyridazine, conveniently isolated as its hydrochloride in 79% yield.[262] In addition, pyridazine (**2**) has been made by regular primary syntheses (see Chapters 1 and 2), the pyrolysis of 3-pyridazinecarboxylic acid (**4**) (no details),[25] and the action of hydrazine and ozone on benzene (**6**) in the vapor state at 25°, probably via maleic aldehyde (**5**) but in only ~1% yield under the conditions used.[1571]




Hexahydropyridazine (piperidazine: **7**) has been partially dehydrogenated to 1,4,5,6-tetrahydropyridazine (**8**) by oxygenation in heptane over a platinum catalyst at 25° for 30 min (65%) or treatment with mercuric oxide in heptane at 25° for 45 min (38%).[5] Although unisolated, 3,4- (**9**) and 4,5-didehydropyridazine (**12**) have been experimentally implicated as intermediates in both the electron impact and pyrolytic fragmentation of 3,4- (**10**) and 4,5-pyridazine dicarboxylic anhydride (**11**), respectively.[201]



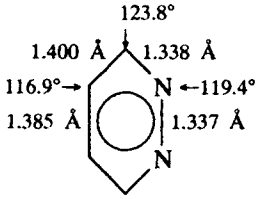




### 3.1.2 Physical Aspects of Pyridazine (*H* 2)

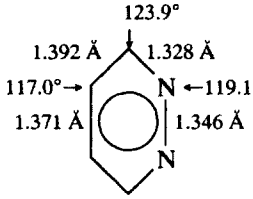
Most of the physical aspects of pyridazine and its usual salts or complexes were measured prior to 1970 and have been summarized (*H* 5) adequately.[2011]

Recently the geometric *structure of pyridazine* in the gas phase has been redetermined by electron-diffraction data[177] and subsequently by a combination of electron diffraction, microwave data, and dipolar couplings in the liquid-

crystal $^1$H nmr spectrum.[1926] The latter set of data (**13**) compared remarkably well with that (**14**) derived from state-of-the-art X-ray crystallography on pyridazine at $-173°$.[1532] The geometry of pyridazine hydrochloride has been measured, also by X-ray crystallography at $-170°$.[180] The conformational flexibility of 1,4-dihydropyridazine has been calculated.[1973]



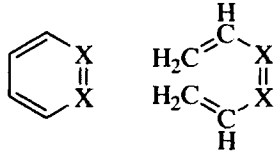

(13)




(14)

General studies on the *spectra of pyridazine* (and its derivatives) have been confined in the main to the effects of substitution on $^{13}$C nmr data and the correlation of such results with $^1$H nmr data and with calculated charge densities.[229,450,829,829,1638] An infrared study,[2078] a mass-spectral study of pyridazine, [2077] a study of the $^1(n,\pi*)$ excited states of pyridazine in water,[1860] and coupled-cluster calculations of its excitation energies[1967] have been reported.

Studies on the elusive concept of *aromaticity*, based on many diverse criteria, have continued to include pyridazine. For example, it has been proposed that a realistic value for the aromaticity of pyridazine (compared with that for the standard, benzene) should be attainable by comparing the ratio, heat of formation for pyridazine (**15**, X = N)/heat of formation for 3,4-diaza-1,3,5-hexatriene (**16**, X = CN, with that of the ratio, heat of formation for benzene (**15**, X = CH)/heat of formation for 1,3,5-hexatriene (**16**, X = CH), all experimentally attainable data.[1502] However, judging from a subsequent extensive review[1433] (based on 12 criteria common to each case), the measurement of aromaticity in heterocycles can be described presently as only semiquantitative. (See also Ref. 2013.)




(15) (16)

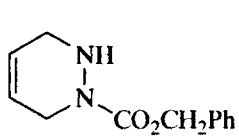
The *polarographic reduction* of pyridazine in acetonitrile proved to be far from simple and has been studied in detail;[579] the polarographic picture in aqueous buffer was distinctly different.[1189]

Calculations on 3,4- (**9**) and 4,5-*didehydropyridazine* (**12**) by three groups agree that the latter lies lower in energy than its 3,4 isomer.[1191,1927,1928]

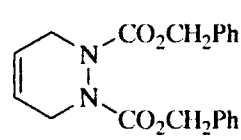
### 3.1.3 Reactions of Pyridazine (*H* 16)

Pyridazine and its hydro derivatives undergo a surprising number of reactions, as illustrated by recent examples in the following paragraphs:

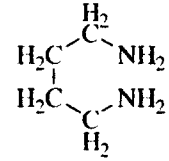
REDUCTION The polarography of pyridazine has been mentioned in Section 3.1.2. A partial reduction of pyridazine to the tetrahydro stage has been achieved by treatment with sodium cyanoborohydride and benzyl chloroformate in methanol at 25° for 16 h: the reduced product was trapped as a separable mixture of benzyl 1,2,3,6-tetrahydro-1-pyridazinecarboxylate (**17**) (12% yield) and dibenzyl 1,2,3,6-tetrahydro-1,2-pyridazinedicarboxylate (**18**) (28% yield).[1543] In contrast, reduction of pyridazine in dilute alkali with NiAl alloy over 2.5 h gave 1,4-diaminobutane (**19**), isolated as its hydrochloride in 61% yield;[348] pyridazine underwent a nonreductive ring fission by the carbene, $(OC)_5Cr{=}C(OMe)Me$, to give 1,4-bis(α-methoxyethylideneamino)butane in 35% initial yield, reduced to 5% during isolation.[626]




(17)

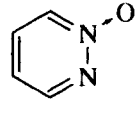



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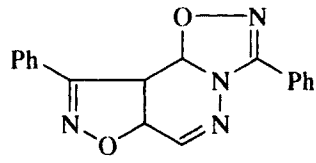



(19)

OXIDATION. Pyridazine underwent only mono-*N*-oxidation on treatment with 30% hydrogen peroxide at 100° for 8 h in the presence of sodium tungstate to give pyridazine 1-oxide (**20**) in 50% yield; at 55°, the yield was only 8.5%.[783] Pyridazine with perfluoro-2-butyl-3-propyloxaziridine in chloroform at −60° gave a separable mixture of pyridazine 1-oxide (**20**) (70%) and so-called *N*-(perfluorobutanoyl)pyridazinium 1-aminide (20%).[1944] Benzonitrile oxide (PhCN→O) reacted with pyridazine in refluxing benzene for 3 h to give mainly the tricyclic adduct (**21**) but also a little 3(2*H*)-pyridazinone (**22**).[1809] When

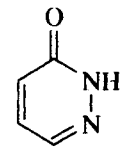



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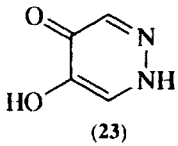



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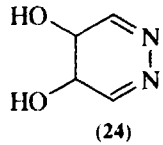



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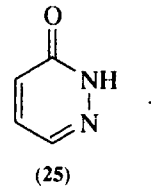

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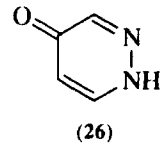

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(26)

pyridazine was fed to rats, metabolites in the urine included 5-hydroxy-4(1*H*)-pyridazinone (**23**), 4,5-dihydro-4,5-pyridazinediol (**24**), and "two isomeric monohydroxypyridazines," presumably 3(2*H*)- (**25**) and 4(1*H*)-pyridazinone (**26**).[1040]

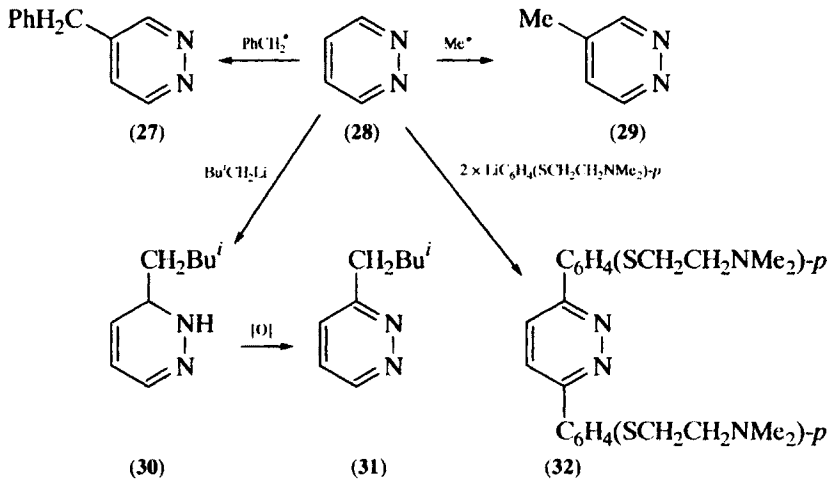
ALKYLATION Processes for the *C*-alkylation and *C*-arylation of pyridazine are illustrated in the examples that follow but for *N*-alkylation by the formation of *N*-alkylpyridazinium salts (or ylides), see Sections 3.3.1 and 3.3.2. Grignard additions have been studied.[188]

### Using Alkyl Radicals

Pyridazine (**28**) gave 4-benzylpyridazine (**27**) [$PhCH_2CO_2H$, $AgNO_3$, 1M $H_2SO_4$, 70°; $(NH_4)_2S_2O_8\downarrow$, 20 min; 90°, 30 min: 25%].[72, 1098]

Pyridazine (**28**) gave mainly 4-methylpyridazine (**29**) but also six other methylated pyridazines in small amounts (dilute $H_2SO_4$, 15°; $Bu^tO_2H\downarrow$, $FeSO_4\downarrow$, 60 min: 60%).[211]

Also other examples.[261]




### Using Alkyllithium Reagents

Pyridazine (**28**) gave crude 3-isopentyl-2,3-dihydropyridazine (**30**) ($Bu^iCH_2Li$, $Et_2O$, −15°, 3 h, then 25°, 12 h) and then 3-isopentylpyridazine (**31**) ($KMnO_4$, AcMe: 93% overall).[1660]

Pyridazine gave 3-perfluorohexyl-2,3-dihydropyridazine (pyridazine, $C_6F_{13}I$, $BF_3 \cdot Et_2O$, $Et_2O$, −78°; then MeI·LiBr, 178°, 1 h: 71%) and then 3-perfluorohexylpyridazine (aerial oxidation: >95%).[1403]

Pyridazine (**28**) with *p*-(β-dimethylaminoethylthio)phenyllithium gave 3-[*p*-(β-dimethylaminoethylthio)phenyl]pyridazine (40%), which reacted with

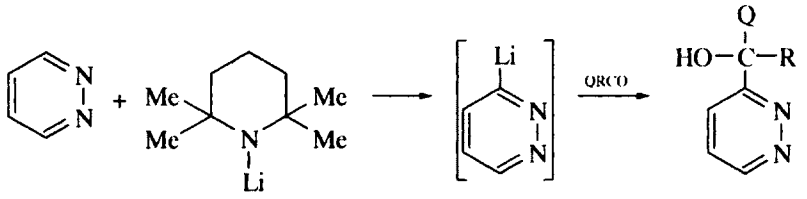
the same reagent again to afford 3,6-bis[*p*-(β-dimethylaminoethylthio)-phenyl]pyridazine (**32**) (36%).[1740]

Also examples of heteroarylation.[452, 1235, 1317]

### Using 3-Lithiopyridazine as Substrate

Pyridazine (**33**) with LTMP (**34**) gave 3-lithiopyridazine (**35**) (THF, −75°, 6 min: unisolated) and then 3-α-hydroxyethylpyridazine (**36**, Q = Me, R = H) (MeCHO, THF, −75°, 1 h: 26%).[1387]

Somewhat similarly, pyridazine (**33**) gave 3-α-hydroxybenzylpyridazine (**36**, Q = Ph, R = H) (LTMP, PhCHO, THF, 75°, 90 min: 31%) or 3-(α-hydroxy-α-phenylbenzyl)pyridazine (**36**, Q = R = Ph) (LTMP, $Ph_2CO$, THF, −75°, 2 h: 47%).[1387]




(**33**) (**34**) (**35**) (**36**)

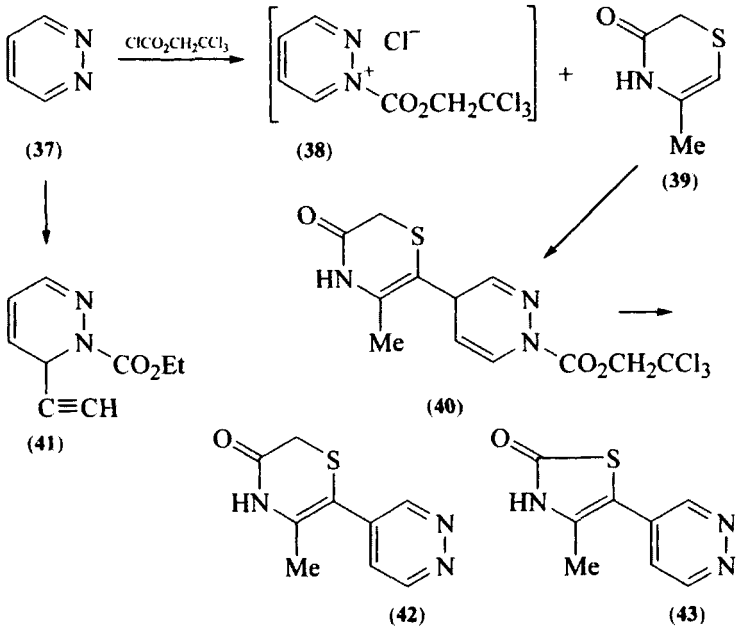
### Using an N-Acylpyridazinium Substrate

*Note*: This indirect route has been used mainly for *C*-heteroarylation of pyridazine and its simple derivatives.

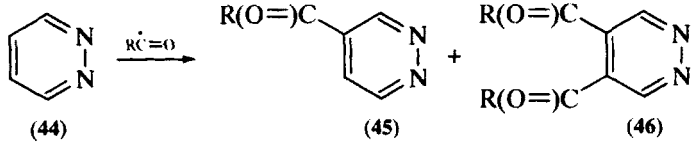
Pyridazine (**37**) and β,β,β-trichloroethyl chloroformate gave a solution of the quaternary intermediate (**38**) (MeCN, pyridine, 0°, 20 min), which underwent arylation by 5-methyl-2*H*-1,4-thiazin-3(4*H*)-one (**39**) to afford 5-methyl-6-(2-β,β,β-trichloroethoxycarbonyl-1,4-dihydropyridazin-4-yl)-2*H*-1,4-thiazin-3(4*H*)-one (**40**) (MeCN, pyridine, 0°, 20 min; then 25°, 5 h: 63%) and then (by oxidative deacylation) 5-methyl-6-pyridazin-4′-yl-2*H*-1,4-thiazin-3(4*H*)-one (**42**) (S, $Me_2NCHO$, reflux, 90 min: 23%);[883] 4-methyl-5-pyridazin-4′-yl-2(3*H*)-thiazolone (**43**) was made similarly in 19% yield.[889]

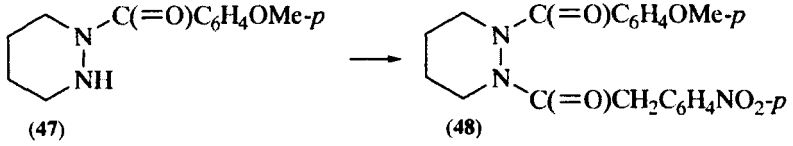
Somewhat similarly, pyridazine (**37**), bis(tributylstannyl)acetylene ($Bu_3SnC{\equiv}CSnBu_3$), and ethyl chloroformate gave ethyl 3-ethynyl-1,6-dihydro-1-pyridazinecarboxylate (**41**) ($CH_2Cl_2$, 25°, >30 h; then $F_3CCO_2H$, 30 min: 71%);[1430,1582] likewise, allyltributyltin gave a separable mixture of ethyl 4-allyl-1,4-dihydro- and ethyl 6-allyl-1,6-dihydro-1-pyridazinecarboxylate (14% and 63%, respectively).[1627]

ACYLATION Pyridazine undergoes *C*-acylation on treatment with acyl radicals to afford 4-acyl- or more often 4,5-diacylpyridazines; hexahydropyridazine undergoes *N*-acylation or even *N,N′*-diacylations.[227] These processes are illustrated in the following examples:



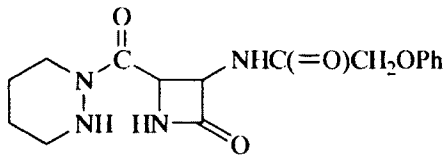
Pyridazine (**44**) gave a separable mixture of 4-benzoyl- (**45**, R = Ph) and 4,5-dibenzoylpyridazine (**46**, R = Ph) (PHCHO, 1M $H_2SO_4$, AcOH; then $Bu^tO_2H\downarrow$, $FeSO_4/H_2O\downarrow$, 10°, 1 h: 3% and 31%, respectively);[72,79,81] *p*-anisaldehyde gave only 4,5-di-*p*-anisoylpyridazine (**46**, R = $C_6H_4OMe$-*p*) (56%), but aliphatic aldehydes gave poor yields, for instance, 4,5-diisobutyrylpyridazine (**46**, R = $Pr^i$) (9%).[79,81] (See also Sections 8.2.1 and 8.8)





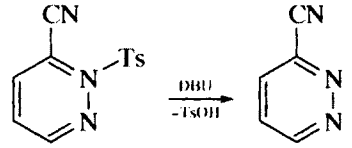
Hexahydropyridazine gave 1-*p*-anisoylhexahydropyridazine (**47**) ($MeOC_6H_4CO_2H$-*p*, $C_6H_{11}N{=}C{=}NC_6H_{11}$, 4-$Me_2N$-pyridine, PhH, 40°, 16 h: 37%) and then 1-*p*-anisoyl-2-(*p*-nitrophenylacetyl)hexahydropyridazine (**48**) (*p*-$O_2NC_6H_4CH_2COCl$, PhMe, reflux, 12 h: ?%).[23]

Hexahydropyridazine and 4-oxo-3-phenoxyacetamido-2-azetidinecarboxylic acid gave 1-(4-oxo-3-phenoxyacetamido-2-azetidinecarbonyl)hexahydropyridazine (**49**) [$C_6H_{11}N{=}C{=}NC_6H_{11}$ (?), THF, 25°, 18 h: 46%].[258]



(49)

REISSERT REACTIONS Pyridazine reacted readily with trimethylsilyl cyanide in the presence of *p*-toluenesulfonyl chloride and aluminum chloride to give 2-tosyl-2,3-dihydro-3-pyridazinecarbonitrile (**50**) (84%), which underwent oxidation by loss of *p*-toluenesulfonic acid on stirring with 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU) in THF for 1 h to afford 3-pyridazinecarbonitrile (**51**) in 92% yield;[220] 4- and 6-methyl-3-pyridazinecarbonitrile were made similarly in ~45 and ~80% yields, respectively.[220]



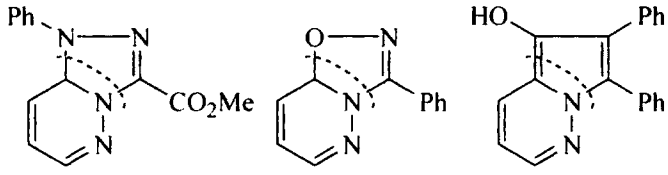
(50) (51)

CYCLOADDITIONS Several types of cycloaddition (in both the strict and broad senses of the word) have involved pyridazine or hexahydropyridazine, as illustrated in the following examples:

Pyridazine and methyl 2-chloro-2-phenylhydrazonoacetate gave methyl 1-phenyl-1,8a-dihydro-1,2,4-triazolo[4,3-*b*]pyridazine-3-carboxylate (**52**) ($Et_3N$, $CHCl_3$, 25°, 24 h: 65%).[937]

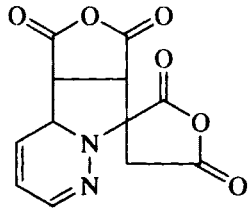
Pyridazine and benzonitrile oxide gave 3-phenyl-8a *H*-1,2,4-oxadiazolo[4,5-*b*]pyridazine (**53**) [PhCN→O (made in situ), $Et_2O$, $Et_3N$, 0°→25°, 2 days: 60%].[1878]

Pyridazine and diphenylcyclopropenone gave 6,7-diphenylpyrrolo[1,2-*b*]pyridazin-5-ol (**54**) (MeOH, reflux, A, 1 h: 70%).[1379]



(52) (53) (54)

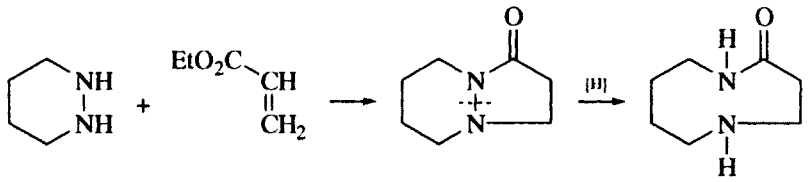
Pyridazine and maleic anhydride gave, not the simple diadduct as reported previously,[1929] but the isomeric spiro compound (**55**) ($CHCl_3$, 25°, 2 days; then 40°, 2 days: 85%).[953]



(**55**)

Hexahydropyridazine (**56**) and ethyl acrylate (**57**) gave 2,3,5,6,7,8-hexahydro-1*H*-pyrazolo[1,2-*a*]pyridazin-1-one (**58**) (70%) and then perhydro-1,5-diazonin-2-one (**59**) (liquid $NH_3$, Na: 87%).[630]

Also other examples.[225,284,431,1946]



(**56**) (**57**) (**58**) (**59**)

## Metal Complex Formation

Pyridazine has been involved, usually as a coligand, in the formation of complexes with the following metals: Ag,[900] Cd,[1091] Co,[1091,1202] Cr,[626] Cu,[1091,1546,1658] Fe,[1091,1133,1202] Ge,[1249] Mn[1091,1202] Mo[478,1093] Ni,[1091,1202] Pd,[900] Rh[454] Ru[490] Si,[1249] Sn,[1349] Ti[1349] V,[2115] W,[478] and Zn.[1091,1202]

## Other Reactions

As well as undergoing the alkylations (discussed earlier in this Section), 3-lithiopyridazine (**35**) (prepared in situ in THF) afforded 3-phenylthiopyridazine (PhSSPh, THF, −75°, 2 h: 7%), 3-iodopyridazine (I, THF, −75°, 40 min: 16%), 3-deuteropyridazine (DCl–EtOD, −75°, 10 min: 32%), or 4(*not* 3)-*t*-butyldimethylsilylpyridazine ($Bu^tMe_2SiCl$, THF, −75°, 90 min: 9%).[1387] The *C* or *N* amination of pyridazine is discussed in Section 7.2.1.

Pyridazine acted as an effective catalyst in the successive reductive dechlorinations of tetrachlorodimethyldisilane ($Cl_2MeSiSiMeCl_2$) by tributylstannane in toluene during 2 days.[1647]

Pyridazine underwent profound decomposition on pyrolysis.[2082]

## 3.2 *C*-ALKYL- AND *C*-ARYLPYRIDAZINES

In earlier days, alkyl and aryl groups attached to pyridazine were clearly considered of little importance. However, it is evident now that such groups do undergo a variety of reactions, have marked steric and electronic effects on the reactivity of functional groups attached to the same pyridazine nucleus, and profoundly affect the bioactivity of such pyridazines. The formation and reactions of *N*-alkylpyridazinium salts and ylides are treated in Section 3.3.

### 3.2.1 Preparation of *C*-Alkyl- and *C*-Arylpyridazines

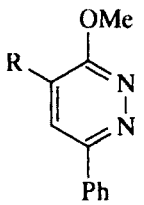
The following treatment is not confined entirely to methods for the preparation of simple alkyl- or arylpyridazines. It does include typical examples in which the resulting molecule may bear one or more functional passenger groups that have remained unaffected during the procedure(s) involved. The many *primary syntheses* of alkyl- and arylpyridazines have been covered in Chapters 1 and 2.

#### *3.2.1.1 By Direct Alkylation*

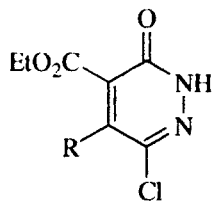
The alkylation of pyridazine has been discussed in Section 3.1.3. Direct alkylation or arylation of other pyridazines are illustrated in the following examples:

**Classical *C*-Alkylations**

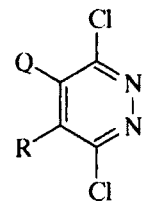
3-Methoxy-6-phenylpyridazine (**60**, R = H) gave its unisolated 4-lithio derivative (**60**, R = Li) (BuLi, 2,2,6,6-$Me_4$-piperidine, THF, $-30° \rightarrow 0°$, 30 min; then substrate ↓, $-70°$, 90 min) and then 3-methoxy-4-methyl-6-phenylpyridazine (**60**, R = Me) (MeI, $-70°$, 2 h: 81%);[1569] also analogous examples.[358, 1569]



(**60**)



(**61**)



(**62**)

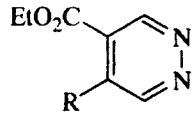
Ethyl 6-chloro-3-oxo-2,3-dihydro-4-pyridazinecarboxylate (**61**, R = H) gave its 5-methyl derivative (**61**, R = Me) ($CH_3NO_2$, $Et_3N$ or $K_2CO_3$, $Me_2SO$,

25°, 3 days: 96% or 63%, respectively); also the 5-ethyl homologue (**61**, R = Et) ($EtNO_2$, similarly: 36% or 38%, respectively).[821,cf.30]

3,6-Dichloropyridazine (**62**, Q = R = H) gave 3,6-dichloro-4-tosylmethylpyridazine (**62**, $Q = CH_2Ts$, R = H) ($ClCH_2Ts$, $Bu^tOK$, THF, −75°, 1 h: 98%).[1506,1625]

### Homolytic or Irradiation-Assisted *C*-Alkylations

3,6-Dichloropyridazine (**62**, Q = R = H) gave 3,6-dichloro-4-methylpyridazine (**62**, Q = Me, R = H) [AcOH, $AgNO_3$, dilute $H_2SO_4$, $(NH_4)_2S_2O_8$, 70°: 87%]; similar treatment using $Pr^iCO_2H$ for AcOH gave a separable mixture of 3,6-dichloro-4-isopropyl- (**62**, $Q = Pr^i$, R = H) and 3,6-dichloro-4,5-diisopropylpyridazine (**62**, $Q = R = Pr^i$) (84% and 12%, respectively).[1223]



(**63**)

Ethyl 4-pyridazinecarboxylate (**63**, R = H) gave ethyl 5-phenoxymethyl-4-pyridazinecarboxylate (**63**, $R = CH_2OPh$) [$PhOCH_2CO_2H$, $AgNO_3$, dilute $H_2SO_4$, PhMe, $(NH_4)_2S_2O_8$, 90°, vigorous stirring, 1 h: 72%; use of an homogeneous mixture of deletion of PhMe led to a poor yield]; the 5-phenylthiomethyl analogue was made similarly (63%).[1635]

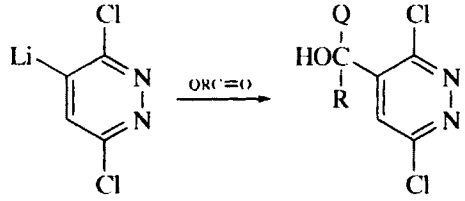
3,6-Dichloropyridazine (**62**, Q = R = H) gave a separable mixture of 3,6-dichloro-4-methylpyridazine (**62**, Q = Me, R = H), 3,6-dichloro-4,5-dimethylpyridazine (**62**, Q = R = Me), and three very minor products (MeOH, HCl gas, *hν*, 6 h: ~20%, ~10%, and <3% for each of the others).[755,762]

Also other examples.[24,758,761,762,1676]

### *C*-Alkylations with Aldehydes or Ketones

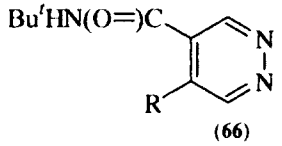
*Note:* The course of these alkylations is determined by the type of substrate and the ratio of reactants. Pyridazines (after lithiation) afford *C*-α-hydroxyalkylpyridazines; 4,5-dihydropyridazies give 4- or 5-monoalkylpyridazines by loss of water or 4,5-dialkylidenedihydropyridazines by loss of 2 × water. For Mannich *C*-alkylations, see the next subsection.

3,6-Dichloro-4-lithiopyridazine (**64**) (prepared in situ) gave 3,6-dichloro-4-α-hydroxyethylpyridazine (**65**, Q = me, R = H) (MeCHO, THF, −70°, 90 min: 65%) or 3,6-dichloro-4-(α-hydroxydiphenylmethyl)pyridazine (**65**, Q = R = Ph) (BzPh, THF, −70°, 90 min: 45%).[1669]



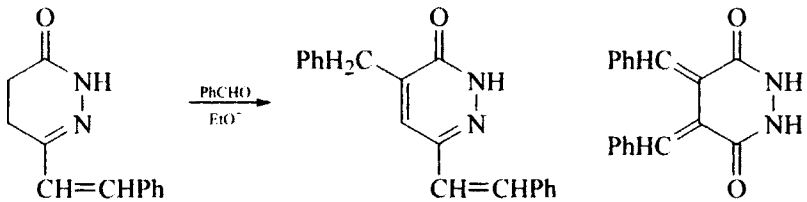
(64) (65)

*N*-*t*-Butyl-4-pyridazinecarboxamide (**66**, R = H) gave its 5-lithio derivative (**66**, R = Li) (BuLi, THF, −30°; then 2,2,6,6-$Me_4$-piperidine, 0°, 38 min; then substrate↓, −70°, 2 h) and then *N*-*t*-butyl-5-α-hydroxybenzyl-4-pyridazinecarboxamide [**66**, R = CH(OH)Ph](PhCHO, 170°, 30 min: 79%).[1609]



(66)

6-Styryl-4,5-dihydro-3(2*H*)-pyridazinone (**67**) gave 4-benzyl-6-styryl-3(2*H*)-pyridazinone (**68**) (PhCHO, EtOK, EtOH, reflux, 3 h: 48%).[1282]



(67) (68) (69)

6-*p*-Methoxyphenyl-4,5-dihydro-3(2*H*)-pyridazinone gave 4-*p*-methoxybenzyl-6-*p*-methoxyphenyl-3(2*H*)-pyridazinone ($MeOC_6H_4CHI$-*p*, EtOK, EtOH, reflux, 2 h: 73%); also analogues.[1253]

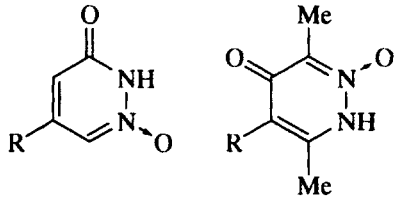
4,5-Dihydro-3,6(1*H*,2*H*)-pyridazinedione gave 4,5-dibenzylidene-4,5-dihydro-3,6(1*H*,2*H*)-pyridazinedione (**69**) (PhCHO, $Ac_2O$, AcONa, AcOH, reflux, 3 h: 69%).[372,374]

Also other examples.[358,363,370,868,922,992,1019,1039,1251,1263,1276,1410,1421,1553,1569,1610,1721,1867,1868,1880,2035]

## *C*-Alkylation by the Mannich Reaction

3(2*H*)-Pyridazinone 1-oxide (**70**, R = H) gave 6-dimethylaminomethyl-3(2*H*)-pyridazinone 1-oxide (**70**, R = $Me_2NCH_2$)($CH_2O/H_2O$, $Me_2NH$, EtOH, 0° → 25°, 12 h: 75%, as hydrochloride); also several analogues.[769]

3,6-Dimethyl-4(1*H*)-pyridazinone 2-oxide (**71**, R = H) gave 3,6-dimethyl-5-piperidinomethyl-4(1*H*)-pyridazinone 2-oxide [**71**, $R = CH_2N(CH_2)_5$] [$CH_2O/H_2O$, $HN(CH_2)_5$, EtOH, 25°, 2 days: 71%, as hydrochloride].[791]



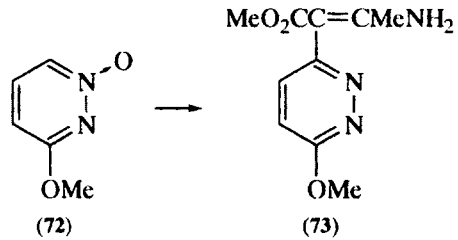
(70) (71)

## Dehydrative *C*-Alkylation of Pyridazine N-Oxides

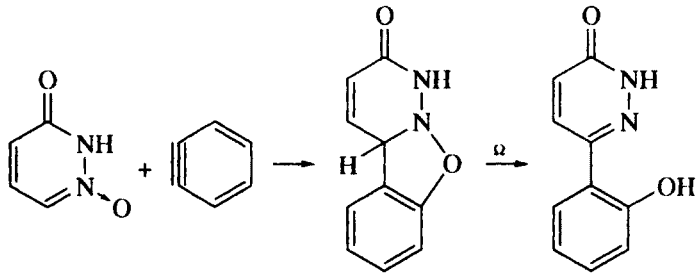
3-Methoxypyridazine 1-oxide (**72**) gave 3-(2-amino-1-methoxycarbonyl-prop-1-enyl)-6-methoxypyridazine (**73**) [$MeC(NH_2)=CHCO_2Me$, $BzCl,CHCl_3$, 0° → 25°, 24 h: 48%].[962]

3-Ethoxypyridazine 1-oxide gave 3-ethoxy-6-pyridin-2′-ylpyridazine and 2,2′-bipyridine (pyridine, Pt + Pd/C, ~125°, 4 h: <5% each).[536]

In contrast, 3(2*H*)-pyridazinone 1-oxide (**74**) with benzyne (**75**) gave 6-*o*-hydroxyphenyl-3(2*H*)-pyridazinone (**77**) (92%), probably via the tricyclic intermediate (**76**).[817]



(72) (73)

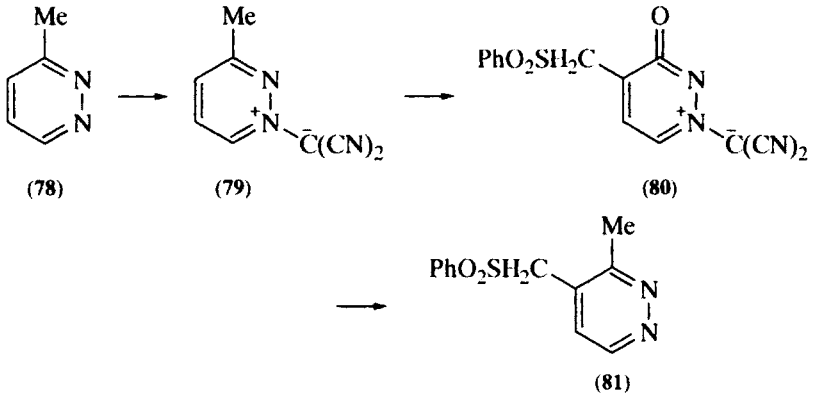


(74) (75) (76) (77)

## Alkylation of Zwitterionic Pyridazines

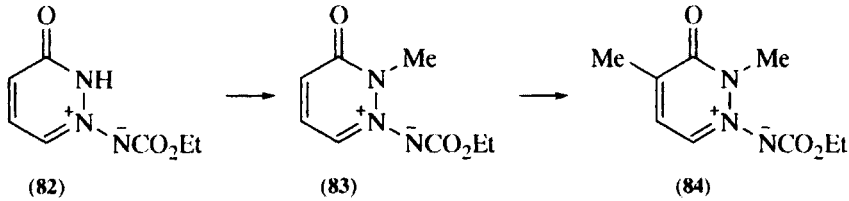
3-Methylpyridazine (**78**) gave successively 3-methylpyridazinium-1-dicyanomethylide (**79**) (tetracyanoethylene oxide, THF, 0°, ~3 h: 78%),

3-methyl-4-phenylsulfonylmethylpyridazinium-1-dicyanomethylide (**80**) ($ClCH_2SO_2Ph$, Bu'OK, THF, 0°, 15 min; then substrate↓, $Me_2NCHO$, 0°, 10 min: 59%), and 3-methyl-4-phenylsulfonylmethylpyridazine (**81**) [$(NH_4)_2S_2O_8$, MeOH, reflux, 1 h: 88%]; also analogues.[1816]



(78) (79) (80) (81)

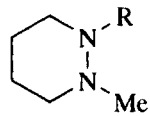
3-Oxo-2,3-dihydropyridazinium-1-ethoxyformamidate (**82**) gave its 2-methyl derivative (**83**) ($CH_2N_2$, $Et_2O$, 25°, 1 h: 42%) and thence the 2,4-dimethyl derivative (**84**) ($CH_2N_2$, $Et_2O$-$CH_2Cl_2$, 25°, 1 h: 77%).[877]



(82) (83) (84)

### *N*-Alkylation of Hexahydropyridazines

1-Methylhexahydropyridazine (**85**, R = H) gave 1-ethyl-2-methylhexahydropyridazine (**85**, R = Et) (MeCHO, $NaBH_3CN$, MeCN, $H_2O$, AcOH gradually, 7 h: ~50%).[604]



(85)

## *3.2.1.2 By Nuclear Addition Reactions*

Most of these alkylations occur by the addition of an appropriate Grignard, lithium, or tin reagent across one double bond of a pyridazine to afford an alkyl-

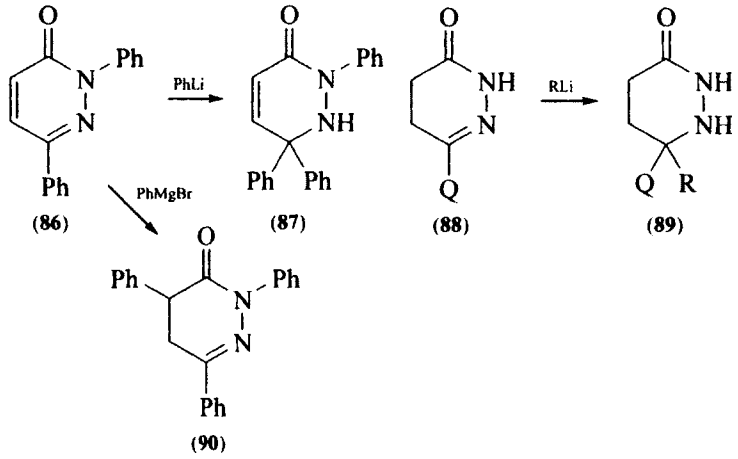
or aryldihydropyridazine, many of which may be oxidized to the corresponding alkyl- or arylpyridazines. However, some nonmetallic reagents have been so used. The following examples, classified according to the type of reagent, illustrate these additive alkylations and arylations:

### Using Alkyl- or Aryllithiums

2,6-Diphenyl-3(2*H*)-pyridazinone (**86**) gave 2,6,6-triphenyl-1,6-dihydro-3(2*H*)-pyridazinone (**87**) (PhLi, $Et_2O$-PhH, $-50°$; then reflux, 5 h: 75%).[113]

6-*p*-Tolyl-4,5-dihydro-3(2*H*)-pyridazinone (**88**, Q = $C_6H_4Me$-*p*) and phenyllithium gave 6-phenyl-6-*p*-tolyl-1,4,5,6-tetrahydro-3(2*H*)-pyridazinone (**89**, Q = $C_6H_4Me$-*p*, R = Ph) ($Et_2O$-PhH, reflux, 8 h; 85%); 6-phenyl-4,5-dihydro-3(2*H*)-pyridazinone (**88**, Q = Ph) and *p*-tolyllithium gave the same product (**89**, Q = Ph, R = $C_6H_4Me$-*p*) (similarly: 70%).[366]

Also other examples.[452,453,1199,1201]



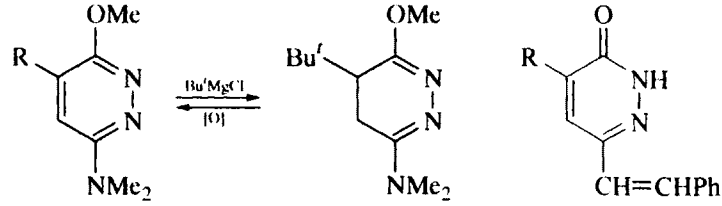

### Using Grignard Reagents

2,6-Diphenyl-3(2*H*)-pyridazinone (**86**) gave 2,4,6-triphenyl-4,5-dihydro-3(2*H*)-pyridazinone (**90**) [PhMgBr, $Et_2O$–PhH, reflux, 8 h: 64%; cf. the isomer (**87**) produced by PhLi (see preceding subsection)].[113]

3-Dimethylamino-6-methoxypyridazine (**91**, R = H) gave 4-*t*-butyl-6-dimethylamino-3-methoxy-4,5-dihydropyridazine (**92**) ($Bu^tMgCl$, $Et_2O$, 25°, 1 h: ~70%) and then 4-*t*-butyl-6-dimethylamino-3-methoxypyridazine (**91**, R = $Bu^t$)[Br, $H_2O$; crude product, MeONa, MeOH, reflux, 10 min (dehydrobromination): ~75%];[137] also analogous reactions.[136,137]

6-Styryl-3(2*H*)-pyridazinone (**93**, R = H) gave 4-phenyl-6-styryl-3(2*H*)-pyridazinone (**93**, R = Ph) (excess PhMgBr, $Et_2O$–PhH, reflux,

8 h: 70%; presumably via a dihydro derivative);[369] also analogous reactions.[369, 375, 634]



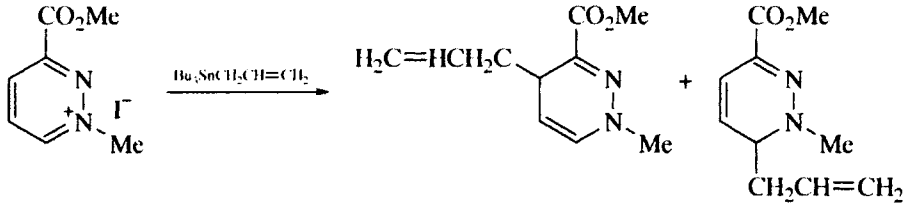

(91) (92) (93)

3-Azido-4-pyridazinecarbonitrile gave 3-azido-5-phenylpyridazine, presumably by loss of HCN from the intermediate 3-azido-5-phenyl-4,5-dihydro-4-pyridazinecarbonitrile (PhMgCl, THF, A, 0° → 25°, 4 h: 90%).[1408, cf. 1595]

Also other examples.[366, 370, 780, 843, 1102, 1245, 1247, 1253, 1263, 1277, 1278, 1329, 1346]

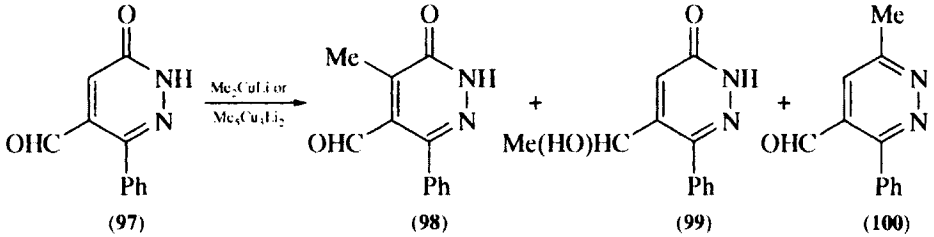
### Using Other Organometallic Reagents

Methyl 3-pyridazinecarboxylate 1-methiodide (**94**) with allyltributyltin gave a separable mixture of methyl 4-allyl-1-methyl-1,4-dihydro- (**95**) and methyl 6-allyl-1-methyl-1,6-dihydro-3-pyridazinecarboxylate (**96**) (MeCN, 0°, 5 h: 18% and 52%, respectively); also analogues.[1469]




(94) (95) (96)

6-Oxo-3-phenyl-1,6-dihydro-4-pyridazinecarbaldehyde (**97**) with the typical methyllithium–methylcopper complex, $Me_2CuLi$ or $Me_5Cu_3Li_2$, gave a separable mixture of 5-methyl-6-oxo-3-phenyl-1,6-dihydro-4-pyridazinecarbaldehyde (**98**), 5-$\alpha$-hydroxyethyl-6-phenyl-3(2*H*)-pyridazinone (**99**), and 6-methyl-3-phenyl-4-pyridazinecarbaldehyde (**100**) [metallic complex (made in situ), $Et_2O$–$C_5H_{12}$, −75°, 2 h: ~80% prior to separation].[370]

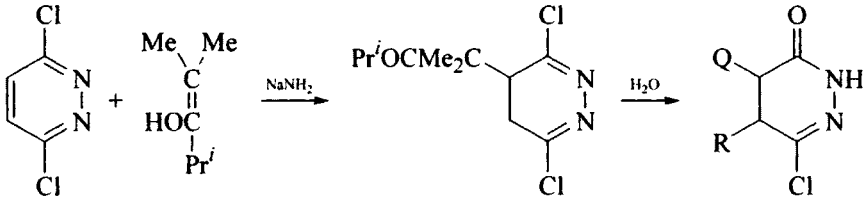



(97) (98) (99) (100)

**Using Nonmetallic Reagents**

3,6-Dichloropyridazine (**101**) and diisopropyl ketone (enol: **102**) gave 3,6-dichloro-4-(α-isobutyryl-α-methylethyl)-4,5-dihydropyridazine (**103**) ($NaNH_2$, liquid $NH_3$–$Et_2O$, 15 min?), characterized by partial hydrolysis to a separable mixture of 6-chloro-4-(**104**, Q = $CMe_2COPr^t$, R = H) and 6-chloro-5-(α-isobutyryl-α-methylethyl)-4,5-dihydro-3(2*H*)-pyridazinone (**104**, Q = H, R = $CMe_2COPr^i$) wet silica gel, $Et_2O$, 25°, 24 h).[334]

Also other examples.[2103]




(**101**) (**102**) (**103**) (**104**)

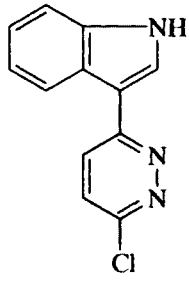
### *3.2.1.3 By Displacement Reactions*

Pyridazines bearing an halogeno or other such leaving group can be converted into the corresponding alkyl- or arylpyridazine by treatment with a hydrocarbon in the presence of a suitable catalyst, by treatment with a carbanion, or by treatment with an organometallic or other such "active" reagent; in all these procedures, the entering alkyl or aryl substituent may bear passenger groups. The following examples illustrate the foregoing possibilities:

**From Halogenopyridazines with Hydrocarbons ($AlCl_3$-Assisted)**

3,6-Dichloropyridazine and indole gave 3-(6-chloropyridazin-3-yl)indole (**105**) ($AlCl_3$, $ClCH_2CH_2Cl$, 20–35°, 90 min; then reflux, 30 min: 30%).[359]

Also other examples.[359,1772]



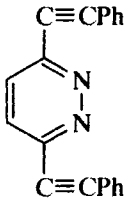

(**105**)

### From Halogenopyridazines with Hydrocarbons (Pd/Cu-Assisted)

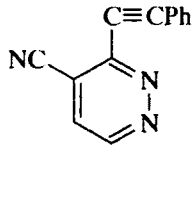
3,6-Diiodopyridazine and phenylacetylene gave 3,6-bisphenylethynylpyridazine (**106**) [$Et_3N$, $Me_2SO$, $(Ph_3P)_2PdCl_2 + CuI$, 25°, A, 2 days: 61%].[1638]

3-Chloro-4-pyridazinecarbonitrile and phenylacetylene gave 3-phenylethynyl-4-pyridazinecarbonitrile (**107**) [$Et_3N$, $(PH_3P)_2PdCl_2 + CuI$, 25°, 7 h: 78%].[1791]

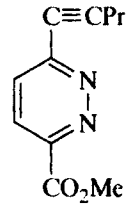
Methyl 6-chloro-3-pyridazinecarboxylate and pent-1-yne gave methyl 6-pent-1′-ynyl-3-pyridazinecarboxylate (**108**) [$Et_3N$, $(Ph_3P)_2PdCl_2CuI$, 50°, 20 h: 80%] and then methyl 6-pentyl-3-pyridazinecarboxylate ($H_2$, Pd/C, MeOH: 96%).[864,1592]



(**106**)



(**107**)



(**108**)

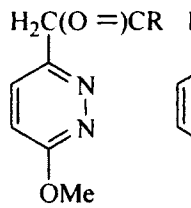
3-Iodo-6-methoxypyridazine and prop-2-yn-1-ol gave 3-(3-hydroxyprop-2-ynyl)-6-methoxypyridazine [$Et_3N$, $(Ph_3P)_2PdCl_2 + CuI$, THF, 25°, 8 h: 71%].[1385]

Also other examples.[122,191,399,834]

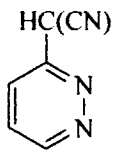
### From Halogenopyridazines with Carbanions

3-Chloro-6-methoxypyridazine and acetone gave 3-acetonyl-6-methoxypyridazine (**109**, R = Me) ($NaNH_2$, liquid $NH_3$–$Et_2O$, dark, 15 min: 60%) or with pinacolone gave 3-methoxy-6-pivaloylmethylpyridazine (**109**, R = Bu′) ($NaNH_2$, liquid $NH_3$–$Et_2O$, *hν*, 15 min: 72%).[326]

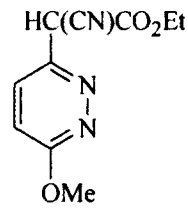
3,6-Dichloropyridazine with malononitrile gave 3-chloro-6-dicyanomethylpyridazine (**110**, R = CN) (KOH/$H_2O$, $Me_2SO$ or $Me_2NCHO$, 80°, 4 h: >50%);[74] with *o*-methoxyphenylacetonitrile it gave 3-chloro-6-α-cyano-*o*-methoxybenzylpyridazine (**110**, R = $C_6H_4OMe$-*o*) (Bu′OK, Bu′OH,



(**109**) (**110**)



(**111**)



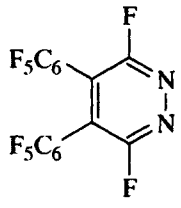
(**112**)

35°, 90 min: 84%);[750,1197] with 2-(3-benzyloxy-6-methoxyphenyl) acetonitrile it gave 3-(3-benzyloxy-α-cyano-6-methoxybenzyl)-6-chloropyridazine [**110**, R = $C_6H_3(OCH_2Ph)OMe$-3,6] (NaH, $Me_2NCHO$, ?°, 2 h: 64%);[749] or with phenylacetonitrile it gave 3-chloro-6-α-cyanobenzylpyridazine (**110**, R = Ph) ($PhCH_2CN$, PhMe, $NaNH_2$, 0°, 10 min; then substrate↓, 20°, 5 h: 67%).[1354]

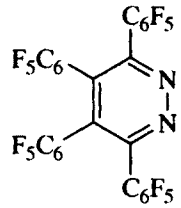
3-Chloropyridazine with phenylacetonitrile gave 3-α-cyanobenzylpyridazine (**111**) ($NaNH_2$, PhH, <5°; then 25°, 6 h; then reflux, 4 h: 65%).[839]

3-Chloro-6-methoxypyridazine with ethyl cyanoacetate gave 3-(α-cyano-α-ethoxycarbonylmethyl)-6-methoxypyridazine (**112**) [NaH, $(Me_2N)_3PO$, 100°, 2 h: 50%].[1184]

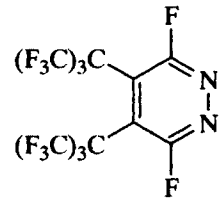
Tetrafluoropyridazine (1 mol) with pentafluorophenyllithium (2 mol; made in situ) gave perfluoro-4,5-diphenylpyridazine (**113**) ($Et_2O$–$C_6H_{14}$, −78° → 25°, 14 h: 30%) or with the same reagent (>4 mol), perfluorotetraphenylpyridazine (**114**) (same conditions: 86%).[653]



(**113**)



(**114**)



(**115**)

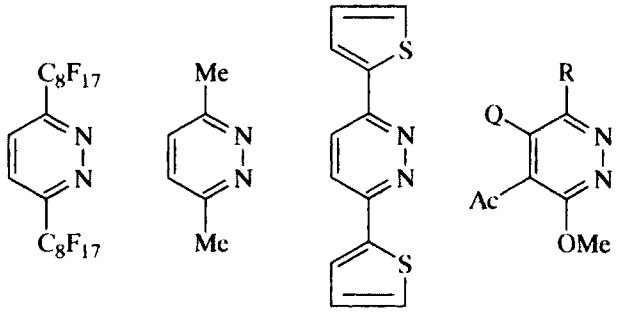
Tetrafluoropyridazine with perfluoroisobutene gave perfluoro-3,6-di-*t*-butylpyridazine (**115**) [CsF, tetrahydrothiophene dioxide as solvent, 80°, 10 h; 75%; note the formation of a carbanion thus: $(F_3C)_2C{=}CF_2 + F^- \rightarrow (F_3C)_3C^-$];[649] for other such fascinating but specialized reaction of perfluorocarbanions, see References 456, 457, 469, 643, 645, 647, 649, 653, 658, 660, 745.

Also other examples.[534,857,1030,1377,1441,1547,1628,1631,1698,1798,2098]

## From Halogenopyridazines with Other Reagents

3,6-Dichloropyridazine with perfluorooctyl iodide gave 3,6-bis(perfluorooctyl)pyridazine (**116**) and a little 3-chloro-6-(perfluorooctyl)pyridazine (Cu, 2,2′-bipyridine, $Me_2SO$, $C_6F_6$, 85°, 6 days: 68% and <5%, respectively; akin to an Ullmann biaryl synthesis).[1577]

3-Chloro-6-methylpyridazine gave 3,6-dimethylpyridazine (**117**) (MeMgI, $Ph_2PCH_2CH_2CH_2PPh_2 \cdot NiCl_2$, $Et_2O$, substrate↓ slowly, reflux, $N_2$, 3 h: 71%); 3,6-dichloropyridazine similarly gave the same product (**117**) (19%)[823] or 3,6-dithien-2′-ylpyridazine (**118**) (thien-2-yl-MgBr, $Ph_2PCH_2CH_2CH_2PPh_2 \cdot NiCl_2$, $Et_2O$, substrate↓ slowly, <25°, 48 h; 90%).[1062] Contrast these results with the addition reactions that occur in the absence the Ni catalyst (Section 3.2.1.2).

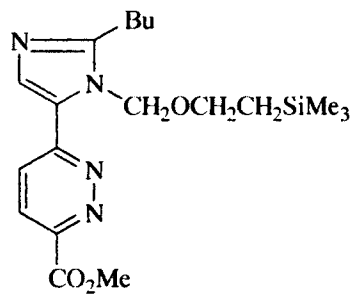


(116) (117) (118) (119)

3-Chloro- or 3-iodo-6-methoxypyridazine gave 3-methoxy-6-phenylpyridazine [$PhB(OH)_2$, $Pd(PPh_3)_4$, 2M $Na_2CO_3$, EtOH, reflux, 12 h: 75%].[1385, 1569]

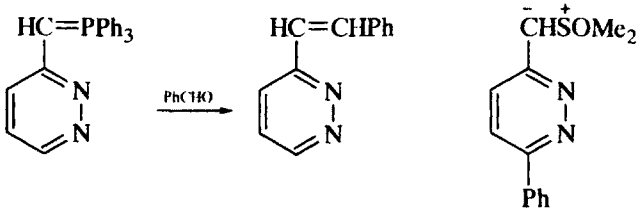
4-Acetyl-6-chloro-5-iodo-3-methoxypyridazine (**119**, Q = I, R = Cl) and *p*-chlorophenylboronic acid (1 mol) gave 4-acetyl-6-chloro-5-*p*-chlorophenyl-3-methoxypyridazine (**119**, Q = $C_6H_4Cl$-*p*, R = Cl) [$K_2CO_3$, EtOH–PhMe; $Pd(Ph_3P)_4$ ↓, A, 110°, 3 days: 87%] and then, with phenylboronic acid (1 mol), 4-acetyl-5-*p*-chlorophenyl-3-methoxy-6-phenylpyridazine (**119**, Q = $C_6H_4Cl$-*p*, R = Ph) (same conditions: 87%); note the apparent preference for displacement of the iodo substituent, although positional factors could be relevant.[1610]

Methyl 6-chloro-3-pyridazinecarboxylate and 2-butyl-4-tributylstannyl-3-(β-trimethylsilylethoxymethyl)imidazole gave methyl 6-[2-butyl-3-(β-trimethylsilylethoxymethyl)imidazol-4-yl]-3-pyridazinecarboxylate (**120**) [$Pd(PPh_3)_4$, 2,6-$Bu^t_2$-4-Me-phenol, dioxane, reflux, 7 h: 76%] and then methyl 6-(2-butylimidazol-4-yl)-3-pyridazinecarboxylate (HCl, MeOH, reflux, 4 h: 62%);[1754] also other examples using stannyl reagents.[1385, 1574, 1831]



(120)

3-Chloropyridazine with methylenetriphenylphosphorane ($H_2C{=}PPh_3$) gave the unisolated intermediate (**121**) that reacted with benzaldehyde to give 3-styrylpyridazine (**122**) (PhH, reflux, 4 h; then PhCHO ↓, 25°, 30 min: 88%).[26]



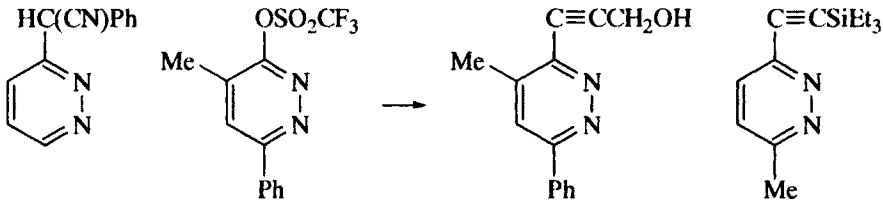

(121) (122) (123)

3-Chloro-6-phenylpyridazine with so-called dimethyloxosulfonium methylide ($Me_2\overset{+}{S}O\overset{-}{C}H_2$) gave dimethyloxosulfonium 6-phenylpyridazin-3-ylmethylide (**123**) (THF, reflux, $N_2$, 5 h: 81%), a useful substrate for side-chain elaboration.[836]

## From Alkoxy- or Acyloxypyridazines

3-Methoxypyridazine gave 3-α-cyanobenzylpyridazine (**124**) [$PhCH_2CN$, NaH, THF, reflux, 30 min; then substrate↓, reflux, until complete (tlc): 58%]; 4-α-cyanobenzylpyridazine was made similarly.[1586]

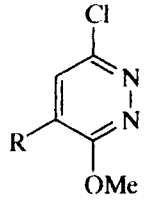
4-Methyl-6-phenyl-3-trifluoromethanesulfonyloxypyridazine (**125**) gave 3-(3-hydroxyprop-1-ynyl)-4-methyl-6-phenylpyridazine (**126**) [$HC{\equiv}CCH_2OH$ (2 mol), $(PhP)_2PdCl_2 + CuI$, $Pr^i_2NH$, THF, 0°, 5 h: 88%]; other substituted-alkynyl derivatives, including 4-methyl-6-triethylsilylethynylpyridazine (**127**) (89%), were made likewise.[1629]




(124) (125) (126) (127)

## From Trialkylsilylpyridazines

3-Chloro-6-methoxy-5-trimethylsilylpyridazine (**128**, R = $SiMe_3$) and acetaldehyde gave 3-chloro-5-α-hydroxyethyl-6-methoxypyridazine [**128**, R = CH(OH)Me] with 3-chloro-6-methoxypyridazine (**128**, R = H) as a byproduct (THF, $Bu_4NF$ in three portions, 20°, 4 h: 78% and 22%, respectively);[1610] propionaldehyde and benzaldehyde likewise gave the 5-α-hydroxypropyl [**128**, R = CH(OH)Et] (66%) and 5-α-hydroxybenzyl homologue [**128**, R = CH(OH)Ph] (71%), respectively.[1610]



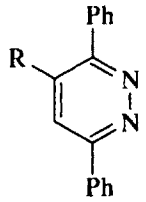
(128)

### From Trialkylstannylpyridazines

4-Tributylstannyl- gave 4-phenylpyridazine [PhI, $Pd(PPh_3)_4$, PhMe, reflux, 48 h: 51%];[2046] 3,5-diphenylpyridazine likewise.[2093]

3,6-Diphenyl-4-tributylstannylpyridazine (**129**, $R = SnBu_3$) gave 3,4,6-triphenylpyridazine (**129**, $R = Ph$) [PhI, $(Ph_3P)_2PdCl_2$, $Et_4NCl$, $Me_2NCHO$, 80°, 30 h: 39%; PhBr, similarly: 25%].[1591]

*From quaternized alkylthiopyridazines* See Section 6.2.2.2



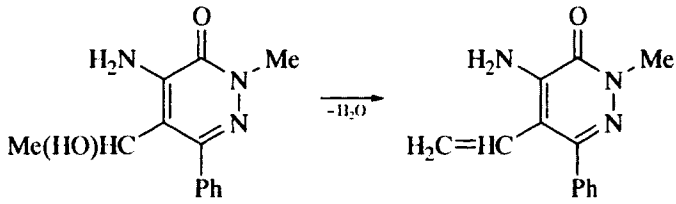
(129)

## *3.2.1.4 By Elimination of Functional Groups*

This diverse set of preparative methods is used mainly to produce simple (unsubstituted-alkyl)pyridazines, as illustrated in the following examples:

### Eliminations from Side Chains

4-Amino-5-α-hydroxyethyl-2-methyl-6-phenyl- (**130**) gave 4-amino-2-methyl-6-phenyl-5-vinyl-3(2*H*)-pyridazinone (**131**) (96% $H_2SO_4$, 25°, 2 h: 48%).[1832]

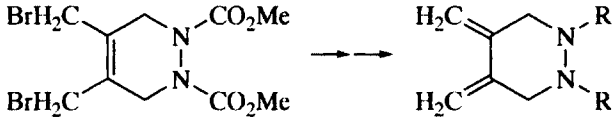


(130) (131)

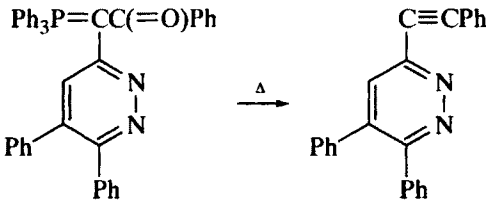
3-β-Hydroxyethylpyridazine (crude) gave 3-vinylpyridazine (distillation from solid NaOH at 120°/1 mm, radical inhibitor: ~20%).[494]

2-β-Chloroethyl-3(2*H*)-pyridazinone gave 2-vinyl-3(2*H*)-pyridazinone MeONa, MeOH, reflux, 3 h: 70%).[1179]

Dimethyl 4,5-bisbromomethyl-1,2,3,6-tetrahydro-1,2-pyridazinedicarboxylate (**132**) gave a product formulated as dimethyl 4,5-dimethylenehexahydro-1,2-pyridazinedicarboxylate (**133**, R = $CO_2Me$) [ZnCu, $Et_2O$-$(Me_2N)_3PO$, reflux, 2 h: 72%, unstable but characterized] and then a second product formulated as 4,5-dimethylenehexahydropyridazine (**133**, R = H) (KOH, $H_2O$–MeOH, reflux, A, 4 h; $CO_2\uparrow$ on $H^+\downarrow$: 62% unstable in air).[323]



(132) (133)



(134) (135)

3,4-Diphenyl-6-[α-(triphenylphosphoranylidene)phenacyl]pyridazine (**134**) gave 3,4-diphenyl-6-phenylethynylpyridazine (**135**) (neat, 230°, sealed, 15 min: 96%; loss of $Ph_2PO$).[330]

4-Carboxymethyl-6-chloro- gave 6-chloro-4-methyl-3(2*H*)-pyridazinone (Cu powder, 150°, 20 min: 71%).[947]

4-α-Hydroxyethylpyridazine underwent thermal disproportionation to 4-ethyl- and 4-acetylpyridazine [neat, 120°, 5 h: products identified by mass spectroscopy (ms)].[73]

Also other examples.[92,433,434,769,1547,1605]

## Eliminations from the Pyridazine Nuclei

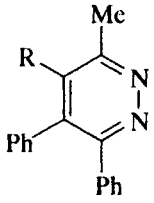
3-Methyl-5,6-diphenyl-4-pyridazinecarboxylic acid (**136**, R = $CO_2H$) gave 3-methyl-5,6-diphenylpyridazine (**136**, R = H) (neat, 200°, 30 min: >95%);[314] also an analogous example.[330]

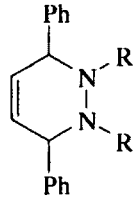
Diethyl 3,6-diphenyl-1,2,3,6-tetrahydro-1,2-pyridazinedicarboxylate (**137**, R = $CO_2Et$) gave crude dipotassium 3,6-diphenyl-1,2,3,6-tetrahydro-

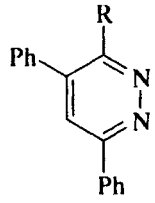
1,2-pyridazinedicarboxylate (**137**, R = $CO_2K$) (KOH, MeOH, reflux, $N_2$, 24 h) and then 3,6-diphenyl-1,2,3,6-tetrahydropyridazine (**137**, R = H) (AcOH, $H_2O$, 20°, 30 min: 95% overall).[1204] also a related example.[1146]

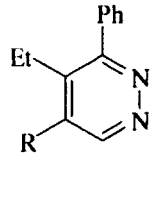
3-Benzoyl-4,6-diphenylpyridazine (**138**, R = Bz) gave 3,5-diphenylpyridazine (**138**, R = H) (Bu$^t$OK, dioxane, trace $H_2O$, 20°, $N_2$, 4 h: 82%).[404]

4-Ethyl-3-phenyl-5-trimethylsilylpyridazine (**139**, R = $SiMe_3$) gave 4-ethyl-3-phenylpyridazine (**139**, R = H) (KOH, EtOH–$H_2O$, 60°, 12 h: 57%);[12] analogues likewise.[12]


(136)

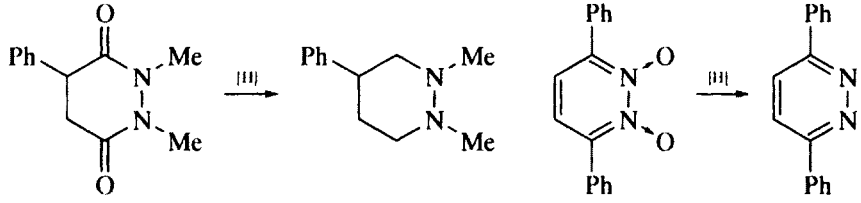

(137)


(138)


(139)

1,2-Dimethyl-4-phenyl-4,5-dihydro-3,6(1*H*,2*H*)-pyridazinedione (**140**) gave 1,2-dimethyl-4-phenylhexahydropyridazin (**141**) ($LiAlH_4$, THF, 25°; then reflux, 20 h: 67%);[1328] also related examples.[1051,1833]

3,6-Diphenylpyridazine 1,2-dioxide (**142**) gave 3,6-diphenylpyridazine 1-oxide and 3,6-diphenylpyridazine (**143**) ($H_2$, Pd/C, EtOH: 89% and 6%, respectively; reaction probably not optimized);[321] other appropriate *N*-oxides also gave their parent alkylpyridazines by a variety of methods, usually in poor yield.[230,411,426,625,1193]


(140) (141) (142) (143)

3-Bromo-6-butyl-3,6-dimethyl-3,4,5,6-tetrahydropyridazine gave 3-butyl-3,6-dimethyl-3,4-dihydropyridazine (1,5-diazabicyclo[4.3.0]non-5-ene, THF, $N_2$, 25°, 10 h: ~20%; by elimination of HBr) and then the isomeric 3-butyl-3,6-dimethyl-2,3-dihydropyridazine (PhH, 125°, sealed, 12 h: unisolated but identified by nmr).[1201]

3-Ethoxy-6-phenyl-2-tosyl-2,3,4,5-tetrahydropyridazine gave 3-phenylpyridazine [EtONa, EtOH, 60°, 18 h: 81%; by elimination of EtOH and TsH].[1237]

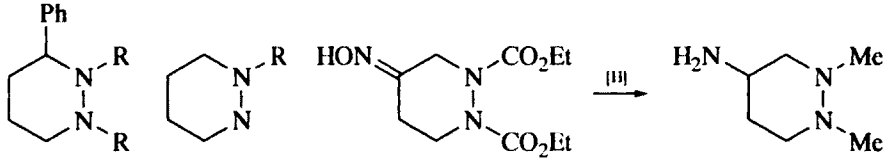
### 3.2.1.5 *By Modification of Aldehydes, Ketones, or Esters*

Most such modifications involve reduction to alkyl- or aralkylpyridazines or the conversion of pyridazine aldehydes or ketones into (substituted-vinyl)pyridazines by condensation with methylene reagents. The following examples illustrate these routes to alkylpyridazines:

**Reductions**

Diethyl 3-phenylhexahydro-1,2-pyridazinedicarboxylate (**144**, R = $CO_2Et$) gave 1,2-dimethyl-3-phenylhexahydropyridazine (**144**, R = me) ($LiAlH_4$, $Et_2O$, 20 h: 93%).[1329]

Ethyl hexahydro-1-pyridazinecarboxylate (**145**, R = $CO_2Et$) gave 1-methylhexahydropyridazine (**145**, R = Me)($LiAlH_4$, $Et_2O$, 25°, 2.5 h, then reflux, 4 h: 42%;[604] likewise but reflux 16 h: ~90%)[972]

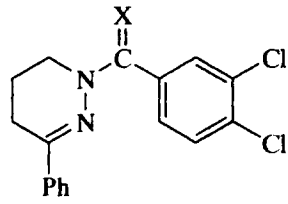



(144) (145) (146) (146a)

Diethyl 4-hydroxyiminohexahydro-1,2-pyridazinedicarboxylate (**146**) gave 1,2-dimethylhexahydro-4-pyridazinamine (**146a**) ($LiAlH_4$, $Et_2O$, reflux, 28 h: 60%).[1699]

1-(3,4-Dichlorobenzoyl)-(**147**, X = O) gave 1-(3,4-dichlorobenzyl)-3-phenyl-1,4,5,6-tetrahydropyridazine (**147**, X = $H_2$) ($LiAlH_4$, THF, $N_2$, 5° → 20°, 1 h: ~55%).[1834]

Also other examples.[91,790,1671]



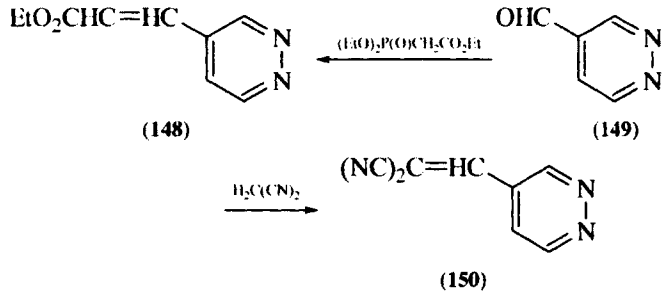

(147)

**Condensations with methylene reagents**

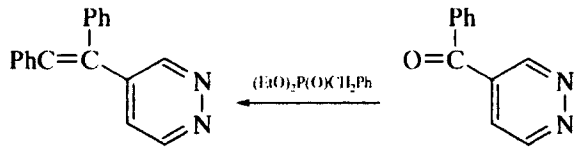
4-Pyridazinecarbaldehyde (**149**) with triethyl phosphonoacetate gave 4-β-ethoxycarbonylvinylpyridazine (**148**) (NaH, $Me_2NCHO$, 25°, 2 h: 49%)

or with malononitrile gave 4-$\beta,\beta$-dicyanovinylpyridazine (**150**) (EtOH, 25°, 2 h: 80%); also analogues.[1668]

4-Benzoylpyridazine (**152**) with diethyl benzylphosphonate gave 4-$\alpha$-phenylstyrylpyridazine (**151**) (NaH, $Me_2NCHO$, 25°, 16 h: 70%); also analogues.[1668]

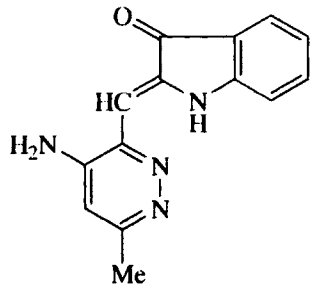


(148) (149) (150)



(151) (152)

4-Amino-6-methyl-3-pyridazinecarbaldehyde with 1-acetyl-3-indolinone gave 2-(4-amino-6-methylpyridazin-3-ylmethylene)-3-indolinone (**153**) (EtOH, trace KOH, 25°, 24 h: ~35%).[1020]



(153)

3-Pyridazinecarbaldehyde 2-oxide with 3-methylpyridazine 2-oxide gave $\alpha,\beta$-bis(2-oxidopyridazin-3-yl)ethylene (MeONa, MeOH, reflux, 1 h: 21%) and then $\alpha,\beta$-bispyridazin-3′-ylethylene ($PCl_3$, $CHCl_3$, reflux, 48 h: 19%).[767]

Also other examples[67,511,960,1603,1811]

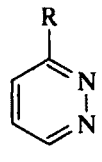
### 3.2.1.6 *By Interconversion of Simple Alkyl Substituents*

Alkylpyridazines, usually with no functional group(s) on their alkyl substituents, may be converted into other such alkyl-, alkenyl-, or aralkylpyridazines in several ways, as illustrated in the following examples.

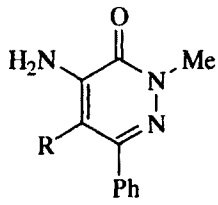
**Reduction**

3-Styrylpyridazine (**154**, R = CH:CHPh) gave 3-phenethylpyridazine (**154**, R = $CH_2CH_2Ph$)($H_2$, Pd/C, EtOH, 25°: 91%);[1791] likewise 4-styryl- gave 4-phenethylpyridazine (87%).[67]

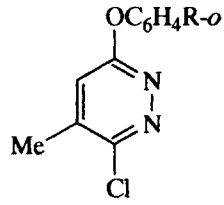
4-Amino-2-methyl-6-phenyl-5-vinyl-3(2*H*)-pyridazinone (**155**, R = CH:$CH_2$) gave 4-amino-5-ethyl-2-methyl-6-phenyl-3(2*H*)-pyridazinone (**155**, R = Et) ($H_2$, Pd/C, EtOH, 25°, 45 min: 66%);[1832] likewise 6-methyl-2-vinyl- gave 2-ethyl-6-methyl-3(2*H*)-pyridazinone ($H_2$, Pt, $F_3CCO_2H$, 20°, 2 h: >95%; or $Et_3SiH$, $F_3CCO_2H$, 65°, 25 h: 67%).[1134]



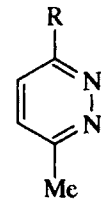

(**154**)




(**155**)




(**156**)




(**157**)

3-*o*-Allylphenoxy-6-chloro-5-methylpyridazine (**156**, R = $CH_2CH:CH_2$) gave 3-chloro-4-methyl-6-*o*-propylphenoxypyridazine (**156**, R = Pr) ($H_2$, Pd/C, $NH_4OH$MeOH: 98%).[766]

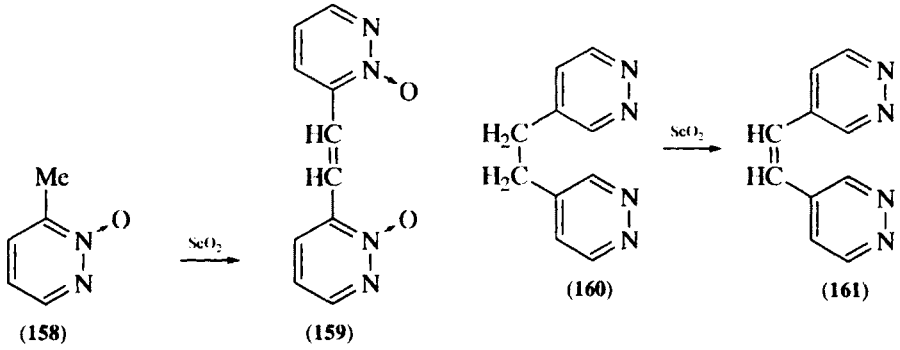
3-Methyl-6-phenylethynylpyridazine (**157**, R = CH : CPh) gave 3-methyl-6-phenethylpyridazine (**157**, R = $CH_2CH_2Ph$) ($H_2$, Pd/C, EtOH–$H_2O$: 97%);[834] likewise, 3,4-diphenyl-6-phenylethynyl- gave 3-phenethyl-5,6-diphenylpyridazine ($H_2$, Pt, EtOAc, 3 atm, 4 h: 93%).[330]

Also other examples.[6,122,160,767,1385,1734]

**Oxidation**

3-Methylpyridazine 2-oxide (**158**) gave α,β-bis(2-oxidopyridazin-3-yl)ethylene (**159**) ($SeO_2$, pyridine, reflux, 6 h: 49%; probably by partial conversion into the pyridazinecarbaldehyde oxide and condensation with unchanged methylpyridazine oxide; see end of Section 3.2.1.5).[767]

α,β-Dipyridazin-4-ylethane (**160**) gave α,β-dipyridazin-4-ylethylene (**161**) ($SeO_2$, $H_2O$, 70°, 3 h: 76%).[73]




(158) (159) (160) (161)

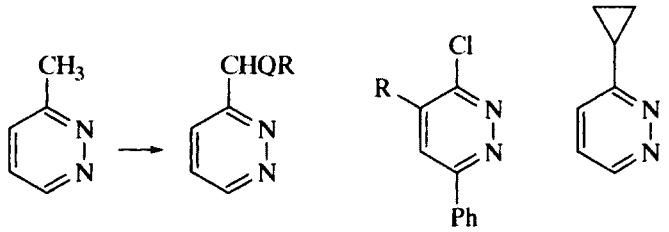
### By Extranuclear Alkylation or Arylation

3-Methylpyridazine (**162**) afforded a separable mixture of 3-ethyl- (**163**, Q = H, R = Me) and 3-isopropylpyridazine (**163**, Q = R = Me) [LiNPr$^i$ (made in situ), THF, substrate↓, −70° → 25°, 1 h; then MeI, −70° → 25°, 3 h: 40% and 15%, respectively];[822] likewise, 3-methyl- (**162**) gave 3-phenethyl- (**163**, Q = H, R = $CH_2Ph$), and 3-(dibenzylmethyl)pyridazine (**163**, Q = R = $CH_2Ph$) ($PhCH_2Cl$: 52% and 6%, respectively).[822]

3-Methylpyridazine (**162**) gave 3-benzylpyridazine (**163**, Q = H, R = Ph) [$NaNH_2$ (made in situ), liquid $NH_3$, PhBr, −78°, 15 min: 17%].[123]

3-Chloro-4-methyl-6-phenylpyridazine (**164**, R = Me) gave 3-chloro-4-pentyl-6-phenylpyridazine (**164**, R = $C_5H_{11}$) [$LiNPr^i_2$ (made in situ), THF, substrate↓, −70°, 90 min; then BuBr, 25°, 12 h: 18%].[1632]

3-Vinylpyridazine gave 3-cyclopropylpyridazine (**165**) ($Me_2\overset{+}{S}O\overset{-}{C}H_2$ or $Me_2\overset{+}{S}\overset{-}{C}H_2$, no details: 42% or 20%, respectively; essentially an addition to the vinyl double bond; the original named the product as its 2 isomer).[178]




(162) (163) (164) (165)

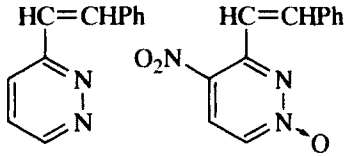
Also other examples in the foregoing references and elsewhere.[537]

### By Extranuclear Alkylidenation

3-Methylpyridazine gave 3-styrylpyridazine (**166**) (neat PhCHO, $ZnCl_2$, 150°, 6 h: 66%).[77]

4-Methylpyridazine gave 4-styrylpyridazine (neat PhCHO, $ZnCl_2$, reflux, 5 h: 64%;[67] PhCHO, NaOH, $Me_2SO$-$H_2O$, 25°, 5 h: 83%).[509]

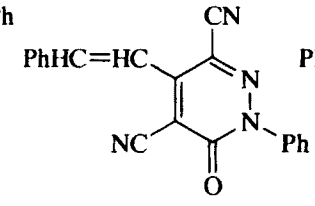
3-Methyl-4-nitropyridazine 1-oxide gave 4-nitro-3-styrylpyridazine 1-oxide (**167**) [PhCHO, $HN(CH_2)_5$, MeOH, reflux, 20 h: 59%];[904] 3-chloro-6-methoxy-5-nitro-4-styrylpyridazine 2-oxide from its 4-methyl analogue similarly (reflux, 6 h: 75%).[902]



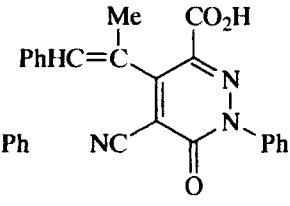
(166)



(167)



(168)



(169)

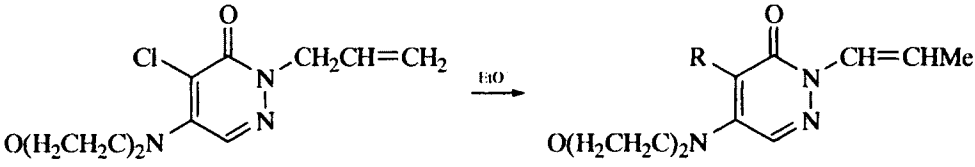
4-Methyl-6-oxo-1-phenyl-1,6-dihydro-3,5-pyridazinedicarbonitrile gave 6-oxo-1-phenyl-4-styryl-1,6-dihydro-3,5-pyridazinedicarbonitrile (**168**) (PhCHO, pyridine, reflux, 4 h: 70%;[1217] or neat PhCHO, 100°, 5 h: 70%).[395]

5-Cyano-4-ethyl-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylic acid gave 5-cyano-4-α-methylstyryl-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylic acid (**169**) (PhCHO, pyridine, reflux, 6 h: 69%).[1808]

Also other examples.[527,853,1363,1645]

### By Prototropy

2-Allyl-4-chloro-5-morpholino-3(2*H*)-pyridazinone (**170**) with ethoxide ion gave a separable mixture of 5-morpholino-2-prop-1′-enyl-3(2*H*)-pyridazinone (**171**, R = H) and its 4-ethoxy derivative (**171**, R = OEt) (EtONa, EtOH, reflux, 3 h: 10% and 25%, respectively; note isomerization of the alkenyl group in each product).[1269]

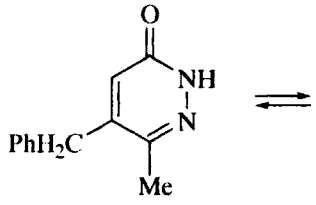


(170) (171)

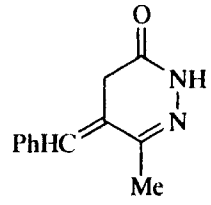
## 3.2.2 Properties and Reactions of *C*-Alkyl- and *C*-Arylpyridazines

Some interesting new material on physical aspects of alkylpyridazines has been reported. Both 3- and 4-methylpyridazine have been included in a broad

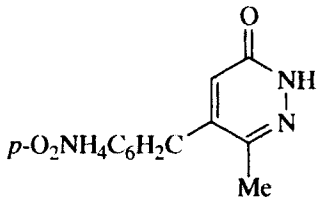
nmr study of bond orders in heteroaromatic systems (based on *o*-benzylic coupling constants) that may be used to measure the relative degree of aromaticity in such systems.[1686] The electron-impact ionization ms and tandem ms of pyridazine, 3- and 4-benzylpyridazine, and 3- and 4-phenethylpyridazine show marked differences between the fragmentation patterns of the 3- and the 4-substituted derivatives.[1666] Despite earlier claims that benzylpyridazines, such as 5-benzyl-4-methyl-3(2*H*)-pyridazinone (**172**), existed in their benzylidenedihydro forms, including 5-benzylidene-6-methyl-4,5-dihydro-3(2*H*)-pyridazinone (**173**),[1067,1446,1667] a more recent interpretation of the nmr and ms data suggested that this was not the case;[1827] moreover, the X-ray crystal structure of one such compound, 6-methyl-5-*p*-nitrobenzyl-3(2*H*)-pyridazinone (**174**), precluded the nitrobenzylidenedihydro form in the solid state.[1827] However, there has been no challenge to the formulation of 4,5-diisopropylidene-1,2-dimethylhexahydropyridazine (**175**).[589] The preferred conformation(s) of 1,2-dimethylhexahydropyridazine appear(s) to be still an open question.[600,609,618,1125,1528,1859] Several metal complexes have been reported.[472,1570]



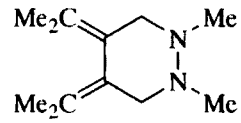

(172)




(173)




(174)




(175)

Alkyl and aryl groups attached to pyridazine undergo a variety of reactions. Of these, the conversion of one simple alkyl group into another (by reduction, oxidation, alkylation, alkylidenation, or prototropy) has been covered already in Section 3.2.1.6. Other reactions, including the foregoing processes in which a functional passenger group is borne on the starting or finishing alkyl group, are discussed in the following sections.

### *3.2.2.1 Oxidative Reactions (H, 365, 409)*

Alkylpyridazines may be oxidized to aldehydes, ketones, or carboxylic acids according to substrate, reagent, and conditions; they may also undergo nuclear

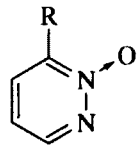
oxidation, oxidative ring fission, oxidative hydroxylation, and so on. These reactions are illustrated in the following examples:

### Oxidation to Aldehydes

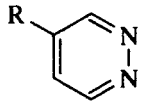
3-Methylpyridazine 2-oxide (**176**, R = Me) gave 3-pyridazinecarbaldehyde 2-oxide (**176**, R = CHO) ($SeO_2$, pyridine, reflux, 6 h: 45%).[767] Also other such oxidations.[1111, 1261]

4-Styrylpyridazine (**177**, R = CH:CHPh) gave 4-pyridazinecarbaldehyde (**176**, R = CHO) ($NaIO_4$, trace $OsO_4$, $H_2O$–dioxane, 25°, $N_2$, 5 h: 87%).[67] 3-Styrylpyridazine likewise gave the 3-aldehyde (< 20%).[77]

3,6-Distyrylpyridazine gave 3,6-pyridazinedicarbaldehyde (AcMe, $O_3\downarrow$, −50°, 60 min: 64%).[1894]



(176)



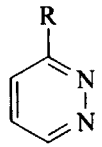
(177)

### Oxidation to Ketones

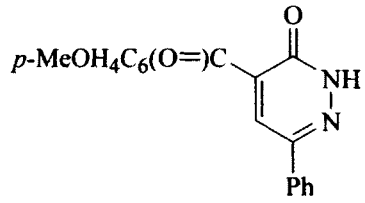
3-Benzylpyridazine (**178**, R = $CH_2$Ph) gave 3-benzoylpyridazine (**178**, R = Bz) ($SeO_2$, AcOH, 100°, 1 h: 85%).[1098, 1108]

4-Benzylpyridazine (**177**, R = $CH_2$Ph) gave 4-benzoylpyridazine (**177**, R = Bz) ($SeO_2$, AcOH, 100°, 1 h: 87%; or $KMnO_4$, 1M $H_2SO_4$, 100°, 3.5 h: 51%)[72]

4-*p*-Methoxybenzyl- gave 4-*p*-methoxybenzoyl-6-phenyl-3(2*H*)-pyridazinone (**179**) ($Na_2Cr_2O_7$, AcOH, reflux, 8 h: 53%); also analogues.[1716]



(178)



(179)

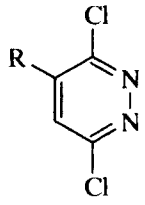
### Oxidation to Carboxylic Acids

3,6-Dichloro-4-methylpyridazine (**180**, R = Me) gave 3,6-dichloro-4-pyridazinecarboxylic acid (**180**, R = $CO_2$H) ($K_2Cr_2O_7$, concentrated $H_2SO_4$, 30°, 10 h; then 25°, 15 h: 83%;[65] similarly, 40°, 2 h: ~75%).[1748]

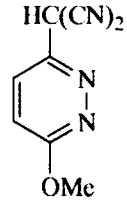
3-Bromo-6-methylpyridazine furnished 6-bromo-3-pyridazinecarboxylic acid ($K_2Cr_2O_7$, concentrated $H_2SO_4$, 50°, 2 h: ~85%).[1127]

3-Dicyanomethyl-6-methoxypyridazine (**181**) gave 6-methoxy-3-pyridazinecarboxylic acid (75% $H_2O_2$, KOH, MeOH, 25°, 24 h: 28%).[74]

Also other examples.[1459]



(**180**)



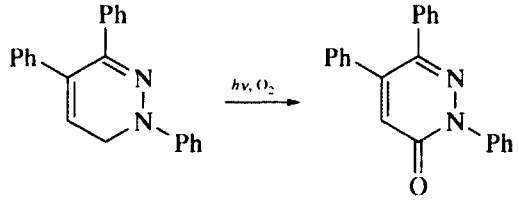
(**181**)

## Miscellaneous Oxidations

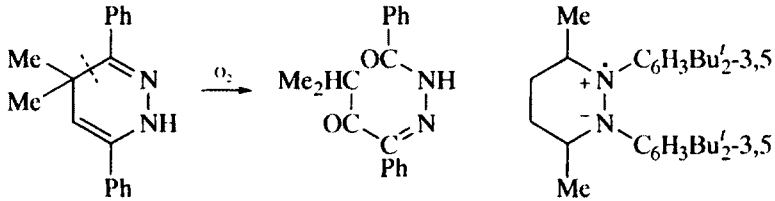
3,6-Dimethylhexahydropyridazine gave 3,6-dimethyl-1,4,5,6-tetrahydropyridazine ($O_2$, Pt, $C_7H_{16}$, 25°, ~30 min: 59%).[5]

1,3,4-Triphenyl-1,6-dihydropyridazine (**182**) gave 2,5,6-triphenyl-3(2*H*)-pyridazinone (**183**) ($O_2$, EtOH, *hν*, 2.5 h: ~25%),[931] whereas 3,6-diphenylpyridazine resisted photooxidation completely.[1265]

In contrast, even recrystallization of 4,4-dimethyl-3,6-diphenyl-1,4-dihydropyridazine (**184**) in the presence of air caused oxidative ring fission to 1-benzoylhydrazono-3-methyl-1-phenylbutan-2-one (**185**).[1029]



(**182**) (**183**)



(**184**) (**185**) (**186**)

Oxidation of *cis*- or *trans*-1,2-bis(3,5-di-*t*-butylphenyl)-3,6-dimethylhexahydropyridazine gave a relatively stable violet radical cation (**186**), characterised in solution by its electron spin resonance (esr) spectrum [$Pb(OAc)_4$, $CH_2Cl_2$–$F_3CCO_2H$, −20°].[251]

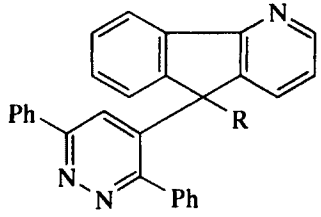
### *3.2.2.2 Extranuclear Alkylation and Alkylidenation*

Simple cases, in which the final alkyl grouping bears no functional substituent, have been discussed in Section 3.2.1.6. Less simple cases, including extranuclear Mannich alkylations, are illustrated in the following examples:

#### Regular Alkylations

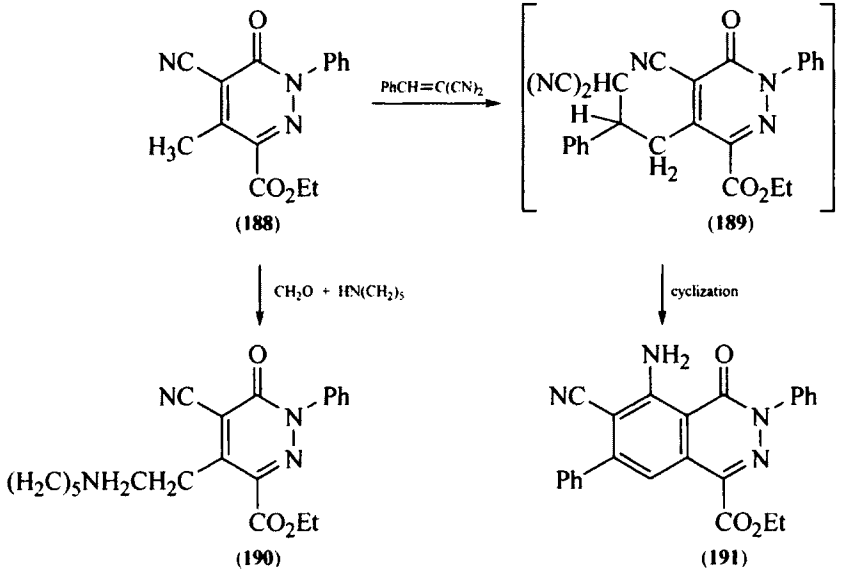
These alkylations appear to be unrepresented by any simple examples.

5-(3,6-Diphenylpyridazin-4-yl)-5*H*-indeno[1,2-*b*]pyridine (**187**, R = H) gave its 5-β-butoxycarbonylethyl derivative (**187**, R = $CH_2CH_2CO_2Bu$) [$BuO_2CCH{=}CH_2$,$Me_2PhHNOEt$ (Rodionov's catalyst), PhH, reflux, 7 h: 11%].[1491]




(**187**)

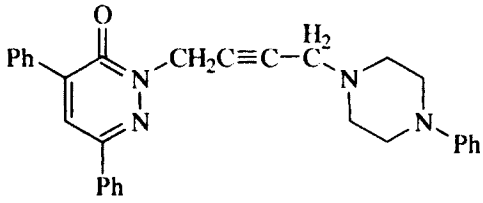
Ethyl 5-cyano-4-methyl-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate (**188**) and benzylidenemalononitrile gave presumably the 4-(3,3-dicyano-2-phenylpropyl) derivative (**189**), which underwent spontaneous cyclization to afford ethyl 5-amino-6-cyano-4-oxo-3,7-diphenyl-3,4-dihydrophthalazine-1-carboxylate (**191**) [trace $HN(CH_2)_5$, EtOH, reflux, 30 min: 70%]; also analogues, somewhat similarly.[505, 1216, 1937]



### Mannich alkylations

The foregoing substrate (**188**) with formalin and piperidine gave ethyl 5-cyano-6-oxo-1-phenyl-4-$\beta$-piperidinoethyl-1,6-dihydro-3-pyridazinecarboxylate (**190**) (MeOH, reflux, 3 h: 80%).[1216]

4,6-Diphenyl-2-prop-2′-ynyl-3(2*H*)-pyridazinone with formalin and 1-phenylpiperazine gave 4,6-diphenyl-2-[4-(4-phenylpiperazin-1-yl)but-2-ynyl]-3(2*H*)-pyridazinone (**192**) [trace CuCl, THF, (conditions?): 38%]; also analogues.[1837]

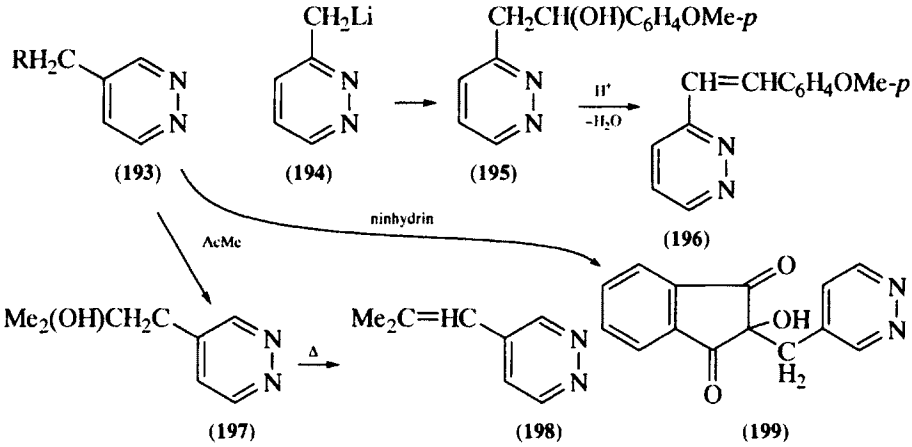


(192)

### Hydroxyalkylations

*Note:* The alkylidenation of an activated methyl- or methylenepyridazine with an aldehyde or ketone gives first an intermediate hydroxyalkylpyridazine that usually loses water spontaneously under the conditions of reaction to afford an alkenylpyridazine. However, the intermediate is sometimes isolable when appropriate reagents are used under gentle conditions, as illustrated in this subsection.

4-(Lithiomethyl)pyridazine (**193**, R = Li) and anhydrous formaldehyde gave 4-$\beta$-hydroxyethylpyridazine (**193**, R = $CH_2OH$) (THF, $-78° \rightarrow 25°$, 1 h: 82%);[1581] 3-$\beta$-hydroxyethylpyridazine (40%) was made similarly.[1581] An earlier procedure using formalin and piperidine[494] appears to have given mainly polymeric material.[1581]



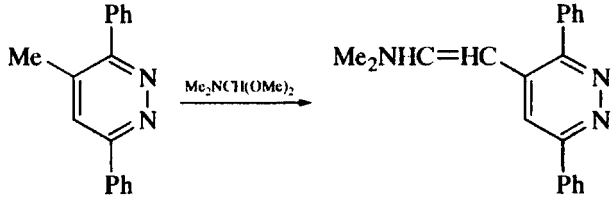
3-Methylpyridazine, as its lithio derivative (**194**), gave 3-(β-hydroxy-*p*-methoxyphenethyl)pyridazine (**195**) (*p*-$MeOC_6H_4CHO$, THF, −20°→25°, 20 h: 68%) and thence 3-*p*-methoxystyrylpyridazine (**196**) (HCl, MeOH, 25°, 7 h: 95%).[1605]

4-Methylpyridazine, as its lithium derivative (**193**, R = Li), and acetone gave 4-(β-hydroxy-β-methylpropyl)pyridazine (**197**) (THF, −70°, $N_2$, 10 min: 92%) and then 4-(2-methylprop-1-enyl)pyridazine (**198**) ($Me_2SO$, 200°, 1 h: 50%); also analogues.[827]

4-Methylpyridazine (**193**, R = H) and ninhydrin (2,2-dihydroxy-1,3-indandione, i.e., the hydrate of 1,2,3-indantrione) gave 4-(2-hydroxy-1,3-dioxoindan-2-ylmethyl)pyridazine (**199**) (xylene, 105°, A, 8 h: 61%) but no subsequent dehydration product.[1910]

## Alkylidenations with Dimethylformamide Dialkyl Acetals

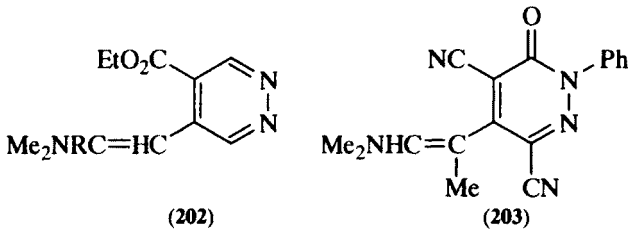
4-Methyl-3,6-diphenylpyridazine (**200**) gave 4-β-dimethylaminovinyl-3,6-diphenylpyridazine (**201**) [neat $Me_2NCH(OEt)_2$, $N_2$, reflux, 24 h: 58%].[43]




(**200**) (**201**)

Ethyl 5-methyl-4-pyridazinecarboxylate gave ethyl 5-β-dimethylaminovinyl-4-pyridazinecarboxylate (**202**, R = H) [neat $Me_2NCH(OMe)_2$, 100°, 4 h: 90%].[1095]

4-Ethyl- gave 4-(β-dimethylamino-α-methylvinyl)-6-oxo-1-phenyl-1,6-dihydro-3,5-pyridazinedicarbinitrile (**203**) [$Me_2NCH(OMe)_2$, $Ac_2O$, reflux, 10 min: 71%].[1808]




(**202**) (**203**)

Ethyl 5-benzyl-4-pyridazinecarboxylate gave ethyl 5-(β-dimethylamino-α-phenylvinyl)-4-pyridazinecarboxylate (**202**, R = Ph) [neat $Me_2NCH(OMe)_2$, 100°, 3 h: 77% (crude)].[1081]

Ethyl 3-methyl-4-pyridaziecarboxylate gave ethyl 3-β-dimethylaminovinyl-4-pyridazinecarboxylate [$Me_2NC(OBu')NMe_2$, $Me_2NCHO$, 110°, $N_2$, 9 h, water-removal trap: 71%].[1678]

Also other examples.[1642, 1830, 1902]

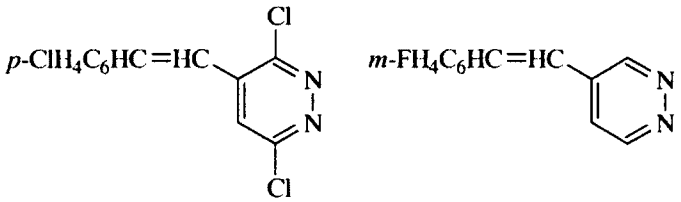
**Alkylidenations with Aromatic Aldehydes**

3,6-Dichloro-4-methylpyridazine gave 3,6-dichloro-4-*p*-chlorostyrylpyridazine (**204**) (*p*-$ClC_6H_4CHO$, $AcOH–H_2SO_4$, reflux, 24 h: 60%).[1739]

4-Methylpyridazine gave 4-*m*-fluorostyrylpyridazine (**205**) (*m*-$FC_6H_4CHO$, NaOH, $Me_2SO–H_2O$, 25°, dark, 6 h: 55%).[1047]

3-Acetamido-6-methylpyridazine and 5-methylsulfonyl-2-furaldehyde gave 3-acetamido-6-[β-(5-methylsulfonylfuran-2-yl)vinyl]pyridazine (**206**) [$AcOH–Ac_2O$, 160° (sealed?), 3 h: 10%].[1259]

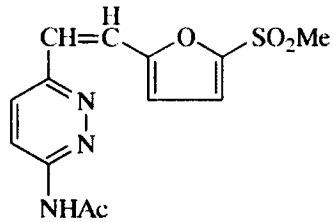
Also other examples.[379, 509, 1217, 1786, 2012, 2118]




(**204**)




(**205**)

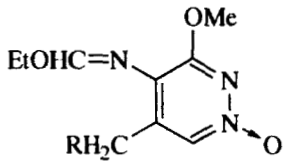



(**206**)

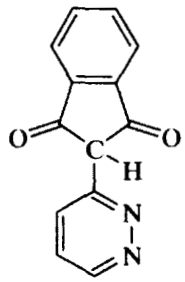
### *3.2.2.3 Extranuclear Acylation*

Extranuclear *C*-acylation of alkylpyridazines is illustrated by the conversion of 4-ethoxymethyleneamino-3-methoxy-5-methylpyridazine 1-oxide (**207**, R = H) into 4-ethoxalylmethyl-5-ethoxymethyleneamino-6-methoxypyridazine 2-oxide (**207**, R = $COCO_2Et$) (90%) by condensation with diethyl oxalate in ethanolic ether containing potassium ethoxide at 25° during 5 h;[902] by the conversion of 3-methylpyridazine into 3-acetonylpyridazine (21%) by initial lithiation of the substrate in THF and subsequent treatment with ethyl acetate at −70° and then at 25° for 3 h;[825] and by a variety of analogous preparations.[537, 825] An interesting diacylation at a single methyl group involved condensation of

3-methylpyridazine with phthalic anhydride at 170° during 3 h to afford 3-(1,3-dioxoindan-2-yl)pyridazine (**208**) in 19% yield.[1756,1775]




(207)



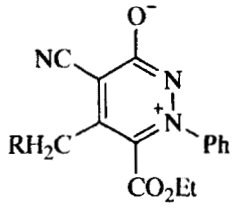

(208)

### 3.2.2.4 *Extranuclear Halogenation, Nitration, and Diazo Coupling*

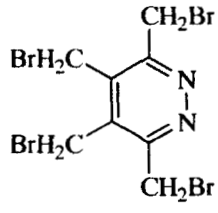
Although alkyl- and arylpyridazines are quite responsive to such electrophilic attack, little use has been made of these reactions in recent years. Typical examples follow; for other such halogenations, see Section 4.3.2:

4-Cyano-6-ethoxycarbonyl-5-methyl- (**209**, R = H) gave 5-bromomethyl-4-cyano-6-ethoxycarbonyl-1-phenylpyridazinium-3-olate (**209**, R = Br) ($Br_2$, AcOH, 25°, 4 h: 81%).[1645]

3,4,5,6-Tetramethylpyridazine gave the unstable 3,4,5,6-tetrakisbromomethylpyridazine (**210**) (*N*-bromosuccinimide, *hν*, $CCl_4$: ~10%).[747]




(209)

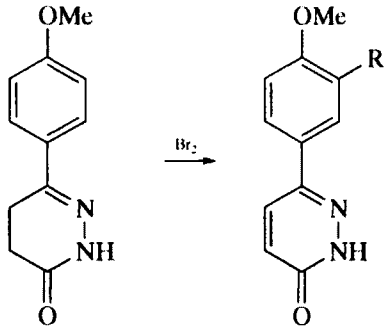



(210)

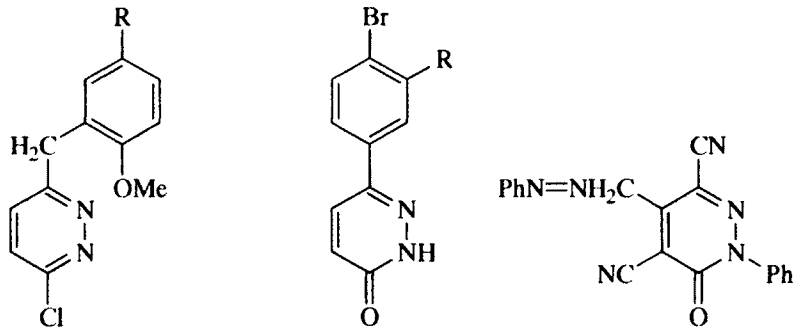
6-*p*-Methoxyphenyl-4,5-dihydro-3(2*H*)-pyridazinone (**211**) gave a separable mixture of 6-*p*-methoxyphenyl-3(2*H*)-pyridazinone (**212**, R = H) (by oxidation) and 6-(3-bromo-4-methoxyphenyl)-3(2*H*)-pyridazinone (**212**, R = Br) (by subsequent bromination) ($Br_2$, AcOH, 60°: 56% and 31%, respectively; optimized?).[919]

3-Chloro-6-*o*-methoxybenzylpyridazine (**213**, R = H) gave 3-chloro-6-(3-iodo-6-methoxybenzyl)pyridazine (**213**, R = I) [$H_2SO_4$, AcOH, I, $(NH_4)_2S_2O_8$; then substrates↓, 50°, 90 min: 84%; best of several procedures].[61]

6-*p*-Bromophenyl-3(2*H*)-pyridazinone (**214**, R = H) gave 6-(4-bromo-3-nitrophenyl)-3(2*H*)-pyridazinone (**214**, R = $NO_2$) ($H_2SO_4$, $HNO_3$, < 0°, 4 h: 76%);[919] also other such nitrations.[1453]




(211) (212)




(213) (214) (215)

The extranuclear nitration of 3-thien-2′-yl-, 4-thien-2′-yl-, and 4-thien-3′-ylpyridazine has been studied in a nonpreparative way.[1205, 1235]

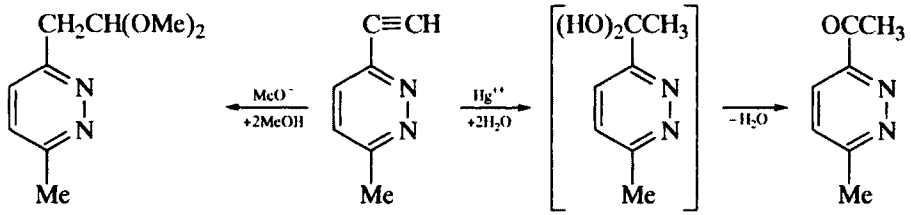
4-Methyl-6-oxo-1-phenyl-1,6-dihydro-3,5-pyridazinedicarbonitrile gave 6-oxo-1-phenyl-4-phenylazomethyl-1,6-dihydro-3,5-pyridazinedicarbonitrile (**215**) (formulated as the 4-phenylhydrazonomethyl tautomer) ($PhN_2Cl$, AcONa, EtOH, < 5°, 1 h: 80%).[395] Also less simple examples, involving the α carbon of an ylide.[1956]

### *3.2.2.5 Miscellaneous Reactions*

Alkylpyridazines undergo a number of other interesting minor reactions, mentioned briefly in the following paragraphs.

*Addition of Methanol* 3-Ethynyl-6-methylpyridazine (**217**) added two molecules of methanol on refluxing in methanolic sodium methoxide

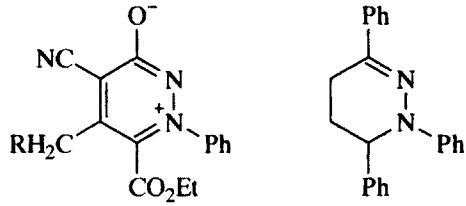
for 5 h to give 3-β,β-dimethoxyethyl-6-methylpyridazine (**216**) in 63% yield;[125] 4-β,β-dimethoxyethyl-3,6-dimethylpyridazine (60%) was made similarly.[125]




(216) (217) (218) (219)

*Addition of Water* In contrast, treatment of 3-ethynyl-6-methylpyridazine (**217**) in refluxing aqueous acetonic mercuric sulfate for 2 h gave 3-acetyl-6-methylpyridazine (**219**) (58%), presumably via the dihydroxyethyl intermediate (**218**);[125] 4-acetyl-3,6-dimethylpyridazine (22%) was made similarly.[125]

*Addition to Phenyl Isothiocyanate* Addition of the methyl group of 4-cyano-6-ethoxycarbonyl-5-methyl-1-phenylpyridazinium-3-olate (**220**, R = H) to the N:C bond of phenyl isothiocyanate (in refluxing pyridine during 3 h) afforded 4-cyano-6-ethoxycarbonyl-1-phenyl-5-*N*-phenyl(thiocarbamoyl)methylpyridazinium-3-olate [**220**, R = C(=S)NHPh] in 90% yield.[1645]

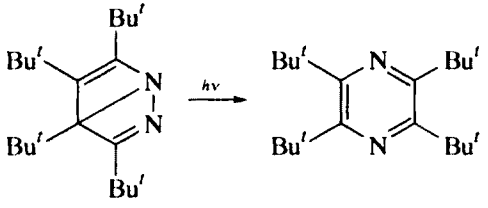



(220) (221)

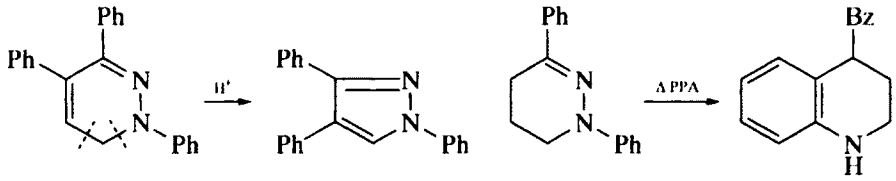
*Nuclear Reduction.* Hydrogenation of 1,3,6-triphenyl-1,4-dihydropyridazine in ethyl acetate over palladium gave the corresponding 1,4,5,6-tetrahydro derivative (**221**) (53%);[1541] also others.[1204]

*Conversion into Other Heterocyclic Systems* The irradiation of 3,4,5,6-tetra-*t*-butylpyridazine gave first the Dewar isomer, 3,4,5,6-tetra-*t*-butyl-1,2-diazabicyclo[2.2.0]hexa-2,5-diene (**222**) (characterized by nmr, ir, and ms), and then 2,3,5,6-tetra-*t*-butylpyridazine (**223**), isolated in 18% yield.[1438] 1,3,4-Triphenyl-1,6-dihydropyridazine (**224**) underwent a complicated transformation (in dioxane-hydrochloric acid at 25° during 24 h) probably involving ring fission, addition of water, recyclization, loss of formaldehyde, and final oxidation to 1,3,4-triphenylpyrazole (**225**) in ~30% yield.[931] 1,3-Diphenyl-1,4,5,6-tetrahydropyridazine (**226**) was converted into 4-benzoyl-1,2,3,4-tetrahydroquinoline (**227**) (35%) and

other minor products on heating in polyphosphoric acid (PPA) at 125°; a likely mechanism was devised.[279]

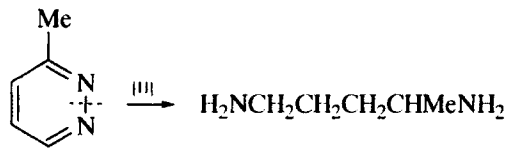


(222) (223)

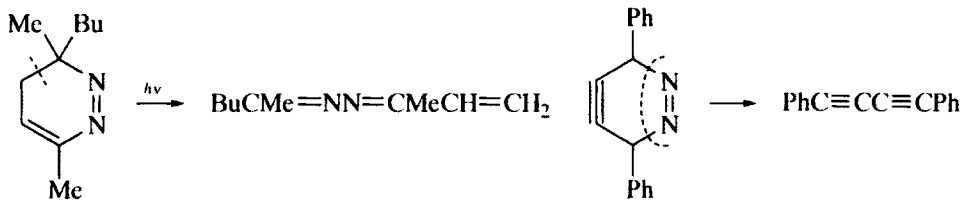


(224) (225) (226) (227)

*Cleavage to Acyclic Products* Reduction of 3-methylpyridazine (**228**) in alkali with NiAl alloy during 3 h afforded 1,4-diaminopentane (**229**), isolated as its dihydrochloride in 56% yield.[348] Irradiation of 3-butyl-3,6-dimethyl-3,4-dihydropyridazine (**230**) in benzene at 10° produced a mixture of the *cis–cis* and *cis–trans* isomers of 4,5-diazadeca-1,3,5-triene (**231**), which isomerized above 20° into the *trans–cis* and *trans–trans* isomers; all were sufficiently stable in solution for nmr characterization.[1203] When 3,6-diphenyl-4,5-didehydropyridazine (**232**) was generated in the vapor phase, it rapidly lost nitrogen to afford diphenylbutadiyne (**233**).[476,669] Thermal and photochemical fragmentations of other hydropyridazines have been studied.[594,598,1661]



(228) (229)



(230) (231) (232) (233)

# 3.3 *N*-ALKYLPYRIDAZINIUM SALTS AND RELATED YLIDES

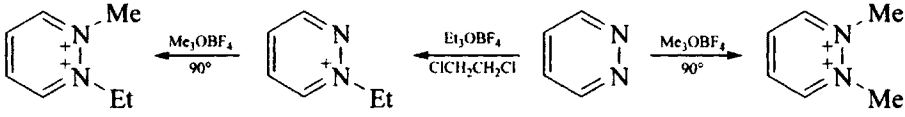
Pyridazine and many of its derivatives are converted easily into the corresponding *N*-alkylpyridazinium salts by treatment with alkyl halides or the like; when the entering alkyl grouping incorporates an electron-withdrawing entity, such as carbonyl, the resulting salt may be deprived of its halide or other gegenion by treatment with a base to afford a pyridazinium ylide, in which the negative charge resides on a carbon atom of the side chain. Pyridazinium salts or ylides undergo few reactions specifically associated with their ionic nature.

## 3.3.1 Preparation of *N*-Alkylpyridazinium Salts

The monoquaternization of simple pyridazines is usually easy, but the formation of diquaternary salts requires vigorous conditions, and yields are poor. The presence of a bulky substituent, especially in the 3 or 6 position adjacent to a ring-nitrogen atom, makes quaternization more difficult or even impossible at the hindered site(s);[138,243] bulky alkyl halides and similar compounds have a similar negative effect on quaternization. The relative rates for methiodide formation from some common unsubstituted azines and diazines have been studied under standardized conditions.[575]

The following examples illustrate the conditions needed for the preparation of typical pyridazinium salts, which may bear functional passenger groups:

Pyridazine (**236**) gave 1-ethylpyridazinium tetrafluoroborate (**235**) ($EtBF_4 \cdot Et_2O$, $ClCH_2CH_2Cl$, reflux, $N_2$, 15 min: 60%) and then 1-ethyl-2-methylpyridazinediium bistetrafluoroborate (**234**) (neat $MeBF_4 \cdot Me_2O$, 90°, $N_2$, 45 min: 4%);[294] pyridazine gave 1,2-dimethylpyridazinediium bistetrafluoroborate (**237**) (neat $MeBF_4 \cdot Me_2O$, 90°, $N_2$, 30 min: 21%).[294]




(**234**) (**235**) (**236**) (**237**)

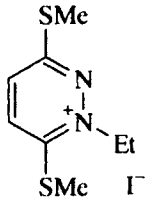
3,6-Bismethylthiopyridazine gave 1-ethyl-3,6-bismethylthiopyridazinium iodide (**238**) (EtI, EtOH, 100°, sealed, 2 h: ~80%).[95]

Pyridazine gave 1-*p*-toluoylmethylpyridazinium bromide (**239**) ($BrCH_2CO C_6H_4Me$-*p*, MeCN, 25°, 24 h: ?%)[1321] or 1-benzylpyridazinium chloride ($PhCH_2Cl$, MeOH, reflux, 1 h: ~70%).[773]

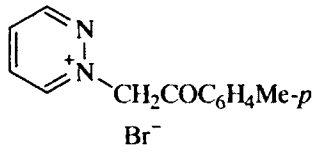
3-Pyrrol-2′-ylpyridazine gave only 1-ethyl-3-pyrrol-2′-ylpyridazinium tetrafluoroborate ($Et_3OBF_4$, $CH_2Cl_2$, 25°, ?h: ?%; identified by X-ray analysis).[1804]

Pyridazine hydrobromide and acrylamide gave 1-β-carbamoylethylpyridazinium bromide (**240**) (EtOH, reflux, 3 h: 65%; note the unusual combination of reagents).[498]

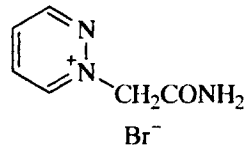
Also other examples.[682, 690, 708, 773, 790, 1435, 1835, 2097]




(238)



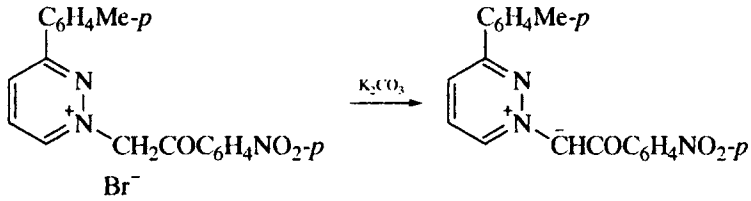

(239)




(240)

### 3.3.2 Preparation of Pyridazinium Ylides

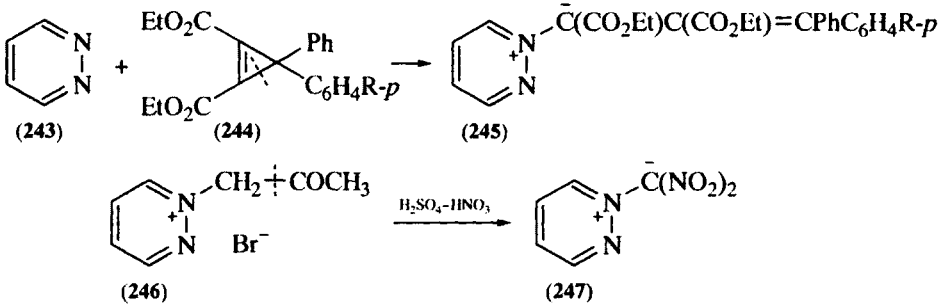
Pyridazinium ylides are usually made from appropriate pyridazinium salts (by simply treating a solution with base) and then promptly used without characterization, often for the formation of adducts with acetylenic or ethylenic reagents (see Sections 3.3.3 and 3.3.4).[708, 714, 773, 777, 1413, 1435] However, 3-*p*-tolylpyridazinium-1-(*p*-nitrobenzoylmethylide) (**242**), from 1-*p*-nitrophenacyl-3-*p*-tolylpyridazinium bromide (**241**), has been reported as stable for several weeks in the absence of light and moisture.[638]




(241) (242)

Properly characterized pyridazinium ylides have been made in other ways. Thus pyridazine (**243**) reacted with diethyl 3,3-diphenylcyclopropene-1,2-dicarboxylate (**244**, R = H) in ether at 25° during < 36 h to give pyridazinium-1-(1,2-diethoxycarbonyl-3,3-diphenylprop-2-enylide) (**245**, R = H) (58%), of X-ray proven structure;[22] appropriately substituted cyclopropenes likewise gave several analogues such as pyridazinium-1-(1,2-diethoxycarbonyl-3-*p*-nitrophenyl-3-phenylprop-2-enylide) (**245**, R = $NO_2$) (36%);[22] and tetracyanoethylene oxide afforded products such as pyridazinium-1-dicyanomethylide (70%) or its 3-methoxy derivative (83%).[756, 1949] Quite differently, treatment of a solution of

1-acetonylpyridazinium bromide (**246**) in concentrated sulfuric acid at < 10° with fuming nitric acid and subsequent warming to 60° for 30 min afforded pyridazinium-1-dinitromethylide (**247**) in 20% yield;[1653] according to kinetic data, successive dinitration at the methylene group occurred prior to removal of the acetyl residue.[1653] Other examples have been reported.[681,1828]

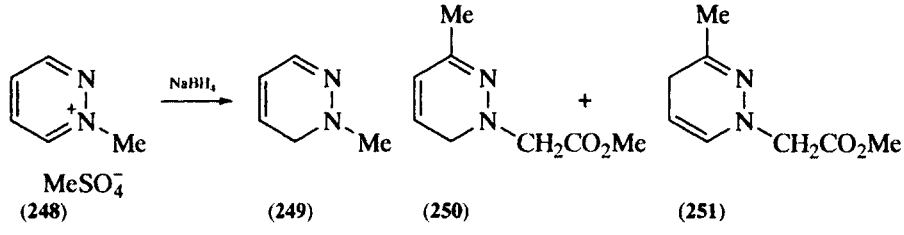



### 3.3.3 Reactions of *N*-Alkylpyridazinium Salts

Apart from the conversion of appropriate *N*-alkylpyridazinum salts into the corresponding ylides (see Section 3.3.2), such salts undergo only a few reactions of interest, exemplified here:

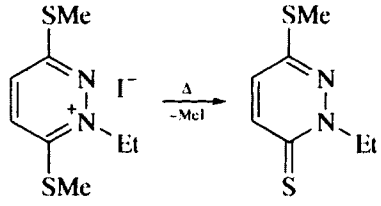
#### Reduction

The reduction of (crude) methyl 1-methylpyridazinium sulfate (**248**) with sodium borohydride in water below 5° for 5 h furnished 1-methyl-1,6-dihydropyridazine (**249**) in 46% yield;[790] analogues, such as 1,3-dimethyl-1,6-dihydropyridazine (63%) or 3-methoxy-1-methyl-1,6-dihydropyridazine (58%), were made similarly.[790] Moreover, both quaternization and reduction may be done at the same time: for example, treatment of a mixture of 3-methylpyridazine and sodium borohydride in tetrahydrofuran at < 5° with methyl chloroformate during 3 h gave a separable mixture of methyl 3-methyl-1,6-dihydro-1-pyridazinecarboxylate (**250**) and its 1,4-dihdyro isomer (**251**) in 43% and 8% yield, respectively.[790]



### Hilbert–Johnson Reaction

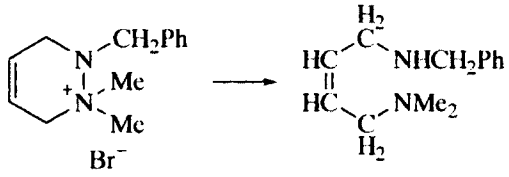
A thio version of the Hilbert–Johnson reaction[1930] has been reported: thermolysis of 1-ethyl-3,6-bismethylthiopyridazinium iodide (**252**) in refluxing *t*-butyl alcohol during 18 h gave 2-ethyl-6-methylthio-3(2*H*)-pyridazinethione (**253**) (~60%) with loss of methyl iodide.[95]




(**252**) (**253**)

### Ring Fission

2-Benzyl-1,1-dimethyl-1,2,3,6-tetrahydropyridazinium bromide (**254**) appears to have suffered rupture of the N–N bond to afford 1-benzylamino-4-dimethylaminobut-2-ene (**254a**) (35%) on treatment with base under unspecified conditions.[281]



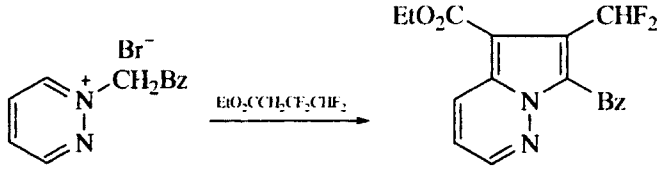

(**254**) (**254a**)

### Association of 1-Methylpyridazinium Ions

An interesting comparison of the association of arenium and *N*-methylheteroarenium ions in ethanol and acetonitrile has been made, using conductometric and uv spectral methods; the 1-methylpyridazinium ion was included in the results.[1891]

### Cyclization

Treatment of 1-phenacylpyridazinium bromide (**255**) with ethyl 3,3,4,4-tetrafluorobutyrate, triethylamine, and potassium carbonate in dimethylformamide at 50° for 6 h afforded ethyl 7-benzoyl-6-difluoromethylpyrrolo[1,2-*b*]pyridazine-5-carboxylate (**255a**) in 75% yield: analogues were made similarly.[2094]



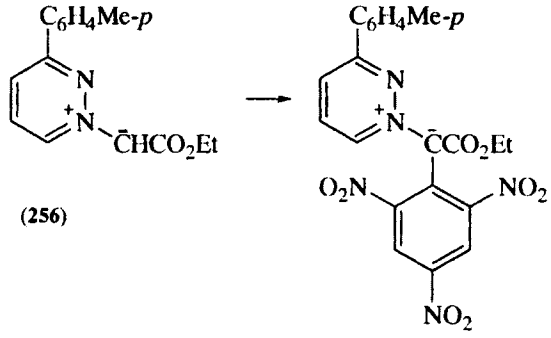

(255) (255a)

### 3.3.4 Reactions of Pyridazinium Ylides

Such ylides undergo side-chain alkylation or arylation, cycloaddition reactions, and photochemical ring-contractions, as illustrated in the following paragraphs:

#### Side-Chain Alkylations or Arylations

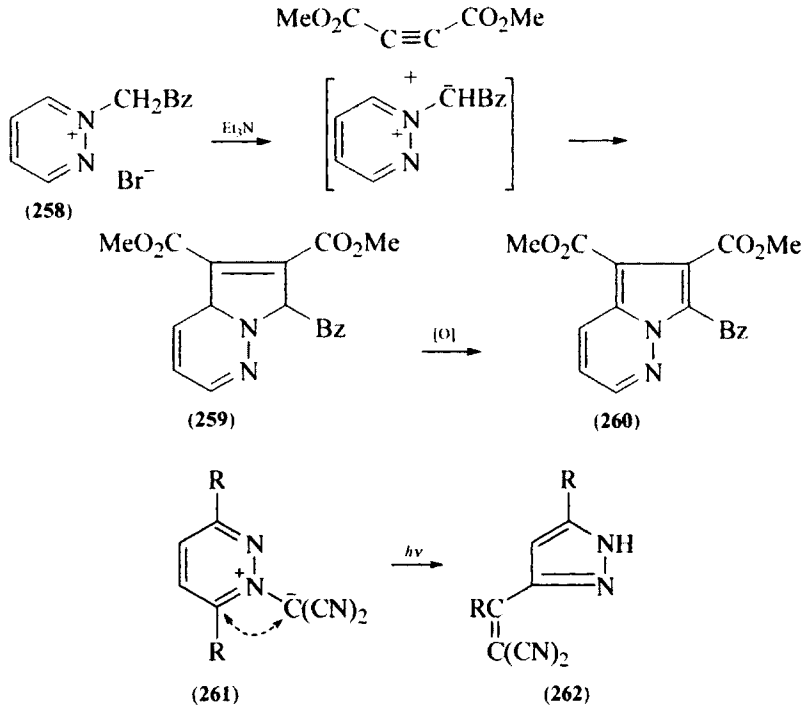
4-*p*-Tolylpyridazinium-1-ethoxycarbonylmethylide (**256**) and picryl chloride gave 3-*p*-tolylpyridazinium-1-(α-ethoxycarbonyl-2,4,6-trinitrobenzylide) (**257**);[708] several analogues were made similarly.[714,732]




(256) (257)

#### Cycloadditions

The required ylide is often made in situ for such cycloadditions, typified in the following integrated procedure. A mixture of 1-phenacylpyridazinium bromide (**258**) and dimethyl acetylenedicarboxylate in dimethylformamide was treated with triethylamine at 25° during 3 h to afford the crude cycloadduct (**259**), which was oxidized by chloranil (tetrachloro-*o*-benzoquinone) in refluxing benzene for 1 h to furnish dimethyl 7-benzoylpyrrolo[1,2-*b*]-pyridazine-5,6-dicarboxylate (**260**) in 84% yield overall;[773] numerous analogues were made in much the same way.[36,195,263,638,704,773,777,1413,1879,1949,2022]




## Ring Contractions

Pyridazinium-1-dicyanomethylide (**261**, R = H) underwent ring contraction to 3-$\beta$,$\beta$-dicyanovinylpyrazole (**262**, R = H) in 15% yield on irradiation in benzene;[474] 3,6-dimethylpyridazinium-1-dicyanomethylide (**261**, R = Me) likewise gave 3-($\beta$,$\beta$-dicyano-$\alpha$-methylvinyl)-5-methylpyrazole (**262**, R = Me) (20%).[474]

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D. J. Brown
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CHAPTER 4

# Halogenopyridazines (*H* 219)

Whether a halogeno substituent occupies the 3/6 or the 4/5 position on a pyridazine, it is strongly activated by one of the ring nitrogen atoms; accordingly, its reactivity resembles that in *o*- or *p*-chloronitrobenzene, although such reactivity may be affected substantially by the presence of any other electron-withdrawing, electron-releasing, or bulkyl group(s) appropriately placed in the molecule. Extranuclear halogeno substituents are little affected by the pyridazine nucleus, and their reactivities are reasonably predictable. Because of their convenient reactivities, halogenopyridazines have been used extensively as intermediates in pyridazine metatheses; since there is little difference between reactivities of corresponding fluoro-, chloro-, bromo- or iodopyridazines, the more easily available chloro derivatives are the ones normally employed in such processes.

## 4.1 PREPARATION OF NUCLEAR HALOGENOPYRIDAZINES (*H* 221)

Apart from those nuclear halogenopyridazines made by *primary syntheses* (see Chapters 1 and 2), most such chloropyridazines have been made by the action of a phosphorus chloride on (tautomeric) pyridazinones, whereas most such fluoro-, bromo-, and iodopyridazines have been made either by direct halogenation or from the corresponding chloropyridazines by transhalogenation.

### 4.1.1 Nuclear Halogenopyridazines from Pyridazinones (*H* 221)

Although most such transformations have been done with neat phosphoryl chloride (or bromide), sometimes it appears to have been more effective to use a phosphorus pentahalide of a combination of reagents.

#### *4.1.1.1 Using Neat Phosphoryl Chloride (H 221)*

Many tautomeric pyridazinones react with warm neat phosphoryl chloride, usually over a period of 1–3 h, to afford good yields of the corresponding chloropyridazines; phosphoryl bromide is less readily available and (as it is a

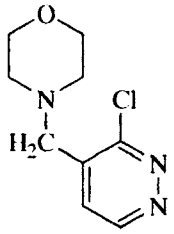
solid at room temperature) less convenient to use (see Section 4.1.1.5). The formation of the following chloropyridazines, in each case from the corresponding pyridazinone or pyridazinedione unless otherwise indicated, illustrates the range of conditions required and the yields to be expected in the presence of various passenger groups:

**From Unsubstituted, Alkyl, or Arylpyridazinones**

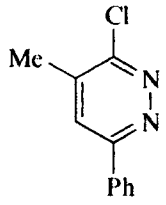
*Note:* Substrates in this group also include (substituted alkyl)- and (substituted-aryl)pyridazinones because such extranuclear substitution rarely affects the reaction.

3-Chloro-4-morpholinomethylpyridazine (**1**) (95°, 1 h: 64%).[769]

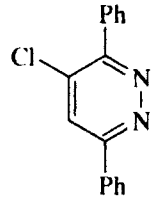
3-Chloro-4-methyl-6-phenylpyridazine (**2**) (80°, 3 h: 95%;[1632] 95°, 1 h: ~90%;[1104] 90°, 6 h: 94%).[1569]



(1)



(2)



(3)

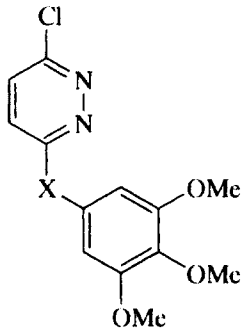
4-Benzyl-3-chloro-6-phenylpyridazine (reflux, 2 h: 71%).[238]

4-Chloro-3,6-diphenylpyridazine (**3**) (reflux, 2 h: 85%).[451]

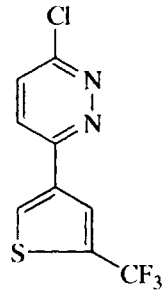
3-Chloro-6-(3,4,5-trimethoxyphenethyl)pyridazine (**4**, X = $CH_2CH_2$) (100°, 1 h: 73%);[1734] likewise, 3-chloro-6-(3,4,5-trimethoxystyryl)pyridazine (**4**, X = CH:CH) (72%).[1734]

3-Chloro-6-(5-trifluoromethylthien-3-yl)pyridazine (**5**) (reflux, 2 h: 86%).[514]

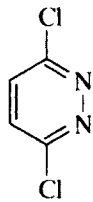
3,6-Dichloropyridazine (**6**) [from 3,6(1*H*,2*H*)-pyridazinedione; 80°, 4.5 h: 85%;[1345] reflux, 5 h: 86–87%;[99,636,1098,cf.1925] 100°, 15 min: ?%].[163]



(4)



(5)



(6)

3,6-Dichloro-4-methoxycarbonylmethylpyridazine (60°, 10 min: 38%).[947]

4,5-Dichloropyridazine [from 5-hydroxy-4(1*H*)-pyridazinone; reflux, 2.5 h: 60%].[923]

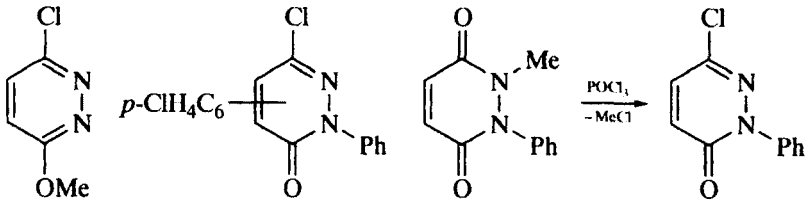
Also many other examples.[152,311,363,446,461,465,467,525,779,837,922,936,961,1034,1039,1064,1067,1082,1263,1267,1268,1276,1278,1282,1309,1339,1394,1411,1423,1455,1462,1557,1574,1727,1746,1871,1959,2069]

## From Pyridazones Bearing Alkoxy or Nontautomeric Oxo Substituents

3-Chloro-5-methoxypyridazine (**7**) (80° until homogeneous: 78%;[1821] reflux, 5 min: 54%).[90]

6-Chloro-4/5-*p*-chlorophenyl-2-phenyl-3(2*H*)-pyridazinone (**8**) (reflux, 3 h: 70%).[462]

6-Chloro-2-phenyl-3(2*H*)-pyridazinone (**10**), in this case from 1-methyl-2-phenyl-3,6(1*H*,2*H*)-pyridazinedione (**9**) with loss of the *N*-methyl group (as MeCl?).[878]




(**7**) (**8**) (**9**) (**10**)

3,4-Dichloro-5-methoxypyridazine (reflux, 15 min: 29%).[90]

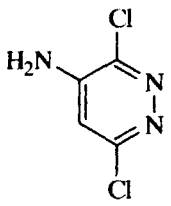
## From Pyridazinones Bearing Amino or Azido Substituents

3,6-Dichloro-4-pyridazinamine (**11**) [120°, sealed, 15 h: 76%; reflux gave only 4-amino-6-chloro-3(2*H*)-pyridazinone].[1228]

3,6-Dichloro-4-piperidinopyridazine (reflux, 30 min: variable yield, lower with prolonged reflux).[641]

4-Azido-6-chloro-3-phenylpyridazine (70°, 30 min: 85%).[133]

Also other examples.[1450]

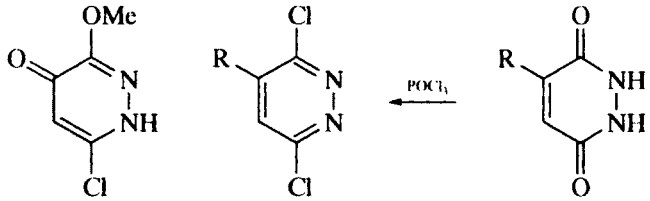



(**11**)

### From Pyridazinones Bearing a Halogeno Substituent

3,5-Dichloro-6-methoxypyridazine [from 6-chloro-3-methoxy-4(1*H*)-pyridazinone (**12**); 80°, until homogeneous (~5 min): 90%];[881] likewise, the isomeric 4,5-dichloro-3-methoxypyridazine (87%).[882]

4-Bromo-3,6-dichloropyridazine (**14**, R = Br) [from 4-bromo-3,6(1*H*,2*H*)-pyridazinedione (**13**, R = Br); reflux, 5 h: 70%];[654, cf., 614, 1925] likewise, 4-fluoro-3,6-dichloropyridazine (**14**, R = F).[917]




(12) (13) (14)

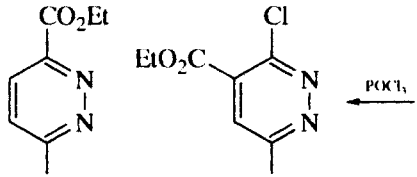
### From Pyridazinones Bearing Ester Groupings

Ethyl 6-chloro-3-pyridazinecarboxylate (**15**) (100°, 2 h: 84%).[1459]

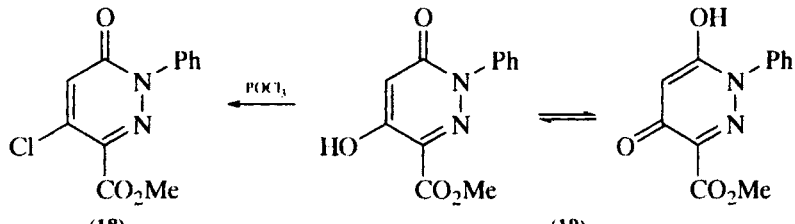
Ethyl 3,6-dichloro-4-pyridazinecarboxylate (**16**) [from ethyl 6-chloro-3-oxo-2,3-dihydro-4-pyridazinecarboxylate (**17**); reflux, 90 min; 82%].[1099]

Methyl 4-chloro-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate (**18**) [from methyl 4-hydroxy-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate (**19**); 95°, 3 h: 91%].[1117]

Also other examples.[759, 1117, 1127, 1689]




(15) (16) (17)

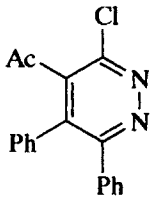



(18) (19)

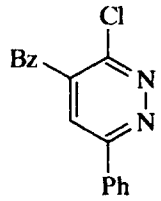
## From Pyridazinones Bearing a Ketonic Substituent

4-Acetyl-3-chloro-5,6-diphenylpyridazine (**20**) (95°, 1 h: 75%).[377]

4-Benzoyl-3-chloro-6-phenylpyridazine (**21**) (95°, 1 h: 56%); also substituted-phenyl analogues likewise.[1716]




(**20**)



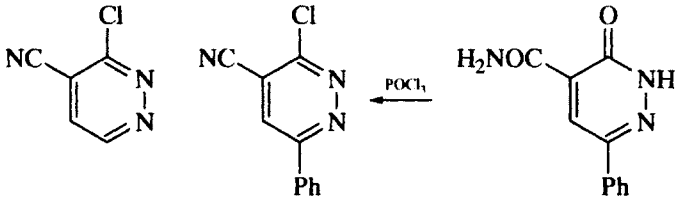

(**21**)

## From Pyridazinones Bearing an Amide or Nitrile Grouping

3-Chloro-4-pyridazinecarbonitrile (**22**) (110°, 1 h: 75%).[820]

3-Chloro-6-phenyl-4-pyridazinecarbonitrile (**23**) [from 3-oxo-6-phenyl-2,3-dihydro-4-pyridazinecarboxamide (**24**); 90°, 3 h: 60%; note additional dehydration of the amide grouping].[1319]

Also other examples.[1056, 1398]



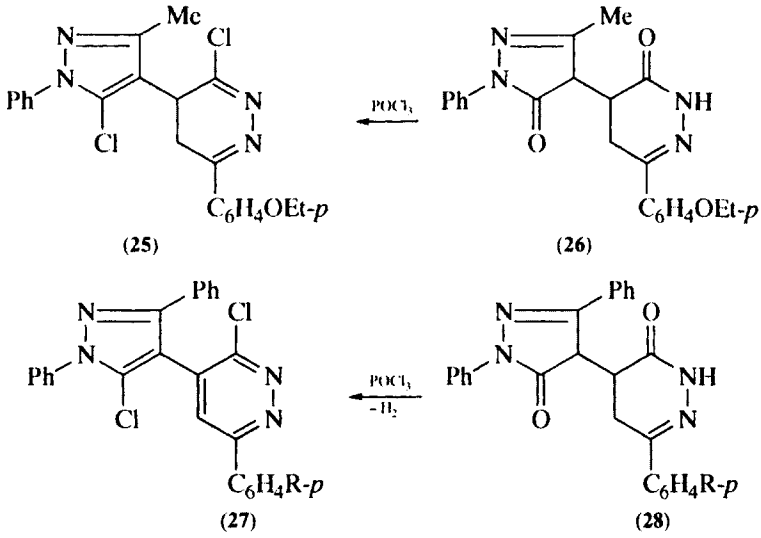

(**22**) (**23**) (**24**)

## From Pyridazinones Bearing Oxoheteroaryl Substituents

*Note:* At least some such substrates undergo conversion of both their nuclear and extranuclear oxo substituents by phosphoryl chloride.

3-Chloro-4-(3-chloro-5-methyl-2-phenylpyrazol-4-yl)-6-*p*-ethoxyphenyl-4,5-dihydropyridazine (**25**) [from 6-*p*-ethoxyphenyl-4-(3-methyl-5-oxo-1-phenyl-2-pyrazolin-4-yl)-4,5-dihydro-3(2*H*)-pyridazinone (**26**); reflux, 30 min: 45%].[1371]

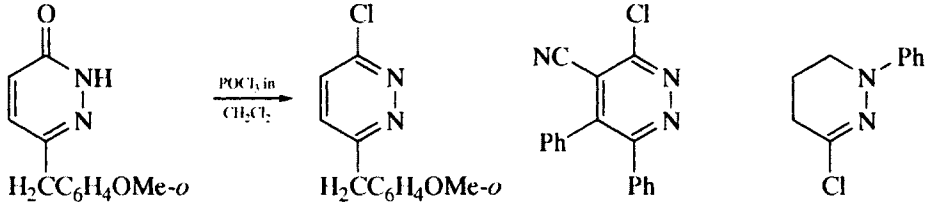
In contrast, 3-chloro-4-(3-chloro-2,5-diphenylpyrazol-4-yl)-6-*p*-ethoxyphenylpyridazine (**27**, R = OEt; not the dihydro derivative akin to **26**) [from 6-*p*-ethoxyphenyl-4-(5-oxo-1,3-diphenyl-2-pyrazolin-4-yl)-4,5-dihydro-3(2*H*)-pyridazinone (**28**, R = OEt); reflux, 30 min: 44%; note concomitant dehydrogenation];[1103] also the 6-*p*-chlorophenyl (**27**, R = Cl) (45%) and 6-*p*-tolyl analogue (**27**, R = Me) (43%) likewise.[1373]




(25) (26) (27) (28)

### *4.1.1.2 Using Phosphoryl chloride in a Solvent*

No reasons have been given for the occasional use of phosphoryl chloride in an inert solvent to convert pyridazinones into the corresponding chloropyridazines. However, 6-*o*-methoxybenzyl-3(2*H*)-pyridazinone (**29**) gave 3-chloro-6-*o*-methoxybenzylpyridazine (**30**) in 87% yield by refluxing in phosphoryl chloride/dichloromethane for 1 h;[750] 3-oxo-5,6-diphenyl-2,3-dihydro-4-pyridazinecarbonitrile gave 3-chloro-5,6-diphenyl-4-pyridazinecarbonitrile (**31**) in 63% yield on refluxing in phosphoryl chloride/dioxane for 3 h;[1726] and 1-phenyl-1,4,5,6-tetrahydro-3(2*H*)-pyridazinone gave 3-chloro-1-phenyl-1,4,5,6-tetrahydropyridazine (**32**) in 61% yield on warming with phosphoryl chloride/toluene at 80° for 1 h.[60]




(29) (30) (31) (32)

### *4.1.1.3 Using Phosphorus Pentachloride in Phosphoryl Chloride (H 224)*

Warming pyridazinones with phosphorus pentachloride in phosphoryl chloride remains a quite popular route to the correspinding chloropyridazines.

However, in most cases the phosphorus pentachloride makes little difference to the desired reaction, whereas it does introduce the real possibility of additional *C*-chlorination, especially if the temperature is too high or the reaction time is prolonged. Accordingly, it would appear that phosphorus pentachloride should not be added unless phosphoryl chloride alone has proved inadequate.

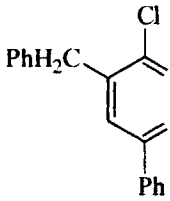
The following examples of the use of phosphorus pentachloride in phosphoryl chloride are divided into two groups; those in which additional *C*-chlorination has not been observed (or reported), and those in which *C*-chlorination has proved appreciable:

### Without Additional C-Chlorination

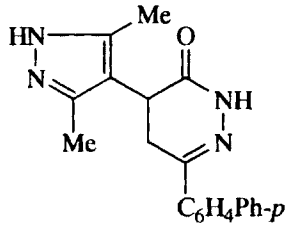
4-Benzyl-6-phenyl-3(2*H*)-pyridazinone gave 4-benzyl-3-chloro-6-phenylpyridazine (**33**) (95°, 3 h: 60%);[1251] 3-chloro-6-*p*-chlorophenylpyridazine likewise (70%).[1266]

6-Biphenyl-4′-yl-4-(3,5-dimethylpyrazol-4-yl)-4,5-dihydro-3(2*H*)-pyridazinone (**34**) gave 3-biphenyl-4′-yl-6-chloro-5-(3,5-dimethylpyrazol-4-yl)pyridazine (**35**), (95°, 3 h: 56%; note the concomitant dehydrogenation of the pyridazine ring).[1712]

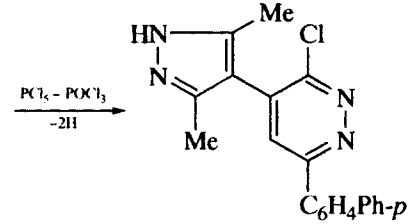
Also other examples.[915, 1022, 1046, 1474, 1703, 1711, 1871]




(33)




(34)



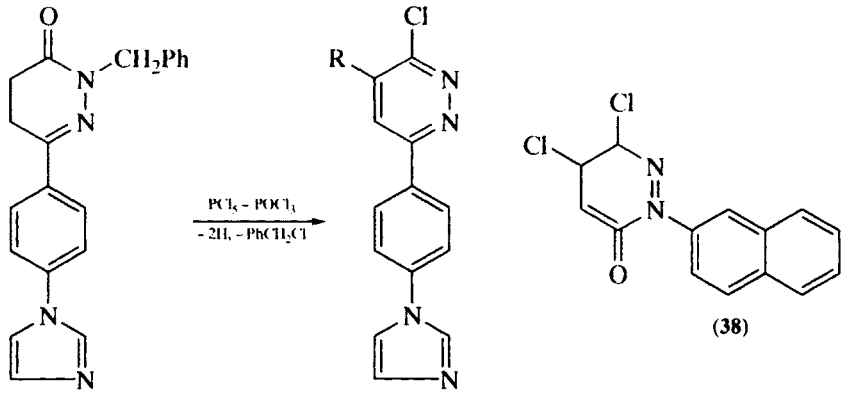

(35)

### With Additional C-Chlorination

2-Benzyl-6-[*p*-(imidazol-1-yl)phenyl]-4,5-dihydro-3(2*H*)-pyridazinone (**36**) gave a separable mixture of 3-chloro- (**37**, R = H) and 3,4-dichloro-6-[*p*-(imidazol-1-yl)phenyl]pyridazine **37**, R = Cl) (reflux, 24 h: <15% and 15%, respectively; note loss of the benzyl group as $PhCH_2Cl$ (?) and dehydrogenation).[1073]

1-Naphthalen-2′-yl-3,6(1*H*,2*H*)-pyridazinedione gave 5,6-dichloro-2-naphthalen-2′-yl-3(2*H*)-pyridazinone (**38**) (reflux, ?h: 41%).[726]

Also other exmples, including some in which two extra chloro substituents were introduced under sufficiently vigorous conditions.[784,787,871,938]

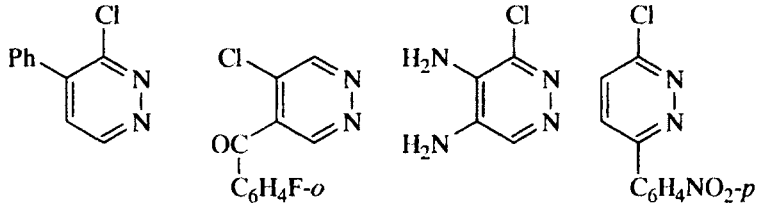



### *4.1.1.4 Using Phosphoryl Chloride and a Tertiary Base*

Although this procedure has been used extensively in the pyrimidine series,[1930b] it has not been employed much to convert pyridazinones into chloropyridazines. However, the following examples do indicate that it is highly effective and may even be the method of choice in the presence of passenger amino groups:

4-Phenyl-3(2*H*)-pyridazinone gave 3-chloro-4-phenylpyridazine (**39**) ($POCl_3$, pyridine, reflux, 2h: 94%).[1408]

5-*o*-Fluorobenzoyl-4(1*H*)-pyridazinone gave 4-chloro-5-*o*-fluorobenzoyl-pyridazine (**40**) ($POCl_3$, pyridine, 60°, 45 min: 50%).[240]




4,5-Diamino-3(2*H*)-pyridazinone gave 3-chloro-4,5-pyridazinediamine (**41**) ($POCl_3$, $Me_2NPh$, reflux, 10h: 50%).[973]

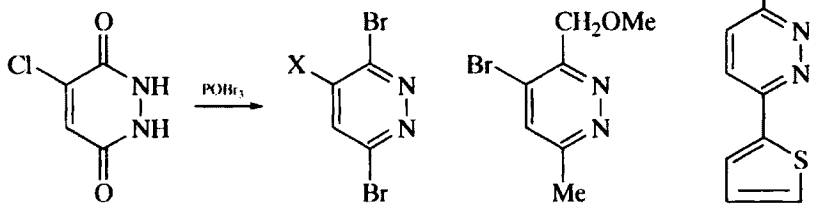
6-*p*-Nitrophenyl-3(2*H*)-pyridazinone gave 3-chloro-6-*p*-nitrophenylpyridazine (**42**) ($POCl_3$, $Me_2NPh$, reflux, 7h: 95%).[1186]

Also other examples.[963]

### *4.1.1.5 Using Phosphoryl Bromide or Phosphorus Pentabromide (H 233)*

Little use has been made of the bromides of phosphorus to convert pyridazinones into bromopyridazines. This is probably because phosphoryl bromide is less readily available and less convenient to use (because it is a solid) than is the corresponding chloride; moreover, the useful device[1930c] of using phosphoryl bromide or phosphorus pentabromide in (liquid) phosphorus tribromide or in an inert solvent appears to have been neglected in the pyridazine series. Present practice is illustrated in the following examples:

4-Chloro-3,6(1*H*,2*H*)-pyridazinedione (**43**) gave 3,6-dibromo-4-chloropyridazine (**44**, X = Cl) (neat $PBr_5$, 95°, 8 h: 74%) or 3,4,6-tribromopyridazine (**44**, R = Br) (neat $POBr_3$, 140°, 3 h: 56%; note additional transhalogenation).[447]




(43) (44) (45) (46)

3-Methoxymethyl-6-methyl-4(1*H*)-pyridazinone gave 4-bromo-3-methoxymethyl-6-methylpyridazine (**45**) (neat $POBr_3$, 35°, 1 h: 20%).[446]

6-Thien-2′-yl-3(2*H*)-pyridazinone gave 3-bromo-6-thien-2′-ylpyridazine (**46**) ($POBr_3$, solvent (?), "warmed gently," 1 h; then reflux, 1 h: 66%).[1831]

Also other examples.[465]

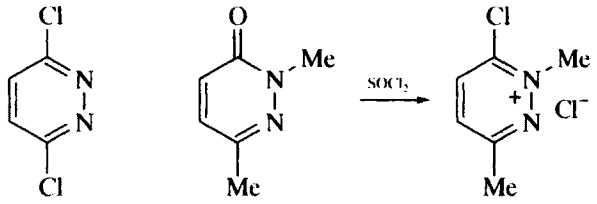
### *4.1.1.6 Using Other Reagents*

Several other reagents have been used occasionally to convert pyridazinones into halogenopyridazines, as illustrated in the following examples:

#### Thionyl chloride

3,6(1*H*,2*H*)-Pyridazinedione gave 3,6-dichloropyridazine (**47**) [$SOCl_2$, (9 equiv), $MeSO_3H$ (3 equiv), reflux, 48 h: 90%; without $MeSO_3H$, no reaction].[1196]

2,6-Dimethyl-3(2*H*)-pyridazinone (**48**) gave 6-chloro-1,3-dimethylpyridazinium chloride (**49**) ($SOCl_2$, $CHCl_3$, reflux, 2 h: 82%).[115]



(47) (48) (49)

## Vilsmeier Reagent

4-Bromo-2-*p*-methoxyphenyl-1-methyl-3,6(1*H*,2*H*)-pyridazinedione (**50**) gave 4,6-dichloro-2-*p*-methoxyphenyl-3(2*H*)-pyridazinone (**51**) with loss of MeCl ($SO_2Cl$/$Me_2NCHO$, 25°, ? h; note concomitant trans-halogenation).[862]

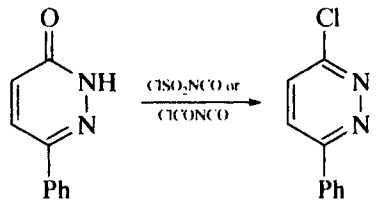


(50) (51)

Somewhat similarly, 1-*p*-bromophenyl-2-methyl-5-morpholino-3,6(1*H*,2*H*)-pyridazinedione gave 2-*p*-bromophenyl-4,6-dichloro-3(2*H*)-pyridazinone ($POCl_3$/$Me_2NCHO$, 150°, 30 min: 70%; note loss of MeCl and displacement of the morpholino by chloro substituent) or the expected product, 2-*p*-bromophenyl-6-chloro-4-morpholino-3(2*H*)-pyridazinone (neat $POCl_3$, reflux, 1 h: 64%; loss of MeCl only).[985]

## Chloroacyl Isocyanates

6-Phenyl-3(2*H*)-pyridazinone (**52**) gave 3-chloro-6-phenylpyridazine (**53**) ($ClSO_2NCO$, MeCN, 55°, 3.5 h, then 25°, 12 h: ~68%;[59] ClCONCO,



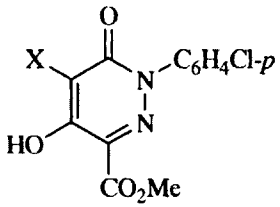
(52) (53)

likewise: 71%);[1222] also four substituted-phenyl analogues in comparable yields.[59,1222]

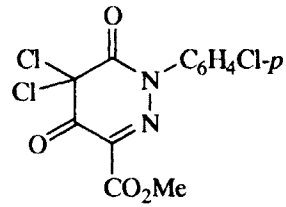
### 4.1.2 Nuclear Halogenopyridazines by Direct Halogenation (*H* 228, 236)

The direct halogenation of pyridazines, usually at the 4/5 position(s), can clearly occur by several well-known mechanisms, although it is often difficult to determine which of these is involved in any particular case. Accordingly, no attempt has been made to classify the following typical examples according to their mechanisms:

Methyl 1-*p*-chlorophenyl-4-hydroxy-6-oxo-1,6-dihydro-3-pyridazinecarboxylate (**54**, X = H) gave its 5-bromo derivative (**54**, X = Br) ($Br_2$, EtOH, 25°, 5 min: 85%), its 5-chloro derivative (**54**, X = Cl) ($SO_2Cl_2$, dioxane, 50° → 65°, 10 min: 91%), or methyl 5,5-dichloro-1-*p*-chlorophenyl-4,6-dioxo-1,4,5,6-tetrahydro-3-pyridazinecarboxylate (**55**) (excess, $Cl_2\downarrow$, $CH_2Cl_2$, 25°, 30 min: 90%).[1549]



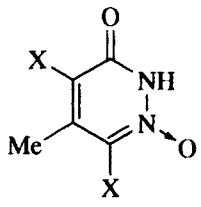
(54)



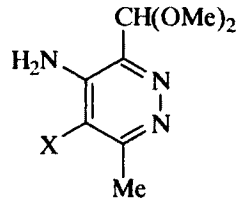
(55)

5-Methyl-3(2*H*)-pyridazinone 1-oxide (**56**, X = H) gave its 4,6-dichloro derivative (**56**, X = Cl) ($Cl_2\downarrow$, $H_2O$, 25°: ~80%);[833] also analogous examples.[833]

4-Amino-6-methyl-3-pyridazinecarbaldehyde dimethyl acetal (**57**, X = H) gave its 5-bromo derivative (**57**, X = Br) ($Br_2$, $CCl_4$, reflux, 30 min: 93%).[996]

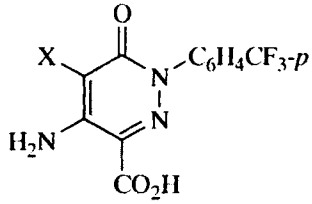


(56)



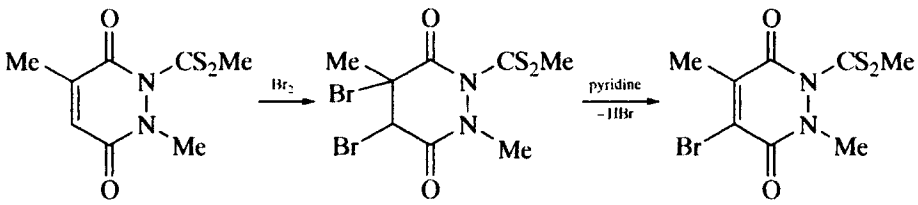
(57)

4-Amino-6-oxo-1-*p*-(trifluoromethyl)phenyl-1,6-dihydro-3-pyridazinecarboxylic acid (**58**, X = H) gave its 5-iodo derivative (**58**, X = I) (I, $Na_2CO_3$, $H_2O$–dioxane, reflux, briefly: 92%);[1117] also analogues likewise.[1117].



(58)

Methyl 2,5-dimethyl-3,6-dioxo-1,2,3,6-tetrahydro-1-pyridazinecarbodithioate (**59**) gave methyl 4,5-dibromo-2,5-dimethyl-3,6-dioxo-1,2,3,4,5,6-hexahydro-1-pyridazinecarbodithioate (**60**) ($Br_2$, $CHCl_3$, reflux: 76%) and thence methyl 4-bromo-2,5-dimethyl 3,6-dioxo-1,2,3,6-tetrahydro-1-pyridazinecarbodithioate (**61**) (pyridine, $CHCl_3$: 66%).[1460]



(59) (60) (61)

3-Butyl-3,6-dimethyl-2,3,4,5-tetrahydropyridazine gave 3-bromo-6-butyl- or 3-butyl-6-chloro-3,6-dimethyl-3,4,5,6-tetrahydropyridazine (Bu$^t$OBr, PhH, 10° → 25°, briefly: 60–70%; or Bu$^t$OCl, likewise: 90%; in each case, a mixture of stereoisomers).[1201]

6-Methoxy-2-*m*-methoxyphenyl-3(2*H*)-pyridazinone gave its 4-chloro derivative ($PCl_5$–$POCl_3$);[851] for other such chlorinations, see References 529 and 938 and Section 4.1.1.3.

Also other examples.[447,655,685,703,789,791,794,826,984,1225]

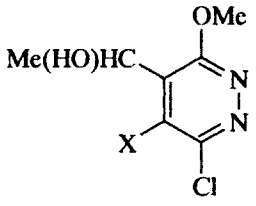
For examples of a rarely used route involving *C*-halogenation and deoxygenation of pyridazine *N*-oxides by phosphoryl halides, see Section 5.2.2.

### 4.1.3 Nuclear Halogenopyridazines from Metallopyridazines

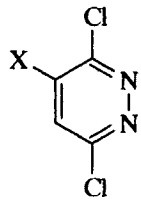
Lithio- and trialkylstannylpyridazines are made easily, and both afford the corresponding halogenopyridazines conveniently on treatment with elemental halogen. Although this route has not been used extensively, it is illustrated well by the following examples:

3-Chloro-5-α-hydroxyethyl-6-methoxypyridazine (**62**, X = H) gave its 4-lithio derivative (**62**, X = Li) (BuLi, THF, 2,2,6,6-$Me_4$–piperidine, −70° → 0°, 15 min, then substrate ↓, −70° → −30°, 30 min: not isolated) and thence the 4-iodo derivative (**62**, R = I) (I, THF, −70°, 1 h: 86% overall);[1610] likewise 3,6-dichloropyridazine (**63**, X = H) gave its lithio

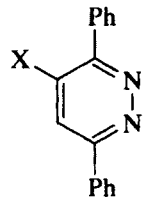
derivative (**63**, R = Li) and then 3,6-dichloro-4-iodopyridazine (**63**, X = I) (32%).[1669]



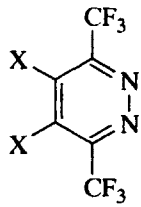

(62)




(63)




(64)




(65)

3,6-Diphenyl-4-tributylstannylpyridazine (**64**, X = $SnBu_3$) (from a primary synthesis) gave its 4-iodo analogue (**64**, X = I) (I, THF, 25°, 24 h: 77%).[1591]

3,6-Bistrifluoromethyl-4,5-bistrimethylstannylpyridazine (**65**, X = $SnMe_3$) (by a primary synthesis: 78%) gave 4,5-dichloro- (**65**, X = Cl) ($Cl_2$, $CCl_4$, 196° → 25°, sealed, 4 days: 82%) or 4,5-diiodo-3,6-bistrifluoromethylpyridazine (**65**, X = I) (I, $CCl_4$, 70°, 3 days: 85%).[696]

Also other examples.[2093]

### 4.1.4 Nuclear Halogenopyridazines from Nitropyridazines

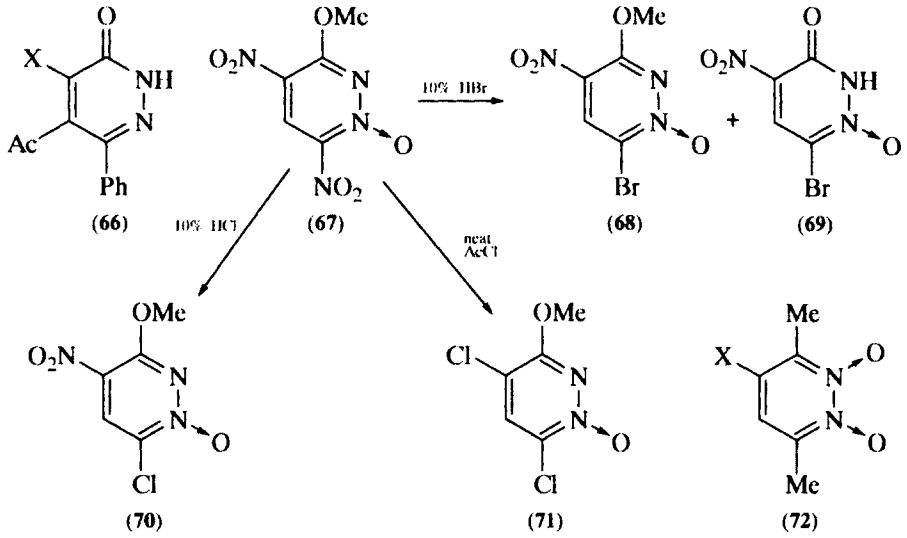
The nitro substituent(s) of some nitropyridazines or their *N*-oxides can act as leaving groups in favor of chloro or bromo substituents when treated with an appropriate hydrohalide, phosphorus halide, Vilsmeier reagent, or acyl halide. This replcement has not been studied systematically, but the following examples indicate its potential utility:

5-Acetyl-2-methyl-4-nitro-6-phenyl-3(2*H*)-pyridazinone (**66**, X = $NO_2$) gave 5-acetyl-4-chloro- (**66**, X = Cl) (6M HCl, AcMe, reflux, 90 min: 65%;[1766] $PhCH_2Et_3NCl$, MeCN, reflux, 1 h: >90%)[1561] or 5-acetyl-4-bromo-2-methyl-6-phenyl-3(2*H*)-pyridazinone (**66**, X = Br) (48% HBr, AcMe, reflux, 15 min: 71%).[1766]

3-Methoxy-4,6-dinitropyridazine 1-oxide (**67**) gave a separable mixture of 3-bromo-6-methoxy-5-nitropyridazine 2-oxide (**68**) and 6-bromo-4-nitro-3(2*H*)-pyridazinone 1-oxide (**69**) (10% HBr, 90°, 3 h: 20% and 4%, respectively, after separation); 3-chloro-6-methoxy-5-nitropyridazine 2-oxide (**70**) only (10% HCl, 90°, 3 h: 40%); or 3,5-dichloro-6-methoxypyridazine 2-oxide (**71**) (neat AcCl, reflux, 3 h: 51%).[905]

3,6-Dimethyl-4-nitropyridazine 1,2-dioxide (**72**, X = $NO_2$) gave 4-chloro-3,6-dimethylpyridazine 1,2-dioxide (**72**, X = Cl) (10M HCl, 95°, 7 h: 35%; HCl gas, trace urea, EtOH, reflux, 4.5 h: 80%; neat AcCl, 50°, 2 h: 39%; or neat $POCl_3$, 95°, 5 h: 8%) or 4-bromo-3,6-dimethylpyridazine 1,2-dioxide (**72**, X = Br) (47% HBr, 95°, 7 h: 21%).[796]

Also other examples.[794,833,1938]



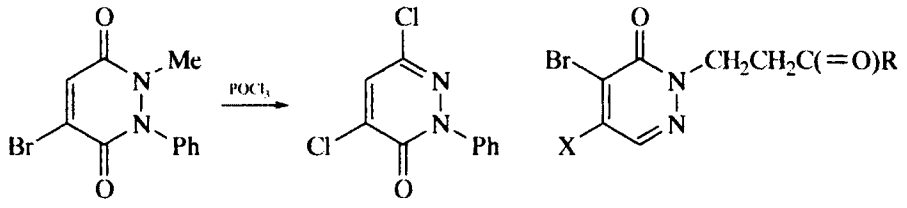

## 4.1.5 Nuclear Halogenopyridazines by Transhalogenation (*H* 240, 241)

The replacement of one halogeno substituent by another is a useful way to make halogenopyridazines, especially the less accessible fluoro and iodo derivatives. The following examples illustrate procedures recently employed:

### Bromo- to chloropyridazines

4-Bromo-1-methyl-2-phenyl-3,6(1*H*,2*H*)-pyridazinedione (**73**) gave 4,6-dichloro-2-phenyl-3(2*H*)-pyridazinone (**74**) ($POCl_3$).[878]

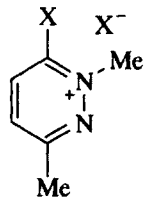
4,5-Dibromo-2-β-carboxyethyl-3(2*H*)-pyridazinone (**75**, R = OH, X = Br) gave 4-bromo-2-β-carbamoylethyl-5-chloro-3(2*H*)-pyridazinone (**75**, R = $NH_2$, X = Cl) ($SOCl_2$, PhH, reflux, 4 h; then $NH_4OH$).[587]



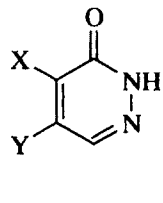

### Chloro- to bromopyridazines

6-Chloro-1,3-dimethylpyridazinium chloride (**76**, X = Cl) gave 6-bromo-1,3-dimethylpyridazinium bromide (**76**, X = Br) (HBr gas↓, $CHCl_3$, 0°, 3 h: 72%).[115]

4,5-Dichloro-3(2*H*)-pyridazinone (**77**, X = Y = Cl) gave 4-bromo-5-chloro- (**77**, X = Br, Y = Cl) (47% HBr, reflux, 2 h: 92%) or 4,5-dibromo-3(2*H*)-pyridazinone (**77**, X = Y = Br) (47% HBr, reflux, 20 h: 90%).[542]



(**76**)



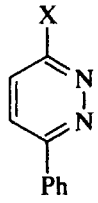
(**77**)

## Chloro- to fluoropyridazines

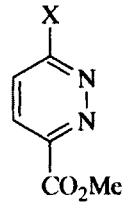
3-Chloro- (**78**, X = Cl) gave 3-fluoro-6-phenylpyridazine (**78**, X = F) (neat $Bu_4PF \cdot HF$, 100°, 2 h: 89%).[1593]

Methyl 6-chloro- (**79**, X = Cl) gave methyl 6-fluoro-3-pyridazinecarboxylate (**79**, X = F) (excess neat KF, 185°, 2 h: <15%).[1127]

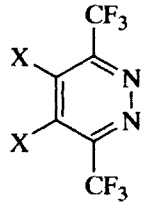
4,5-Dichloro- (**80**, X = Cl) gave 4,5-difluoro-3,6-bistrifluoromethylpyridazine (**80**, X = F) (neat KF, 160°, sealed, 4 days: 94%).[696]



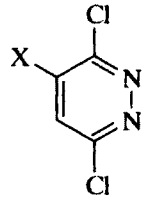
(**78**)



(**79**)



(**80**)



(**81**)

3,6-Dichloro- gave 3,6-difluoropyridazine (neat KF, 250°, sealed, 7 h: 64%)[448] or a 4:6 mixture of 3-chloro-6-fluoro- and 3,6-difluoropyridazine (anhydrous HF↓, 100°, ? h: 92% before separation).[1650]

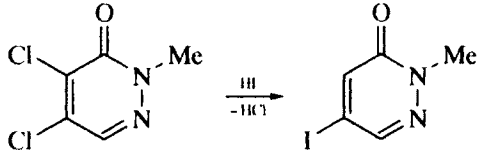
3,4,6-Trichloro- (**81**, X = Cl) gave 3,6-dichloro-4-fluoropyridazine (**81**, X = F) (KF, propylene glycol–MeCN, reflux, 15 h: 59%).[1053]

Also other examples.[459, 1018]

## Chloro- to Iodopyridazines

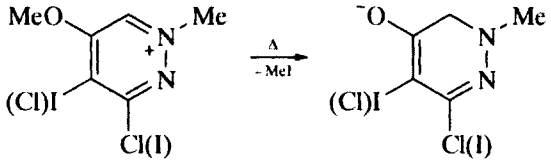
3-Chloro- gave 3-iodopyridazine (NaI, HI, AcMe, 1 h: ?%);[1127] likewise, methyl 6-iodo-3-pyridazinecarboxylate (**79**, X = I) (~40%)[1127] and 4-iodo-3-methoxymethyl-6-methylpyridazine (AcEt, 65°, 2 h: 20%).[446, cf. 1931]

4,5-Dichloro-2-methyl-3(2*H*)-pyridazinone (**82**) gave 5-iodo-2-methyl-3(2*H*)-pyridazinone (**83**) (HI, 150°, 24 h: ?%; note the reductive 4-dechlorination)[1602]



(82) (83)

3,4-Dichloro-5-methoxypyridazine gave 3/4-chloro-4/3-iodo-5-methoxy-1-methylpyridazinium iodide (**84**) (MeI, reflux, 4 h: ~65%) and then 6/5-chloro-5/6-iodo-2-methylpyridazinium-4-olate (**85**) (dioxane, reflux, 3 h: ~90%);[689] also an analogous example.[690]



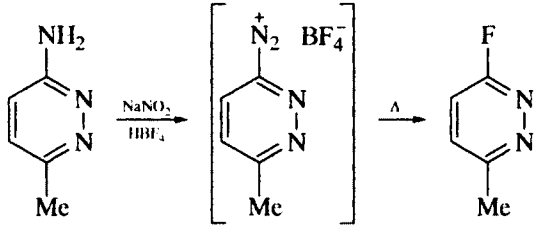
(84) (85)

### Iodo- to fluoropyridazines

3,6-Diiodo- gave 3-fluoro-6-iodopyridazine (KF, $Me_2SO$, 140°, 6 h: 90%).[1385]

## 4.1.6 Nuclear Halogenopyridazines from Pyridazinamines (*H* 227, 235)

This traditional route to halogeno derivatives appears to be represented by only two examples in the recent pyridazine literature. Thus 6-methyl-3-pyridazinamine (**86**) underwent diazotization by sodium nitrite in fluoroboric acid at 0° during 30 min and subsequent warming to 50° for 30 min produced 3-fluoro-6-methylpyridazine (**87**) in ~75% yield;[1127] and diazotization of 6-methylthio-3-pyridazinamine 2-oxide in ~8M hydrochloric acid, followed by the addition of copper powder, gave 3-chloro-6-methylthiopyridazine 2-oxide but in only 7% yield.[679]



(86) (87)

## 4.2 REACTIONS OF NUCLEAR HALOGENOPYRIDAZINES (*H* 245)

Because they undergo nucleophilic displacement reactions effectively, nuclear halogenopyridazine have been widely used as intermediates in the preparation of other pyridazines.[1477,1575] Single-crystal X-ray diffraction studies have been reported for 4,5-dichloro-2-phenyl-3(2*H*)-pyridazinone (also quadrupole resonance data),[1126] 3-chloro-5-*p*-toluenesulfonylmethylpyridazine,[1476] 4-chloro-3,6-bischloromethylpyridazine,[174] and 3,6-dichloropyridazine (also microwave spectroscopy).[177,2020] Like their pyrimidine analogues,[1930b] simple chloropyridazines can be irritating to the eyes and skin; the primary irritant effect and subsequent sensitization of skin to 3,4,5-trichloropyridazine has been studied in some detail using guinea pigs.[1003,1005] Some polarography has been reported.[1983]

The conversion of nuclear *halogenopyridazines into alkyl- or arylpyridazines* has been discussed in Section 3.2.1.3. The other important reactions of such halogenopyridazines are summarized in the following sections.

### 4.2.1 Aminolysis of Nuclear Halogenopyridazines (*H* 248, 465)

This is by far the most used reaction of halogenopyridazines. Because nuclear passenger groups are significant factors in the ease or otherwise of such aminolyses,[458] the typical examples in each subsection are grouped according to the type of passenger group(s) present. The order of nucleophilicity for amines is hydrazine > primary or secondary amines > ammonia; tetriary amines form a special category.

#### *4.2.1.1 3/6-Monohalogenopyridazines as Substrates (H 248)*

Although they usually undergo aminolysis quite readily, the 3/6-monohalogeno substrates appear to be marginally less reactive than their 4/5-isomers: this perceived difference probably arises because the halogeno leaving group is activated by an *ortho* ring nitrogen in the case of 3/6-halogenopyridazines but more effectively by a *para* ring nitrogen in the case of 4/5-halogenopyridazines; the modest steric effect of the lone-pair electrons on an *ortho* ring- nitrogen may also play its part. Kinetic data on the aminolysis of 3-chloro-6-phenyl-, 3-bromo-6-phenyl-, 3-chloro-4-methyl-6-phenyl-, and 3-bromo-4-methyl-6-phenylpyridazine have been reported.[914]

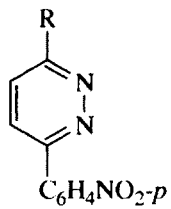
**From Substrates with Only Passenger Alkyl or Aryl Groups**

*Note:* It should be remembered that an alkyl group has a slightly deactivating effect on the halogen, whereas an aryl group (especially one with an

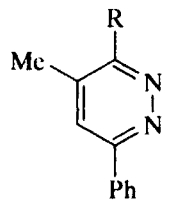
electron-withdrawing substituent) has a slightly activating effect thereon; also that an aryl or bulky alkyl group can have a substantial hindering effect on the aminolysis of an adjacent halgeno substituent. Unfortunately, such effects are seldom reflected in the conditions reported for preparative aminolyses, simply because of the almost universal tendency for workers to "overkill" in this respect to increase the chances of a good yield.

3-Chloro-6-*p*-nitrophenylpyridazine (**88**, R = Cl) gave 6-*p*-nitrophenyl-3-pyridazinamine (**88**, R = $NH_2$) ($NH_4OH$, $NH_4Cl$, $H_2O$, 200°, sealed, 12 h: 74%).[1309]

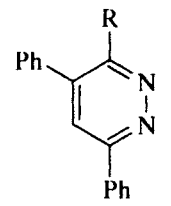
3-Chloro-4-methyl-6-phenylpyridazine (**89**, R = Cl) gave 3-*β*-aminoethylamino-4-methyl-6-phenylpyridazine (**89**, R = $NHCH_2CH_2NH_2$) (excess neat $H_2NCH_2CH_2NH_2$, 100°, $N_2$, 3 h: 80%),[1319] 4-methyl-3-*β*-morpholinoethylamino-6-phenylpyridazine [**89**, R = $NHCH_2CH_2N(CH_2CH_2)_2O$] [neat $H_2NCH_2CH_2N(CH_2CH_2)_2O$ + its hydrochloride, 170°, A, 2 h: 69%],[1569] 4-methyl-3-octylamino-6-phenylpyridazine (**89**, R = $HC_8H_{17}$) (neat $C_8H_{17}NH_2$ + its hydrochloride, 170°, A, 75 min: 93%),[914] 4-methyl-6-phenyl-3-pyridazinamine (**89**, R = $NH_2$) ($NaNH_2$, xylene, reflux, 48 h: 48%),[1279] or 3-hydrazino-4-methyl-6-phenylpyridazine (**89**, R = $NHNH_2$) ($H_2NNH_2 \cdot H_2O$, BuOH, reflux, 12 h: ~75%).[1104]



(**88**)



(**89**)



(**90**)

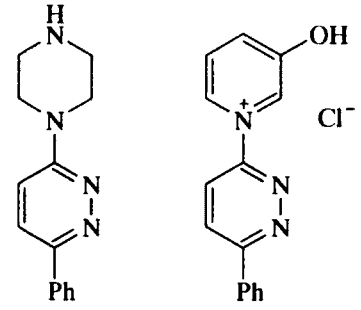
3-Chloro- (**90**, R = Cl) gave 3-dimethylamino-4,6-diphenylpyridazine (**90**, R = $NMe_2$) (neat $Me_2NH$, 160°, sealed, 6 h; 96%).[1082]

3-Chloro-6-phenylpyridazine gave 3-(*γ*-diethylamino-*β*-hydroxypropyl)amino-6-phenylpyridazine [$Et_2NCH_2CH(OH)CH_2NH_2$, trace KI, PhOH as solvent, < 125°, 30 min; then 165°, 10 h: 59%],[936] 3-phenyl-6-piperazin-1′-ylpyridazine (**91**) [excess $HN(CH_2CH_2)_2NH$, $AcBu^i$, reflux, 12 h: 91%],[1768] or 3-(3-hydroxypyridinio)-6-phenylpyridazine chloride (**92**) (3-pyridinol, BuOH, reflux, 6 h: 80%).[1236]

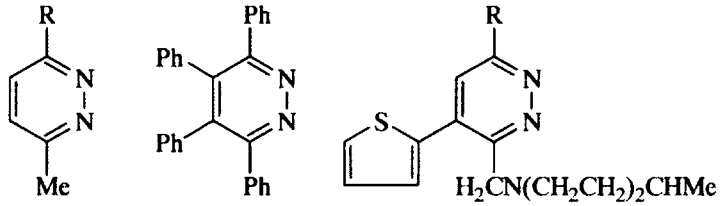
3-Chloro- (**93**, R = Cl) gave 3-hydrazino-6-methylpyridazine (**93**, R = $NHNH_2$) (neat $H_2NNH_2 \cdot H_2O$, 95°, 6 h: 75%);[1127] study of mechanism.[1037]

3-Chloro- (**94**, R = Cl) gave 3-hydrazino-4,5,6-triphenylpyridazine (**94**, R = $NHNH_2$) ($H_2NNH_2 \cdot H_2O$, BuOH, reflux, 4 h: 60%).[1362]

3-Chloro- (**95**, R = Cl) gave 3-hydrazino-5-(4-methylpiperidinomethyl)-6-thien-2′-ylpyridazine (**95**, R = $NHNH_2$) ($H_2NNH_2 \cdot H_2O$, EtOH, reflux, 3 h: 80%).[1727]



(91) (92)



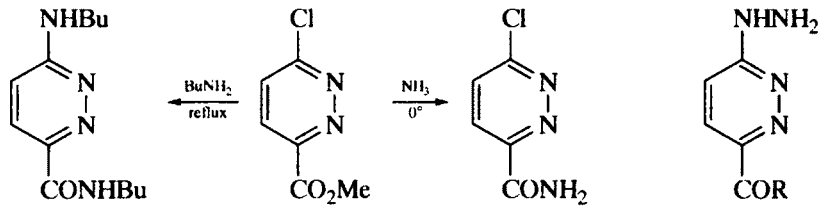
(93) (94) (95)

Also other examples.[18,138,152,158,363,514,779,808,837,886,922,936,946,975,1039,1064,1067,1103,1144,1248,1250,1251,1266,1275,1281,1284,1307,1333,1339,1394,1431,1703,1712,1734,1746,1807,2041,2069]

## From Substrates with Passenger Ester, Amide or Nitrile Groups

*Note:* Not only do these passenger groups activate halogeno substituents toward aminolysis, but they may themseleves react with amines under appropriate conditions.

Methyl 6-chloro-3-pyridazinecarboxylate (**97**) gave *N*-butyl-6-butylamino-3-pyridazinecarboxamide (**96**) (neat $BuNH_2$, reflux, 16 h: 84%) or only 6-chloro-3-pyridazinecarboxamide (**98**) ($NH_3$, MeOH, 0°, 30 min: 97%).[1592]



(96) (97) (98) (99)

Ethyl 6-chloro-3-pyridazinecarboxylate gave a separable mixture of ethyl 6-hydrazino-3-pyridazinecarboxylate (**99**, R = OEt), 6-hydrazino-3-pyridazinecarboxylic acid (**99**, R = OH), 6-hydrazino-3-pyridazinecarbohydrazide (**99**, $R = NHNH_2$), and 6-chloro-3-pyridazinecarbohydrazide

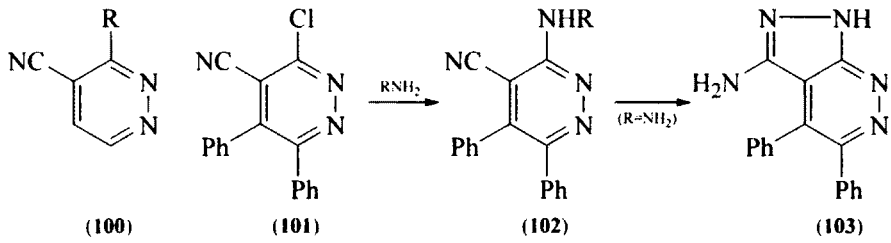
($H_2NNH_2$, EtOH, 60°, 3 h: 30%, 10%, 3%, and 22%, respectively);[1459] however, the same substrate with morpholine gave only ethyl 6-morpholino-3-pyridazinecarboxylate (PhMe, reflux, 3 h: 58%).[1338]

6-Chloro-3-pyridazinecarboxamide (**98**) gave 6-benzylamino-3-pyridazinecarboxamide ($PhCH_2NH_2$, dioxane, reflux, >15 h: 70%);[1773] also homologues likewise.[1592,1773]

3-Chloro-4-pyridazinecarbonitrile (**100**, R = Cl) gave 3-benzylamino- (**100**, R = $NHCH_2Ph$) ($PhCH_2NH_2$, EtOH, reflux, 5 h: 80%),[1079] 3-isopropylamino- (**100**, R = $NHPr^i$) (likewise but only 30%),[1079] or 3-(*N*-β-ethoxycarbonylethyl-*N*-ethylamino)-4-pyridazinecarbonitrile [**100**, R = $N(CH_2CH_2CO_2Et)Et$] [$HNC(CH_2CH_2CO_2Et)Et$, $Me_2NCHO$, 100°, 18 h: 50%].[523]

3-Chloro-5,6-diphenyl-4-pyridazinecarbonitrile (**101**) gave 3-anilino-5,6-diphenyl- (**102**, R = Ph) ($PhNH_2$, AcOH, reflux, 5 h: 65%) or 5,6-diphenyl-3-phenylhydrazino-4-pyridazinecarbonitrile (**102**, R = NHPh) ($PhNHNH_2$ likewise: 70%);[1726] in contrast, hydrazinolysis under the same conditions gave 4,5-diphenyl-1*H*-pyrazolo[3,4-*c*]pyridazine-3-amine (**103**) (65%), presumably by spontaneous cyclization of the initial product (**102**, R = $NH_2$).[1046,1398,1726]

Also other examples.[759,807,1271,1338,2085]




(**100**) (**101**) (**102**) (**103**)

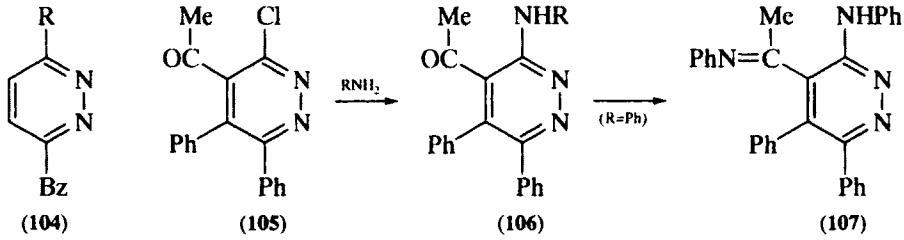
## From Substrates Bearing Ketonic Groups

*Note:* The ketonic passenger groups on these substrates activate the halogeno substituents but may themselves react, at least with primary aromatic amines.

3-Benzoyl-6-chloropyridazine (**104**, R = Cl) gave 3-benzoyl-6-(4-methylpiperazin-1-yl)pyridazine [**104**, R = $N(CH_2CH_2)NMe$] [$MeN(CH_2CH_2)_2NH$, reflux, 6 h: 77%].[1698]

4-Acetyl-3-chloro-5,6-diphenylpyridazine (**105**) gave 4-acetyl-3-anilino- (**106**, R = Ph) ($PhNH_2$, EtOH, reflux, 3 h: 74%) or its Schiff base, 3-anilino-4-α-phenyliminoethyl-5,6-diphenylpyridazine (**107**) (neat $PhNH_2$, 160°, 1 h: 74%);[377] in contrast, the same substrate (**105**) gave only 4-acetyl-3-butylamino-5,6-diphenylpyridazine (**106**, R = Bu) ($BuNH_2$, neat or in EtOH, reflux, 1–3 h: 74%).[377]

Also other examples.[1367]

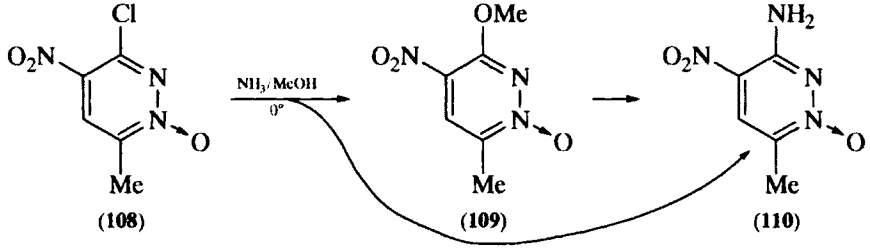



(104) (105) (106) (107)

## From Substrate N-Oxides Bearing a Nitro Group

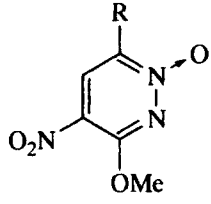
*Note*: The halogeno substituent in such substrates is highly activated.

3-Chloro-6-methyl-4-nitropyridazine 1-oxide (**108**) gave a separable mixture of 3-methoxy-6-methyl-4-nitropyridazine 1-oxide (**109**) and 6-methyl-4-nitro-3-pyridazinamine 1-oxide (**110**) ($NH_3$, MeOH, 0°, 1 h: 32% and 38%, respectively); treatment of the methoxy derivative (**109**) in methanolic ammonia at 0° gradually gave the amino analogue (**110**) (9% after 1 h). The balance of evidence suggests that products **109** and **110** are both formed directly and rapidly in the original aminolysis of substrate (**108**), with concomitant slow aminolysis of the methoxypyridazine (**109**).[952]




(108) (109) (110)

3-Chloro-6-methoxy-5-nitropyridazine 2-oxide (**111**, R = Cl) gave 3-benzylamino-6-methoxy-5-nitropyridazine 2-oxide (**111**, R = $NHCH_2Ph$) ($Ph_2NH_2$, EtOH, reflux, 3 h: 31%).[905]



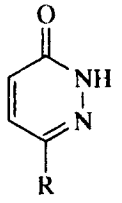

(111)

## From Substrates Bearing Oxy or Thioxy Substituents

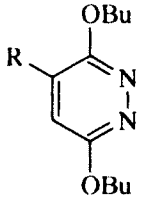
6-Chloro- (**112**, R = Cl) gave 6-hydrazino-3(2*H*)-pyridazinone (**112**, R = $NHNH_2$) (neat $H_2NNH_2 \cdot H_2O$, 95°, 3 h: 27%).[1338]

4,6-Dibutoxy-3-chloropyridazine (**113**, R = Cl) gave 4,6-dibutoxy-3-morpholinopyridazine [**113**, R = $N(CH_2CH_2)_2O$] [neat anhydrous $O(CH_2CH_2)_2NH$, reflux, 3 h: ?%].[640]

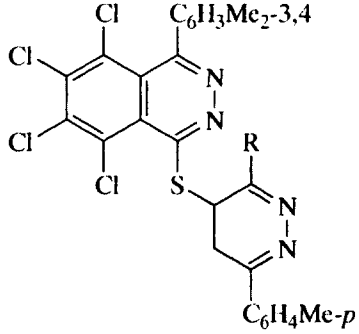
5,6,7,8-Tetrachloro-1-(3-chloro-6-*p*-tolyl-4,5-dihydropyridazin-4-ylthio)-4-(3,4-dimethylphenyl)phthalazine (**114**, R = Cl) gave the 1-(3-hydrazino-6-*p*-tolyl-4,5-dihydropyridazin-4-ylthio) analogue (**114**, R = $NHNH_2$) ($H_2NNH_2 \cdot HCl$, EtOH, reflux, 6 h: 67%).[1711]



(112)



(113)



(114)

Also other examples.[1041]

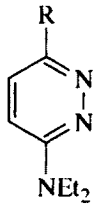
## From Substrates Bearing Amino Groups

*Note:* The examples in this category broadly indicate deactivation of each halogeno substituent by the passenger amino group(s).

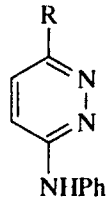
3-Chloro-6-diethylaminopyridazine (**115**, R = Cl) gave 3-diethylamino-6-γ-diethylaminopropylaminopyridazine (**115**, R = $NHCH_2CH_2CH_2NEt_2$) (neat $Et_2NCH_2CH_2CH_2NH_2$, 150°, 24 h: 62%).[924]

3-Anilino-6-chloropyridazine (**116**, R = Cl) gave 3-anilino-6-hydrazinopyridazine (**116**, R = $NHNH_2$) (neat $H_2NNH_2 \cdot H_2O$, reflux, 5 h: ~35%).[1161]

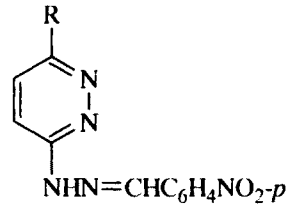
3-Chloro- (**117**, R = Cl) gave 3-morpholino-6-*p*-nitrobenzylidenehydrazinopyridazine [**117**, R = $N(CH_2CH_2)_2O$] [$O(CH_2)_2NH$, $OP(NMe_2)_3$, 150°, 24 h: 85%].[57]



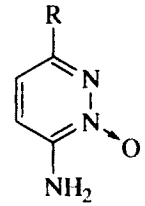
(115)



(116)



(117)



(118)

5-Amino-6-chloro- gave 5-amino-6-hydrazino-3(2*H*)-pyridazinone (neat $H_2NNH_2 \cdot H_2O$, reflux, 4 h: 73%).[1597]

6-Chloro-3-pyridazinamine 2-oxide (**118**, R = Cl) gave 6-*o*-chlorobenzylamino-3-pyridazinamine 2-oxide (**118**, R = $NHCH_2C_6H_4Cl$-*o*) (neat $H_2NCH_2CH_2C_6H_4Cl$-*o*, 142°, sealed, 40 h: 62%);[1527] also analogues likewise.[1161, 1524, 1527]

Also other examples.[554, 955, 1101, 1258, 1283, 1616, 1962]

### *4.2.1.2 4/5-Monohalogenopyridazines as Substrates (H 248)*

The halogeno substituents in these substrates appear to be activated or deactivated by their passenger substituents in much the same way as are their 3/6-halogeno counterparts (Section 4.2.1.1). However, since most categories of passenger groups are poorly represented or even unrepresented in the 4/5-halogenopyridazines available as examples, direct comparisons are virtually impossible. Aminolyses ($NaNH_2$/liquid $NH_3$ or even amines/solvent) have sometimes led to *cine* replacements with the 4/5-chloropyridazines—*beware*! The following examples illustrate typical aminolyses:

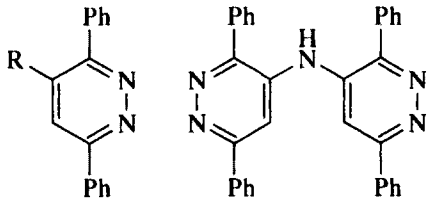
**From Substrates with Only Passenger Alkyl or Aryl Groups**

4-Chloro-3,6-diphenylpyridazine (**119**, R = Cl) gave 3,6-diphenyl-4-pyridazinamine (**119**, R = $NH_2$) (PhOH, trace $CuSO_4$, $NH_3\downarrow$, reflux, 4 h: 60%) or a separable mixture of **119** (R = $NH_2$) and bis(3,6-diphenylpyridazin-4-yl)amine (**120**) ($KNH_2$, liquid $NH_3$, −33°, 7 h: 21% and 10%, respectively).[451]

4-Chloro- gave 4-dimethylamino-6-methyl-3,5-diphenylpyridazine ($Me_2NH$, $Me_2SO$, 115°, sealed, 5 days: ~20%).[311]

4-Chloro- (**121**, R = Cl), 4-bromo- (**121**, R = Br), or 4-iodo-3-methoxymethyl-6-methylpyridazine (**121**, R = I) gave in each case a separable mixture of 3-methoxymethyl-6-methyl-4-pyridazinamine (**121**, R = $NH_2$) and the *cine*-substitution product, 6-methoxymethyl-3-methyl-4-pyridazinamine (**122**), in a ratio of 17:83 ($NaNH_2$, liquid $NH_3$, −33°, 30 min); in contrast, the same chloro substrate (**121**, R = Cl) gave only the expected product (**121**, R = $NH_2$) on regular aminolysis ($NH_3$, EtOH, 130°, sealed, 20 h: 67%).[446]

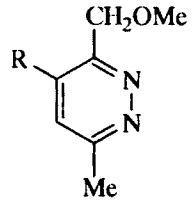
Also other examples.[1985]



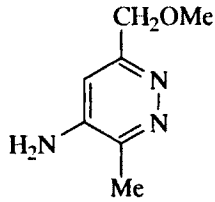
(119)



(120)



(121)

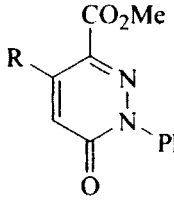


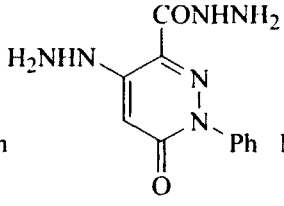
(122)

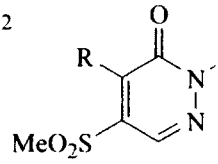
### From Substrates Bearing Electron-Withdrawing Passenger Groups

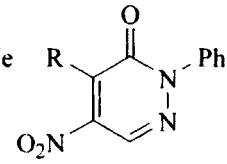
*Note:* Relatively gentle conditions are employed in most examples.

Methyl 4-chloro- (**123**, R = Cl) gave methyl 4-benzylamino-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate (**123**, R = $NHCH_2Ph$) ($PhCH_2NH_2$, $ClC_6H_4Cl$-*o*, reflux, 2.5 h: 65%);[1117] in contrast, the same substrate (**123**, R = Cl) gave methyl 4-hydrazino-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate (**123**, R = $NHNH_2$) ($H_2NNH_2 \cdot H_2O$, EtOH, 40°, 10 min: 94%) or 4-hydrazino-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarbohydrazide (**124**) ($H_2NNH_2 \cdot H_2O$, EtOH, reflux, 15 min: 84%), according to conditions.[1117]


(123)


(124)


(125)


(126)

4-Chloro- (**125**, R = Cl) gave 4-amino-2-methyl-5-methylsulfonyl-3(2*H*)-pyridazinone (**125**, R = $NH_2$) ($NH_4OH$, EtOH, 25°, 12 h: 80%; note survival of the methylsulfonyl group); likewise the 2-phenyl analogue (78%).[533]

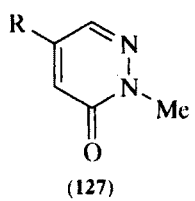
4-Chloro- (**126**, R = Cl) gave 4-cyclohexylamino-5-nitro-2-phenyl-3(2*H*)-pyridazinone (**126**, R = $NHC_6H_{11}$) ($C_6H_{11}NH_2$: >70%).[601]

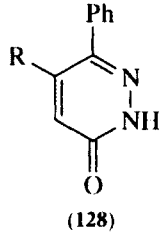
Also other examples.[240]

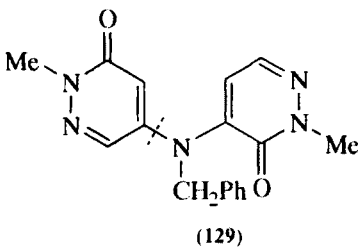
### From Substrates with One Oxo Passenger Group

5-Iodo-2-methyl-3(2*H*)-pyridazinone (**127**, R = I) gave 2-methyl-5-morpholino-3(2*H*)-pyridazinone [**127**, R = $N(CH_2CH_2)_2O$] [$O(CH_2CH_2)_2NH$, EtOH, reflux, ? h: 65%].[1603]

5-Chloro-6-phenyl-3(2*H*)-pyridazinone (**128**, R = Cl) gave 6-phenyl-5-piperazin-1′-yl-3(2*H*)-pyridazinone [**128**, R = $N(CH_2CH_2)_2NH$] [HN $(CH_2CH_2)_2NH$, BuOH, reflux, 16 h: 63%].[1588]


(127)


(128)

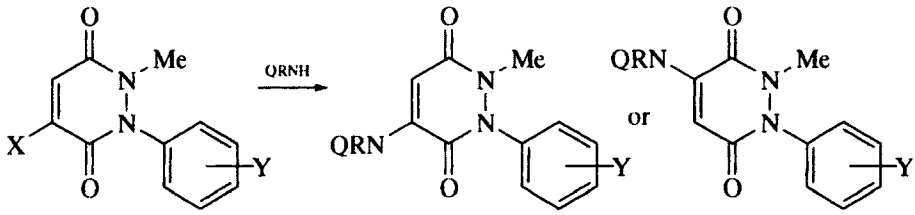

(129)

5-Chloro-2-methyl-3(2*H*)-pyridazinone and 4-benzylamino-2-methyl-3(2*H*)-pyridazinone gave *N*-benzyl-*N*-(1-methyl-6-oxo-1,6-dihydropyridazin-4-yl)- *N*-(2-methyl-3-oxo-2,3-dihydropyridazin-4-yl)amine (**129**) ($K_2CO_3$, $Me_2NCHO$, 150°, 5 h: 43%).[867]

Also other examples.[578,584,768,848,1512]

## From Substrates with Two Oxo Passenger Groups

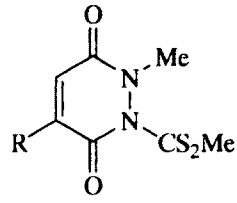
*Note*: Treatment, for example, of 4-halogeno-1-methyl-2-phenyl-3,6(1*H*,2*H*)-pyridazinediones (**130**) with amines in a solvent has been reported to give either the regular aminolytic product (**131**) or the *cine*-product (**132**): criteria for the production of one or other product are by no means clear yet. Examples of both phenomena follow.




(**130**) (**131**) (**132**)

4-Bromo-2-*p*-bromophenyl-1-methyl- (**130**, X = Br, Y = *p*-Br) gave 1-*p*-bromophenyl-2-methyl-5-morpholino-3,6(1*H*,2*H*)-pyridazinedione (**131**, QR = $CH_2CH_2OCH_2CH_2$, Y = *p*-Br) [$O(CH_2CH_2)_2NH$, EtOH, reflux, 2 h: 50%; structure confirmed by X-ray analysis].[985]

Methyl 5-bromo- (**133**, R = Br) gave methyl 5-amino-2-methyl-3,6-dioxo-1,2,3,6-tetrahydro-1-pyridazinecarbodithioate (**133**, R = $NH_2$) ($NH_3$, see original) or likewise the 5-morpholino analogue [**133**, R = $N(CH_2CH_2)_2O$].[984]




(**133**)

In contrast to the foregoing examples of regular aminolysis:

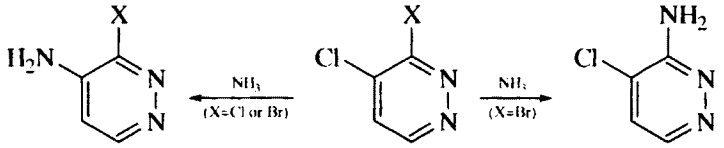
4-Bromo-2-*p*-bromophenyl-1-methyl- (**130**, X = Br, Y = *p*-Br) gave 1-*p*-bromophenyl-4-diethylamino-2-methyl-3,6(1*H*,2*H*)-pyridazinedione (**132**, Q = R = Et, Y = *p*-Br) ($Et_2NH$, MeOH/EtOH);[678,703,787,985] the structures of both substrate and product were confirmed by X-ray analyses.[965]

Other 4-chloro- or 4-bromo-2-(substituted-phenyl) substrates (**130**) also provided only *cine*-aminolysis products (**132**).[620,685,705]

### *4.2.1.3 3,4/5,6-Dihalogenopyridazines as Substrates (H 249)*

In such 3,4-dihalogenopyridazines, the 4-halogeno (activated by a *para* ring nitrogen) should be more easily aminolyzed than the 3-halogeno substituent (activated by an *ortho* ring nitrogen). In almost every recent example of the monoaminolysis of 3,4-dihalogenopyridazines, it was only the 4-halogeno substituent that suffered replacement; moreover, diaminolysis was significantly absent in the reports, suggesting that the first amino group to enter the molecule then powerfully deactivated the remaining halogeno substituent next to it. The following examples illustrate these points:

3,4-Dichloropyridazine (**135**, X = Cl) gave only 3-chloro-4-pyridazinamine (**134**, X = Cl) ($NH_3$, EtOH, 125°, sealed, 6 h: 52%);[447] likewise, 3-bromo-4-chloropyridazine (**135**, X = Br) gave 3-bromo-4-pyridazinamine (**134**, X = Br), but is contained ~ 10% of 4-chloro-3-pyridazinamine (**136**),[447] perhaps because bromo is intrinsically a little better than chloro as a leaving group.




(134) (135) (136)

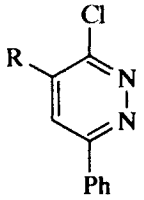
3,4-Dichloro-6-phenylpyridazine (**137**, R = Cl) gave only 3-chloro-4-dimethylamino-6-phenylpyridazine (**137**, R = $NMe_2$) ($Me_2NH$, EtOH, reflux, 5 h: 90%); also 4-butylamino-3-chloro-6-phenylpyridazine (**137**, R = NHBu) (likewise: 77%).[1041]

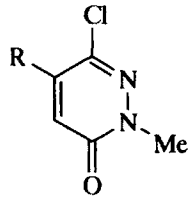
5,6-Dichloro-2-methyl-3(2*H*)-pyridazinone (**138**, R = Cl) gave only 6-chloro-2-methyl-5-α-methylhydrazino-3(2*H*)-pyridazinone (**138**, R = $NMeNH_2$) ($MeHNNH_2$, MeOH, 25°, 5 h: 53%).[1061]

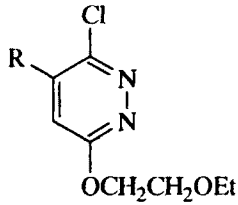
3,4-Dichloro- (**139**, R = Cl) gave 3-chloro-4-dimethylamino-6-β-ethoxyethylpyridazine (**139**, R = $NMe_2$) (PhH, $Me_2NH\downarrow$, < 40°: ~80%, as hydrochloride).[640]

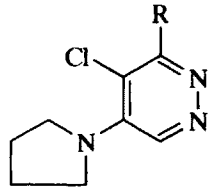
In contrast, 3,4-dichloro- (**140**, R = Cl) gave only 4-chloro-3-hydrazino-5-pyrrolidin-1′-ylpyridazine (**140**, R = $NHNH_2$) (95% $H_2NNH_2$, reflux, briefly: 88%; presumably the steric and electronic effect of the pyrrolidinyl group on the adjacent 4-chloro substituent caused preferential 3-aminolysis).[1058]

Also other examples.[224,861,1025]


(137)


(138)


(139)

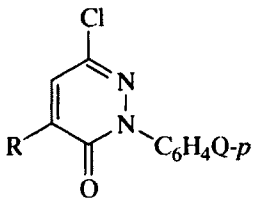

(140)

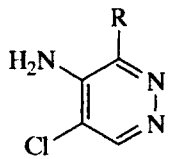
### *4.2.1.4 3,5/4,6-Dihalogenopyridazines as Substrates (H 249)*

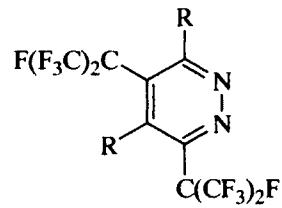
For reasons given in Section 4.2.1.3, the 4/5-halogeno should be more easily displaced than the 3/6-halogeno substituent from these substrates. Most of the reported examples are consistent with this postulate, as indicated in the following aminolyses.

4,6-Dichloro-2-phenyl-3(2*H*)-pyridazinone (**141**, Q = H, R = Cl) gave 6-chloro-4-γ-dimethylaminopropylamino-2-phenyl-3(2*H*)-pyridazinone [**141**, Q = H, R = $NH(CH_2)_3NMe_2$] [$Me_2N(CH_2)_3$ $NH_2$, EtOH, reflux, 13 h: 80%];[540] likewise analogues.[540]

4,6-Dichloro-2-*p*-nitrophenyl-3(2*H*)-pyridazinone (**141**, Q = $NO_2$, R = Cl) gave 6-chloro-4-hydrazino-2-*p*-nitrophenyl-3(2*H*)-pyridazinone (**141**, Q = $NO_2$, R = $NHNH_2$) ($H_2NNH_2 \cdot H_2O$, MeOH, reflux, 6 h: 61%).[539]


(141)


(142)


(143)

In contrast, 3,5-dichloro-4-pyridazinamine (**142**, R = Cl) gave 5-chloro-3-α-methylhydrazino- (**142**, R = $NMeNH_2$) (neat $MeHNNH_2$, 95°, 2.5 h: 51%) or 5-chloro-3-α-(*o*-fluorobenzyl)hydrazino-4-pyridazinamine [**142**, R = $N(CH_2C_6H_4F\text{-}o)NH_2$] (neat $H_2NNHCH_2C_6H_4F\text{-}o$, 100°, $N_2$, 13 h: 29%);[1820] the reason for preferential replacement of the 3-chloro substituent is not evident, but the fact remains.

Exceptionally, 3,5-difluoro- (**143**, R = F) gave 3,5-bisdimethylamino-4,6-bis(perfluoroisopropyl)pyridazine [**143**, R = $C(CF_3)_2F$] ($Me_2NH$, $Me_2NCHO$, 25°, ? h: 66%).[491]

Also other examples.[792,938,1471]

### *4.2.1.5 3,6-Dihalogenopyridazines as Substrates (H 249)*

In 3,6-dihalogenopyridazines, the halogeno substituents are equally prone to aminolysis except when a passenger group preferentially affects one such

leaving group by electronic or steric means. The examples that follow are therefore classified according to the type of passenger group(s) present on the substrate:

### From the Parent Substrates

3,6-Dichloropyridazine (**145**) gave the following products:

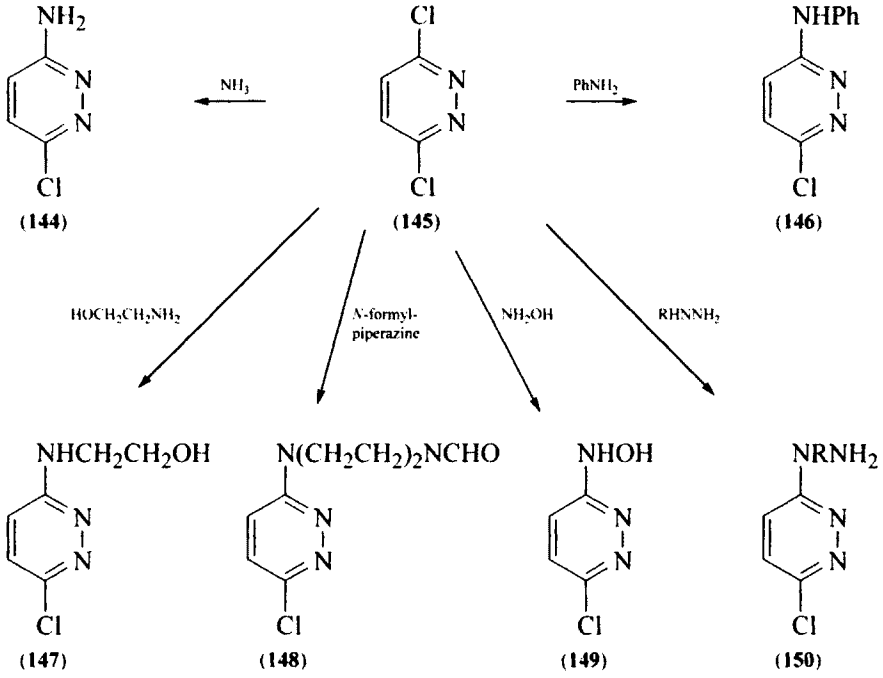
6-Chloro-3-pyridazinamine (**144**) ($NH_4OH$, 100°, sealed, 6 h: 49%;[1283] likewise, 130°, 12 h: 75%).[336]

3-Anilino-6-chloropyridazine (**146**) ($PhNH_2$, EtOH, reflux, ? h: ?%),[1161] 3-chloro-6-(4-methyl-3-nitroanilino)pyridazine (4-Me-3-$NO_2$-aniline, EtOH, reflux, 3 h: 86%),[1330] or analogues.[1291]

3-Chloro-6-(β-hydroxyethylamino)pyridazine (**147**) ($H_2NCH_2CH_2ONa$, $H_2NCH_2CH_2OH$, 25°, 24 h: 69%)[1169] or 3,6-bis(β-hydroxyethylamino)-pyridazine (neat $H_2NCH_2CH_2OH$, 160°, 5 h: 90%).[107] also analogues.[163,1733]

3-Chloro-6-(4-formylpiperazin-1-yl)pyridazine (**148**) [$HN(CH_2CH_2)_2NCHO$, EtOH, reflux, 16 h: 50%];[1761] also analogues.[522,1254,2055]

3-Chloro-6-hydroxyaminopyridazine (**149**) [$HOHN_2$, $H_2O$–EtOH, reflux, 6.5 h: 28%).[196,412,966]




3-Chloro-6-hydrazinopyridazine (**150**, R = H) [$H_2NNH_2 \cdot H_2O$, MeOH, reflux, 24 h: 85%;[636] $H_2NNH_2$, $NH_4OH$, $H_2O$, reflux, 1 h: 99%;[1224]

$H_2NNH_2 \cdot H_2O$, $NaHCO_3$, $H_2O$, reflux, 6.5 h: 80%, isolated as 2,4-bis(6-chloropyridazin-3-ylhydrazono)hexane];[1345] or 3-chloro-6-α-methylhydrazinopyridazine (**150**, R = Me) ($MeHNNH_2$, EtOH, reflux, briefly, then 25°, until complete: 52%).[1183]

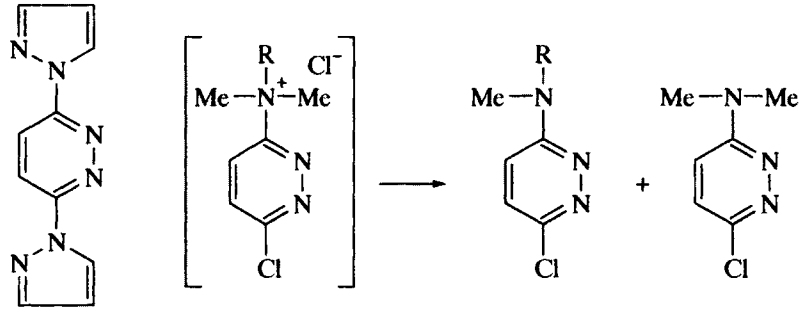
3,6-Bispyrazol-1′-ylpyridazine (**151**) (excess pyrazole, EtOH, reflux, 2 h: 65%),[977] 3-chloro-6-pyrazol-1′-ylpyridazine (K-pyrazole, THF, 20° → reflux, 4 h: 74%),[2081] or complicated analogues.[1807]

Also other examples.[554,955,1239,1293,1730,1818,1886,2059,2101]

3,6-Difluoropyridazine gave 6-fluoro-3-pyridazinamine ($NH_3$, EtOH, 100°, sealed, 15 h: ~45%).[1145]

3,6-Diiodopyridazine gave 3-dimethylamino-6-iodopyradazine ($Me_2NH$, $H_2O$, reflux, 12 h: >95%).[1385]

In addition, 3,6-dichloropyridazine with the tertiary amine, trimethylamine, gave a separable mixture of 3-chloro-6-trimethylammoniopyridazine chloride (**152**, R = Me) and its decomposition product, 3-chloro-6-dimethylaminopyridazine (**154**) ($Me_3N$, PhH, <25°; ~10% and ~5%, respectively);[1796] with isopropyldimethylamine gave a separable mixture of 3-chloro-6-(*N*-isopropyl-*N*-methylamino)- (**153**, R = $Pr^i$) and 3-chloro-6-dimethylaminopyridazine (**154**) [THF, 100°, sealed, 4 days: 82% and 6%, respectively; presumably by loss of MeCl or $Pr^iCl$, respectively, from the common intermediate (**152**, R = $Pr^i$)];[1352] with *N*-cyclohexyl-*N*,*N*-dimethylamine likewise gave mainly 3-chloro-6-(*N*-cyclohexyl-*N*-methylamino)pyridazine (**153**, R = $C_6H_{11}$);[1352] or with *N*-benzyl-*N*,*N*-dimethylamine likewise gave (in contrast) mainly 3-chloro-6-dimethylaminopyridazine (**154**).[1352] The reactions of 3,6-dichloropyridazine with pyridine have been explored.[1981]




(151) (152) (153) (154)

## From Substrates with Only Passenger Alkyl or Aryl Groups

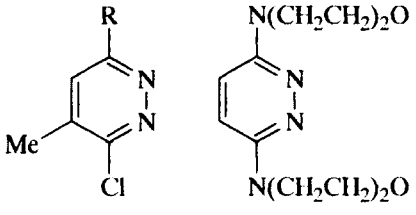
3,6-Dichloro-4-methylpyridazine (**155**, R = Cl) gave two isomeric chloromethylpyridazinamines from which the less soluble major(?) compo-

nent, 6-chloro-5-methyl-3-pyridazinamine (**155**, R = $NH_2$), was separated easily ($NH_3$, EtOH, 140°, sealed, 8 h).[1145]

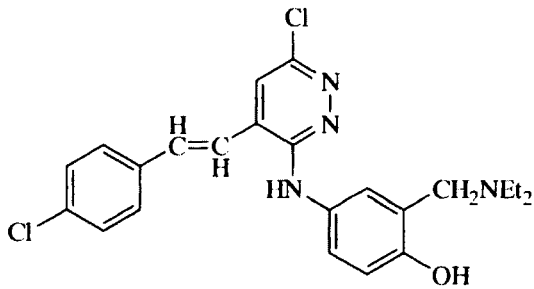
The same substrate (**155**, R = Cl) gave only 3-chloro-6-di(β-hydroxyethyl)amino-4-methylpyridazine [**155**, R = $N(CH_2CH_2OH)_2$] [$(HOCH_2CH_2)_2NH$, $C_6H_{11}OH$, reflux, 4 h: ~30%] or 4-methyl-3,6-dimorpholinopyridazine (**156**) [neat $O(CH_2CH_2)_2NH$, reflux, 6 h: ~60%].[544]

3,6-Dichloro-4-*p*-chlorostyrylpyridazine and 4-amino-2-diethylaminomethylphenol were reported to give only 3-chloro-5-*p*-chlorostyryl-6-(3-diethylaminomethyl-4-hydroxyanilino)pyridazine (**157**) (EtOH, reflux, 24 h: 70%, structure in line with mild electron withdrawal by the chlorostyryl group but at variance with the appreciable steric factors).[1739]

Also other examples.[1009]



(155) (156)

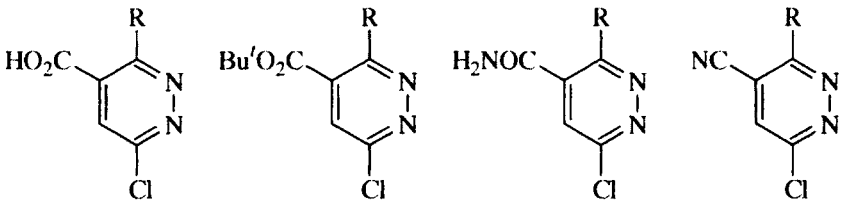


(157)

## From Substrates with Passenger Carbonyl-Containing or Nitrile Groups

3,6-Dichloro-4-pyridazinecarboxylic acid (**158**, R = Cl) gave only 6-chloro-3-hydrazino-4-pyridazinecarboxylic acid (**158**, R = $NHNH_2$ $H_2NNH_2 \cdot H_2O$, EtOH, reflux, 1 h: 93%).[1228]

*t*-Butyl 3,6-dichloro-4-pyridazinecarboxylate (**159**, R = Cl) gave *t*-butyl 6-chloro-3-hydrazino-4-pyridazinecarboxylate (**159**, R = $NHNH_2$) ($H_2NNH_2$, 25°, 90 min; 61%).[945]



(158) (159) (160) (161)

3,6-Dichloro-4-pyridazinecarboxamide (**160**, R = Cl) gave a separable mixture of 3-amino-6-chloro- (**160**, R = $NH_2$), the isomeric 6-amino-3-chloro-, and 6-chloro-3-ethoxy-4-pyridazinecarboxamide (**160**, R = OEt) ($NH_3$, EtOH, 100°, sealed, 2 h: ~60%, ~20%, and ~20%, respecti-

vely)[1099] or only 6-chloro-3-hydrazino-4-pyridazinecarboxamide (**160**, R = $NHNH_2$) ($H_2NNH_2 \cdot H_2O$, EtOH, reflux, 45 min: 63%).[765]

3,6-Dichloro-4-pyridazinecarbonitrile (**161**, R = Cl) gave 6-chloro-3-piperidino-4-pyridazinecarbonitrile [**161**, R = $N(CH_2)_5$] and a little of the isomeric 3-chloro-6-piperidino-4-pyridazinecarbonitrile [$(CH_2)_5NH$, MeOH, reflux, 1 h: 75% and 7%, respectively].[820]

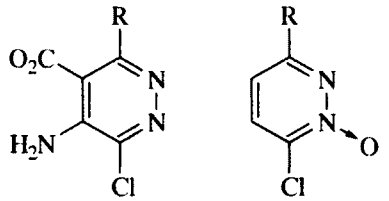
Also other examples.[820, 1479, 1954, 1984, 2110]

### From Substrates with a Passenger Nitro Group or N-Oxide

3,6-Dichloro-5-nitro-4-pyridazinamine (**162**, R = Cl) gave 3-chloro-6-hydrazino-5-nitro-4-pyridazinamine (**162**, R = $NHNH_2$) ($H_2NNH_2$, EtOH, <5°, briefly: 93%).[1001]

3,6-Dichloropyridazine 1-oxide (**163**, R = Cl) gave 6-chloro-3-pyridazinamine 1-oxide (**163**, R = $NH_2$) (liquid $NH_3$, 80°, sealed, 6 h: 70%, after separation from the isomeric byproduct)[891] or 3-chloro-6-hydrazinopyridazine 2-oxide (**163**, R = $NHNH_2$) ($H_2NNH_2 \cdot H_2O$, EtOH, reflux, 1 h: 46–60%).[546, 891]

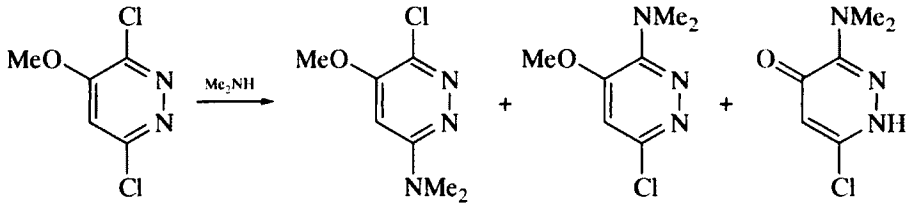
Also other examples.[555]




(**162**) (**163**)

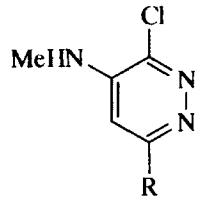
### From Substrates with Passenger Oxo or Amino Substituents

3,6-Dichloro-4-methoxypyridazine (**164**) gave a separable mixture of at least five products from which were isolated initially 3-chloro-6-dimethylamino-4-methoxypyridazine (**165**), 3-chloro-6-dimethylamino-5-methoxypyridazine (**166**), and 6-chloro-3-dimethylamino-4(1*H*)-pyridazinone (**167**) ($Me_2NH$, PhH, 120°, sealed, 6 h: ~12%, ~15%, and ~25%, respectively).[640]




(**164**) (**165**) (**166**) (**167**)

3,6-Dichloro-4-methylaminopyridazine (**168**, R = Cl) gave 6-chloro-5-methylamino-3-pyridazinamine (**168**, R = $NH_2$) ($KNH_2$, liquid $NH_3$, − 33°, ?h: 63%)[447] or 3-chloro-4-methylamino-6-α-methylhydrazinopyridazine (**168**, R = $NMeNH_2$) ($MeHNNH_2$, $H_2O$, 95°, 1 h: ~45%).[1138]

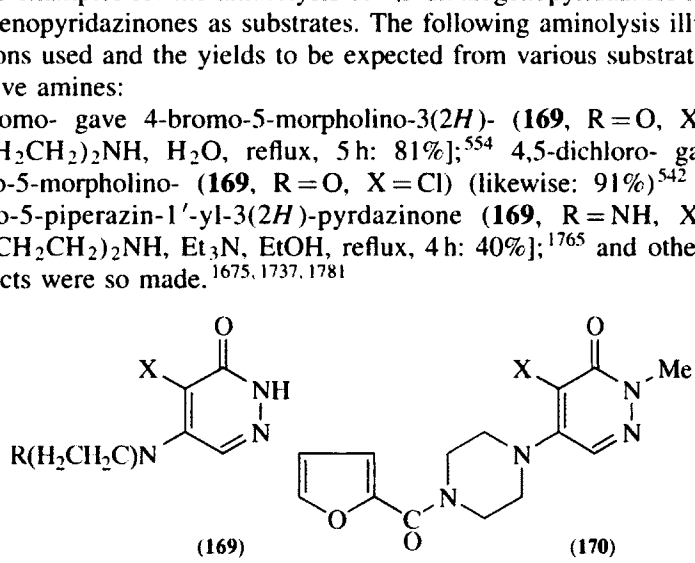



(**168**)

### *4.2.1.6 4,5-Dihalogenopyridazines as Substrates (H 249)*

The remarks on aminolysis of 3,6-dihalogenopyridazines (Section 4.2.1.5) apply equally well to their 4,5 counterparts. However, no direct comparison of the two systems is possible because, in contrast with 3,6-dihalogenopyridazines, all available examples for the aminolysis of 4,5-dihalogenopyridazines involve 4,5-dihalogenopyridazinones as substrates. The following aminolysis illustrate the conditions used and the yields to be expected from various substrates and representative amines:

4,5-Dibromo- gave 4-bromo-5-morpholino-3(2*H*)- (**169**, R = O, X = Br) [$O(CH_2CH_2)_2NH$, $H_2O$, reflux, 5 h: 81%];[554] 4,5-dichloro- gave 4-chloro-5-morpholino- (**169**, R = O, X = Cl) (likewise: 91%)[542] or 4-chloro-5-piperazin-1′-yl-3(2*H*)-pyrdazinone (**169**, R = NH, X = Cl) [$HN(CH_2CH_2)_2NH$, $Et_3N$, EtOH, reflux, 4 h: 40%];[1765] and other such products were so made.[1675, 1737, 1781]

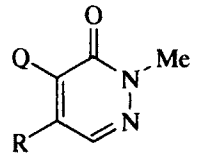



(**169**) (**170**)

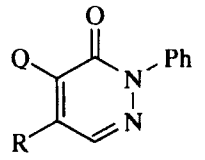
4,5-Dibromo-2-methyl-3(2*H*)-pyridazinone and 4-fur-2′-oylpiperazine gave 4-bromo-5-[4-(fur-2-oyl)piperazin-1-yl]-2-methyl-3(2*H*)-pyridazinone (**170**, X = Br) ($K_2CO_3$, $Me_2NCHO$, reflux, 6 h: 61%);[515] 4,5-dichloro- likewise gave 4-chloro-5-[4-(fur-2-oyl)piperazin-1-yl]-2-methyl-3(2*H*)-pyridazinone (**170**, X = Cl) (55%).[515]

4,5-Dichloro- (**171**, Q = R = Cl) gave a separable mixture of 5-amino-4-chloro- (**171**, Q = Cl, R = $NH_2$) and 4-amino-5-chloro-2-methyl-3(2*H*)-

pyridazinone (**171**, Q = $NH_2$, R = Cl) ($NH_4OH$, 145°, sealed, 12 h: 40% and 9%, respectively);[1677] a separable mixture of 4-chloro 5-dimethylamino- (**171**, Q = Cl, R = $NMe_2$) and 5-chloro-4-dimethylamino-2-methyl-3(2*H*)-pyrdazinone (**171**, Q = $NMe_2$, R = Cl) ($Me_2NH$, PhH, 25°, 3 months: ~35% and ~40%, respectively;[641] $Me_2NH$, PhMe, reflux, 7 h; 35% and 51%, respectively);[148] or other such separable mixtures.[148, 532, 850, 867, 1054, 1269, 1601, 1677, 1685, 1903]



(171)

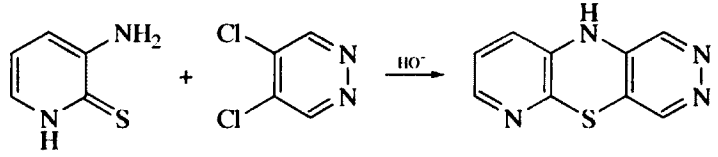


(172)

4,5-Dichloro- (**172**, Q = R = Cl) gave 5-amino-4-chloro-2-phenyl-3(2*H*)-pyridazinone (**172**, Q = Cl, R = $NH_2$) [$NH_4OH$, $Me_2SO$, 100°, sealed, 8 h: 54%, after separation from an isomeric(?) byproduct;[464] a separable mixture of 4-chloro-5-cyclopropylamino- (**172**, Q = Cl, R = $NHC_3H_5$) and 5-chloro-4-cyclopropylamino-2-phenyl-3(2*H*)-pyridazinone (**172**, Q = $NHC_3H_5$, R = Cl) ($C_3H_5NH_2$, EtOH, reflux, 4.5 h: 83% and 6%, respectively; in contrast, $C_3H_5NH_2$, $Et_3N$, THF, reflux, 54 h: 44% and 40%, respectively);[956] or other such mixtures.[687, 873]

4,5-Dichloropyridazine (**174**) and 3-amino-2(1*H*)-pyridinethione (**173**) gave 10*H*-pyridazino[4,5-*b*]pyrido[3,2-*e*][1,4]thiazine (**175**) (KOH, EtOH, 90°, 10 min, then 25°, 14 h: 63%; probably by initial sulfide formation followed by intramolecular aminolysis);[1033] analogues likewise.[205, 1033]

Also other examples from 4,5-dihalogeno-2-substituted (alkyl, aryl, or heteroaryl)-3(2*H*)-pyridazinones with a variety of amines.[534, 846, 932, 1234, 1600]



(173) (174) (175)

### *4.2.1.7 3,4,5/4,5,6-Trihalogenopyridazines as Substrates (H 252)*

In 3,4,5-trihalogenopyridazines, electronic factors would place the 4-halogeno substituent marginally ahead of the 5-halogeno substituent in activity toward aminolysis, but steric considerations would favor the reverse order. Most of the typical examples that follow can be rationalized in the foregoing terms. 3,4,5-Trichloropyridazine (**178**) gave the following products:

A separable mixture of 3,5-dichloro- (**176**, R = H) and 5,6-dichloro-4-pyridazinamine (**177**, R = H) ($NH_3$, EtOH, 125°, sealed, 5 h: 33% and 26%, respectively).[1820]

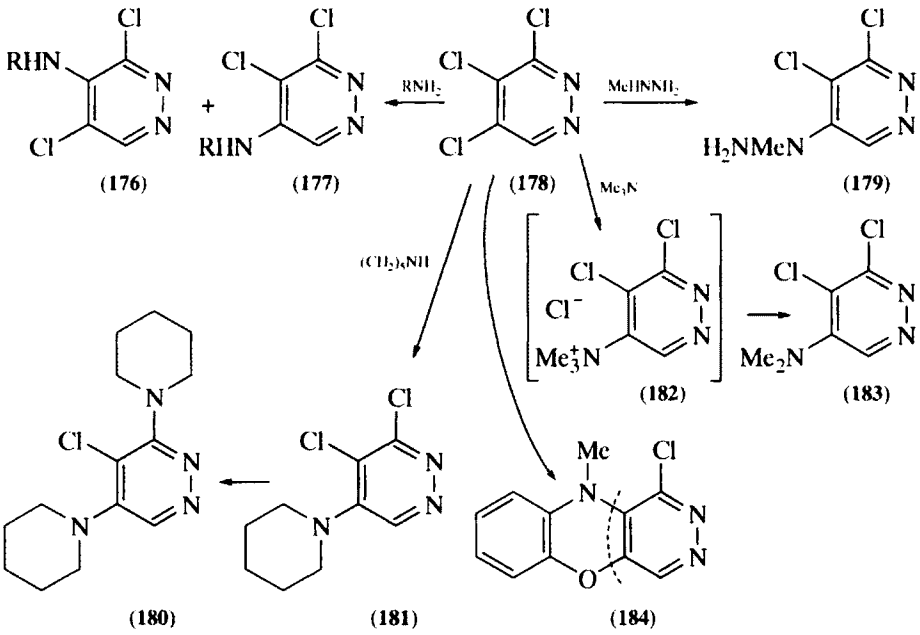
A separable mixture of 3,5-dichloro-4-isopropylamino- (**176**, R = $Pr^i$) and 3,4-dichloro-5-isopropylaminopyridazine (**177**, R = $Pr^i$) ($Pr^iNH_2$, PhMe, reflux, 3 h: ~40% and ~45%, respectively).[1917]

3,4-Dichloro-5-α-methylhydrazinopyridazine (**179**) ($MeHNNH_2$, MeOH, 0° → 25°, 5 h: 48%).[1061]

3,4-Dichloro-5-piperidinopyridazine (**181**) [$(CH_2)_5NH$, EtOH, 25°, 1 h: 50%] or 4-chloro-3,5-dipiperidinopyridazine (**180**) [neat $(CH_2)_5NH$, 95°, 2 h: 52%].[1058]

3,4-Dichloro-5-dimethylaminopyridazine (**183**) [$Me_3N$, PhH, < 25°, 2 days: ~3%; via the intermediate (**182**)].[1796]

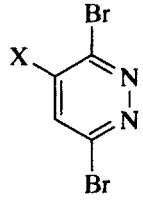
1-Chloro-10-methyl-10*H*-pyridazino[4,5-*b*][1,4]benzoxazine (**184**) ($HOC_6H_4NHMe$-*o*, see original for conditions: > 23%); likewise homologues.[701]




## *4.2.1.8 3,4,6/3,5,6-Trihalogenopyridazines as Substrates (H 252)*

Subject to steric considerations, 3,4,6-trihalogenopyridazines should undergo aminolysis first at the 4 position and subsequently at the 6 position; this postulate is confirmed by the very limited number of recent reports, typified in the following examples:

3,4,6-Tribromo- (**185**, X = Br) or 3,6-dibromo-4-chloropyridazine (**185**, X = Cl) gave 3,6-dibromo-4-pyridazinamine (**185**, X = $NH_2$) ($NH_3$, EtOH, 100°, sealed, 2 or 5 h respectively: 64% or 60%, respectively).[447]

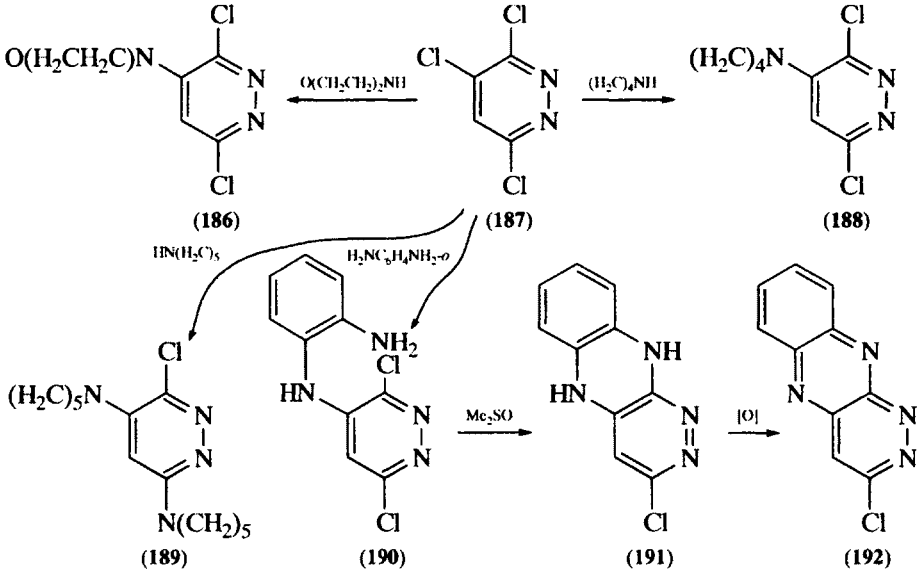


(**185**)

3,4,6-Trichloro- (**187**) gave 3,6-dichloro-4-morpholinopyridazine (**186**) [$O(CH_2CH_2)_2NH$, EtOH, reflux, 30 min: < 10%;[544] $O(CH_qCH_2)_2NH$, PhH, reflux, 4 h: ~90%],[637] 3,6-dichloro-4-pyrrolidin-1′-yl-pyridazine (**188**) [$(CH_2)_4NH$, $Na_2CO_3$, $CH_2Cl_2$, 25°, 15 h: ~55%],[410] or 3-chloro-4,6-dipiperidinopyridazine (**189**) [$(CH_2)_5NH$, EtOH, reflux, 4 h; ~30%].[544]

3,4,6-Trichloro- (**187**) gave mainly 3,6-dichloro-4-dimethylaminopyridazine ($Me_3N$, PhH, 25°, 2 days: ~15%; produced via the corresponding dichlorotrimethylammoniopyridazine chloride).[1796]

3,4,6-Trichloropyridazine (**187**) gave 4-*o*-aminoanilino-3,6-dichloropyridazine (**190**) ($H_2NC_6H_4NH_2$-*o*, AcONa, EtOH: > 30%)[610, 1065, cf. 1185] and then 3-chloro-5,10-dihydropyridazino[3,4-*b*]quinoxaline (**191**) ($Me_2SO$, 25°, 2 days: > 95%).[1065] which underwent oxidation to 3-chloropyridazino[3,4-*b*]quinoxaline (**192**) ($Na_2Cr_2O_7$, $H_2SO_4$, 25°, 3 h: 30%;[1065] also analogous cyclizations.[723, 730]



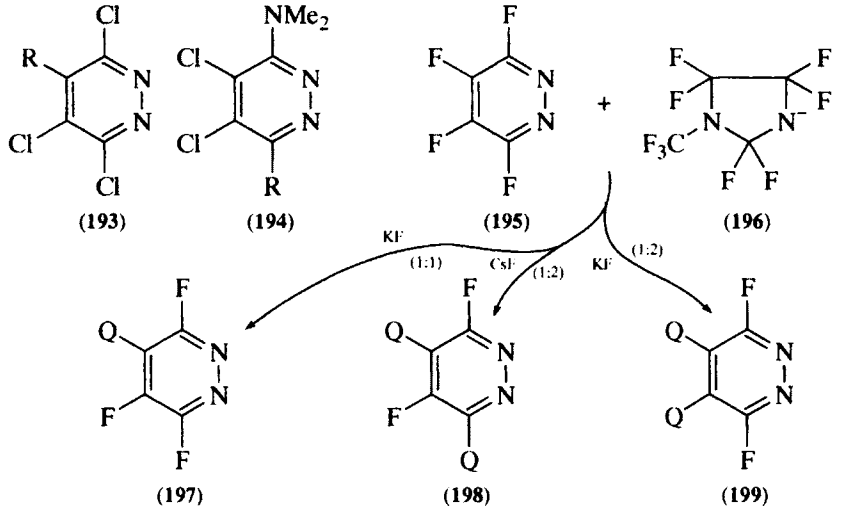
### *4.2.1.9 3,4,5,6-Tetrahalogenopyridazines as Substrates (H 254)*

The 4- or 5-halogeno substituent might be expected to undergo aminolysis more readily than the 3 or 6 substituent in tetrahalogenopyridazines but there are insufficient recent examples even to confirm this postulate. Available experimental data follow:

3,4,5,6-Tetrachloropyridazine (**193**, R = Cl) gave only 3,4,6-trichloro-5-morpholinopyridazine [**193**, R = $O(CH_2CH_2)_2N$] [$O(CH_2CH_2)_2NH$, PhH, reflux, 4 h: ?%].[637] or, in contrast, a separable mixture of 3,4,5-trichloro-6-dimethylamino- (**194**, R = Cl) and 4,5-dichloro-3,6-bisdimethylaminopyridazine (**194**, R = $NMe_2$) ($Me_3N$, PhH, 8 days; major and minor products, respectively; clearly via appropriate quaternary intermediates).[1796]

3,4,5,6-Tetrafluoropyridazine (**195**) with the anion (**196**) (generated in situ) gave 3,4,6-trifluoro-5-perfluoro(3-methylimidazolidin-1-yl)pyridazine (**197**) (reactants 1:1, KF, tetrahydrothiophene dioxide, 60°, 24 h: 57%), 3,5-difluoro-4,6-bis[perfluoro(3-methylimidazolidin-1-yl)]pyridazine (**198**) (reactants 1:2, CsF, MeCN, 25°, 22 h: 83%; similarly but 85°, 5 h; 92%), or 3,6-difluoro-4,5-bis[perfluoro(3-methylimidazolidin-1-yl)]pyridazine (**199**) (reactants 1:2, KF, MeCN, 25°, 17 h; 71%).[460]

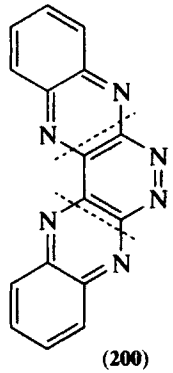
3,4,5,6-Tetrachloropyridazine with *o*-phenylenediamine (2 equiv) gave pyridazino[3,4-*b*:5,6-*b'*]diquinoxaline (**200**) via spontaneous aerial oxidation of its 5,8,13,14-tetrahydro derivative (*N*-methylpyrrolidin-2-one, 115°, air, 17 h; 14% after separation from a second product).[257,1233,1932]




(Q = perfluoro-3-methylimidazoliden-1-yl: from **196**)

### 4.2.2 Hydrolysis of Nuclear Halogenopyridazines (*H* 256)

Probably because most halogenopyridazines are made from corresponding pyridazinones, the reverse process of hydrolysis is seldom used. When it has been employed recently, near-glacial acetic acid has been used more often than has aqueous/alcoholic mineral acid or aqueous/alcoholic alkali; selective monohydrolysis of a di- or trihalogenopyridazine is possible sometimes, and the only reported kinetics are for the alkaline hydrolysis of several 2-alkyl/aryl-4,5-dichloro-3(2*H*)-pyridazinones (**201**, X = Cl) into 2-alkyl/aryl-4-chloro-5-hydroxy-3(2*H*)-pyridazinones (**201**, X = OH).[1044] The following examples illustrate the three main direct hydrolytic procedures for the conversion of halogenopyridazines into the corresponding pyridazinones: the choice of method is often clearly indicated by the presence of a sensitive passenger group:

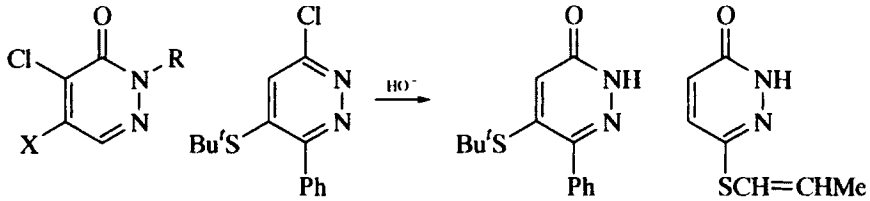


(**200**)

#### Using Aqueous/Ethanolic Alkali

4-*t*-Butylthio-6-chloro-3-phenylpyridazine (**202**) gave 5-*t*-butylthio-6-phenyl-3(2*H*)-pyridazinone (**203**) (NaOH, PrOH–$H_2O$, reflux, 24 h: 42%).[1087]

3-Allylthio-6-chloropyridazine gave 6-prop-1′-enylthio-3(2*H*)-pyridazinone (**204**) (KOH, MeOH–$H_2O$, reflux, $N_2$, 2 h: 52%; note concomitant isomerization of the allylthio group).[557]



(**201**) (**202**) (**203**) (**204**)

4,5-Dichloro- gave 4-chloro-5-hydroxy-3(2*H*)-pyridazinone (8M, KOH, reflux, 6 h: 54%).[90]

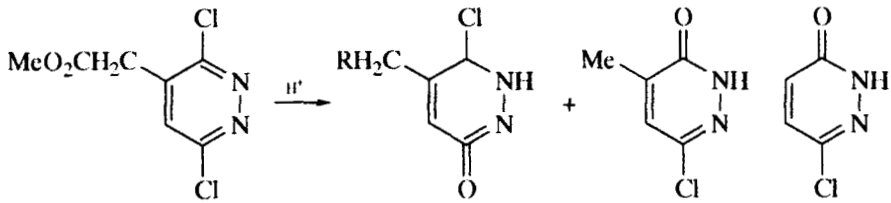
Also other examples.[553, 694, 776, 871, 1439]

### Using Aqueous / Alcoholic Mineral Acid

3,6-Dichloro-4-methoxycarbonylmethylpyridazine (**205**) gave a separable mixture of 5-carboxymethyl-6-chloro-3(2*H*)-pyridazinone (**206**, R = $CO_2H$), 6-chloro-5-methyl-3(2*H*)-pyridazinone (**206**, R = H), and 6-chloro-4-methyl-3(2*H*)-pyridazinone (**207**) (6M HCl, 90°, 1 h; 47%, 15%, and 31%, respectively; note nonselective hydrolysis of one chloro substituent, ester hydrolysis, and decarboxylation).[947]

3,6-Dichloropyridazine gave 6-chloro-3(2*H*)-pyridazinone (**208**) (aqueous acid, for details, see original).[644]

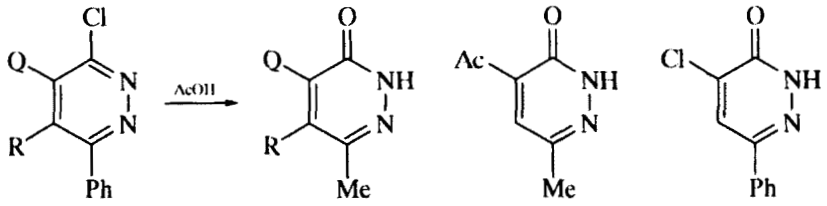
Also other somewhat irregular examples.[710, 755, 909]




(**205**) (**206**) (**207**) (**208**)

### Using Neat or Buffered Acetic Acid

3-Chloro-5-methyl-6-phenylpyridazine (**209**, Q = H, R = Me) gave 5-methyl-6-phenyl-3(2*H*)-pyridazinone (**210**, Q = H, R = Me) (AcOH, reflux, 3 h: 63%);[1557] also the 4-methyl substrate (**209**, Q = Me, R = H) gave the 4-methyl product (**210**, Q = Me, R = H) [AcONa, AcOH, 185° (bath?), 90 min: ~75%].[1104]




(**209**) (**210**) (**211**) (**212**)

4-Acetyl-3-chloro-6-methylpridazine gave 4-acetyl-6-methyl-3(2*H*)-pyridazinone (**211**) (AcOH, reflux, < 8 h: 97%); also analogues.[1514]

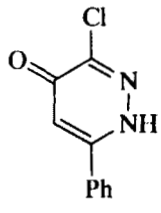
3,4-Dichloro-6-phenylpyridazine gave a separable mixture of 4-chloro-6-phenyl-3(2*H*)-pyridazinone (**212**) and 3-chloro-6-phenyl-4(1*H*)-pyridazi-

none (**213**) (AcOH, reflux, 6 h: ~45% and ~12%, respectively; note the apparent preference for hydrolysis of the 3-chloro substituent);[1041] 3,4-dichloro-6-*p*-(imidazol-1-yl)phenylpyridazine likwise gave 4-chloro-6-*p*-(imidazol-1-yl)phenyl-3(2*H*)-pyridazinone as the main product (AcOH, reflux, 5 h: 67%).[1073]

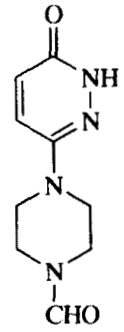
3-Chloro-6-(4-formylpiperazin-1-yl)pyridazine gave 6-(4-formylpiperazin-1-yl)-3(2*H*)-pyridazinone (**214**) (AcOH, reflux, 8 h: 60%).[1761]

7-(6-Chloropyridazin-3-yl)-1,3-dimethyl-2,6(1*H*,3*H*)-purinedione gave 1,3-dimethyl-7-(6-oxo-1,6-dihydropyridazine-3-yl)-2,6-(1*H*,3*H*)-purinedione (**215**) (AcOH, reflux, 8 h: 80%).[1730]

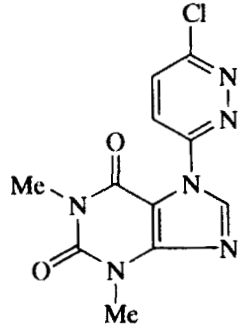
Also other examples.[522,654,1197,1733,1737,2055]



(213)



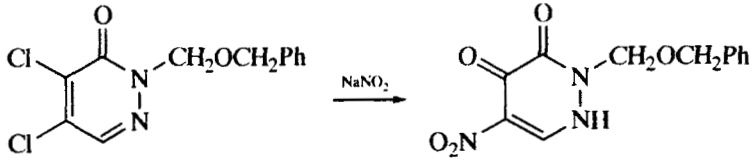
(214)



(215)

## Using Miscellaneous Procedures

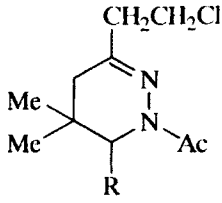
2-Benzyloxymethyl-4,5-dichloro-3(2*H*)-pyridazinone (**216**) gave 2-benzyloxymethyl-5-nitro-3,4(1*H*,2*H*)-pyridazinedione (**217**) ($NaNO_2$, $Me_2NCHO$-$H_2O$, 85°, 24 h: 93%).[1048]



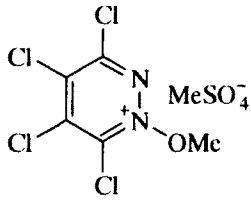
(216) (217)

1-Acetyl-6-chloro-3-β-chloroethyl- (**218**, R = Cl) gave 1-acetyl-3-β-chloroethyl-6-hydroxy-5,5-dimethyl-1,4,5,6-tetrahydropyridazine (**218**, R = OH) (chromatography, silica gel, EtOH–$C_6H_{14}$: ~85%).[1824]

Crude 3,4,5,6-tetrachloro-1-methoxypyridazinium methyl sulfate (**219**) gave a separable mixture of six products, including 4,5,6-trichloro-2-methoxy-3(2*H*)-pyridazinone as the main component ($H_2O$, 10°, 1 h: 57%).[893]



(218)



(219)

### 4.2.3 Alcoholysis of Nuclear Halogenopyridazines (*H* 260)

As the method of choice for preparing alkoxy- and aryloxypyridazines, the alcoholysis of halogenopyridazines is a frequently used process. Alkoxide ions are excellent nucleophiles that attack almost all halogenopyridazines quite readily; indeed, it is often difficult in practice to achieve selectivity on a di- or polyhalogenopyridazine. Although no kinetic studies have been reported recently, older rate studies[1935] and qualitative observations do suggest that passenger groups can play significant roles in the alcoholysis of halogenopyridazines. These points are illustrated in the following examples, grouped according to the position(s) of the halogeno substituents:

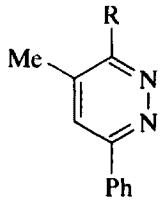
#### 3/6-Monohalogenopyridazines as Substrates

3-Chloro- (**220**, R = Cl) gave 3-methoxy- (**220**, R = OMe) (MeONa, MeOH, reflux, 6 h; 92%;[1104] MeONa, $Me_2NCHO$, reflux, 12 h: 90%)[1632] or 3-butoxy-4-methyl-6-phenylpyridazine (**220**, R = OBu) ($NaNH_2$, BuOH, reflux, 48 h: 82%).[1279]

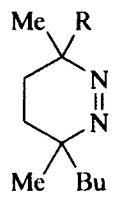
3-Bromo-6-butyl- (**221**, R = Br) gave 3-butyl-6-methoxy-3,6-dimethyl-3,4,5,6-tetrahydropyridazine (**221**, R = OMe) (MeONa, MeOH, reflux, $N_2$, 5 h: 60%).[1201]

3-Benzoyl-6-chloropyridazine (**222**, R = Cl) gave 3-benzoyl-6-β-dimethylaminoethoxypyridazine (**222**, R = $OCH_2CH_2NMe_2$) (neat $Me_2NCH_2CH_2OH$, NaH, A, 25°, 30 min; 61%).[1698]

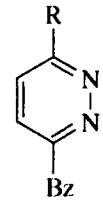
3-Chloro- (**223**, R = Cl) gave 3-methoxy-4-pyridazinecarbonitrile (**223**, R = OMe) (MeONa, MeONa, THF, 25°, ?h: only 7% after purification).[1300]



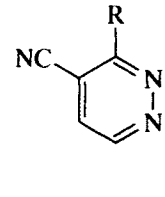
(220)



(221)



(222)



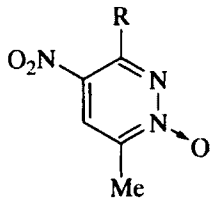
(223)

3-Chloro- (**224**, R = Cl) gave 3-methoxy-6-methyl-4-nitropyridazine 1-oxide (**224**, R = OMe (MeONa, MeOH, −5°, 1 h: 62%; note the highly activated chloro substituent).[952]

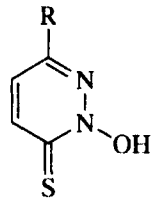
6-Chloro-2-hydroxy- (**225**, R = Cl) gave 2-hydroxy-6-methoxy-3(2*H*)-pyridazinone (**225**, R = OMe) (MeONa, MeOH, reflux, 2 h: 54%).[679]

3-Chloro-5-methoxy- (**226**, R = Cl) gave 3,5-dimethoxypyridazine (**226**, R = OMe) (KOH, MeOH, $N_2$, 25°, 20 h: 98%).[1821]

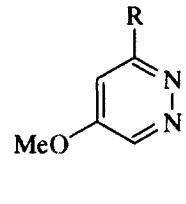
3-Chloro- (**227**, R = Cl) gave 3-benzyloxy-6-dimethylaminopyridazine (**227**, R = $OCH_2Ph$) ($PhCH_2ONa$, $PhCH_2OH$, 190°, 4 h: 80%; note extreme conditions because of deactivation by the dimethylamino group)[94] or 3-dimethylamino-6-methoxypyridazine (**227**, R = OMe) (MeONa, MeOH, reflux, 2 days: 65%).[137]



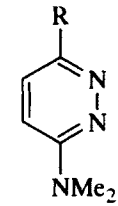
(**224**)



(**225**)



(**226**)



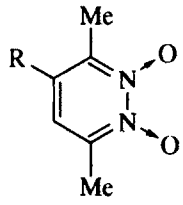
(**227**)

6-Chloro-3-pyridazinamine gave 6-phenoxy-3-pyridazinamine (PhONa, $H_2O$, 170°, sealed, 16 h: ~40%),[1150] numerous 6-(substituted phenoxy) analogues (likewise: comparable yields)[1150,1155,1157,1159] or 6-propoxy-3-pyridazinamine (PrONa, PrOH, 145°, sealed, 14 h: 96%).[1841]

Also other examples.[304,363,377,465,517,605,606,673,688,707,761,769,801,805,844,913,924,975,1087,1103,1250,1251,1266,1275,1311,1330,1367,1371,1592,1628,1703]

## 4/5-Monohalogenopyridazines as Substrates

4-Chloro- (**228**, R = Cl) gave 4-methoxy- (**228**, R = OMe) (MeONa, MeOH, reflux, 1 h: 40%) or 4-benzyloxy-3,6-dimethylpyridazine 1,2-dioxide (**228**, R = $OCH_2Ph$) ($PhCH_2ONa$, $PhCH_2OH$, 25°, 2 days; 5% after purification).[796]



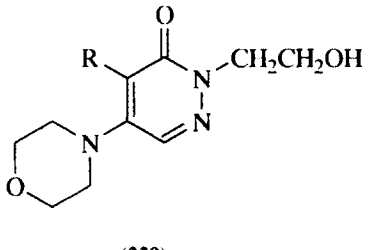
(**228**)

4-Chloro- (**229**, R = Cl) gave 4-ethoxy-2-β-hydroxyethyl-5-morpholino-3(2*H*)-pyridazinone (**229**, R = OEt) (EtONa, EtOH, reflux, 6 h: 48%);[534]

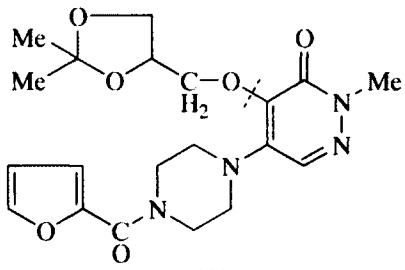
likewise, the analogous 4-ethoxy-2-methyl-5-morpholino-3(2*H*)-pyridazinone (70%).[1269]

4-Bromo- or 4-chloro-5-[4-(fur-2-oyl)piperazin-1-yl]-2-methyl-3(2*H*)-pyridazinone with 4-hydroxymethyl-2,2-dimethyl-1,3-dioxolane (isopropylideneglycerol) gave 4-(2,2-dimethyl-1,3-dioxolan-4-yl)methoxy-5-[4-(fur-2-oyl)piperazin-1-yl]-2-methyl-3(2*H*)-pyridazinone (**230**) (NaH, $Me_2NCHO$, 90°, 30 min; then substrate ↓, 90°, 2 h: 90% or 88%, respectively);[515] also analogous reactions.[48]

Also other examples.[529,540,705,794,848]



(229)



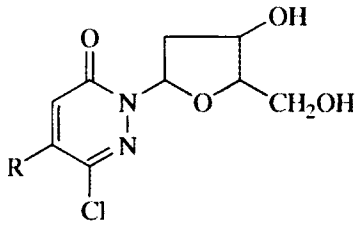
(230)

## 3,4/5,6-Dihalogenopyridazines as Substrates

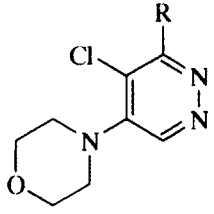
5,6-Dichloro- (**231**, R = Cl) gave 6-chloro-5-methoxy-2-β-D-ribofuranosyl-3(2*H*)-pyridazinone (**231**, R = OMe) (MeONa, MeOH–THF, 10°, 48 h: 91%).[1025]

3,4-Dichloro- (**232**, R = Cl) gave 3-butoxy-4-chloro-5-morpholinopyridazine (**232**, R = OBu) (BuONa, BuOH, reflux, 10 h: ~90%; note unexpected activity of the 3-Cl, probably due to the deactivating and steric effects of the morpholino group on the adjacent 4-Cl substituent).[637]

Also other examples.[861,881]



(231)



(232)

## 3,5/4,6-Dihalogenopyridazines as Substrates

3,5-Dichloro-6-methoxypyridazine gave 3-chloro-5,6-dimethoxypyridazine (MeONa, MeOH, reflux, 1 h: 91%).[881]

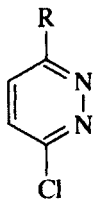
### 3,6-Dihalogenopyridazines as Substrates

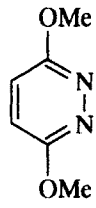
3,6-Dichloro- (**233**, R = Cl) gave 3-chloro-6-ethoxy- (**233**, R = OEt) (EtONa, EtOH, exothermic, 30 min: 90%),[99,cf.1258,1933] 3-chloro-6-β-methoxyethoxy- (**233**, R = $OCH_2CH_2OMe$) ($MeOCH_2CH_2ONa$, THF, A, 25°, 30 min: 88%),[1421] or 3-β-anilinoethoxy-6-chloropyridazine (**233**, R = $OCH_2CH_2NHPh$) ($PhNHCH_2CH_2OH$, $K_2CO_3$, $Me_2NCHO$, 100°, 10 h: 50%).[1258]

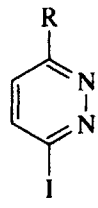
3,6-Dichloro- (**233**, R = Cl) gave 3,6-dimethoxypyridazine (**234**) (NaOH, MeOH, "prolonged refluxing": ?%).[644,cf.1933]

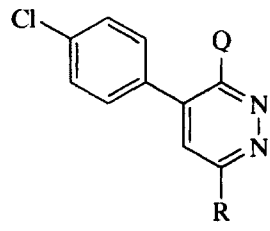
3,6-Diiodo- (**235**, R = I) gave 3-iodo-6-methoxypyridazine (**235**, R = OMe) (MeONa, MeOH, reflux, 12 h: 92%).[1385]

3,6-Dichloro- (**233**, R = Cl) gave 3-*o*-allyloxyphenoxy-6-chloro- (**233**, R = $OC_6H_4OCH_2CH{:}CH_2$-*o*) ($H_2C{=}CHCH_2C_6H_4OH$-*o*, KOH, $Me_2SO$, 50°, 3 h: 85%),[830] 3-chloro-6-*o*-hydroxyphenoxypyridazine (**233**, R = $OC_6H_4OH$-*o*) ($HOC_6H_4OH$-*o*, $K_2CO_3$, $Me_2NCHO$, 100°, 1 h: 47%),[1466] or 3-but-3′-ynyloxy-6-chloropyridazine (**233**, R = $OCH_2$ $CH_2C{\equiv}CH$) [$HC{\equiv}CCH_2CH_2ONa$ (made in situ), THF, 0° → 25°, 2 h: 82%].[336]


(233)


(234)

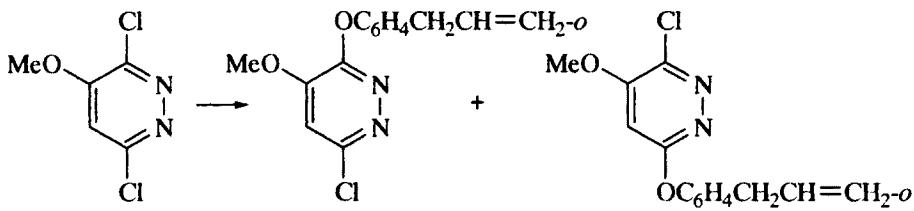

(235)


(236)

3,6-Dichloro-4-*p*-chlorophenyl- (**236**, Q = R = Cl) gave 3-chloro-4-*p*-chlorophenyl-6-methoxy- (**236**, Q = Cl, R = OMe) (MeONa, MeOH, reflux, 30 min: 83%) or 4-*p*-chlorophenyl-3,6-dimethoxypyridazine (**236**, Q = R = OMe) (MeONa, MeOH, 145°, sealed, 24 h: 62%).[461]

3,6-Dichloro-4-methoxypyridazine (**237**) with sodium *o*-allylphenoxide gave a separable mixture of 3-*o*-allylphenoxy-6-chloro-4-methoxypyridazine (**238**) and 3-*o*-allylphenoxy-6-chloro-5-methoxypyridazine (**239**) (PhMe, 50°, 2 h; then reflux 1 h: 19% and 46%, respectively).[766]

Also other examples.[359,636,641,654,677,881,1169,1209,1478,1519,1899,2102]


(237) (238) (239)

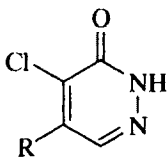
### 4,5-Dihalogenopyridazines as Substrates

*Note*: All available recent examples in this category are 4,5-dihalogenopyridazinones.

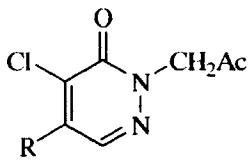
4,5-Dichloro- (**240**, R = Cl) gave only 4-chloro-5-methoxy-3(2*H*)-pyridazinone (**240**, R = OMe) (MeONa, MeOH, reflux, 3 days: ~95%).[90]

2-Acetonyl-4,5-dichloro- (**241**, R = Cl) gave, not the expected nitrile (**241**, R = CN), but 2-acetonyl-4-chloro-5-methoxy-3(2*H*)-pyridazinone (**241**, R = OMe) (KCN, MeOH, 40°, 2 h: 53%).[1673]

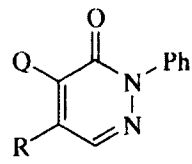
4,5-Dichloro- (**242**, Q = R = Cl) gave 4-chloro-5-ethoxy (**242**, Q = Cl, R = OEt) (EtONa, EtOH, reflux, 30 min: 95%), 5-chloro-4-ethoxy- (**242**, Q = OEt, R = Cl) (EtONa, EtOH–dioxane, 25°, 16 h: 78%), or a separable mixture of 4-chloro-5-isopropoxy- (**242**, Q = Cl, R = $OPr^i$ and 5-chloro-4-isopropoxy-2-phenyl-3(2*H*)-pyridazinone (**242**, Q = $OPr^i$, R = Cl) (NaH, $Pr^iOH$, 25°, 30 min: 38% and 50%, respectively);[1089] the reaction medium appears to have been crucial to selectivity in the foregoing and related examples.[1089]



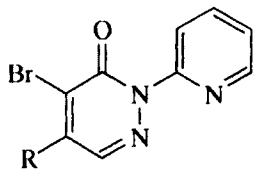
(**240**)



(**241**)



(**242**)



(**243**)

4,5-Dibromo- (**243**, R = Br) gave 4-bromo-5-methoxy-2-pyridin-2′-yl-3(2*H*)-pyridazinone (**243**, R = OMe) (MeONa, MeOH, reflux, >24 h: 81%).[1234]

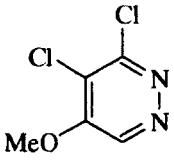
4,5-Dichloro- gave 4,5-diethoxy-2-methyl-3(2*H*)-pyridazinone (excess EtONa, EtOH, reflux, 6 h: 66%).[531]

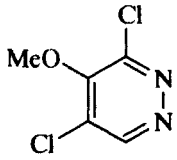
Also other examples.[764,932,1688,1936]

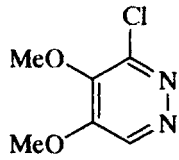
### 3,4,5 / 4,5,6-Trihalogenopyridazines as Substrates

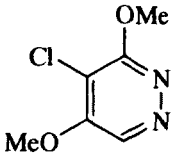
3,4,5-Trichloropyridazine (**244**) gave a separable mixture of 3,4-dichloro-5-methoxy- (**245**) and 3,5-dichloro-4-methoxypyridazine (**246**) [MeONa (1 equiv), MeOH, 0° → 25°, 1 h: 55% and 24%, respectively];[882, cf.689] a separable mixture of 3-chloro-4,5-dimethoxy- (**247**), 4-chloro-3,5-dimethoxy- (**248**), and 3,4,5-trimethoxypyridazine (**249**) [MeONa (2 equiv), MeOH, 25° → reflux, 90 min: ~42%, ~25%, and ~2%, respectively];[689] or a separable mixture of 4-chloro-3,5-dimethoxy- (**248**) and 3,4,5-trimethoxy-pyridazine (**249**) (excess MeONa, MeOH, 25°, 15 h: ~24% and ~35%, respectively).[689]

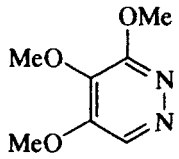

(244)

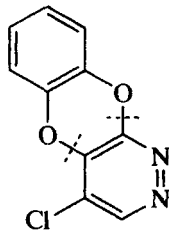

(245)


(246)


(247)


(248)


(249)


(250)

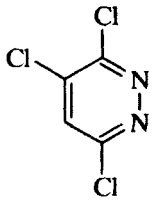
3,4,5-Trichloropyridazine (**244**) gave 4-chloro[1,4]benzodioxino[2,3-*c*]pyridazine (**250**) (neat $HOC_6H_4OH$-*o*, slowly to 195°: 44%).[1691]

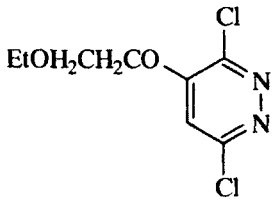
Also other examples.[640]

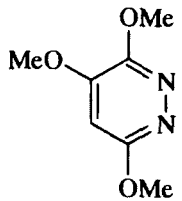
### 3,4,6/3,5,6-Trihalogenopyridazines as Substrates

3,4,6-Trichloro- (**251**) gave 3,6-dichloro-4-β-ethoxyethoxy- (**252**) ($EtOCH_2CH_2ONa$, $EtOCH_2CH_2OH$, 18°, 2 h: ~60%)[1796] or 3,4,6-trimethoxypyridazine (**253**) (excess MeONa, MeOH, 5° → reflux, 10 h: 55%, after separation from a byproduct).[881]

Also other examples.[640,917]


(251)


(252)


(253)


(254)

### 3,4,5,6-Tetrahalogenopyridazines as Substrates

3,4,5,6-Tetrafluoropyridazine (**254**, R = F) gave 3,4,6-trifluoro-5-heptafluoroisopropoxypyridazine [**254**, $R = CF(CF_3)_2$] [$(F_3C)_2CO$, CsF, $Me_2NCHO$, − 196°, then 110°, 3 days: 31%].[1664]

## 4.2.4 Thiolysis of Nuclear Halogenopyridazines (*H* 265)

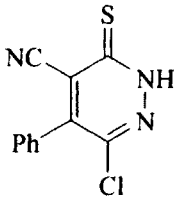
The conversion of halogenopyridazines into pyridazinethiones may be done by treatment with sodium hydrogen sulfide, thioures (via the isothiouronium

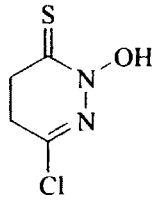
intermediate), or phosphorus pentasulfide (by a yet unresolved mechanism). These procedures are illustrated in the following recent examples.

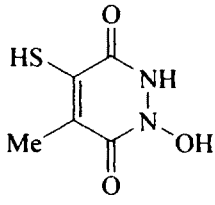
### Using Sodium Hydrogen Sulfide

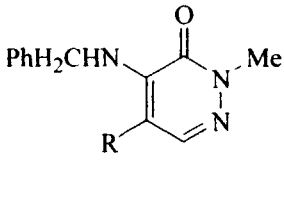
3-Chloro-5,6-diphenyl-4-pyridazinecarbonitrile gave 5,6-diphenyl-3-thioxo-2,3-dihydro-4-pyridazinecarbonitrile (**255**) (NaHS, EtOH, 25°, 30 min: 92%; note survival of the nitrile group under these mild conditions).[1913]

3,6-Dichloropyridazine 1-oxide gave 6-chloro-2-hydroxy-3(2*H*)-pyridazinethione (**256**) ($Na_2S$, $H_2O$–dioxane, 25°, 10 h: 76%; KHS, MeOH, 25°, 5 h: 70%).[679]


(255)


(256)


(257)


(258)

4,6-Dibromo-5-methyl-3(2*H*)-pyridazinone 1-oxide gave 1-hydroxy-4-mercapto-5-methyl-6-thioxo-1,6-dihydro-3(2*H*)-pyridazinone (**257**) (KHS, EtOH–$Me_2NCHO$, reflux, 2 h; ~40%).[833]

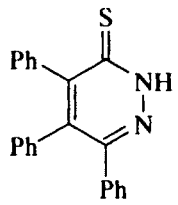
4-Benzylamino-5-chloro- (**258**, R = Cl) gave 4-benzylamino-5-mercapto-2-methyl-3(2*H*)-pyridazinone (**258**, R = SH) (NaHS, $Me_2NCHO$, 90°, 10 h: 71%).[867]

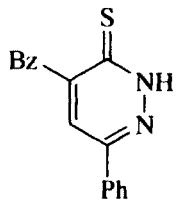
Also other examples.[315,539,923,1226,1724]

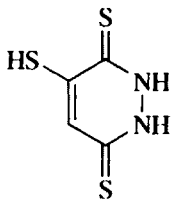
### Using Thiourea

3-Chloro-4,5,6-triphenylpyridazine gave 4,5,6-triphenyl-3(2*H*)-pyridazinethione (**259**) ($H_2NCSNH_2$, EtOH, 20°, 3 h; then 2.5M NaOH, reflux, 1 h; 70%).[1362]

4-Benzoyl-3-chloro-6-phenylpyridazine gave 4-benzoyl-6-phenyl-3(2*H*)-pyridazinethione (**260**) ($H_2NCSNH_2$, $H_2O$–EtOH, reflux, 10 h: 61%; no alkaline treatment needed to decompose isothiouronium salt).[1367]


(259)


(260)


(261)

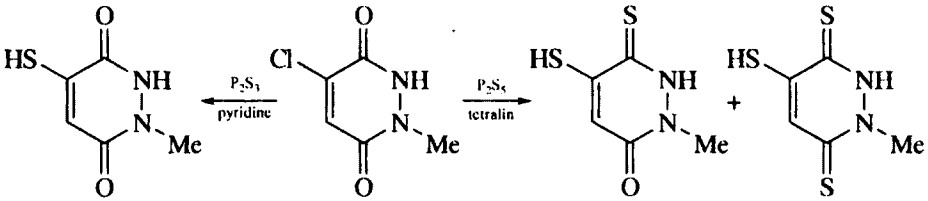
3,4,6-Trichloropyridazine gave 4-mercapto-3,6-(1*H*,2*H*)-pyridazinedithione (**261**) (excess $H_2NCSNH_2$, EtOH, reflux, 15 min; then 1M NaOH, 95°, 10 min: <65%).[1129]

Also other examples.[112,365,371,377,946,1812]

### Using Phosphorus Pentasulfide

4-Chloro-1-methyl-3,6(1*H*,2*H*)-pyridazinedione (**263**) gave 4-mercapto-1-methyl-3,6(1*H*,2*H*)-pyridazinedione (**262**) ($P_2S_5$, pyridine, reflux, 90 min: ~70%) or a mixture of 5-mercapto-2-methyl-6-thioxo-1,6-dihydro-3(2*H*)-pyridazinone (**264**) and 4-mercapto-1-methyl-3,6(1*H*,2*H*)-pyridazinedithione (**265**), both identified subsequently by *S*-methylation to known compounds ($P_2S_5$, tetralin, 180°, 3 h: ~70% as initial mixture);[1129] also other examples.[1129]

3-Amino-6-chloro-4-pyridazinecarboxamide gave 3-amino-6-thioxo-1,6-dihydro-4-pyridazinecarbothioamide ($P_2S_5$, pyridine, reflux, 90 min: 66%; note concomitant thiation of the amide grouping).[1099]




(262) (263) (264) (265)

## 4.2.5 Alkanethiolysis of Nuclear Halogenopyridazines (*H* 269, 760)

The conversion of halogeno- into alkylthio- or arylthiopyridazines occurs readily, but it is usually possible to achieve selective partial alkanethiolysis of di-, tri-, or even tetrahalogenopyridazines. The following typical examples, grouped according to the position(s) of the halogeno substituents, illustrate the conditions required for reasonable yields:

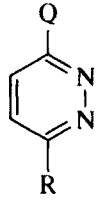
### 3/6-Monohalogenopyridazines as Substrates

3-Chloro-6-methoxypyridazine (**266**, Q = Cl, R = OMe) gave a separable mixture of 3-*t*-butylthio-6-methoxy- (**266**, Q = SBu$^t$, R = OMe) and 3,6-bis-*t*-butylthiopyridazine (**266**, Q = R = SBu$^t$) [Bu$^t$SNa] (made in situ), THF, reflux, 3 h: 56% and 10%, respectively; prolonged reflux gave a higher proportion of the latter product.[1219]

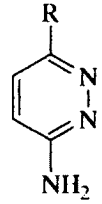
6-Chloro-3-pyridazinamine (**267**, R = Cl) gave 6-ethylthio- (**267**, R = SEt) (EtSH, 1M NaOH, 130°, sealed, 16 h: ~85%), 6-cyclohexylthio- (**267**, R = $SC_6H_{11}$) ($C_6H_{11}SH$, 0.6M NaOH, 140°, sealed, 20 h: ~80%), or

6-phenylthio-3-pyridazinamine (**267**, R = SPh) (PhSH, 1.7M NaOH, 130°, sealed, 18 h: ~75%);[1147] also many homologues.[1147, 1152, 1842]

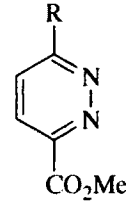
Methyl 6-chloro-3-pyridazinecarboxylate (**268**, R = Cl) gave methyl 6-ethylthio-3-pyridazinecarboxylate (**268**, R = SEt) (EtSNa, THF, reflux, 22 h: 62%).[1592]



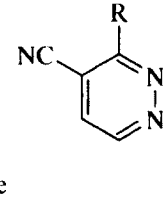

(**266**)




(**267**)




(**268**)




(**269**)

3-Chloro-4-pyridazinecarbonitrile (**269**, R = Cl) gave 3-*o*-aminophenylthio-4-pyridazinecarbonitrile (**269**, R = $SC_6H_4NH_2$-*o*) ($H_2NC_6H_2SH$-*o*, $NaHCO_3$, EtOH, 25°, A, 2 h: 67%; not enhanced activation of the chloro substituent by the adjacent cyano gruop).[1551]

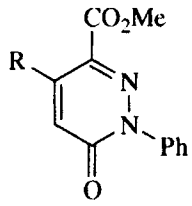
Also other examples.[146, 304, 517, 924, 1087, 1250, 1266, 1398, 1726, 2032, 2089]

## 4/5-Monohalogenopyridazines as Substrates

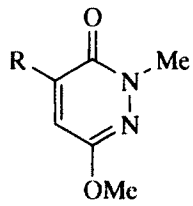
Methyl 4-chloro-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate (**270**, R = Cl) gave methyl 4-ethylthio- (**270**, R = SEt) (EtSH, $K_2CO_3$, $Me_2NCHO$, 25°, 1 h: 74%) or methyl 4-butylthio-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate (**270**, R = SBu) (BuSH, likewise: 96%);[1117] similarly homologues.[1117]

4-Chloro-6-methoxy-2-methyl- (**271**, R = Cl) gave 6-methoxy-2-methyl-4-phenylthio-3(2*H*)-pyridazinone (**271**, R = SPh) (PhSH, $Et_3N$, $CHCl_3$, 25°, 30 min: 75%);[1672] the isomeric 5-chloro substrate (**272**, R = Cl) likewise gave 6-methoxy-2-methyl-5-phenylthio-3(2*H*)-pyridazinone (**272**, R = SPh).[1672]

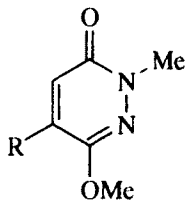
Also other examples.[848, 939, 1007, 1028, 1409, 1418, 1436]




(**270**)




(**271**)



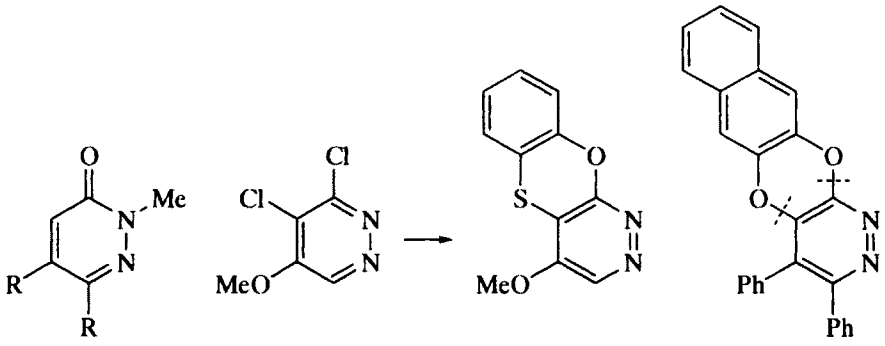

(**272**)

## 3,4/5,6-Dihalogenopyridazines as Substrates

5,6-Dichloro-2-methyl- (**273**, R = Cl) gave 2-methyl-5,6-bismethylthio-3(2*H*)-pyridazinone (**273**, R = SMe) (MeSNa, MeOH, 100°, sealed, 4 h: ~60%).[1129]

3,4-Dichloro-5-methoxypyridazine (**274**) with the disodium salt of *o*-mercaptophenol gave a single cyclocondensation product, identified as 4-methoxy[1,4]benzoxathiino[2,3-*c*]pyridazine (**275**) ($Me_2NCHO$, reflux, 9 h: 18%).[989]

Somewhat similarly, 3,4-dichloro-5,6-diphenylpyridazine with 2,3-naphthalenediol gave 3,4-diphenylnaphtho[2′,3′–5,6][1,4]dioxino[2,3-*c*]pyridazine (**276**) (see original for conditions and yield).[861]



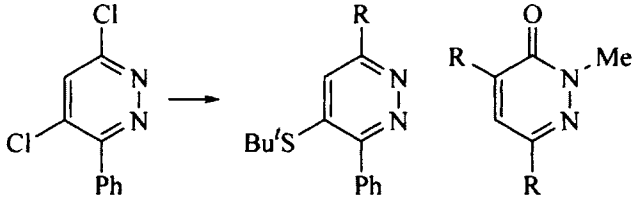
(273) (274) (275) (276)

## 3,5/4,6-Dihalogenopyridazines as Substrates

3,5-Dichloro-6-phenylpyridazine (**277**) gave either 4-*t*-butylthio-6-chloro-3-phenylpyridazine (**278**, R = Cl) [Bu′SNa (1 equiv), Pr′OH, 0° → 25°, 1 h: 74%] or 3,5-di-*t*-butylthio-6-phenylpyridazine (**278**, R = SBu′) [Bu′SNa (2 equiv), Pr′OH, 0° → reflux, 90 min: 76%].[1087]

4,6-Dichloro-2-methyl- (**279**, R = Cl) gave 2-methyl-4,6-bismethylthio-3(2*H*)-pyridazinone (**279**, R = SMe) (MeSNa, MeOH, 100°, sealed, 4 h: ~75%).[1129]

Also other examples.[863]



(277) (278) (279)

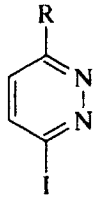
## 3,6-Dihalogenopyridazines as Substrates

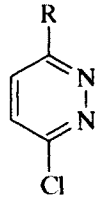
3,6-Diiodo- (**280**, R = I) gave 3-iodo-6-methylthiopyridazine (**280**, R = SMe) (MeSNa, EtOH, reflux, 12 h: 67%).[1385]

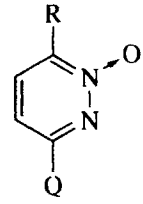
3,6-Dichloropyridazine (**281**, R = Cl) gave 3-chloro-6-*o*-hydroxyphenethylthiopyridazine (**281**, R = $SCH_2CH_2C_6H_4OH$-*o*) ($NaSCH_2CH_2C_6H_4OH$-*o*, PhH, 25°, 30 min: 70%)[1466] or 3-(β-amino-β-methylpropylthio)-6-chloropyridazine (**281**, R = $SCH_2CMe_2NH_2$) ($NaSCH_2Me_2NH_2$, Bu'OH–PhH, 30°, 1 h: 91%).[1478] Also analogues.[1116]

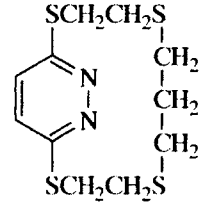
3,6-Dichloropyridazine 1-oxide (**282**, Q = R = Cl) gave 3-benzylthio-6-chloropyridazine 2-oxide (**282**, Q = Cl, R = $SCH_2Ph$) ($PhCH_2SH$, NaOH, $H_2O$–dioxane, ~5°, 2 h: 63%)[679] or 3,6-bismethylthiopyridazine pyridazine 1-oxide (**282**, Q = R = SMe) (MeSNa, EtOH, 5° → < 65°, then 75°, 3 h: 86%).[304]

3,6-Dichloropyridazine with 3,7-dithianonane-1,9-dithiol gave 2,5,9,12-tetrathia-14,15-diazabicyclo[11.2.2]heptadeca-13,15,16-triene (**283**) (EtONa, EtOH, 25°, 18 h: 72%; see original for further details);[1640] also analogous cyclic sulfides,[1639, 1641, 1657] all used as ligands for Ag and Cu complexes.


(280)


(281)


(282)

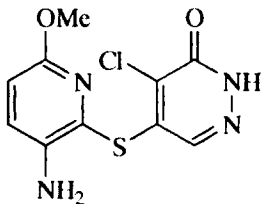

(283)

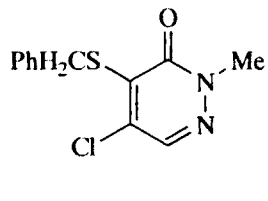
## 4,5-Dihalogenopyridazines as Substrates

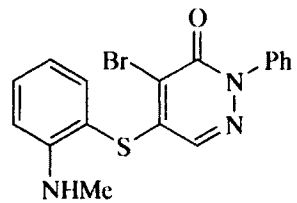
4,5-Dichloro-3(2*H*)-pyridazinone with 3-amino-6-methoxy-2(1*H*)-pyridinethione gave 5-(3-amino-6-methoxypyridin-2-ylthio)-4-chloro-3(2*H*)-pyridazinone (**284**) (NaOH, $H_2O$–$Me_2SO$, reflux, 1 h: 62%).[1024]

In contrast, 4,5-dichloro-2-methyl-3(2*H*)-pyridazinone with sodium phenylmethanethiolate (made in situ) gave 4-benzylthio-5-chloro-2-methyl-3(2*H*)-pyridazinone (**285**) ($PhCH_2SNa$, PhMe, 25°, 7 h: 74%).[1903]

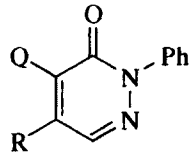
4,5-Dibromo-2-phenyl-3(2*H*)-pyridazinone with *o*-methylaminophenol gave 4-bromo-5-*o*-methylaminophenylthio-2-phenyl-3(2*H*)-pyridazinone (**286**) (NaOH, EtOH–$H_2O$, 25°, 12 h: 92%);[545] also analogues.[545, 548, 550]


(284)


(285)


(286)

4,5-Dichloro-2-phenyl-3(2*H*)-pyridazinone (**287**, Q = R = Cl) gave either 4-chloro-5-ethylthio- (**287**, Q = Cl, R = SEt) (EtSH, KOH, $H_2O$–dioxane, 25°, 15 min: 84%) or 5-chloro-4-ethylthio-2-phenyl-3(2*H*)-pyridazinone (**287**, Q = SEt, R = Cl) [EtSNa (made in situ), dioxane, 25°, 1 h: 78%];[1089] the same substrate (**287**, Q = R = Cl) gave either a separable mixture of 4-chloro-5-*o*-nitrophenylthio- (**287**, Q = Cl, R = $SC_6H_4NO_2$-*o*) and 4,5-bis(*o*-nitrophenylthio)-2-phenyl-3(2*H*)-pyridazinone (**287**, Q = R = $SC_6H_4NO_2$-*o*) ($HSC_6H_4NO_2$-*o*, MeONa, MeOH, 20°, 75 min: ~40% and ~10%, respectively)[768] or only the second product (**287**, Q = R = $SC_6H_4NO_2$-*o*) [$HSC_6H_4NO_2$-*o* (2 equiv), MeONa, MeOH, 50°, 1 h; 75%].[1774]

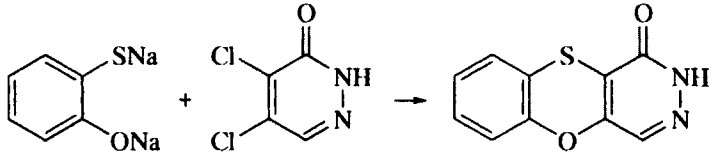


(**287**)

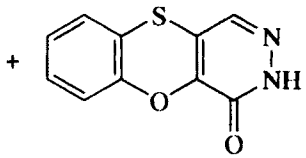
4,5-Dichloro-2-methyl- gave 2-methyl-4,5-bismethylthio-3(2*H*)-pyridazinine (MeSNa, EtOH–$H_2O$, 120°, sealed, 4 h: ~60%).[689]

4,5-Dichloro-3(2*H*)-pyridazinone (**289**) with the disodium salt of *o*-mercaptophenol (**288**) gave a separable mixture of [1,4]benzoxathiino[2,3-*d*]pyridazin-1(2*H*)-one (**290**) and [1,4]benzoxathiino[2,3-*d*]pyridazin-4(3*H*)-one (**291**) ($Me_2NCHO$, −15°, then 25°, 18 h: 22% and 26%, respectively; a single intermediate was postulated!).[1014]

Also other examples.[205,528,533,547,549,556,892,1234]



(**288**) (**289**) (**290**)



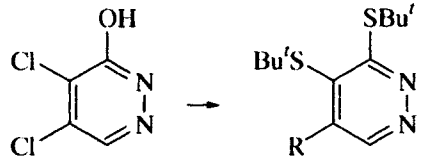
(**291**)

## 3,4,5 / 4,5,6-Trihalogenopyridazines as Substrates

3,4,5-Trichloropyridazine (**292**) gave a separable mixture of 3,4-bis-*t*-butylthio-5-chloro- (**293**, R = Cl) and 3,4,5-tris-*t*-butylthiopyridazine

(**293**, R = SBu$^t$) [Bu$^t$SNa (made in situ), THF, reflux, 5 min; 16% and 34%, respectively; both products unstable].[1219]

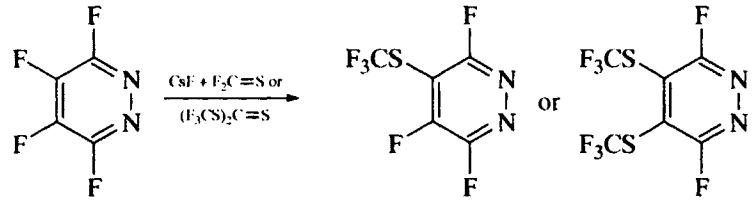
Also other relevant material.[993]



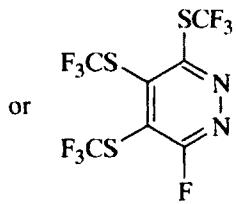
(**292**) (**293**)

### 3,4,5,6-Tetrahalogenopyridazines as Substrates

3,4,5,6-Tetrafluoropyridazine (**294**) gave 3,4,6-trifluoro-5-trifluoromethylthiopyridazine (**295**) [CsF, $F_2C{=}S$ (1 equiv), MeCN, −10° → 20°, 90 min: 89%; CsF, $(F_3CS)_2C{=}S$ (1 equiv), −5° → 20°, 1 h: 72%], 3,6-difluoro-4,5-bistrifluoromethylthiopyridazine (**296**) [CsF, $F_2C{=}S$ (2 equiv), MeCN, −10° → 20°, 12 h: 82%; CsF, $(F_3CS)_2C{=}S$ (2 eqiuiv), −5° → 20°, 1 h: 61%], or a separable 9:1 mixture of the foregoing product (**296**) and 3-fluoro-4,5,6-tristrifluoromethylthiopyridazine (**297**), from which the latter was isolated in < 10% yield [CsF, $(F_3CS)_2C{=}S$ (4 equiv), −5 → 20°, 12 h].[748]



(**294**) (**295**) (**296**)

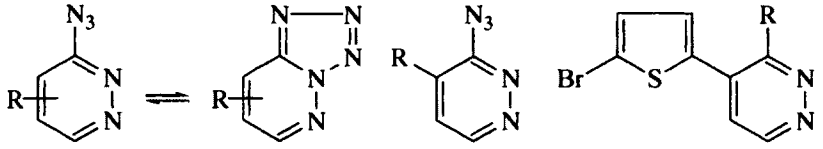


(**297**)

## 4.2.6 Conversion of Nuclear Halogeno- into Azidopyridazines (*H* 270)

Halogenopyridazines are easily converted into corresponding azidopyridazines by treatment of sodium azide in an appropriate solvent. The

azidopyridazines (**298**), resulting from 3/6-halogenopyridazines with an unsubstituted ring nitrogen adjacent to the halogeno substituent, are generally considered to exist as their tertazolo[1,5-*b*]pyridazine tautomers (**299**) see Section 7.3.4 but (for the sake of simplicity) such compounds are named and formulated as azidopyridazines in this book. The following examples illustrate the conditions needed for such conversions:



(298) (299) (300) (301)

### 3/6-Monohalogenopyridazines as Substrates

3-Chloro-4-phenyl- gave 3-azido-4-phenylpyridazine (**300**, R = Ph) ($NaNH_3$, $Me_2NCHO$, 120°, 20 h: 68%).[1408]

3-Chloro- gave 3-azido-4-pyridazinecarbonitrile (**300**, R = CN) ($NaN_3$, $Me_2NCHO$, 25°, 4 h: 80%).[1079]

4-(5-Bromothien-2-yl)-3-chloropyridazine (**301**, R = Cl) gave 3-azido-4-(5-bromothien-2-yl)pyridazine (**301**, R = $N_3$) ($NaN_3$, $Me_2NCHO$, 95°, 12 h: 63%; note survival of the ω-Br substituent).[975]

Also other examples.[133,377,1022,1034,1250,1251,1275,1335,1371,1417,1871,1959,2019]

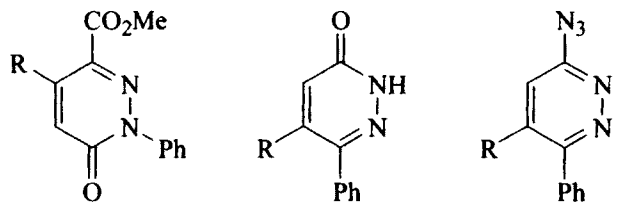
### 4/5-Monohalogenopyridazines as Substrates

Methyl 4-chloro- (**302**, R = Cl) gave methyl 4-azido-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate (**302**, R = $N_3$), ($NaN_3Me_2NCHO$, 25°, 2 h: 85%);[1117] also several analogues.[1117]

5-Chloro- (**303**, R = Cl) gave 5-azido-6-phenyl-3(2*H*)-pyridazinone (**303**, R = $N_3$) ($NaN_3$, $Me_2NCHO$, 25°, 2 h: 88%).[134]

3-Azido-5-chloro- (**304**, R = Cl) gave 3,5-diazido-6-phenylpyridazine (**304**, R = $N_3$) ($NaN_3$, $Me_2NCHO$, < 80°, 15 min; 70%).[134]

Also other examples.[848]

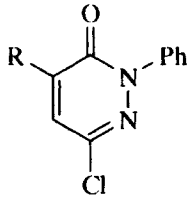


(302) (303) (304)

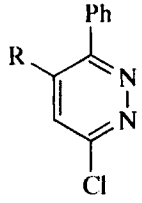
### 3,5/4,6-Dihalogenopyridazines as Substrates

4,6-Dichloro- (**305**, R = Cl) gave 4-azido-6-chloro-2-phenyl-3(2*H*)-pyridazinone (**305**, R = $N_3$) ($NaN_3$, $H_2O$–EtOH, reflux, 4 h: ~65%).[401]

3,5-Dichloro-6-phenylpyridazine (**306**, R = Cl) gave 4-azido-6-chloro-3-phenylpyridazine (**306**, R = $N_3$) ($NaN_3$, $Me_2NCHO$, 25°, 2 h: 88%).[134]



(**305**)

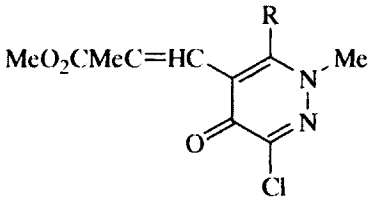


(**306**)

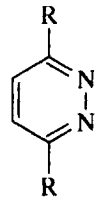
### 3,6-Dihalogenopyridazines as Substrates

3,6-Dichloro- (**307**, R = Cl) gave 6-azido-3-chloro-5-(2-methoxycarbonylprop-1-enyl)-1-methyl-4(1*H*)-pyridazinone (**307**, R = $N_3$) ($NaN_3$, $Me_2SO$, 25°, 2 h: 94%).[967]

3,6-Dichloro- (**308**, R = Cl) gave 3,6-diazidopyridazine (**308**, R = $N_3$) $NaN_3$, $H_2O$–EtOH, reflux, ?: explosive, kept wet).[1355, cf. 1934]



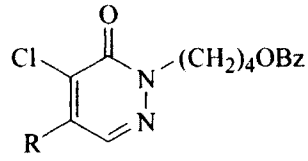
(**307**)



(**308**)

### 4,5/5,6-Dihalogenopyridazines as Substrates

2-δ-Benzoyloxybutyl-4,5-dichloro- (**309**, R = Cl) gave 5-azido-2-δ-benzoyloxybutyl-4-chloro-3(2*H*)-pyridazinone (**309**, R = $N_3$) ($NaN_3$, $Me_2SO$, reflux, 6 h: 56%).[1700]

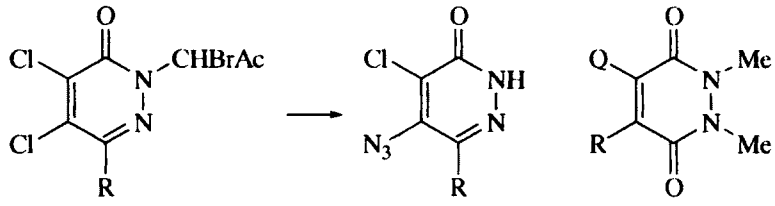


(**309**)

In contrast, 2-α-bromoacetonyl-4,5-dichloro-3(2*H*)-pyridazinone (**310**, R = H) gave 5-azido-4-chloro-3(2*H*)-pyridazinone (**311**, R = H) ($NaN_3$, AcMe–$Me_2NCHO$, 25°, 8 h: 78%; note *N*-dealkylation);[1680] 2-α-bromoacetonyl-4,5-dichloro-6-nitro- (**310**, $R = NO_2$) likewise gave 5-azido-4-chloro-6-nitro-3(2*H*)-pyridazinone (**311**, $R = NO_2$) (92%).[1680]

4,5-Dibromo-1,2-dimethyl- (**312**, Q = R = Br) has been reported to give 4-azido-1,2-dimethyl-3,6(1*H*,2*H*)-pyridazinedione (**312**, $Q = N_3$, R = H) [$NaN_3$ (2 equiv), $Me_2NCHO$, 25°, 48 h: 80%; the mechanism would be more easily rationalized if the 4,5-dihydro substrate had been used].[402]

Also other examples.[1884]




### 4.2.7 Hydrogenolysis of Nuclear Halogenopyridazines (*H* 245)

Dehalogenation of halogenopyridazines is a useful process, usually carried out by catalytic hydrogenation at 25° and 1–4 atm in the presence of added base to avoid nuclear reduction. Selectivity in the partial hydrogenolysis of di- or polyhalogenopyridazines is generally difficult but has been achieved occasionally. The following examples illustrate the techniques and conditions employed recently:

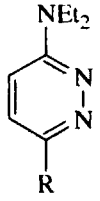
#### Monochloropyridazines with a Palladium Catalyst

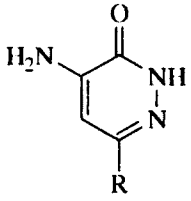
3-Chloro-6-diethylaminopyridazine (**313**, R = Cl) gave 3-diethylaminopyridazine (**313**, R = H) ($H_2$, Pd/C, NaOH, EtOH: 87%).[924] likewise 6-chloro-3-pyridazinamine 1-oxide gave 3-pyridazinamine 1-oxide (MeOH: 95%).[891]

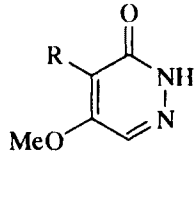
4-Amino-6-chloro-3(2*H*)-pyridazinone (**314**, R = Cl) gave 4-amino-3(2*H*)-pyridazinone (**314**, R = H) ($H_2$, Pd/C NaOH, $H_2O$: 75%).[1597]

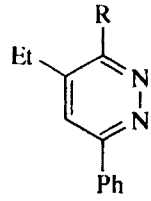
4-Chloro-5-methoxy-3(2*H*)-pyridazinone (**315**, R = Cl) gave 5-methoxy-3(2*H*)-pyridazinone (**315**, R = H) ($H_2$, Pd/C, NaOH, $H_2O$, 19 h: 85%).[90]

3-Chloro-4-ethyl-6-phenylpyridazine (**316**, R = Cl) gave 4-ethyl-6-phenylpyridazine (**316**, R = H) ($H_2$, Pd/C, $NH_4OH$, EtOH: 94%); also analogues.[1676]


(313)


(314)


(315)


(316)

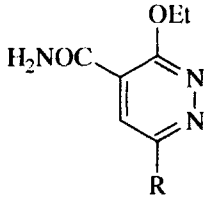
6-Chloro-3-ethoxy-4-pyridazinecarboxamide (**317**, R = Cl) gave 3-ethoxy-4-pyridazinecarboxamide (**317**, R = H) ($H_2$, Pd/C, $NH_4OH$, EtOH: 80%); also analogues.[1099]

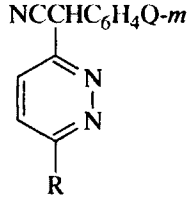
6-Chloro-5-methylamino-3-pyridazinamine gave 5-methylamino-3-pyridazinamine ($H_2$, Pd/C, AcONa, AcOH–EtOH, > 1 h: 62%).[447]

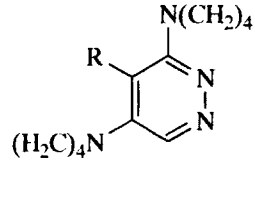
3-Chloro-6-α-cyanobenzylpyridazine (**318**, Q = H, R = Cl) gave 3-α-cyanobenzylpyridazine (**318**, Q = R = H) [$H_2$, Pd/C, MgO, EtOH–$H_2O$: 67%;[1547] A (no $H_2$ required), Pd/C, $HCO_2NH_4$, MeOH, 48°, until complete (tlc): 95%];[1354] also by the latter method, 3-chloro-6-[α-cyano-*m*-(trifluoromethyl)benzyl]pyridazine (**318**, Q = $CF_3$, R = Cl) gave 3-[α-cyano-*m*-(trifluoromethyl)benzyl]pyridazine (**318**, Q = $CF_3$, R = H) [$N_2$ (not $H_2$), Pd/C, $HCO_2NH_4$, MeOH, reflux, until complete (tlc): 41%].[1798]

4-Chloro-3,5-dipyrrolidin-1′-ylpyridazine (**319**, R = Cl) gave 3,5-dipyrrolidin-1′-ylpyridazine (**319**, R = H) ($H_2$, Pd/C, EtOH: 95%; note lack of added base);[1058] also analogous examples without added base.[769,1331]

Also may other examples.[224,410,446,531,534,544,546,555,558,641,762,766,821,833,882,902,956,1021,1025,1041,1061,1104,1186,1239,1254,1269,1254,1269,1421,1700,1760,2102]


(317)


(318)


(319)

## Monochloropyridazines with Raney-Nickel Catalyst

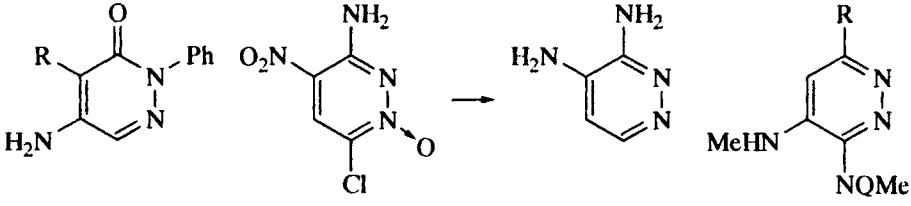
5-Amino-4-chloro-2-phenyl-3(2*H*)-pyridazinone (**320**, R = Cl) gave 5-amino-2-phenyl-3(2*H*)-pyridazinone (**320**, R = H) ($H_2$, Raney Ni, $NH_4OH$, $H_2O$, high pressure: 90%).[1344]

3-Amino-6-chloro-4-nitropyridazine 1-oxide (**321**) gave 3,4-pyridazinediamine (**322**) ($H_2$, Raney Ni + Pd/C, MeOH, 15 h: 51%; note lack of added base and concomitant *N*-deoxidation and reduction of the nitro group).[449]

3-Chloro-5-methylamino-6-α-methylhydrazinopyridazine (**323**, Q = $NH_2$, R = Cl) gave 3,4-bismethylaminopyridazine (**323**, Q = R = H) ($H_2$,

Raney Ni, EtOH; then Pd/C↓, NaOH↓, $H_2$: ?%, characterized as picrate).[1138]

Also other examples.[956]

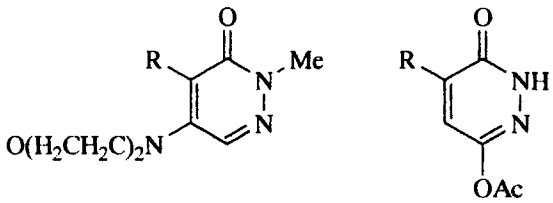


(320) (321) (322) (323)

## Monobromopyridazines with a Palladium Catalyst

4-Bromo-2-methyl-5-morpholino- (**324**, R = Br) or the chloro analogue (**324**, R = Cl) gave 2-methyl-5-morpholino-3(2*H*)-pyridazinone (**324**, R = H) ($H_2$, Pd/C, 60%).[542]

6-Acetoxy-4-bromo-3(2*H*)-pyridazinone (**325**, R = Br) gave 6-acetoxy-4-tritio-3(2*H*)-pyridazinone (**325**, R = $^3$H) ($^3H_2$ gas, Pd/C, EtOH, 20°, 10 min: see original for details).[163]



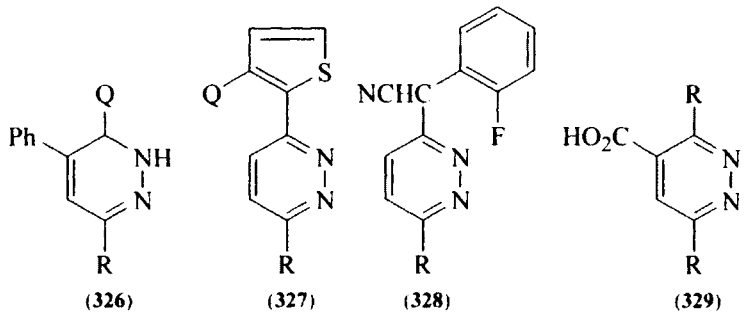
(324) (325)

## Di- or Trihalogenopyridazines with a Palladium Catalyst

3,6-Dichloro-4-phenylpyridazine (**326**, Q = R = Cl) gave either 4-phenylpyridazine (**326**, Q = R = H) (unlimited $H_2$, Pd/C, AcONa, EtOH: 91%) or a separable mixture of 4-phenyl- (**326**, Q = R = H) and 3-chloro-4-phenylpyridazine (**326**, Q = Cl, R = H) [$H_2$ (1 equiv): 19% and 16%, respectively, after separation; the lack of any 4-chloro isomer (**326**, Q = H, R = Cl) suggested some selectivity].[1186]

3-(5-Bromothien-2-yl)-6-chloropyridazine (**327**, Q = Br, R = Cl) gave 3-thien-2′-ylpyridazine (**327**, Q = R = H) ($H_2$, Pd/C, $Na_2CO_3$, EtOH: 77%);[975] in contrast, 3-chloro-6-(α-cyano-*o*-fluorobenzyl)pyridazine (**328**, R = H) gave 3-(α-cyano-*o*-fluorobenzyl)pyridazine (**328**, R = H) without loss of the fluoro substituent [A (no $H_2$), Pd/C, $HCO_2NH_4$, MeOH, 40°: 77%].[1628]

3,6-Dichloro-4-pyridazinecarboxylic acid (**329**, R = Cl) gave 4-pyridazinecarboxylic acid (**329**, R = H) ($H_2$), Pd/C, $NH_4OH$, MeOH, ~5 h: 92%).[65]



(326) (327) (328) (329)

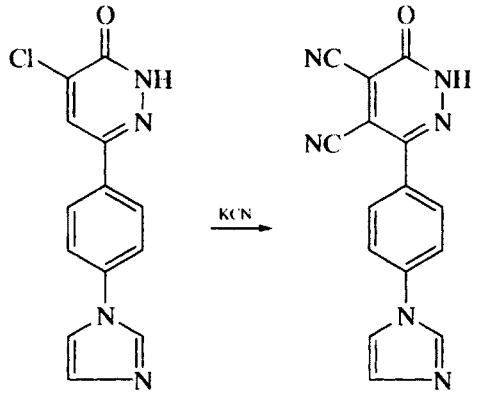
3,4,6-Trichloro-5-morpholinopyridazine gave 5-morpholinopyridazine ($H_2$, Pd/C, no base, EtOH: ?%, isolated as picrate).[637]

Also other examples in the foregoing references and elsewhere.[605,1115]

### 4.2.8 Other Displacement Reactions of Nuclear Halogenopyridazines (*H* 271)

Several other displacement reactions of halogenopyridazines have been observed, as illustrated by the isolated examples that follow:

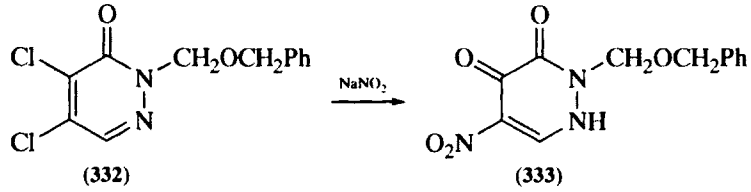
4-Chloro-6-*p*-imidazol-1′-ylphenyl-3(2*H*)-pyridazinone (**330**) gave 3-*p*-imidazol-1′-ylphenyl-6-oxo-1,6-dihydro-4,5-pyridazinedicarbonitrile (**331**) (KCN, $Me_2SO$, 60°, 1 h; then 25°, 16 h: 15%; the mechanism probably involved displacement, addition, and oxidation in a yet undetermined order).[1073]



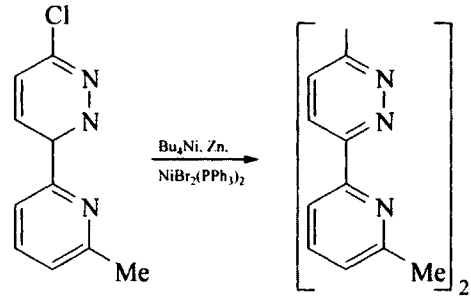
(330) (331)

2-Benzyloxymethyl-4,5-dichloro-3(2*H*)-pyridazinone (**332**) gave 2-benzyloxymethyl-5-nitro-3,4(1*H*,2*H*)-pyridazinedione (**333**) ($NaNO_2$, $Me_2NCHO$–$H_2O$, 85°, 24 h: 93%; possibly by displacement of 5-Cl followed by hydrolysis of 4-Cl during aqueous workup?).[1048]

3-Chloro-6-(6-methylpyridin-2-yl)pyridazine (**334**) gave 6,6′-bis(6-methylpyridin-2-yl)-3,3′-bipyridazine (**335**) [$Bu_4Ni + Zn + NiBr(PPh_3)_2$, $Me_2NCHO$, 55°: 80%];[1574] also another such example,[1899] as well as a Cu-induced example towards the end of Section 3.2.1.3.

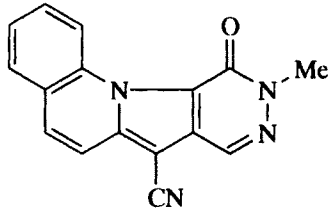



(332) (333)




(334) (335)

4,5-Dibromo-2-methyl-3(*H*)-pyridazinone with 2-cyanomethylquinoline gave 10-methyl-11-oxo-10,11-dihydropyridazino[4′,5′:4,5]pyrrolo[1,2-*a*]quinoline-2-carbonitrile (**336**), by cyclization of a pyridazinylmethylquinoline intermediate (for details, see original: >40% overall).[1441]




(336)

3,6-Dichloropyridazine gave 3,6-bis(diphenylphosphino)pyridazine ($Ph_2PLi$, THF, 5°, 3 h: 61%),[2120] used subsequently as a ligand in Ag, Cr, and Mo complexes.[2080,2120]

Also other examples.[1941,2056,2095,2113]

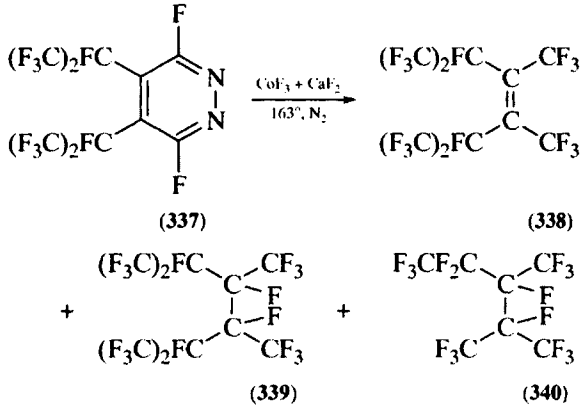
## 4.2.9 Reactions Involving Ring Fission of Nuclear Halogenopyridazines

Many ring fissions of halogenopyridazines are followed spontaneously by reassembly into halogeno derivatives of pyridine, pyrimidine, or pyrazine. Most

of these reactions are more of interest than preparative value. The following examples illustrate typical reaction conditions and products:

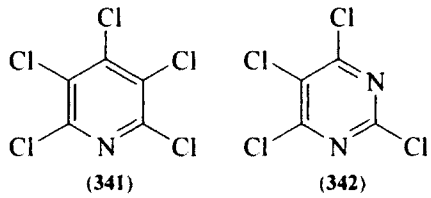
### Aliphatic Products

Perfluoro-4,5-diisopropylpyridazine (**337**) gave a separable mixture of perfluoro-2,3,4,5-tetramethylhex-3-ene (**338**; $E+Z$), perfluoro-2,3,4,5-tetramethylhexane (**339**), and perfluoro-2,3-dimethylpentane (**340**) (neat substrate, $CoF_3 + CaF_2$ 163°: 53%, 12%, and 9%, respectively);[709] perfluoro-3,5-diisopropylpyridazine gave only *E*- and *Z*-perfluoro-2,3,6-trimethylhept-3-ene (likewise at 132°: 25% each).[709]



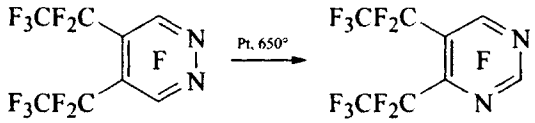

### Pyridines as Products

3,4,5,6-Tetrachloropyridazine gave pentachloropyridine (**341**), hexachlorobenzene, pentachlorobenzonitrile, tetrachlorophthalonitrile, octachlorostyrene, hexachlorobutadiene, tetrachloroethylene, dichlorofumaronitrile, dichloromaleonitrile, and other products (pyrolysis on silica wool at 680°).[653]




### Pyrimidines as Products

3,4,5,6-Tetrachloropyridazine gave the products listed in the preceding paragraph plus an appreciable amount (6%) of 2,4,5,6-tetrachloropyrimidine (**342**) (pyrolysis in a sealed tube at 600°).[484]




(343) (344)

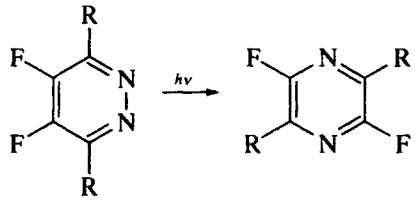
Perfluoro-4,5-diethylpyridazine (**343**) gave mainly perfluoro-4,5-diethylpyrimidine (**344**) (pyrolysis, Pt foil, 650°: ~70%).[658]

Also other such examples.[245,486,487,706]

### Pyrazines as Products

Perfluoro-3,6-dimethylpyridazine (**345**, R = $CF_3$) gave perfluoro-2,5-dimethylpyrazine (**346**, R = $CF_3$) (neat substrate, A, *hν*, 90 h: ~45%).[696]

Tetrafluoropyridazine gave tetrafluoropyrazine (**346**, R = F) (neat substrate, sealed, *hν*, 6 days: ~60%).[663]




(345) (346)

Also other examples in the foregoing references and elsewhere.[329,658,666,706]

## 4.3 PREPARATION OF EXTRANUCLEAR HALOGENOPYRIDAZINES

Although some extranuclear halogenopyridazines have been made by *primary syntheses* (see Chapters 1 and 2), most have been prepared recently from extranuclear hydroxypyridazines: by *C*-halogenation of alkyl or aryl groups already attached to pyridazines; or by attachment to a pyridazine of an alkyl or aryl group already bearing a passenger halogeno substituent.

### 4.3.1 Extranuclear Halogenopyridazines from Corresponding Hydroxypyridazines

Most such transformations have been done by treatment with thionyl chloride in an inert solvent. However, phosphoryl chloride, thionyl bromide, or phosphorus tribromide have been used also as appropriate and indirect routes have been employed occasionally. The following examples illustrate the various procedures:

### Using Thionyl Chloride

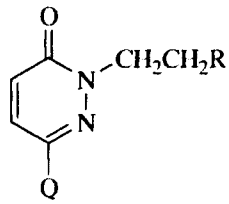
4-Hydroxymethylpyridazine gave 4-chloromethylpyridazine (neat $SOCl_2$, 5°, then 25°, 3 h: 87%, as hydrochloride).[66]

3-α-Hydroxybenzylpyridazine gave 3-α-chlorobenzylpyridazine ($SOCl_2$, PhMe, 0° → 25°, 12 h: 84%).[1108]

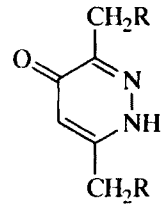
2-β-Hydroxyethyl- (**347**, Q = R, R = OH) gave 2-β-chloroethyl-3(2*H*)-pyridazinone (**347**, Q = H, R = Cl) ($SOCl_2$, $CHCl_3$, 25° → reflux, 1 h: 88%);[1179] likewise, 2-β-hydroxyethyl-6-phenyl- (**347**, Q = Ph, R = OH) gave 2-β-chloroethyl-6-phenyl-3(2*H*)-pyridazinone (**347**, Q = Ph, R = Cl) ($SOCl_2$, $CHCl_3$, −5° → reflux, 1 h: 89%).[1292]

3,6-Bishydroxymethyl- (**348**, R = OH) gave 3,6-bischloromethyl-4(1*H*)-pyridazinone (**348**, R = Cl) (substrate·HCl, $SOCl_2$, 0°, 2 h: 45%).[446]

Also other examples.[107,237,441,534,578,707,850,1357,1491,1600,1675,1752,1903]



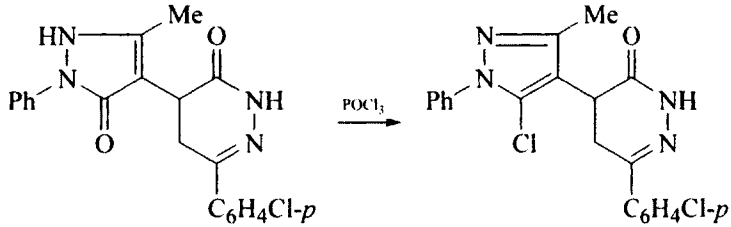
(**347**)



(**348**)

### Using Phosphoryl Chloride

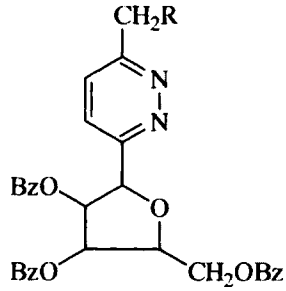
6-*p*-Chlorophenyl-4-(3-methyl-5-oxo-1-phenyl-3-pyrazolin-4-yl)-4,5-dihydro-3(2*H*)-pyridazinone (**349**) has been reported to afford 4-(3-chloro-5-methyl-2-phenylpyrazol-4-yl)-6-*p*-chlorophenyl-4,5-dihydro-3(2*H*)-pyridazinone (**350**), apparently by selective replacement of the oxo substituent on the five-membered ring ($POCl_3$: ?%).[1416]



(**349**) (**350**)

### Using Thionyl Bromide

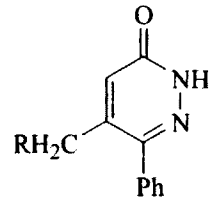
3-Hydroxymethyl- (**351**, R = OH) gave 3-bromomethyl-6-(2,3,5-tri-*O*-benzoyl-β-D-ribofuranosyl)pyridazine (**351**, R = Cl) ($SOBr_2$, PhH, 0° → 25°, 1 h: 74%).[1539]



(351)

### Using Phosphorus Tribromide

5-Hydroxymethyl- (**352**, R = OH) gave 5-bromomethyl-6-phenyl-3(2*H*)-pyridazinone (**352**, R = Br) ($PBr_3$, $Et_2O$, 0° → 25°, 2 h: 80%).[1588]



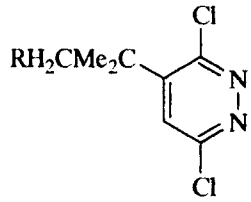
(352)

### Using a Vilsmeier Reagent

4,5-Dichloro-2-hydroxymethyl- gave 2-bromomethyl-4,5-dichloro-3(2*H*)-pyridazinone ($SOBr_2$-$Me_2NCHO$, reflux, 4 h: 94%).[1950]

### Using an Indirect Route

3,6-Dichloro-4(β-hydroxy-α,α-dimethylethyl)pyridazine (**353**, R = OH) gave 3,6-dichloro-4-(α,α-dimethyl-β-*p*-toluenesulfonyloxyethyl)pyridazine (**353**, R = OTs) (TsCl, pyridazine, 25°, $N_2$, 17 h: 80%) and then 4-(β-bromo-α,α-dimethylethyl)-3,6-dichloropyridazine (**353**, R = Br) (LiBr, $Me_2SO$, 110°, $N_2$, 2 h: 91%).[354]

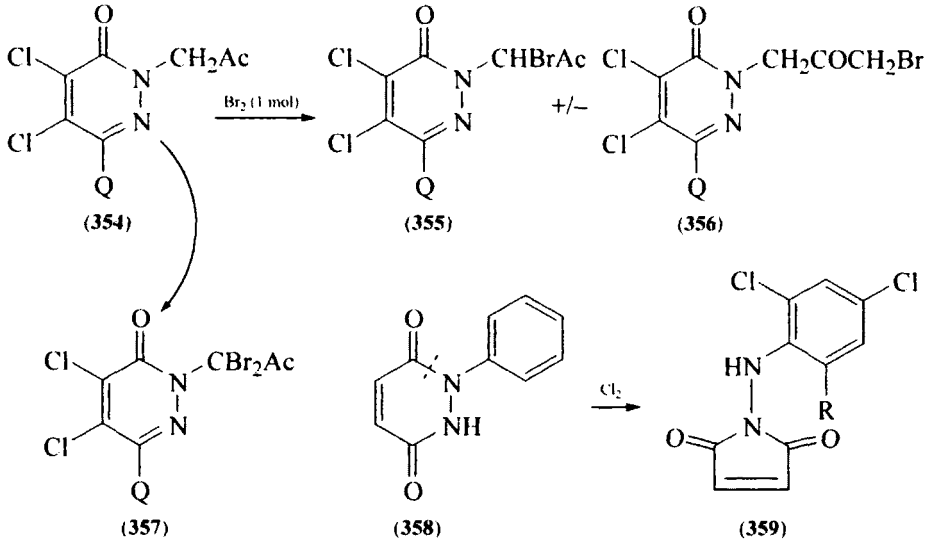


(353)

### 4.3.2 Extranuclear Halogenopyridazines by Halogenation

The direct halogenation of a side chain attached to pyridazine is not always simple; unwanted halogenation may occur at an unoccupied position on the pyridazine ring, it is not easy to control the site(s) of halogenation on an aliphatic or even aromatic side chain, and other untoward modification(s) of the pyridazine may ensue. In addition to those already given (Section 3.2.2.4), the following examples illustrate some regular and irregular results.

2-Acetonyl- (**354**, Q = H) gave only 2-α-bromoacetonyl-4,5-dichloro-3(2*H*)-pyridazinone (**355**, Q = H) ($Br_2$, AcONa, AcOH–$CHCl_3$, 25°, 3 h: 60%) or a separable mixture of 2-α-bromoacetonyl- (**355**, Q = H) and the isomeric 2-γ-bromoacetonyl-4,5-dichloro-3(2*H*)-pyriazinone (**356**, Q = H) ($Br_2$, $CHCl_3$, reflux, 30 min: 10% and 61%, respectively);[1673] the 6-nitro substrate (**354**, Q = $NO_2$) behaved similarly to give only the product (**355**, Q = $NO_2$) (62%) or a separable mixture of isomers (**355**, Q = $NO_2$) and (**356**, Q = $NO_2$) (53% and 39%, respectively).[1673] The foregoing substrates (**355**, Q = H or $NO_2$) also gave 4,5-dichloro-2-α,α-dibromoacetonyl-3(2*H*)-pyridazinone (**357**, Q = H) or its 6-nitro derivative (**357**, Q = $NO_2$), respectively (2 × $Br_2$, AcONa AcOH–$CHCl_3$, 25°, ~15 h: both ~88%).[1673]

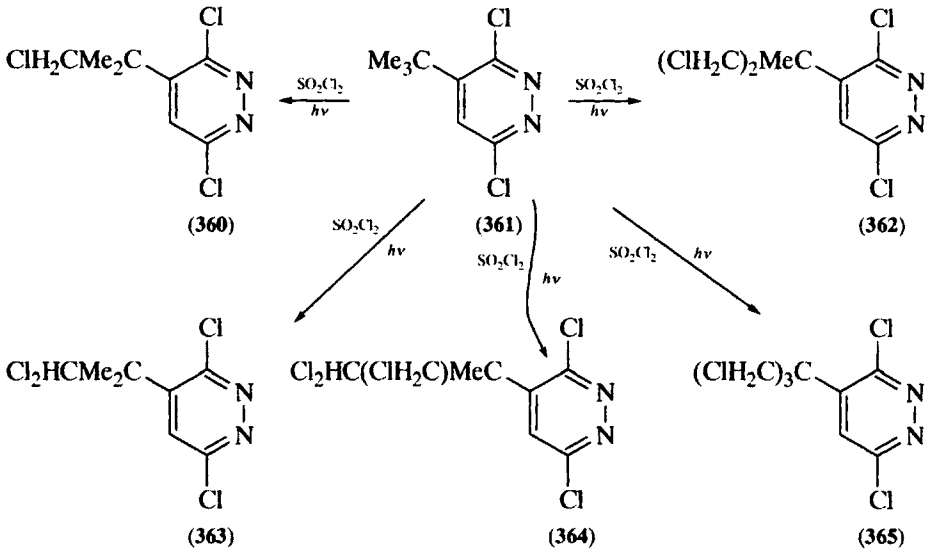



1-Phenyl-3,6(1*H*,2*H*)-pyridazinedione (**358**) gave 1-(2,4-dichloroanilino)- (**359**, R = H) ($Cl_2$↓, $Me_2NCHO$, 80°, ~30 min: ~5%) or 1-(2,4,6-trichloroanilino)-3-pyrroline-2,5-dione (**359**, R = Cl) ($Cl_2$↓, $Me_2NCHO$, 50°, 105 min: 53%; note ring contraction).[1105]

4-*t*-Butyl-3,6-dichloropyridazine (**361**) gave 3,6-dichloro-4-(β-chloro-α,α-dimethylethyl)- (**360**), 4-(α,α-bischloromethylethyl)-3,6-dichloro- (**362**),

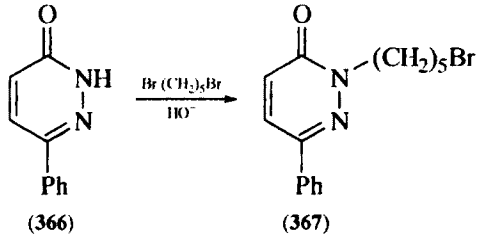
3,6-dichloro-4-(β,β-dichloro-α,α-dimethylethyl)- (**363**), 3,6-dichloro-4-(β,β-dichloro-α-chloromethyl-α-methylethyl)- (**364**), and 3,6-dichloro-4-(β-chloro-α,α-bischloromethylethyl)pyridazine (**365**) ($SO_2Cl_2$, $Bz_2O_2$, $CCl_4$, $N_2$, 58°, *hν*, 1.5–19 h: chlorination increased with time; all products isolated and characterized).[354]

Also other examples.[789,838,862,1979]




### 4.3.3 Extranuclear Halogenopyridazines by Passenger Halogenation

Halogeno substituents are often carried as passengers when other groups are attached to the pyridazine nucleus in all sorts of ways that are covered elsewhere in this book. For example, an extranuclear halogenopyridazine was produced when 6-phenyl-3(2*H*)-pyridazinone (**366**) underwent *N*-alkylation by an excess of α,ε-dibromopentane in benzene containing potassium hydroxide and tetrabutylammonium bromide at 25° during 8 h to afford 2-ε-bromopentyl-6-phenyl-3(2*H*)-pyridazinone (**367**) in 69% yield.[1288,1289]



## 4.4 REACTIONS OF EXTRANUCLEAR HALOGENOPYRIDAZINES

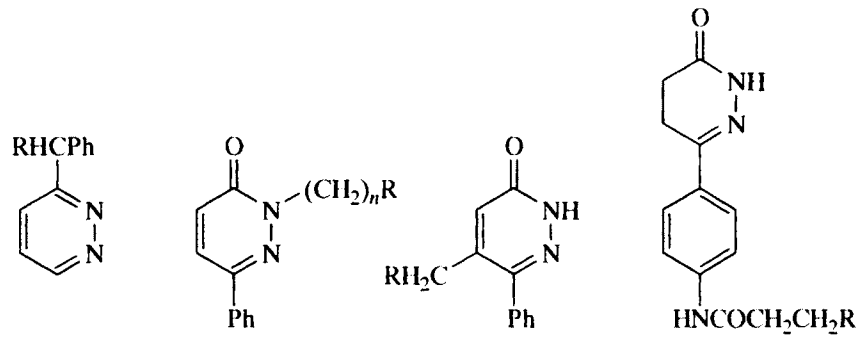
These halogenopyridazines undergo all the reactions to be expected of homocyclic analogues such as benzyl chloride. The reactivity of the halogeno substituent appears to be virtually unaffected by the heterocyclic ring or by groups attached thereto but, of course, it is affected significantly by appropriate groups (e.g., carbonyl) adjacent to it on the side chain. Because such aliphatic chemistry is so familiar, treatment here is accordingly brief.

### 4.4.1 Aminolysis of Extranuclear Halogenopyridazines

The aminolytic replacement of a halogeno substituent on an aliphatic side chain is done easily. However, when the halogeno substituent is attached to an aromatic entity, replacement is very difficult unless there is activation by an appropriate adjacent group. Occasional *tele*-aminolyses have been reported. The following examples illustrate conditions for some typical aminolyses:

3-α-Chlorobenzylpyridazine (**368**, R = Cl) gave 3-α-piperidinobenzylpyridazine [**368**, R = $N(CH_2)_5$] [$(CH_2)_5NH$, PhMe, reflux, A, 6 h: 71%]; also several analogues.[1108]

2-(7-Bromoheptyl)- (**369**, $n = 7$, R = Br) gave 2-(7-aminoheptyl)-6-phenyl-3(2*H*)-pyridazinone (**369**, $n = 7$, R = $NH_2$) ($NH_4OH$, EtOH, 40°, 24 h: 75%);[1337] likewise, 2-δ-bromobutyl- (**369**, $n = 4$, R = Br) gave 2-δ-methylaminobutyl-6-phenyl-3(2*H*)-pyridazinone (**369**, $n = 4$, R = NHMe) ($MeNH_2$, MeOH, 50°, 20 h: 68%).[1289,1337]




(368) (369) (370) (371)

2-β-Chloroethyoxymethyl- gave 2-β-aminoethoxymethyl-6-phenyl-3(2*H*)-pyridazinone ($NH_3$, MeOH–$H_2O$, 150°, sealed, 24 h: 55%).[1292]

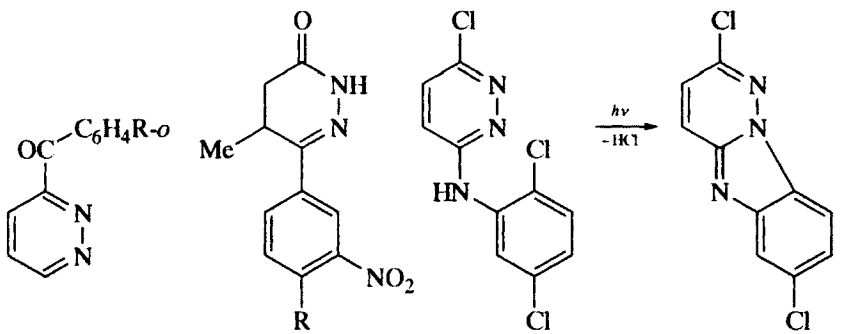
5-Bromomethyl- (**370**, R = Br) gave 5-(4-benzoylpiperazin-1-ylmethyl)-3(2*H*)-pyridazine [**370**, R = $N(CH_2CH_2)_2NBz$] [$HN(CH_2CH_2)_2NBz$, MeOH, 25°, 12 h: 94%].[1588]

6-*p*-(3-Bromopropionamido)phenyl-4,5-dihydro-3(2*H*)-pyridazinone (**371**, R = Br) gave 6-*p*-[3-(benzylamino)propionamido]phenyl-4,5-dihydro-3(2*H*)-pyridazinone (**371**, R = $NHCH_2Ph$) ($PHCH_2NH_2$, PrOH, reflux, 16 h: 42%).[1313]

3-*o*-Fluorobenzoylpyridazine (**372**, R = F) gave 3-*o*-(benzylamino)benzoylpyridazine (**372**, R = $NHCH_2Ph$) ($PhCH_2NH_2$, $K_2CO_3$, PhMe, reflux, 36 h: 82%; note evident activation of F(fluorine) by adjacent C=O);[1628] 4-*o*-fluorobenzoylpyridazine likewise gave 4-*o*-(butylamino)benzoylpyridazine ($BuNH_2$, $K_2CO_3$, PhMe, 90°, 72 h: 70%).[53]

6-(4-Chloro-3-nitrophenyl)-5-methyl-4,5-dihydro-3(2*H*)-pyridazinone (**373**, R = Cl) gave 6-(4-benzylamino-3-nitrophenyl)-5-methyl-4,5-dihydro-3(2*H*)-pyridazinone (**373**, R = $NHCH_2Ph$) ($PhCH_2NH_2$, BuOH, reflux, 20 h: 86%; note the activation of Cl substituent by $NO_2$ group).[1762]

3-Chloro-6-(2,5-dichloroanilino)pyridazine (**374**) underwent an aminolytic cyclization to give 2,7-dichloropyridazino[1,6-*a*]benzimidazole (**375**) ($Bu^tOH$-$H_2O$, *hν*, 6 h: ~15%).[574]




(372) (373) (374) (375)

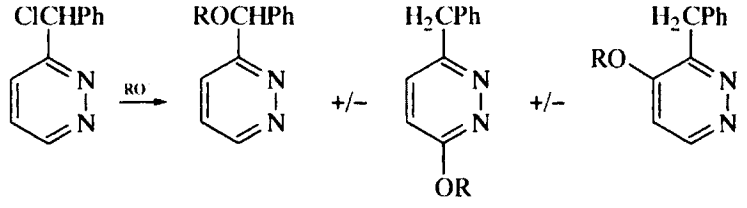
4-α-Chlorobenzylpyridazine gave not only the regular product, 4-α-morpholinobenzylpyridazine, but also the *tele*-aminolytic product, 4-benzyl-5-morpholinopyridazine [$O(CH_2CH_2)_2NH$, PhMe, 100°, 3 h: 49% and 21%, respectively].[82]

Also other examples.[209,578,862,1049,1314,1752,1760]

### 4.4.2 Alcoholysis of Extranuclear Halogenopyridazines

Although some extranuclear halogenopyridazines undergo regular alcoholysis or phenolysis by alkoxide or aryloxide ion to afford the corresponding alkoxy- or aryloxypyridazines, some undergo *tele*-alcoholysis usually to afford nuclear alkoxypyridazines instead of (or as well as) the expected products; the formation of such irregular products appears to be suppressed by preinsertion of an electron-donating group into the pyridazine ring.[1357,1625] The following examples illustrate the foregoing possibilities:

3-α-Chlorobenzylpyridazine (**376**) gave only 3-(α-phenoxybenzyl)pyridazine (**377**, R = Ph) (PhONa, PhMe, reflux, 12 h: 46% 0;[1357] in contrast, the same substrate (**376**) gave a separable mixture of 3-α-methoxybenzyl- (**377**, R = Me), 3-benzyl-6-methoxy- (**378**, R = Me), and 3-benzyl-4-methoxypyridazine (**379**, R = Me) (MeONa, MeOH, reflux, 12 h: 19%, 55%, and 12% respectively).[1357]

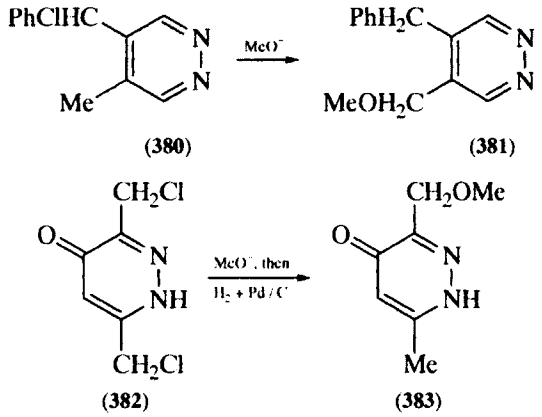



(**376**) (**377**) (**378**) (**379**)

4-Chloromethylpyridazine gave a separable mixture of 4-methoxymethylpyridazine and 4-methoxy-5-methylpyridazine (MeONa, PhMe, reflux, 2 h: 26% and 45%, respectively);[82] in contrast, 4-α-chlorobenzyl-5-methylpyridazine (**380**) gave only 4-benzyl-5-methoxymethylpyridazine (**381**) (MeONa, MeOH, reflux, 2 h: >72%).[82]

3,6-Bischloromethyl-4(1*H*)-pyridazinone (**382**) gave 3-methoxymethyl-6-methyl-4(1*H*)-pyridazinone (**383**) [MeONa (1 equiv), MeOH, 20°, 24 h; then $H_2$, Pd/C, AcONa, AcOH: 90%; note selective alcoholysis prior to dehalogenation].[446]

See also Section 4.4.6 for an intramolecular alcoholysis.




(**380**) (**381**)

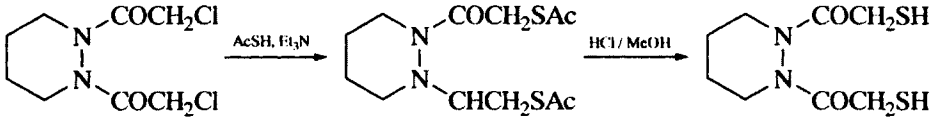
(**382**) (**383**)

### 4.4.3 Thiolysis of Extranuclear Halogenopyridazines

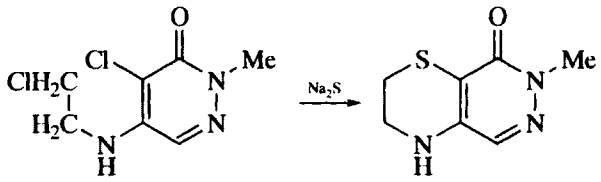
This reaction is usually avoided, probably because the resulting aliphatic thiols tend to undergo rapid aerial oxidation to disulfides, especially in basic

media. To avoid this, the thiol group(s) may be introduced as their acetate esters and then converted into free thiols under acidic conditions. For example, 1,2-bischloroacetylhexahydropyridazine (**384**) with thioacetic acid and triethylamine in dichloromethane at 23° for 5 h afforded 72% of 1,2-bis[α-(acetylthio)acetyl]hexahydropyridazine (**385**), which underwent deacetylation in methanolic hydrogen chloride at 23° during 20 h to furnish 1,2-bismercaptoacetylhexahydropyridazine (**386**) in 90% yield.[1382]

Another example, which must involve the thiolysis of a nuclear and/or an extranuclear chloro substituent, is the conversion of 4-chloro-5-β-chloroethylamino-2-methyl-3(2*H*)-pyridazinone (**387**) into 20% of 7-methyl-3,4-dihydro-2*H*-pyridazino[4,5-*b*]-1,4-thiazin-8(7*H*)-one (**388**) by treatment with ethanolic sodium sulfide nonahydrate under reflux for 4 h.[850]



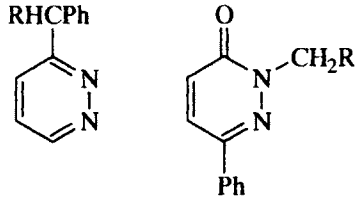

(384) (385) (386)




(387) (388)

### 4.4.4 Alkanethiolysis of Extranuclear Halogenopyridazines

Such alkanethiolyses are typified by the conversion of 3-α-chlorobenzylpyridazine (**389**, R = Cl) into 3-α-(β-dimethylaminoethylthio)benzyl- (**389**, R = $SCH_2CH_2NMe_2$) or 3-α-(phenylthio)benzylpyridazine (**389**, R = SPh) ($Me_2NCH_2CH_2SH$ or PhSh, NaH, PhMe, 15 min; then substrate ↓ reflux, 3 h: 65% or 71%, respectively);[1108] or of 2-chloromethyl- (**390**, R = Cl) into 2-(β-aminoethylthio)methyl-6-phenyl-3(2*H*)-pyridazinone (**390**, R = $SCH_2CH_2NH_2$) ($NaSCH_2CH_2NH_2$, EtOH, reflux, 5 h: 60%).[1292]

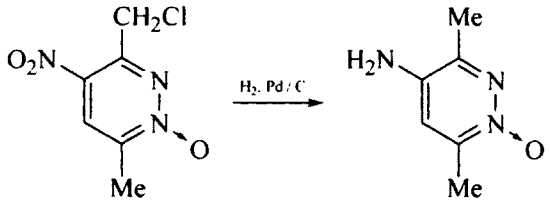



(389) (390)

### 4.4.5 Hydrogenolysis of Extranuclear Halogenopyridazines

Althoug not used very often, hydrogenolysis of both halogenoalkyl- and halogenoarylpyridazines can be done in a mildly alkaline medium over a palladium catalyst; naturally, other reducible passenger groups may be affected. The following examples illustrate typical processes used recently:

3-Chloromethyl-6-methyl-4-nitropyridazine 1-oxide (**391**) gave 3,6-dimethyl-4-pyridazinamine (**392**) ($H_2$, Pd/C, MeOH, $NH_4OH$, >2 h, ?%).[796]

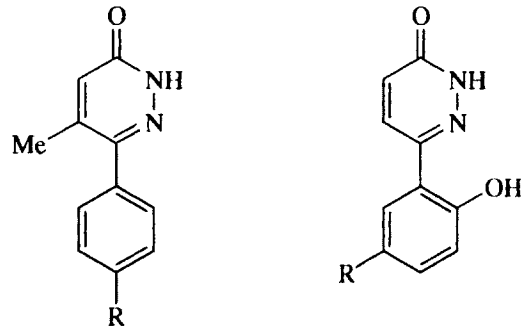



(391) (392)

6-*p*-Bromophenyl-5-methyl-3(2*H*)-pyridazinone (**393**, R = Br) gave 5-methyl-6-phenyl-3(2*H*)-pyridazinone (**393**, R = H) [$H_2$ (3 atm), Pd/C, EtOH–$NH_4OH$, 50°, 4 h: 93%].[961]

6-(3-Bromo-6-hydroxyphenyl)-3(2*H*)-pyridazinone (**394**, R = Br) gave 6-*o*-hydroxyphenyl-3(2*H*)-pyridazinone (**394**, R = H) [$H_2$ (4 atm), Pd/C, 1.5M NaOH, 25°, 5 h: 89%].[159]

Also other examples.[975]




(393) (394)

### 4.4.6 Other Displacement Reactions of Extranuclear Halogenopyridazines

Several other reactions of these halogenopyridazines have proved useful from time to time, as illustrated by the following examples:

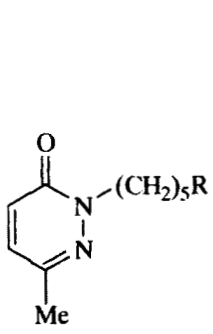
### With Cyanide Ion

2-ε-Bromopentyl- (**395**, R = Br) gave 2-ε-cyanopentyl-6-phenyl − 3(2*H*)-pyridazinone (**395**, R = CN) (KCN, $Bu_4NBr$, PhH, 55°, 10 h: 81%).[1288]

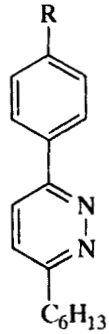
3-*p*-Bromophenyl- (**396**, R = Br) gave 3-*p*-cyanophenyl-6-hexylpyridazine (**396**, R = CN) (CuCN, $Me_2NCHO$, 190°, 3.5 h: 39%; note the vigorous conditions required in an aromatic environment).[1576]

3,6-Bis-*p*-bromophenylpyridazine gave 3,6-bis-*p*-cyanophenylpyridazine (CuCN, quinoline, reflux, 2 h: 18%).[1270]

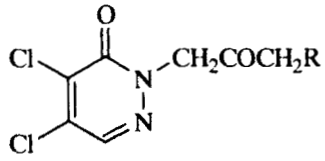
Also other examples.[534, 1257]




(**395**)




(**396**)



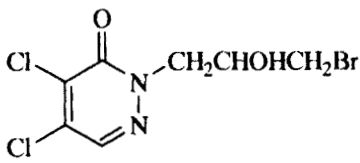

(**397**)

### With Azide Ion

2-γ-Bromoacetonyl- (**397**, R = Br) gave 2-γ-azidoacetonyl-4,5-dichloro-3(2*H*)-pyridazinone (**397**, R = $N_3$) ($NaN_3$, AcMe–$Me_2NCHO$, 25°, 5 h: 91%; note selectivity for ω-Br under these mild conditions).[1680]

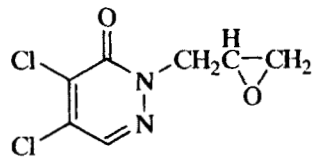
### Hydrolysis via an Epoxide

2-(γ-Bromo-β-hydroxypropyl)-4,6-dichloro- (**398**) gave 4,6-dichloro-2-(β,γ-epoxypropyl)- (**399**) (KOH, $H_2O$–THF–AcMe, 25°, 45 min; 89%) and



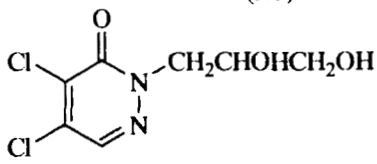

(**398**)

HO⁻




(**399**)

HO⁻

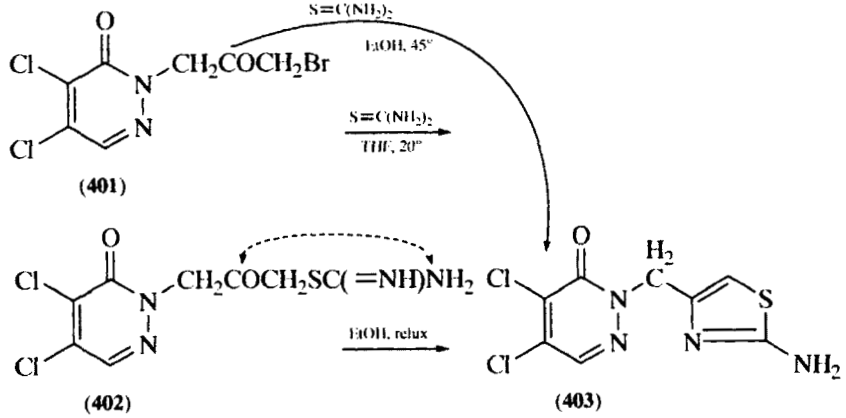



(**400**)

then 4,6-dichloro-2-(β,γ-dihydroxypropyl)-3(2*H*)-pyridazinone (**400**) (dilute HCl, 40°, 5 h: 56%).[1680]

### With Thiourea

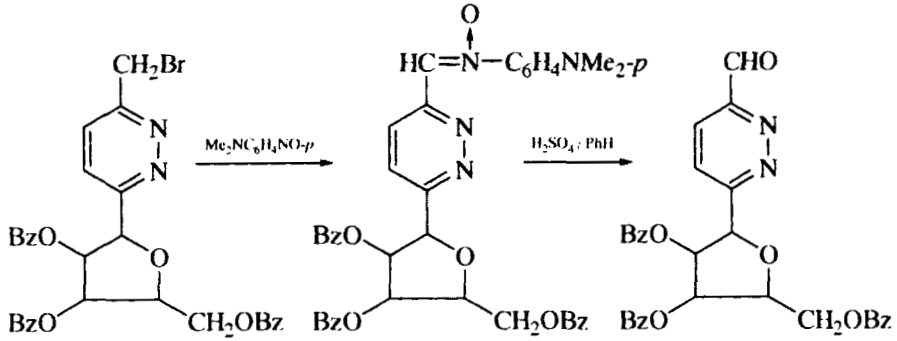
2-γ-Bromoacetonyl- (**401**) gave 2-γ-(amidinothio)acetonyl- (**402**) [S=C(NH$_2$)$_2$, THF, 25°, 20 min: 54%] and thence 2-(2-aminothiazol-4-yl)methyl-4,5-dichloro-3(2*H*)-pyridazinone (**403**) (EtOH, reflux, 20 min: 72%);[1680] the substrate (**401**) gave the final product (**403**) more effectively without isolation of the intermediate (**402**) [S=C(NH$_2$)$_2$, EtOH, 45°, 20 min: 72%].[1680]




(**401**) (**402**) (**403**)

### With *N,N*-Dimethyl-*p*-nitrosoaniline

3-Bromomethyl-6-(2,3,5-tri-*O*-benzoyl-β-D-ribofuranosyl)pyridazine (**404**) gave the nitrone, *p*-dimethylamino-*N*-[6-(2,3,5-tri-*O*-benzoyl-β-D-ribofuranosyl)pyridazin-3-ylmethyl]aniline *N*-oxide (**405**) (substrate, pyridine, 25°, 1 h; then pyridine↑; then *p*-Me$_2$NC$_6$H$_4$NO↓, MeOH, 25°, 10 min; then Na$_2$CO$_3$, H$_2$O, 25°, 1 h: 48%; via an unisolated pyridinio intermediate) and thence 6-(2,3,5-tri-*O*-benzoyl-β-D-ribofuranosyl)-3-pyridazinecarbaldehyde (**405a**) (H$_2$SO$_4$, PhH, 5°→20°, 3 h: 79%).[1539]

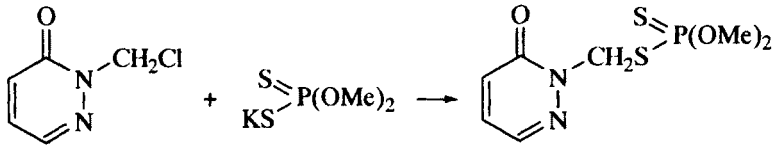



(**404**) (**405**) (**405a**)

## Acyloxylation

*Note:* Although no simple examples using carboxylates appear to be available, similar reactions with phosphates and thiophosphates have been reported.

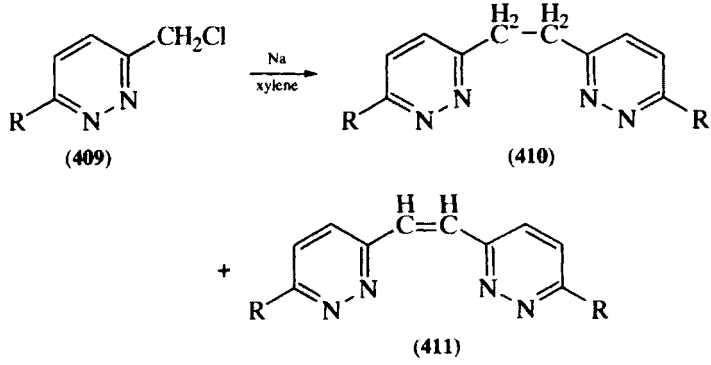
2-Chloromethyl-3(2*H*)-pyridazinone (**406**) with the potassium salt of *O,O*-dimethyl dithiophosphate (**407**) gave 2-(dimethoxyphosphinothioylthiomethyl)-3(2*H*)-pyridazinone (**408**) ($H_2O$, 60°, vigorous stirring, 2 h: 84%);[441] also many analogues of interest as potential insecticides.[441]



(406) (407) (408)

## Würtz Coupling

3-Chloromethylpyridazine (**409**, R = H) underwent Würtz coupling[1852c] to give α,β-bispyridazin-3-ylethane (**410**, R = H) as the only isolable product (Na, xylene, reflux, 12 h: 3%);[767] in contrast, 3-chloromethyl-6-



(409) (410)

(411)

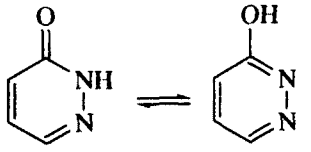
methoxypyridazine (**409**, R = OMe) gave a separable mixture of α,β-bis(6-methoxypyridazin-3-yl)ethane (**410**, R = OMe) and the corresponding ethylene (**411**, R = OMe) (likewise: 12% and 7%, respectively).[767]

*Chemistry of Heterocyclic Compounds, Volume 57*
D. J. Brown
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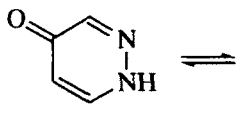
CHAPTER 5

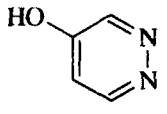
# Oxypyridazines (*H* 23)

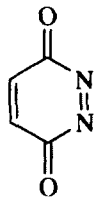
The conveniently general term *oxypyridazines* is employed here to include derivatives such as the cycloamidic tautomeric pyridazines (**1** or **2**), pyridazine quinones (**3**), the alcoholic hydroxylalkylpyridazines (**4**), the etherial alkoxypyridazines (**5**, **6**, or **7**), the ester-like acyloxypyridazines (**8** or **9**), the cycloamidic nontautomeric pyridazinones (**10** or **11**), and the pyridazine *N*-oxides (**12**).

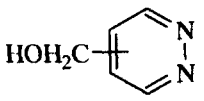

(1a)

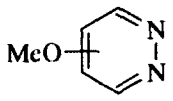

(1b)

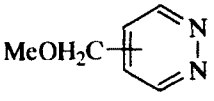

(2a)

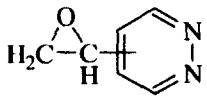

(2b)

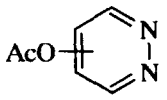

(3)

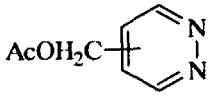

(4)

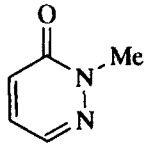

(5)

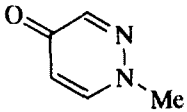

(6)

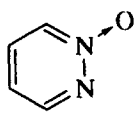

(7)


(8)


(9)


(10)


(11)

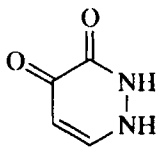

(12)

Recent contributions to the chemistry of each such group are covered in appropriate sections of this chapter. More general ancillary information of oxypyridazines is collected in a final section; this includes brief indications of recent theoretical, physical, spectral, and structural studies, as well as an alphabetical list of recently used trivial names for many pyridazine drugs, agrochemicals, natural products, and so on

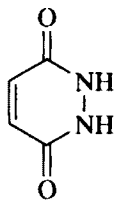
Several comprehensive general reviews, covering oxypyridazines inter alia, have appeared in recent years.[1188,1575,1853] In addition, detailed reviews of pharmacologically active pyridazines (mainly oxypyridazines) have appeared in the 1990s,[1575,1777,1782] thereby precluding any necessity to cover such material in this book. Agrochemical pyridazines have been reviewed much less thoroughly.[933] Synthetic routes to 5-hydroxy-3(2*H*)-pyridazinones and derivatives have been reviewed thoughtfully.[2064]

## 5.1 TAUTOMERIC PYRIDAZINONES (*H* 23)

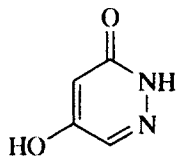
Both 3- (**1b**) and 4-pyridazinol (**2b**) are confidently considered to exist almost entirely as the pyridazinones (**1a**) and (**2a**), respectively; 3,4- and 3,6-pyridazinediol can adopt dione forms (**13**) and (**14**), respectively, but the experimental evidence seems to favor an hydroxypyridazinone form in each case (see Section 5.7.2); 3,5- and 4,5-pyridazinediol can adopt the hydroxypyridazinone forms (**15**) and (**16**), respectively, but not regular dione forms; and the pyridazinetriols and pyridazinetetrol can adopt dione forms. For obvious pragmatic reasons, all such entities are named as pyridazinediones when possible or otherwise as lactamic pyridazinones or lactamic hydroxypyridazinones as appropriate.



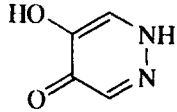

(13)




(14)




(15)




(16)

### 5.1.1 Preparation of Tautomeric Pyridazinones (*H* 24)

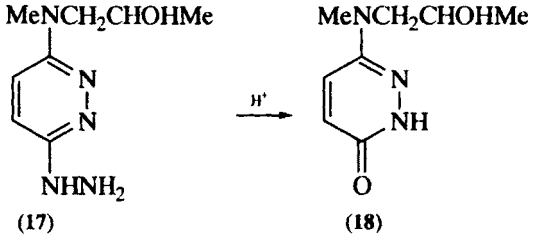
Most such pyridazinones have been made by *primary syntheses* (see Chapters 1 and 2) or by *hydrolysis of halogenopyridazines* (see Section 4.2.2); the remainder have been made by various hydrolytic displacement reactions or other miscellaneous procedures, as illustrated in the following examples:

## From Pyridazinamines

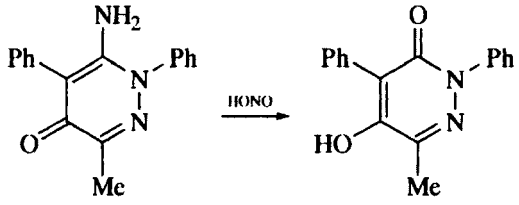
3-Hydrazino-6-(*N*-β-hydroxypropyl-*N*-methylamino)pyridazine (**17**) gave 6-(*N*-β-hydroxypropyl-*N*-methylamino)-3(2*H*)-pyridazinone (**18**) (1M HCl, reflux, 1 : 25%).[1331]

6-Amino-3-methyl-1,5-diphenyl-4(1*H*)-pyridazinone (**19**) gave 5-hydroxy-6-methyl-2,4-diphenyl-3(2*H*)-pyridazinone (**20**) ($HNO_2$: ?%).[911]

Also other examples.[588,713]




(17) (18)



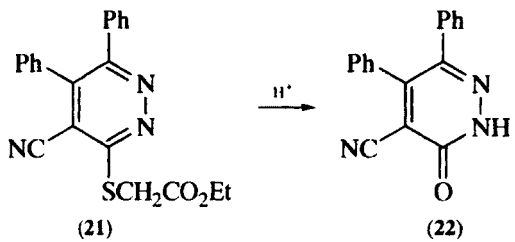

(19) (20)

## From Alkylthiopyridazines

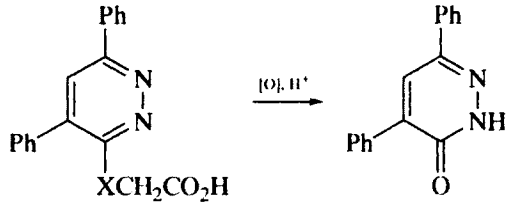
3-Ethoxycarbonylmethylthio-5,6-diphenyl-4-pyridazinecarbonitrile (**21**) gave 3-oxo-5,6-diphenyl-2,3-dihydro-4-pyridazinecarbonitrile (**22**) (HCl–$H_2O$, reflux, 2 h: ?%).[1390]

3-Carboxymethylthio-4,6-diphenylpyridazine (**23**, X = S) gave 4,6-diphenyl-3(2*H*)-pyridazinone (**24**) [AcOH, 30% $H_2O_2$, 25°, 4 h: ?%; probably by hydrolysis of an intermediate sulfoxide (**23**, X = SO) or sulfone (**23**, $X = SO_2$)].[1264]

Also other examples.[1116,1264]




(21) (22)



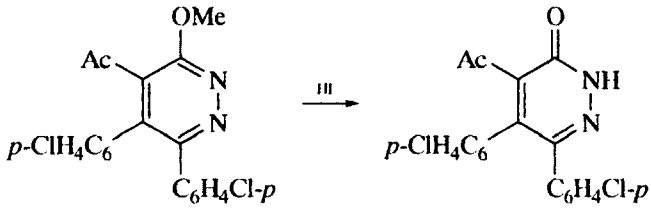

(23) (24)

## From Alkoxypyridazines

With hydriodic acid:

4-Acetyl-5,6-bis-*p*-chlorophenyl-3-methoxypyridazine (**25**) gave 4-acetyl-5,6-bis-*p*-chlorophenyl-3(2*H*)-pyridazinone (**26**) (HI, MeOH, 80°, 1 h: 95%).[1610]

Also other examples.[1279]




(25) (26)

With hydrobromic acid:

5-Methoxy- (**27**, R = Me) gave 5-hydroxy-3(2*H*)-pyridazinone (**27**, R = H) (47% HBr–AcOH, reflux, 10 h: 94%).[90]

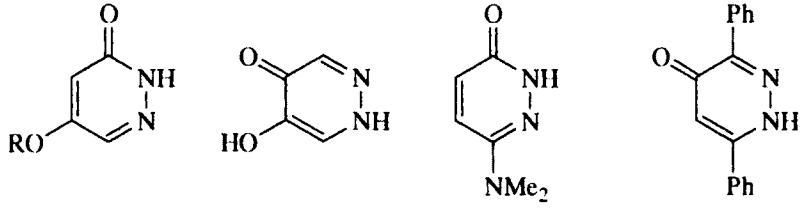
4,5-Dimethoxypyridazine gave 5-hydroxy-4(1*H*)-pyridazinone (**28**) (48% HBr, reflux, 3 h: 89%).[923]

Also other examples.[523,905,1574]

With hydrochloric acid:

3-Benzyloxy-6-dimethylaminopyridazine gave 6-dimethylamino-3(2*H*)-pyridazinone (**29**) (5M HCl, reflux, 4 h: 43%).[94]

4-Ethoxy-3,6-diphenylpyridazine gave 3,6-diphenyl-4(1*H*)-pyridazinone (**30**) (10M HCl, 150°, sealed 6 h: 92%).[451]

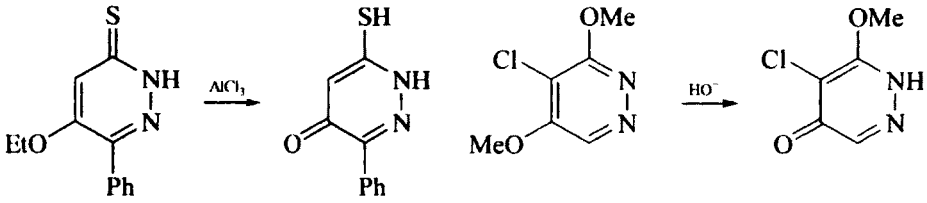



(27) (28) (29) (30)

Also other examples.[1013,1087,1169,1826]

With aluminium chloride:

5-Ethoxy-6-phenyl-3(2*H*)-pyridazinethione (**31**) or 3-*t*-butylthio-5-ethoxy-6-phenylpyridazine gave 6-mercapto-3-phenyl-4(1*H*)-pyridazinone (**32**) ($AlCl_3$, PhMe, reflux, 1 h: 85% or 70%, respectively).[1087]



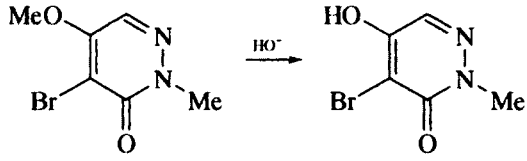

(31) (32) (33) (34)

With alkali or in other basic media:

4-Chloro-3,5-dimethoxypyridazine (**33**) gave only 5-chloro-6-methoxy-4(1*H*)-pyridazinone (**34**) (2.5M NaOH, reflux, 4.5 h: 87%; the reason for such selectivity is not evident).[882]

4-Bromo-5-methoxy- (**35**) gave 4-bromo-5-hydroxy-2-methyl-3(2*H*)-pyridazinone (**36**) [KOH (1 equiv), $H_2O$, reflux, 3 h: ~90%]; also many homologues.[1936]

Also other examples.[224,531,694,760,766,791,877,1597]

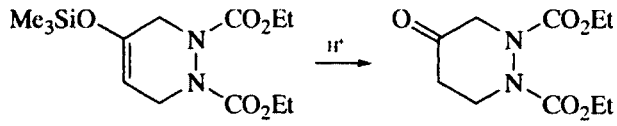



(35) (36)

## From Trialkylsiloxypyridazines

Diethyl 4-trimethylsiloxy-1,2,3,6-tetrahydro-1,2-pyridazinedicarboxylate (**37**) gave diethyl 4-oxohexahydro-1,2-pyridazinedicarboxylate (**38**) (MeOH, $F_3CCO_2H$, 25°, 24 h: ~90%).[1699]

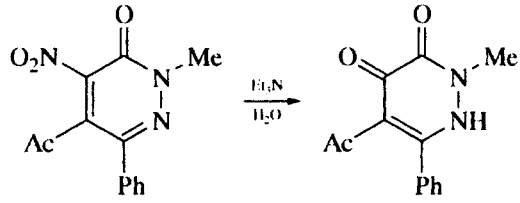
Also other examples.[19]




(37) (38)

## From Nitropyridazines

5-Acetyl-2-methyl-4-nitro-6-phenyl-3(2*H*)-pyridazinone (**39**) gave 5-acetyl-2-methyl-6-phenyl-3,4(1*H*,2*H*)-pyridazinedione (**40**) ($Et_3N$, MeCN–$H_2O$, 25°, 2 h: >67%).[156]

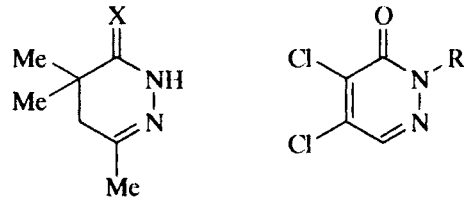


(**39**) (**40**)

## From Miscellaneous Substrates

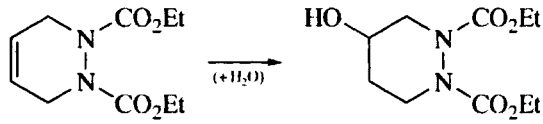
4,4,6-Trimethyl-4,5-dihydro-3(2*H*)-pyridazinone oxime (**41**, X = NOH) gave the free pyridazinone (**41**, X = O) (6M HCl, reflux, 2 h: 88%).[4]

4,5-Dichloro-2-hydroxymethyl-3(2*H*)-pyridazinone (**42**, R = $CH_2OH$) gave 4,5-dichloro-3(2*H*)-pyridazinone (**42**, R = H) ($K_2CO_3$, MeCN, reflux, 2 h: >90%; with loss of $CH_2O$).[1901]



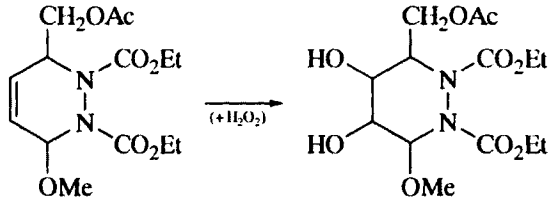
(**41**) (**42**)

Diethyl 1,2,3,6-tetrahydro-1,2-pyridazinedicarboxylate (**43**) gave diethyl 4-hydroxyhexahydro-1,2-pyridazinedicarboxylate (**44**) [$BH_3$, THF, 0–5°, 2.5 h; then $H_2O\downarrow$; then NaOH, $H_2O_2$, < 5°, 2 h: 66%; or $Hg(OAc)_2$, $H_2O$–THF, 25°, 4 h; then $NaBH_4$, NaOH, $H_2O$: 75%].[1671]



(**43**) (**44**)

Diethyl 3-acetoxymethyl-6-methoxy-1,2,3,6-tetrahydro-1,2-pyridazinedicarboxylate (**45**) gave diethyl 3-acetoxymethyl-6-methoxy-4,5-dihydroxyhexahydro-1,2-pyridazinedicarboxylate (**46**) [$O(CH_2CH_2)_2N(\rightarrow O)$ Me·$2H_2O$, AcMe–$H_2O$–Bu$^t$OH, trace $OsO_4$, 25°, 5 days: 83%].[734]




(45) (46)

2-Benzyl-4-ethoxy-5-morpholino-3(2*H*)-pyridazinone (a fixed pyridazinone) gave 4-ethoxy-5-morpholino-3(2*H*)-pyridazinone [Pd/C, AcOH, $H_2$, (3–4 atm), 36 h: 31%];[1269] 5-methoxy-2-tetrahydropyran-2′-yl-3(2*H*)-pyridazinone gave 5-methoxy-3(2*H*)-pyridazinone (HCl, $H_2O$–MeOH, reflux, 2 h: 65%);[1821] and like reactions.[218,240,1794]

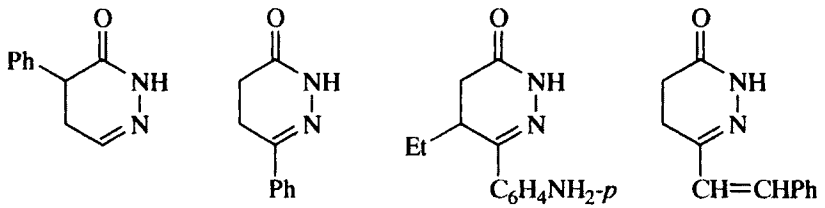
**From Hydropyridazinones**

4-Phenyl-4,5-dihydro- (**47**) gave 4-phenyl-3(2*H*)-pyridazinone ($Br_2$, AcOH, 70°, 2 min: ~80%);[738] 6-phenyl-4,5-dihydro- (**48**) gave 6-phenyl-3(2*H*)-pyridazinone ($CuCl_2$, MeCN, reflux, 30 min: ~88%).[1823]

6-*p*-Aminophenyl-5-ethyl-4,5-dihydro- (**49**) gave 6-*p*-aminophenyl-5-ethyl-3(2*H*)-pyridazinone ($O_2NC_6H_4SO_3Na$-*m*, 0.7M NaOH, reflux, 6.5 h: 67%).[1310]

6-Styryl-4,5-dihydro- (**50**) gave 6-styryl-3(2*H*)-pyridazinone ($SeO_2$ EtOH, reflux, 8 h: 87%).[1282]

Also other examples.[1267,1371,1405,2001]




(47) (48) (49) (50)

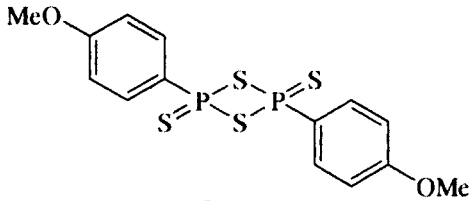
## 5.1.2 Reactions of Tautomeric Pyridazinones (*H* 37)

The important conversion of *pyridazinones into halogenopyridazines* was covered in Section 4.1.1. The remaining reactions are discussed in subsections hereafter. Selected reactions have been reviewed.[2063]

### *5.1.2.1 Conversion into Pyridazinethiones (H 764)*

Most tautomeric pyridazinones undergo thiation quite readily on treatment with phosphorus pentasulfide in hot pyridine, toluene, or the like; it usually pays

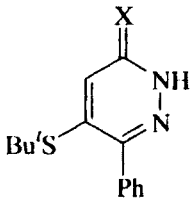
to use good-quality phosphorus pentasulfide or to purify poor-quality reagent beforehand.[1977] Lawesson's reagent, 2,4-bis-*p*-methoxyphenyl-1,3-dithia-2,4-diphosphetane-2,4-disulfide (**51**),[1986.1987] has been used occasionally. Typical thiations are illustrated in the following examples:



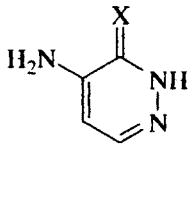
(51)

5-*t*-Butylthio-6-phenyl-3(2*H*)-pyridazinone (**52**, X = O) gave the corresponding thione (**52**, X = S) ($P_2S_5$, PhMe, reflux, 30 min: 72%).[1087]

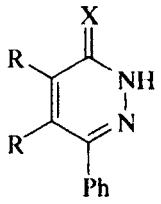
4-Amino-3(2*H*)-pyridazinone (**53**, X = O) gave the corresponding thione (**53**, X = S) ($P_2S_5$, pyridine, reflux, 14 h: 62%).[923]



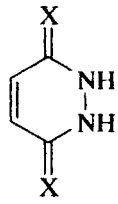
(52)



(53)



(54)

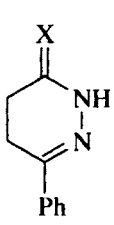


(55)

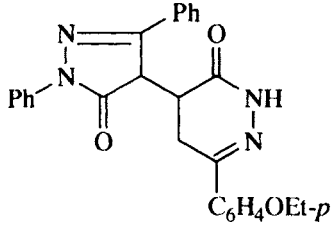
6-Phenyl- (**54**, R = H, X = O) and 4,5,6-triphenyl-3(2*H*)-pyridazinone (**54**, R = Ph, X = O) gave the corresponding thiones (**54**, R = H, X = S) ($P_2S_5$, pyridine, reflux, 3 h: 60%)[413] and (**54**, R = Ph, X = S) (likewise, 4 h: 56%).[1362]

3,6(1*H*,2*H*)-Pyridazinedione (**55**, X = O) gave the corresponding dithione (**55**, X = S) ($P_2S_5$, pyridine, reflux, 45 min: 64%).[365]

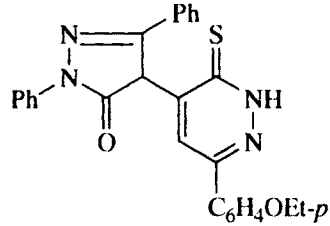
6-Phenyl-4,5-dihydro-3(2*H*)-pyridazinone (**56**, X = O) gave the corresponding dihydrothione (**56**, X = S) [Lawesson's reagent (**51**), PhH, reflux, 105 min: >95%].[184]



(56)



(57)



(58)

6-*p*-Ethoxyphenyl-4-(5-oxo-1,3-diphenyl-2-pyrazolin-4-yl)-4,5-dihydro-3(2*H*)-pyridazinone (**57**) gave 6-*p*-ethoxyphenyl-4-(1,3-diphenyl-5-thioxo-2-pyrazolin-4-yl)-3(2*H*)-pyridazinethione (**58**) [Lawesson's reagent (**51**), xylene, reflux, 6 h: 70%; note the additional thiation in the five-membered ring and dehydrogenation of the six-membered ring].[1103, cf. 1371]

Also other examples.[112,159,249,315,362,371,373,375,539,808,811,819,946,973,1056,1064,1127, 1278,1390,1450,1462,1712,1726,1758,2019]

### *5.1.2.2 Conversion into O- or N-Alkyl Derivatives (H 37)*

Irrespective of the type of reagent or the conditions used, alkylation of a tautomeric 3(2*H*)- or 4(1*H*)-pyridazinone usually gives an *N*-alkylpyridazinone sometimes accompanied by a smaller amount of the isomeric alkoxypyridazine. Occasionally, the alkoxypyridazine may predominate, especially when a diazoalkane is used or when the steric and/or electronic factors associated with the reagent or substrate are favorable; in this regard, appropriately placed electron-withdrawing passenger groups or partial reduction of the ring seems, particularly prone to affect the outcome appreciably.

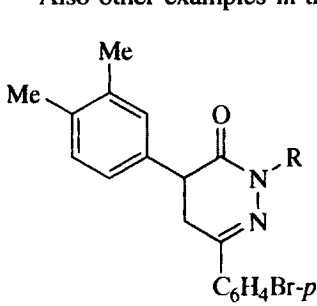
The formation of the following products illustrate the results to be expected on alkylation of various types of tautomeric pyridazinone using a variety of reagents and conditions. The examples are grouped according to the type of substrate and the given percentages represent isolated yields (for some trialkylsilylation reactions, see Section 5.4.2.3):

**From Simple 3(2*H*)-pyridazinones: *O*-Alkylation**

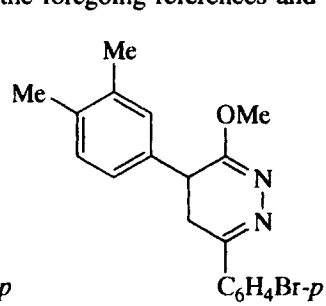
6-*p*-Bromophenyl-4-(3,4-dimethylphenyl)-4,5-dihydro-3(2*H*)-pyridazinone (**59**, R = H) gave its 2-methyl derivative (**59**, R = Me) and 3-*p*-bromophenyl-5-(3,4-dimethylphenyl)-6-methoxy-4,5-dihydropyridazine (**60**) ($Me_2SO_4$, $K_2CO_2$, AcMe, reflux, 10 h: 50% and 30%, respectively).[1278]

6-*p*-Bromophenyl-4-(3-isopropyl-6-methylphenyl)-4,5-dihydro-3(2*H*)-pyridazinone gave 3-*p*-bromophenyl-6-ethoxycarbonylmethoxy-5-(3-isopropyl-6-methylphenyl)-4,5-dihydropyridazine (**61**) ($EtO_2CCH_2Cl$, $K_2CO_3$, AcMe, reflux, 10 h: 28%; no isomer mentioned).[1267]

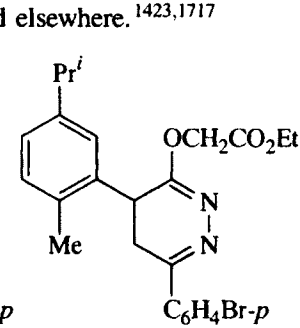
Also other examples in the foregoing references and elsewhere.[1423,1717]




(59)



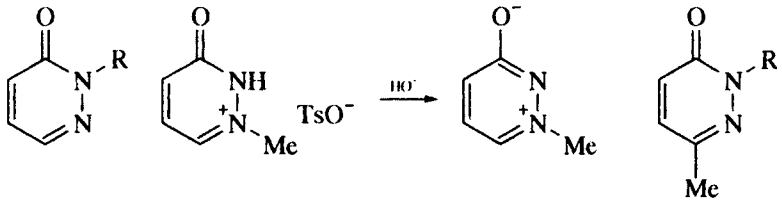

(60)




(61)

## From Simple 3(2*H*)-Pyridazinones: *N*-Alkylation

3(2*H*)-Pyridazinone (**62**, R = H) gave 2-carboxymethyl-3(2*H*)-pyridazinone (**62**, R = $CH_2CO_2H$) ($ClCH_2CO_2H$, NaOH, $H_2O$, reflux, 1 h: 67%),[147] 2-β-hydroxyethyl-3(2*H*)-pyridazinone (**62**, R = $CH_2CH_2OH$) (ethylene oxide, trace MeONa, $Me_2NCHO$, 90°, 4 h: 70%),[1179] or 1-methyl-3-oxo-2,3-dihydropyridazinium *p*-toluenesulfonate (**63**) [TsOMe,petroleum (b.p. >130°), 130°, 12 h: 45%] and then 1-methylpyridazinium-3-olate (**64**) [Amberlite IRA-401 ($HO^-$), $H_2O$: 70%].[668]



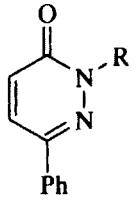

(**62**) (**63**) (**64**) (**65**)

6-Methyl-3(2*H*)-pyridazinone (**65**, R = H) gave 6-methyl-2-phenacyl-3(2*H*)-pyridazinone (**65**, R = $CH_2Bz$) ($BzCH_2Br$, $NaHCO_3$, MeCN, reflux, 14 h: 95%;[1594] $BzCH_2Cl$, KOH, $Me_2NCHO$: 40%).[530]

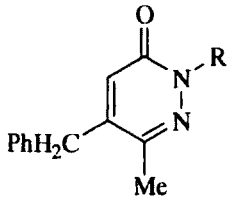
6-Phenyl-3(2*H*)-pyridazinone (**66**, R = H) gave 2-methyl-6-phenyl- (**66**, R = Me) (MeI, MeOH, 100°, sealed, 6 h: 84%),[919] 2-hydroxymethyl-6-phenyl- (**66**, R = $CH_2OH$) ($CH_2O$, $H_2O$, reflux, 1 h: 88%),[1292] 2-β-hydroxyethyl-6-phenyl- (**66**, R = $CH_2CH_2OH$) (substrate K salt, $HOCH_2CH_2Cl$, $Me_2NCHO$–PhH, 0° → 120°, 24 h: 74%),[1292] 2-β-bromoethyl-6-phenyl- (**66**, R = $CH_2CH_2Br$) (excess $BrCH_2CH_2Br$, KOH, trace $Bu_4NBr$, PhH, 25°, 6 h: 84%),[1337] 6-phenyl-2-vinyl- (**66**, R = CH : $CH_2$) [AcOCH = $CH_2$, trace $Hg(OAc)_2$, trace $H_2SO_4$, $CHCl_3$, <60°, 8 h: 60%;[1179] or HC≡CH, dioxane or THF, pressure, ?°: <60%],[693] 2-γ-cyanopropyl-6-phenyl- (**66**, R = $CH_2CH_2CH_2CN$) ($BrCH_2CH_2CH_2$ CN, KOH, trace $Bu_4NBr$, PhH, 25°, 6 h: 88%),[1288] or 2-β-cyanoethyl-6-phenyl-3(2*H*)-pyridazinone (**66**, R = $CH_2CH_2CN$) ($H_2C$=CHCN, $PhCH_2Me_3NOH$, EtOH, reflux, 8 h: 76%);[1288] also analogues.[368]

5-Benzyl-6-methyl-3(2*H*)-pyridazinone (**67**, R = H) gave 5-benzyl-2-ethoxycarbonylmethyl-6-methyl- (**67**, R = $CH_2CO_2Et$) (substrate Na salt, $BrCH_2CO_2Et$, EtOH, 25° → reflux, 24 h: 98%),[1446] 5-benzyl-6-methyl-2-mopholinomethyl- [**67**, R = $CH_2N(CH_2CH_2)_2O$] [$CH_2O$, $HN(CH_2CH_2)_2O$, AcOH, $H_2O$–EtOH, reflux, 6 h: 69%; a Mannich aminoalkylation],[884] or 5-benzyl-2-β-dimethylaminoethyl-6-methyl-3(2*H*)-pyridazinone (**67**, R = $CH_2CH_2NMe_2$) ($ClCH_2CH_2NMe_2$, EtONa, EtOH, 25° → reflux, 12 h: 96%, as hydrochloride).[884]

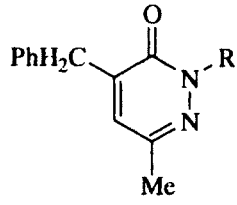
4-Benzyl-6-methyl-3(2*H*)-pyridazinone (**68**, R = H) gave 4-benzyl-2-β-cyanoethyl-6-methyl-3(2*H*)-pyridazinone (**68**, R = $CH_2CH_2CN$) ($H_2C$=CHCN, trace NaOH, EtOH, reflux, 12 h: 82%).[112]



(66)



(67)



(68)

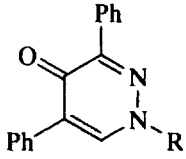
Also other examples.[118,162,350,433,434,441,530,698,775,855,874,888,990,994,998,1006,1035,1039,1244,1253,1268,1276,1282,1289,1323,1343,1425,1475,1716,1771,1837,1912]

### From Simple 4(1*H*)-Pyridazinones: *N*-Alkylation

No examples in this category have been reported recently.

### From simple 4(1*H*)-Pyridazinones: *N*-Alkylation

3,5-Diphenyl-4(1*H*)-pyridazinone (**69**, R = H) gave 1-methyl-3,5-diphenyl-4(1*H*)-pyridazinone (**69**, R = Me) ($Me_2SO_4$, pH 12 buffer, ?°, ?h: > 95%: MeI, NaH, $Me_2NCHO$, ?°, ?h: > 95%);[249] also some substituted-phenyl and/or *N*-ethyl (or propyl) analogues.[249]



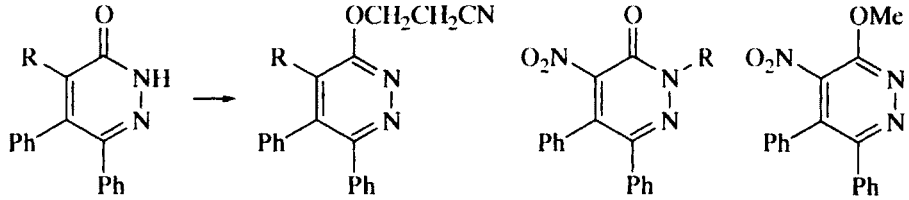
(69)

### From Functionally Substituted 3(2*H*)-Pyridazinones: *O*-Alkylation

3-Oxo-5,6-diphenyl-2,3-dihydro-4-pyridazinecarbonitrile (**70**, R = CN) gave 3-*β*-cyanoethoxy-5,6-diphenyl-4-pyridazinecarbonitrile (**71**, R = CN) ($H_2C$=CHCN, trace pyridine or NaOH, EtOH, reflux, 12 h: 73%); ethyl 3-oxo-5,6-diphenyl-2,3-dihydro-4-pyridazinecarboxylate (**70**, R = $CO_2$ Et) gave ethyl 3-*β*-cyanoethoxy-5,6-diphenyl-4-pyridazinecarboxylate (**71**, R = $CO_2$Et) (likewise: 75%).[389]

4-Nitro-5,6-diphenyl-3(2*H*)-pyridazinone (**72**, R = H) gave a separable mixture of its 2-methyl derivative (**72**, R = Me) and 3-methoxy-4-nitro-5,6-diphenylpyridazine (**73**) ($CH_2N_2$, $Et_2O$, 25°).[795]

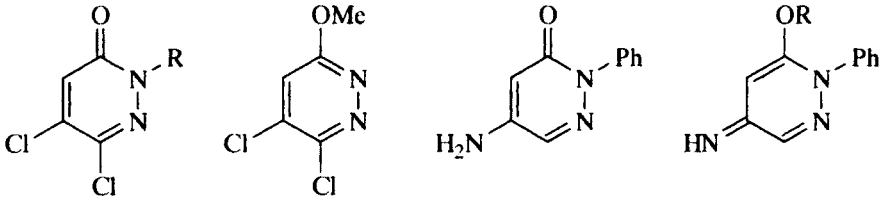
5,6-Dichloro-3(2*H*)-pyridazinone (**74**, R = Me) gave a separable mixture of its 2-methyl derivative (**74**, R = Me) and 3,4-dichloro-6-methoxypyridazine (**75**) ($CH_2N_2$, $Et_2O$ –AcMe, 25°, until no $N_2$ ↑: 52% and 16%, respectively; MeI, $K_2CO_3$, $Me_2NCHO$, 25°, 3 h: 67% and 0%, respectively).[881]



(70) (71) (72) (73)

5-Amino-2-phenyl-3(2*H*)-pyridazinone (**76**), which is tautomeric with 5-imino-2,5-dihydro-2-phenyl-3-pyridazinol (**77**, R = H), gave 6-methoxy-1-phenyl-4(1*H*)-pyridazinimine (**77**, R = Me) ($Me_2SO_4$ $CHCl_3$, reflux, 2 h: 61%, initially as the methyl hydrogen sulfate salt and thence as hydrochloride).[1344]

Also other examples.[390,547,552,595,608,851,1056,1326,1918,1950,2058]



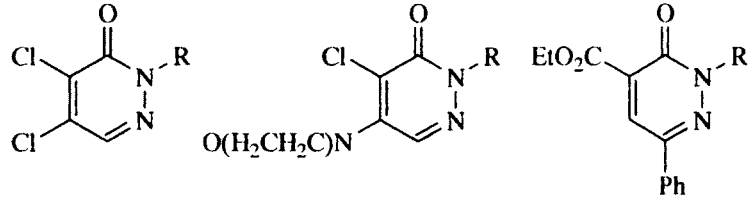
(74) (75) (76) (77)

## From Functionally Substituted 3(2*H*)-Pyridazinones: *N*-Alkylation

4,5-Dichloro-3(2*H*)-pyridazinone (**78**, R = H) gave 4,5-dichloro-2-methyl- (**78**, R = Me) (MeI, $K_2CO_3$, $Me_2NCHO$, 50°, 2 h: > 86%),[1936] 4,5-Dichloro-2-γ-dimethylaminopropyl- (**78**, R = $CH_2CH_2CH_2NMe_2$) (substrate Na salt, $ClCH_2CH_2CH_2NMe_2$, EtOH, reflux, 1 h: 58%, as hydrochloride),[533] or benzyloxymethyl-4,5-dichloro-3(2*H*)-pyridazinone (**78**, R = $CH_2OCH_2Ph$) ($ClCH_2OCH_2Ph$, 1,8-diazabicyclo[5.4.0]undec-7-ene, $Me_2NCHO$, 0° → 25°, 2 h: 58%).[1048]

4-Chloro-5-morpholino-3(2*H*)-pyridazinone (**79**, R = H) gave 2-allyl-4-chloro-5-morpholino-3(2*H*)-pyridazinone (**79**, R = $CH_2CH{:}CH_2$) ($BrCH_2CH{=}CH_2$, $K_2CO_3$, AcMe, reflux, 4 h: 84%).[1269]

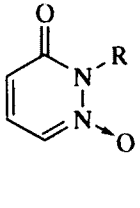
Ethyl 3-oxo-6-phenyl-2,3-dihydro-4-pyridazinecarboxylate (**80**, R = H) gave ethyl 2-ethoxycarbonylmethyl-3-oxo-6-phenyl-2,3-dihydro-4-pyridazinecarboxylate (**80**, R = $CH_2CO_2Et$) ($BrCH_2CO_2Et$, $K_2CO_3$, AcMe, reflux, 24 h: 91%).[1767]



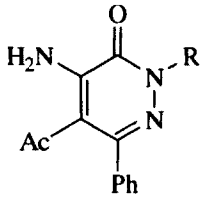
(78) (79) (80)

3(2*H*)-Pyridazinone 1-oxide (**81**, R = H) gave 2-methyl-3(2*H*)-pyridazinone 1-oxide (**81**, R = Me) ($CH_2N_2$, $Et_2O$–$CH_2Cl_2$, 25°, until homogeneous: >70%); also homologues.[789]

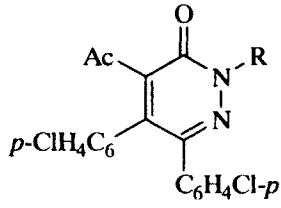
5-Acetyl-4-amino-6-phenyl-3(2*H*)-pyridazinone (**82**, R = H) gave 5-acetyl-2-allyl-4-amino-6-phenyl-3(2*H*)-pyridazinone (**82**, R = $CH_2CH{:}CH_2$) ($BrCH_2CH{=}CH_2$, $K_2CO_3$, $Me_2NCHO$, 25°, 30 min: 75%).[1832]



(**81**)

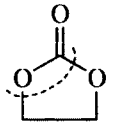


(**82**)

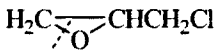


(**83**)

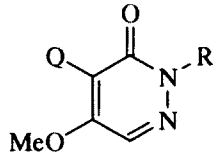
4-Acetyl-5,6-bis-*p*-chlorophenyl-3(2*H*)-pyridazinone (**83**, R = H) gave 4-acetyl-5,6-bis-*p*-chlorophenyl-2-β-hydroxyethyl-3(2*H*)-pyridazinone (**83**, R = $CH_2CH_2OH$) [ethylene carbonate (**84**), $K_2CO_3$ or KOH, $Me_2NCHO$, 90–95°, 2 h: 99%][156,1332] or a separable mixture of 4-acetyl-2-(γ-chloro-β-hydroxypropyl)-5,6-bis-*p*-chlorophenyl- (**83**, R = $CH_2CHOHCH_2Cl$) and 4-acetyl-5,6-bis-*p*-chlorophenyl-2-(2,3-epoxypropyl)-3(2*H*)-pyridazinone (**83**, R = 2,3-epoxypropyl)[epichlorhydrin (**85**), $K_2CO_3$, $Me_2NCHO$, 80°, 2 h: 10% each; the first by fission of the epoxide, the second by a regular alkylation process].[1272]



(**84**)



(**85**)



(**86**)

5-Methoxy-3(2*H*)-pyridazinone (**86**, Q = R = H) gave 5-methoxy-2-methyl-3(2*H*)-pyridazinone (**86**, Q = H, R = Me) (neat MeI, $K_2CO_3$, reflux, 3 h: 51%;[90] MeI, $K_2CO_3$, $Me_2NCHO$, 50°, $N_2$, 15 h: 18%;[1821] $Me_2SO_4$, $K_2CO_3$, PhMe, reflux, 8 h: 55%;[90] structure confirmed by X-ray analysis);[90] 4-chloro-5-methoxy-3(2*H*)-pyridazinone (**86**, Q = Cl, R = H) gave 4-chloro-5-methoxy-2-methyl-3(2*H*)-pyridazinone (**86**, Q = Cl, R = Me) ($Me_2SO_4$, NaOH, $H_2O$, 25°, 48 h: 51%).[90]

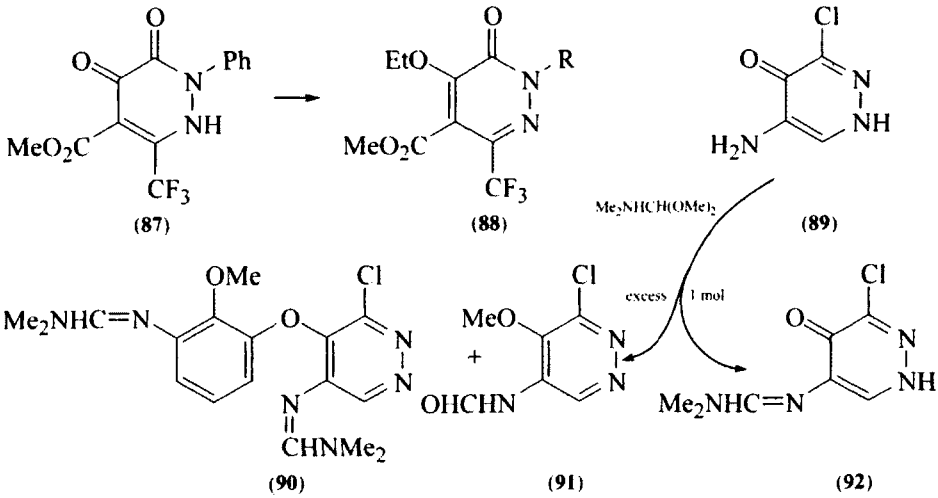
Also other examples.[534,593,676,814,994,999,1004,1055,1450,1460,1540,1742,1749,1780,1781,1964,2039]

## From Functionally Substituted 4(1*H*)-Pyridazinones: *O*-Alkylation

Methyl 5,6-dioxo-1-phenyl-3-trifluoromethyl-1,2,5,6-tetrahydro-4-pyridazinecarboxylate (**87**) gave methyl 5-ethoxy-6-oxo-1-phenyl-3-trifluoromethyl-

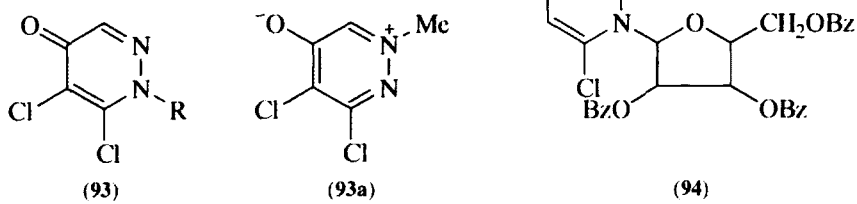
1,6-dihydro-4-pyridazinecarboxylate (**88**) (EtI, $K_2CO_3$, AcMe, reflux, 36 h: 83%).[1696]

5-Amino-3-chloro-4(1*H*)-pyridazinone (**89**) gave either 3-chloro-5-dimethylaminomethyleneamino-4(1*H*)-pyridazinone (**92**) [$Me_2NCH(OMe)_2$ (1 equiv), PhMe, 25°, 20 h: 73%] or a separable mixture of 3-chloro-5-formamido-4-methoxypyridazine (**91**) and 3-chloro-5-dimethylaminomethyleneaminopyridazin-4-yl 5-dimethylaminomethyleneamino-4-methoxypyridazin-3-yl ether (**90**) [excess $Me_2NCH(OMe)_2$, PhMe, reflux, 8 h: 27% and 35%, respectively; both probably formed from the initial product (**92**) by easily rationalized processes including *O*-alkylations].[83]




## From Functionally Substituted 4(1*H*)-Pyridazinones: *N*-Alkylation

5,6-Dichloro-4(1*H*)-pyridazinone (**93**, R = H) gave 5,6-dichloro-1-methyl-4(1*H*)-pyridazinone (**93**, R = Me) ($Me_2SO_4$, MeONa, MeOH, 20° → reflux, 1 h: ~3%, after a complicated purification)[141] or 5,6-dichloro-2-methylpyridazinium-4-olate (**93a**) ($Me_2SO_4$, PhMe, reflux, 4 h; then $H_2O$: 55%).[699]

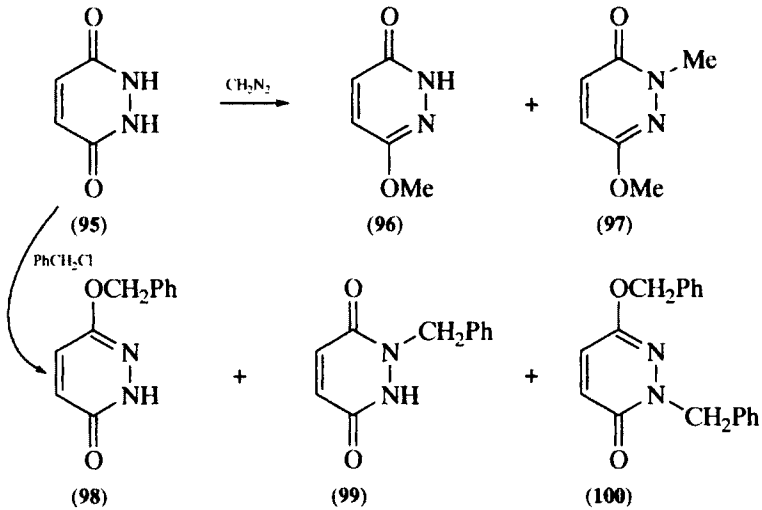


3,6-Dichloro-4(1*H*)-pyridazinone gave 3,6-dichloro-1-(2,3,5-tri-*O*-benzoyl-β-D-ribofuranosyl)-4(1*H*)-pyridazinone (**94**) [1-*O*-Acetyl-2,3,5-tri-*O*-benzoyl-β-D-ribofuranose, trace $ClCH_2CO_2H$, fusion; 170°, ?h: 52%;[1115] also analogues.[94]

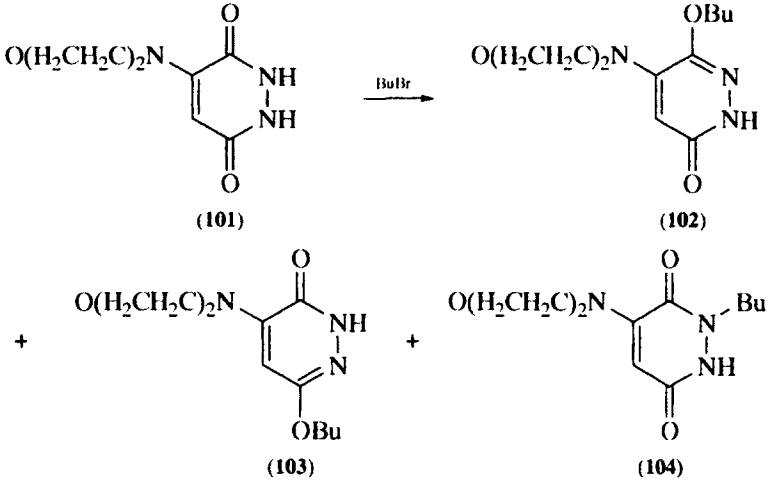
## From 3,6(1*H*,2*H*)-Pyridazinediones: *O*- and/or *N*-Alkylation

*Note*: It is clear from existing data that the alkylation of such diones can afford *O*-alkyl, *N*-alkyl, *O*,*N*-dialkyl, and *N*,*N*-dialkyl derivatives, in amounts depending not only on passenger groups but also on the reagents and conditions used. No definitive guidelines for the formation of specific types of derivative can be offered at this stage. The following examples represent some of the more useful data reported in recent years.

3,6-(1*H*,2*H*)-Pyridazinedione (**95**) gave a separable mixture of 6-methoxy-2-methyl-3(2*H*)-pyridazinone (**97**) [excess $CH_2N_2$, $Et_2O$, 25°, dark, 12h: 16% and 45%, respectively, after separation from a third (bicyclic) product];[332] a separable mixture of 6-benzyloxy-3(2*H*)-pyridazinone (**98**), 1-benzyl-3,6-(1*H*,2*H*)-pyridazinedione (**99**), and 2-benzyl-6-benzyloxy-3(2*H*)-pyridazinone (**100**) [$PhCH_2Cl$ (1 equiv), $Me_2NCHO$, 140°, 3h: 43%, 31%, 0%; $PhCH_2Cl$ (excess), $Me_2NCHO$, 140°, 8h: 26%, 42%, 10%; likewise plus $Et_3N$: 42%, 33%, 9%];[1498] or a variety of other such mixtures by using different alkylating agents.[674,718,876,1483,1753,1975]

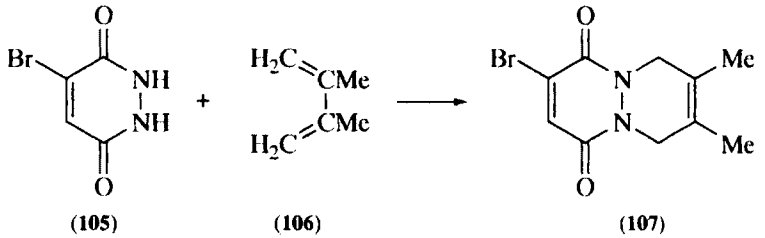



4-Morpholino-3,6(1*H*,2*H*)-pyridazinone (**101**) gave a separable mixture of 6-butoxy-5-morpholino-3(2*H*)-pyridazinone (**102**), 6-butoxy-4-morpholino-3(2*H*)-pyridazinone (**103**), and 1-butyl-5-morpholino-3,6-(1*H*,2*H*)-pyridazinedione (**104**) (substrate Na salt, BuBr, $Me_2NCHO$, 100°, 8h: ~40%, ~15%, ~3% respectively).[641]



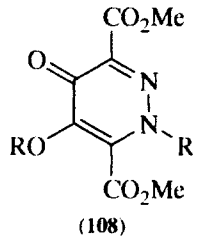

4-Bromo-3,6(1*H*,2*H*)-pyridazinedione (**105**) gave 2-bromo-7,8-dimethyl-1,4,6,9-tetrahydropyridazino[1,2-*a*]pyridazine-1,4-dione (**107**) [the diene (**106**); see original for details].[858]

Also other examples.[648,726,980,997,1194,1287]




### From 5-Hydroxy-4(1*H*)-Pyridazinones: *O*- and/or *N*-Alkylation

Dimethyl 4-hydroxy-5-oxo-2,5-dihydro-3,6-pyridazinedicarboxylate (**108**, R = H) gave dimethyl 4-methoxy-2-methyl-5-oxo-2,5-dihydro-3,6-pyridazinedicarboxylate (**108**, R = Me) ($CH_2N_2$, $Et_2O$, 25°, 2 days: ~ 15%).[406]




(**108**)

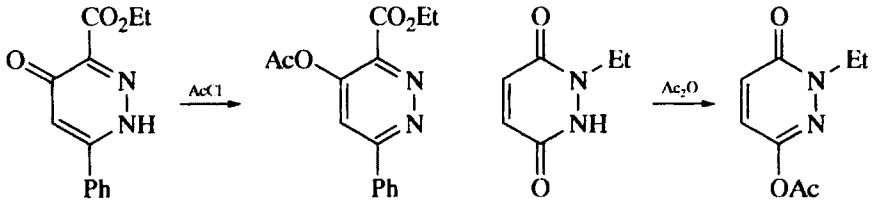
### *5.1.2.3 Conversion into O- or N-Acyl Derivatives (H 41)*

Treatment of a tautomeric pyridazinone with an appropriate acylating reagent derived from a carboxylic or phosphorus acid appears to give invariably an *O*-acyl derivative; however, the use of a reagent derived from a sulfur acid can give either an *O*- or an *N*-acyl derivative. A ring nitrogen that bears a proton by virtue of nuclear reduction will naturally undergo *N*-acylation by any of the foregoing types of reagent. All these reactions are illustrated in the following examples:

**Acylation with Alkylcarbonyl Chlorides or the Like**

Ethyl 4-oxo-6-phenyl-1,4-dihydro-3-pyridazinecarboxylate (**109**) gave ethyl 4-acetoxy-6-phenyl-3-pyridazinecarboxylate (**110**) (AcCl, $Et_3N$, PhMe, 25°, 14 h: 66%).[1969]

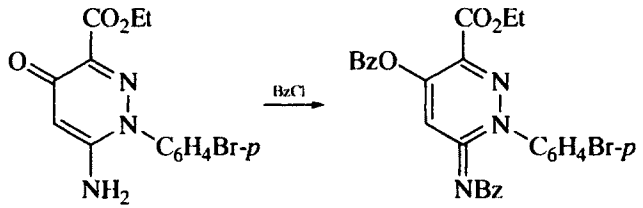
1-Ethyl-3,6(1*H*,2*H*)-pyridazinedione (**111**) gave 6-acetoxy-2-ethyl-3(2*H*)-pyridazinone (**112**) (neat $Ac_2O$, reflux, 1 h: 85%).[292]




(**109**) (**110**) (**111**) (**112**)

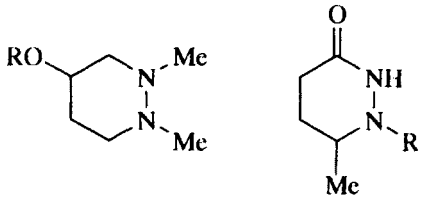
Ethyl 6-amino-1-*p*-bromophenyl-4-oxo-1,4-dihydro-3-pyridazinecarboxylate (**113**) gave ethyl 6-benzoylimino-4-benzoyloxy-1-*p*-bromophenyl-1,6-dihydro-3-pyridazinecarboxylate (**114**) (BzCl, pyridine, reflux, 40 min: 68%).[1304]

1,2-Dimethylhexahydro-4-pyridazinol (**115**, R = H) gave 1,2-dimethyl-4-*o*-tolylcarbamoyloxyhexahydropyridazine (**115**, R = $CONHC_6H_4Me$-*o*) *o*-$MeC_6H_4NCO$, PhMe, reflux, 19 h: 80%).[1671]




(**113**) (**114**)

6-Methyl-1,4,5,6-tetrahydro-3(2*H*)-pyridazinone (**116**, R = H) gave 1-iodoacetyl-6-methyl-1,4,5,6-tetrahydro-3(2*H*)-pyridazinone (**116**, R = $COCH_2I$) ($ICH_2CO_2H$, $C_6H_{11}N{=}C{=}NC_6H_{11}$, $CH_2Cl_2$–THF, 25°, 24 h: 86%).[350]



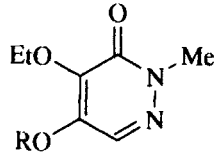
(115) (116)

Also other examples.[163,217,462,466,851,939,1016,1042,1090,1580,1598,2111]

### Acylation with Dialkyl Phosphorochloridates or the Like

4-Ethoxy-5-hydroxy-2-methyl-3(2*H*)-pyridazinone (**117**, R = H) gave 5-diethoxyphosphinyloxy-4-ethoxy-2-methyl-3(2*H*)-pyridazinone [**117**, R = P(:O)(OEt)$_2$] [substrate Na salt, ClP(=O)(OEt)$_2$, MeCN, 10° → 40°, 4 h: 91%], 5-diethoxyphosphinothioyloxy-4-ethoxy-2-methyl-3(2*H*)-pyridazinone [**117**, R = P(:S)(OEt)$_2$] [substrate Na salt, ClP(=S)(OEt)$_2$, MeCN, reflux, >2 h: 87%], or 4-ethoxy-5-ethoxy(ethyl)phosphinothioyloxy-2-methyl-3(2*H*)-pyridazinone [**117**, R = P(:S)EtOEt] [ClP(=S)EtOEt, likewise: 79%].[142]

Also many analogous examples.[141,142,144,145,646,788,895,897]

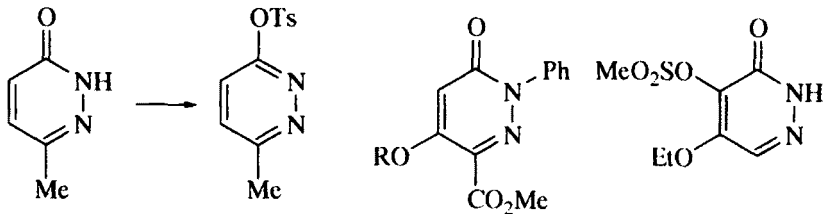


(117)

### Acylation with Arenesulfonyl Chlorides or the Like

6-Methyl-3(2*H*)-pyridazinone (**118**) gave 3-methyl-6-*p*-toluenesulfonyloxypyridazine (**119**) (TsCl, pyridine, 25°, 16 h: 79%).[437]

Methyl 4-hydroxy-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate (**120**, R = H) gave methyl 4-benzenesulfonyloxy-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate (**120**, R = $SO_2Ph$) ($PhSO_2Cl$, $Et_3N$, trace 4-$Me_2N$-pyridine, $Me_2NCHO$, 60°, 6 h: 62%).[1090]

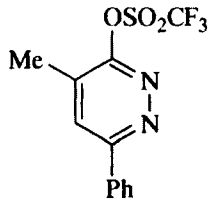


(118) (119) (120) (121)

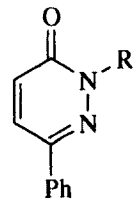
5-Ethoxy-3,4(1*H*,2*H*)-pyridazinedione gave 5-ethoxy-4-methanesulfonyloxy-3(2*H*)-pyridazinone (**121**) ($MeSO_2Cl$, pyridine, $CHCl_3$, reflux, 30 min: 77%) and then 5-ethoxy-3(2*H*)-pyridazinone ($H_2$, Pd/C, pyridine–MeOH, 66%; deacyloxylation!).[531]

4-Methyl-6-phenyl-3(2*H*)-pyridazinone gave 4-methyl-6-phenyl-3-trifluoromethanesulfonyloxypyridazine (**122**) [$(F_3CSO_2)_2O$, pyridine, A, 0° → 25°, 6 h: 72%; substrate Li salt, $(F_3CSO_2)_2$, THF, − 70°, > 10 min: ?%];[1629] also analogous examples.[1629,1907]

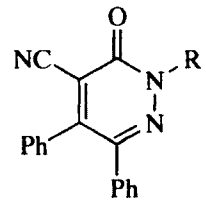
In contrast, 6-phenyl-3(2*H*)-pyridazinone (**123**, R = H) gave 6-phenyl-2-*p*-toluenesulfonyl-3(2*H*)-pyridazinone (**123**, R = Ts) (substrate Na salt, TsCl, NaH, THF, 0° → 25°, 90 min: 70%);[1063] 3-oxo-5,6-diphenyl-2,3-dihydro-4-pyridazinecarbonitrile (**124**, R = H) gave its 2-benzenesulfonyl derivative (**124**, R = $SO_2Ph$) ($PhSO_2Cl$, $K_2CO_3$, AcMe, reflux, 5 h: 60%);[1726] and other such *N*-acylations have been reported.[1398,1726]



(122)



(123)

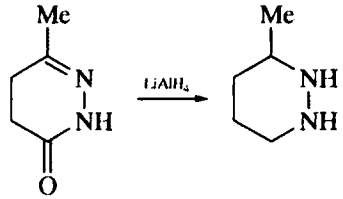


(124)

## *5.1.2.4 Reductive Reactions (H 49)*

Reduction of tautomeric pyridazinones usually results in nuclear reduction, sometimes followed by deoxygenation of ring fission. The following examples briefly illustrate such reactions:

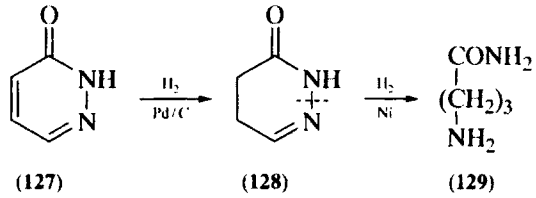
6-Methyl-4,5-dihydro-3(2*H*)-pyridazinone (**125**) gave 3-methylhexahydropyridazine (**126**) ($LiAlH_4$: > 30%).[720]



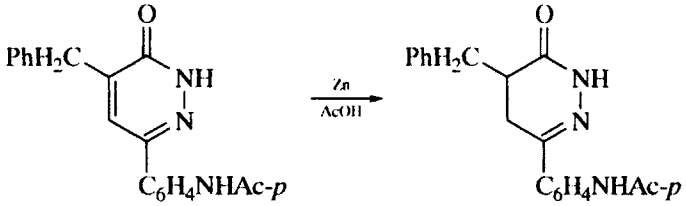
(125) (126)

3(2*H*)-Pyridazinone (**127**) gave 1,4,5,6-tetrahydro-3(2*H*)-pyridazinone (**128**) [$H_2$ (3 atm), $H_2O$, Pd/C, 25°, 42 h: 39%] and thence 4-aminobutyramide (**129**) [$H_2$ (3 atm), Raney Ni, EtOH, 25°, 24 h: 14%, as hydrochloride].[155]

6-*p*-Acetamidophenyl-4-benzyl-3(2*H*)-pyridazinone (**130**) gave its 4,5-dihydro derivative (**131**) (Zn dust, AcOH, reflux, 3 h: >75%).[493]



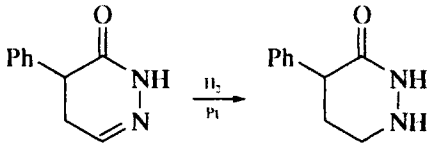
(**127**) (**128**) (**129**)



(**130**) (**131**)

4-Phenyl-4,5-dihydro-3(2*H*)-pyridazinone (**132**) gave 4-phenyl-1,4,5,6-tetrahydro-3(2*H*)-pyridazinone (**133**) ($H_2$, $PtO_2$, EtOH–AcOH, 6 h: ~85%).[738]

Also other examples,[313,496,543,1395] including the polarographic reduction of 3,6(1*H*,3*H*)-pyridazinedione.[2047]

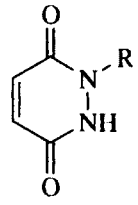


(**132**) (**133**)

## *5.1.2.5 Miscellaneous Reactions (H 51)*

Tautomeric pyridazinones also undergo a variety of other reactions, most of which are inadequately exemplified in recent literature but are of potentual use. These are discussed briefly in the following paragraphs.

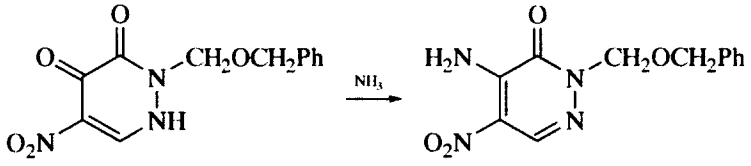
*N*-AMINATION Treatment of 3,6(1*H*,2*H*)-pyridazinedione (**134**, R = H) with hydroxylamine-*O*-sulfonic acid gave 37% of 2-amino-3(2*H*)-pyridazinone (**134**,



(**134**)

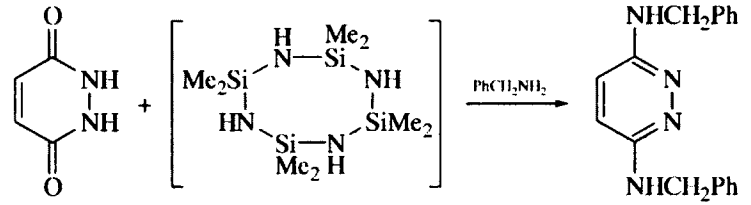
R = $NH_2$) or, when benzaldehyde was included in the reaction mixture, 2-benzylideneamino-3(2*H*)-pyridazinone (**134**, R = N:CHPh).[903]

AMINOLYSIS The direct aminolysis of the oxo substituent of a tautomeric pyridazinone is unusual, but 2-benzyloxymethyl-5-nitro-3,4(1*H*,2*H*)-pyridazinedione (**135**) has been converted into 4-amino-2-benzyloxymethyl-5-nitro-3(2*H*)-pyridazinone (**136**) (80%) by simply heating with methanolic ammonia at 130° in an autoclave for 22 h.[1048] An indirect silicon-assisted aminolysis



(135) (136)

method has been described: 3,6(1*H*,2*H*)-pyridazinedione (**137**) with benzylamine (2 equiv), octamethylcyclotetrasilazane (**138**) (1 equiv), and a little *p*-toluenesulfonic acid at 200° for 5 h gave 62% of 3,6-bisbenzylaminopyridazine (**139**), probably via an unformulated *O*,*O*′-disilylated intermediate.[16] The formation of an oxime from a pyridazinone is, of course, an aminolysis; for

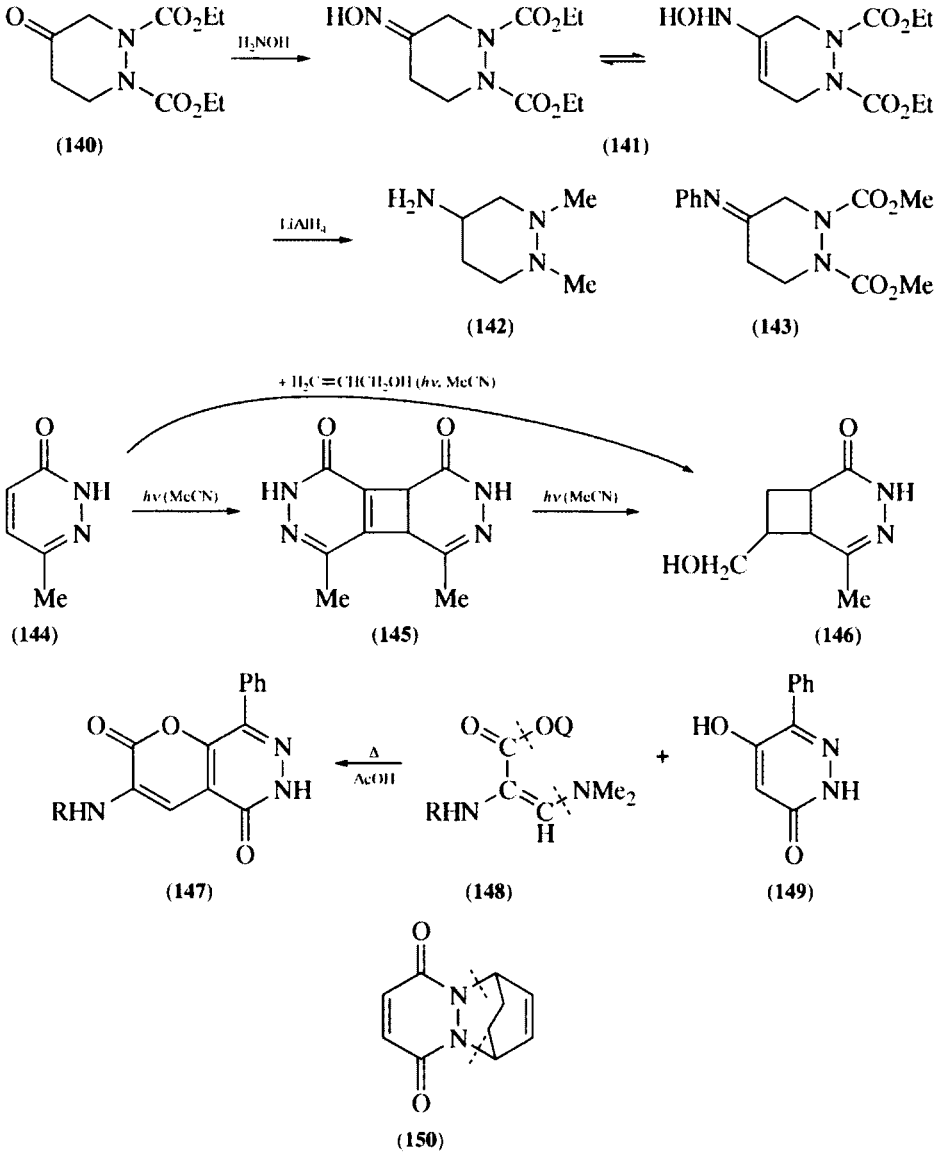


(137) (138) (139)

example, diethyl 4-oxohexahydro-1,2-pyridazinedicarboxylate (**140**) afforded its oxime (**141**) (89%, by heating in ethanolic hydroxylamine containing pyridine for 3 h) and then 1,2-dimethylhexahydro-4-pyridazinamine (**142**) in 60% yield by refluxing with lithium aluminum hydride in ether for 28 h.[1699] Other minor aminolytic processes gave, for example, the Schiff base dimethyl 4-phenyliminohexahydro-1,2-pyridazinedicarboxylate (**143**).[774,1456]

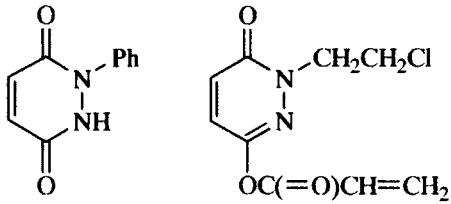
CYCLOADDITION REACTIONS Irradiation of 6-methyl-3(2*H*)-pyridazinone (**144**) gave initially the *cis–syn* "cyclobutane dimer" (**145**) and then 7-hydroxymethyl-5-methyl-3,4-diazabicyclo[4.2.0]oct-4-en-2-one (**146**), which was also formed by irradiation of the same substrate (**144**) with allyl alcohol;[166] analogous examples have been reported.[166,233,786,1988] 5-Hydroxy-6-phenyl-3(2*H*)-pyridazinone (**149**) underwent a cycloaddition by methyl 2-acetamido-3-dimethylaminoacrylate (**148**, Q = Me, R = Ac) in refluxing acetic acid during 1 h to afford 3-acetamido-8-phenyl-2*H*-pyrano[2,3-*d*]pyridazine-2,5(6*H*)-dione (**147**) in 34% yield;[1951] the same substrate (149) with ethyl 2-*β*,*β*-

di(ethoxycarbonyl)vinylamino-3-dimethylaminoacrylate [**148**, Q = Et, R = $CH:C(CO_2Et)_2$] in refluxing acetic acid during 25 h gave the analogous 3-$\beta,\beta$-diethoxycarbonylvinylamino-8-phenyl-2*H*-pyrano[2,3-*d*]pyridazine-2,5(6*H*)-dione [**147**, $R = CH:C(CO_2Et)_2$].[1953] 3,6(1*H*,2*H*)-Pyridazinedione suffered oxidative cycloaddition by 1,3-cyclohexadiene in the presence of lead tetraacetate, in dichloromethane at 25° during 24 h, to furnish 1,4,6,9-tetrahydro-1,4-ethanopyridazino[1,2-*a*]pyridazine-6,9-dione (**150**) in 48% yield;[615] and other such adducts have been reported.[667]



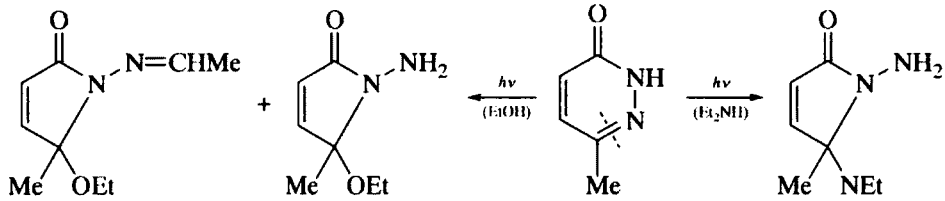

(140) (141) (142) (143) (144) (145) (146) (147) (148) (149) (150)

POLYMERIZATION The copolymerization of 1-phenyl-3,6-(1*H*,2*H*)-pyridazinedione (**151**) or a related substrate with styrene or acrylonitrile has been studied.[1084] The homopolymerization of 6-acryloyloxy-2-$\beta$-chloroethyl-3(2*H*)-pyridazinone (**152**) and its copolymerization with styrene or methyl methacrylate have also been investigated.[1112]



(**151**) (**152**)

RING CONTRACTION TO *N*-AMINOPYRROLES Irradiation of 6-methyl-3(2*H*)-pyridazinone (**155**) in ethanol gave a separable mixture of 1-amino-5-ethoxy-5-methyl-3-pyrrolin-2-one (**154**) (28% yield) and 5-ethoxy-1-ethylideneamino-5-methyl-3-pyrrolin-2-one (**153**) (5% yield), but in diethylamine-dichloromethane only 1-amino-5-diethylamino-5-methyl-3-pyrrolin-2-one (**156**) was isolated in 7% yield;[785] homologous substrates gave similar results.[785] Other examples have been reported.[920]



(**153**) (**154**) (**155**) (**156**)

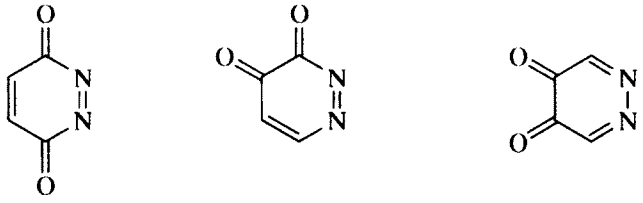
OXIDATIVE REACTIONS The chemiluminescent oxidation of 3,6(1*H*,2*H*)-pyridazinedione and several derivatives (by molecular oxygen in dimethyl sulfoxide containing potassium *t*-butoxide) have been shown to afford fumaric acid or an appropriate derivative.[200] The radicals formed by one-electron oxidation of 3,6(1*H*,2*H*)-pyridazinedione have been studied by esr spectra.[265]

## 5.2 PYRIDAZINE QUINONES

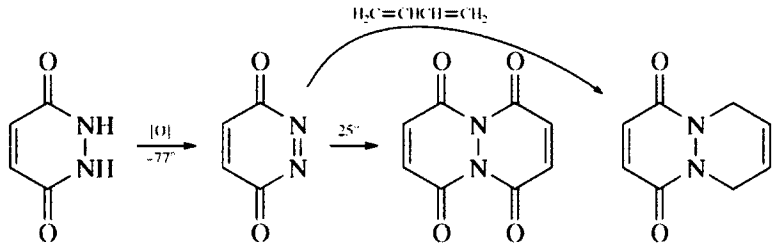
Of the three pyridazine quinone systems (**157–159**) that can be envisaged, only the *para* system (**157**) has been investigated to any extent; the *ortho* systems (**158**) and (**159**) appear to be each represented at present by a single derivative.

Following seminal work in the phthalazine series,[1989] the parent pyridazine *para*-quinone, 3,6-dihydro-3,6-pyridazinedione (**161**), was generated in solution by oxidation of the monopotassium salt of 3,6(1*H*,2*H*)-pyridazinedione (**160**)

with *t*-butyl hypochlorite in acetone at − 77° during 3 h: the product could not be isolated, but it was characterized as either 1,4,6,9-tetrahydropyridazino[1,2-*a*]pyridazine-1,4,6,9-tetrone (**162**) (by loss of nitrogen on warming the solution to 25°) or the regular Diels–Alder adduct with butadiene, 1,4,6,9-tetrahydropyridazino[1,2-*a*]pyridazine-1,4-dione (**163**).[1990] This pioneering work and subsequent contributions up to 1977 have been reviewed in detail.[192,cf.1991]

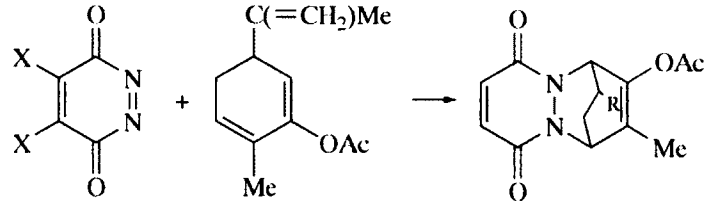


(157) (158) (159)



(160) (161) (162) (163)

Progress in pyridazine quinones since 1977 has been far from spectacular. However, the parent *para*-quinone, 3,6-dihydro-3,6-pyridazinedione (**164**, X = H), has been shown to undergo cycloaddition with 2-acetoxy-6-isopropenyl-3-methylcyclohexa-1,3-diene (**165**) to give a 16% yield of 2-acetoxy-12-isopropenyl-3-methyl-1,4,6,9-tetrahydro-1,4-ethanopyridazino[1,2-*a*]pyridazine-6,9-dione [**166**, R = C(:$CH_2$)Me][235] or with dibenzo[*b*,*n*]perylene to give, not

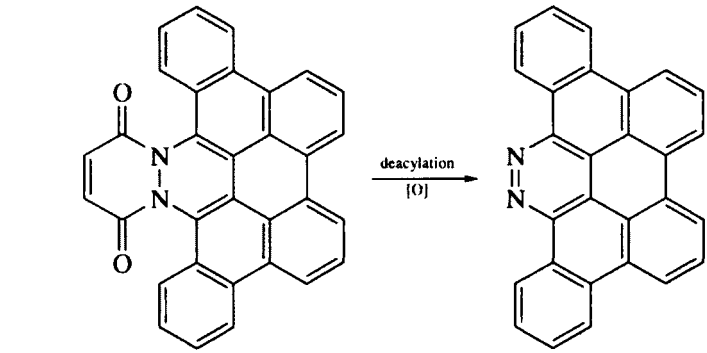


(164) (165) (166)

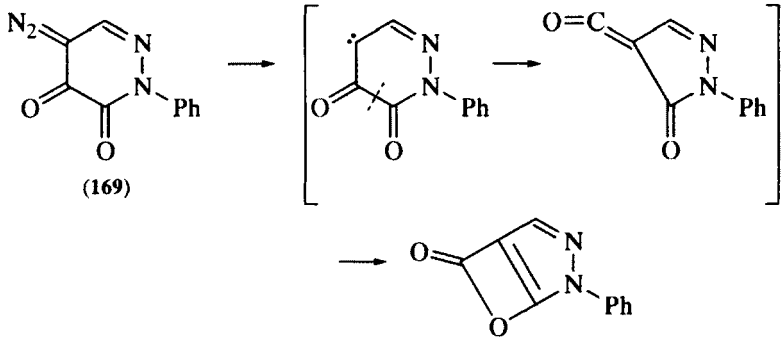
the expected adduct (**167**), but its deacylated and dehydrogenated derivative, diphenanthro[9,10,1-*d*,*e*,*f*:1′,10′,9′-*h*,*i*,*j*]phthalazine (**168**), in 36% yield;[120] as might be expected, 4,5-dichloro-3,6-dihydro-3,6-pyridazinedione (**164**, X = Cl) gave the same product (**168**) (in 46% yield).[120]

5-Diazo-2-phenyl-4,5-dihydro-3,4(2*H*)-pyridazinedione (**169**) arguably qualifies as one type of *ortho*-quinone in the pyridazine series. It is made easily be

diazotization of 5-amino-2-phenyl-3,4(1*H*,2*H*)-pyridazinedione under anhydrous conditions[1992] and has been shown more recently to undergo an interesting quantitative transformation in the solid state (during 10 days at 25° in the dark) to afford 2-phenyl-4,3-pyrazolecarbolactone (**170**),[194] from which other pyrazoles could be derived.[194]



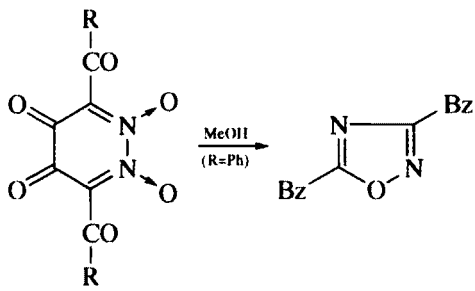

(**167**) (**168**)



(**169**)

(**170**)

The second type of *ortho*-quinone in the pyridazine series has long been represented by a compound of putative structure, 3,6-dibenzoyl-4,5-dihydro-4,5-pyridazinedione 1,2-dioxide (**171**, R = Ph), originally made[1993] by primary




(**171**) (**172**)

synthesis (see Section 1.2.2.1.4). More recently, the original synthesis was repeated and extended to make several analogues, for example, 3,6-dithen-2′-oyl-4,5-dihydro-4,5-pyridazinedione 1,2-dioxide (**171**, R = 2-thienyl),[506] and the structure (**171**, R = Ph) was confirmed by $^{13}C$ nmr spectra;[250] moreover, the unlikely degradation product, formed in 40% yield by treatment of (**171**, R = Ph) with methanol at 25° for 24 h,[1993] has been confirmed as 3,5-dibenzoyl-1,2,4-oxadiazole (**172**).[250]

## 5.3 EXTRANUCLEAR HYDROXYPYRIDAZINES (*H* 353)

These important hydroxyalkyl- and hydroxyarylpyridazines must be viewed as regular alcohols or phenols; indeed, their methods of preparation, properties, and reactions are usually little affected by the presence of the pyridazine ring.

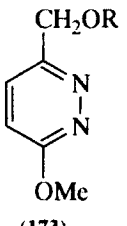
### 5.3.1 Preparation of Extranuclear Hydroxypyridazines (*H* 369, 389)

Many such hydroxypyridazines have been made by a variety of *primary syntheses* (see Chapters 1 and 2), some by *C-hydroxyalkylation* procedures (see Sections 3.1.3, 3.2.1.1, and 3.2.2.2), and a few by direct or indirect *hydrolysis of extranuclear halogenopyridazines* (see Section 4.4.6). The remaining preparative routes are illustrated in the following examples:

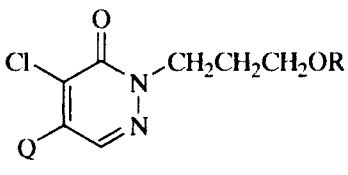
#### By Deacylation of Extranuclear Acyloxypyridazines

3-Acetoxymethyl-6-methoxypyridazine (**173**, R = Ac) gave 3-hydroxymethyl-6-methoxypyridazine (**173**, R = H) (dilute HCl, reflux, ?min: 87%).[913]

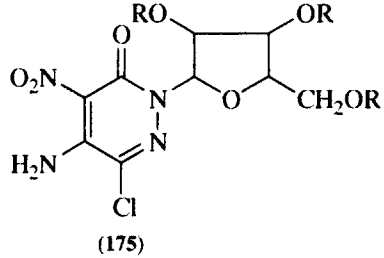
2-δ-Benzoyloxybutyl-4,5-dichloro-3(2*H*)-pyridazinone (**174**, Q = Cl, R = Bz) gave 4,5-dichloro-2-δ-hydroxybutyl-3(2*H*)-pyridazinone (**174**, Q = Cl, R = H) [6-hydroxy-2(1*H*)-pyridinone, $Me_2NCHO$, reflux, 4 h: 46%; a transacylation process, presumably used to avoid affecting the chloro substituents].[1700]



(173)



(174)



(175)

5-Amino-2-δ-benzoyloxybutyl-4-chloro-3(2*H*)-pyridazinone (**174**, Q = $NH_2$, R = Bz) gave 5-amino-4-chloro-2-δ-hydroxybutyl-3(2*H*)-pyridazinone

(**174**, Q = $NH_2$, R = H) [MeONa, MeOH, 25°, 24 h; then Amberlite ($H^+$ form of TRC-50)↓, 25°, 12 h: 95%].[1700]

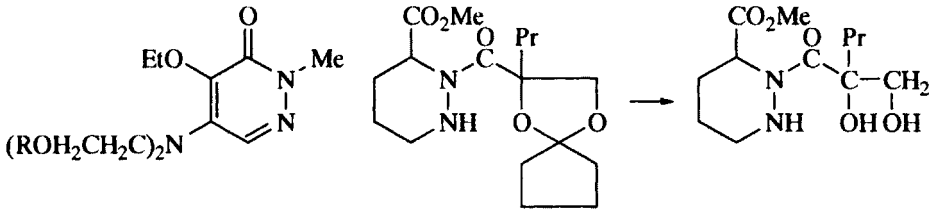
5-Amino-6-chloro-4-nitro-2-(2,3,5-tri-*O*-benzoyl-β-D-ribofuranosyl)-3(2*H*)-pyridazinone (**175**, R = Bz) gave 5-amino-6-chloro-4-nitro-2-(β-D-ribofuranosyl)-3(2*H*)-pyridazinone (**175**, R = H) [MeONa, MeOH, reflux, 3 h: then Dowex ($H^+$ form of 50W-X8)↓, to pH 7: 65%; structure confirmed by X-ray analysis].[1004]

Also other examples.[778,994,1043,1894]

## From Extranuclear Alkoxypyridazines

5-Bis(β-benzyloxyethyl)amino-4-ethoxy-2-methyl-3(2*H*)-pyridazinone (**176**, R = $CH_2Ph$) gave 5-bis(β-hydroxyethyl)amino-4-ethoxy-2-methyl-3(2*H*)-pyridazinone (**176**, R = H) ($H_2$, Pd/C, EtOH: 82%; a useful procedure when hydrolysis would affect another group in the molecule).[532]

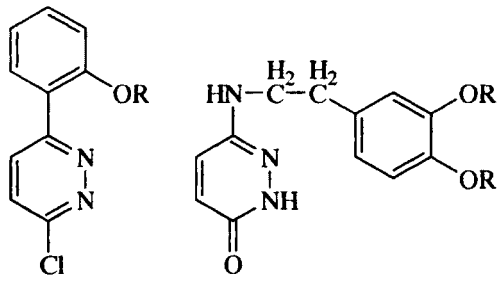
Methyl 2-(2,3-cyclopentylidenedioxy-2-propylpropionyl)hexahydro-3-pyridazinecarboxylate (**177**) gave methyl 2-(2-hydroxy-2-hydroxymethylvaleryl)-hexahydro-3-pyridazinecarboxylate (**178**) ($HClO_4$, THF–$H_2O$, 23°, 12 h: 81%).[1810]



(**176**) (**177**) (**178**)

3-Chloro-6-*o*-methoxybenzylpyridazine (**179**, R = Me) gave 3-chloro-6-*o*-hydroxybenzylpyridazine (**179**, R = H) ($BBr_3$, $CH_2Cl_2$, −50°→25°, 30 min: 80%; note survival of the Cl substituent using this method).[750,1197]

6-(3,4-Dimethoxyphenethylamino)-3(2*H*)-pyridazinone (**180**, R = Me) gave 6-(3,4-dihydroxyphenethylamino)-3(2*H*)-pyridazinone (**180**, R = H) (48% HBr, reflux, 2 h: 50%).[1733]

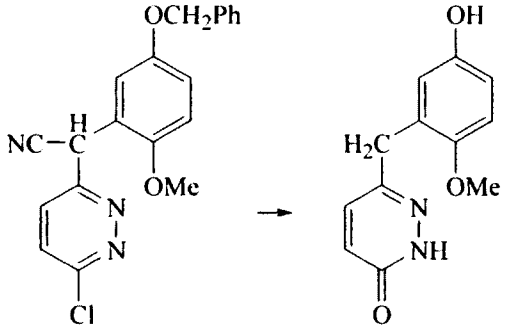


(**179**) (**180**)

6-*p*-Methoxyphenyl-3(2*H*)-pyridazinone gave 6-*p*-hydroxyphenyl-3(2*H*)-pyridazinone (10M HCl, 130°, sealed, 5 h: 64%).[919]

3-(3-Benzyloxy-α-cyano-6-methoxybenzyl)-6-chloropyridazine (**181**) gave 6-(3-hydroxy-6-methoxybenzyl)-3(2*H*)-pyridazinone (**182**) (HCl–AcOH, reflux, 20 min; then residue from evaporation, 6M HCl, reflux, 6 h: 82%; involved hydrolysis of Cl, hydrolysis of CN with subsequent decarboxylation, and selective hydrolysis of the $OCH_2Ph$).[749,cf.1698]

Also other examples.[1090,1585,1605]

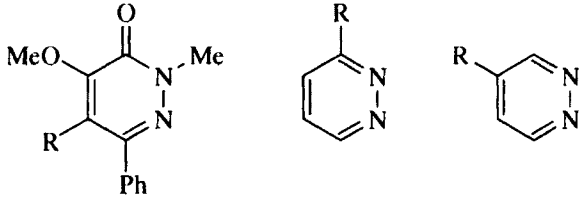



(**181**) (**182**)

## From Pyridazinecarbaldehydes or Ketones

5-Acetyl- (**183**, R = Ac) gave 5-α-hydroxyethyl-4-methoxy-6-phenyl-3(2*H*)-pyridazinone [**183**, R = CH(OH)Me] ($NaBH_4$, MeOH, 25°, 75 min: 72%).[1832]

3-Benzoyl- (**184**, R = Bz) gave 3-α-hydroxybenzylpyridazine [**184**, R = CH(OH)Ph] ($NaBH_4$, MeOH, 25°, 75 min: 95%);[1108] likewise 3-α-hydroxybenzyl-6-methoxypyridazine (96%).[1698]




(**183**) (**184**) (**185**)

4-Pyridazinecarbaldehyde (**185**, R = CHO) gave 4-hydroxymethylpyridazine (**185**, R = $CH_2OH$) ($NaBH_4$, $H_2O$, 25°, 30 min: 94%).[66]

4-Benzoyl- (**185**, R = Bz) gave 4-α-hydroxybenzylpyridazine [**185**, R = CH(OH)Ph] ($NaBH_4$, MeOH, 25°, until complete: 97%);[72,cf.1098] analogues likewise.[66,218,1060,1732]

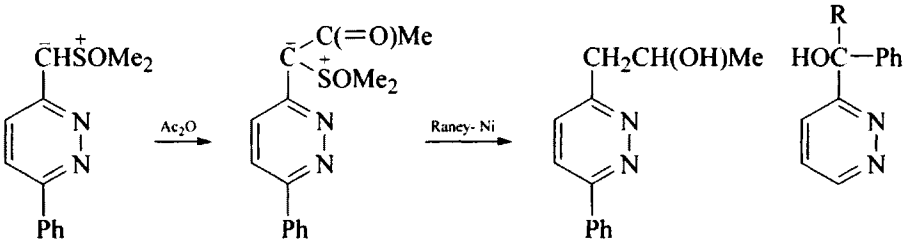
3-Pyridazinecarbaldehyde (**184**, R = CHO) gave a separable mixture of 3-hydroxymethylpyridazine (**184**, R = $CH_2OH$) and 3-pyridazinecarboxylic

acid (**184**, R = $CO_2H$) by a Cannizzaro reaction (0.5M KOH, reflux, 5 h: 78% and 19%, respectively, after purification).[77]

Dimethyloxosulfonium 6-phenylpyridazine-3-ylmethylide (**186**) (for preparation see Section 3.2.1.3) underwent *C*-acetylation to give dimethyloxosulfonium *C*-acetyl-*C*-(6-phenylpyridazin-3-yl)methylide (**187**) ($Ac_2O$, dioxane, 0° → 25°, 90 min: 98%) and then 3-β-hydroxypropyl-6-phenylpyridazine (**188**) (Raney Ni, MeOH, reflux, 2 h: 33%).[836]

3-Benzoylpyridazine (**184**, R = Bz) gave 3-(α-hydroxy-α-methylbenzyl)pyridazine (**189**, R = Me) (MeMgCl, THF, − 5° → 25°, 45 min: 71%) or 3-α-hydroxydiphenylmethylpyridazine (**189**, R = Ph) (PhMgCl, THF–$Et_2O$, − 5° → 25°, 3.5 h: 76%).[1357]

Also other examples.[373,702,1060,1680,2039]

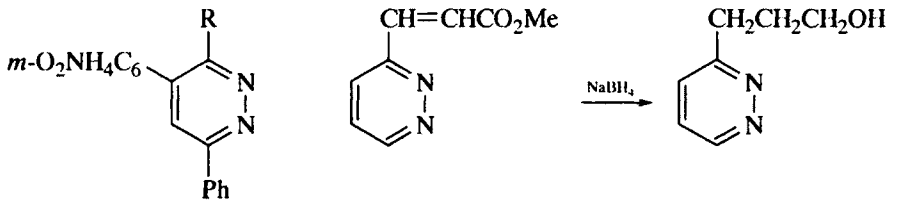



(186) (187) (188) (189)

## From Pyridazinecarboxylic Esters

Ethyl 4-*m*-nitrophenyl-6-phenyl-3-pyridazinecarboxylate (**190**, R = $CO_2Et$) gave 3-hydroxymethyl-4-*m*-nitrophenyl-6-phenylpyridazine (**190**, R = $CH_2OH$) ($NaBH_4$, EtOH–THF, 25°, 3 h: 22%; note survival of the nitro group).[1458]

3-β-Methoxycarbonylvinylpyridazine (**191**) gave 3-γ-hydroxypropylpyridazine (**192**) ($NaBH_4$, MeOH, 25° → reflux, 2 h: 81%; note reduction of the side-chain double bond).[1778]

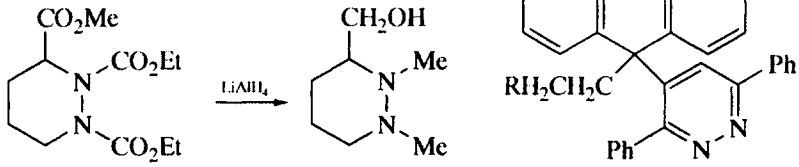



(190) (191) (192)

Ethyl 4-pyridazinecarboxylate (**185**, R = $CO_2Et$) gave a separable mixture of 4-hydroxymethylpyridazine (**185**, R = $CH_2OH$) and ethyl 2,5-dihydro-4-pyridazinecarboxylate ($LiAlH_4$, $Et_2O$, 1 h: 67% and 16%, respectively).[66]

Diethyl 3-methoxycarbonylhexahydro-1,2-pyridazinedicarboxylate (**193**) gave 3-hydroxymethyl-1,2-dimethylhexahydropyridazine (**194**) ($LiAlH_4$, $Et_2O$, 25°, 18 h; then reflux 24 h: 80%; note the interesting differential reduction of *C*- and *N*-ester groupings).[1342]

5-*β*-Butoxycarbonylethyl-5-(3,6-diphenylpyridzin-4-yl)- (**195**, R = $CO_2Bu$) gave 5-(3,6-diphenylpyridazin-4-yl)-5-*γ*-hydroxypropyl-5*H*-indeno[1,2-*b*]pyridine (**195**, R = $CH_2OH$) ($LiAlH_4$, THF, reflux, 3 h: 61%).[1491]




(193) (194) (195)

### By Direct Hydroxyalkylation of Like Processes

*Note*: Extranuclear hydroxy groups may be introduced as passenger group during hydroxyalkylation or other such processes described elsewhere in appropriate sections. Examples are *C*-hydroxyalkylation (Section 3.2.1.1), *O*- or *N*-hydroxyalkylation (Section 5.1.2.2), or hydroxyalkylaminolysis (Section 4.2.1.5) and so on.

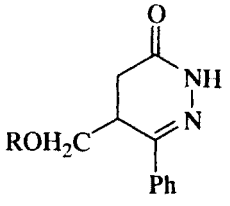
## 5.3.2 Reactions of Extranuclear Hydroxypyridazines (*H* 382, 392)

Extranuclear hydroxypyridazines undergo the reactions that might be expected of alcohols or phenols. Of these, the conversion of *hydroxyalkyl- into alkenylpyridazines* by elimination of water (Section 3.2.1.4) and the conversion of *hydroxyalkyl- into halogen alkylpyridazines* (Section 4.3.1) have been discussed already. The remaining reactions are illustrate briefly in the following examples.

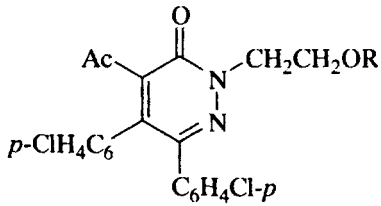
### *O*-Acylation

5-Hydroxymethyl- (**196**, R = H) gave 5-acetoxymethyl-6-phenyl-4,5-dihydro-3(2*H*)-pyridazinone (**196**, R = Ac) ($Ac_2O$, pyridine, 25°, 12 h: >95%).[1588]

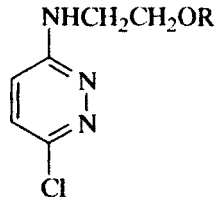
4-Acetyl-5,6-bis-*p*-chlorophenyl-2-*β*-hydroxyethyl-3(2*H*)-pyridazinone (**197**, R = H) gave 2-*β*-acetoxyethyl-4-acetyl-5,6-bis-*p*-chlorophenyl-3(2*H*)-pyridazinone (**197**, R = Ac) (AcCl, $Et_3N$, THF, 20°, 48 h: 63%)[1273] or 4-acetyl-5,6-bis-*p*-chlorophenyl-2-*β*-formyloxyethyl-3(2*H*)-pyridazinone (**197**, R = CHO) (88% $HCO_2H$, 55°, 3 h: 38%).[1332]



(196)



(197)



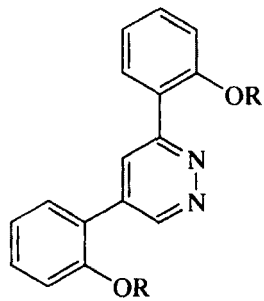
(198)

3-Chloro-6-β-hydroxyethylaminopyridazine (**198**, R = H) gave 3-β-acetoxyethylamino-6-chloropyridazine (**198**, R = Ac) (neat $Ac_2O$, 40° → 25°, 12 h: ~85%).[1169]

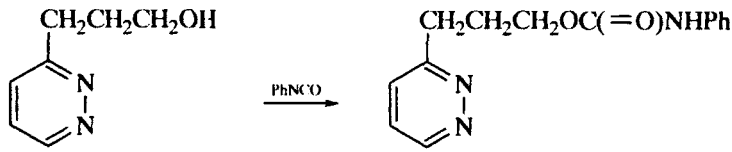
3,5-Bis-*o*-hydroxyphenylpyridazine (**199**, R = H) gave 3,5-bis-*o*-acetoxyphenylpyridazine (**199**, R = Ac) ($Ac_2O$, pyridine, 25°, 12 h: 81%).[356]

3-γ-Hydroxypropylpyridazine (**200**) gave 3-γ-(phenylcarbamoyloxy)propylpyridazine (**201**) (PhNCO, MeCN, 70°, 70 min; then 25°, 12 h: 69%);[1778] also analogous reactions.[1788]

Also other examples.[47,159,335,1280,1300,1342,1480,1733,1901,2018,2029,2045]



(199)



(200) (201)

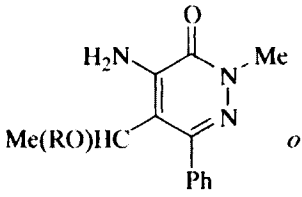
## *O*-Alkylation

4-Amino-5-α-hydroxyethyl- (**202**, R = H) gave 4-amino-5-α-ethoxyethyl-2-methyl-6-phenyl-3(2*H*)-pyridazinone (**202**, R = Et) (EtOH, polyphosphoric acid, 100°, sealed?, 15 min: 60%).[1832]

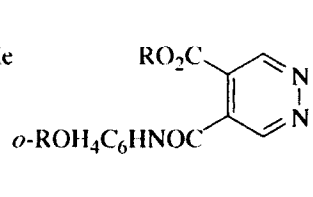
5-*o*-Hydroxyphenylcarbamoyl-4-pyridazinecarboxylic acid (**203**, R = H) gave methyl 5-*o*-methoxyphenylcarbamoyl-4-pyridazinecarboxylate (**203**, R = Me) (excess $CH_2N_2$, $Et_2O$–MeOH, 25°, 132 h: ~60%; note the additional esterification).[393]

4-(5-Hydroxy-3-methyl-1-phenylpyrazol-4-yl)- (**204**, R = H) gave 4-(5-methoxy-3-methyl-1-phenylpyrazol-4-yl)-6-methyl-2-phenyl-3(2*H*)-pyridazinone (**204**, R = Me) ($CH_2N_2$, $Et_2O$, 25°, 1 week: 51%).[378]

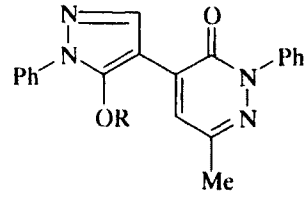
Also other examples.[356,582]



(**202**)



(**203**)



(**204**)

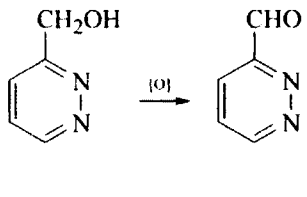
## Oxidation

*Note*: Primary pyridazinyl alcohols are oxidized to aldehydes, secondary alcohols to ketones, but both types may afford carboxylic acids under sufficiently severe conditions.

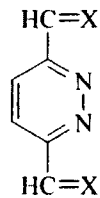
3-Hydroxymethylpyridazine (**205**) gave 3-pyridazinecarbaldehyde (**206**) (activated $MnO_2$, $CHCl_3$, 25°, 15 h: 67%;[77] $SeO_2$, dioxane, reflux, 2 h: 52%);[439] also analogues.[913,2018]

3,6-Bishydroxymethyl-pyridazine gave 3,6-pyridazinedicarbaldehyde (**207**, X = O) [MnO2 or a Swern[1852b] oxidation: 48% or 90%; isolated as the dioxime (**207**, X = NoH)].[1545,1894,1939]

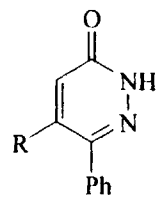
5-Hydroxymethyl-6-phenyl-3(2*H*)-pyridazinone (**208**, R = $CH_2OH$) gave 6-oxo-3-phenyl-1,6-dihydro-4-pyridazinecarbaldehyde (**208**, R = CHO) [$Pb(OAc)_4$, pyridine, 25°, 5 h: 65%].[370]



(**205**) (**206**)



(**207**)



(**208**)

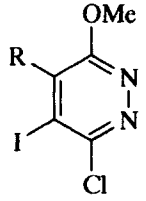
3-Chloro-5-α-hydroxyethyl-4-iodo-6-methoxypyridazine [**209**, R = CH(OH)Me] gave 4-acetyl-6-chloro-5-iodo-3-methoxypyridazine [**209**, R = Ac] (pyridinium chlorochromate, 3 Å molecular sieve, $CH_2Cl_2$, 25°, 30 h: 95%; or $MnO_2$, PhMe, reflux, water removal, 3 h: 77%).[1610]

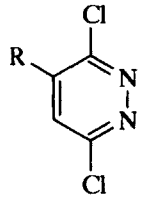
3,6-Dichloro-4-α-hydroxybenzylpyridazine [**210**, R = CH(OH)Ph] gave 4-benzoyl-3,6-dichloropyridazine (**210**, R = Bz) ($MnO_2$, PhMe, reflux, water removal, 3 h: 68%).[1669]

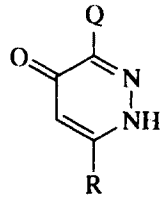
3-α-Hydroxybenzylpyridazine underwent thermal disproportionation to 3-benzoyl- and 3-benzylpyridazine (neat, 160°, vacuum, 6 h: 44% and 37%, respectively).[1357]

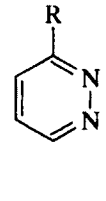
6-Hydroxymethyl-5-oxo-2,5-dihydro-3-pyridazinecarboxylic acid (**211**, $Q = CH_2OH$, $R = CO_2H$) or its isomer, 6-hydroxymethyl-4-oxo-1,4-dihydro-3-pyridazinecarboxylic acid (**211**, $Q = CO_2H$, $R = CH_2OH$), gave 4-oxo-1,4-dihydro-3,6-pyridazinedicarboxylic acid (**211**, $Q = R = CO_2H$) ($KMnO_4$, $H_2O$, 75°: ~75%).[778]

3-$\alpha,\beta$-Dihydroxyethylpyridazine [**212**, $R = CH(OH)CH_2OH$] gave 3-pyridazinecarboxylic acid (**212**, $R = CO_2H$) ($KMnO_4$, $H_2O$, 75°, 15 min: 83%).[925,935]


(209)


(210)

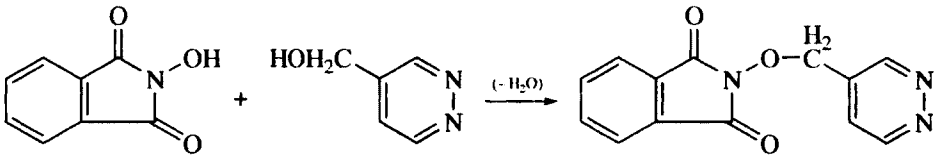

(211)


(212)

**Miscellaneous Reactions**

3-Hydroxymethylpyridazine (**214**) and *N*-hydroxyphthalimide (**213**) gave 3-phthalimidooxymethylpyridazine (**215**) [$EtO_2CN{=}NCO_2Et$, $Ph_3P$, THF, 25°, 2 h: 65%; akin to a mixed ether formation].[1230]

4-$\alpha$-hydroxy- gave 4-$\alpha$-mercaptoduphenylmethyl-5,6-diphenyl-3-(2*H*)-pyridazinethione ($P_2S_5$, xylene, reflux, 3 h: 62%).[373]


(213) (214) (215)

## 5.4 NUCLEAR AND EXTRANUCLEAR ALKOXY- OR ARYLOXYPYRIDAZINES (*H* 52)

Both types of pyridazine ether are made easily, but the nuclear ethers are good substrates for nucleophilic displacement reactions. The present discussion includes a few epoxides which are also quite reactive.

### 5.4.1 Preparation of Alkoxy- and Aryloxypyridazines (*H* 52)

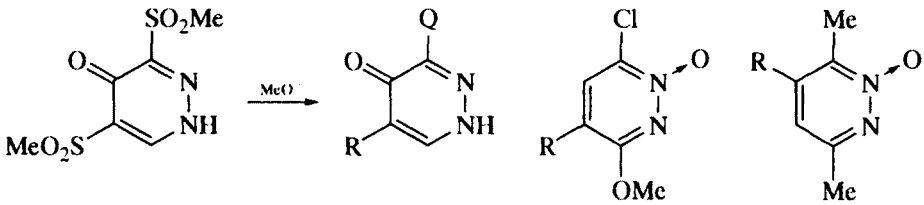
Most such ethers are made by *primary synthesis* (see Chapters 1 and 2), by *alcoholysis or phenolysis of halogenopyridazines* (see Sections, 4.2.3 and 4.4.2), or by *O-alkylation of tautomeric pyridazinones or extranuclear hydroxypyr-*

*idazines* (see Sections 5.1.2.2 and 5.3.2). However, an appreciable number of these ethers have been made by the alcoholysis of other (minor) substrates and epoxides by oxidative or other processes; all such reactions are illustrated by the following examples.

### From Alkylthio- or Alkylsulfonylpyridazines

3-Acetonylphio- gave 3-ethoxy-6-phenylpyridazine (EtONa, $Me_2NCHO$, 50°, 1 h: >95%).[413]

3,5-Bismethylsulfonyl-4(1*H*)-pyridazinone (**216**) gave either 3/5-methoxy-5/3-methylsulfonyl-4(1*H*)-pyridazinone (**217**, Q = OMe, R = $SO_2Me$; or Q = $SO_2Me$, R = OMe) (MeONa, MeOH, reflux, 4 h: ~60%) or 3,5-dimethoxy-4(1*H*)-pyridazinone (**217**, Q = R = OMe) (MeONa, MeOH, 155°, sealed, 3 h: ~30%).[689]




(216) (217) (218) (219)

### From Nitropyridazines

3-Chloro-6-methoxy-5-nitropyridazine 2-oxide (**218**, R = $NO_2$) gave 3-chloro-5,6-dimethoxypyridazine 2-oxide (**218**, R = OMe) (MeONa, MeOH, 25°, 3 h: 93%).[905]

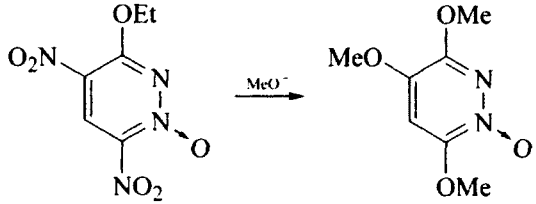
3,6-Dimethyl-4-nitropyridazine 2-oxide (**219**, R = $NO_2$) gave 4-methoxy-3,6-dimethylpyridazine 2-oxide (**219**, R = OMe) (MeONa, MeOH, 25°, 3 h: 75%).[791]

Also other examples, not necessarily involving substrate *N*-oxides.[695,757,809,814,952]

### From Other Alkoxypyridazines (Transalkoxylation)

3-Ethoxy-4,6-dinitropyridazine 1-oxide (**220**) gave 3,4,6-trimethoxypyridazine 1-oxide (**221**) (MeONa, MeOH, reflux, 1 h: 80%).[757]

Also other examples.[673]

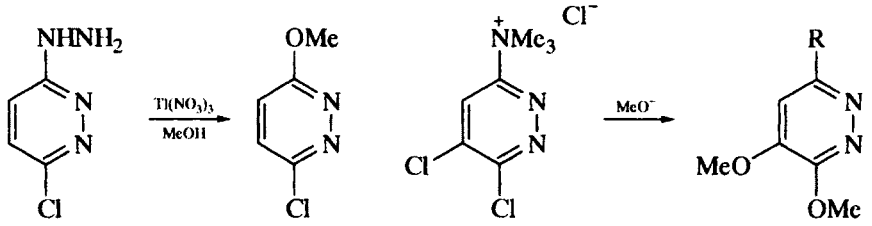



(220) (221)

## From Pyridazinamines

3-Chloro-6-hydrazinopyridazine (**222**) gave 3-chloro-6-methoxypyridazine (**223**) [Tl $(NO_3)_3 \cdot 3H_2O$, MeOH, 25°, 15 min: 68%].[1386]

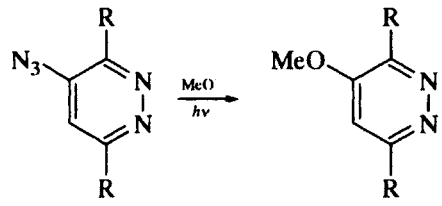
3,4-Dichloro-6-trimethylammoniopyridazine chloride (**224**) gave a separable mixture of 3,4-dimethoxy-6-trimethylammoniopyridazine chloride (**225**, $R = \overset{+}{N}Me_2\ Cl^-$) and 3,4,6-trimethoxypyridazine (**225**, R = OMe) (MeONa, MeOH, reflux, 90 min: yields?).[640]




(**222**) (**223**) (**224**) (**225**)

## From Other Substrates

4-Azidopyridazine (**226**, R = H) or its 3,6-dimethyl derivative (**226**, R = Me) gave 4-methoxy- (**227**, R = H) or 4-methoxy-3,6-dimethylpyridazine (**227**, R = Me), respectively (MeONa, MeOH–dioxane, $N_2$, *hν*, ~90 min: 8% each).[1443]

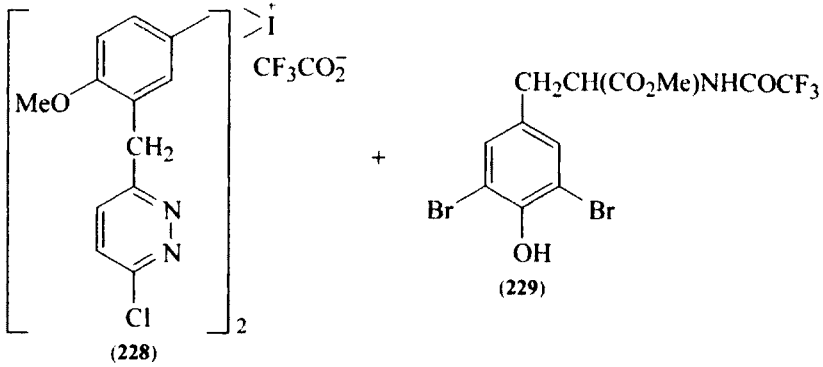



(**226**) (**227**)

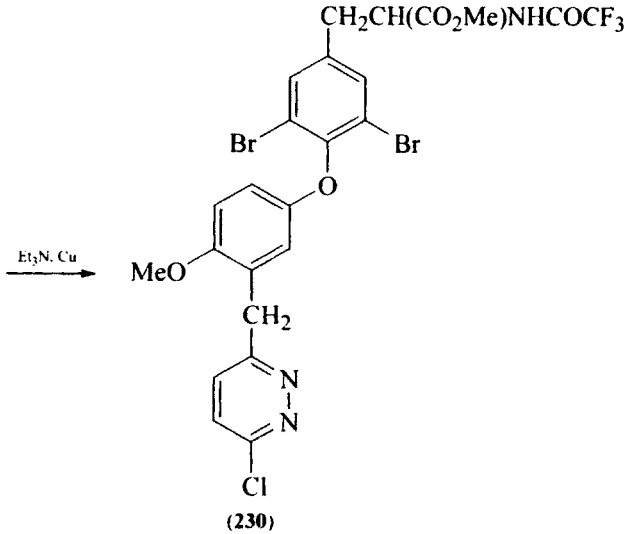
Bis{3-[(6-chloropyridazin-3-yl)methyl]-4-methoxyphenyl}iodonium trifluoroacetate (**228**) with methyl 3-(3,5-dibromo-4-hydroxyphenyl)-2-trifluoroacetamidopropionate (**229**) gave 3-chloro-6-{3-[2,6-dibromo-4-(β-methoxycarbonylethyl-β-trifluoroacetamido)phenoxy]-6-methoxybenzyl}-pyridazine (**230**) ($Et_3N$, Cu bronze, $Ch_2Cl_2$, 25°, 18 h: 55%).[751]

## By Epoxidation Reactions

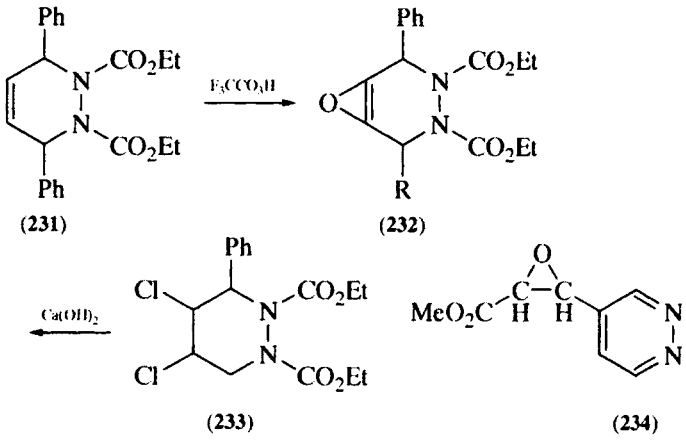
Diethyl 3,6-diphenyl-1,2,3,6-tetrahydro-1,2-pyridazinedicarboxylate (**231**) gave diethyl 3,6-diphenylhexahydro-4,5-epoxypyridazine-1,2-dicarboxylate (**232**, R = Ph) ($F_3CCO_3H$, $CH_2Cl_2$, 40°, 30 min: 92%).[1148]




(228) (229)




(230)




(231) (232) (233) (234)

Diethyl 4,5-dichloro-3-phenylhexahydro-1,2-pyridazinedicarboxylate (**233**) gave diethyl 3-phenylhexahydro-4,5-epoxypyridazine-1,2-dicarboxylate (**232**, R = H) [$Ca(OH)_2$, EtOH–$H_2O$, 75°, 24 h: 37%].[1153]

4-Pyridazinecarbaldehyde gave 4-(2-methoxycarbonyl-1,2-epoxyethyl)pyridazine (**234**) ($ClCH_2CO_2Me$, MeONa, MeOH, 0°, 2 h; then 25°, 4 days: 30%).[1906]

Also other examples.[1028,1858]

### 5.4.2 Reactions of Alkoxy- and Aryloxypyridazines (*H* 57)

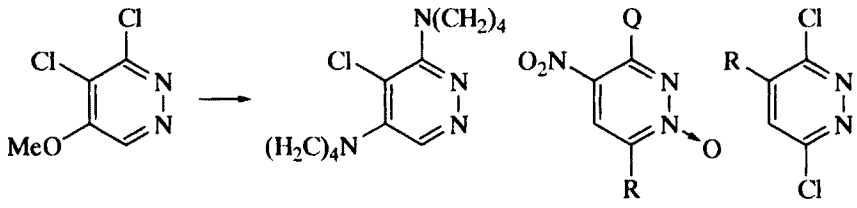
Such pyridazine ethers, both nuclear and extranuclear, undergo several useful reactions. Their *hydrolysis to tautomeric pyridazinones or hydroxyalkylpyridazines* has been discussed already (Sections 5.1.1 and 5.3.1, respectively) and their other reactions are covered in the subsections that follow.

#### *5.4.2.1 Aminolysis and Related Processes (H 64, 475)*

Although there is an unfortunate lack of simple cases in recent reports, alkoxypyridazines undergo aminolysis (and especially hydrazinolysis) reasonably easily in the following typical examples:

3,4-Dichloro-5-methoxypyridazine (**235**) gave 4-chloro-3,5-dipyrrolidin-1′-ylpyridazine (**236**) [neat(?) $HN(CH_2)_4$, reflux, ? h: 88%].[224]

3-Methoxy-4,6-dinitropyridazine 1-oxide (**237**, Q = OMe, R = $NO_2$) gave a separable mixture of 3-benzylamino-4,6-dinitropyridazine 1-oxide (**237**, Q = $NHCH_2Ph$, R = $NO_2$) and 3-benzylamino-6-methoxy-5-nitropyridazine 2-oxide (**237**, Q = OMe, R = $NHCH_2Ph$) ($PhCH_2NH_2$, EtOH, reflux, 3 h: 35% and 36%, respectively).[905]



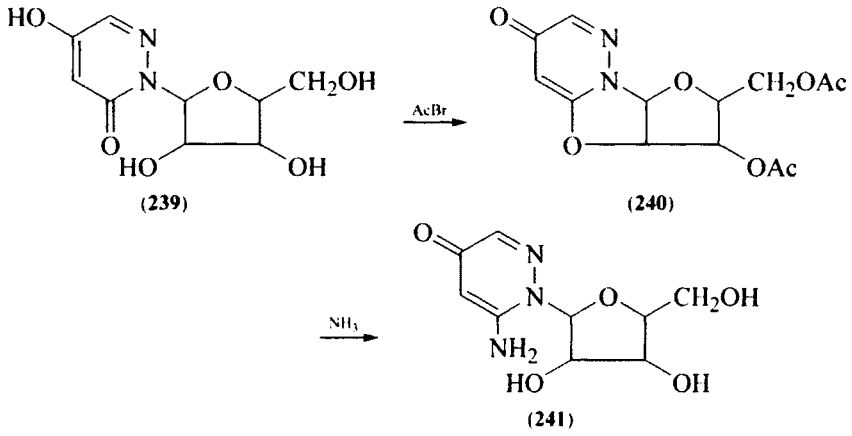

(**235**) (**236**) (**237**) (**238**)

3,6-Dichloro-4-methoxypyridazine (**238**, R = OMe) gave only 3,6-dichloro-4-pyridazinamine (**238**, R = $NH_2$) ($NH_3$, MeOH 105°, sealed, 5 h: ?%) or only 3,6-dichloro-4-hydrazinopyridazine (**238**, R = $NHNH_2$) ($H_2NNH_2 \cdot H_2O$, MeOH, reflux, 1 h: ~80%).[640]

5-Amino-6-methoxy- gave 5-amino-6-hydrazino-3(2*H*)-pyridazinone (neat $H_2NNH_2$, 125°, sealed, 4 h: 71%);[654] cf. the failure of an isomeric substrate to undergo such hydrazinolysis.[1597]

Also other examples.[449,636,760,952,1056]

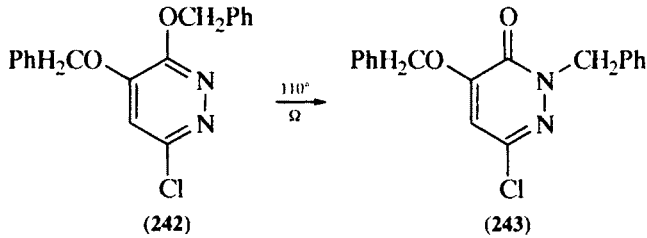
The cyclic ether, 6,2′-anhydro-1-(3′,5′-di-*O*-acetyl-β-D-arabinofuranosyl)-6-hydroxy-4(1*H*)-pyridazinone i.e. 3-acetoxy-2-acetoxymethyl-2,3,3a,9a-tetrahydro-6*H*-furo[2′,3′:4,5]oxazolo[3,2-*b*]pyridazin-6-one (**240**) was easily made from 5-hydroxy-2-β-D-ribofuranosyl-3(2*H*)-pyridazinone (**239**) in 88% yield by treatment with acetyl bromide in acetonitrile at 25° for 2 h. It subsequently underwent ring fission, by warming with liquid ammonia in a sealed vessel at 110° for 34 h, to give 6-amino-1-β-D-arabinofuranosyl-4(1*H*)-pyridazinone (**241**) in 62% yield.[335]




(239) (240) (241)

### 5.4.2.2 *Thermal Rearrangement (H 59)*

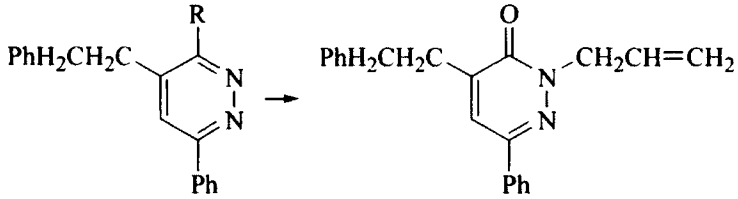
The thermal rearrangement of alkoxypyridazines into *N*-alkylpyridazinones is represented meagrely in recent literature. Thus 3,4-dibenzyloxy-6-chloropyridazine (**242**) gave 2-benzyl-4-benzyloxy-6-chloro-3(2*H*)-pyridazinone (**243**) in unstated yield by simply heating at 110°;[665] and 3-chloro-4-phenethyl-6-phenylpyridazine (**244**, R = Cl) with sodium allyloxide in refluxing allyl alcohol for 18 h gave not the allyloxy product (**244**, R = $OCH_2CH{:}CH_2$) but 2-allyl-4-phenethyl-6-phenyl-3(2*H*)-pyridazinone (**245**) in 58% yield.[1959]



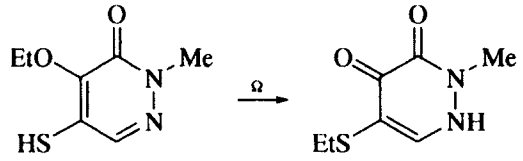

(242) (243)

Other interesting rearrangements have also been reported: 4-ethoxy-5-mercapto-2-methyl-3(2*H*)-pyridazinone (**246**) gave 5-ethylthio-2-methyl-3,4(1*H*,2*H*)-pyridazinedione (**247**) in 72% yield by simply heating in refluxing

pyridine, triethylamine, dimethylformamide, or dimethyl sulfoxide for 4 h:[1896] and deacylation of 3-chloro-6-$\beta$-phthalimidoethoxypyridazine in refluxing ethanolic hydrazine for 2 h afforded 3-choro-6-$\beta$-hydroxyethylaminopyridazine (59%),[1169] apparently by a Smiles rearrangement.[1852d]



(244) (245)

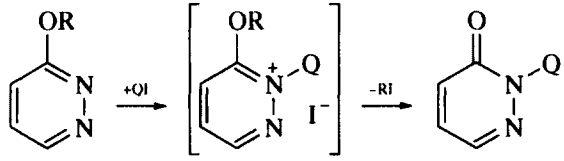


(246) (247)

### *5.4.2.3 Hilbert–Johnson Reaction (H 59)*

Although developed in the pyrimidine series,[380–388,1930d] this useful reaction has been used frequently in the pyridazine series. Typically, treatment of an alkoxy- (**248**, R = alkyl) or trialkylsiloxypyridazine (**248**, R = Si-trialkyl) with an alkyl halide (QI) affords a quaternary salt (**249**), which generally loses the alkyl or trialkylsilyl halide (RI) readily to furnish a (fixed) *N*-alkylpyridazinone (**250**). A trialkylsiloxy substrate is usually favored now, simply because tautomeric pyridazinones tend to undergo both *N*- and *O*-alkylation whereas they undergo only *O*-trialkylsilylation, so making the latter more easily available.[1172]

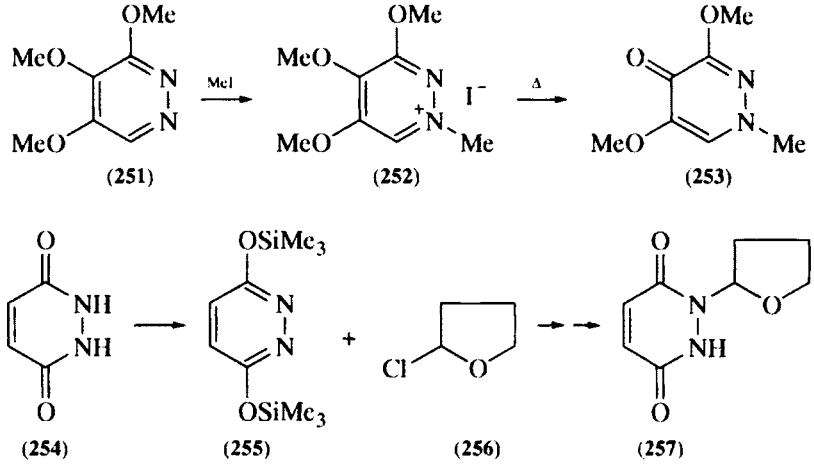
The following examples illustrate both the regular and silyl version of this reaction:



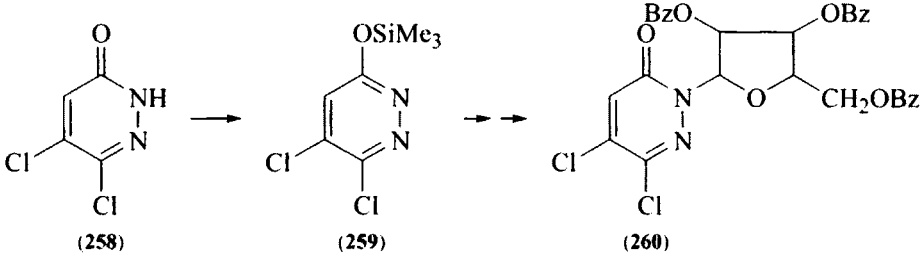
(248) (249) (250)

3,4,5-Trimethoxypyridazine (**251**) gave 3,4,5-trimethoxy-1-methylpyridazinium iodide (**252**) (MeI, PhH, 25°, 12 h: ~75%) and then 3,5-dimethoxy-1-methyl-4(1*H*)-pyridazinone (**253**) (PhH, reflux, 1 h: >90%).[689]

3,6(1*H*,2*H*)-Pyridazinedione (**254**) gave 3,6-bistrimethylsiloxypyridazine (**255**) ($Me_3SiNHSiMe_3 + ClSiMe_3$, 165°, 4 h: 79%), which reacted with 2-chlorotetrahydrofuran (**256**) to afford 1-tetrahydrofuran-2′-yl-3,6(1*H*,2*H*)-pyridazinedione (**257**) (PhH, −15°, 8 h, evaporation, EtOH: ↓: ~50%; no intermediate isolated);[1168] the 1-adamant-1′-yl analogue was made somewhat similarly.[856]



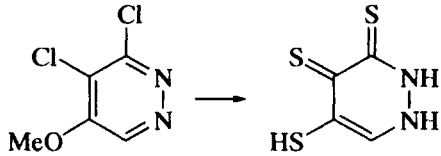

5,6-Dichloro-3(2*H*)-pyridazinone (**258**) gave 3,4-dichloro-6-trimethylsiloxypyridazine (**259**) [$Me_3SiNHSiMe_3$, trace $(NH_4)_2SO_4$, reflux, 2 h: crude] and thence 5,6-dichloro-2-(2,3,5-tri-*O*-benzoyl-β-D-ribofuranosyl)-3(2*H*)-pyridazinone (**260**) (1-*O*-acetyl-2,3,5-tri-*O*-benzoyl-β-D-ribofuranose, $SnCl_4$, $CH_2Cl_2$, 0° → reflux, 1 h: 92% overall: note the use of an acetate plus Lewis acid in place of the usual halide).[1025] Other such pronucleosides were made similarly.[492,932,1285,1300]




### 5.4.2.4 *Other Reactions Including Those of Epoxides*

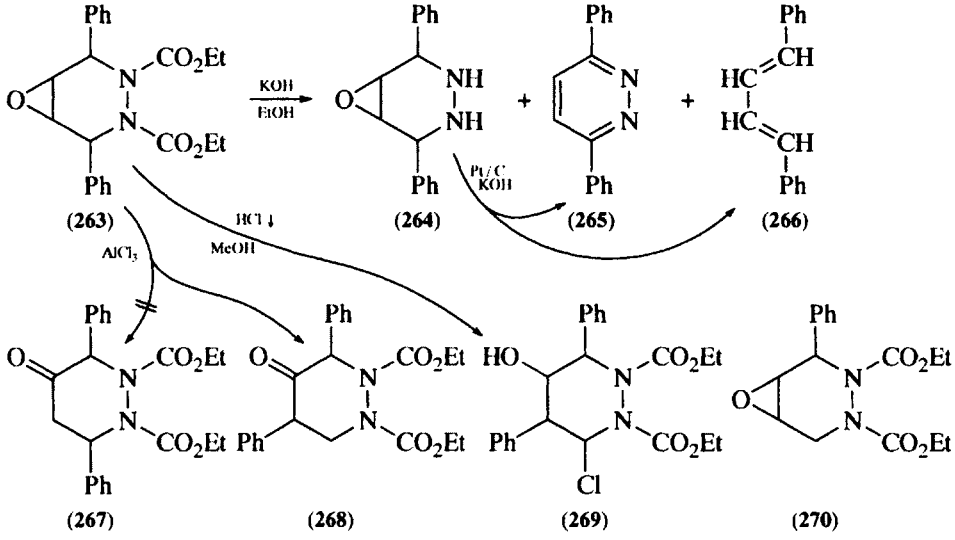
The conversion of methoxypyridazines into tautomeric pyridazinethiones has been reported. Treatment of 3,4-dichloro-5-methoxypyridazine (**261**) with phosphorus pentasulfide in refluxing pyridine for 4 h gave 5-mercapto-

3,4(1*H*,2*H*)-pyridazinedithione (**262**) (77%);[689] and 6-chloro-3-methoxy-4-pyridazinamine gave 4-amino-6-chloro-3(2*H*)-pyridazinethione (85%) on refluxing with thiourea in ethanol for 4 h followed by alkaline treatment.[654] The conversion of an alkoxy- into an alkylthiopyridazine has also been reported: 3-chloro-6-methoxy- gave 3,6-bis-*t*-butylthiopyridazine (23%) by prolonged refluxing with sodium *t*-butylmercaptide in tetrahydrofuran.[1219]



(**261**) (**262**)

Of the few known pyridazine epoxides, diethyl 3,6-diphenylhexahydro-4,5-epoxypyridazine-1,2-dicarboxylate {diethyl 2,5-diphenyl-7-oxo-3,4-diazabicyclo[4.1.0]heptane-3,4-dicarboxylate (**263**)} has been best explored in respect of reactions. Thus treatment in refluxing ethanolic alkali gave a separable mixture of 3,6-diphenylhexahydro-4,5-epoxypyridazine (**264**) (19%), 3,6-diphenylpyridazine (**265**) (38%), and 1,4-diphenylbuta-1,3-diene (**266**);[1148] treatment with aluminum chloride in refluxing carbon tetrachloride for 8 h gave, not the expected product (**267**), but the isomeric diethyl 4-oxo-3,5-diphenylhexahydro-1,2-pyridazinedicarboxylate (**268**) in 80% yield;[1151] and treatment with hydrogen chloride in refluxing methanol for 70 h gave diethyl 3-chloro-5-hydroxy-4,6-diphenylhexahydro-1,2-pyridazinedicarboxylate (**269**) (37%, after separation from a major unidentified byproduct).[1153] Furthermore, the above-mentioned 3,6-diphenylhexahydro-4,5-epoxypyridazine (**264**) underwent a strongly exothermic degradation in neat potassium hydroxide containing



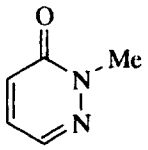
(**263**) (**264**) (**265**) (**266**)

(**267**) (**268**) (**269**) (**270**)

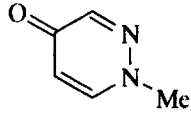
platinum/carbon catalyst from 40° to afford a separable mixture of 3,6-diphenylpyridazine (**265**) (60%) and 1,4-diphenylbuta-1,3-diene (**266**) (>12%).[1148] Diethyl 3-phenylhexahydro-4,5-epoxypyridazine-1,2-dicarboxylate (**270**) underwent a somewhat similar series of reactions.[1858] Much remains to be done with pyridazine epoxides.

## 5.5 NONTAUTOMERIC PYRIDAZINONES AND *N*-ALKYLPYRIDAZINIUMOLATES

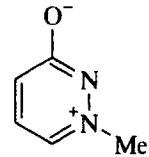
Tautomeric pyridazinones may be rendered nontautomeric (fixed) by *O*-alkylation to afford alkoxypyridazines (Section 5.4) or by *N*-alkylation to furnish compounds typified by 2-methyl-3(2*H*)-pyridazinone (**271**), 1-methyl-4(1*H*)-pyridazinone (**271a**), or 1-methylpyridazinium-3-olate (**272**) (included here for convenience). Such fixed pyridazinones undergo a surprising variety of reactions, not all of which are of much use.




(271)




(271a)



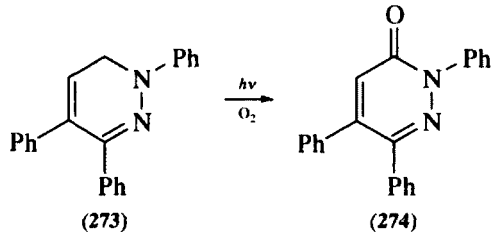

(272)

### 5.5.1 Preparation of Nontautomeric Pyridazinones

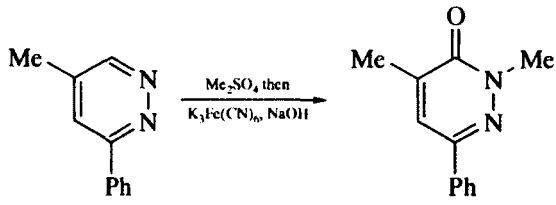
Most such pyridazinones have been made by *primary syntheses* (see Chapters 1 and 2), by *N-alkylation of tautomeric pyridazinones* (see Section 5.1.2.2), or *from alkoxypyridazines by thermal rearrangement or the Hilbert-Johnson reaction* (see Section 5.4.2.2 or 5.4.2.3, respectively). However, there are several other minor ways to make fixed pyridazinones, as illustrated in the following paragraphs:

#### By Oxidation

It is possible to introduce a *C*-oxo substituent by direct oxidation of a pyridazine. For example, irradiation of 1,3,4-triphenyl-1,6-dihydropyridazine (**273**) in 95% ethanol open to the air for 2.5 h afforded 2,5,6-triphenyl-3(2*H*)-pyridazinone (**274**) in ~30% yield;[931] in a different way, 4-methyl-6-phenylpyridazine (**275**) underwent a one-pot methylation and oxidation to 2,4-dimethyl-6-phenyl-3(2*H*)-pyridazinone (**276**) (~40%) by initial quaternization with dimethyl sulfate (without solvent) followed by simultaneous treatment with aqueous potassium ferricyanide and aqueous alkali at >5°.[1104]



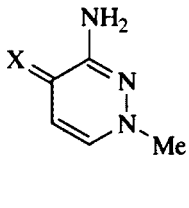

(273) (274)



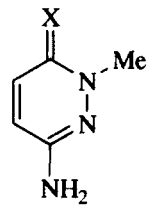

(275) (276)

## From Fixed Pyridazinimines

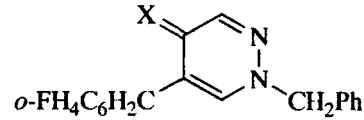
This route is useful when Dimroth rearrangement of the imine (see Section 7.4) may be precluded on structural grounds or very slow because of electron-donating substituents. For example, the hydriodide of 4-imino-1-methyl-1,4-dihydro-3-pyridazinamine (**277**, X = NH) in 1M sodium hydroxide at 95° for 75 min gave 3-amino-1-methyl-4(1*H*)-pyridazinone (**277**, X = O) in ~60% yield;[1132] the hydriodide of 6-imino-1-methyl-1,6-dihydro-3-pyridazinamine (**278**, X = NH) likewise gave 6-amino-2-methyl-3(2*H*)-pyridazinone (**278**, X = O) in unstated yield;[675] and the hydrobromide of 1-benzyl-5-*o*-fluorobenzoyl-4(1*H*)-pyridazinimine (**279**, X = NH) in aqueous methanolic alkali at 25° for 12 h furnished 1-benzyl-5-*o*-fluorobenzoyl-4(1*H*)-pyridazinone (**279**, X = O) in 90% yield.[746] Other examples have been reported.[746,1829,1862]




(277)




(278)

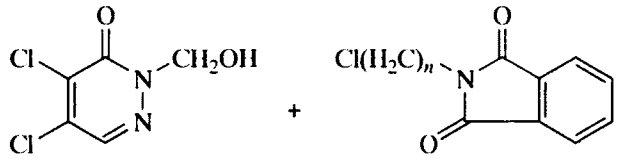



(279)

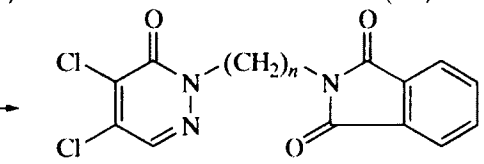
## By *N*-Transalkylation

The conversion of one *N*-alkylpyridazinone into another with a different alkyl group is clearly possible but seldom used. However, 4,5-dichloro-2-hydroxymethyl-3(2*H*)-pyridazinone (**280**) with *N*-(chloromethyl)phthali-

mide (**281**, $n = 1$) in refluxing acetonitrile containing potassium carbonate during >2.5 h gave 4,5-dichloro-2-phthalimidomethyl-3(2*H*)-pyridazinone (**282**, $n = 1$) in 97% yield;[1904] homologous products (**282**, $n = 2$–6)[1904] and some analogues[2037] were made similarly.



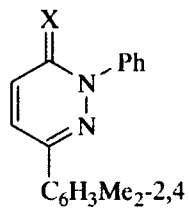
(**280**) (**281**)



(**282**)

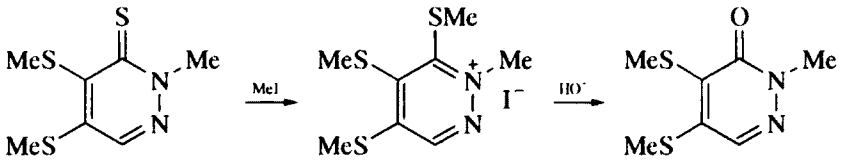
## From Fixed Pyridazinethiones or Alkylthiopyridazines

Isolated examples of these processes have been reported in recent years. For example, 6-(2,4-dimethylphenyl)-2-phenyl-3(2*H*)-pyridazinethione (**283**,



(**283**)

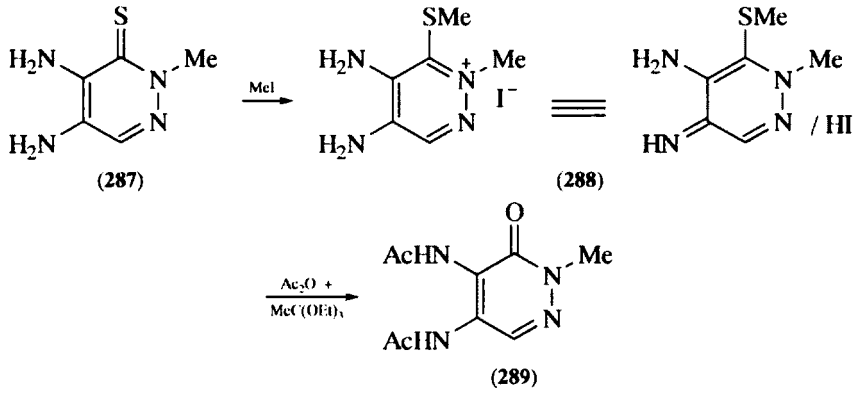
X = S) underwent dye-sensitized photooxygenation to afford the corresponding pyridazinone (**283**, X = O).[1578] In a less direct way, 2-methyl-4,5-bismethylthio-3(2*H*)-pyridazinethione (**284**) and methyl iodide in benzene at 25° for 12 h gave 1-methyl-4,5,6-trismethylthiopyridazinium iodide (**285**)



(**284**) (**285**) (**286**)

(~35%), which underwent alkaline hydrolysis in a few minutes at 25° to give 2-methyl-4,5-bismethylthio-3(2*H*)-pyridazinone (**286**) in excellent

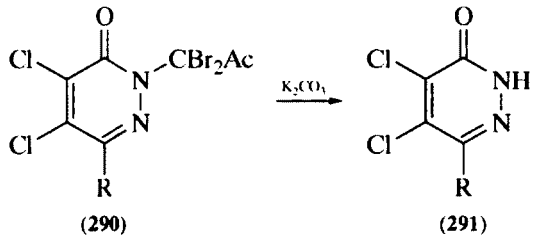
yield;[689] somewhat similarly, 4,5-diamino-2-methyl-3(2*H*)-pyridazinethione (**287**) in refluxing methyl iodide for 24 h gave its methiodide [5-imino-2-methyl-3-methylthio-2,5-dihydro-4-pyridazinamine hydriodide (**288**)] (>95%) and thence, on an attempted cyclization in acetic anhydride-triethyl orthoacetate under reflux for 3 h, 4,5-diacetamido-2-methyl-3(2*H*)-pyridazinone (**289**) in 38% yield.[325]




## 5.5.2 Reactions of Nontautomeric Pyridazinones

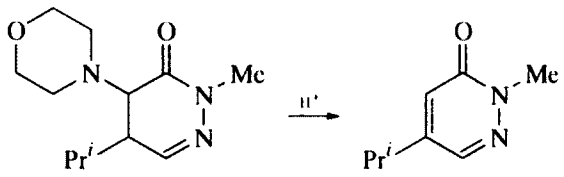
Although the reactions illustrated in the following paragraphs are mainly those that affect the oxo substituent of fixed pyridazinones, a few others are included for obvious reasons.

*N*-DEALKYLATION The dealkylation of an *N*-alkylpyridazinone to afford a tautomeric pyridazinone is possible sometimes. Thus treatment of 4,5-dichloro-2-(1′,1′-dibromoacetonyl)-3(2*H*)-pyridazinone (**290**, R = H) or its 6-nitro derivative (**290**, $R = NO_2$) in aqueous tetrahydrofuran containing potassium carbonate for 2 h at room temperature gave 4,5-dichloro-3(2*H*)-pyridazinone (**291**, R = H) or 4,5-dichloro-6-nitro-3(2*H*)-pyridazinone (**291**, $R = NO_2$), respectively, each in >95% yield.[1673] Other examples have been given toward the beginning of Section 4.1.1.1 and toward the end of Section 5.1.1.



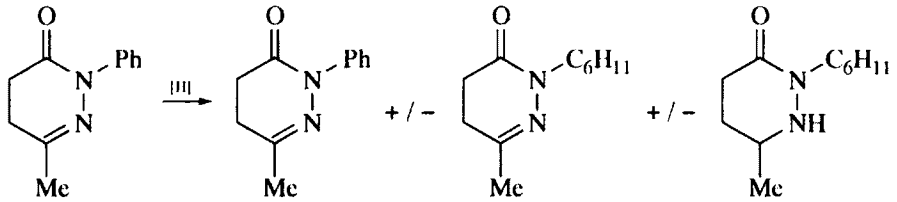

OXIDATIVE AND REDUCTIVE REACTIONS Nuclear oxidation is exemplified in the conversion of 5-isopropyl-4-morpholino-2-methyl-4,5-dihydro-3(2*H*)-pyr-

idazinone (**292**) (made by primary synthesis) into 5-isopropyl-2-methyl-3(2*H*)-pyridazinone (**293**) by elimination of morpholine on warming in 10M hydrochloric acid for 15 min.[327]



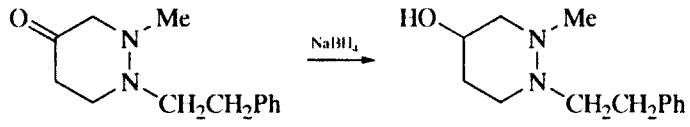
(**292**) (**293**)

Reduction may result in nuclear hydrogenation, formation of a pyridazinol, or deoxygenation.[496] Thus sodium cyanoborohydride reduction of 6-methyl-2-phenyl-4,5-dihydro-3(2*H*)-pyridazinone (**294**) gave only 6-methyl-2-phenyl-1,4,5,6-tetrahydro-3(2*H*)-pyridazinone (**295**) (40%), but hydrogenation over platinum gave a separable mixture of the same product (**295**), 2-cyclohexyl-6-methyl-4,5-dihydro-3(2*H*)-pyridazinone (**296**), and 2-cyclohexyl-6-methyl-1,4,5,6-tetrahydro-3(2*H*)-pyridazinone (**297**), in ratios according to conditions.[691]



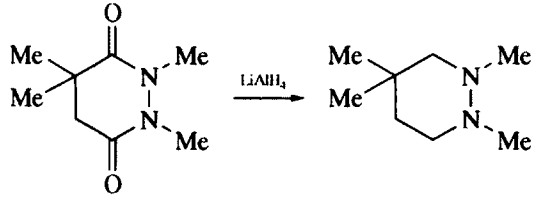
(**294**) (**295**) (**296**) (**297**)

In contrast, the hydrochloride of 2-methyl-1-phenethyl-2,3,5,6-tetrahydro-4(1*H*)-pyridazinone (**298**) with sodium borohydride in methanol for 5 h gave 2-methyl-1-phenethylehexahydro-4-pyridazinol (**299**) in >95% yield.[1826]



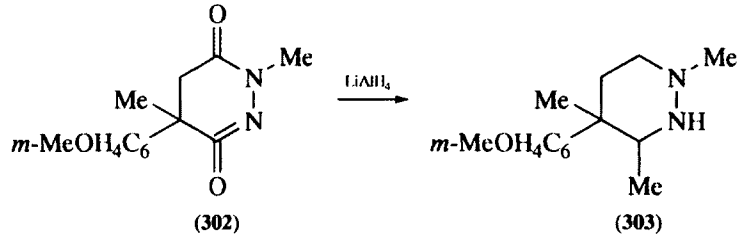
(**298**) (**299**)

Finally, 1,2,4,4,-tetramethyl-4,5-dihydro-3,6(1*H*,2*H*)-pyridazinedione (**300**) suffered reductive deoxygenation to 1,2,4,4-tetramethyl hexahydropyridazine (**301**) (52%) on treatment with lithium aluminum hydride in refluxing tetrahydrofuran for 3 days;[93] likewise, but with dioxane as solvent, 5-*m*-methoxyphenyl-2,5,6-trimethyl-4,5-dihydro-3(2*H*)-pyridazinone (**302**) gave 4-*m*-methoxyphenyl-1,3,4-trimethylhexahydropyridazine (**303**), isolated as hydrochloride in 50% yield;[906] and 2-methyl-3(2*H*)-pyridazinone with lithium aluminum hydride in tetrahydrofuran for 3 h afforded 1-methyl-1,6-dihydropyridazine, albeit in only 10% yield.[790]

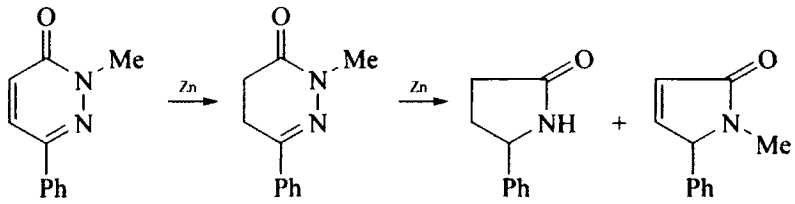


(300) (301)

The reduction of 2-methyl-6-phenyl-3(2*H*)-pyridazinone (**304**) with a single equivalent of zinc dust in boiling acetic acid for 3 h gave only 2-methyl-6-phenyl-4,5-dihydro-3(2*H*)-pyridazinone (**305**) (>75%), but when 3 equiv of zinc were used, a ring contraction occurred to afford a mixture of 5-phenylpyrrolidin-2-one (**306**) and 1-methyl-5-phenyl-3-pyrrolin-2-one (**307**) in about equal amounts; a rational mechanism was proposed.[493]



(302) (303)



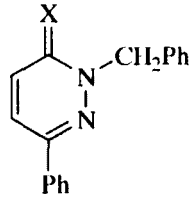
(304) (305) (306) (307)

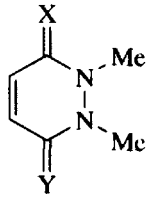
THIATION Fixed pyridazinones appear too undergo thiation at least as easily as do tautomeric pyridazinones. Although phosphorus pentasulfide in refluxing solvents such as pyridine or xylene usually gives reasonable results, Lawesson's reagent (**51**) may be particularly suited to the thiation of fixed pyridazinones (cf. Section 5.1.2.1). The following examples illustrate typical conditions and results:

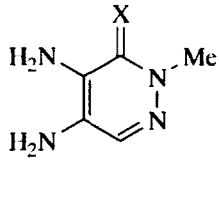
2-Benzyl-6-phenyl-3(2*H*)-pyridazinone (**308**, X = O) gave the corresponding pyridazinethione (**308**, X = S) ($P_2S_5$, xylene, reflux, 2 h: 54%).[1674]

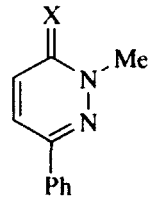
1,2-Dimethyl-3,6(1*H*,2*H*)-pyridazinedione (**309**, X = Y = O) gave a separable mixture of the corresponding pyridazinedithione (**309**, X = Y = S) and 1,2-dimethyl-6-thioxo-1,6-dihydro-3(2*H*)-pyridazinone (**309**, X = O, Y = S) ($P_2S_5$, PhH, reflux, 8 h: ~10% each).[95,cf.672]

4,5-Diamino-2-methyl-3(2*H*)-pyridazinone (**310**, X = O) gave the pyridazinethione (**310**, X = S) ($P_2S_5$, $\beta,\beta$-picoline, reflux, 5 h: 60%).[325]


(308)


(309)


(310)

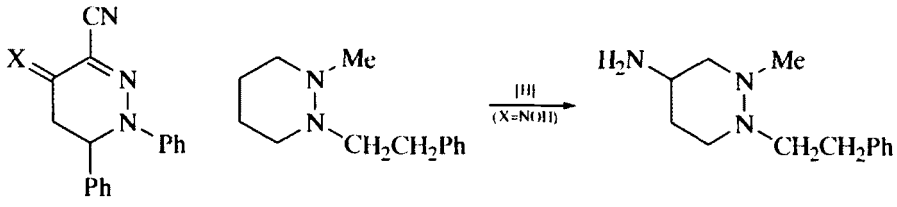

(311)

2-Methyl-5,6-bismethylthio-3(2*H*)-pyridazinone gave the corresponding pyridazinethione ($P_2S_5$, pyridine, reflux, 25 h: ~90%).[1129]

2-Methyl-6-phenyl-3(2*H*)-pyridazinone (**311**, X = O) gave the pyridazinethione (**311**, X = S) (Lawesson's reagent, PhH, reflux, 1 h: >95%).[202]

Also other examples.[371,689,735,1288]

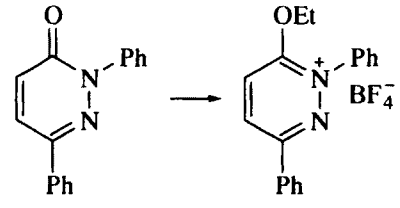
AMINOLYSIS Fixed pyridazinones can undergo displacement of their oxo substituent(s) by hydrazine or hydroxylamine to afford the corresponding hydrazones or oximes. For example, 4-oxo-1,6-diphenyl-1,4,5,6-tetrahydro-2-pyridazinecarbonitrile (**312**, X = O) with hydrazine hydrate and a little sulfuric acid in tetrahydrofuran at 25° for 2 days gave its hydrazone, 4-hydrazono-1,6-diphenyl-1,4,5,6-tetrahydro-3-pyridazinecarbonitrile (**312**, $X = NNH_2$) (60%);[1369] likewise, treatment of a methanolic solution of 2-methyl-1-phenethyl-2,3,5,6-tetrahydro-4(1*H*)-pyridazinone (**313**, X = O) and hydroxylamine hydrochloride with aqueous potassium carbonate at 25° for 30 min afforded the oxime, 4-hydroxyimino-2-methyl-1-phenethylhexahydropyridazine (**313**, X = NOH) (81%) and then, by reduction with lithium aluminum hydride in ether for 6 h, 2-methyl-1-phenethylhexahydro-4-pyridazinamine (**314**) (72%).[1826]


(312) (313) (314)

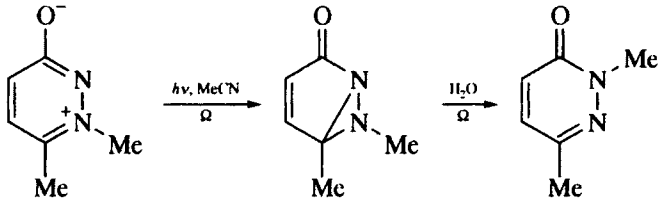
*O*-ALKYLATION It is possible to *O*-alkylate fixed pyridazinones with reagents such as triethyloxonium tetrafluoroborate ($Et_3O \cdot BF_4$) to give quaternary products. For example, 2,6-diphenyl-3(2*H*)-pyridazinone (**315**) and the foregoing reagent in dichloromethane at 25° for a week afforded a 58% yield of 6-ethoxy-1,3-diphenylpyridazinium tetrafluoroborate (**316**), which was converted into the corresponding perchlorate for characterization,[566] several analogues were made similarly.[564,566]

ISOMERIZATION OR RING CONTRACTION *N*-Alkylpyridaziniumolates undergo interesting photorearrangements. Thus irradiation of 1,6-dimethylpyridazinium-

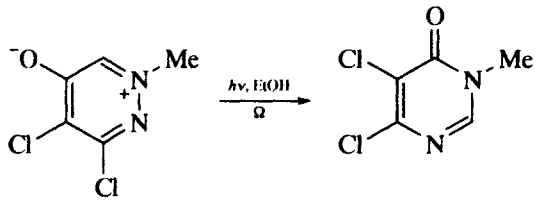
3-olate (**317**) in acetonitrile gave the isolable bridged product, 5,6-dimethyl-1,6-diazabicyclo[3.1.0]hex-3-en-2-one (**318**), which in cold water afforded 2,6-dimethyl-3(2*H*)-pyridazinone (**318a**), both steps in reasonable yield.[397,699] In contrast, irradiation of 5,6-dichloro-2-methylpyridazinium-4-olate (**319**) in ethanol produced 5,6-dichloro-3-methyl-4(3*H*)-pyrimidinone (**320**) in 95% yield;[699] analogues behaved similarly to give isomeric pyrimidinones.[247,699]



(315) (316)

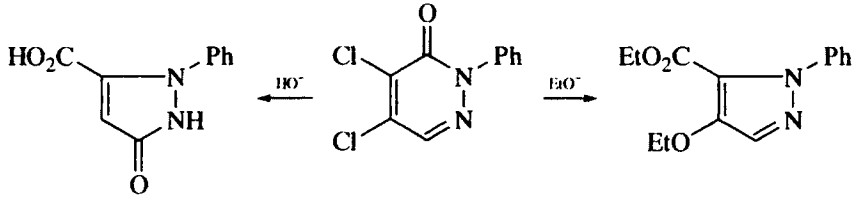


(317) (318) (318a)



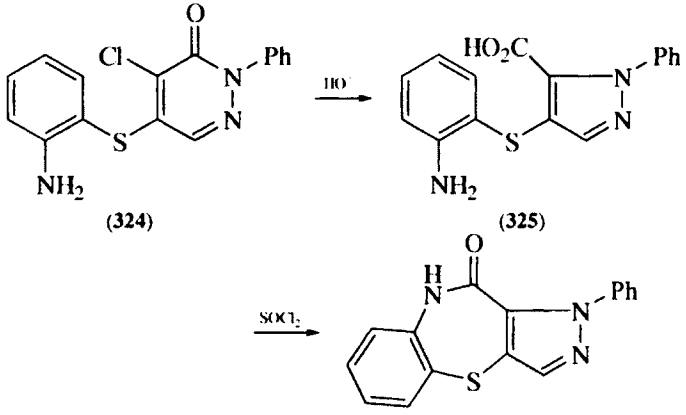
(319) (320)

Work on the previously reported ring contraction of regular fixed pyridazinones[1994,1995] to pyrazoles (*H* 53) has been extended considerably. Thus, whereas 4,5-dichloro-2-phenyl-3(2*H*)-pyridazinone with aqueous alkali in a sealed vessel at 130° for 5 h gave 5-oxo-2-phenyl-3-pyrazoline-3-carboxylic acid (**321**) (~50%).[1996] the same substrate (**322**) in refluxing ethanolic sodium ethoxide for 7 h gave ethyl 4-ethoxy-1-phenyl-5-pyrazolecarboxylate (**323**) as



(321) (322) (323)

the major product in ~40% yield.[764] Related substrates have been shown to undergo this rearrangement also. For example, 5-*o*-aminophenylthio-4-chloro-2-phenyl-3(2*H*)-pyridazinone (**324**) with aqueous alkali at 150° for 10 h afforded 4-*o*-aminophenylthio-1-phenyl-5-pyrazolecarboxylic acid (**325**) (~60%), which then suffered cyclization on treatment with thionyl chloride in refluxing chloroform during 1 h to give 1-phenyl-1*H*-pyrazolo[4,3-*b*][1,5]benzothiazepin-10(9*H*)-one (**326**) (~75%).[776] The mechanisms of such contractions have been studied.[1994-1996]




(324) (325) (326)

CYCLOADDITION REACTIONS 2-Alkyl-3(2*H*)-pyridazinones undergo cycloaddition to their 4,5-bond by diazomethane, 1,3-dienes, or the like. However, the same pyridazinones may also act as dienes with the dienophile, diethyl azodicarboxylate. The following examples illustrate these reactions.

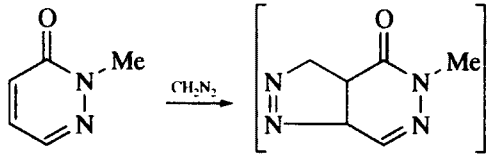
2-Methyl-3(2*H*)-pyridazinone (**327**) and diazomethane slowly gave a separable mixture of 2,5-dimethyl-2*H*- (**329**) and 1,5-dimethyl-1*H*-pyrazolo[3,4-*d*]pyridazin-4(5*H*)-one (**330**), both via (**328**) (excess $CH_2N_2$, $Et_2O$–MeOH, 0°, 15 days: ~60% and ~30%, respectively; also a trace of an isomer produced by the reverse addition of $CH_2N_2$ to the substrate).[199] Analogues behaved similarly.[231]

5-Ethylsulfonyl-2-methyl-3(2*H*)-pyridazinone (**332**) with 2,3-dimethyl-1,3-butadiene gave 4a-ethylsulfonyl-2,6,7-trimethyl-4a,5,8,8a-tetrahydro-1(2*H*)-phthalazinone (**331**) (PhH, reflux, 10 days: 90%);[1427] the same substrate (**332**) with 1-methoxy-1,3-butadiene gave (regioselectively) 4a-ethylsulfonyl-5-methoxy-2-methyl-4a,5,8,8a-tetrahydro-1(2*H*)-phthalazinone (**333**) (PhH, reflux, 4 days: 90%);[1427] and many more examples have been reported.[1427,1510,1633] *Note*: For most such Diels–Alder reactions, the presence of an electron-withdrawing group in the pyridazinone was essential.

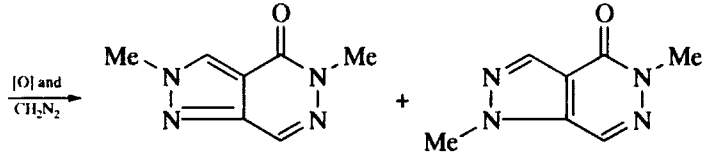
2-Methyl-6-phenyl-3(2*H*)-pyridazinone (**334**) and diethyl azodicarboxylate (**335**) gave diethyl 6-methyl-5-oxo-7-phenyl-1,2,3,6-tetraazabicy-

clo[2.2.2]oct-7-ene-2,3-dicarboxylate (**336**) (EtOH–$H_2O$, baker's yeast, pH 7.2, 37°, 24 h: 76%);[1649] also homologues likewise.[1622,1649]

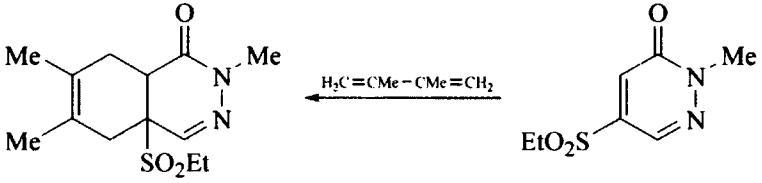
REACTION WITH PHOSPHORYL CHLORIDE Treatment of 2,6-dimethyl-3(2*H*)-pyridazinone (**337**) with phosphoryl chloride gave the quaternary salt, 6-chloro-1,3-dimethylpyridazinium phosphorodichloridate (**338**), which proved useful as a reagent to convert alcohols into alkyl chlorides.[406]



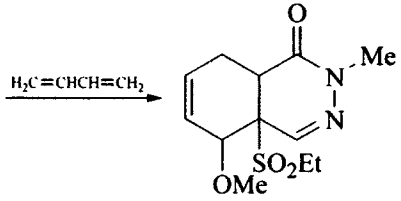
(**327**) (**328**)



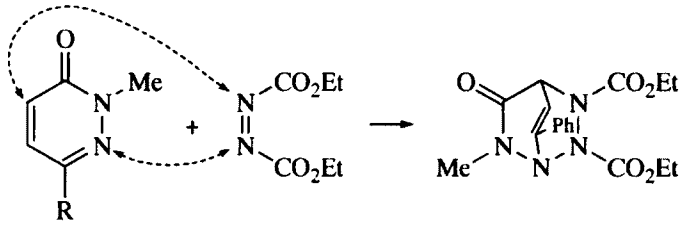
(**329**) (**330**)



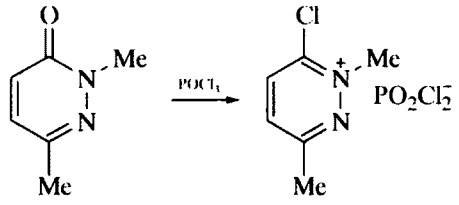
(**331**) (**332**)



(**333**)



(**334**) (**335**) (**336**)




(337) (338)

## 5.6 PYRIDAZINE *N*-OXIDES (*H* 675)

Most of the fundamental chemistry of pyridazine *N*-oxides was reported prior to 1970, so that the following update of information appearing thereafter is composed largely of improved or somewhat complicated examples of known preparations or reactions. A thoughtful theoretical approach to the tautomerism of pyridazinol/pyridazinone *N*-oxides has appeared.[2079]

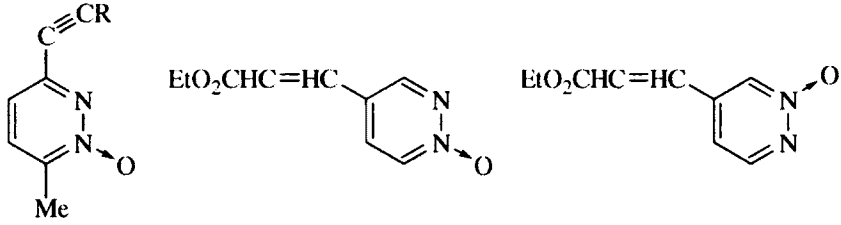
### 5.6.1 Preparation of Pyridazine *N*-Oxides (*H* 677)

Some pyridazine *N*-oxides have been made by *primary syntheses* (see Chapters 1 and 2), but most have been prepared by *N-oxidation of existing pyridazines* with a variety of reagents, of which *m*-chloroperoxybenzoic acid in chloroform appears to be the most generally applicable. The following examples illustrate the reagents and procedures used to make pyridazine *N*-oxides, in each case from the corresponding deoxy-precursor unless otherwise indicated:

**Using *m*-Chloroperoxybenzoic Acid**

3,6-Diphenylpyridazine 1-oxide (*m*-$ClC_6H_4CO_3H$, $CHCl_3$, 25°, 3 days: 87%).[581]

3-Hex-1′-ynyl-6-methylpyridazine 1-oxide (**339**, R = Bu) (*m*-$ClC_6H_4CO_3H$, $CH_2Cl_2$, 25°, 6 h: 64%; note regioselectivity);[191,834] also 3-methyl-6-phenylethynylpyridazine 2-oxide (**339**, R = Ph) (likewise: 92%).[191]

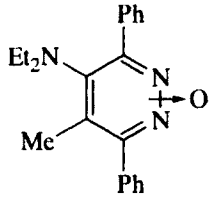



(339) (340) (341)

4-β-Ethoxycarbonylvinylpyridazine 1-oxide (**340**) and the 2-oxide (**341**) (*m*-$ClC_6H_4CO_3H$, $CH_2Cl_2$, reflux, 24 h: 30% and 40%, respectively, after separation).[1906]

4-Diethylamino-5-methyl-3,6-diphenylpyridazine 1 + 2-oxide (**342**) [*m*-$ClC_6H_4CO_3H$, $CH_2Cl_2$, 0° → 25°, 12 h: 88% unseparated; cf. $MeCO_3H$, (made in situ), 73°, 15 h: only 4-ethylamino-5-methyl-3,6-diphenylpyridazine (29%)!].[311]

Also other examples.[123,577,640]



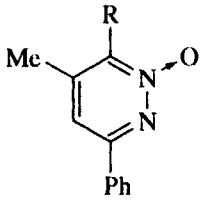
(**342**)

## Using *p*-Nitroperoxybenzoic Acid

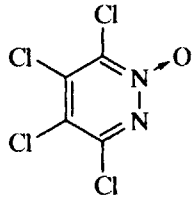
3-Methoxy-4-methyl-6-phenylpyridazine 2-oxide (**343**, R = OMe) (*p*-$O_2NC_6H_4CO_3H$, $CHCl_3$, 25°, 3 days: > 75%); also 3-chloro-4-methyl-6-phenylpyridazine 2-oxide (likewise, 2 days: > 70%).[1104]

## Using Trifluoroperoxyacetic Acid

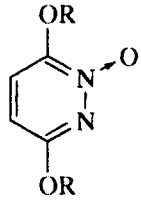
3,4,5,6-Tetrachloropyridazine 1-oxide (**344**) ($F_3CCO_2H$, 90% $H_2O_2$, 25° → reflux, 15 min: 87%); also 3,4,6-trichloropyridazine 1 + 2-oxide (likewise: > 85% unseparated).[893]



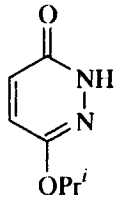
(**343**)



(**344**)



(**345**)



(**346**)

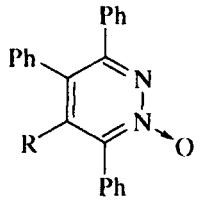
## Using Peroxyacetic Acid

3,6-Bis-β-ethoxyethoxypyridazine 1-oxide (**345**, R = $CH_2CH_2OEt$) (AcOH, 30% $H_2O_2$, 70°, 6 h: > 80%);[640] in contrast, similar treatment of 3,6-diisopropoxypyridazine gave, not the oxide (**345**, R = $Pr^i$), but mainly 6-isopropoxy-3(2*H*)-pyridazinone (**346**).[640]

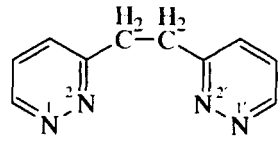
3,4,5,6-Tetraphenylpyridazine 1-oxide (**347**, R = Ph) (AcOH, 30% $H_2O_2$, 70°, 6 h: ~ 80%);[763] also 3,4,6-triphenylpyridazine 1-oxide (**347**, R=H) (likewise: ~ 75%, apparently without any of the isomeric 2-oxide).[763]

α,β-Bispyridazin-3-ylethane (**348**) gave a separable mixture of its 1,1′-, 1,2′-, and 2,2′-dioxide (AcOH, 30% $H_2O_2$, 70°, 3 h: 18%, 60%, and 6%, respectively).[767]

Also other examples.[97,833]



(347)



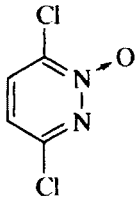
(348)

### Using Monoperoxymaleic Acids

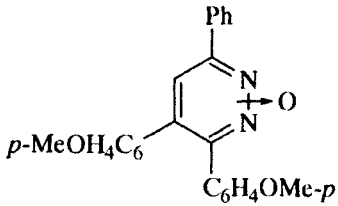
3,6-Dichloropyridazine 1-oxide (**349**) (maleic anhydride, 74% $H_2O_2$, $CHCl_3$, 5°, 7 days: 50%;[891] α,β-dichloromaleic anhydride, 90% $H_2O_2$, $CH_2Cl_2$, 7°, 96 h: 86%).[103]

3-Chloro-5,6-bis-*p*-methoxyphenylpyridazine 1 + 2-oxide (**350**) (maleic anhydride, 32% $H_2O_2$, $CHCl_3$, 5°, 15 h; 97% unseparated; separation gave 17% and 3%, respectively).[1335]

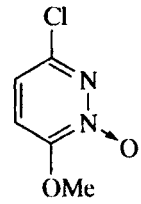
3-Chloro-6-methoxypyridazine 2-oxide (**351**) (likewise, 7 days: 93%, apparently a regioselective oxidation).[449]



(349)



(350)



(351)

### Using Hydrogen Peroxide

3-Azidopyridazine 1-oxide (**352**) (polyphosphoric acid, 85% $H_2O_2$, 25°, dark, 4 days: 2.5% after purification).[1008]

1,2-Dimethyl-1,2,3,6-tetrahydropyridazine 1-oxide (**353**) (MeOH, 30% $H_2O_2$, 25°, 2 days: unstable but identity confirmed by $^{13}C$ nmr).[285]

Also other examples.[913]

## 5.6.2 Reactions of Pyridazine *N*-Oxides (*H* 683)

Pyridazine *N*-oxides undergo a variety of reactions, not all of which affect the oxido entity directly. Both types of reaction are illustrated in the following examples:

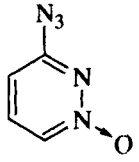
## Deoxygenation by Hydrogenation

6-(4-Methylpiperidino)methyl-3(2*H*)-pyridazinone from its 1-oxide (**354**) (substrate.HCl, MeOH, $H_2$, Pd/C: 93% as hydrochloride).[769]

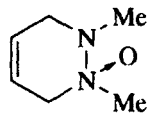
6-*m*-Methoxybenzylamino-3-pyridazinamine from its 2-oxide (**355**) (MeOH, HCl, $H_2$, Pd/C, 25°, 2 h: ~50%).[1526]

4-Amino-6-methyl-3(2*H*)-pyridazinone from 6-methyl-4-nitro-3(2*H*)-pyridazinone 1-oxide (**356**) (MeOH, $H_2$, Pd/C: 73%; note concomitant reduction of the nitro group).[760]

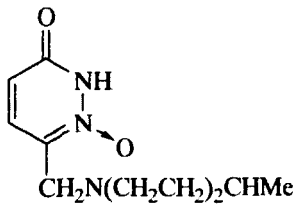
A separable mixture of 3,6-dimethyl-4-pyridazinamine 1-oxide and 2-oxide from 3,6-dimethyl-4-nitropyridazine 1,2-dioxide (**357**) [EtOH, $H_2$ (limited to 4 mol), Pd/C: ~40% each].[796]



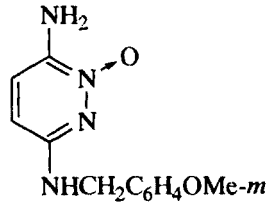
(**352**)



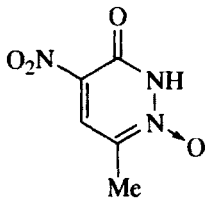
(**353**)



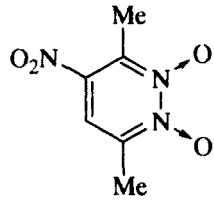
(**354**)



(**355**)



(**356**)



(**357**)

## Deoxygenation by Chemical Reduction

3-Methylpyridazine from its 2-oxide (**358**) [PhOH, MeCN, *hν*, 20 min: 45% of product and 45% of 1,2- + 1,3- + 1,4-$(HO)_2C_6H_4$;[1538] $Ph_3P=S$, MeCN, *hν*: ~35% of both product and $Ph_3P=O$];[1193] also a deoxygenation with styrene in the presence of a Ru-porphyrin catalyst.[1504]

3-Di(β-hydroxyethyl)aminopyridazine from 3-chloro-6-di(β-hydroxyethyl)aminopyridazine 1/2-oxide (**359**) (neat $H_2NNH_2 \cdot H_2O$, reflux, 5 h: ~25%,

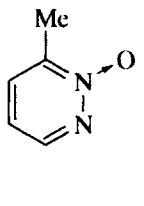
as hydrochloride; note additional dechlorination rather than hydrazinolysis of Cl).[555]

3-Chloro-6-methylthiopyridazine from its 2-oxide [$MoCl_3$ (made in situ), THF–$H_2O$, reflux, 45 min: 64%].[111]

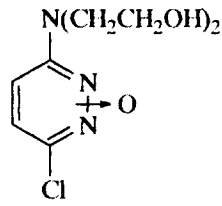
5-(5-Amino-3,6-diphenylpyridazin-4-ylazo)-3,6-diphenyl-4-pyridazinamine from the corresponding azoxy compound (**360**) ($SnCl_2$, AcOH–HCl, 50°, 1 h: 75%; note that the azoxy substrate may be considered as an extranuclear *N*-oxide!).[1358]

4-Azidopyridazine 1-oxide gave 4-azidopyridazine ($PCl_3$, $CHCl_3$, 5° → 25°, 8 h: 70%); analogues likewise.[1443]

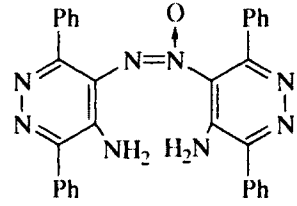
Also other examples.[97,527]




(358)




(359)

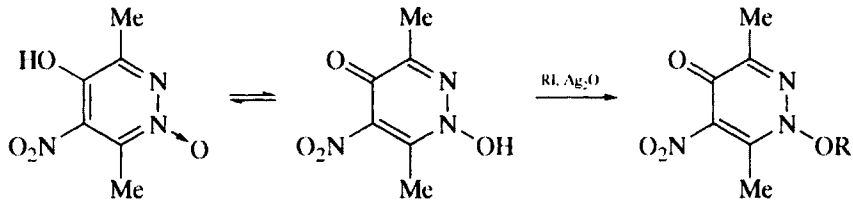



(360)

## *O*-Alkylation Reactions

*Note*: Tautomeric pyridazine *N*-oxide give *N*-alkoxypyridazines by the usual alkylation processes, but nontautomeric pyridazine *N*-oxides can do so only by forming a pyridazinium salt.

3,6-Dimethyl-5-nitro-4-pyridazinol 1-oxide (**361**), usually named as the tautomeric 1-hydroxy-3,6-dimethyl-5-nitro-4(1*H*)-pyridazinone (**361a**), gave 1-methoxy- (**362**, R = Me) (MeI, $Ag_2O$, MeOH, 90°, sealed, 4 h: 89%) or 1-benzyloxy-3,6-dimethyl-5-nitro-4(1*H*)-pyridazinone (**362**, R = $CH_2Ph$) (benzyl halide, likewise: 45%).[794]

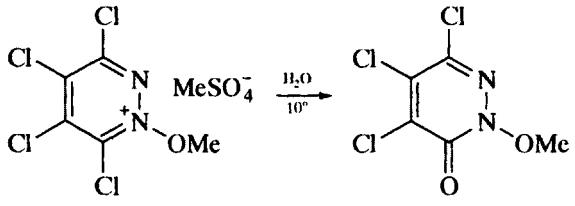



(361) (361a) (362)

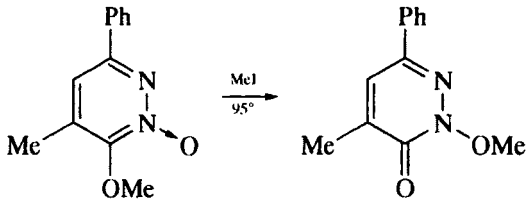
3,4,5,6-Tetrachloropyridazine 1-oxide gave 3,4,5,6-tetrachloro-1-methoxypyridazinium methyl sulfate (**363**) (neat $Me_2SO_4$, 95°, 6 h: crude) and thence 4,5,6-trichloro-2-methoxy-3(2*H*)-pyridazinone (**364**) ($H_2O$, 10°, 1 h: 57%, after separation from minor products).[893]

3-Methoxy-4-methyl-6-phenylpyridazine 2-oxide (**365**) gave the isomeric 2-methoxy-4-methyl-6-phenyl-3(2*H*)-pyridazinone (**366**), presumably by loss of MeI from a quaternary intermediate (neat MeI, 95°, sealed, 18 h: 5%, after extensive purification).[1104]

Also other examples.[798]



(**363**) (**364**)



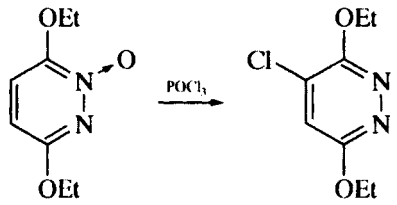
(**365**) (**366**)

## Reaction with Phosphoryl Halides

*Note*: Pyridazine *N*-oxides suffer deoxygenation and *C*-halogenation on treatment with phosphoryl halides.

3,6-Diethoxypyridazine 1-oxide (**367**) gave 4-chloro-3,6-diethoxypyridazine (**368**) ($POCl_3$, $CHCl_3$, 0° → 25°, 4 h: ~75%); also homologues.[640]

3,6-Dimethylpyridazine 1-oxide (**369**) gave an unseparated mixture of 4-chloro-3,6-dimethyl- (**370**, R = Cl) and 3-chloromethyl-6-methylpyridazine (**371**, R = Cl), which was treated immediately with methoxide ion to afford



(**367**) (**368**)

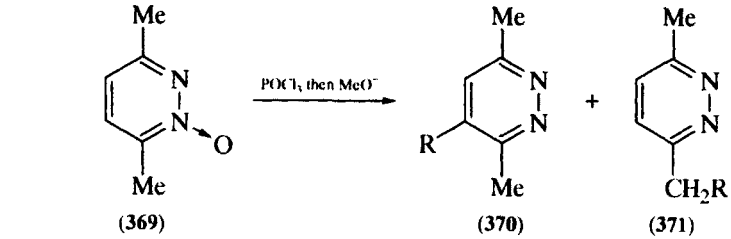
a separable mixture of 4-methoxy-3,6-dimethyl- (**370**, R = OMe) and 3-methoxymethyl-6-methylpyridazine (**371**, R = OMe) ($POCl_3$, 70°, ? h; then MeONa, MeOH, ? h: each 12% after separation);[529] also many analogous data using $POBr_3$, $H_2SO_4$, $Ac_2O$, etc.[529,913]

## Action of Cyanide Ion

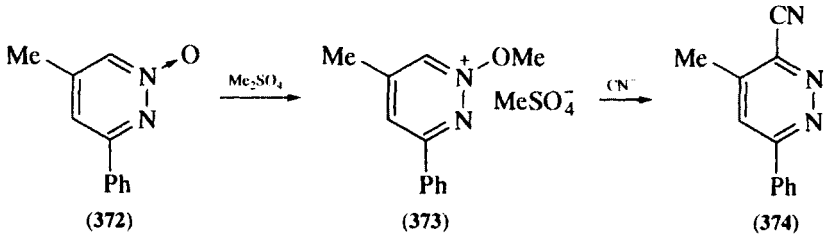
*Note*: Available examples used *N*-alkoxypyridazinium salts (made in situ) as substrates. Direct treatment of pyridazine 1-oxide with cyanide ion failed.[296]

4-Methyl-6-phenylpyridazine 2-oxide (**372**) gave 4-methyl-6-phenyl-3-pyridazinecarbonitrile (**374**) via the quaternary intermediate (**373**) [neat $Me_2SO_4$, 95°, 2 h: then KCN, $H_2O$, 25° (?), 30 min: ~55%].[1104]

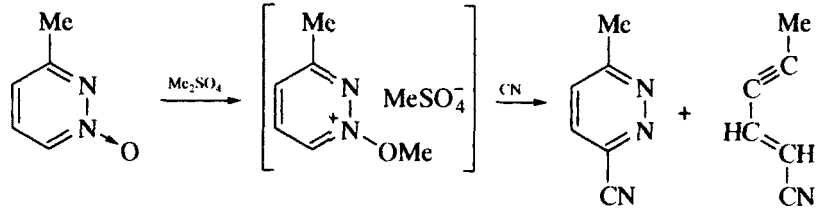
3-Methylpyridazine 1-oxide (**375**) gave the intermediate (**376**) and then a separable mixture of 6-methyl-3-pyridazinecarbonitrile (**377**) and (*cis* + *trans*)-hex-2-en-4-ynenitrile (**378**) (neat $Me_2SO_4$, 95°, 3 h; then KCN, $H_2O$–THF, 5°, 15 min: 3% and 30%, respectively;[498] also related reactions).[798]




(369) (370) (371)




(372) (373) (374)



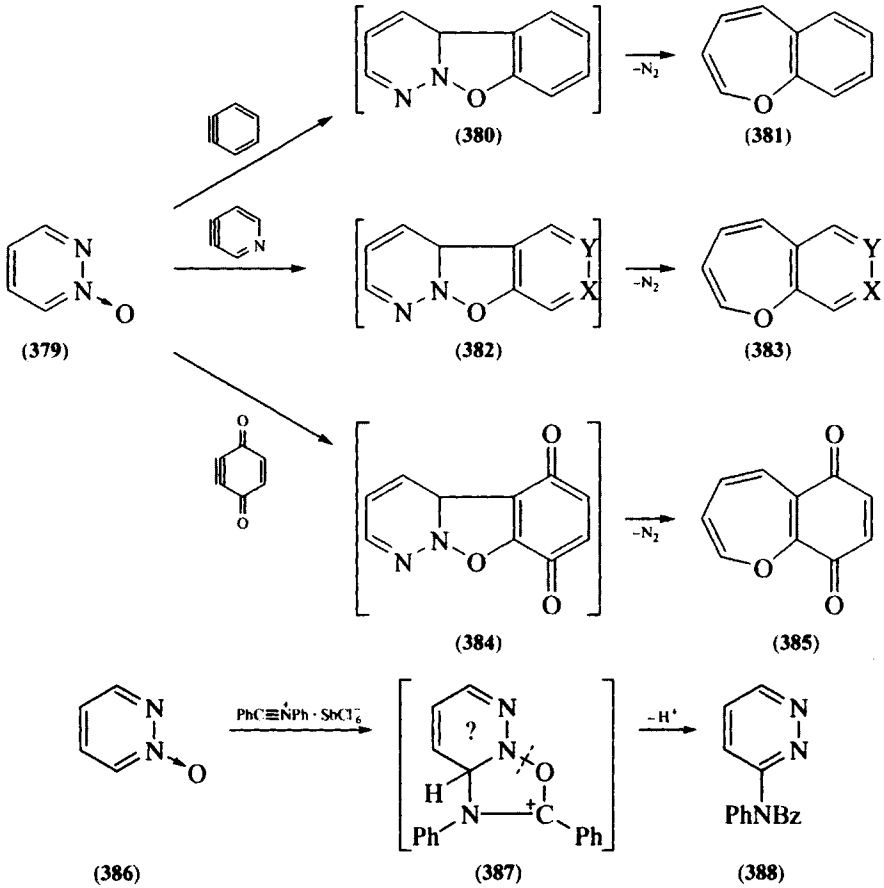

(375) (376) (377) (378)

## Cycloadditions and Subsequent Reactions

Pyridazine 1-oxide (**379**) gave an unisolated adduct (**380**) and then 1-benzoxepine (**381**) [benzyne (made in situ), $CH_2Cl_2$–AcMe, 45°, 2 h: >60%];[797] two unisolated adducts (**382**, X = N, Y = CH; X = CH, Y = N) and then oxepino[2,3-*c*]pyridine (**383**, X = N, Y = CH) and oxepino[3,2-

*c*]pyridine (**383**, X = CH, Y = N) [2,3-didehydropyridine (made in situ), $CH_2Cl_2$, 0°, ? h: > 10% and > 15%, respectively];[1587] or an unisolated adduct (**384**) and thence 6,9-dihydro-1-benzoxepine-6,9-dione (**385**) [2,3-didehydro-*p*-benzoquinone (made in situ), $CH_2Cl_2$, 0°, ? h: 10%].[1908] Alkylated derivatives of the foregoing products were made similarly.[797,1587,1908]

Pyridazine 1-oxide (**386**) gave an unisolated adduct (**387**) and then 3-*N*-benzoylanilinopyridazine (**388**) ($PhC{=}\overset{+}{N}HPh \cdot SbCl_6^-$, $ClCH_2CH_2Cl$, reflux, 17 h: 18%).[306]



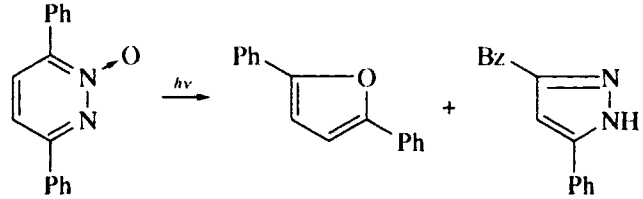

## Photochemical Reactions

*Note*: Irradiation of pyridazine *N*-oxides in solution usually furnishes furans, often accompanied by pyrazoles and/or other cyclic or acyclic products. However, no general pattern can be formulated yet.

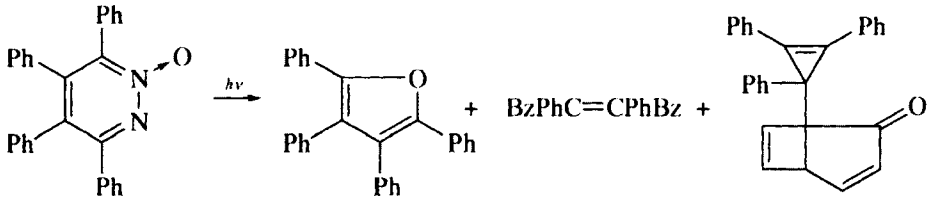
3,6-Diphenylpyridazine 1-oxide (**389**) gave mainly 2,5-diphenylfuran (**390**) and 3-benzoyl-5-phenylpyrazole (**391**) (*hν*, AcMe, 90 min: ~5% and ~50%;

*hν*, AcMe–$C_6H_{11}$, $N_2$, 30 min: 67% and 27%; or *hν*, EtOH, –70°: 43% and 0%).[581]

3,4,5,6-Tetraphenylpyridazine 1-oxide (**392**) gave 2,3,4,5-tetraphenylfuran (**393**), α,β-dibenzoylstilbene (**394**) (*cis* and *trans* separated), 1-(1,2,3-triphenylcyclopropenyl)bicyclo[3.2.0]hepta-3,6-dien-2-one (**395**), and 3,4,5,6-tetraphenylpyridazine (*hν*, AcMe or $CH_2Cl_2$, 3 h: 10%, 20%, 25%, and 15%, respectively).[763]



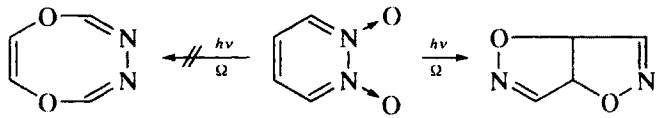
(389) (390) (391)



(392) (393) (394) (395)

Pyridazine 1,2-dioxide (**397**) gave not the dioxadiazocine (**396**), as initially reported,[483] but the isomeric 3a,6a-dihydroisoxazolo[5,4-*d*]isoxazole (**398**) (*hν*, $CH_2Cl_2$, $N_2$, >4 h: 37%; structure confirmed by X-ray analysis).[485]

Also other examples.[187,403,471,577]



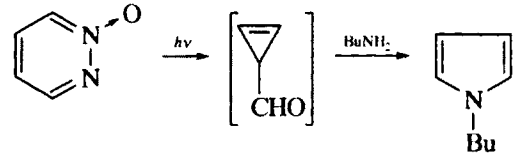
(396) (397) (398)

## Photoassisted Reaction with Amines

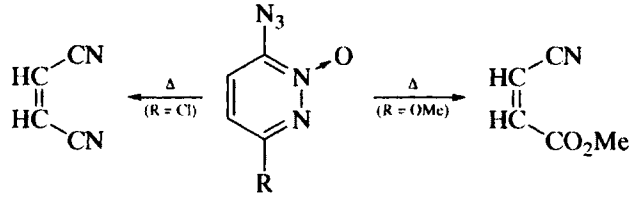
Pyridazine 1-oxide (**399**) and butylamine gave 1-butylpyrrole (**401**), probably via 3-cyclopropenecarbaldehyde (**400**), (*hν*, $CH_2Cl_2$: < 10%;) homologues behaved similarly.[471,770]

## Thermal or Chemical Ring Fission

3-Azido-6-chloropyridazine 2-oxide (**403**, R = Cl) gave maleonitrile (**402**) (PhMe, reflux, ? h: 85%); in contrast, 3-azido-6-methoxypyridazine 2-oxide (**403**, R = OMe) gave methyl 3-cyanoacrylate (**404**) (PhH, reflux, ? h: 89%); possible mechanisms were discussed.[480]

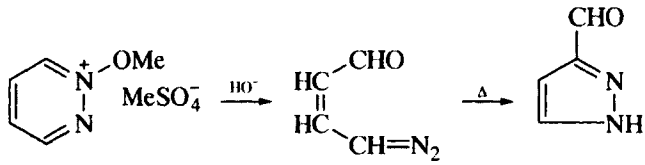


(399) (400) (401)



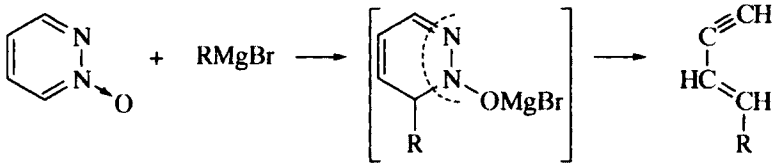
(402) (403) (404)

1-Methoxypyridazinium methyl sulfate (405) gave (stable) 4-diazocrotonaldehyde (406) (KOH, $H_2O$, < 5°, 5 min: > 70%), which underwent thermal cyclization to 3-pyrazolecarbaldehyde (**407**) (Ph, reflux, ?h: > 60%);[479] homologues behaved similarly.[479]



(405) (406) (407)

Pyridazine 1-oxide (**408**) with phenylmagnesium bromide (**409**, R = Ph) gave 1-phenylbut-1-en-3-yne (**411**, R = Ph) (THF, 25°, 30 min: 76%)[700] or with hept-1-ynylmagnesium bromide (**409**, $R = C_5H_{11}C{\equiv}C$) gave a mixture of *cis*- and *trans*-undec-3-en-1,5-diyne (**411**, $R = C{\equiv}CC_5H_{11}$) (likewise: 16%, in ratio 1:5).[272]



(408) (409) (410) (411)

## 5.7 ANCILLARY ASPECTS OF OXYPYRIDAZINES (*H* 36)

Many individual papers on the physical properties, spectra, metal complexes, fine structure, and other such aspects of oxypyridazines involve various types

thereof. Accordingly, it seemed appropriate to mention recent sources of such information in one central location.

### 5.7.1 X-Ray Structural Analyses of Oxypyridazines

The following structures for oxypyridazines have been determined or confirmed by X-ray analyses (others that have been mentioned in the text are noted in the Index):

6-(4-Acetamido-2-methoxyphenyl)-3(2*H*)-pyridazinone[1535]

6-*p*-Acetamidophenyl-3(2*H*)-pyridazinone[1535]

3-Amino-6-methyl-1-*m*-trifluoromethylphenyl-4(1*H*)-pyridazinone (polymorphs: powder X-ray patterns only)[1770]

6-*p*-Aminophenyl-4,5-dihydro-3(2*H*)-pyridazinone[1535]

6-*p*-Aminophenyl-5-methyl-4,5-dihydro-3(2*H*)-pyridazinone, tartrate salt[1535]

6-*p*-Aminophenyl-5-methyl-3(2*H*)-pyridazinone[1535]

6-*o*-Aminophenyl-3(2*H*)-pyridazinone[1535]

6-*p*-Aminophenyl-3(2*H*)-pyridazinone[1535]

5-Benzoyl-2,6-diphenyl-3,4(1*H*,2*H*)-pyridazinedione[85]

5-Benzyl-5-hydroxy-4,4,6-trimethyl-4,5-dihydro-3(2*H*)-pyridazinone[1520]

3,6-Bishydroxymethylpyridazine[173]

6-*p*-(3-Bromopropionamido)phenyl-5-methyl-4,5-dihydro-3(2*H*)-pyridazinone[1535]

3-*o*-(γ-*t*-Butylamino-β-hydroxypropoxy)phenyl-6-hydrazinopyridazine (salt)[1534]

5-*o*-Chlorobenzyl-6-methyl-3(2*H*)-pyridazinone[1579]

5-[β-Chloroethoxy(isobutoxy)phosphinothioyloxy]-4-methylthio-2-phenyl-3(2*H*)-pyridazinone (**412**)[149]

4,5-Dichloro-3,6(1*H*,2*H*)-pyridazinedione[139]

4,5-Dichloro-3(2*H*)-pyridazinone (two polymorphs)[170]

4,5-Dihydro-3,6(1*H*,2*H*)-pyridazinedione (at − 165°)[181,183]

1,2-Dimethyl-4,5-dihydro-3,6(1*H*,2*H*)-pyridazinedione (at − 165°)[182]

3,3-Dimethyl-5-(6-oxo-1,4,5,6-tetrahydropyridazin-3-yl)-2-indolinone[2001]

1,2-Dimethyl-3,6(1*H*,2*H*)-pyridazinedione[140] and Fe complex (**413**)[1207]

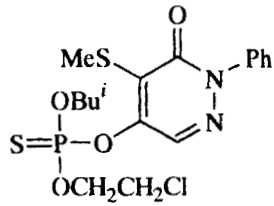
3,6-Dimethylpyridazine 1-oxide, $CuCl_2$ complex (**414**)[260]

Ethyl 4-acetoxy-6-*p*-tolyl-3-pyridazinecarboxylate[2111]

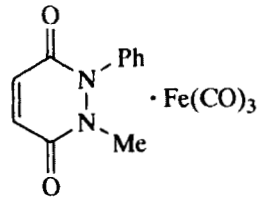
5-Hydroxy-2-phenyl-4-tributylphosphonio-3(2*H*)-pyridazinone chloride[2113]

5-Hydroxy-3(2*H*)-pyridazinone[90]

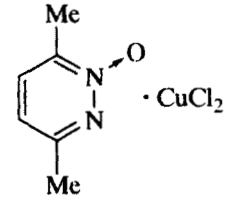
5-Hydroxy-4,4,5,6-tetramethyl-4,5-dihydro-3(2*H*)-pyridazinone[1520]



(412)



(413)



(414)

6-*p*-(Imidazol-1-yl)phenyl-4,5-dihydro-3(2*H*)-pyridazinone, maleate salt[2002]

6-*p*-(Imidazol-1-yl)phenyl-5-methyl-4,5-dihydro-3(2*H*)-pyridazinone, maleate salt[2002]

4-Methoxy-2-methyl-1-phenethyl-1,2,3,6-tetrahydropyridazine[1011]

4-[5-(5-Methoxy-3-methyl-1-phenylpyrazol-4-yloxy)-3-methyl-1-phenylpyrazol-4-yl]-6-methyl-2-phenyl-3(2*H*)-pyridazinone (**415**)[1784]

5-Methoxy-2-methyl-3(2*H*)-pyridazinone[90]

6-Methoxy-2-methyl-3(2*H*)-pyridazinone[175]

5-Methoxy-3(2*H*)-pyridazinone[90]

3-Methoxy-6-sulfanilamidopyridazine[1998]

6-Methyl-4,5-dihydro-3(2*H*)-pyridazinone[1535]

Methyl 6-oxohexahydro-4-pyridazinecarboxylate[1544]

1-Methyl-3,6(1*H*,2*H*)-pyridazinone[176]

4-Methyl-3(2*H*)-pyridazinone[1890]

5-Methyl-3(2*H*)-pyridazinone[1890]

6-Methyl-3(2*H*)-pyridazinone[1890]

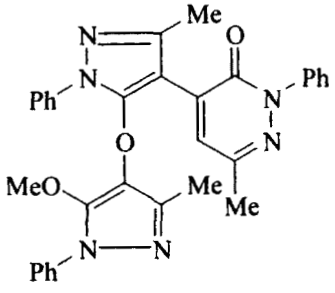
2-*p*-Methylsulfonylphenyl-6-phenylhexahydro-4-pyridazinol[1513]

6-Phenyl-3(2*H*)-pyridazinone, acetate salt[1535]

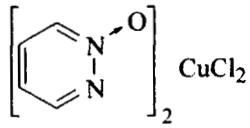
3,6(1*H*,2*H*)-Pyridazinedione (new polymorph)[1533]

Pyridazine 1-oxide, $CuCl_2$ complex (**416**)[260]

2-*p*-Tolyl-3(2*H*)-pyridazinone[1890]



(415)

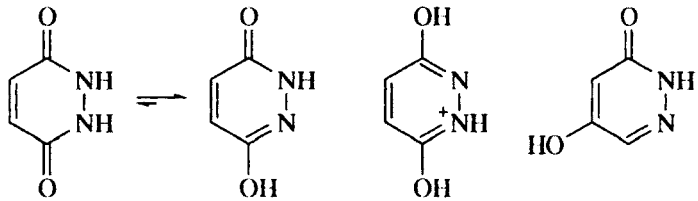


(416)

### 5.7.2 Studies of Fine Structure in Oxypyridazines (*H* 24)

The fact that most pyridazinols existed predominantly as the corresponding pyridazinones had been established firmly by 1970.[1999,2000] However, some doubts, mainly about the tautomeric forms of pyridazinediols and pyridazinetriols, remained to be clarified subsequently by X-ray analysis (for solids) and by ionization and/or spectral studies (for solutions and gases). Some of these results, as well as related findings on aromaticity and conformational forms, are illustrated in the following list:

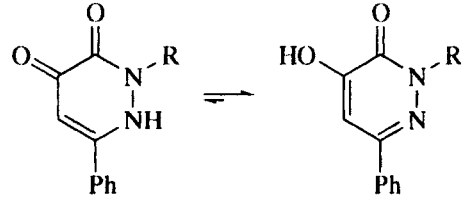
3,6(1*H*,2*H*)-Pyridazinedione (**417**) exists mainly as 6-hydroxy-3(2*H*)-pyridazinone (**417a**) in aqueous media (ionization and uv spectral evidence),[95,1548;cf.1348] in the gas phase,[1052,1548] or in the solid state.[1533] The cation appeared to have the structure (**418**) on uv evidence,[95] but later nmr work suggested that other forms could be significant.[1128]



(**417**) (**417a**) (**418**) (**419**)

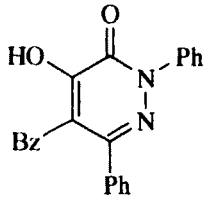
5-Hydroxy-3(2*H*)-pyridazinone (**419**) probably exists as such, rather than as 6-hydroxy-4(1*H*)-pyridazinone, according to a molecular orbital study of possible tautomers.[1052]

6-Phenyl- (**420**, R = H) and 2,6-diphenyl-3,4(1*H*,2*H*)-pyridazinedione (**420**, R = Ph) exist mainly as 4-hydroxy-6-phenyl- (**420a**, R = H) and 4-hydroxy-2,6-diphenyl-3(2*H*)-pyridazinone (**420a**, R = Ph), respectively, as judged from ir spectral evidence.[942] similarly, 5-benzoyl-2,6-diphenyl-3,4(1*H*,2*H*)-pyridazinedione exists as 5-benzoyl-4-hydroxy-2,6-diphenyl-3(2*H*)-pyridazinone (**421**) on X-ray and $^{13}C$ nmr evidence.[85]

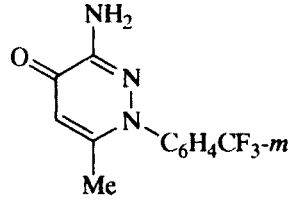


(**420**) (**420a**)

Two polymorphs of 3-amino-6-methyl-1-*m*-trifluoromethylphenyl-4(1*H*)-pyridazinone (**422**) have been characterized thoroughly be nmr and several other techniques, but their respective fine structures remain unformulated.[1770]



(421)



(422)

Conformational allocations for 6-phenyl-4,5-dihydro- and 5-methyl-6-*p*-tolyl-4,5-dihydro-3(2*H*)-pyridazinone derived from molecular mechanics calculations agree with allocations for closely similar compounds derived by X-ray analysis.[2002]

Other solid-state data (see Section 5.7.1) and several confirmatory studies on the tautomerism of simple pyridazinones[570,1392,1656,2048] have been reported.

Aromaticity indices have been calculated for pyridazine and some tautomeric or nontautomeric pyridazinones.[417,1415]

### 5.7.3 General Spectral and Related Studies on Oxypyridazines

Only papers that describe essentially nonroutine applications of spectral or related physicochemical methods to oxypyridazines are cited in the following list:

#### Ionization and Ultraviolet Spectral Studies

The p$K_a$ values for 3(2*H*)-pyridazinone[94,95,1167] and its *N*- and *O*-methyl derivatives;[95] for some 4-, 5-, or 6-substituted analogues;[94,98,1167] and for 3,6(1*H*,2*H*)-pyridazinedione[95,98] have been measured and in some cases compared with values to be expected from the use of Hammett constants.[94]

Full uv data for several of the foregoing pyridazinones have been reported.[95,1190]

#### Nuclear magnetic resonance spectra studies

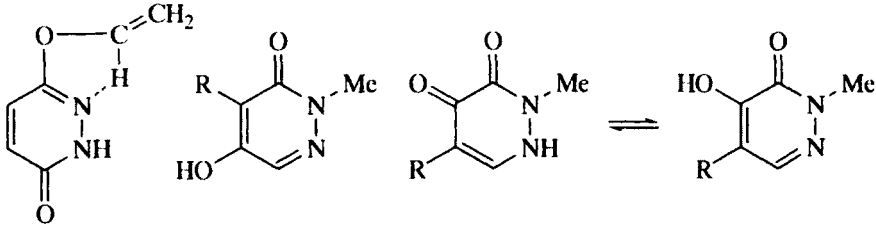
The effects of 2-, 4-, 5-, or 6-substituents on the $^{13}C$ nmr spectrum of 3(2*H*)-pyridazinone have been studied,[719,1213] and a comparison of the $^{13}C$ nmr spectra for 3- and/or 6-substituted pyridazines with those for the corresponding pyridazine *N*-oxides has revealed some interesting substituent effects.[450]

In contrast to $^{1}H$ nmr spectra, $^{13}C$ nmr measurements have been shown to distinguish clearly between *O*- and *N*-methylation of 6-chloro-3(2*H*)-pyridazinone or related heterocyclic lactams.[1349]

The $^{1}H$, $^{13}C$, $^{15}N$, and $^{17}O$ nmr spectra of 6-vinyloxy-3(2*H*)-pyridazinone (**423**) and many heterocyclic analogues clearly indicate hydrogen bonding between the $\alpha$-carbon and adjacent ring nitrogen.[1897]

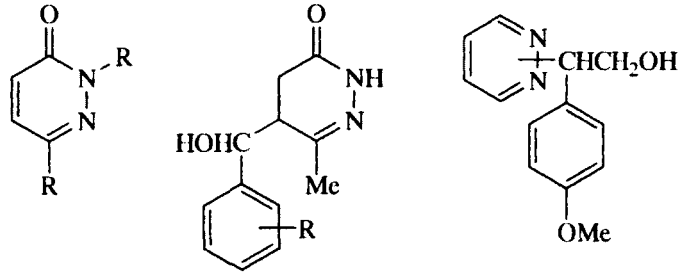
## Other Spectral Studies

The ir, uv, and $^1$H nmr data for > 50 homlogues or analogues of 5-hydroxy-2-methyl-3(2*H*)-pyridazinone (**424**) and 2-methyl-3,4(1*H*,2*H*)-pyridazinedione (**425**) have been collected and used to diagnose cases of intramolecular hydrogen bonding.[143]



(423) (424) (425)

Mass-spectral studies have been reported for series of 2,6-di(alkyl/aryl)-4,5-dihydro-3(2*H*)-pyridazinones (**426**);[502] 5-(α-hydroxy-*o*, *m*, or *p*-substituted-benzyl)-6-methyl-4,5-dihydro-3(2*H*)-pyridazinones (**427**);[1131] and compounds related to 4/5-(β-hydroxy-α-methoxyphenylethyl)pyridazine (**428**).[1970]



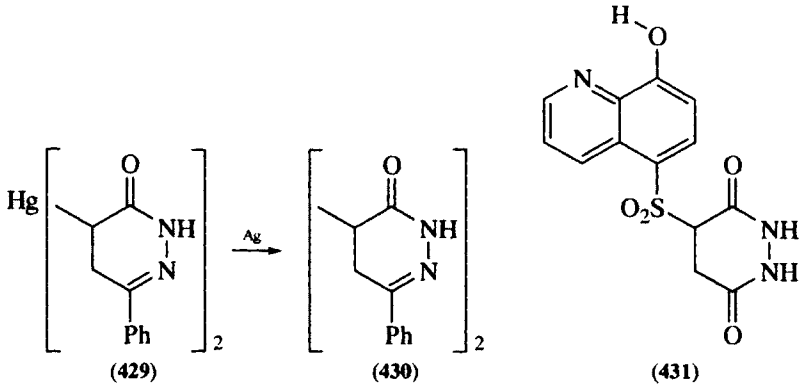
(426) (427) (428)

## Metal Complex Studies

*Note*: A great many metal complexes with oxypyridazines as ligands or coligands have been reported. Only a few typical examples are mentioned here.

Treatment of 6-phenyl-4,5-dihydro-3(2*H*)-pyridazinone with mercuric acetate in boiling acetic acid gave bis(3-oxo-6-phenyl-2,3,4,5-tetrahydropyridazin-4-yl)mercury (**429**) (98%), which underwent demercuration with metallic silver in acetic acid to afford 3,3′-dioxo-6. 6′-diphenyl-2,2′, 3,3′, 4,4′, 5,5′,-octahydro-4,4′-bipyridazine (**430**) ( ~ 99%).[1094]

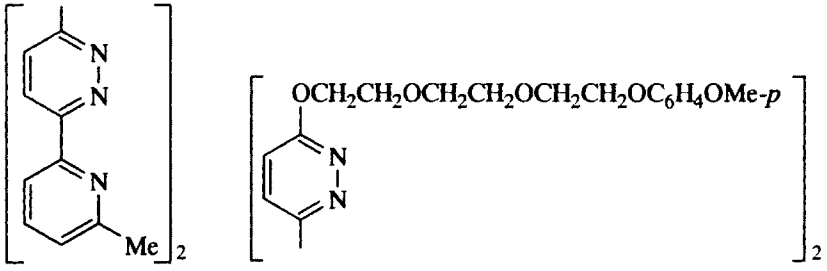
5-(3,6-Dioxohexahydropyridazin-1-ylsulfonyl)-8-quinolinol (**431**) gave metal complexes of the type $L_2Cu$, $L_2Hg$, and $L_2FeNO_3$ that were screened for antibacterial activity.[1393]




(429) (430) (431)

An interesting self-assembled structure has been reported: X-ray crystallography and spectra indicated that it was composed of nine silver ions sandwiched between two layers, each composed of three parallel hexadentate ligands [6,6′-bis(6-methylpyridin-2-yl)-3,3′-bipyridazine (**432**)] and arranged so that the ligands in the first layer were at ~90° to those in the second.[1574]

Several "octopus"-shaped (actually hexapodal!) ruthenium complexes have been made for elaboration into biomimetic models for photosynthesis. A typical complex ($RuL_3Cl_2$) employed 6,6′-bis(10-*p*-methoxyphenyl-1,4,7,10-tetraoxadecyl)-3,3′-bipyridazine (**433**) as each of the three bidentate dipodal ligands.[1899]




(432) (433)

## 5.7.4 Trivial Names for Pyridazine Derivatives

Most of the trivial names commonly used in recent years for pharmaceutical, agrochemical, naturally occurring, and other pyridazines are here listed alphabetically. Each entry is accompanied by its chemical name, type of activity (if applicable), and leading reference(s) or CAS number as appropriate.

Acetylsulfamethoxypyridazine, 3-*p*-acetamidobenzenesulfonamido-6-methoxypyridazine, antibacterial[1782]

Amezinium metilsulfate, 6-methoxy-1-phenyl-4(1*H*)-pyridazinimine methyl hydrogen sulfate, antihypotensive[1575,1777]

Amipizone, 6-*p*-(2-chloropropionamido)phenyl-4-methyl-4,5-dihydro-3(2*H*)-pyridazinone, antihypertensive and platelet-aggregation inhibitor[1777,1782]

Antrimycin(s), structure(s),[1782] natural antibiotic(s)[1375]

Atensil, *see* Pildralazine

Aurantimycin(s), structure(s),[1874] natural antibiotic(s)

1-Azafagomine, (3,4-*trans*-4,5-*trans*)-3-hydroxymethylhexahydro-4,5-pyridazinediol, inhibitor of enzymatic glycoside cleavage[2029,2050]

Azinothrizin, structure,[1782] natural antibiotic

Azintamide, 3-chloro-6-(*N*,*N*-diethylcarbamoylmethylthio)pyridazine, choleretic[1777]

Azinthiamide, *see* Azintamide

Biloral, *see* Azintamide

Brompyrazon, 5-amino-4-bromo-2-phenyl-3(2*H*)-pyridazinone, herbicide, CAS 3042-84-0

Cadralazine, 3-$\beta$-ethoxycarbonylhydrazino-6-(*N*-ethyl-*N*-$\beta$-hydroxypropylamino)pyridazine, antihypertensive[1777]

Cadraten, *see* Cadralazine

Cadrilan, *see* Cadralazine

Cantor, *see* Minaprine

Catos, *see* Medazonamide

Catohexazine, 4,6-dimethyl-3(2*H*)-pyridazinone, hypnotic[1782]

Chloridazon(e), 5-amino-4-chloro-2-phenyl-3(2*H*)-pyridazinone, herbicide[90] CAS 1698-60-8

Delibryl, *see* Ridaflone

Denpidazone, 4-butyl-5-hydroxy-1,2-diphenyl-3,6(1*H*,2*H*)-pyridazinedione, muscle relaxant[1782]

Diclomezine, 6-(3,5-dichloro-4-methylphenyl)-3(2*H*)-pyridazinone, agrofungicide, CAS 62865-36-5

Donnaienin(s), structure(s),[1968] plant products.

Emorfazone, 4-ethoxy-2-methyl-5-morpholino-3(2*H*)-pyridazinone, analgesic and antiinflammatory[1269,1782]

Fenridazon, 1-*p*-chlorophenyl-6-methyl-4-oxo-1,4-dihydro-3-pyridazinecarboxylic acid, agrochemical CAS68254-10-14

Himastatin, structure,[1805] natural antitumor antibiotic

Hydracarbazine, 6-hydrazino-3-pyridazinecarboxamide, antihypertensive[1777]

Imazodan, 6-*p*-(imidazol-1-yl)phenyl-4,5-dihydro-3(2*H*)-pyridazinone, cardiotonic[1449,1451,1777;cf.1306]

Indolidan, 3,3-dimethyl-5-(6-oxo-1,4,5,6-tetrahydropyridazin-3-yl)-2-indolinone, cardiotonic[1451,1777]

Isamfazone, 2-(*N*-α-benzylethyl-*N*-methylcarbamoyl)methyl-6-phenyl-3(2*H*)-pyridazinone, analgesic and antiinflammatory[1782]

Isopulsan, *see* Minaprine

Kantor, *see* Minaprine

Lismont, *see* Amezinium metilsulfate

Luzopeptin, structure,[1806] natural antitumor antibiotic

Malazin, 3-*p*-maleoylaminobenzenesulfonamido-6-methoxypyridazine, antibacterial[1782] CAS 13010-48-5

Maleic hydrazide, 3,6(1*H*,2*H*)-pyridazinedione

Maleohydrazide, 3,6(1*H*,2*H*)-pyridazinedione

Matlystatin-B (also others), 1-[2-(*N*-hydroxycarbamoylmethyl)heptanoyl]-6-[*N*-(2-methyl-1-propionylbutyl)carbamoyl]hexahydropyridazine, natural product[1511,1652,1814;cf.2087,2100]

Medazoamide, *see* Medazonamide

Medazonamide, 1-methyl-6-oxo-1,4,5,6-tetrahydro-3-pyridazinecarboxamide, bronchodilator[1777]

Meribendan, 5-methyl-6-(2-(pyrazol-3-yl)benzimidazol-5-yl-4,5-dihydro-3(2*H*)-pyridazinone, cardiotonic, CAS 119322-27-9

Metamfazone, 4-amino-6-methyl-2-phenyl-3(2*H*)-pyridazinone, analgesic[1782]

Metflurazone, 4-chloro-5-dimethylamino-2-*m*-trifluoromethylphenyl-3(2*H*)-pyridazinone, herbicide CAS 23576-23-0

Minaprine, 4-methyl-3-(β-morpholinoethyl)amino-6-phenylpyridazine dihydrochloride, antidepressant[886,1782] (analogues[2062])

Minoten, *see* Oxdralazine

Mopidralazine, 3-(2,5-dimethylpyrrol-1-yl)-6-morpholinopyridazine, antihypertensive[1777]

Motapizone, 6-[4-(imidazol-1-yl)thien-2-yl]-5-methyl-4,5-dihydro-3(2*H*)-pyridazinone, antihypertensive and antithrombotic[1777,1782]

Nandron, *see* Emorfazone

Nifurprazine, 6-β-(5-nitrofuran-2-yl)vinyl-3-pyridazinamine, antibacterial[1782]

Norflurazone, 4-chloro-5-methylamino-2-*m*-trifluoromethylphenyl-3(2*H*)-pyridazinone, herbicide[90,724] CAS 127314-13-2

Nortimic, *see* Minaprine

Ofunack, *see* Pyridaphenthion

Oragal, *see* Azintamide

Oragallin, see Azintamide

Oxdralazine, 3-di(β-hydroxyethyl)amino-6-hydrazinopyridazine, antihypertensive[1777]

Pentoil, *see* Emorfazone

Pentoyl, *see* Emorfazone

Phenazon(e), 5-amino-4-chloro-2-phenyl-3(2*H*)-pyridazinone, herbicide[865,1130]

Phthalylsulfapyridazine, 3-methoxy-6-(*p*-phthalimidobenzenesulfonamido)pyridazine, antibacterial[1782]

Pildralazine, 3-hydrazino-6-(*N*-β-hydroxypropyl-*N*-methylamino)pyridazine, antihypertensive[1331,1777]

Pimobendan, 6-(2-*p*-methoxyphenylbenzimidazol-5-yl)-5-methyl-4,5-dihydro-3(2*H*)-pyridazinone, cardiotonic[1777,1972;cf.1451]

Piperazic acid, hexahydro-3-pyridazinecarboxylic acid

Piperidazine, hexahydropyridazine

Prizidilol, 3-*o*-(γ-*t*-butylamino-β-hydroxypropoxy)phenyl-6-hydrazinopyridazine,[1534] antihypertensive[1777]

Propildazine, *see* Pildralazine

Pydanon, 4-carboxymethyl-4-hydroxy-4,5-dihydro-3,6(1*H*,2*H*)-pyridazinedione, herbicide CAS 22571-07-9

Pyrazon(e), *see* Phenazon(e); the name *pyrazon*(*e*), has also been used for a pharmaceutical of quite different composition.

Pyridaben, 2-*t*-butyl-5-(*p*-*t*-butylbenzyl)thio-4-chloro-3(2*H*)-pyridazinone, agroinsecticide CAS 96489-71-3

Pyridafenthion, *see* Pyridaphenthion

Pyridaphenthion, 6-diethoxyphosphinothioyloxy-2-phenyl-3(2*H*)-pyridazinone, anticholinesterase[1777]

Pyridate, 3-chloro-5-(octylthio)carbamoyloxy-6-phenylpyridazine, herbicide[90]

Pyridazocidin, 1-(δ-amino-δ-carboxybutyl)-4-carboxypyridazinium chloride, natural phytotoxin[2010]

Pyridazomycin, 1-(δ-amino-δ-carboxybutyl)-4-carbamoylpyridazinium chloride, natural fungicide[1229,1575,1782;cf.1971]

Regulton, *see* Anezinium metilsulfate

Ridaflone, 4-phenyl-2-β-pyrrolidinoethyl-6-*m*-trifluoromethylphenyl-3(2*H*)-pyridazinone, tranquillizer[1782]

Ridazolol, 4-chloro-5-[β-(γ-*o*-chlorophenyl-β-hydroxypropylamino)ethylamino]-3(2*H*)-pyridazinone, cardioselective β-blocker[1777]

Ridiflone, *see* Ridaflone

Rogalin, *see* Azintamide

Salazosulfapyridazine, 3-*p*-(3-carboxy-4-hydroxyphenylazo)benzenesulfonamido-6-methoxypyridazine, antibacterial[1782]

Sulfamethoxypyridazine, 3-methoxy-6-sulfanilamidopyridazine, antibacterial[1782,1998]

Sulfethoxypyridazine, 3-ethoxy-6-sulfanilamidopyridazine, antibacterial[1782]

Supratonin, see Amezinium metilsulfate

Triladine, 2,5,6-trimethyl-3-oxo-2,3-dihydro-4-pyridazinecarbonitrile, analgesic[1782]

Verucopeptin, structure,[1651] natural antitumor antibiotic (also several analogues)

Zardaverine, 6-(4-difluoromethoxy-3-methoxyphenyl)-3(2*H*)-pyridazinone, cardiotonic CAS 101975-10-4

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D. J. Brown
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CHAPTER 6

# Thiopyridazines (*H* 755)

The term *thiopyridazines* is used to cover any pyridazine with a sulfur-containing substituent that is attached directly or indirectly to the nucleus through its sulfur atom. Relationships within the thiopyridazine family are simple. A parent tautomeric pyridazinethione, pyridazinethiol, or mercaptoalkylpyridazine may undergo *S*-alkylation to an alkylthiopyridazine (thioether, RSR′) which may then be oxidized to an alkylsulfinyl- [sulfoxide, RS(=O)R′] or an alkylsulfonylpyridazine [sulfone, $RS(=O)_2R'$]; alternatively, the parent may be oxidized directly to a dipyridazinyl disulfide (RSSR), a pyridazinesulfenic acid [*S*-oxide, RSOH], pyridazinesulfinic acid ($RSO_2H$), or a pyridazinesulfonic acid ($RSO_3H$). The nontautomeric pyridazinethiones and dipyridazinyl sulfides are also included in this chapter, but any thiocyanatopyridazines (RSC≡N) have been relegated to Chapter 8, in which isothiocyanatopyridazines (RN=C=S) and pyridazinecarbonitriles are discussed. Inexplicably, some of the foregoing types of thiopyridazine are poorly represented or even unrepresented in the post-1970 literature, a situation in bold contrast with that in the pyrimidine series.[1930e]

## 6.1. PYRIDAZINETHIONES AND PYRIDAZINETHIOLS (*H* 756)

Although this section is mainly about tautomeric pyridazinethiones, the small amount of available information on nontautomeric (fixed) pyridazinethiones and no nuclear and extranuclear pyridazinethiols is included also.

### 6.1.1 Preparation of Pyridazinethiones and Pyridazinethiols (*H* 758)

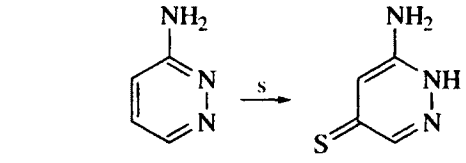
Most tautomeric pyridazinethiones have been made by *primary synthesis* (Chapters 1 and 2), *thiation of pyridazinones* (see Section 5.1.2.1), or *thiolysis of halogenopyridazines* (see Section 4.2.4); a few nontautomeric pyridazinethiones have been made by *primary synthesis* (see Chapters 1 and 2) but most by *thiation of fixed pyridazinones* (See Section 5.5.2); and virtually all extranuclear pyridazinethiols have been made by *thiolysis of corresponding halogenoalkylpyridazines* (see Section 4.4.3) with a single example by thiation of an extranuclear pyridazinol (see end of Section 5.3.2). The remaining

synthetic routes to pyridazinethiones and pyridazinethiols are illustrated in the following examples:

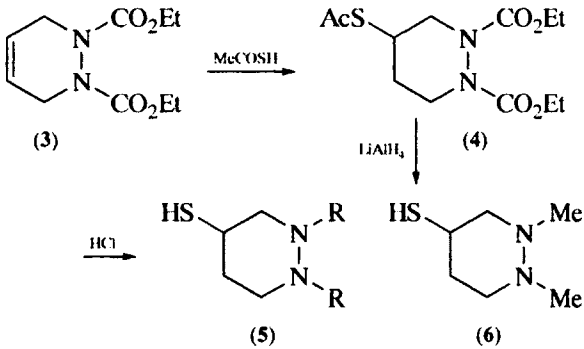
### Direct Introduction of Sulfur

3-Pyridazinamine (**1**) gave 6-amino-4(1*H*)-pyridazinethione (**2**) [neat S, trace 2-Me-pyridine or $(EtO_2C)_2CH_2$, 170°, sealed, 6 h: 45%].[930]

Diethyl 1,2,3,6-tetrahydro-1,2-pyridazinedicarboxylate (**3**) gave diethyl 4-acetylthiohexahydro-1,2-pyridazinedicarboxylate (**4**) (neat MeCOSH, $Bu^tO_2H$, *hν*, 25° → 95°, 90 min: 83%; a radical addition) and then diethyl 4-mercaptohexahydro-1,2-pyridazinedicarboxylate (**5**, R = $CO_2Et$) (HCl gas, EtOH, reflux, $N_2$, 4 h: 76%), hexahydro-4-pyridazinethiol (**5**, R = H) (HCl, AcOH, reflux, $N_2$, 48 h: 43%), or 1,2-dimethylhexahydro-4-pyridazinethiol (**6**) ($LiAlH_4$, $Et_2O$, 25°, 2 h: 54%).[970]




(1) (2)

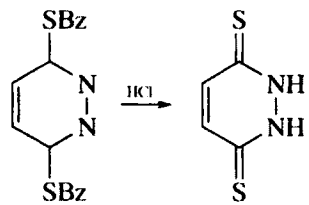



(3) (4) (5) (6)

### From Acylthiopyridazines

3,6-Bisbenzoylthiopyridazine (**7**) gave 3,6(1*H*,2*H*)-pyridazinedithione (**8**) (5M HCl, reflux, 2 h: ~75%).[95]

Also other examples in the preceding paragraph.



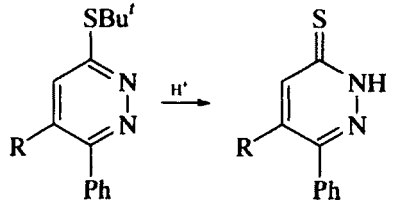

(7) (8)

### From Nitropyridazines

3-Chloro-6-methoxy-5-nitropyridazine 2-oxide gave 6-chloro-1-hydroxy-3-methoxy-4(1*H*)-pyridazinethione (excess NaHS, EtOH, reflux, 15 h: 47%).[905]

### From Alkylthiopyridazines

3-*t*-Butylthio-5-ethoxy-6-phenylpyridazine (**9**, R = OEt) gave 5-ethoxy-6-phenyl-3(2*H*)-pyridazinethione (**10**, R = OEt) (10M HCl, reflux, 1 h: 86%);[1087] 3,5-bis-*t*-butylthio-6-phenylpyridazine (**9**, R = SBu$^t$) gave 5-mercapto-6-phenyl-3(2*H*)-pyridazinethione (**10**, R = SH) ($AlCl_3$, PhMe, reflux, 30 min: 77%).[1087]

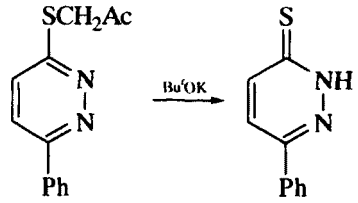



(**9**) (**10**)

3-Acetonylthio-6-phenylpyridazine (**11**) gave 6-phenyl-3(2*H*)-pyridazinethione (**12**) (Bu$^t$OK, $Me_2NCHO$, 60°, 2 h: 90%).[413]

3-*β*-Cyanoethylthio-6-methyl-4-*p*-methylbenzylpyridazine gave 6-methyl-4-*p*-methylbenzyl-3(2*H*)-pyridazinethione (5M NaOH, reflux, 10 h: 65%).[112]

Also other examples.[95,659,689]




(**11**) (**12**)

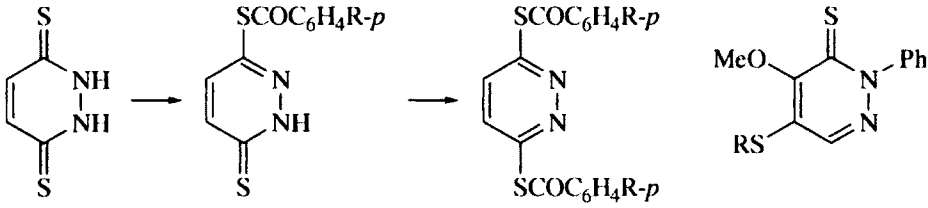
## 6.1.2 Reactions of Pyridazinethiones and Pyridazinethiols (*H* 767)

With one exception (see Section 5.5.1), the *direct hydrolysis of pyridazinethiones or thiols* into pyridazinones or pyridazinols has been avoided recently in favor of indirect conversions, usually via the derived alkylthiopyridazines (see Section 5.1.1). The other reactions of pyridazinethiones or thiols are discussed in the following subsections.

### *6.1.2.1 S-Acylation (H 787)*

This mundane but seldom-used process is applicable to tautomeric pyridazinethiones and thiols but normally not to nontautomeric pyridazinethiones. It is illustrated in the following examples.

3,6(1*H*,2*H*)-Pyridazinethione (**13**) gave an easily separable mixture of 6-benzoylthio-3(2*H*)-pyridazinethione (**14**, R = H) and 3,6-bisbenzoylthiopyridazine (**15**, R = H) (BzCl, 5M NaOH, 25°, brief shaking: each ~30%); retreatment of the first product (**14**, R = H) gave the second (**15**, R = H) (~80%).[95] The same substrate (**13**) also gave 3,6-bis-*p*-methoxybenzoylthiopyridazine (**15**, R = OMe) (*p*-$MeOC_6H_4COCl$, 2M KOH-dioxane, 25°, 1 h: <64%),[365]




(13) (14) (15) (16)

5-Mercapto- (**16**, R = H) gave 5-acetylthio- (**16**, R = Ac) (neat $Ac_2O$, reflux, 5 h: 69%) or 5-ethoxycarbonylthio-4-methoxy-2-phenyl-3(2*H*)-pyridazinone (**16**, R = $CO_2Et$) ($ClCO_2Et$, xylene, reflux, 7 h: 58%).[1896]

Also other examples.[1480]

### *6.1.2.2 S-Alkylation (H 770)*

With rare exceptions (see Section 6.1.2.3), tautomeric pyridazinethiones undergo *S*- rather than *N*-alkylation; extranuclear pyridazinethiols and fixed pyridazinethiones also undergo *S*-alkylation, the latter with the formation of quaternary products. The ease and resulting extensive use of *S*-alkylation reactions are illustrated in the formation of the following typical alkylthiopyridazines, classified according to the type of alkylthio group(s) in each product; the substrate thiones or thiols are not mentioned except when confusion might arise:

#### Simple Alkylthio Derivatives

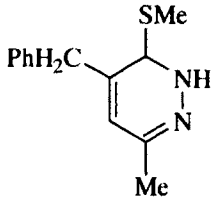
4-Benzyl-6-methyl-3-methylthiopyridazine (**17**) ($Me_2SO_4$, 2.5M NaOH, warm, a few minutes: 72%);[112] 3-ethylthio-6-methyl-4-*p*-methylbenzylpyridazine (EtI, NaOH, EtOH, reflux, 3 h: 82%).[112]

3-*p*-Bromophenyl-6-hexylthiopyridazine ($C_6H_{13}Br$, EtONa, EtOH, reflux, 1 h: <65%).[365]

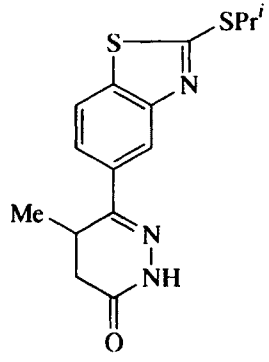
3-Chloro-6-methylthiopyridazine [$Me_2NCH(OMe_2)$, PhMe, reflux, 1 h: 33%].[1135]

3,4,5-Trismethylthiopyridazine [5-mercapto-3,4(1*H*,2*H*)-pyridazinedithione, MeI, 1M NaOH, 30 min: ~80%].[689]

6-(Isopropylthiobenzothiazol-5-yl)-5-methyl-4,5-dihydro-3(2*H*)-pyridazinone (**18**) ($Pr^iI$, 2M NaOH, 25°, 20 min: 62%; alkylation of an extranuclear thione).[1449]



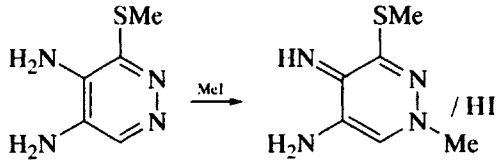

(17)




(18)

3-Methylthio-4,5-pyridazinediamine (**19**) [MeI (1 equiv), 0.5M KOH (1 equiv), 25°, 20 h: 75%][973] or its 1-methiodide: 5-imino-2-methyl-6-methylthio-2,5-dihydro-4-pyridazinamine hydriodide (**20**) (similarly but excess MeI; structure confirmed by X-ray analysis).[197, cf. 2003]

Also other examples.[89, 636, 654, 679, 735, 808, 930, 1064, 1129, 1617, 1813]

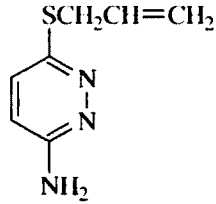


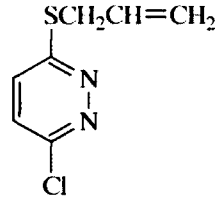

(19) (20)

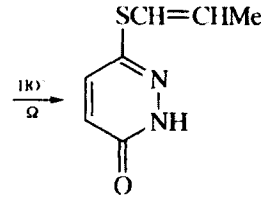
## Simple Alkenylthio Derivatives

6-Allylthio-3-pyridazinamine (**21**) ($H_2C{=}CHCH_2Br$, 1M NaOH, 20°, 12 h: 87%).[1842]

3-Allylthio-6-chloropyridazine (**22**) ($H_2C{=}CHCH_2Br$, MeONa, MeOH, 55°, a few minutes: > 62%) and thence 6-prop-1′-enylthio-3(2*H*)-pyridazinone (**23**) (KOH, $H_2O$–MeOH, reflux, 2 h: 52%; note rearrangement of the alkenylthio group).[557]


(21)


(22)

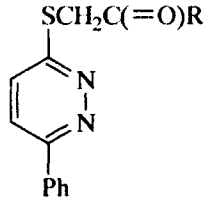

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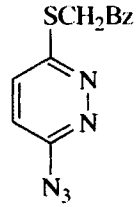
## Acylalkylthio Derivatives

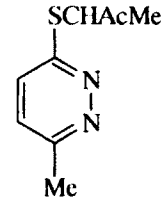
3-Acetonylthio- (**24**, R = Me) ($BrCH_2Ac$, EtONa, EtOH, 25°, a few hours: 50%) or 3-*p*-bromophenacylthio-6-phenylpyridazine (**24**, R = $C_6H_4Br$-*p*) (*p*-$BrC_6H_4COCH_2Br$, likewise: 50%).[413]

3-Azido-6-phenacylthiopyridazine (**25**) ($BzCH_2Br$, EtOH, 25°, 30 min: 84%).[1008]

3-α-Acetylethylthio-6-methylpyridazine (**26**) (ClCHAcMe, KOH, EtOH, 25°, 2 h: 51%); also analogues.[819]


(24)


(25)

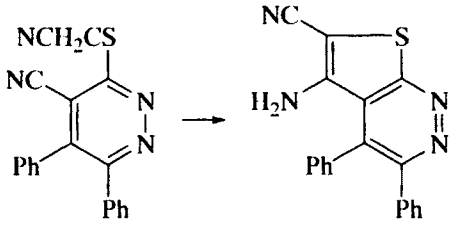

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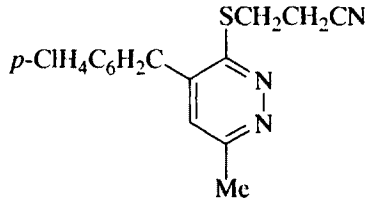
## Cyanoalkylthio derivatives

3-Cyanomethylthio-5,6-diphenyl-4-pyridazinecarbonitrile (**27**) ($ClCH_2CN$, $K_2CO_3$, trace KI, AcMe, 25°, 1 h: 61%) for cyclization to 5-amino-3,4-diphenylthieno[2,3-*c*]pyridazine-6-carbonitrile (**28**) [$(CH_2)_5NH$, AcMe–THF, reflux, 5 h: 70%].[1947]

4-*p*-Chlorobenzyl-3-β-cyanoethylthio-6-methylpyridazine (**29**) ($H_2O$ = CHCN, EtOH, trace NaOH, reflux, 12 h: 77%).[112]

Also other examples.[811,1023,1056]


(27) (28)

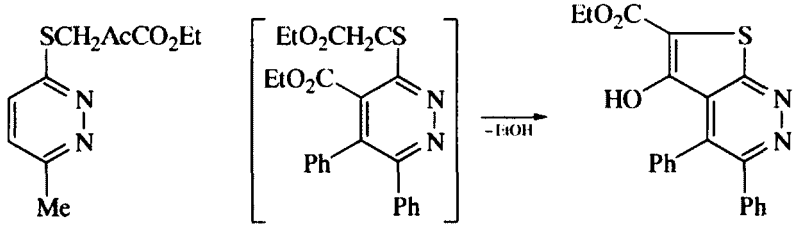

(29)

### Alkoxycarbonylalkylthio Derivatives

3-(1-Ethoxycarbonylacetonyl)thio-6-methylpyridazine (**30**) ($ClCHAcCO_2Et$, KOH, EtOH, 25°, 30 min: 69%).[819]

Ethyl 3-ethoxycarbonylmethylthio-5,6-diphenyl-4-pyridazinecarboxylate (**31**), isolated only as its cyclization product, ethyl 5-hydroxy-3,4-diphenylthieno[2,3-*c*]pyridazine-6-carboxylate (**32**) ($ClCH_2CO_2Et$, EtONa, EtOH, reflux, 3 h: 60%).[390]

Also other examples.[1371]

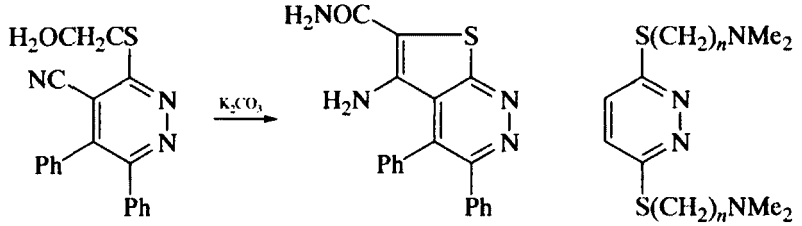



(**30**) (**31**) (**32**)

### Carbamoylalkylthio Derivatives

3-Carbamoylmethylthio-5,6-diphenyl-4-pyridazinecarbonitrile (**33**) ($ClCH_2CONH_2$, $K_2CO_3$, trace KI, MeAc, 25°, 1 h: 66%) and thence 5-amino-3,4-diphenylthieno[2,3-*c*]pyridazine-6-carboxamide (**34**) ($K_2CO_3$, EtOH, reflux, 30 min: 75%).[1913, cf. 1715]

Also other examples.[1142]




(**33**) (**34**) (**35**)

### Aminoalkylthio Derivatives

3,6-Bis(β-dimethylaminoethylthio)pyridazine (**35**, $n = 2$) ($ClCH_2CH_2NMe_2$, 1M NaOH, 25°, 1 h: ~25%) or 3,6-bis(γ-dimethylaminopropylthio)pyridazine (**35**, $n = 3$) ($ClCH_2CH_2CH_2NMe_2$, $NH_3$, EtOH, 25°, 1 h: ~10%).[1142]

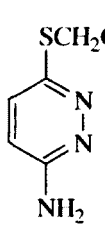
### Benzylthio or Heteroarylthio Derivatives

6-*o*-Methoxybenzylthio-3-pyridazinamine (**36**) ($ClCH_2C_6H_4$ OMe-*o*, 0.3M NaOH, 25°, 4 h: ~70%);[1155] also many analogues.[1152, 1155, 1531]

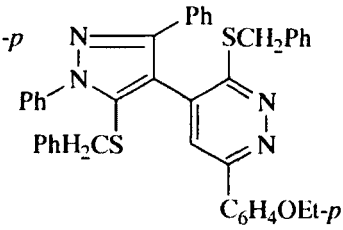
3-Benzylthio-4-(5-benzylthio-1,3-diphenylpyrazol-4-yl)-6-*p*-ethoxyphenylpyridazine (**37**) (corresponding dithione, $PhCH_2Cl$, $NaHCO_3$, $H_2O$–MeAc, 5° → 25°, 2 h: 72%).[1103]

2,6-Dimethyl-5-pyridazin-4′-ylthio-4(3*H*)-quinazolinone (**38**) [4(3*H*)-pyridazinethione, NaH, $Me_2NAc$, 25°, 1 h; then 5-bromo-2,6-dimethyl-4(3*H*)-quinazolinone↓, CuBr, $Cu_2O$, 90°, 4 h: 30%].[1747]

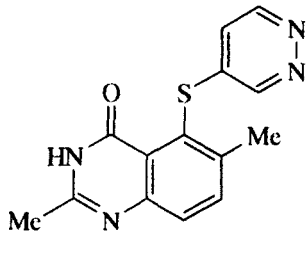
Also other examples.[1226, 1962, 2084]



(36)



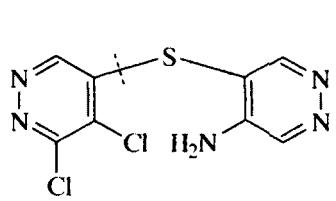
(37)



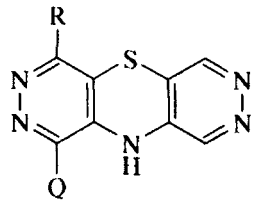
(38)

## Dipyridazinyl Sulfides

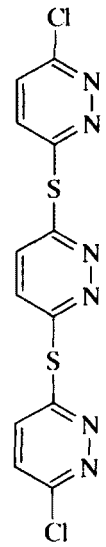
5-(5,6-Dichloropyridazin-4-yl)thio-4-pyridazinamine (**39**) [5-amino-4(1*H*)-pyridazinethione, 3,4,5-trichloropyridazine, KOH, EtOH, 110°, 24 h: 60%], which underwent cyclization to the expected product, 1-chloro-10*H*-dipyridazino[4,5-*b*:4′,5′-*c*][1,4]thiazine (**40**, Q = Cl, R = H) (10M HCl, 90°, 3 h: 58%), or to its unexpected 4-chloro isomer (**40**, Q = H, R = Cl) (AcOH, 90°, 3 h: 57%; said to involve a Smiles[1852d] rearrangement).[923]



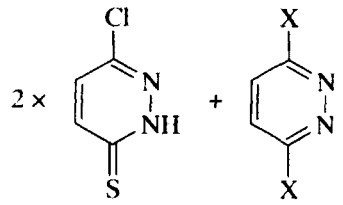
(39)



(40)



(43)



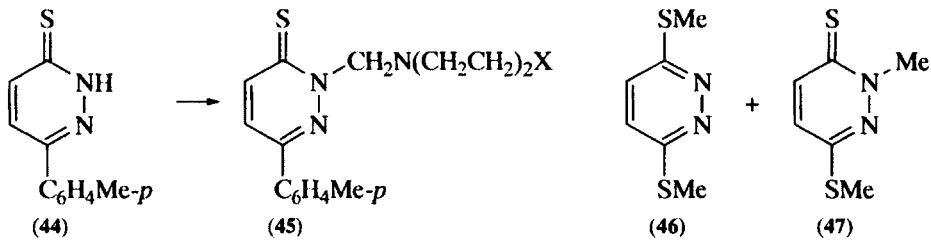
(41) (42)

3,6-Bis(6-chloropyridazin-3-ylthio)pyridazine (**43**) [6-chloro-3(2*H*)-pyridazinethione (**41**) (2 equiv) plus 3,6-dichloro- (**42**, X = Cl) (1 equiv) or 3,6-dibromopyridazine (**42**, X = Br) (1 equiv), MeOH, trace HCl, reflux, 3 h: 68% or 75%, respectively].[315]

Also other examples.[210,867,1087]

### *6.1.2.3 N-Alkylation (H 778)*

Tautomeric pyridazinethiones seldom afford any *N*-alkylated product. However, 6-*p*-tolyl-3(2*H*)-pyridazinethione (**44**) underwent Mannich aminomethylation by formaldehyde and piperidine or morpholine in ethanol at 25° during 12 h to afford 2-piperidinomethyl- (**45**, X = $CH_2$) or 2-morpholinomethyl-6-*p*-tolyl-3(2*H*)-pyridazinethione (**45**, X = O) in 85% or 70%, respectively.[371] Several analogues were made similarly.[371,733] In addition, 3,6(1*H*,2*H*)-pyridazinedithione with dimethyl sulfate in aqueous methanolic sodium methoxide gave a separable mixture of 3,6-bismethylthiopyridazine (**46**) and 2-methyl-6-methylthio-3(2*H*)-pyridazinethione (**47**) in unstated ratio;[95] the possibility that the *N*-methyl derivative (**47**) may have risen indirectly by a Hilbert–Johnson type of reaction (see Sections 5.4.2.3 and 6.2.2.2) was not excluded.[95] The direct (?) formation of *N*-glycosylpyridazinethiones has been reported.[635]

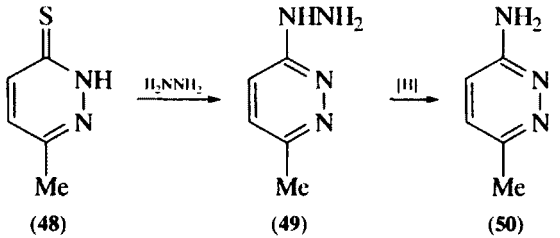


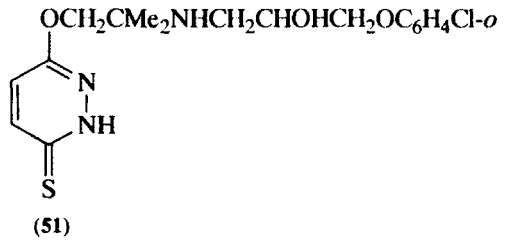

### *6.1.2.4 Aminolysis (H 476, 769)*

Aminolysis of tautomeric pyridazinethiones is usually done indirectly by *S*-alkylation (Section 6.1.2.2) and subsequent aminolysis (Section 6.2.2.2) or by hydrazinolysis (which occurs much more easily than regular aminolyses) and subsequent reductive cleavage of the hydrazino group.

Thus 6-methyl-3(2*H*)-pyridazinethione (**48**) afforded 3-hydrazino-6-methylpyridazine (**49**) by treatment in ethanolic hydrazine hydrate under reflux for 6 h; subsequent hydrogenation over Raney- nickel in ethanol gave 6-methyl-3-pyridazinamine (**50**) in ~65% yield;[1127] Likewise, 3-[2-(3-*o*-chlorophenoxy-2-hydroxypropylamino)-2-methylpropoxy]-6-hydrazinopyridazine was obtained in 65% yield from the corresponding thione (**51**) in refluxing ethanolic

hydrazine hydrate for 3 h:[1813] and other such hydrazinolyses have been reported.[159,636]



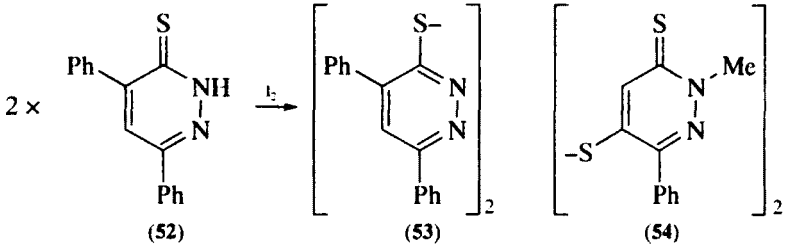




### *6.1.2.5 Oxidation to Dipyridazinyl Disulfides (H 767)*

Tautomeric pyridazinethiones are easily oxidized to the corresponding dipyridazinyl disulfides, as illustrated in the following examples:

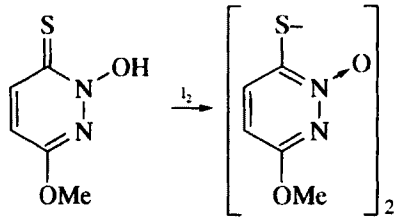
4,6-Diphenyl-3(2*H*)-pyridazinethione (**52**) gave bis(4,6-diphenylpyridazine-3-yl) disulfide (**53**) (NaOH, $H_2O$; $I_2$/KI↓ until color persists: 80%); homologues likewise.[371]

5-Mercapto-2-methyl-6-phenyl-3(2*H*)-pyridazinethione gave bis(1-methyl-3-phenyl-6-thioxo-1,6-dihydropyridazin-4-yl) disulfide (**54**) ($CHCl_3$, air, 25°, 3 days: 60%).[1087]




2-Hydroxy-6-methoxy-3(2*H*)-pyridazinethione (**55**) gave bis(6-methoxypyridazin-3-yl) disulfide 2,2′-dioxide (**56**) ($NaHCO_3$, $H_2O$; $I_2$↓, 25°: 75%).[679]

Also other examples.[112,1578]

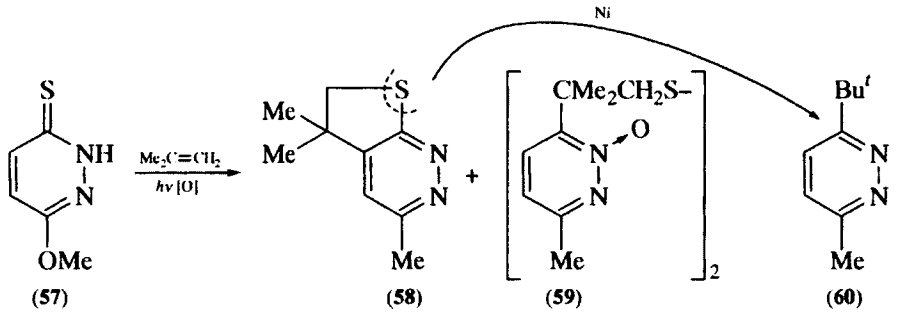


(55) (56)

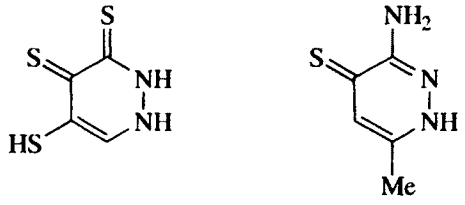
## *6.1.2.6 Other Reactions (H 756, 774)*

Pyridazinethiones can undergo oxidative *photoaddition by olefines*. For example, irradiation of 6-methyl-3(2*H*)-pyridazinethione (**57**) with 2-methylpropene in acetonitrile for 4 h gave a 52% yield of 3,5,5-trimethyl-5,6-dihydrothieno[2,3-*c*]pyridazine (**58**), accompanied by an interesting separable byproduct, bis[2-methyl-2-(6-methylpyridazin-3-yl)propyl] disulfide (**59**) (19%), arising by a rational mechanism; the main product (**58**) gave 3-*t*-butyl-6-methylpyridazine (**60**) on desulfurization with Raney nickel.[880]

Recently described *metal complexes* of pyridazinethiones are typified by those of 5-mercapto-3,4(1*H*,2*H*)-pyridazinedithione (**61**) with Pd, Rh, Ru, and Pt;[1260] also by those of 3-amino-6-methyl-4(1*H*)-pyridazinethione (**62**) with Cu.[463]



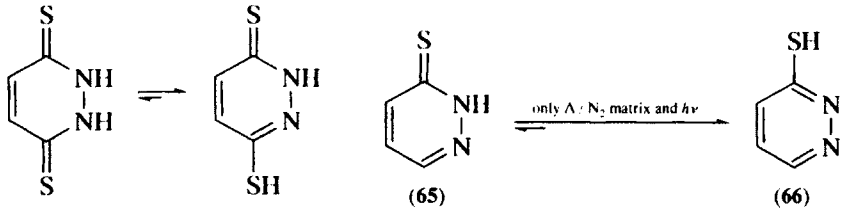
(57) (58) (59) (60)



(61) (62)

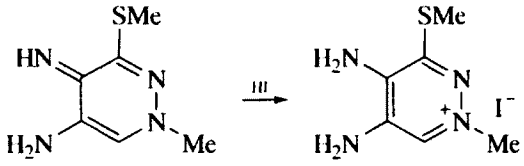
Previous studies on the *tautomerism* of pyridazinethiones have established that, in general, most exist as thiones rather than thiols.[1999,2000;cf.570] However, 3,6(1*H*,2*H*)-pyridazinedithione (**63**), usually named as such for pragmatic

reasons, exists predominantly as 6-mercapto-3(2*H*)-pyridazinethione (**64**) at least in aqueous media,[95] a conclusion consistent with the calculated aromaticity index for tautomer (**64**).[1415] Moreover, even 3(2*H*)-pyridazinethione (**65**) can exist mainly as 3-pyridazinethiol (**66**) when irradiated in a nitrogen or argon matrix.[1653]




## 6.2 ALKYLTHIOPYRIDAZINES AND DIPYRIDAZINYL SULFIDES (*H* 769)

A great many alkylthiopyridazines have been made, not only as end products for biological testing, but also as intermediates for the preparation of other pyridazines. The hydriodide of 5-imino-2-methyl-6-methylthio-2,5-dihydro-4-pyridazinamine (**67**) has been shown to exist as 4,5-diamino-1-methyl-3-methylthiopyridazinium iodide (**68**) in the solid state by X-ray analysis.[165]

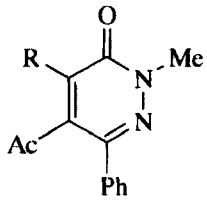



### 6.2.1 Preparation of Alkylthiopyridazines (*H* 769)

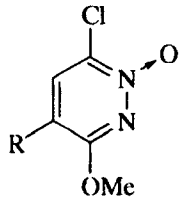
Most of the routes to alkylthiopyridazines have been discussed already: by *primary synthesis* (see Chapters 1 and 2), by *alkanethiolysis of halogenopyridazines* (see Sections 4.2.5 and 4.4.4), by *alkanethiolysis of alkoxypyridazines* (see Section 5.4.2.4), or by *S-alkylation of pyridazinethiones or pyridazinethiols* (see Section 6.1.2.2).

Of the remaining routes, *alkanethiolysis of nitropyridazines* has been used with some success: for example, treatment of 5-acetyl-2-methyl-4-nitro- (**69**, R = $NO_2$) with aqueous ethanolic sodium methanethiolate at 25° for 30 min gave 5-acetyl-2-methyl-4-methylthio-6-phenyl-3(2*H*)-pyridazinone (**69**, R =

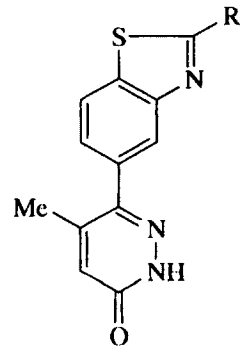
SMe) in 48% yield;[1766] 3-chloro-6-methoxy-5-nitro- (**70**, R = $NO_2$) with sodium ethanethiolate in refluxing ethanol for 3 h afforded 3-chloro-5-ethylthio-6-methoxypyridazine 2-oxide (**70**, R = SEt) in 35% yield;[905] and other such displacements have been reported.[809] In addition, there is an example of *arenethiolysis of an (extranuclear) alkylsulfonylpyridazine*: 5-methyl-6-(2-methylsulfonylbenzothiazol-5-yl)- (**71**, R = $SO_2Me$) with methanolic sodium benzenethiolate at 25° for 1 h afforded 5-methyl-6-(2-phenylthiobenzothiazol-5-yl)-4,5-dihydro-3(2*H*)-pyridazinone (**71**, R = SPh) in 62% yield.[1449] The *photolysis* of 2-*t*-butyl-4-chloro-5-trimethylsilylmethylthio- to 2-*t*-butyl-4-chloro-5-methylthio-3(2*H*)-pyridazinone has been reported.[2051]



(69)



(70)



(71)

## 6.2.2 Reactions of Alkylthiopyridazines (*H* 781)

Several reactions of alkylthiopyridazines have been covered already: *hydrolysis of pyridazinones* (see Section 5.1.1), *alcoholysis to alkoxypyridazines* (see Section 5.4.1), *de-S-alkylation to pyridazinethiones* (see Section 6.1.1), and *rearrangement of S-alkenyl groups* (see Section 6.1.2.2). Other reactions are discussed in the following subsections.

### *6.2.2.1 Oxidation to Sulfoxides or Sulfones (H 785)*

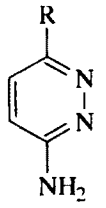
Although alkylthio substituents are reasonably good leaving groups for nucleophilic displacements, the easily derived alkylsulfinyl- or alkylsulfonyl substituents are even better for this purpose. Hence the oxidation of alkylthiopyridazines to corresponding sulfoxides or sulfones is well represented in recent literature. Typical conditions and yields are illustrated by the following examples:

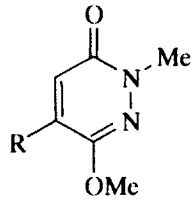
Methyl 6-ethylthio- gave methyl 6-ethylsulfonyl-3-pyridazinecarboxylate ($KMnO_4$, AcOH–PhH, trace, $Bu_4NBr$, 25°, 18 h: 71%).[1592]

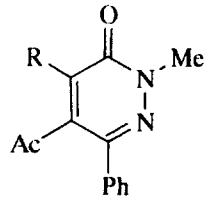
6-Phenylthio- (**72**, R = SPh) gave 6-phenylsulfinyl- (**72**, R = SOPh) [*m*-$ClC_6H_4CO_3H$ (1 equiv), $CH_2Cl_2$, 25°, 24 h: 82%] or 6-phenylsulfonyl-3-

pyridazinamine (**72**, R = $SO_2Ph$) [*m*-$ClC_6H_4CO_3H$ (2.5 equvi), likewise: 44% after separation from some sulfoxide].[1693]

6-Methoxy-2-methyl-5-phenylthio- (**73**, R = SPh) gave 6-methoxy-2-methyl-5-phenylsulfinyl- (**73**, R = SOPh) [AcOH, 70% $H_2O_2$ (~1 equiv), 50°, 3 h: 95%] or 6-methoxy-2-methyl-5-phenylsulfonyl-3(2*H*)-pyridazinone (**73**, R = $SO_2Ph$) (AcOH, excess $H_2O_2$, likewise: 53% after purification).[1672]


(72)


(73)


(74)

5-Acetyl-2-methyl-4-methylthio- (**74**, R = SMe) gave 5-acetyl-2-methyl-4-methylsulfinyl- (**74**, R = SOMe) (AcOH–$H_2O$, limited 30% $H_2O_2$, 15°, 18 h: 61%) or 5-acetyl-2-methyl-4-methylsulfonyl-6-phenyl-3(2*H*)-pyridazinone (**74**, R = $SO_2Me$) (AcOH–$H_2O$, excess 30% $H_2O_2$, 90°, 2 h: 73%).[1766]

3,6-Bismethylthiopyridazine gave 3-methylsulfinyl-6-methylthio- [AcOH, $H_2O_2$ (< 1 equiv), 40°, 2 h: 59%; or $CHCl_3$, $Br_2$: ?%], 3,6-bismethylsulfinyl- (AcOH, limited $H_2O_2$, 50°, 1 h: 54%), 3-methylsulfinyl-6-methylsulfonyl- [$CH_2Cl_2$, limited $HO_2CClC{=}CClCO_3H$ (made in situ), 25°, 1 h: 11% after purification], or 3,6-bismethylsulfonylpyridazine [$CH_2Cl_2$, excess $HO_2CClC{=}CClCO_3H$ (made in situ), 25°, 1 h: 64%; AcOH, excess $H_2O_2$, 50°, 30 min: 64%; or $Na_2WO_4{\cdot}2H_2O$, $H_2O_2$, 5°, 30 min: 89%].[304]

Also other examples.[300, 371, 533, 675, 733, 1028, 1116, 1449, 1755, 1774, 1903, 1974]

### *6.2.2.2 Miscellaneous Minor Reactions (H 781)*

Although some are quite important, the remaining reactions of alkylthiopyridazines must be considered as minor in that they have been used only rarely of recent years. They are illustrated by the following classified examples:

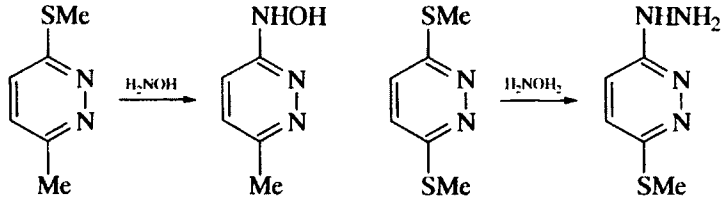
#### Aminolysis

*Note*: It is sometimes advantageous to oxidize alkylthio- to alkylsulfinyl- or alkylsulfonylpyridazines (see Section 6.2.2.1) prior to aminolysis (see Section 6.4).

3-Methyl-6-methylthiopyridazine (**75**) gave 3-hydroxyamino-6-methylpyridazine (**76**) ($H_2NOH$, EtOH, trace HCl, reflux, 9 h: 25%).[969]

3,6-Bismethylthiopyridazine (**77**) gave 3-hydrazino-6-methylthiopyridazine (**78**) (excess neat $H_2NNH_2$, 100°, 20 h; 77%).[636]

Also other examples.[1362,1390]



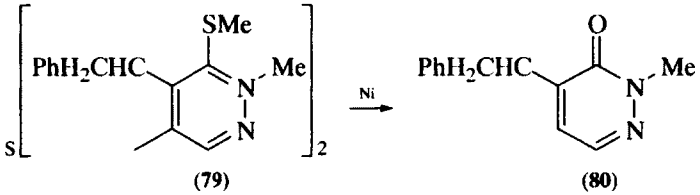

(**75**) (**76**) (**77**) (**78**)

## Desulfurization or Sulfur Extrusion

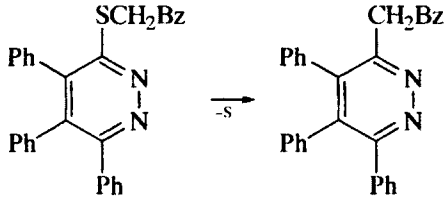
Bis(5-benzylamino-1-methyl-6-oxo-1,6-dihydropyridazin-4-yl) sulfide (**79**) gave 4-benzylamino-2-methyl-3(2*H*)-pyridazinone (**80**) (Raney Ni, $Me_2NCHO$, 150°, 10 h; 83%; a classical reductive desulfurization).[867]

3-Phenacylthio- (**81**) gave 3-phenacyl-4,5,6-triphenylpyridazine (**82**) (pyridine, reflux, 30 min: 55%; an extrusion).[1362]

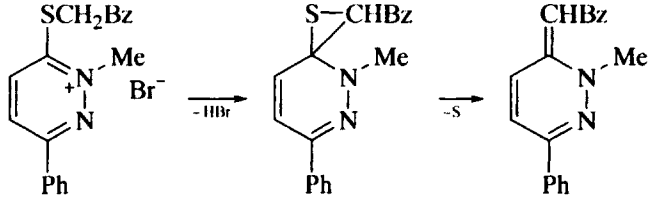
1-Methyl-6-phenacylthio-3-phenylpyridazinium bromide (**83**) gave 1-methyl-6-phenacylidene-3-phenyl-1,6-dihydropyridazine (**85**), presumably via the cyclic sulfide (**84**) ($K_2CO_3$, $Me_2NCHO$, 25°, 30 min: 60%); also analogues similarly.[413]




(**79**) (**80**)




(**81**) (**82**)

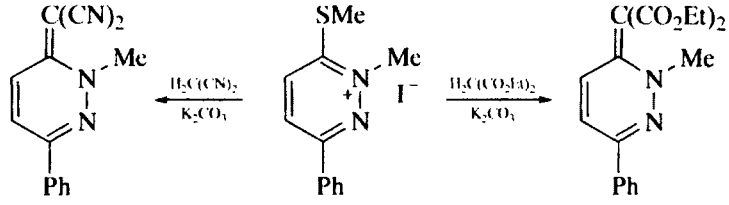



(**83**) (**84**) (**85**)

## Displacement of Alkylthio Groups by Carbanions

*Note*: This can be done provided the substrate is a quaternary salt.

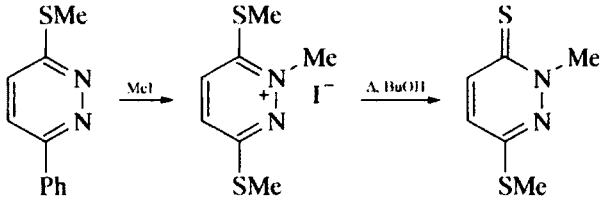
1-Methyl-6-methylthio-3-phenylpyridazinium iodide (**87**) gave 3-dicyanomethylene- (**86**) [$H_2C(CN)_2$, $K_2CO_3$, $Me_2NCHO$, 25°, ?h: 75%] or 3-diethoxycarbonylmethylene-2-methyl-6-phenyl-2,3-dihydropyridazine (**88**) [$H_2C(CO_2Et)_2$, likewise: 46%]; also related products similarly.[266]




(**86**) (**87**) (**88**)

## Hilbert–Johnson-Type Reactions

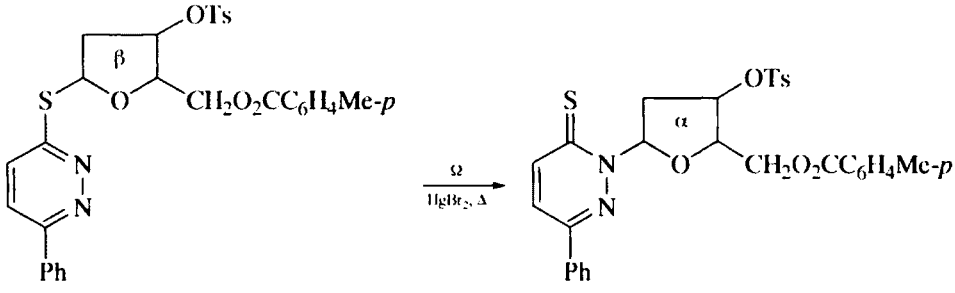
3,6-Bismethylthiopyridazine (**89**) gave 1-methyl-3,6-bismethylthiopyridazinium iodide (**90**) (MeI, MeOH, 20°, 6 days: ~65%) and then 2-methyl-6-methylthio-3(2*H*)-pyridazinethione (**91**) (BuOH, reflux, 4 h: ~15%); the 2-ethyl homologue was made similarly.[95]




(**89**) (**90**) (**91**)

## Thermal Rearrangements

3-Phenyl-6-(5-*O*-*p*-toluoyl-3-*O*-tosyl-2-deoxy-β-D-ribofuranosylthio)pyridazine (**92**) gave 6-phenyl-2-(5-*O*-*p*-toluoyl-3-*O*-tosyl-2-deoxy-α-D-ribofuranosyl)-3(2*H*)-pyridazinethione (**93**) ($HgBr_2$, PhH, reflux: >60%; note additional change from β- to α-anomer).[659]



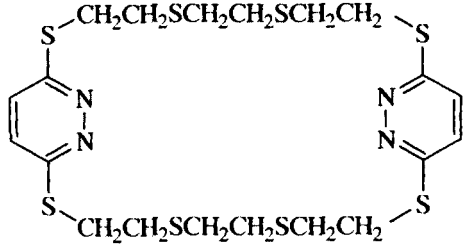

(**92**) (**93**)

### Cyclizations

Appropriate (substituted-alkyl)thio derivatives can undergo intramolecular cyclization. For examples, see Section 6.1.2.2 under *cyanomethylthio derivatives* and subsequent subsections.

### Formation of Metal Complexes

Several macrocyclic pyridazine thioethers [e.g., ligand (**94**)] have been converted into well-characterized copper and silver complexes.[1639, 1641 1657]



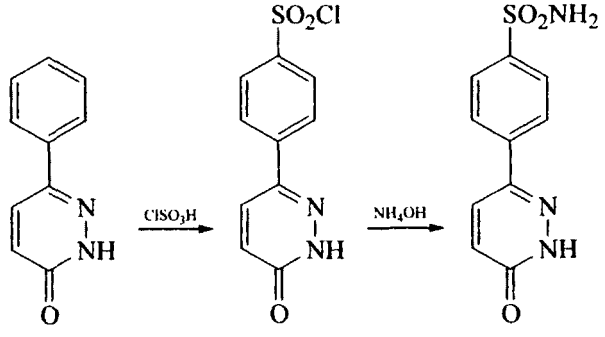
(94)

## 6.3 DIPYRIDAZINYL DISULFIDES AND PYRIDAZINESULFONIC ACIDS OR RELATED COMPOUNDS (*H* 788)

Of these oxidation products from pyridazinethiones or thiols, only the disulfides are represented in recent literature; the sulfenic, sulfinic, and sulfonic acids as well as their derivatives have been almost totally neglected.

The *preparation of disulfides*, done almost invariably by oxidation of tautomeric pyridazinethiones or thiols with air or iodine, has been discussed in Section 6.1.2.5. No *reactions of disulfides* have been reported recently.

Information on *pyridazinesulfonic acids* is confined to the preparation of two extranuclear derivatives. Treatment of 6-phenyl-3(2*H*)-pyridazinon (**95**) with chlorosulfonic acid, initially at 25° for 1 h and subsequently at 60° for



(95) (96) (97)

2 h, afforded crude 6-*p*-chlorosulfonylphenyl-3(2*H*)-pyridazinone (**96**), characterized through conversion into 6-*p*-sulfamoylphenyl-3(2*H*)-pyridazinone (**97**) (89% yield overall) by trituration with concentrated ammonia and ice for a few minutes.[919]

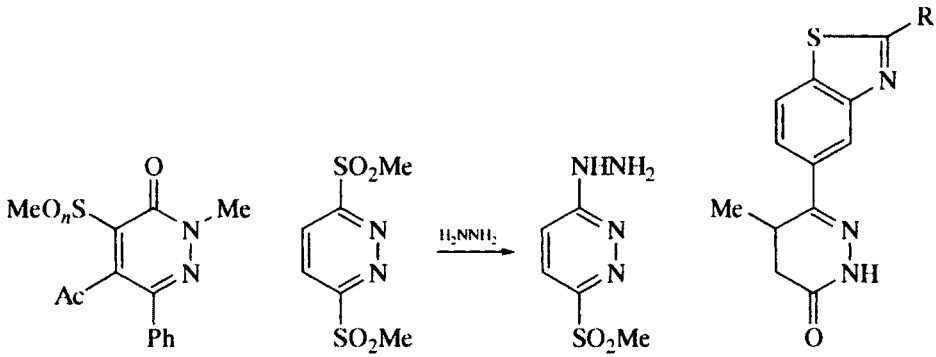
## 6.4 PYRIDAZINE SULFOXIDES AND SULFONES (*H* 477, 788)

The main preparative route to such pyridazine derivatives is by *oxidation of alkylthiopyridazines*, discussed in Section 6.2.2.1. Minor routes are represented in recent literature only by the *oxidation of alkylsulfinyl- to alkylsulfonylpyridazines*: thus 5-acetyl-2-methyl-4-methylsulfinyl- (**98**, $n = 1$) gave 5-acetyl-2-methyl-4-methylsulfonyl-6-phenyl-3(2*H*)-pyridazinone (**98**, $n = 2$) (AcOH, 30% $H_2O_2$, 90°, 1 h: 87%).[1766]

Only a few reactions of sulfoxides or sulfones have been reported recently. *Hydrolysis* is represented by several examples, outlined toward the beinging of Section 5.1.1; *alcoholysis*, by examples in Section 5.4.1; and *alkanethiolysis*, by examples in Section 6.2.1. *Aminolyses* are illustrated in the following examples:

3,6-Bismethylsulfonylpyridazine (**99**) gave 3-hydrazino-6-methylsulfonylpyridazine (**100**) ($H_2NNH_2$, EtOH, reflux, 2 h: 84%) or 3-methylsulfonyl-6-piperidinopyridazine [$HN(CH_2)_5$, $Me_2NCHO$, 60°, 2 h: 90%].[304]

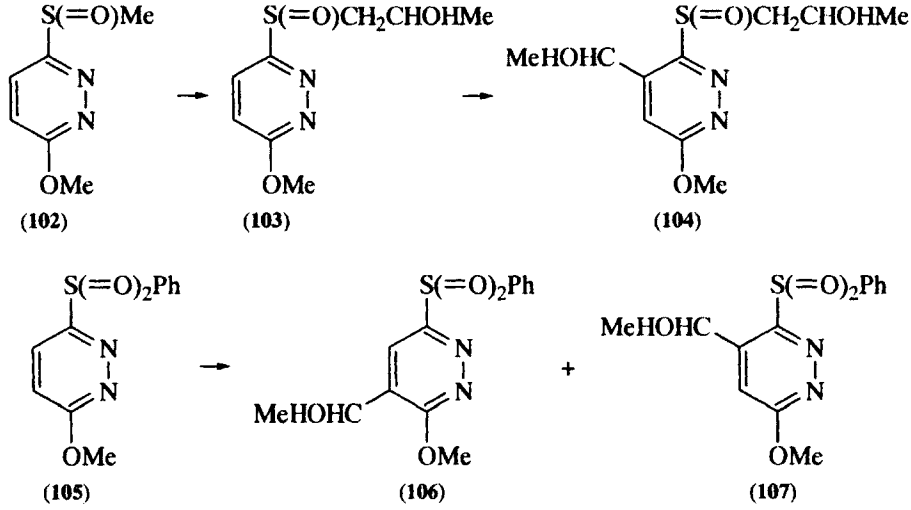
5-Methyl-6-(2-methylsulfonylbenzothiazol-5-yl)- (**101**, R = $SO_2Me$) gave 6-(2-β-aminoethylaminobenzothiazol-5-yl)-5-methyl-4,5-dihydro-3(2*H*)-pyridazinone (**101**, R = $NHCH_2CH_2NH_2$) (excess neat $H_2NCH_2CH_2NH_2$, 100°, 1 h: 87%).[1449]




(98) (99) (100) (101)

*Alkylations* of alkylsulsulfinyl- and alkylsulfonylpyridazines appear to occur first at the α carbon and subsequently at a ring carbon; alkylations of arylsulfinyl- and arylsulfonylpyridazines occur only at a ring carbon of the pyridazine nucleus. For example, mono- or dilithiation of 3-methoxy-6-methylsulfinylpyridazine (**102**) and subsequent treatment with acetaldehyde gave

mainly 3-(β-hydroxypropyl)sulfinyl-6-methoxypyridazine (**103**) or mainly 4-α-hydroxyethyl-3-(β-hydroxypropyl)sulfinyl-6-methoxypyridazine (**104**), respectively; in contrast, monoalkylation of 3-methoxy-6-phenylsulfonylpyridazine (**105**) gave a mixture of 4-α-hydroxyethyl-3-methoxy-6-phenylsulfonylpyridazine (**106**) and 4-α-hydroxyethyl-6-methoxy-3-phenylsulfonylpyridazine (**107**).[2035]




*Resolution* of 5-acetyl-2-methyl-4-methylsulfinyl-6-phenyl-3(2*H*)-pyridazinone proved possible by virtue of its sulfur stereogenic center. Chromatography on an appropriate Chiralcel or related column afforded the entiomers: that of (−)(*S*) configuration was confirmed in structure by X-ray analysis.[2075]

*Chemistry of Heterocyclic Compounds, Volume 57*
D. J. Brown
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CHAPTER 7

# Nitro-, Amino-, and Related Pyridazines (*H* 463, 629)

This chapter covers pyridazines bearing nitrogenous substituents that are joined directly or indirectly to the nucleus through their nitrogen atom; exceptionally, isocyanato- and isothiocyanatopyridazines are relegated to Chapter 8 in order to be included close to pyridazinecarbonitriles and the like.

## 7.1 NITROPYRIDAZINES (*H* 629)

The nuclear mono- and dinitropyridazines as well as various extranuclear nitropyridazines are important intermediates for reduction to the corresponding pyridazinamines. Some nuclear nitropyridazines also undergo nucleophilic displacement of their nitro groups to afford a variety of useful pyridazines.

### 7.1.1 Preparation of Nitropyridazines (*H* 629)

The majority of nitropyridazines have been made by *primary syntheses*, covered in Chapters 1 and 2.

A less predictable approach to both nuclear and extranuclear nitropyridazines involves *direct nitration* of existing pyridazines, as illustrated in the following examples:

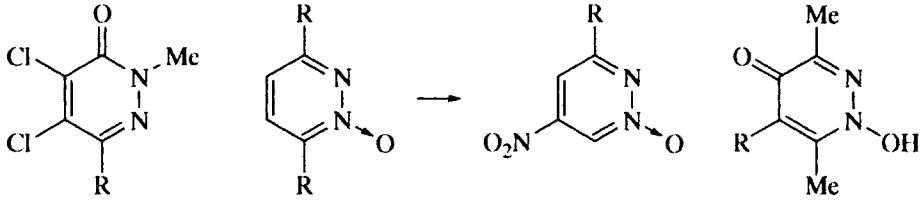
4,5-Dichloro-2-methyl-3(2*H*)-pyridazinone (**1**, R = H) gave 4,5-dichloro-2-methyl-6-nitro-3(2*H*)-pyridazinone (**1**, R = $NO_2$) [$KNO_3$, 95% $H_2SO_4$, reflux(!), 4.5 h: 64%].[1938]

3-Chloro- (**2**, R = Cl) or 3-methoxy-6-methylpyridazine 1-oxide (**2**, R = OMe) gave 3-chloro-6-methyl-5-nitro- (**3**, R = Cl) or 3-methoxy-6-methyl-5-nitropyridazine 1-oxide (**3**, R = OMe), respectively (BzCl, $CHCl_3$, $AgNO_3\downarrow$, − 10°, 4 h; then 25°, 4 days: 32% or 43%, respectively, after separation from unchanged substrate).[904]

1-Hydroxy-3,6-dimethyl-4(1*H*)-pyridazinone (**4**, R = H) gave 1-hydroxy-3,6-dimethyl-5-nitro-4(1*H*)-pyridazinone (**4**, R = $NO_2$) (95% $H_2SO_4$, 98% $HNO_3$, 5° → 25°, 12 h: 67%).[794]

3-Ethoxypyridazine 1-oxide gave a separable mixture of 3-ethoxy-4-nitro- and 3-ethoxy-4,6-dinitropyridazine 1-oxide (95% $H_2SO_4$, 98% $HNO_3$, 60°, 4 h; then 75°, 4 h: 14% and 25%, respectively).[757]

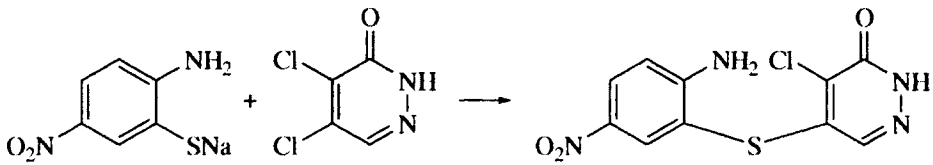
6-Benzyloxy-3(2*H*)-pyridazinone gave 6-*p*-nitrobenzyloxy-3(2*H*)-pyridazinone [nitration (for conditions, see original): 82%]; 2-*p*-nitrobenzyl-3,6(1*H*,2*H*)-pyridazinedione (94%), likewise.[1453]



(1) (2) (3) (4)

Also other examples.[527,534,833,902]

Extranuclear nitropyridazines may also be made by the introduction of *passenger nitro groups* in a variety of ways. For example, sodium 2-amino-5-nitrobenzenethiolate (**5**) (made in situ) reacted with ethanolic 4,5-dichloro-3(2*H*)-pyridazinone (**6**) at 25° to afford 5-(2-amino-5-nitrophenylthio)-4-chloro-3(2*H*)-pyridazinone (**7**) in excellent yield;[549] and 1,2-dimethylhexahydro-4-pyridazinamine underwent acylation by *p*-nitrobenzoyl chloride in chloroform at 25° during 12 h to afford 1,2-dimethyl-4-*p*-nitrobenzamidohexahydropyridazine in 67% yield.[1699]



(5) (6) (7)

## 7.1.2 Reactions of Nitropyridazines (*H* 631)

The most important reaction of nitropyridazines is their reduction to pyridazinamines. In addition, some nitropyridazines undergo diverse displacement reactions, in which the nitro substituent acts as a leaving group

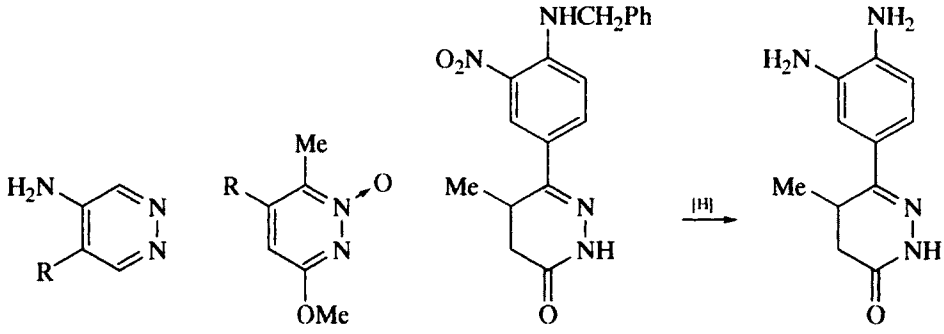
### *7.1.2.1 Reduction (H 478)*

The reduction of both nuclear and extranuclear nitropyridazines can be done with a variety of reagents, as illustrated in the following examples:

## Hydrogenation over Palladium

5-Nitro-4-pyridazinamine (**8**, $R = NO_2$) gave 4,5-pyridazinediamine (**8**, $R = NH_2$) ($H_2$, Pd/C, EtOH, 30 min: 57%).[1080]

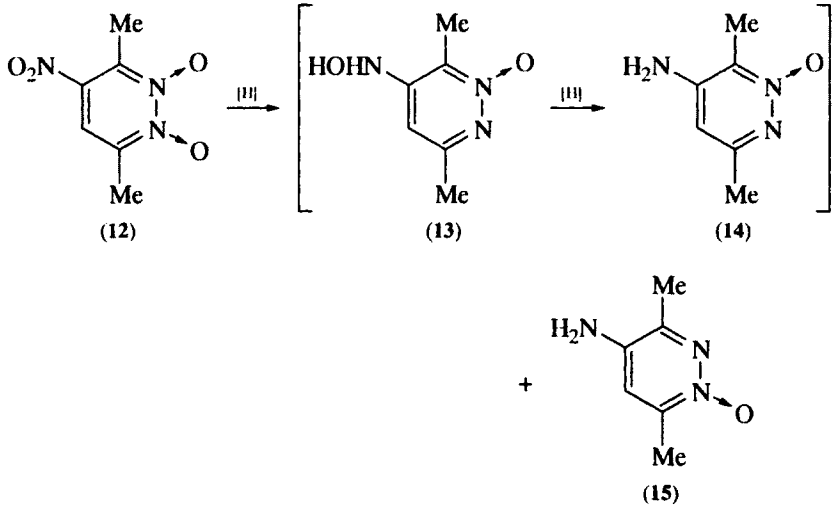
3-Methoxy-6-methyl-5-nitropyridazine 1-oxide (**9**, $R = NO_2$) gave 6-methoxy-3-methyl-4-pyridazinamine 2-oxide (**9**, $R = NH_2$) ($H_2$, Pd/C, EtOH; 86%).[904]




6-(4-Benzylamino-3-nitrophenyl)- (**10**) gave 6-(3,4-diaminophenyl)-5-methyl-4,5-dihydro-3(2*H*)-pyridazinone (**11**) ($H_2$, Pd/C, 1M HCl–MeOH, 2 h: 89%; note additional hydrogenolysis of the benzyl group).[1762]

3,6-Dimethyl-4-nitropyridazine 1,2-dioxide (**12**) gave a separable mixture of 3,6-dimethyl-4-pyridazinamine 1-oxide (**15**) and the isomeric 2-oxide (**14**) [$H_2$ (4 equiv), Pd/C, EtOH, 25°: ~45% and 42%, respectively] or only 4-hydroxyamino-3,6-dimethylpyridazine 2-oxide (**13**), [$H_2$, (3 equiv), likewise: 38%, after separation from unisolable products], which afforded the pyridazinamine (**14**) (94%) on rehydrogenation.[796]

Also other examples.[794, 902, 904, 1699, 1769]



### Hydrogenation over Raney Nickel

*Note*: Raney nickel is particularly useful when hydrogenolysis of a passenger halogeno substituent is to be avoided.

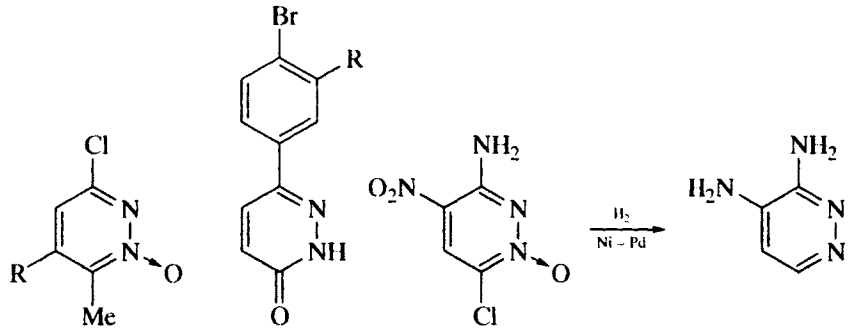
3-Chloro-6-methyl-5-nitropyridazine 1-oxide (**16**, R = $NO_2$) gave 6-chloro-3-methyl-4-pyridazinamine 2-oxide (**16**, R = $NH_2$) ($H_2$, Raney Ni, EtOH, 25° → 50°: ~2 h: 92%).[904]

6-(4-Bromo-3-nitrophenyl)- (**17**, R = $NO_2$) gave 6-(3-amino-4-bromophenyl)-3(2*H*)-pyridazinone (**17**, R = $NH_2$) [$H_2$ (75 atm), Raney Ni, MeOH, 50°, 78%].[919]

2-*o*-Nitrophenyl- gave 2-*o*-aminophenyl-6-phenyl-3(2*H*)-pyridazinone ($H_2$, Raney Ni, THF–EtOH, 25°, 20 min: 79%).[1069]

6-Chloro-4-nitro-3-pyridazinamine 1-oxide (**18**) gave 3,4-pyridazinediamine (**19**) ($H_2$, Raney Ni–Pd/C, MeOH, 25°, 15 h: 51% as the hydrochloride; note reduction, dechlorination, and deoxygenation over the mixed catalysts).[449]

Also other examples.[1021]




(**16**) (**17**) (**18**) (**19**)

### Stannous Chloride Reductions

4-Chloro-5-*o*-nitrophenylthio- (**20**, R = $NO_2$) gave 5-*o*-aminophenylthio-4-chloro-2-phenyl-3(2*H*)-pyridazinone (**20**, R = $NH_2$) ($SnCl_2 \cdot 2H_2O$, 10M HCl, 65°, 3 h: ~40%).[768]

Also other examples.[539,792,838]

### Iron Reductions

4,5-Dichloro-2-methyl-6-nitro- (**21**, R = $NO_2$) gave 6-amino-4,5-dichloro-2-methyl-3(2*H*)-pyridazinone (**21**, R = $NH_2$) (Fe powder, AcOH, reflux, 2 h: 55%;[846] Fe powder, $NH_4Cl$, $H_2O$–$CHCl_3$, 25°, 20 h: 93%).[1938]

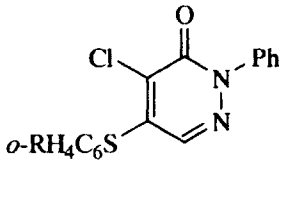
Also other examples.[1453]

**Miscellaneous Methods**

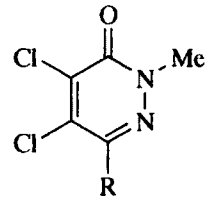
4-Nitro- (**22**, R = $NO_2$) gave 4-amino-5,6-diphenyl-3(2*H*)-pyridazinone (**22**, R = $NH_2$) ($Na_2S_2O_4$; for details, see original paper).[809]

3-Chloro-6-methoxy-5-nitropyridazine 2-oxide (**23**, R = $NO_2$) gave a separable mixture of the expected 4-azido-6-chloro-3-methoxypyridazine 1-oxide (**23**, R = $N_3$) and 6-chloro-3-methoxy-4-pyridazinamine 1-oxide (**23**, R = $NH_2$) ($NaN_3$, $H_2O$–EtOH, reflux, 4 h: 23% and 40%, respectively).[905]

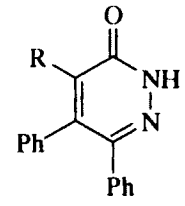
4-Amino-2-methyl-5-nitro- gave 4,5-diamino-2-methyl-3(2*H*)-pyridazinone ($H_2NNH_2 \cdot H_2O$, Pd/C, EtOH, reflux, 3 h: 80%).[1206]



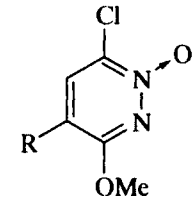
(**20**)



(**21**)



(**22**)



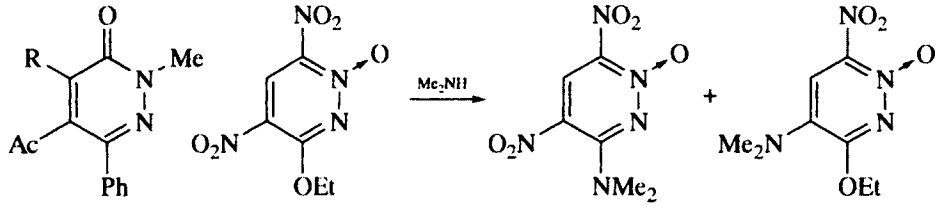
(**23**)

## *7.1.2.2 Displacement Reactions*

Several such displacement of nitro groups have been covered already: direct *conversion into halogenopyridazines* (Section 4.1.4), *hydrolysis to pyridazinones* (Section 5.1.1), *alcoholysis to alkoxypyridazines* (Section 5.4.1), *thiolysis to pyridazinethiones* (Section 6.1.1), and alkanethiolysis to alkylthiopyridazines (Section 6.2.1). The remaining displacement are illustrated in the following examples:

**Aminolysis to Pyridazinamines**

5-Acetyl-2-methyl-4-nitro-6-phenyl-3(2*H*)-pyridazinone (**24**, R = $NO_2$) gave 5-acetyl-4-(*N*-ethoxycarbonylmethyl-*N*-methylamino)- (**24**, R = $NMeCH_2CO_2Et$) ($MeHNCH_2CO_2Et$, EtOH, 25°, 30 min: 76%),[1561] 5-acetyl-4-(*N*-β-cyanoethyl-*N*-methylamino)- (**24**, R = $NMeCH_2CH_2CN$) ($MeHNCH_2CH_2CN$, likewise: 83%),[1561] or 5-acetyl-4-anilino-2-methyl-6-phenyl-3(2*H*)-pyridazinone (**24**, R = NHPh) ($PhNH_2$, likewise: 59%).[1769]



(**24**) (**25**) (**26**) (**27**)

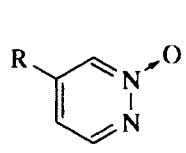
3-Ethoxy-4,6-dinitro- (**25**) gave a separable mixture of the expected product, 3-dimethylamino-4,6-dinitro- (**26**) and 4-dimethylamino-3-ethoxy-6-nitropyridazine 1-oxide (**27**) ($Me_2NH$, MeOH–$H_2O$, 25°, 1 h: 10% and 19%, respectively, after separation).[760]

Also other examples.[809,905,1414]

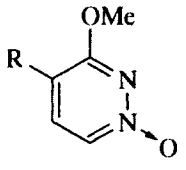
### Conversion into Azidopyridazines

4-Nitro- (**28**, R = $NO_2$) gave 4-azidopyridazine 1-oxide (**28**, R = $N_3$) ($NaN_3$, EtOH–$H_2O$, 95°, 5 h: 88%).[831]

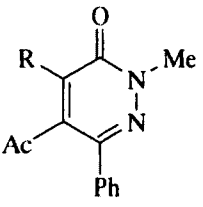
3-Methoxy-4-nitro- (**29**, R = $NO_2$) gave 4-azido-3-methoxypyridazine 1-oxide (**29**, R = $N_3$) ($NaN_3$, EtOH–$H_2O$, reflux, 5 h: 93%); also homologues likewise.[1443]




(28)




(29)




(30)

### Reductive Displacement

5-Acetyl-2-methyl-4-nitro-6-phenyl-3(2*H*)-pyridazinone (**30**, R = $NO_2$) has been reported to afford 5-acetyl-2-methyl-6-phenyl-3(2*H*)-pyridazinone (**30**, R = H) ($NaBH_4$, MeOH, cooled, 30 min: 34%).[1769]

## 7.2 REGULAR AMINOPYRIDAZINES (*H* 463)

This section covers primary, secondary, tertiary, and quaternary aminopyridazines (both nuclear and extranuclear) but not the (functionally substituted-amino)pyridazines such as hydrazino- or hydroxyaminopyridazines.

### 7.2.1 Preparation of Regular Aminopyridazines (*H* 464, 507)

Some synthetic routes to regular aminopyridazines have been discussed already as indicated in the following list, which includes the potential scope of each method:

By *primary synthesis* (nuclear, extranuclear: primary, secondary, and tertiary): Chapters 1 and 2

By *Mannich aminoalkylation* (extranuclear: secondary and tertiary): Section 3.2.2.2

By *aminolysis of halogenopyridazines* (nuclear, extranuclear: primary, secondary, tertiary, and quaternary): Sections 4.2.1 and 4.4.1

By *aminolysis of tautomeric pyridazinones* (nuclear: primary, secondary, and tertiary): Section 5.1.2.5

By *N-amination of tautomeric pyridazinones* (nuclear: primary): Section 5.1.2.5

By *aminolysis of alkoxypyridazines* (nuclear, extranuclear: primary, secondary, and tertiary): Section 5.4.2.1

By *aminolysis of tautomeric pyridzinethiones* (nuclear: primary, secondary, and tertiary but usually done indirectly): Section 6.1.2.4

By *aminolysis of alkylthio-, alkylsulfinyl-, or alkylsulfonylpyridazines* (nuclear: primary, secondary, or tertiary): Sections 6.2.2.2 and 6.4

By *reduction of nitropyridazines* (nuclear, extranuclear: primary): Section 7.1.2.1

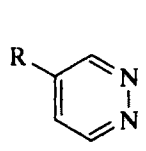
By *aminolysis of nitropyridazines* (nuclear, primary, secondary, and tertiary): Section 7.1.2.2

The remaining methods for the preparation of regular aminopyridazines (and some hydropyridazines) are illustrated by the following classified examples with explanatory notes as required:

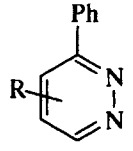
### By *C*-Amination

*Note*: For primary 4/5-aminopyridazines only.

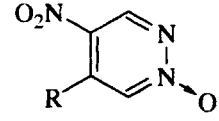
Pyridazine (**31**, R = H) gave only 4-pyridazinamine (**31**, R = $NH_2$) ($NaNH_2$, liquid $NH_3$, −70°, 10 min; then $KMnO_4$, −70°, 10 min: 91%; proceeding by 4,5 addition of $NH_3$ followed by oxidative dehydrogenation);[1012] in contrast, similar treatment of 3-phenylpyridazine (**32**, R = H) gave a separable mixture (**32**, R = $NH_2$) of 3-phenyl-4- (49%), 6-phenyl-4- (18%), and 6-phenyl-3-pyridazinamine (5%).[1012]




(31)



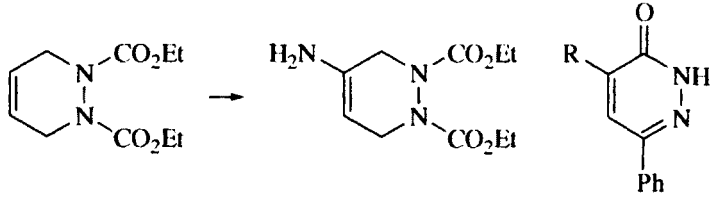

(32)




(33)

4-Nitropyridazine 1-oxide (**33**, R = H) gave 5-nitro-4-pyridazinamine 2-oxide (**33**, R = $NH_2$) (liquid $NH_3$, $KMnO_4$, −70°, 15 min: 75%);[1072] also analogues likewise.[1072, 1080]

Diethyl 1,2,3,6-tetrahydro-1,2-pyridazinedicarboxylate (**34**) gave diethyl 4-aminohexahydro-1,2-pyridazinedicarboxylate (**35**) ($BH_3$, $H_2NOSO_3H$, THF, reflux, 3 h: 10%; or $BH_3$, THF, 0°, 2 h, then 50°, 3 h, then $NH_4OH$ ↓, 0° → 25°, 3 h: 19%).[1699]



(34) (35) (36)

6-Phenyl-3(2*H*)-pyridazinone (**36**, R = $NH_2$) (neat $H_2NNH_2 \cdot H_2O$, reflux, 7 h: 66%);[1585] also many analogous reactions.[213,1585,1597] (See also Section 7.3.1.)

Also other examples.[449,820,1073,1873]

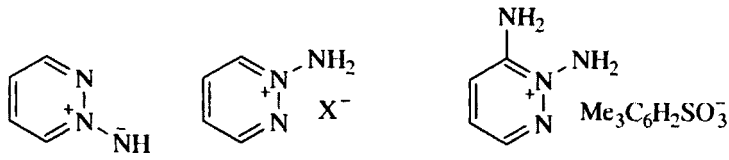
### By *N*-Amination

*Note*: For nuclear *N*-aminopyridazinium salts only; the derived zwitterionic pyridazinium *N*-imides (e.g., **37**), are highly unstable but can undergo cycloadditions and similar reactions.[477]

Pyridazine gave 1-aminopyridazinium chloride (**38**, X = Cl) ($H_2NOSO_3K$, $H_2O$, 70°, 4.5 h; then KCl↓: 71%),[756] the corresponding iodide (**38**, X = I) (likewise but KI↓: 65%),[756] the mesitylenesulfonate (**38**, X = 2,4,6-$Me_3C_6H_2SO_3^-$) (2,4,6-$Me_3C_6H_2SO_2ONH_2$, $CH_2Cl_2$, 0° → 25°, 30 min: 85%),[832] or the nitrate (**38**, X = $NO_3$) [$H_2NOSO_3H$, $Ba(NO_3)_2$, BaO, $H_2O$, exothermic: 34%].[1380]

3-Pyridazinamine gave 2,3-diaminopyridazinium mesitylsulfonate (**39**) (2,4,6-$Me_3C_6H_2SO_2ONH_2$, MeOH or MeOH–$CHCl_3$, 0° → 25°, 15 min: 60% or 72%, respectively).[412,929]

Also other examples in the foregoing references and elsewhere.[782,1359]



(37) (38) (39)

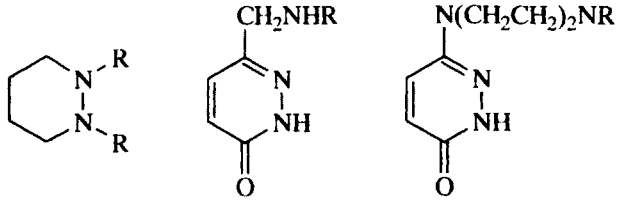
### From Acylaminopyridazines

*Note*: For any primary or secondary *C*-aminopyridazine.

1,2-Diacetylhexahydropyridazine (**40**, R = Ac) gave hexahydropyridazine (**40**, R = H) (hydrolysis: 27%; for details, see original paper).[633]

6-Benzamidomethyl- (**41**, R = Bz) gave 6-aminomethyl-3(2*H*)-pyridazinone (**41**, R = H) (6M HCl, 95°, 16 h: 94%).[731]

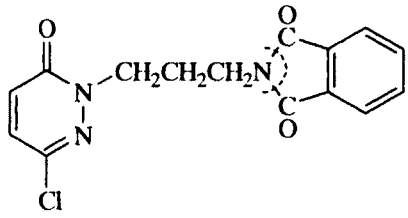
6-(4-Formylpiperazin-1-yl)- (**42**, R = CHO) gave 6-piperazin-1′-yl-3(2*H*)-pyridazinone (**42**, R = H) (HCl, EtOH–$H_2O$, reflux, 12 h: 40%).[1761]



(40) (41) (42)

6-Chloro-2-γ-phthalimidopropyl-3(2*H*)-pyridazinone (**43**) gave 2-γ-aminopropyl-6-chloro-3(2*H*)-pyridazinone ($H_2NNH_2 \cdot H_2O$, EtOH, 50°, 2 h: 38%, as hydrochloride).[1760]

Also other examples.[89,919,1086,1289,1337]



(43)

## From Hydrazino- or Hydroxyaminopyridazines

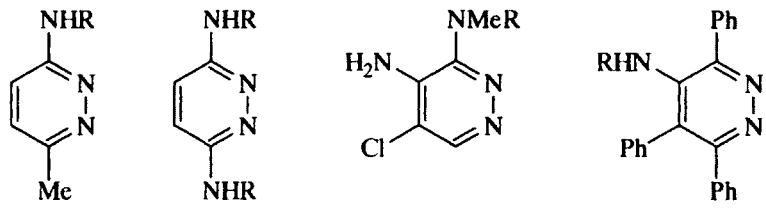
*Note*: For any primary or secondary *C*-aminopyridazine.

3-Hydrazino-6-methylpyridazine (**44**, R = $NH_2$) gave 6-methyl-3-pyridazinamine (**44**, R = H) ($H_2$, Raney-Ni, EtOH–$H_2O$, 20°: ~60%).[1037,1127]

3-Hydrazino-4-methyl-6-phenylpyridazine gave 4-methyl-6-phenyl-3-pyridazinamine ($H_2$, Raney Ni, MeOH, 20°: 60%;[1118] NiAl alloy, KOH, MeOH–$H_2O$, 25°: 76%).[1381]

3,6-Dihydrazinopyridazine (**45**, R = $NH_2$) gave 3,6-pyridazinediamine (**45**, R = H) ($H_2$, Raney Ni, $H_2O$, 25°, 5 h: ~65%).[675]

5-Chloro-3–α-methylhydrazino-4-pyridazinamine (**46**, R = $NH_2$) gave 5-chloro-3-methylamino-4-pyridazinamine (**46**, R = H) ($H_2$, Raney Ni, EtOH, 25°: 68%).[908]



(44) (45) (46) (47)

4-Hydroxyamino-3,5,6-triphenylpyridazine (**47**, R = OH) gave 3,5,6-triphenyl-4-pyridazinamine (**47**, R = H) (Al or $H_2NNH_2$ reduction: for details, see original paper).[588]

Also other examples.[116,1136,1138,1161,1176,1309,1732,1746]

### By Transamination

*Note*: For any primary, secondary, or tetriary *C*-aminopyridazine but very neglected.

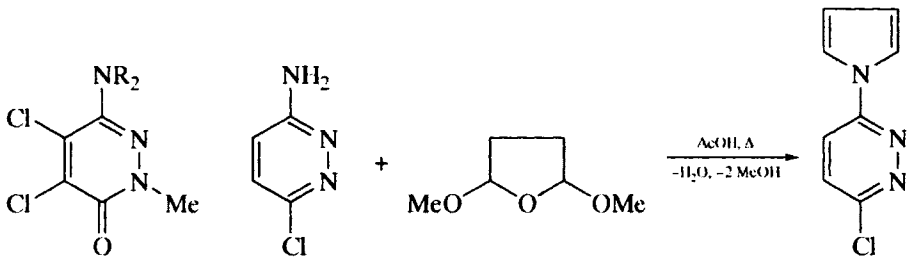
6-Chloro-3-pyridazinamine gave 3-benzylamino-6-chloropyridazine (neat $PhCH_2NH_2$, reflux, 3 h: ~10%; not survival of Cl).[675]

### By *N*-Alkylation or *N*-Dealkylation

*Note*: Applicable to any primary, secondary, tertiary, or quaternary aminopyridazine but usually difficult in practice, especially in the presence of tautomeric passenger substituents. See also Section 7.2.2.3.

6-Amino-4,5-dichloro- (**48**, R = H) gave 4,5-dichloro-6-dimethylamino-2-methyl-3(2*H*)-pyridazinone (**48**, R = Me) ($Me_2SO_4$, $K_2CO_3$, $CHCl_3$, reflux, 23 h: 68%).[1938]

6-Chloro-3-pyridazinamine (**49**) and 4,5-dimethoxytetrahydrofuran (**50**) gave 3-chloro-6-pyrrol-1′-ylpyridazine (**51**) (AcOH, 120°, sealed(?), 1 h: 51%).[1283]




(**48**) (**49**) (**50**) (**51**)

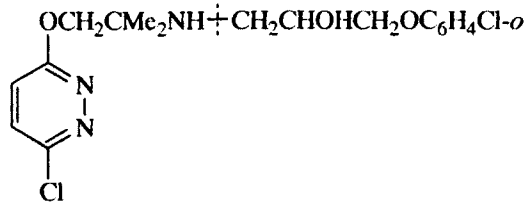
6-Amino-3(2*H*)-pyridazinone and dimethyl acetylenedicarboxylate ($MeO_2CC{\equiv}CCO_2Me$) gave 6-(α,β-dimethoxycarbonylvinylamino)-3(2*H*)-pyridazinone (**52**) (MeOH, reflux, 10 h: 55%).[1085]

3-(β-Amino-β-methylpropoxy)-6-chloropyridazine and 3-*o*-chlorophenoxy-1,2-epoxypropane gave 3-chloro-6-{β-[γ-(*o*-chlorophenoxy)-β-hydroxypropylamino]-β-methylpropoxy}pyridazine (**53**) (Bu′OH, 60°, 12 h: 72%, as hydrochloride).[1813]

6-(4-Benzylamino-3-nitrophenyl)- (**10**) gave 6-(3,4-diaminophenyl)-5-methyl-4,5-dihydro-3(2*H*)-pyridazinone (**11**) (by hydrogenolysis: see Section 7.1.2.1).[1762]



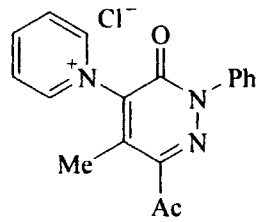
(52)



(53)

6-Acetyl-5-methyl-2-phenyl-4-pyridinio-3(2*H*)-pyridazinone chloride (**54**) gave 6-acetyl-4-amino-5-methyl-2-phenyl-3(2*H*)-pyridazinone [$(CH_2)_5NH$, EtOH, 60°, 30 min: 74%; by fission of the pyridine ring!].[1789]

Also other examples.[1157, 1688, 1700, 1741, 1905, 1978]



(54)

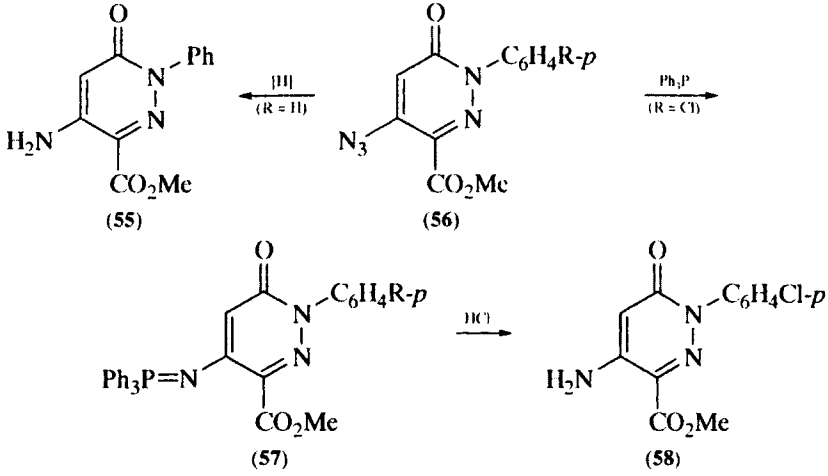
## From Azidopyridazines

*Note*: The conversion of any azidopyridazine into the corresponding primary aminopyridazine may be done by direct reduction or via a triphenylphosphoranylideneamino intermediate.

Methyl 4-azido- (**56**, R = H) gave methyl 4-amino-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate (**55**) ($H_2$, Pd/C, AcOH, 60°: 89%);[1117] in contrast, perhaps to avoid possible dechlorination, methyl 4-azido-1-*p*-chlorophenyl-6-oxo-1,6-dihydro-3-pyridazinecarboxylate (**56**, R = Cl) was converted into methyl 1-*p*-chlorophenyl-6-oxo-4-triphenylphosphoranylideneamino-1,6-dihydro-3-pyridazinecarboxylate (**57**) ($Ph_3P$, PhH, 25°, 30 min: 66%) and thence into methyl 4-amino-1-*p*-chlorophenyl-6-oxo-1,6-dihydro-3-pyridazinecarboxylate (**58**) (HCl, AcMe: 89%).[1117]

3,6-Diazidopyridazine (**59**, $R = N_3$) gave 6-azido-3-pyridazinamine (**59**, $R = NH_2$) [$MoCl_3$ (made in situ), THF, 15 min: 66%].[116]

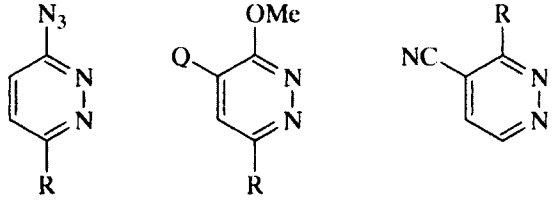
4-Azido-6-chloro-3-methoxypyridazine (**60**, $Q = N_3$, R = Cl) gave 6-chloro-3-methoxy-4-pyridazinamine (**60**, $Q = NH_2$, R = Cl) [$(CH_2)_5NH$, MeOH, $H_2S\downarrow$, 10°, 30 min: 40%] or 3-methoxy-4-pyridazinamine (**60**, $Q = NH_2$, R = H) ($H_2$, Pd/C, EtOH, 2 h: 84%; note additional dechlorination).[1990]




4-Azido- has been reported to give 4-amino-6-chloro-2-phenyl-3(2*H*)-pyridazinone (PhMe, reflux, 4 h: ~70%; mechanism unclear).[401]

3-Azido- (**61**, R = $N_3$) gave 3-triphenylphosphoranylideneamino- (**61**, R = N:$PPh_3$) ($Ph_3P$, PhCl, reflux, 2 h: 88%) and then 3-amino-4-pyridazinecarbonitrile (**61**, R = $NH_2$) (80% AcOH, reflux, 20 min: 73%).[1079]

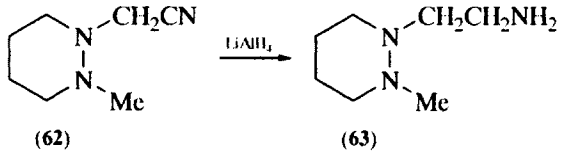
Also other examples, some using different reducing agents.[133,402,580,1355,1700,1884]




## By Reduction of Pyridazinecarbonitriles

*Note*: For extranuclear primary aminopyridazines only.

1-Cyanomethyl-2-methylhexahydropyridazine (**62**) gave 1-β-aminoethyl-2-methylhexahydropyridazine (**63**) ($LiAlH_4$, $Et_2O$, reflux, 12 h: 65%).[1699]

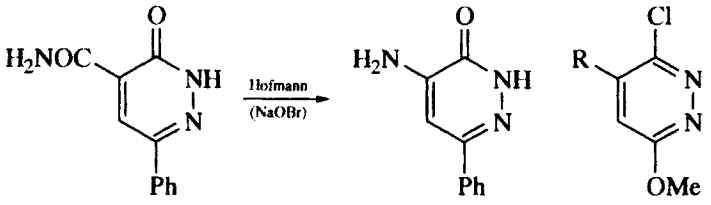



3-Anilino-4-pyridazinecarbonitrile gave 4-aminomethyl-3-anilinopyridazine ($H_2$, Pd/C, HCl–$H_2O$–EtOH, 2 h: 55%).[1079]

**By Hofmann-, Curtius-, or Schmidt-Type Reactions**

*Note*: For any primary aminopyridazines.

3-Oxo-6-phenyl-2,3-dihydro-4-pyridazinecarboxamide (**64**) gave 4-amino-6-phenyl-3(2*H*)-pyridazinone (**65**) ($Br_2$, NaOH, $H_2O$, 0°, 10 min; then substrate↓, 0° → 95°, 4 h: 80%).[1041]



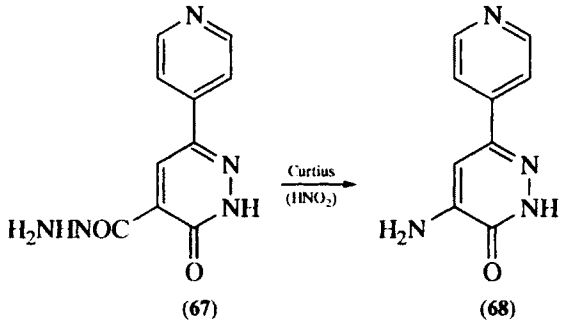

(64) (65) (66)

3-Chloro-6-methoxy-4-pyridazinecarboxamide (**66**, R = $CONH_2$) gave 3-chloro-6-methoxy-4-pyridazinamine (**66**, R = $NH_2$) (as preceeding example, 40 min: ~70%).[1099]

3-Oxo-6-pyridin-4′-yl-2,3-dihydro-4-pyridazinecarbohydrazide (**67**) gave 4-amino-6-pyridin-4′-yl-3(2*H*)-pyridazinone (**68**) ($NaNO_2$, 6M HCl, < 5° → 60°, 4 h: 63%).[1589]

5-Benzoyl-4-pyridazinecarbonyl azide (**69**) gave 5-benzoyl-4-pyridazinamine (**70**) (PhH, reflux, 4 h; then HCl↓, reflux, 2 h: 67%).[46]

Also other examples in the foregoing references and elsewhere.[958, 996, 1073]




(67) (68)

(69) (70)

## 7.2.2 Reactions of Regular Aminopyridazines (*H* 487)

Several reactions of regular aminopyridazines have been discussed already. For the conversion of *primary aminopyridazines into halogenopyridazines*, see

Section 4.1.6; for the conversion of *aminopyridazines into pyridazinones*, see Section 5.1.1; and for the *interconversion of aminopyridazines* by transamination, alkylation, or dealkylation, see Section 7.2.1.

The remaining reactions of aminopyridazines, both nuclear and extranuclear, are covered in the following subsections.

### *7.2.2.1 N-Acylation and Subsequent Reactions (H 487)*

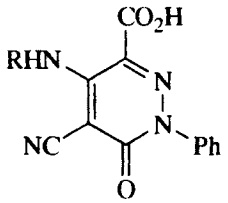
The *N*-acylation of an aminopyridazine or of a reduced-pyridazine (at its ring NH) may be undertaken simply to further characterize the amine but, especially when a functionally substituted acyl group is introduced, the reason may be for subsequent intramolecular cyclization or other elaboration(s). The following examples illustrate the process of acylation and a few typical cyclizations:

**Acylation of Nuclear Aminopyridazines**

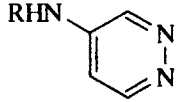
4-Amino- (**71**, R = H) gave 4-acetamido-5-cyano-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylic acid (**71**, R = Ac) ($Ac_2O$–AcOH, reflux, 2 h: 80%).[129]

4-Pyridazinamine (**72**, R = H) gave 4-pivalamidopyridazine (**72**, R = $COBu^t$ $Bu^tCOCl$, $Et_3N$, $CH_2Cl_2$, 0° → 25°, 1 h: 83%) or 4-*t*-butoxycarbonylaminopyridazine (**72**, R = $CO_2Bu^t$) [$O(CO_2Bu^t)_2$, THF, 25°, 90 min: >95%].[1609]

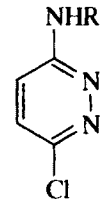
6-Chloro-3-pyridazinamine (**73**, R = H) gave 3-chloro-6-methoxycarbonylaminopyridazine (**73**, R = $CO_2Me$) ($ClCO_2Me$, $K_2CO_3$, THF, 25°, 18 h: 68%),[338] 3-chloro-6-formamidopyridazine (**73**, R = CHO) [neat $HC(NHCHO)_3$, 165°, sealed, 25 min: 90%;[198] or indirectly via 3-chloro-6-dimethylaminomethyleneaminopyridazine: ~50% overall],[295] or 3-chloro-6-dimesylaminopyridazine (**74**) ($MeSO_2Cl$, pyridine, 10° → 25°, 30 h: ?%), which then gave 3-chloro-6-mesylaminopyridazine (**73**, R = $SO_2Me$) (near polyphosphoric acid, 120°, 40 min: ?%).[89]



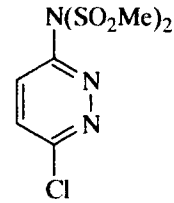
(71)



(72)

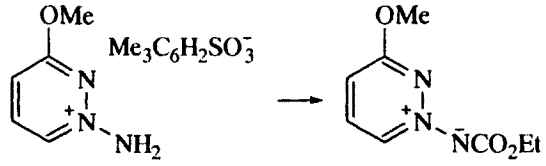


(73)



(74)

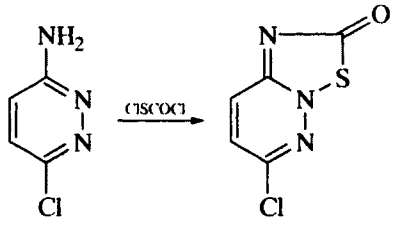
1-Amino-3-methoxypyridazinium mesitylenesulfonate (**75**) gave 3-methoxypyridazinium 1-ethoxycarbonylimide (**76**) ($ClCO_2Et$, $K_2CO_3$, EtOH, 25°, 20 h: 80%);[877] also analogues likewise.[832,877]



(75) (76)

6-Chloro-3-pyridazinamine (**77**) and chlorothioformyl chloride gave 6-chloro-2*H*-[1,2,4]thiadiazolo[2,3-*b*]pyridazin-2-one (**78**) ($Et_3N$, THF, 5°, 2.5 h; then reflux, 6 h: 37%; presumably by an initial *N*-acylation followed by cyclization).[300]

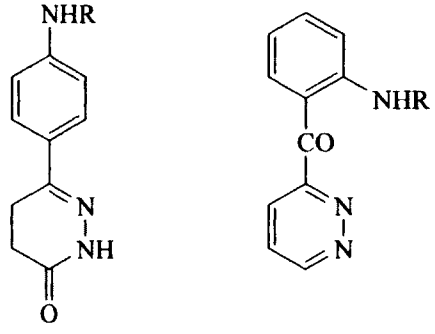
Also other examples.[42,206,545,602,768,911,1221,1700,1729,1815,2089,2119]



(77) (78)

## Acylation of Extranuclear Aminopyridazines

6-*p*-Aminophenyl- (**79**, R = H) gave 6-*p*-formamidophenyl- (**79**, R = CHO) 77% ($HCO_2H$, reflux, 1 h: 177)[1290] or 6-*p*-(3-bromopropionamido)-phenyl-4,5-dihydro-3(2*H*)-pyridazinone (**79**, R = $BrCH_2CH_2CO$) ($BrCH_2CH_2COCl$, PhMe, reflux, 16 h: 91%).[1313]



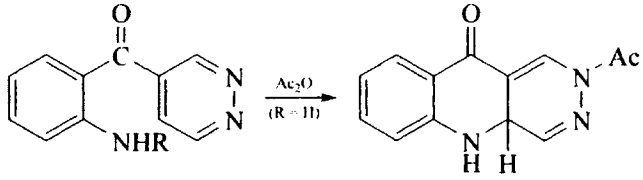
(79) (80)

3-*o*-Aminobenzoyl- (**80**, R = H) gave 3-*o*-acetamidobenzoylpyridazine (**80**, R = Ac) ($Ac_2O$, 100°, 20 min: 58%);[1628] however, similar treatment of the isomeric substrate, 4-*o*-aminobenzoylpyridazine (**81**, R = H), caused cyclization to 2-acetyl-2,4a-dihydropyridazino[4,5-*b*]quinolin-10(5*H*)-one (**82**) (40%) although the expected 4-*o*-acetamidobenzoylpyridazine

(**81**, R = Ac) could be obtained with a different acylating reagent [neat $MeC(OEt)_3$, 160°, 6 h: 42%].[1732]

6-Piperazin-1′-yl- gave 6-(4-benzoylpiperazin-1-yl)-3(2*H*)-pyridazinone (BzCl, $NaHCO_3$, $CHCl_3$, 25°, 24 h: 30%).[1761]

Also other examples.[159,528,541,1340,1463]

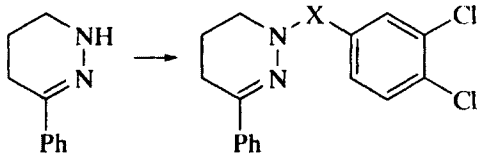


(**81**) (**82**)

## Acylation at Ring Nitrogen of Hydropyridazines

*Note*: This subsection gives examples of the acylation of ring NH groupings that bear a proton by virtue of nuclear reduction rather than tautomerism.

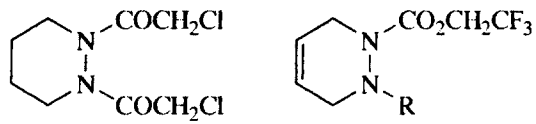
3-Phenyl-1,4,5,6-tetrahydropyridazine (**83**) gave 1-(3,4-dichlorobenzoyl)- [**84**, X = -C(:O)-] (3,4-$Cl_2C_6H_3COCl$, PhMe, reflux, 90 min: 44%) or 1-(3,4-dichlorobenzenesulfonyl)-3-phenyl-1,4,5,6-tetrahydropyridazine [**84**, X = -S(:O)$_2$-] (3,4-$Cl_2C_6H_3SO_2Cl$, pyridine, 15° → 22°, 4 h: ~15%).[1834]



(**83**) (**84**)

Hexahydropyridazine gave 1,2-bis(chloroacetyl)hexahydropyridazine (**85**) [substrate·HCl, neat $(ClCH_2CO)_2O$, 85°, 30 min: >90%].[1382]

1,2,3,6-Tetrahydropyridazine gave 1-(β,β,β-trifluoroethoxycarbonyl)- 1,2,3,6-tetrahydropyridazine (**86**, R = H) [$(F_3CCH_2CO)_2CO$, $Et_2O$, 25°,



(**85**) (**86**)

90 min: ?%] and then ethyl 2-(β,β,β-trifluoroethoxycarbonyl)-12,3,6- tetrahydro-1-pyridazinecarboxylate (**86**, R = $CO_2Et$) ($ClCO_2Et$, lutidene, <5°, 2 h: 89%).[629]

Also other examples.[297,633,728,741,912,1110,1512,1764,1810,2045]

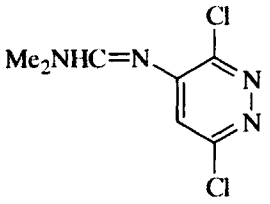
### *7.2.2.2 N-Alkylidenation and Subsequent Reactions (H 494)*

The formation of Schiff base–type products from primary pyridazinamines has been used extensively, often for subsequent intramolecular cyclization. The following examples illustrate such reactions:

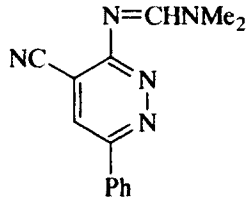
**Simple *N*-Alkylidenations**

3,6-Dichloro-4-pyridazinamine gave 3,6-dichloro-4-dimethylaminomethyleneaminopyridazine (**87**) [$Me_2NCH(OMe)_2$, PhMe, reflux, 2 h: 50%].[84]

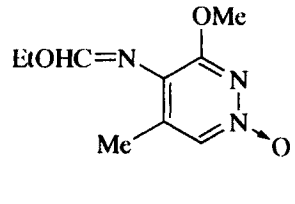
3-Amino- gave 3-dimethylaminomethyleneamino-5-phenyl-4-pyridazinecarbonitrile (**88**) [$Me_2NCH(OMe)_2$, PhMe, reflux, 3 h: 85%; or $POCl_3$, $Me_2NCHO$, 25°, 12 h: 52%].[130]



(**87**)



(**88**)



(**89**)

3-Methoxy-5-methyl-4-pyridazinamine 1-oxide gave 4-ethoxymethyleneamino-3-methoxy-5-methylpyridazine 1-oxide (**89**) [$HC(OEt)_3$, $Me_2NCHO$, EtOH, HCl gas↓ to pH 1, 155°, EtOH↑, 90 min: 90%].[902]

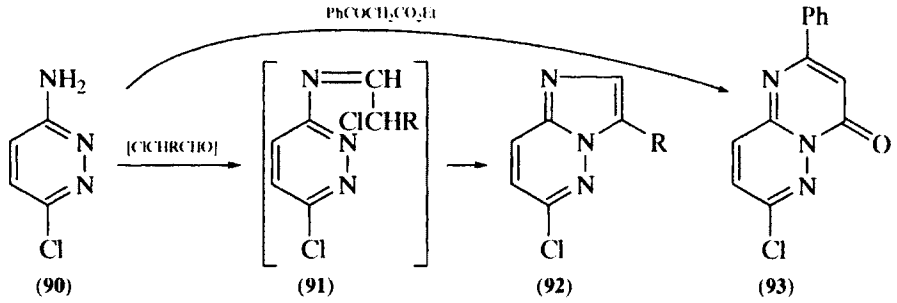
Also other examples.[212,904,910,1010,1681]

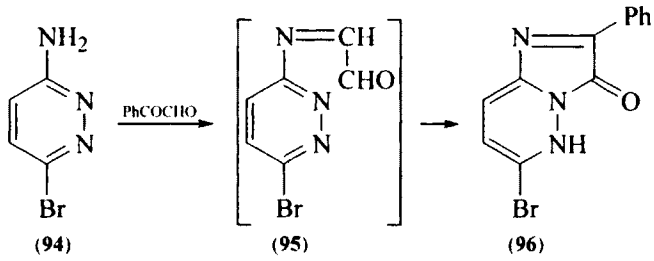
**Cyclizations Involving Initial Alkylidenations**

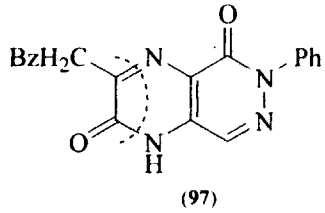
6-Chloro-3-pyridazinamine (**90**) gave 6-chloroimidazo[1,2-*b*]pyridazine (**92**, R = H) [$ClCH_2CH(OEt)_2$, 4M HCl, reflux, 1 h; then (**90**)↓, EtOH, reflux, 30 min: 78%; probably via the Schiff intermediate (**91**, R = H)][154] or 6-chloro-3-methylimidazo[1,2-*b*]pyridazine (**92**, R = Me) (MeCHBrCHO, EtOH, reflux, 2 h: 83%).[1619] A great variety of analogues, some of which are highly active in the central nervous system and/or in the peripheral nervous system,[2030] have been made in this way from appropriately substituted 3-aminopyridazines with α-halogenated aldehydes or α-halogenated ketones.[551,1523,1525,1529,1530,1843; cf. 1145]

6-Chloro-4-pyridazinamine (**90**) gave, somewhat similarly, 7-chloro-2-phenyl-4*H*-pyrimido[1,2-*b*]pyridazin-4-one (**93**) ($BzCH_2CO_2Et$, polyphosphoric acid, 120°, 5 h: 54%).[214]

6-Bromo-3-pyridazinamine (**94**) gave 6-bromo-2-phenylimidazo[1,2-*b*]pyridazin-3(5*H*)-one (**96**), probably via the Schiff intermediate (**95**) (BzCHO, HCl, EtOH, 25°, 3 days: ~60%);[1145] also many analogues by this general method.[1145,1147,1149,1840,2023]



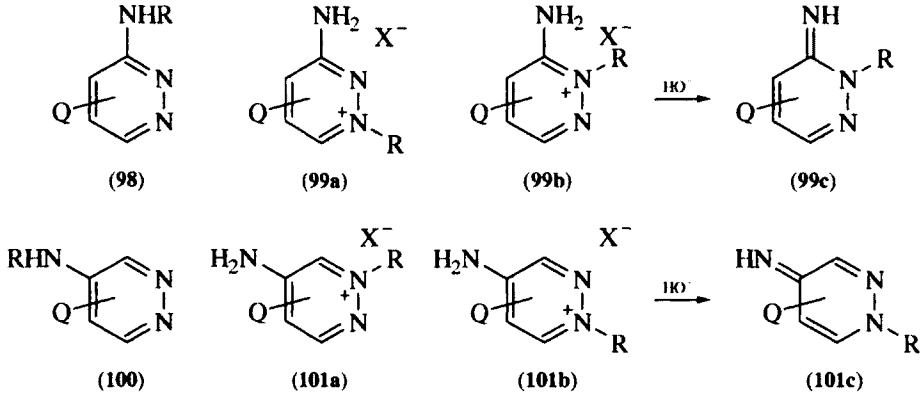




4,5-Diamino-2-phenyl-3(2*H*)-pyridazinone gave 3-phenacyl-6-phenylpyrazino[2,3-*d*]pyridazine-2,5(1*H*,6*H*)-dione (**97**) ($BzCH_2COCO_2H$, EtOH, reflux, 15 min: 86%).[652]

Also other examples.[126, 204, 676, 716, 815, 1020, 1050, 1241, 1404, 1484, 1572, 1636]

### *7.2.2.3 N-Alkylation Reactions (H 497, 507)*

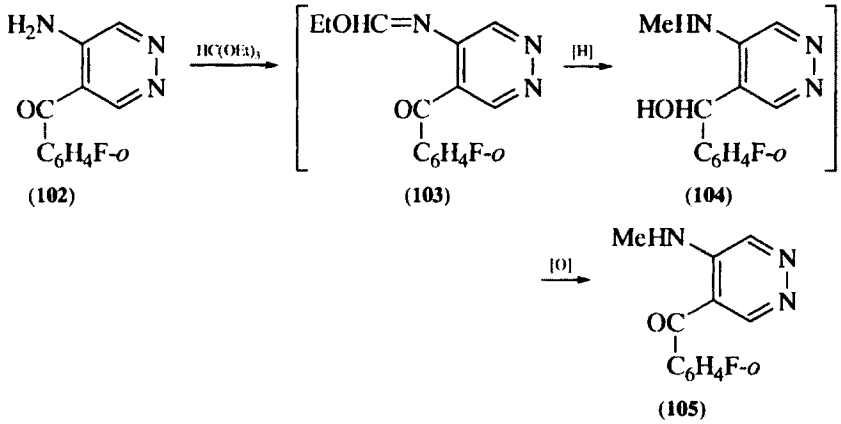
In principle, nuclear primary or secondary aminopyridazines may undergo alkylation on the amino group to give alkylaminopyridazines of the type (**98/100**) or on a ring nitrogen to give amino-1/2-alkylpyridazinium salts of the type (**99a/b** or **101a/b**); the course of each reaction will be controlled or influenced by passenger groups (Q) and the conditions employed. Moreover, products of the type (**99a/101b**) can be isolated as free imines (**99c/101c**), and the first of these could undergo Dimroth rearrangement[2005] to alkylamines of the type **98**, although no recent examples have been reported. As a rule, extranuclear aminopyridazines alkylate on the amino group, usually the most basic center of the molecule. The following examples clarify the situation.



**Alkylations at the Amino Group**

*Note*: Some examples of direct alkylation at the amino group have been given in Section 7.2.1. Others, both direct and indirect, are summarized here.

5-*o*-Fluorobenzoyl-4-pyridazinamine (**102**) gave 4-*o*-fluorobenzoyl-5-methylaminopyridazine (**105**) via the intermediates **103** and **104** [$HC(OEt)_3$, trace $F_3CCO_2H$, 100°, 3 h; $NaBH_4$, EtOH, reflux, 2 h; $KMnO_4$, 1M $H_2SO_4$, 25°, 2 h: 43% overall];[746] also analogues similarly.[218,746]

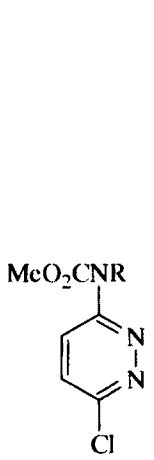


3-Chloro-6-methoxycarbonylaminopyridazine (**106**, R = H) gave 3-(*N*-but-3-ynyl-*N*-methoxycarbonylamino)-6-chloropyridazine (**106**, R = $CH_2CH_2C{\equiv}C$) ($HC{\equiv}CCH_2CH_2OH$, $Ph_3P$, THF, 0°, then $EtO_2CN{=}NCO_2Et\downarrow$, 25° 24 h: 47%); also analogues likewise.[336]

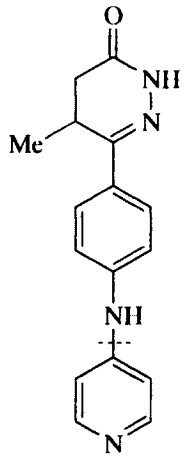
6-*p*-Aminophenyl-5-methyl- gave 5-methyl-6-*p*-(pyridin-4-ylamino)phenyl-4,5-dihydro-3(2*H*)-pyridazinone (**107**) [4-Cl-pyridine, $MeN(CH_2)_4$, $Et_3N$, 90°, 2 h: 68%].[1310]

6-Piperazin-1′-yl-3(2*H*)-pyridazinone gave 6-(4-β-phenoxyethylpiperazin-1-yl)-3(2*H*)-pyridazinone (**108**) ($PhOCH_2CH_2Cl$, EtOH, reflux, 8 h: 40%); several analogues likewise.[1761]

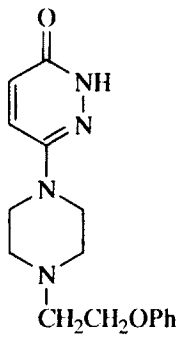
Also other examples.[522, 1783, 2116]




(**106**)




(**107**)




(**108**)

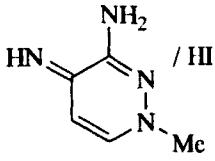
## Alkylations at a Ring Nitrogen

3-Pyridazinamine (**98**, Q = R = H) gave a separable mixture of 3-amino-1-methylpyridazinium iodide (**99a**, Q = H, R = Me, X = I) and 6-amino-1-methylpyridazinium iodide (**99b**, Q = H, R = Me, X = I), the hydriodide of 2-methyl-3(2*H*)-pyridazinimine (**99c**, Q = H, R = Me) (MeI, MeOH, 20°, 12 h: ~40% and ~50%, respectively);[675] 4-pyridazinamine (**100**, Q = R = H) likewise gave 5-amino-1-methylpyridazinium iodide (**101a**, Q = H, R = Me, X = I) and 4-amino-1-methylpyridazinium iodide (**101b**, Q = H, R = Me, X = I), the hydriodide of 1-methyl-4(1*H*)-pyridazinimine (**101c**, Q = H, R = Me)[675]

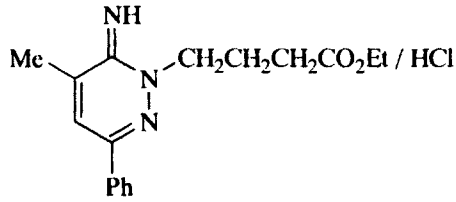
3,4-Pyridazinediamine gave only one methiodide, 4-imino-1-methyl-1,4-dihydro-3-pyridazinamine hydriodide (**109**) (MeI, MeOH, 20°, 3 days: ~85%);[675, 1132] also related alkylations.[675]

4-Methyl-6-phenyl-3-pyridazinamine gave 2-γ-ethoxycarbonylpropyl-4-methyl-6-phenyl-3(2*H*)-pyridazinimine, isolated as its hydrochloride (**110**) ($BrCH_2CH_2CH_2CO_2Et$, $Me_2NCHO$, 80°, 2 h; then HBr → HCl: 72%);[1118, 1309] also analogues likewise.[160, 1309]

5-Amino-2-phenyl-3(2*H*)-pyridazinone (**111**), gave 6-methoxy-1-phenyl-4(1*H*)-pyridazinimine (**112**), initially as its methyl hydrogen sulfate ($Me_2SO_4$, $CHCl_3$, reflux, 2 h: 61%; note that *O*-methylation here produced a fixed imine!).[1344]



(109)



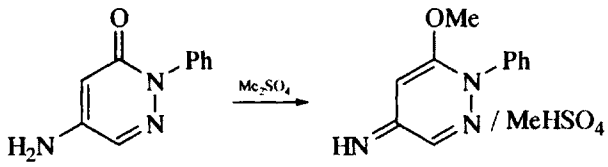
(110)

### 1/2-Alkylations of Hydropyridazines

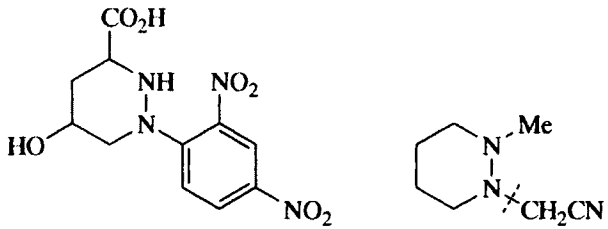
*Note*: It seems appropriate to include at this point some typical examples of alkylations at the ring nitrogen of reduced pyridazines.

5-Hydroxyhexahydro-3-pyridazinecarboxylic acid gave 1-(2,4-dinitrophenyl)-5-hydroxyhexahydro-3-pyridazinecarboxylic acid (**113**) [$FC_6H_3(NO_2)_2$-2,4-, EtOH–$H_2O$, 2 h: ?%].[32]

1-Methylhexahydropyridazine gave 1-cyanomethyl-2-methylhexahydropyridazine (**114**) ($CH_2O$, $NaHSO_3$, $H_2O$, 60° → 35°; then substrate ↓, 25°, > 2 h; then NaCN ↓, 3.5 h: 46%).[1699]



(111) (112)



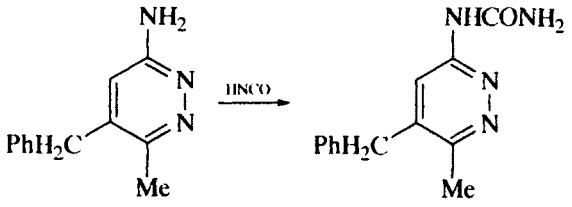
(113) (114)

### *7.2.2.4 Conversion into Ureidopyridazines or Related Products*

Ureidopyridazines may be made by the reaction of isocyanatopyridazines with primary or secondary amines (see Section 8.5) or, more usually, by the reaction of primary or secondary aminopyridazines with isocyanates, as illustrated in the following examples:

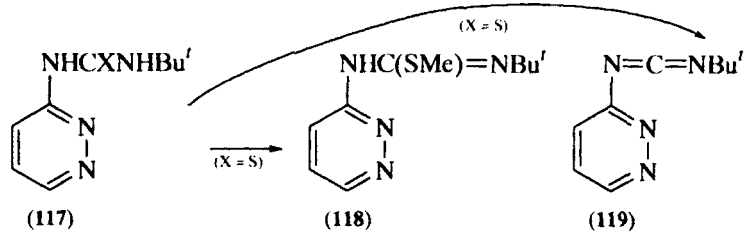
### Using Simple Isocyanates or Isothiocyanates

5-Benzyl-6-methyl-3-pyridazinamine (**115**) gave 4-benzyl-3-methyl-6-ureidopyridazine (**116**) (KOCN, AcOH–$H_2O$, 95°, 24 h: 50%).[1751]




(115) (116)

3-Pyridazinamine gave 3-*N'*-*t*-butylureidopyridazine (**117**, X = O) (substrate, NaH, $Me_2NCHO$, 0°; then Bu'NCO, 0° → 25°, 3 h: 78%) or 3-*N'*-*t*-butyl(thioureido)pyridazine (**117**, X = S) (likewise but Bu'NCS: 76%);[1608] the product (**117**, X = S) then gave 3-(*N'*-*t*-butyl-*S*-methylisothiouriedo)pyridazine (**118**) (substrate, KOH, $H_2O$–PrOH–THF, 25°, 90 min; then MeI↓, 25°, 24 h: 51%) or, surprisingly, *N*-*t*-butyl-*N'*-pyridazin-3'-ylcarbodiimide (**119**) [$Bu_4NBr$, MeI(!), NaOH, $H_2O$–$CH_2Cl_2$, reflux 2 h: 62%].[1608]

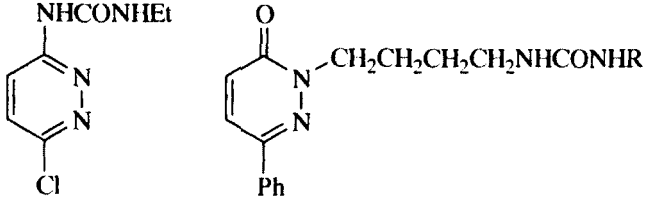



(117) (118) (119)

6-Chloro-3-pyridazinamine gave 3-chloro-6-*N'*-ethylureidopyridazine (**120**) (EtNCO, dioxane, reflux, 9 h: 62%).[1338]

2-*δ*-Aminobutyl-6-phenyl-3(2*H*)-pyridazinone gave 2-*δ*-(*N'*-methylureido)-butyl-6-phenyl- (**121**, R = Me) (MeNCO, $CHCl_3$, 5° → reflux, 2 h: 86%) or 6-phenyl-2-*δ*-(*N'*-*p*-toluenesulfonylureido)butyl-3(2*H*)-pyridazinone (**121**, R = Ts) (likewise but TsNCO: 78%).[1337]

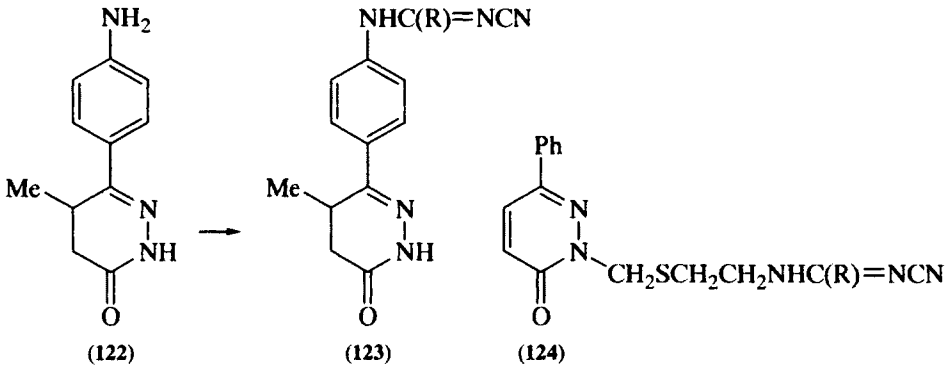
Also other examples.[295, 539, 1109, 1289, 1919, 2060, 2068]




(120) (121)

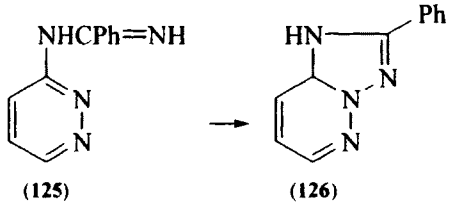
## Using Other Reagents

6-*p*-Aminophenyl- (**122**) gave 6-*p*-(*N'*-cyano-*S*-methylisothioureido)phenyl- (**123**, R = SMe) [$(MeS)_2C{=}NCN$, pyridine, reflux, 3.5 h: 65%] and thence 6-*p*-(*N'*-cyano-*N''*-methylguanidino)phenyl-5-methyl-4,5-dihydro-3(2*H*)-pyridazinone (**123**, R = NHMe) ($MeNH_2$, EtOH, reflux, 2 h: 72%).[1086]



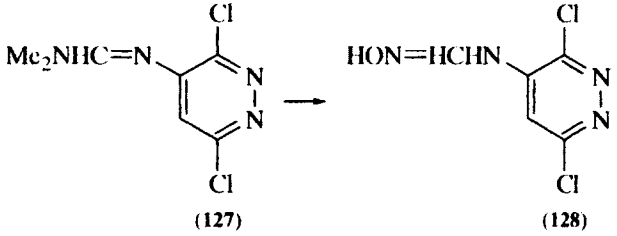

2-$\beta$-Aminoethylthiomethyl- gave 2-$\beta$-(*N'*-cyano-*S*-methylisothioureido) ethylthiomethyl- (**124**, R = SMe) [$(MeS)_2C{=}NCN$, EtOH, 25°, 12 h: 73%] and then 2-$\beta$-(*N'*-cyano-*N''*-methylguanidino)ethylthiomethyl-6-phenyl-3(2*H*)-pyridazinone (**124**, R = NHMe) ($MeNH_2$, MeOH, 60°, 7 h: 84%).[1292]

3-Pyridazinamine gave 3-benzimidoylaminopyridazine (**125**) (PhCN, NaOMe, $Me_2SO$: no details) and then 2-phenyl[1,2,4]triazolo[1,5-*b*]pyridazine (**126**) [$Pb(OAc)_4$, $CH_2Cl_2$; or $Br_2$, AcOH: no details].[244]

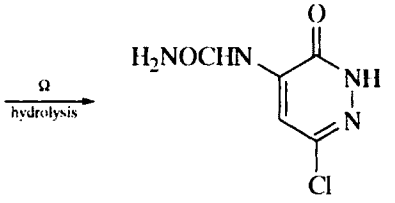



3,6-Dichloro-4-pyridazinamine gave 3,6-dichloro-4-dimethylaminomethyleneaminopyridazine (**127**) [$Me_2NCH(OMe)_2$, PhMe, reflux, 2 h: 50%], which gave 3,6-dichloro-4-hydroxyimnomethylaminopyridazine (**128**) ($HONH_2{\cdot}HCl$, EtOH, reflux, briefly: 44%) or 6-chloro-4-ureido-3(2*H*)-pyridazinone (**129**) (likewise but reflux, 2 h: 70%; note Beckmann rearrangement and hydrolysis of the 3-Cl substituent);[84] the last product (**129**) afforded 4-acetamido-6-chloro-3(2*H*)-pyridazinone (neat $Ac_2O$, reflux, 3 h: 77%).[84]

Also other examples.[164,835,1452]



(127) (128)



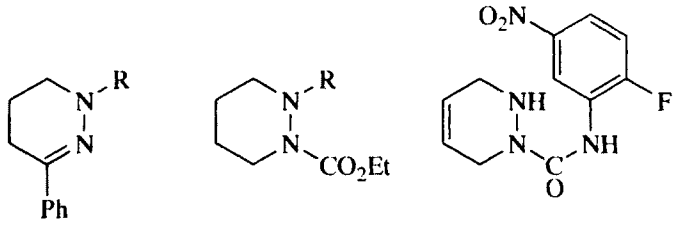
(129)

**Using Reduced Pyridazines as Substrates**

*Note*: It seems appropriate to include here the formation of 1/2-pyridazinecarboxamides from reduced pyridazines.

3-Phenyl-1,4,5,6-tetrahydropyridazine (**130**, R = H) gave *N*-methyl-3-phenyl-1,4,5,6-tetrahydro-1-pyridazinecarbothioamide (**130**, R = CSNHMe) (MeNCS, EtOH, reflux, 3 h: 70%).[1051]

Ethyl hexahydro-1-pyridazinecarboxylate (**131**, R = H) gave ethyl 2-phenylcarbamoylhexahydro-1-pyridazinecarboxylate (**131**, R = CONHPh) (PhNCO, PhH, reflux, 2 h: 70%); several analogues were made similarly.[972]



(130) (131) (132)

1,2,3,6-Tetrahydropyridazine gave *N*-(2-fluoro-5-nitrophenyl)-1,2,3,6-tetrahydro-1-pyridazinecarboxamide (**132**) [substrate hydrochloride, 2-F-5-$NO_2$-$C_6H_3NCO$, $K_2CO_3$, $Me_2NCHO$, 25°, 6 h: 86%].[1690]

Also other examples.[1000, 1182, 1788]

### *7.2.2.5 Miscellaneous Reactions (H 495, 498, 505)*

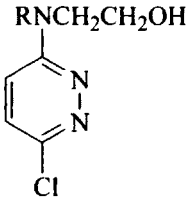
Aminopyridazines undergo several other reactions illustrated in the following classified examples:

### *N*-Nitrosation

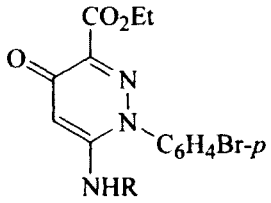
3-Chloro-6-β-hydroxyethylaminopyridazine (**133**, R = H) gave 3-chloro-6-(*N*-α-hydroxyethyl-*N*-nitrosoamino)pyridazine (**133**, R = NO) ($NaNO_2$, 0.5M HCl, 25°: ?%)[1169]

4-Methyl-3-methylamino- gave 4-methyl-3-(*N*-methyl-*N*-nitrosoamino)-6-phenylpyridazine (excess $NaNO_2$, 2.5 M HCl, 80°, ? min: >95%).[1333]

Ethyl 6-amino-1-*p*-bromophenyl- (**134**, R = H) has been reported to give ethyl 1-*p*-bromophenyl-6-nitrosoamino-4-oxo-1,4-dihydro-3-pyridazinecarboxylate (**134**, R = NO) ($NaNO_2$, $AcOH–H_2O$, 15°, 20 h: >95%).[1304]



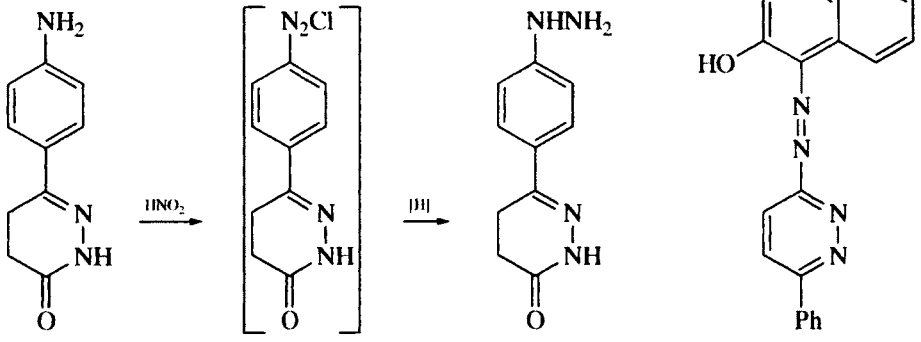

(133)




(134)

### Diazotization and Subsequent Reactions

6-*p*-Aminophenyl- (**135**) gave 6-*p*-hydrazinophenyl-4,5-dihydro-3(2*H*)-pyridazinone (**137**), via the unisolated diazonium salt (**136**) ($NaNO_2$, 2M HCl, 0°, 30 min; then $SnCl_2$↓ HCl↓, 0°, 1 h: 72%); also analogues likewise.[1327]




(135) (136) (137) (138)

6-Phenyl-3-pyridazinamine gave (unisolated) 6-phenyl-3-pyridazinediazonium chloride and then 3-(2-hydroxynaphthalen-1-ylazo)-6-phenylpyridazine (**138**) ($NaNO_2$, 0.5M HCl, 5°; then 2-naphthalenol / 1M NaOH↓: 58%).[1279]

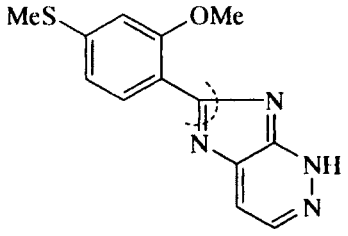
Also other examples.[909, 1453, 2066, 2086]

## Cyclization Reactions

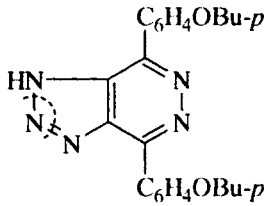
*Note*: Aminopyridazines can clearly undergo cyclization in many ways, some already mentioned in Sections 7.2.2.1 and 7.2.2.2. A few other typical examples are given here.

4,5-Pyridazinediamine and 2-methoxy-4-methylthiobenzoic acid gave 6-(2-methoxy-4-methylthiophenyl)-1*H*-imidazo[4,5-*c*]pyridazine (**139**) ($POCl_3$, reflux, 4 h: 86%).[1755]

3,6-Bis-*p*-butoxyphenyl-4,5-pyridazinediamine and nitrosylsulfuric acid gave 4,7-bis-*p*-butoxyphenyl-1*H*-1,2,3-triazolo[4,5-*d*]pyridazine (**140**) (AcOH, <20°, 9 h: 37%).[1683]

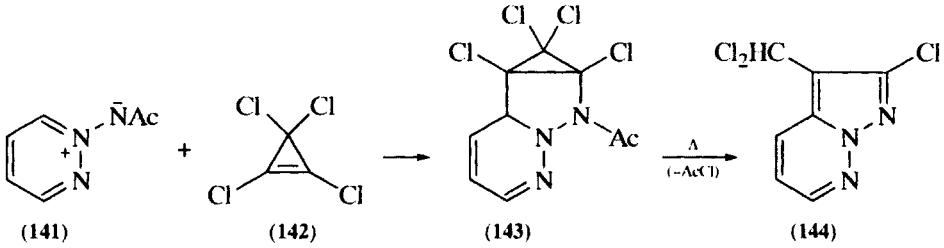



(**139**)

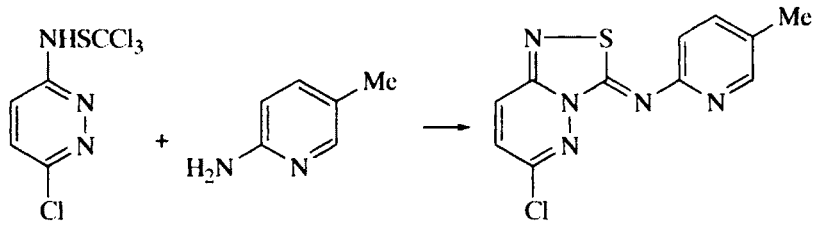



(**140**)

Pyridazinium 1-acetylimide (**141**) and perchlorocyclopropene (**142**) gave 6-acetyl-4b,5,5,5a-tetrachloro-4a,8b,5a,6-tetrahydro-5*H*-cyclopenta[3,4]-pyrazolo[1,5-*b*]pyridazine (**143**) (THF, 0° → 25°, $N_2$, 24 h: 41%) and then 2-chloro-3-dichloromethylpyrazolo[1,5-*b*]pyridazine (**144**) (xylene, 155°, $N_2$, monitored by tlc: 50%); also analogues likewise.[879]




(**141**) (**142**) (**143**) (**144**)




(**145**) (**146**) (**147**)

6-Chloro-3-pyridazinamine gave 3-chloro-6-trichloromethylthioaminopyridazine (**145**) ($Cl_3CSH$, $K_2CO_3$, ice water, 30 min: crude) and then, with 5-methyl-2-pyridinamine (**146**), 6-chloro-3-(5-methylpyridin-2-ylimino)-3*H*-[1,2,4]thiadiazolo[4,3-*b*]pyridazine (**147**) ($Et_3N$, $CHCl_3$, 25°, 24 h: 16%); analogues somewhat similarly.[303]

Also other examples.[421,576,2066,2086,2119]

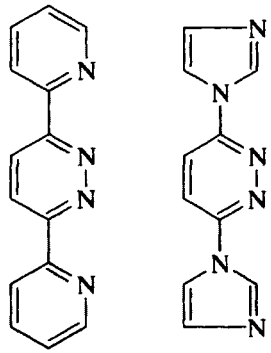
## *7.2.2.6 Ancillary Information (H 486)*

Recently described *metal complexes* involving aminopyridazines (and coligands) are typified by the following random examples:

3,6-Dipyridin-2′-ylpyridazine (**148**) with Cu (X-ray analysis)[168] or with Rh or Ru (syntheses, uv and luminescence spectra).[1865,2088]

3,6-Diimidazol-1′-ylpyridazine (**149**) with Cu (syntheses, X-ray analyses, and ir spectra);[1945] also analogous complexes.[2091,2092]

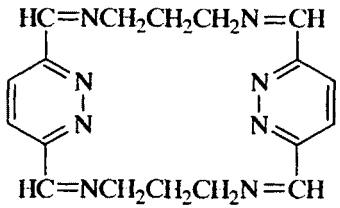
3-Pyridazinamine with Ru (syntheses, antitrypanosome and antitumor activities, and toxicities).[1866]



(**148**)



(**149**)




(**150**)

Macrocyclic aminopyridazines such as the ligand (**150**) with Pb and Mn (syntheses and X-ray analyses for typical complexes).[1894]

Also other examples.[2017,2025,2072]

Several structures of amino- or iminopyridazines have been determined or confirmed by *X-ray analyses*.

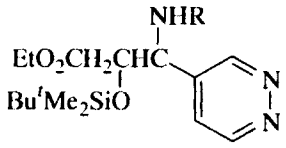
4-[α-Benzylamino-β-(*t*-butyldimethylsiloxy)-β-ethoxycarbonylethyl]pyridazine (**151**, R = $CH_2Ph$; *syn* isomer).[1906]

4-[β-(*t*-Butyldimethylsiloxy)-β-ethoxycarbonyl-α-*p*-methoxyanilinoethyl]-pyridazine (**151**, R = $C_6H_4OMe$-*p*-; *anti* isomer).[1906]

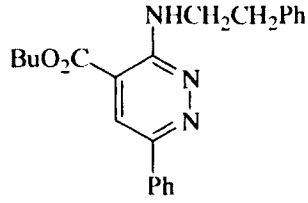
Butyl 3-phenethylamino-6-phenyl-4-pyridazinecarboxylate (**152**).[172]

3-Diphenylphosphinyl-5-*N*-methylanilino-4,6-diphenylpyridazine (**153**).[1123]

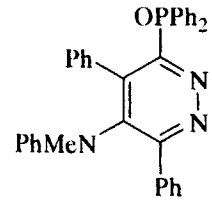
3-(β-Morpholinoethylamino-6-phenyl-4-pyridazinecarboxamide (**154**) as monohydrate.[172]



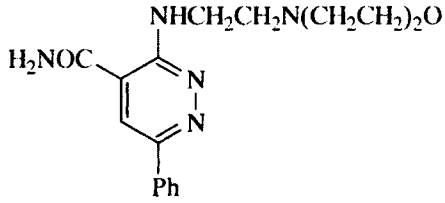
(**151**)



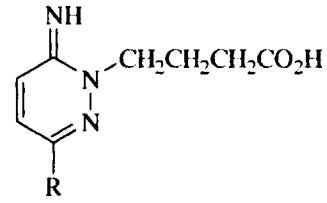
(**152**)



(**153**)



(**154**)



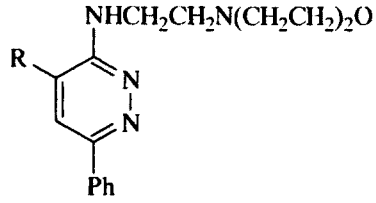
(**155**)

2-γ-Carboxypropyl-6-phenyl- (**155**, R = Ph)[171] and 2-γ-carboxypropyl-6-cyclohexyl-3(2*H*)-pyridazinimine hydrobromide (**155**, R = $C_6H_{11}$).[1192]

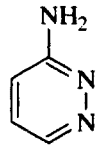
General studies on the *ionization* of regular aminopyridazines have been confined to analogues of Minaprine (**156**, R = Me) (p$K_a$ 7.05, 4.0 representing proton gains at the basic center of the morpholine ring and pyridazine ring, respectively).[102] Thus, replacement of the 4-methyl group by electronically comparable groups (H, Et, $CH_2Ph$, $CH_2OH$) had little effect on either p$K_a$ value;[102] replacement by electron-withdrawing groups (Ph, $CONH_2$, CHO, $CO_2Et$, $CF_3$, Cl, CN, $NO_2$) had a minimal effect on the morpholine center (insulated by $-CH_2CH_2-$) but did have a profound base-weakening effect on the pyridazine center. For example, the values for 3-(β-morpholinoethylamino)-4-nitro-6-phenylpyridazine (**156**, R = $NO_2$) were 6.6 and 0.2,[102] and replacement by an electron-donating group ($NH_2$) did not affect the morpholine center much but increased the basic strength of the pyridazine moiety significantly; 3-(β-morpholinoethylamino-6-phenyl-4-pyridazinamine (**156**, R = $NH_2$) had p$K_a$ values 7.7 and 6.3).[1720]

There is a great increase in basic strength when a 3/4-pyridazinamine undergoes 1/2-alkylation to afford an imino or zwitterionic base; for example, compare 3-pyridazinamine (**157**) (p$K_a$ 5.2) with 2-methyl-3(2*H*)-pyridazinimine (**158**) (p$K_a$ > 10) and with the anhydro pseudobase, 1-methylpyridazinium-3-aminate (**159**) (p$K_a$ > 14);[675] also 4-methyl-3-β-morpholinoethylamino-6-phenylpyridazine (**156**, R = Me) (P$K_a$ 7.05) with 2,5-dimethyl-3-(β-morpholinoethylimino)-6-phenyl-2,3-dihydropyridazine (**160**) (p$K_a$ 10.4).[1720]

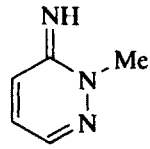
The common *trivial names* used for aminopyridazine are listed with those of other pyridazines in Section 5.7.4.



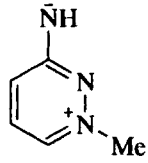
(156)



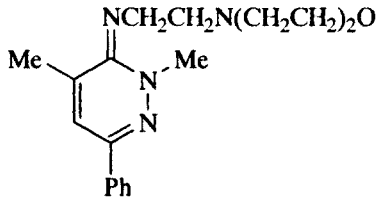
(157)



(158)



(159)



(160)

## 7.3 HYDRAZINO-, HYDROXYAMINO-, AND AZIDOPYRIDAZINES (*H* 631)

Although preparative routes to such (substituted-amino)pyridazines have much in common with those to regular aminopyridazines, the reactions of the two groups differ markedly in many respects. The preparation and metabolic transformations of hydrazinopyridazine drugs and potential drugs have been reviewed.[1481]

### 7.3.1 Preparation of Hydrazinopyridazines ( *H* 635)

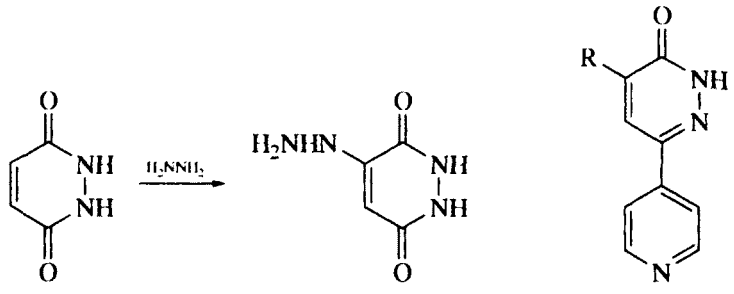
The main routes, that have been used recently to make hydrazinopyridazines, have been covered earlier: by *primary syntheses*, Chapters 1 and 2; *hydrazinolysis of halogenopyridazines*, Sections 4.2.1 and 4.4.1; *hydrazinolysis of alkoxypyridazines*, Section 5.4.2.1; *hydrazinolysis of tautomeric pyridazinethiones*, Section 6.1.2.4 or *hydrazinolysis of alkylthio-, alkylsulfinyl-, or alkylsulfonylpyridazines*, Sections 6.2.2.2 and 6.4.

The remaining minor methods of preparation are illustrated in the following examples:

#### By Hydrazination

3,6(1*H*,2*H*)-Pyridazinedione (**161**) gave 4-hydrazino-3,6(1*H*,2*H*)-pyridazinedione (**162**) (neat $H_2NNH_2 \cdot H_2O$, 100°, 6 days: ~90% apparently by a mechanism involving 4,5 addition, oxidative loss of ammonia, and transamination);[213] in contrast, 6-pyridin-4′-yl-3(2*H*)-pyridazinone (**163**, R = H) gave only 4-amino-6-pyridin-4′-yl-3(2*H*)-pyridazinone (**163**,

R = $NH_2$) (neat $H_2NNH_2 \cdot H_2O$, 100°, 48 h: 74%; by a similar mechanism without the final transamination?).[213]

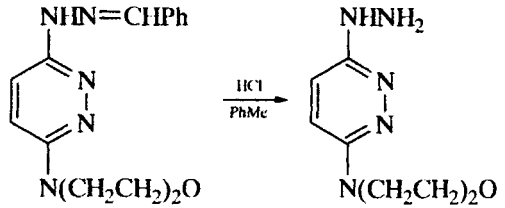


(161) (162) (163)

**From Alkylidenehydrazinopyridazines**

3-Benzylidenehydrazino-6-morpholinopyridazine (**164**) gave 3-hydrazino-6-morpholinopyridazine (**165**) (10M HCl, PhMe, 40°, 90 min: 87%, as hydrochloride);[57] also analogues likewise.[57]

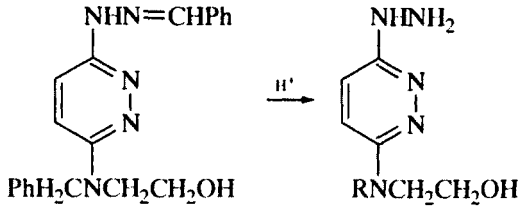
3-(*N*-Benzyl-*N*-β-hydroxylthylamino)-6-benzylidenehydrazinopyridazine (**166**) gave 3-(*N*-benzyl-*N*-β-hydroxyethylamino)-6-hydrazinopyridazine (**167**, R = $CH_2Ph$) (6M HCl, distilled until no further PhCHO: 81%) and then 3-hydrazino-6-β-hydroxyethylaminopyridazine (**167**, R = H) (10M HCl, reflux, 18 h: 36%);[554] likewise several analogues.[554]



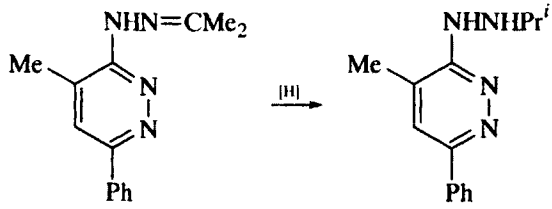
(164) (165)

3-Isopropylidenehydrazino- (**168**) underwent reduction to 3-β-isopropylhydrazino-4-methyl-6-phenylpyridazine (**169**) ($H_2$, Pt, AcOEt, 70 h: 70%, as hydrochloride).[1333]

Also other examples.[1258]



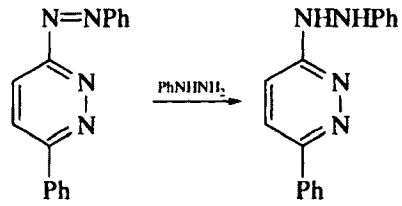
(166) (167)




(168) (169)

### From Arylazopyridazines

3-Phenyl-6-phenylazopyridazine (**170**) gave 3-phenyl-6-β-phenylhydrazinopyridazine (**171**) ($PhNHNH_2$, MeCN, reflux, 3 h: >95%; by replacement or reduction?).[854]




(170) (171)

## 7.3.2 Reactions of Hydrazinopyridazines (*H* 639)

The *reductive fission of hydrazino- to aminopyridazines* has been covered in Section 7.2.1. Other reactions of hydrazinopyridazines are illustrated in the following classified examples; some subsequent reactions, not covered elsewhere in this book, are included here:

### Acylation

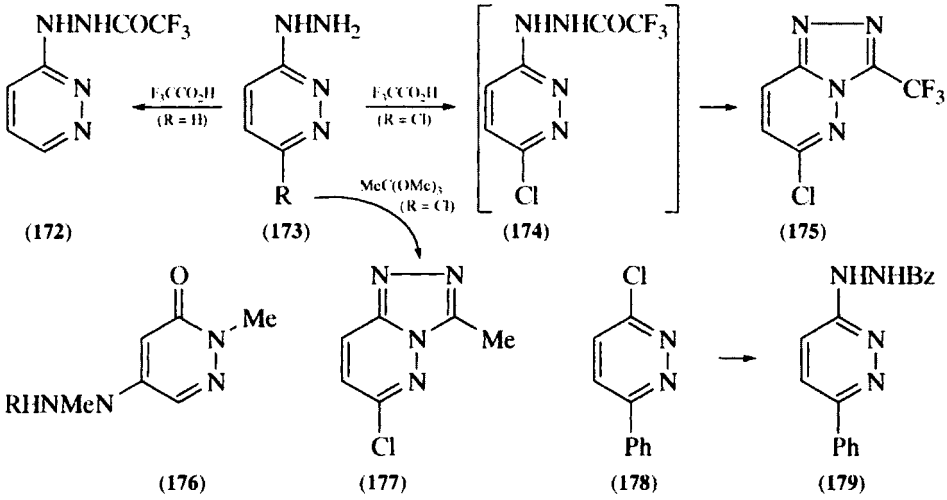
*Note*: Acylation invariably affords a β-acylhydrazinopyridazine unless the position is already blocked, for instance by alkylidenation (see Section 7.4 for an example).

3-Hydrazinopyridazine (**173**, R = H) gave 3-β-trifluoroacetylhydrazinopyridazine (**172**) (neat $F_3CCO_2H$, reflux, 2 h: 87%);[64] in contrast, 3-chloro-6-hydrazinopyridazine (**173**, R = Cl) directly gave 6-chloro-3-trifluoromethyl-1,2,4-triazolo[4,3-*b*]pyridazine (**175**), presumably via the acyl intermediate (**174**) [neat $F_3CCO_2H$, 100° (sealed?), 3 h: 81%].[64]

2-Methyl-5-α-methylhydrazino- (**176**, R = H) gave 5-(β-acetyl-α-methylhydrazino)-2-methyl-3(2*H*)-pyridazinone (**176**, R = Ac) ($Ac_2O$, pyridine, 25°, 10 h: 84%).[873]

3-Chloro-6-hydrazinopyridazine (**173**, R = Cl) gave 6-chloro-3-methyl-1,2,4-triazolo[4,3-*b*]pyridazine (**177**) [neat $MeC(OMe)_3$, reflux, 12 h: 93%; $PhCH_2SC(=NH)Me$, pyridine, 25°, 12 h; then reflux, 2 h: 56%].[981]

Also other examples.[158,711,812,849,1001,1058,1162,1281,1338,1472,2044]

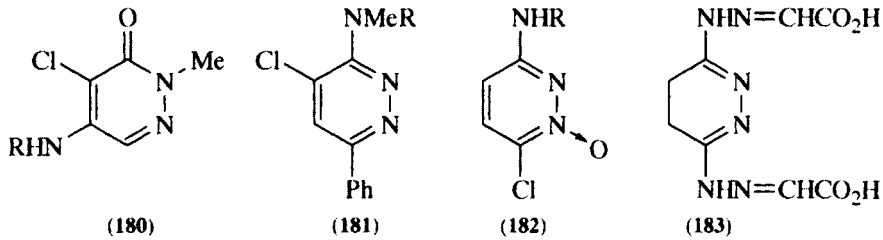



*Note*: A convenient alternative route to acylated hydrazinopyridazines or their cyclized products is by aminolysis of a halogenopyridazine with a carbohydrazide, for example, 3-chloro-6-phenylpyridazine (**178**) gave 3-β-benzoylhydrazino-6-phenylpyridazine (**179**) ($BzNHNH_2$, BuOH, reflux, 5 h: 72%).[1279] Other examples have been reported.[1284]

**Alkylidenation (to Pyridazinylhydrazones)**

4-Chloro-5-hydrazino-2-methyl- (**80**, $R = NH_2$) gave 5-benzylidenehydrazino-4-chloro-2-methyl-3(2*H*)-pyridazinone (**180**, R = N: CHPh) (PhCHO, EtOH, reflux, 2 h: 92%).[1054]

4-Methyl-3-α-methylhydrazino- (**178**, $R = NH_2$) gave 4-methyl-3-(α-methyl-β-methylenehydrazino)-6-phenylpyridazine (**178**, $R = N:CH_2$) 30% $CH_2O$, $Pr^iOH$, 95°, 5 min: 95%.[1333]

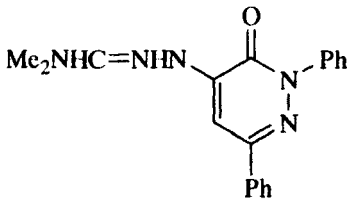


3-Chloro-6-hydrazinopyridazine 2-oxide (**182**, $R = NH_2$) gave 3-chloro-6-isopropylidenehydrazinopyridazine 2-oxide (**182**, $R = N{:}CMe_2$) (AcMe, trace HCl, EtOH, reflux, 30 min: 80%).[546]

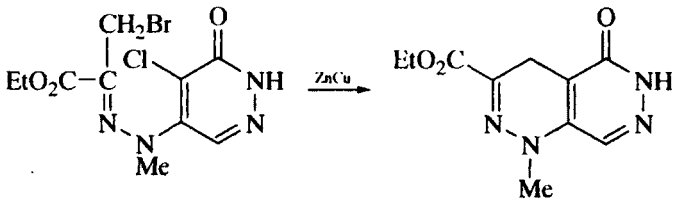
3,6-Dihydrazino-4,5-dihydropyridazine gave 3,6-biscarboxymethylenehydrazino-4,5-dihydropyridazine (**183**) ($HO_2CCHO{\cdot}H_2O$, $H_2O$, $N_2$, 25°: 91%).[639]

6-Chloro-4-hydrazino- gave 6-chloro-4-dimethylaminomethylenehydrazino-2-phenyl-3(2*H*)-pyridazinone (**184**) (Vilsmeier reagent from $Me_2NCHO$–$POCl_3$, $Me_2NCHO$, 20°, 4 h: 75%, initially as the hydrochloride).[1429]

4-chloro-5-α-methylhydrazino-3(2*H*)-pyridazinone gave 5-[β-(β-bromo-α-ethoxycarbonylethylidene)-α-methylhydrazino]-4-chloro-3(2*H*)-pyridazinone (**185**) ($BrCH_2COCO_2Et$, EtOH, 25°, 18 h: 92%) and then ethyl 1-methyl-5-oxo-1,4,5,6-tetrahydropyridazino[4,5-*c*]pyridazine-3-carboxylate (**186**) (Zn-Cu, dioxane, reflux, 18 h: 82%).[1941]



(**184**)

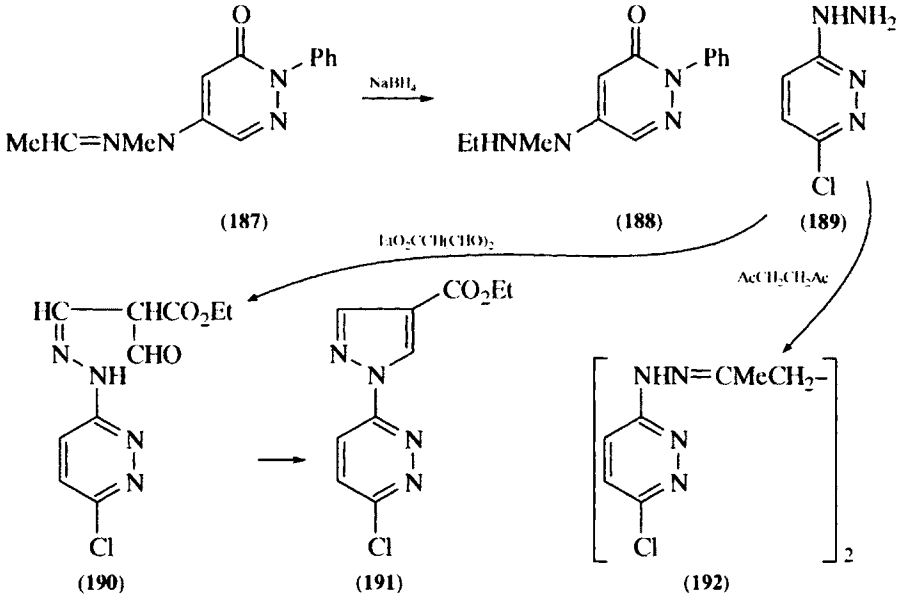


(**185**) (**186**)

5-α-Methylhydrazino- gave 5-(β-ethylidene-α-methylhydrazino)- (**187**) (MeCHO, MeOH 25°, 12 h: 95%) and then 5-(β-ethyl-α-methylhydrazino)-2-phenyl-3(2*H*)-pyridazinone (**188**) ($NaBH_4$, MeOH, 25°, 24 h: 88%);[1540] note that this is a good way to achieve β-alkylation of a hydrazino substrate.

3-Chloro-6-hydrazinopyridazine (**189**) with ethyl diformylacetate gave 3-chloro-6-(4-ethoxycarbonylpyrazol-1-yl)pyridazine (**191**) [EtOH, 0° → 25°, 24 h: 88%, probably via the unisolated intermediate (**190**)];[1693] the same substrate (**189**) (made in situ) with acetonylacetone gave 2,4-bis(6-chloropyridazin-3-ylhydrazono)hexane (**192**) ($H_2O$, 80° → 25°: 80%).[1345]

Also other examples.[18, 209, 432, 495, 539, 555, 671, 793, 954, 1008, 1021, 1227, 1246, 1258, 1262, 1293, 1331, 1336, 1454, 1558, 1583, 1687, 1701, 1711, 1813, 1819, 1909, 1958, 2034, 2036, 2038, 2067, 2070, 2108]

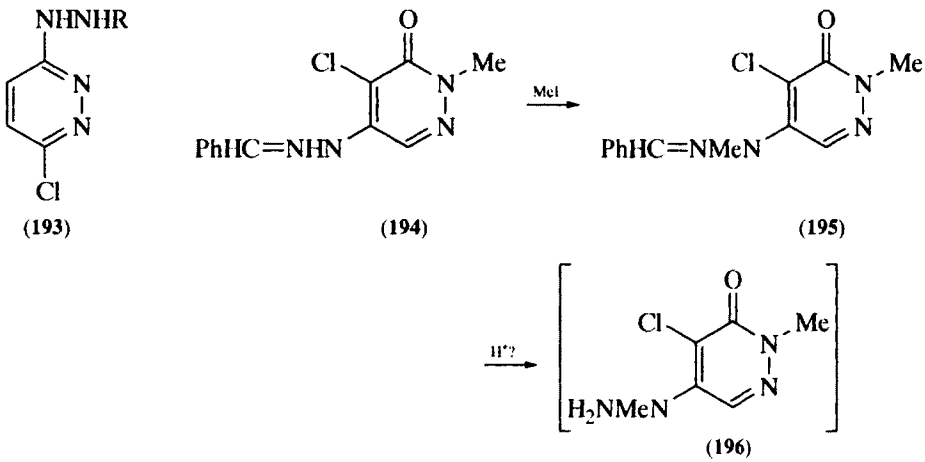



## Alkylation

*Note*: $\beta$-Alkylation may be done directly or by $\beta$-alkylidenation followed by reduction (see preceding subsection); $\alpha$-alkylation may be done directly or by $\beta$-alkylidenation followed by $\alpha$-alkylation and subsequent hydrolytic removal of the unwanted alkylidene group.

3-Chloro-6-hydrazinopyridazine (**193**, R = H) gave 3-chloro-6-$\beta$-tritylhydrazinopyridazine (**193**, R = $CPh_3$) ($ClCPh_3$, $Et_3N$, $CH_2Cl_2$, 20° 1 h: 84%).[1224]

3-Hydrazino- gave 3-$\alpha$-($\beta$-cyanoethyl)hydrazino-6-phenylpyridazine ($H_2C$=CHCN, 2M NaOH, THF, 30° → 60°, 30 min: 32%).[1316]

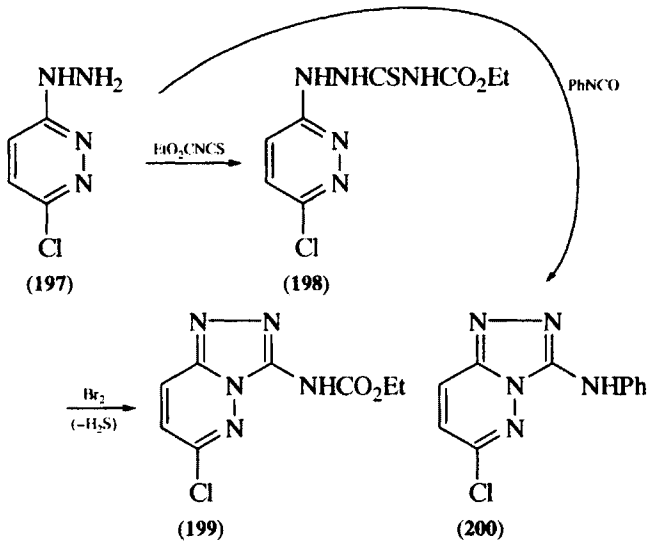


5-Benzylidenehydrazino- (**194**) gave 5-(β-benzylidene-α-methylhydrazino)-4-chloro-2-methyl-3(2*H*)-pyridazinone (**195**) (MeI, $K_2CO_3$, $Me_2NCHO$, 25°, 4 h: 89%),[1054] which should be convertible into 4-chloro-2-methyl-5-α-methylhydrazino-3(2*H*)-pyridazinone (**196**) (not described—for a possible method, see Section 7.3.1).

Also other examples.[2109]

## Conversion into Semicarbazido- or Thiosemicarbazidopyridazines

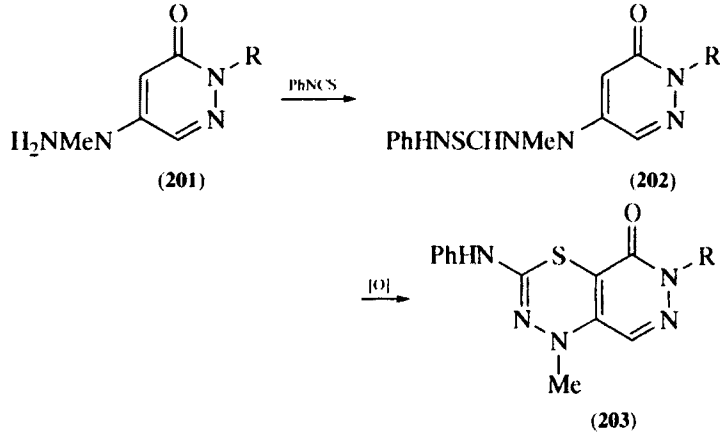
3-Chloro-6-hydrazinopyridazine (**197**) with ethoxycarbonyl isothiocyanate gave 3-chloro-6-[4-ethoxycarbonyl(thiosemicarbazido)]pyridazine (**198**) ($CHCl_3$, reflux, 1 h: 67%) and thence 6-chloro-3-ethoxycarbonylamino-1,2,4-triazolo[4,3-*b*]pyridazine (**199**) ($Br_2$, AcOH, 25°, 3 h: 28%; by an oxidative cyclization with sulfur extrusion);[86] 3-anilino-6-chloro-1,2,4-triazolo[4,3,-*b*]pyridazine (**200**) was made somewhat similarly in 60% yield by using phenyl isocyanate in the presence of triphenylphosphine.[1915]




5-α-Methylhydrazino- (**201**, R = Ph) gave 5-[1-methyl-4-phenyl(thiosemicarbazido)]-2-phenyl-3(2*H*)-pyridazinone (**202**, R = Ph) (PhNCS, MeCN, reflux, 5 h: 49%) and then 3-anilino-1-methyl-6-phenyl-1*H*-pyridazino[4,5-*e*][1,3,4]thiadiazin-5(6*H*)-one (**203**, R = Ph) ($EtO_2CN{=}NCO_2Et$, MeCN, reflux, 4 h: 50%).[1556]

2-Methyl-5-α-methylhydrazino- (**201**, R = Me) gave 2-methyl-5-[1-methyl-4-phenyl(thiosemicarbazido)]-3(2*H*)-pyridazinone (**202**, R = Me) (PhNCS, $Me_2NCHO$, 80°, 3 h: 91%) and then 3-anilino-1,6-dimethyl-1*H*-pyridazino[4,5-*e*][1,3,4]thiadiazin-5(6*H*)-one (**203**, R = Me) (*N*-bromosuccinimide, $CHCl_3$, 25°, 3 h: 63%).[1061]

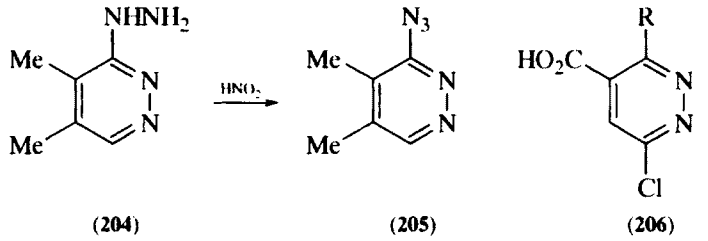
Also other examples in the foregoing references and elsewhere.[721,1722,1751]



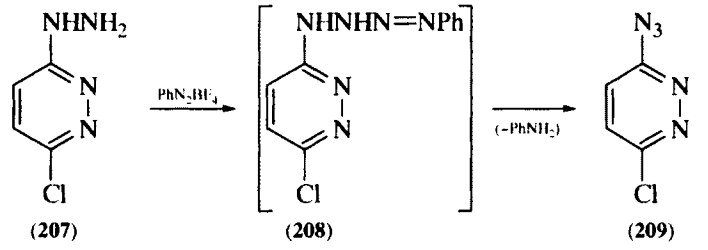

## Conversion into Azidopyridazines

*Note*: Hydrazinopyridazines may be converted into azidopyridazines by the action of nitrous acid or less directly by treatment with a diazonium salt. The ring-chain tautomerism of 3/6-azidopyridazines is discussed in Section 7.3.3.

3-Hydrazino- (**204**) gave 3-azido-4,5-dimethylpyridazine (**205**) ($NaNO_2$, dilute HCl, <5°, 30 min: 90%).[558]




6-Chloro-3-hydrazino- (**206**, R = $NHNH_2$) gave 3-azido-6-chloro-4-pyridazinecarboxylic acid (**206**, R = $N_3$) ($NaNO_2$, dilute HCl, 5–10°, 2 h: 97%).[1228]



3-Chloro-6-hydrazinopyridazine (**207**) gave 3-azido-6-chloropyridazine (**209**), via the detectable but unisolated tetrazene (**208**, or tautomer) ($PhN_2BF_4$, EtOH, 25°, >3 h: 64%).[408]

Also other examples of both methods.[253,363,810,831,1067,1250,1976]

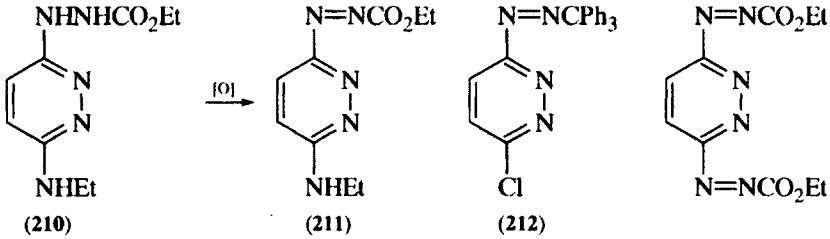
## Conversion into Azopyridazines

*Note*: Hydrazinopyridazines undergo direct oxidation to azopyridazines; an indirect conversion via azidopyridazines is also possible (see Section 7.3.4).

3-*β*-Ethoxycarbonylhydrazino- (**210**) gave 3-ethoxycarbonylazo-6-ethylaminopyridazine (**211**) (Pd/C, PhMe, 25°, dry air↓, 4 h: 98%);[117] also analogues likewise.[117]

3-Chloro-6-*β*-tritylhydrazinopyridazine gave 3-chloro-6-tritylazopyridazine (**212**) (NaOCl, $H_2O-CH_2Cl_2$, $Bu_4NBr$, 25°, 4.5 h: 83%).[1224]

3,6-Bis(*β*-ethoxycarbonylhydrazino)pyridazine gave 3,6-bis(ethoxycarbonylazo)pyridazine (**213**) (neat 70% $HNO_3$, 0°, 5 h: ~80%).[636]

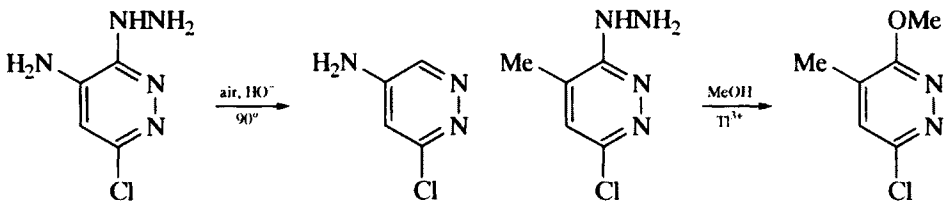



(**210**) (**211**) (**212**) (**213**)

## Miscellaneous Reactions

6-Chloro-3-hydrazino-4-pyridazinamine (**214**) has been reported to give 6-chloro-4-pyridazinamine (**215**) (3M NaOH, air, 85°, 24 h: ~70%).[654]

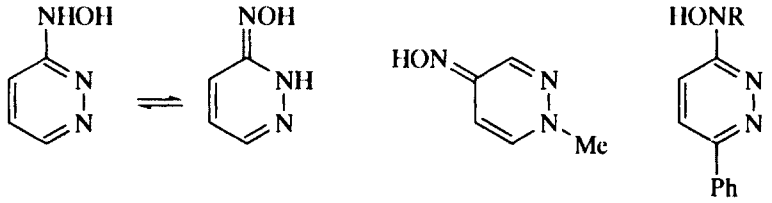
3-Chloro-6-hydrazino-5-methylpyridazine (**216**) gave 3-chloro-6-methoxy-5-methylpyridazine (**217**) [$Tl(NO_3)_3 \cdot 3H_2O$, MeOH, 25°, 20 min: 67%].[1386]




(**214**) (**215**) (**216**) (**217**)

### 7.3.3 Preparation and Reactions of Hydroxyaminopyridazines (*H* 631)

Hydroxyaminopyridazines are sometimes formulated as their tautomeric oximes; for example, 3-hydroxyaminopyridazine (**218**) could be formulated as 3-hydroxyimino-2,3-dihydropyridazine (**219**) which might possibly be called "3(2*H*)-pyridazinone oxime"! Naturally, the presence of an appropriate 1/2-substituent would fix such a compound in the hydroxyimino form, (e.g., **220**).




(218) (219) (220) (221)

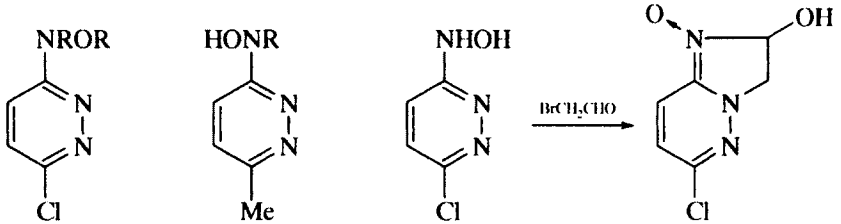
PREPARATIVE METHODS Most recently described hydroxyaminopyridazines have been made by *primary syntheses* (see Chapters 1 and 2), by *hydroxyaminolysis of halogenopyridazines* (see Sections 4.2.1 and 4.4.1), by *hydroxyaminolysis of pyridazinones* (see Section 5.1.2.5), or by *hydroxyaminolysis of alkylthiopyridazines* (see Section 6.2.2.2). In addition, the *deacylation* of 3-(*N*-hydroxyacetamido)-6-phenylpyridazine (**212**, R = Ac) in 10% hydrochloric acid gave 3-hydroxyamino-6-phenylpyridazine (**221**, R = H) in 80% yield.[193]

REACTIONS An example of *reductive deoxygenation* of an hydroxyaminopyridazine to the corresponding pyridazinamine has been given in Section 5.1.2.5. Other reactions are represented by the following examples:

#### Acylation

3-Chloro-6-hydroxyaminopyridazine (**222**, R = H) gave 3-*N*-acetoxyacetamido-6-chloropyridazine (**222**, R = Ac) ($Ac_2O$, $MeOCH_2CH_2OMe$, reflux, 2 h: 51%).[966]

3-(*N*-Hydroxybenzamido)-6-methylpyridazine (**223**, R = Bz) gave 3-(*N*-hydroxyacetamido)-6-methylpyridazine (**223**, R = Ac) ($Ac_2O$, reflux: ?%; a transacylation).[193]




(222) (223) (224) (225)

### Cyclization

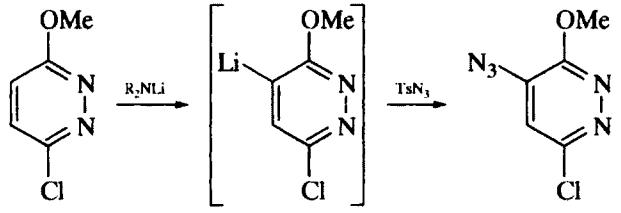
3-Chloro-6-hydroxyaminopyridazine (**224**) gave 6-chloro-2-hydroxy-2,3-dihydroimidazo[1,2-*b*]pyridazine 1-oxide (**225**) [$BrCH_2CH(OMe)_2$, HBr, $H_2O$, 25°d, 14 h: 20%].[196,412]

### Conversion into a Thioureidopyridazine

3-Chloro-6-hydroxyaminopyridazine with ethoxycarbonyl isothiocyanate gave 3-chloro-6-[*N'*-ethoxycarbonyl(thioureido)]pyridazine ($CH_2Cl_2$, 25°, 30 min; 4%; mechanism unclear)[412]

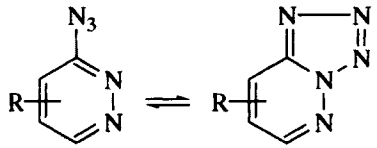
## 7.3.4 Preparation, Isomerization, and Reactions of Azidopyridazines (*H* 645)

PREPARATIVE METHODS Most azidopyridazines have been made from *halogenopyridazines with azide ion* (see Sections 4.2.6 and 4.4.6), from *nitropyridazines with azide ion* (see Section 7.1.2.2), or from *hydrazinopyridazines with nitrous acid or diazonium salts* (see Section 7.3.2). In addition, azidopyridazines have been prepared by *C-metallation and treatment with an acyl azide*. For example, 3-chloro-6-methoxypyridazine (**226**) was treated with lithium *t*-butyl-β-isopropylpentylamide in tetrahydrofuran at −75° during 30 min to give the unisolated intermediate (**227**), which was then allowed to react with tosyl azide at −75° for 2 h to afford 4-azido-6-chloro-3-methoxypyridazine (**228**) in >95% yield.[1900]




(**226**) (**227**) (**228**)

ISOMERISM As in related series,[816] it has long been known that 3/6-azidopyridazines (**229**) and the corresponding isomeric tetrazolo[1,5-*b*]pyridazines (**230**) exist in an equilibrium broadly controlled by solvent, pH of solution




(**229**) (**230**)

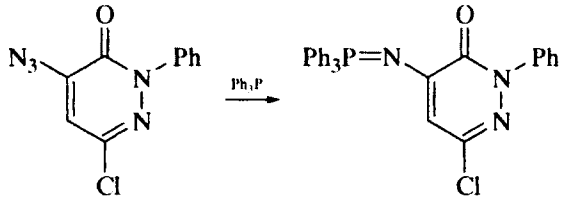
(ionic species), and any passenger groups present.[558] The isolation of a rather esoteric azidopyridazine (as such) and its thermal conversion into the corresponding tetrazolo derivative has been reported.[1711] For obvious pragmatic reasons, all such isomeric pyridazines are named as azidopyridazines throughout this book.

REACTION The direct and indirect *conversion of azido- into aminopyridazines* has been discussed already (Section 7.2.1). Other reactions of azidopyridazines are illustrated in the following examples.

### To Triphenylphosphoranylideneaminopyridazines

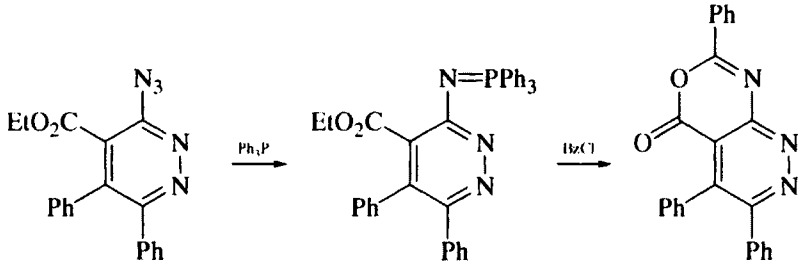
*Note*: Such conversions have been mentioned already as part of an indirect procedure to make pyridazinamines from azidopyridazines (see Section 7.2.1).

4-Azido-6-chloro-2-phenyl-3(2*H*)-pyridazinone (**231**) gave 6-chloro-2-phenyl-4-triphenylphosphoranylideneamino-3(2*H*)-pyridazinone (**232**) ($Ph_3P$, EtOH, 25°, 1 h: 94%);[401] this kinetic of this and related transformations were studied.[401]




(231) (232)

Ethyl 3-azido-5,6-diphenyl-4-pyridazinecarboxylate (**233**) gave ethyl 5,6-diphenyl-3-triphenylphosphoranylideneamino-4-pyridazinecarboxylate (**234**) ($Ph_3P$, xylene, reflux, 12 h: >60%) and then 3,47-triphenyl-5*H*-pyridazino[3,4-*d*][1,3]oxazin-5-one (**235**) (neat BzCl, trace 4-$Me_2N$-pyridine, < 120°, 8 h: 85%).[1417]

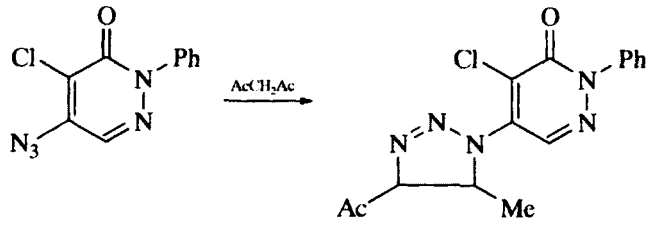



(233) (234) (235)

## To 1,2,3-Triazolylpyridazines

5-Azido-4-chloro-2-phenyl-3(2*H*)-pyridazinone (**236**) gave 5-(4-acetyl-5-methyl-1,2,3-triazol-1-yl)-4-chloro-2-phenyl-3(2*H*)-pyridazinone (**237**) ($AcCH_2Ac$, $Et_3N$, dioxane, 5°, 1 h: 90%).[1166]

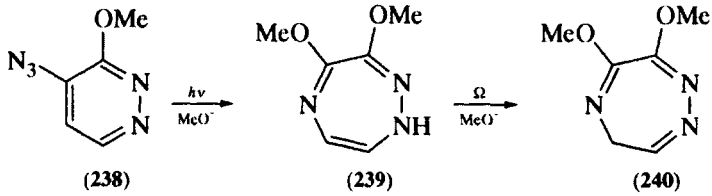
Also other examples.[580,596,599]



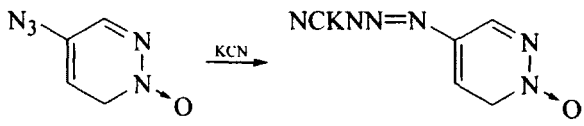

(236) (237)

## Ring Expansion to Triazepines

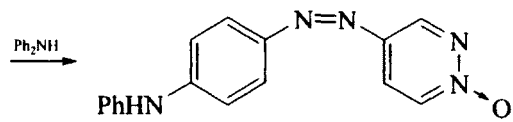
4-Azido-3-methoxypyridazine (**238**) gave 3,4-dimethoxy-1*H*-1,2,5-triazepine (**239**) (MeONa, MeOH-dioxane, *hν*, <5°, 1 h: >50%; unstable but characterized by spectra) and thence the stable isomer, 6,7-dimethoxy-4*H*-1,2,5-triazepine (**240**) (MeONa, MeOH, 25°, 5 h: >30%);[885] also related reactions.[885]




(238) (239) (240)




(241) (242)




(243)

## Miscellaneous Reactions

4-Azidopyridazine 1-oxide (**241**) gave 4-(3-cyanotriazeno)pyridazine 1-oxide as its potassium salt (**242**) (KCN, EtOH–$H_2O$, <5°, 30 min; 91%)

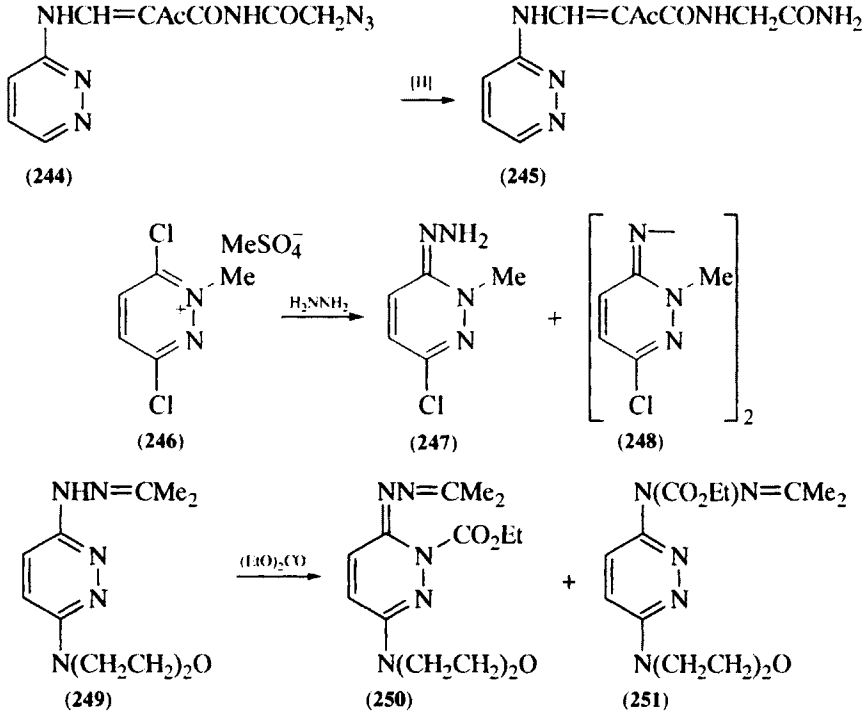
and thence 4-*p*-anilinophenylazopyridazine 1-oxide (**243**) ($Ph_2NH$, HCl, EtOH–$H_2O$, 80°, 10 min: 36%); several analogues likewise.[831]

4-{*β*-Acetyl-*β*-[(azidoacetyl)carbamoyl]vinylamino}pyridazine (**244**) underwent hydrogenation to give a product formulated as 4-{*β*-acetyl-*β*-[(carbamoylmethyl)carbamoyl]vinylamino}pyridazine (**245**) ($H_2$, Pd/C, MeOH, 3.5 atm, 90 min: 56%; mechanism is unclear).[1682]

## 7.4 PREPARATION AND REACTIONS OF NONTAUTOMERIC PYRIDAZINIMINES

Nearly all the recent material in this area has been presented already.

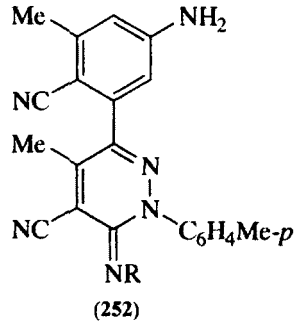
PREPARATIVE ROUTES Such imines have been made by *primary syntheses* (see Chapters 1 and 2) or by *1/2-alkylation of primary or secondary aminopyridazines* (see Section 7.2.2.3). In addition, hydrazinolysis of 3,6-dichloro-1-methylpyridazinium ethyl sulfate (**246**) with methanolic hydrazine at 0° afforded a separable mixture of 3-chloro-6-hydrazono-1-methyl-1,6-dihydropyridazine (**247**) and 3,3′-azinodi(6-chloro-2-methyl-2,3-dihydropyridazine) (**248**) in 25% and 10% yields, respectively.[1183] Also, acylation of 3-isopropylidenehydrazino-6-morpholinopyridazine (**249**) with diethyl carbonate furnished a mixture or ethyl 6-isopropylidenehydrazono-3-morpholino-1,6-



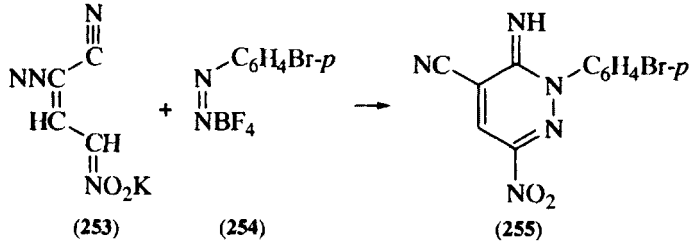
dihydro-1-pyridazinecarboxylate (**250**) and 3-(α-ethoxycarbonyl-β-isopropylidenehydrazino)-6-morpholinopyridazine (**251**).[812]

REACTIONS The *hydrolysis of pyridazinimines* to nontautomeric pyridazinones has been discussed in Section 5.5.1, the *Dimroth rearrangement* of 2-alkyl-3(2*H*)-pyridazinimines has not been studied (cf. *H* 497 and Section 7.2.2.3), and only the following minor reports are of any interest.

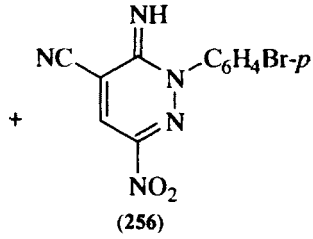
6-(6-Amino-3-cyano-4-methylpyridin-2-yl)-3-imino-5-methyl-2-*p*-tolyl-2,3-dihydro-4-pyridazinecarbonitrile (**252**, R = H) has been reported to react with boiling acetic anhydride during 1 h to afford a single monoacetyl derivative, formulated as 3-acetylimino-6-(6-amino-3-cyano-4-methylpyridin-2-yl)-5-methyl-2-*p*-tolyl-2,3-dihydro-4-pyridazinecarbonitrile (**252**, R = Ac) on ir spectral evidence.[1728] The condensation of the potassium salt of β-nitroethylidenemalononitrile (**253**) with *p*-bromobenzenediazonium tetrafluoroborate (**254**) (cf. Section 1.1.2.9) has been reported to give two isomeric products, *E*- (**255**) and *Z*-2-*p*-bromophenyl-3-imino-6-nitro-2,3-dihydro-4-pyridazinecarbontrile (**256**) in 45–55 ratio;[1495,1497] different *para*- substituents affected the ratio of isomers significantly, according to their electron-withdrawing or electron-donating nature.[1497]



(**252**)



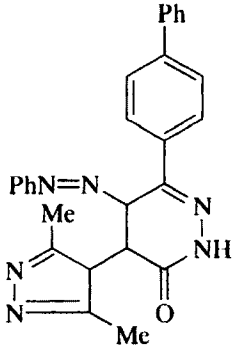
(**253**) (**254**) (**255**)



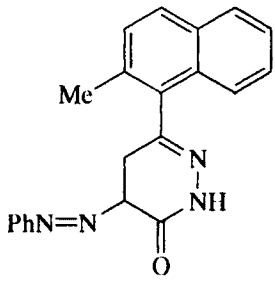
(**256**)

## 7.5 PREPARATION AND REACTIONS OF ARYLAZOPYRIDAZINES (*H* 632)

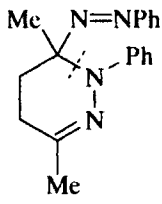
PREPARATIVE ROUTES Most of the known arylazopyridazines have been made by *primary syntheses*[70,80,150,482] (see also Chapters 1 and 2), by *oxidation of (β-substituted hydrazino)pyridazines* (see Section 7.3.2), or by *diazo coupling*, as exemplified in the conversion of 6-(biphenyl-4-yl)-4-(3,5-dimethylpyrazol-4-yl)-4,5-dihydro-3(2*H*)-pyridazinone into 6-(biphenyl-4-yl)-4-(3,5-dimethylpyrazol-4-yl)-5-phenylazo-4,5-dihydro-3(2*H*)-pyridazinone (**257**) in 63% yield by treatment with a solution of benzenediazonium chloride and sodium acetate at <5° for 1 h;[1712] and in the somewhat similar formation of 6-(2-methylnaphthalen-1-yl)-4-phenylazo-4,5-dihydro-3(2*H*)-pyridazinone (**258**) in 55% yield.[837]




(257)




(258)




(259)

[H] → MeC(=NNPh)$CH_2CH_2$C(=NNHPh)Me

(260)

REACTIONS An example of *reduction to a hydrazinopyridazine* has been given in Section 7.3.1. The *ring fission* of arylazopyridazines is exemplified in the hydrogenation of 3,6-dimethyl-1-phenyl-6-phenylazo-1,4,5,6-tetrahydropyridazine (**259**) over a platinum catalyst in acetic acid to give 2,5-hexanedione bisphenylhydrazone (**260**) in 94% yield;[69] several other such fissions have been reported.[69,70]

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D. J. Brown
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CHAPTER 8

# Pyridazinecarboxylic Acids and Related Derivatives (*H* 353, 407)

This chapter covers not only pyridazinecarboxylic acids but also the related esters, acyl halides, amides, nitriles, aldehydes, ketones, and any thio analogues; no isocyanato- or isothiocyanatopyridazines appear to have been investigated in recent years. The direct introduction of "carbon functional groups" into pyridazines has been reviewed.[227] To avoid repetition, interconversions of the foregoing types of pyridazine are discussed only at the first appropriate opportunity: for example, the formation of esters from carboxylic acids is described as a reaction of carboxylic acids rather than as a preparative route to esters, simply because the section on carboxylic acids precedes that on esters. Extensive cross-references have been inserted in order to alleviate any confusion.

## 8.1 PYRIDAZINECARBOXYLIC ACIDS (*H* 407)

As well as regular pyridazinecarboxylic acids, this section includes those acids in which the carboxyl group is attached indirectly to the pyridazine nucleus, as for example, in the extranuclear acid, 3-carboxymethyl-6-phenyl-4(1*H*)-pyridazinone.

### 8.1.1 Preparation of Pyridazinecarboxylic Acids (*H* 408)

Two important routes to pyridazinecarboxylic acids have been discussed already: *by primary syntheses* (see Chapters 1 and 2) and *by oxidation of alkylpyridazines* (see Section 3.2.2.1). Other routes to pyridazinecarboxylic acids, mainly involving hydrolytic processes, are illustrated by the following classified examples:

#### By Hydrolysis of Pyridazinecarboxylic Esters

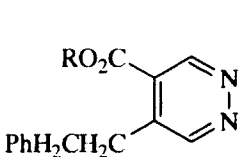
*Note*: Since many such esters have been made by independent primary syntheses, their conversion into carboxylic acids represents an important

route to the latter. The choice between alkaline or acidic hydrolysis may depend on any passenger groups present.

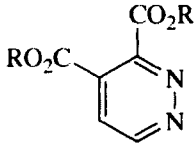
Ethyl 5-phenethyl-4-pyridazinecarboxylate (**1**, R = Et) gave 5-phenethyl-4-pyridazinecarboxylic acid (**1**, R = H) (NaOH, $H_2O$–EtOH, reflux, 2 h: 85%).[1791]

Diethyl 3,4-pyridazinecarboxylate (**2**, R = Et) gave 3,4-pyridazinedicarboxylic acid (**2**, R = H) (1M NaOH, 33°, 15 min: 68%).[1120]

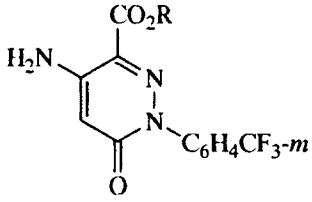
Methyl 4-amino-6-oxo-1-*m*-trifluoromethylphenyl-1,6-dihydro-3-pyridazinecarboxylate (**3**, R = Me) gave 4-amino-6-oxo-1-*m*-trifluoromethylphenyl-1,6-dihydro-3-pyridazinecarboxylic acid (**3**, R = H) (2.5M NaOH, 50° → 25°, briefly: 89%).[1117]



(1)



(2)



(3)

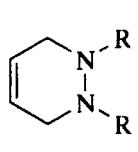
Diethyl 1,2,3,6-tetrahydro-1,2-pyridazinedicarboxylate (**4**, R = $CO_2$Et) gave, not the dicarboxylic acid (**4**, R = $CO_2$H), but its decarboxylation product, 1,2,3,6-tetrahydropyridazine (**4**, R = H) [KOH, EtOH, reflux, 3 h: 76%; further characterized as 1,2-dibenzoyl-1,2,3,6-tetrahydropyridazine (**4**, R = Bz).[405]

Methyl 5-cyano-4-ethyl-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate (**5**, R = Me) gave 5-cyano-4-ethyl-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylic acid (**5**, R = H) (HCl–AcOH, reflux, 1 h: 91%).[1808]

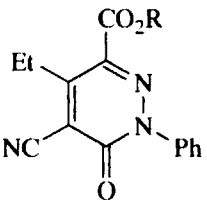
3-Methoxycarbonylmethyl- (**6**, R = Me) gave 3-carboxymethyl-6-phenyl-4(1*H*)-pyridazinone (**6**, R = H) (HCl, $H_2O$–dioxane, reflux, 2 h: 87%).[1489]

Rate constants for the alkaline hydrolysis of methyl 4-pyridazinecarboxylate (**7**) have been determined in 80% methanol and compared with those for comparable diazine esters.[101]

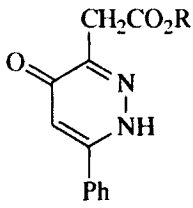
Also other examples.[51,52,55,81,160,314,389,513,534,775,821,864,947,1049,1090,1118,1217,1232,1240,1242,1268,1309,1378,1419,1425,1434,1457,1459,1460,1635,1742,1767,1964]



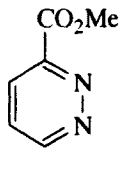
(4)



(5)



(6)

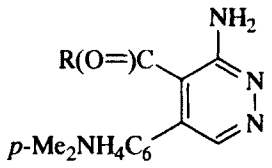


(7)

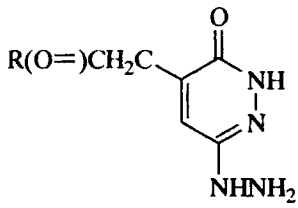
## By Hydrolysis of Pyridazinecarboxamides or Pyridazinecarbohydrazides

3-Amino-5-*p*-dimethylaminophenyl-4-pyridazinecarboxamide (**8**, R = $NH_2$) gave 3-amino-5-*p*-dimethylaminophenyl-4-pyridazinecarboxylic acid (**8**, R = OH) (NaOH, EtOH–$H_2O$, 120°, sealed, 13 h: 80%).[1099]

6-Hydrazino-4-hydrazinocarbonylmethyl- (**9**, R = $NHNH_2$) gave 4-carboxymethyl-6-hydrazino-3(2*H*)-pyridazinone (**9**, R = OH) (6 M HCl, reflux. 3 h: 70%, isolated as hydrochloride).[947]



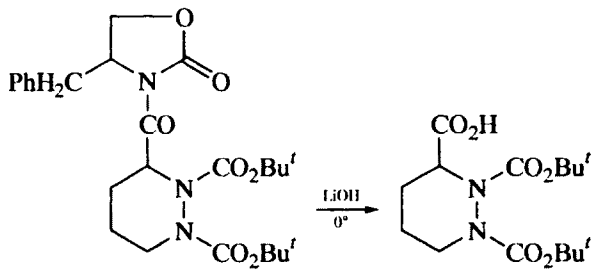

(**8**)




(**9**)

Di-*t*-butyl 3-(4-benzyl-2-oxo-oxazolidin-3-ylcarbonyl)hexahydro-1,2-pyridazinedicarboxylate (**10**) gave 1,2-di-*t*-butoxycarbonylhexahydro-3-pyridazinecarboxylic acid (**11**) (LiOH, THF–$H_2O$, < 0°, 2 h: 89%; saponification of the passenger ester grouping was avoided under these gentle conditions).[1803]

Also other examples.[587, 1365, 1434]



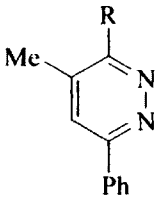

(**10**) (**11**)

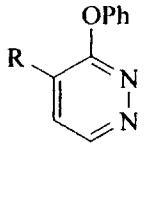
## By Hydrolysis of Pyridazinecarbonitriles

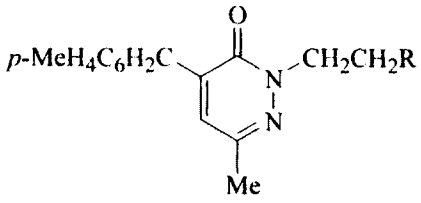
4-Methyl-6-phenyl-3-pyridazinecarbonitrile (**12**, R = CN) gave 4-methyl-6-phenyl-3-pyridazinecarboxylic acid (**12**, R = $CO_2H$) (NaOH, $H_2O$–EtOH, 95°, 8 h: ~75%).[1104]

3-Phenoxy-4-pyridazinecarbonitrile (**13**, R = CN) gave 3-phenoxy-4-pyridazinecarboxylic acid (0.5M NaOH, 60°, 3 h: 90%).[517]

2-β-Cyanoethyl- (**14**, R = CN) gave 2-β-carboxyethyl-6-methyl-4-*p*-methylbenzyl-3(2*H*)-pyridazinone (**14**, R = $CO_2H$) (5M NaOH, reflux, 6 h: 88%).[112]


(12)

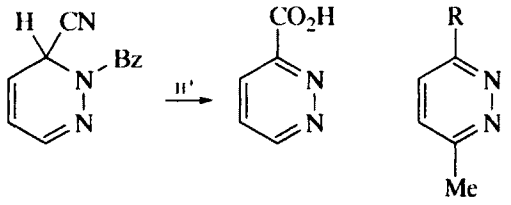

(13)


(14)

The Reissert product, 2-benzoyl-2,3-dihydro-3-pyridazinecarbonitrile (**15**), gave 3-pyridazinecarboxylic acid (**16**) (HBr–AcOH, reflux, 15 min: 24%; by hydrolysis and loss of PhCHO?).[1066]

6-Methyl-3-pyridazinecarbonitrile (**17**, R = CN) gave 6-methyl-3-pyridazinecarboxylic acid (**17**, R = $CO_2H$) (immolized nitrilase from *Rhodococcus* sp. pH 7 phosphate buffer, 25°, 12 days: 70%).[1500,1684]

Also other examples.[389,534,587,775,911,1256,1791]


(15) (16) (17)

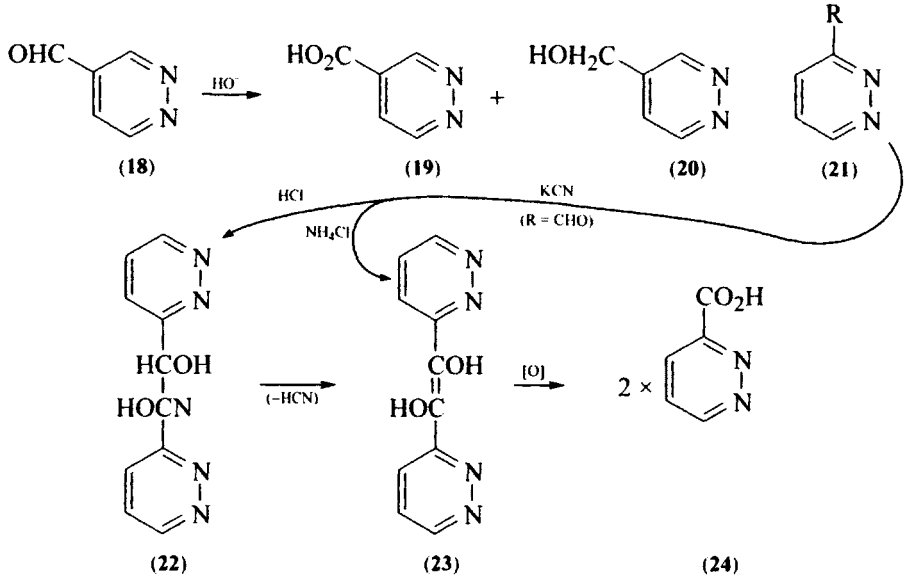
## By Oxidation of Pyridazinecarbaldehydes

6-Oxo-3-phenyl-1,6-dihydro-4-pyridazinecarbaldehyde gave the corresponding 4-carboxylic acid ($AgNO_3$, EtOH–$H_2O$, pH 12, 20°, 16 h: 50%).[2018]

4-Pyridazinecarbaldehyde (**18**) underwent a Cannizzaro reaction[1852e] to give 4-pyridazinecarboxylic acid (**19**) and 4-hydroxymethylpyridazine (**20**) (KOH, $H_2O$, 25°, 3 days: 82% and 79%, respectively);[67] 3-pyridazinecarbaldehyde (**21**, R = CHO) likewise gave 3-pyridazinecarboxylic acid (**21**, R = $CO_2H$) and 3-hydroxymethylpyridazine (**21**, R = $CH_2OH$) (KOH, $H_2O$, reflux, 5 h: 19% and 78%, respectively; perhaps most of the carboxylic acid underwent decarboxylation under these conditions?).[77]

3-Pyridazinecarbaldehyde (**21**, R = CHO) underwent benzoin-type reactions to give either 2,3-dihydroxy-2,3-di(pyridazin-3-yl)propiononitrile (**22**) (KCN, HCl, $H_2O$–dioxane, 0°, 12 h: 37%) or 1,2-di(pyridazin-3-yl)-1,2-ethenediol (**23**, or tautomer) (KCN, $NH_4Cl$ $Et_2O$–$H_2O$, 25°, 10 min: 80%), which was oxidized easily to 3-pyridazinecarboxylic acid (**24**) (e.g., $O_2$, MeOH, 25°, 12 h: >95%).[76]

Also other examples.[1111]

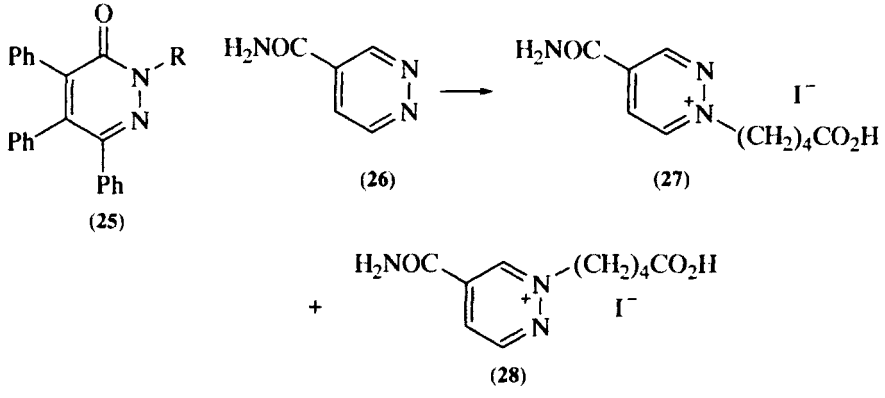



## By Passenger Introduction of Carboxyl Groups

*Note*: Routes to extranuclear carboxypyridazines by passenger introduction of carboxy groups have been mentioned in several sections of this book; a couple of yet unmentioned examples follow.

4,5,6-Triphenyl-3(2*H*)-pyridazinone (**25**, R = H) gave 2-(8-carboxyoctyl)-4,5,6-triphenyl-3(2*H*)-pyridazinone [**25**, $R = (CH_2)_8CO_2H$] [NaH, $Me_2NCHO$, 25–110° as required; then $C_8H_{17}Br\downarrow$, 25–110° as required].[1745]

4-Pyridazinecarboxamide (**26**) gave an unseparated 4:5 mixture of 4-carbamoyl- (**27**) and 5-carbamoyl-1-δ-carboxybutylpyridazinium iodide (**28**) ($ICH_2CH_2CH_2CH_2CO_2H$, MeCN, 80°, > 18 h: 75%).[1779]



### 8.1.2 Reactions of Pyridazinecarboxylic Acids (*H* 414)

The reactions of pyridazinecarboxylic acids seem best illustrated by the following classified examples, accompanied when necessary by brief notes:

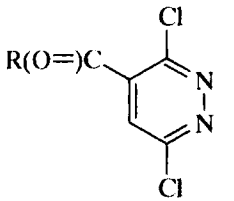
#### To Acyl Chlorides or Anhydrides

*Note*: These chlorides and anhydrides are usually employed as reactive intermediates and are seldom characterized fully.

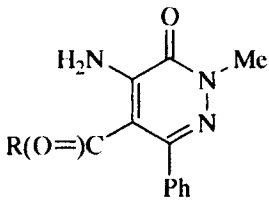
3,6-Dichloro-4-pyridazinecarboxylic acid (**29**, R = OH) gave 3,6-dichloro-4-pyridazinecarbonyl chloride (**29**, R = Cl) ($SOCl_2$, trace $Me_2NCHO$, reflux until HCl↑ and $SO_2$↑ ceased: 90%).[519]

5-Amino-1-methyl-6-oxo-3-phenyl-1,6-dihydro-4-pyridazinecarboxylic acid (**30**, R = OH) gave the corresponding crude carbonyl chloride (**30**, R = Cl) (neat $SOCl_2$, 60°, 30 min) and then 5-amino-1-methyl-6-oxo-3-phenyl-1,6-dihydro-4-pyridazinecarboxamide (**30**, R = $NH_2$) (30% $NH_4OH$, 5°, 1 h: 61% overall);[1832] also analogous examples.[587,1465]

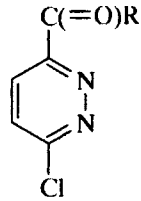
6-Chloro-3-pyridazinecarboxylic acid (**31**, R = OH) gave crude 6-chloro-3-pyridazinecarbonyl chloride (**31**, R = Cl) (neat $SOCl_2$, reflux, A, 2 h) and then 3-benzoyl-6-chloropyridazine (**31**, R = Ph) ($AlCl_3$, PhH, 0° → reflux, 6 h: 89% overall).[1689]



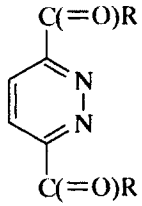
(29)



(30)

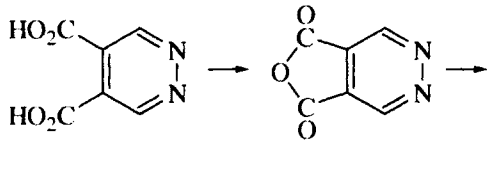


(31)

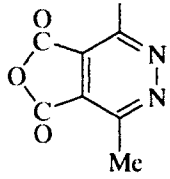


(32)

3,6-Pyridazinedicarboxylic acid (**32**, R = OH) gave 3,6-pyridazinedicarbonyl dichloride (**32**, R = Cl) [PCls, $SOCl_2$, reflux(?): 76%;[435] ClCOCOCl, PhH, reflux, 30 min: 89%;[2091] neat $SOCl_2$, trace $Me_2NCHO$, reflux, 3 h: 98% yield of crude][1545,1939] and then 3,6-dibenzoylpyridazine (**32**, R = Ph) ($AlCl_3$, PhH, 25° → 65°, 5 h: 60%).[1545,1939]



(33) (34) (35)



(36)

4,5-Pyridazinedicarboxylic acid (**33**) gave 4,5-pyridazinedicarboxylic anhydride (**34**) ($C_6H_{11}N{=}C{=}NC_6H_{11}$, THF, 25°, 12 h: 90%).[958] and thence 5-carbamoyl-4-pyridazinecarboxylic acid (**35**, R = $NH_2$) ($NH_3\downarrow$, THF, 25°: 90%)[958] or 5-benzoyl-4-pyridazinecarboxylic acid (**35**, R = Ph) ($AlCl_3$, PhH, 25° → 75°, 18 h: 67%).[702]

3,6-Dimethyl-4,5-pyridazinedicarboxylic acid gave its anhydride (**36**) (neat $Ac_2O$, reflux, briefly: 82%).[927]

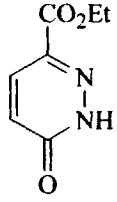
### To Esters or Lactones

*Note*: Most of the convention routes from acids to esters are represented in the pyridazine series.

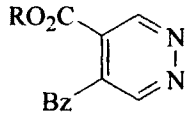
6-Oxo-1,6-dihydro-3-pyridazinecarboxylic acid (**37**, R = H) gave ethyl 6-oxo-1,6-dihydro-3-pyridazinecarboxylate (**37**, R = Et) (EtOH, HCl gas↓, 5°, then reflux, 14 h: 53%).[1459]

6-Amino-3-pyridazinecarboxylic acid gave isopropyl 6-amino-3-pyridazinecarboxylate ($Pr^iOH$, trace 10M HCl, PhH, reflux with Dean–Stark water removal, 24 h; then repeat: 63%).[1689]

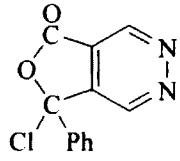
5-Benzoyl-4-pyridazinecarboxylic acid (**38**, R = H) gave methyl 5-benzoyl-4-pyridazinecarboxylate (**38**, R = Me) ($CH_2N_2$, $Et_2O$, 25°, 12 h: 91%)[702] or the lactone (**39**) of 5-(α-chloro-α-hydroxybenzyl)-4-pyridazinecarboxylic acid [neat $SOCl_2$, reflux, 4 h: 90%; lactones of 5-(α,*p*-dichloro-α-hydroxybenzyl)-4-pyridazinecarboxylic acid and other analogues similarly].[46]



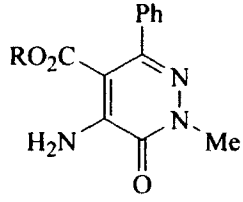
(**37**)



(**38**)



(**39**)



(**40**)

5-Amino-1-methyl-6-oxo-3-phenyl-1,6-dihydro-4-pyridazinecarboxylic acid (**40**, R = H) gave the corresponding methyl pyridazinecarboxylate (**40**, R = Me) (MeI, $K_2CO_3$, $Me_2NCHO$, 25°, 45 min: 65%).[1832]

3,6-Pyridazinedicarboxylic acid (**41**, R = H) gave dimethyl 3,6-pyridazine dicarboxylate (**41**, R = Me) [MeOH, $SOCl_2\downarrow$, −30° → 25°, 77%; substrate $Na_2$ salt (**41**, R = Na), likewise: 69%].[1077]

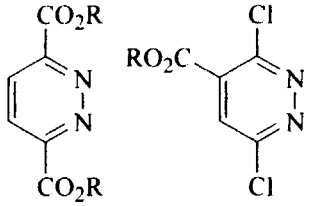
3,6-Dichloro-4-pyridazinecarboxylic acid (**42**, R = H) gave ethyl 3,6-dichloro-4-pyridazinecarboxylate (**42**, R = Et) (EtOH, 4-$Me_2N$–pyridine, $Me_2NCH_2CH_2CH_2N{=}C{=}NEt \cdot HCl$, THF, 25°, 12 h: 61%; note survival or the Cl substituents).[1748]

4-Pyridazinecarboxylic acid (**43**, R = H) gave methyl 4-pyridazinecarboxylate (**43**, R = Me) [1-1′-carbonyldiimidazole, $CH_2Cl_2$, 25°, A, 90 min;

then MeONa/MeOH↓, 25°, 2 h: 60%; presumably via unisolated 4-(imidazol-1-ylcarbonyl)pyridazine].[1347]

1-(2,4-Dinitrophenyl)-5-hydroxyhexahydro-3-pyridazinecarboxylic acid gave 2-acetyl-1-(2,4-dinitrophenyl)hexahydro-3,5-pyridazinecarbolactone (**44**) (neat $Ac_2O$, 25°, 48 h: ~55%; note *N*-acylation additional to lactone formation).[1805]

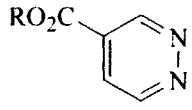
Also other examples.[101, 207, 736, 778, 945, 1066, 1104, 1165, 1264, 1555, 1778]



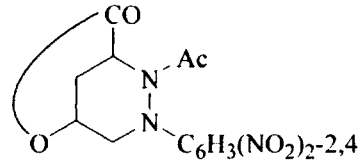

(41)




(42)




(43)

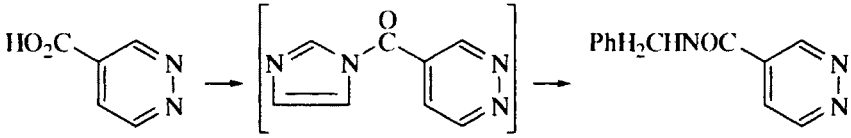



(44)

## To Amides or Hydrazides

*Note*: Pyridazinecarboxylic acids are usually converted into the corresponding amides indirectly via esters (see Section 8.2.2), via acyl chlorides (see preceding examples in this section), or other intermediates (examples following). The rarely used direct transformation of acids to amides is also examplified here.

4-Pyridazinecarboxylic acid (**45**) gave *N*-benzyl-4-pyridazinecarboxamide (**47**) via the unisolated intermediate (**46**) (1,1′-carbonyldiimidazole, $CH_2Cl_2$, 25°, 3 h; then $PhCH_2NH_2$↓, 25°, 1 h: 71%).[1609]

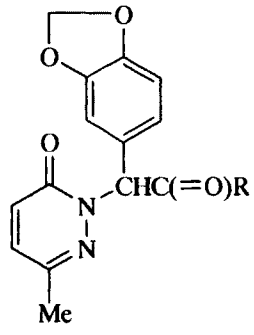



(45) (46) (47)

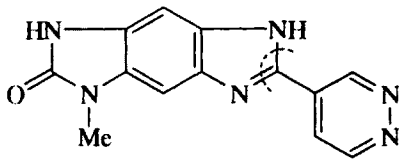
2-[α-(1,3-Benzodioxol-5-yl)-α-carboxymethyl]-6-methyl-3(2*H*)-pyridazinone (**48**, R = OH) gave 2-[α-(*N*-benzenesulfonylcarbamoyl)-α-(1,3-benzodioxol-5-yl)methyl]-6-methyl-3(2*H*)-pyridazineone (**48**, R = $PhSO_2NH$) (1,1′-carbonyldiimidazole, THF, 60°, 1 h; then $PhSO_2NH_2$↓, 1,5-diazabicyclo[5.4.0]undec-7-ene↓, 60°, 3 h: ?%); analogues likewise.[1964]

4-Pyridazinecarboxylic acid and 5,6-diamino-1-methylbenzimidazol-2(3*H*)-one afforded 1-methyl-6-pyridazin-4′-yl-3,5-dihydrobenzo[1,2-*d*:4,5-*d*′]diimidazol-2(1*H*)-one (**49**) (neat crude substrate, polyphosphoric acid, 150°, 1 h: >14%; presumably by ring closure of an intermediate amide).[1320]

Also other examples.[289, 1473, 1511, 1736, 1787, 1814]



(48)



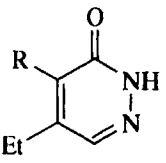
(49)

## Decarboxylation

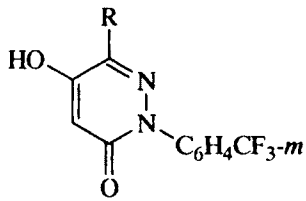
*Note*: For decarboxylations leading to pyridazine or simple alkylpyridazines, see Sections 3.1.1 and 3.2.1.4, respectively. Most other recently reported decarboxylations in the pyridazine series have been done by simple heating, dry distillation in a vacuum, or passing vapor through a hot tube; however, a few have been done in solution. Such processes are illustrated here.

5-Ethyl-3-oxo-2,3-dihydro-4-pyridazinecarboxylic acid (**50**, R = $CO_2H$) gave 5-ethyl-3(2*H*)-pyridazinone (**50**, R = H) (neat, 210°, 30 min: 33%);[821] also analogues somewhat similarly.[1099,1240,1242]

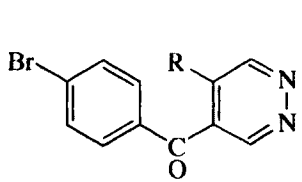
4-Hydroxy-6-oxo-1-*m*-trifluoromethylphenyl-1,6-dihydro-3-pyridazinecarboxylic acid (**51**, R = $CO_2H$) gave 5-hydroxy-2-*m*-trifluoromethylphenyl-3(2*H*)-pyridazinone (**51**, R = H) (neat, 200° until $CO_2\uparrow$ ceased: 64%);[1090] analogues likewise.[1090]



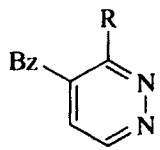
(50)



(51)



(52)



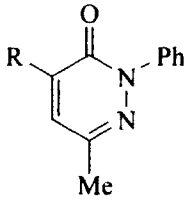
(53)

5-*p*-Bromobenzoyl-4-pyridazinecarboxylic acid (**52**, R = $CO_2H$) gave 4-*p*-bromobenzoylpyridazine (**52**, R = H) (Kugelrohr, 245°, vacuum, 20 min: 44%; Kugelrohr, Cu powder, 120°, vacuum, 30 min: 47%);[513] also analogous examples.[53,81]

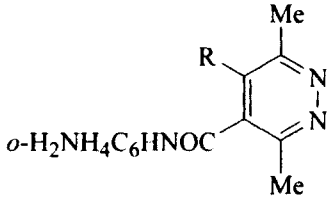
5-Benzoyl-3-pyridazinecarboxylic acid (**53**, R = $CO_2H$) gave 4-benzoylpyridazine (**53**, R = H) (Kugelrohr, 150°, vacuum, 3 h: 90%).[1066]

6-Methyl-3-oxo-2-phenyl-2,3-dihydro-4-pyridazinecarboxylic acid (**54**, R = $CO_2H$) gave 6-methyl-2-phenyl-3(2*H*)-pyridazinone (**54**, R = H) (sublimation at 135°/vacuum into a tube at 550° during 2 h: 86%);[715] analogues likewise (550–850°).[715,719]

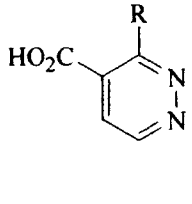
5-*N*-(*o*-Aminophenyl)carbamoyl-3,6-dimethyl-4-pyridazinecarboxylic acid (**55**, R = $CO_2H$) gave *N*-(*o*-aminophenyl)-3,6-dimethyl-4-pyridazinecarboxamide (**55**, R = H) (MeOH, reflux, 18 h: ~75%).[656]



(**54**)



(**55**)



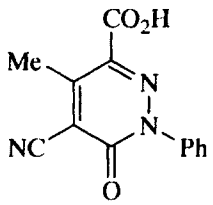
(**56**)

3,4-Pyridazinedicarboxylic acid (**56**, R = $CO_2H$) gave only 4-pyridazin carboxylic acid (**56**, RH = H) (1-methyl-2-pyrrolidinone, 170° until $CO_2\uparrow$, ceased: >95%; note preferential lability of the 3-carboxy group).[1120]

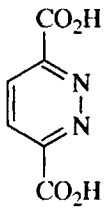
Also miscellaneous examples.[92,1148,1232]

## Ionization and Complex Formation

5-Cyano-4-methyl-6-oxo-1,6-dihydro-3-pyridazinecarboxylic acid (**57**) had p$K_a$ 3.42, enhanced through electron withdrawal by the CN group.[1368]



(**57**)

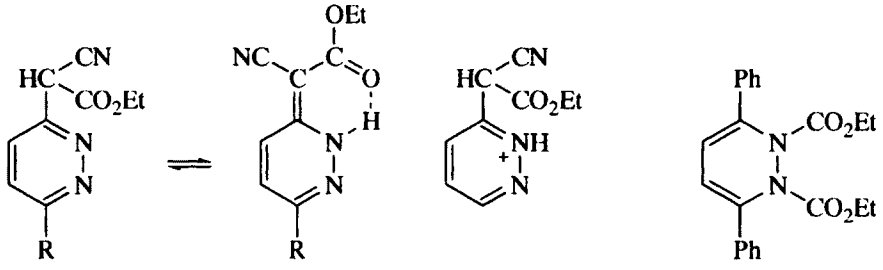


(**58**)

3,6-Pyridazinedicarboxylic acid (**58**) formed well-characterized hydrated dimetal–dinuclear complexes with Ni(II) and CO(II);[1963] however, Mn(II) formed an hydrated monometal–dinuclear complex characterized by X-ray analysis.[1914 cf. 2071]

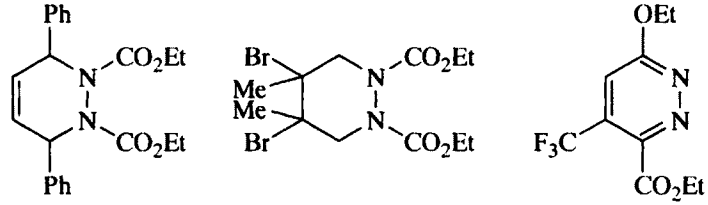
## 8.2 PYRIDAZINECARBOXYLIC ESTERS (*H* 418)

This section covers both nuclear and extranuclear pyridazinecarboxylic esters. A general study of tautomerism in 3-(α-cyano-α-ethoxycarbonylmethyl)pyridazines and related compounds has revealed, for example, that the parent compound (**59**, R = H) exists in $CDCl_3$ entirely as the tautomer (**59a**, R = H) whereas 3-chloro-6-(α-cyano-α-ethoxycarbonylmethyl)pyridazine (**59**, R = Cl) exists in $CDCl_3$–isooctane as a 1 : 3 mixture of the tautomers **59** (R = Cl) and **59a** (R = Cl);[1143] in contrast, the parent cation (**60**) exists in sulfuric acid entirely as such.[1482] These equilibria have been studied further by



(59) (59a) (60) (61)

a molecular orbital pertubation method, and the results agree well with experimental data.[571] The conformations of diethyl 3,6-diphenyl-1,2-dihydro-1,2-pyridazinedicarboxylate (**61**), diethyl 3,6-diphenyl-1,2,3,6-tetrahydro-1,2-pyridazinedicarboxylate (**62**), and diethyl 4,5-dibromo-4,5-dimethylhexahydro-1,2-pyridazinedicarboxylate (**63**) have been determined in the solid state by X-ray analyses[100] and in solution by $^{13}C$ nmr data.[419, cf. 2083] Data for C–H coupling constants between the $CF_3$ and adjacent ring proton in compounds such as ethyl 6-ethoxy-4-trifluoromethyl-3-pyridazinecarboxylate (**64**) have been reported.[1980]



(62) (63) (64)

### 8.2.1 Preparation of Pyridazinecarboxylic Esters (*H* 414)

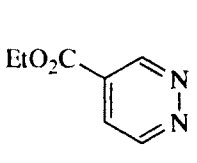
A high proportion of pyridazinecarboxylic esters have been made by *primary syntheses* (see Chapters 1 and 2), *rare reductive alkoxycarbonylations* (see

Section 3.1.3 for examples), or *esterification of carboxylic acids* (see Section 8.1.2). Other routes to such esters are illustrated in the following classified examples:

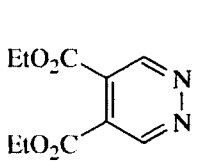
### By Direct Homolytic Alkoxycarbonylation

*Note*: Simple pyridazines undergo such alkoxycarbonylation easily to give a mixture of products in which 4/5-carboxylic esters usually predominate.[416]

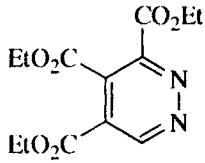
Pyridazine gave a separable mixture, mainly of ethyl 4-pyridazinecarboxylate (**65**) and diethyl 4,5-pyridazinedicarboxylate (**66**) ($AcCH_2CO_2Et$, 30% $H_2O_2$, $-5°$, 15 min; then added to substrate $+H_2SO_4+FeSO_4+H_2O+CH_2Cl_2$, $-5°$, 15 min: 15% and 76%, respectively) or mainly of diethyl 4,5-pyridazinedicarboxylate (**66**) and triethyl 3,4,5-pyridazinetricarboxylate (**67**) (likewise but without $CH_2Cl_2$: 49% and 19%, respectively).[416]



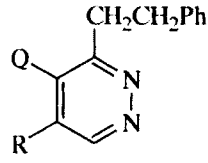
(**65**)



(**66**)



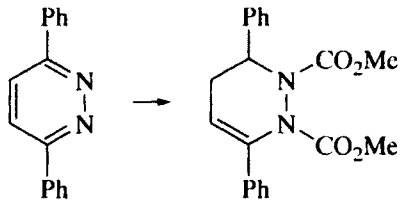
(**67**)



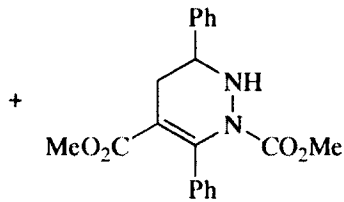
(**68**)

3-Phenethylpyridazine (**68**, Q = R = H) gave mainly methyl 6-phenethyl-4-pyridazinecarboxylate (**68**, $Q=CO_2Me$, R = H) and dimethyl 3-phenethyl-4,5-pyridazinedicarboxylate (**68**, $Q=R=CO_2Me$) (likewise using $AcCH_2CO_2Me$ *with* $CH_2Cl_2$: 14% and 31%, after separation).[1791]

Also many other fascinating examples, some including the formation of dihydro-1/2-ethoxycarbonyl derivatives.[228,416,420]



(**69**) (**70**) (**71**)

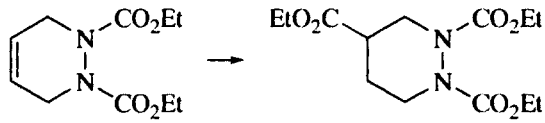


(**72**)

### By Indirect Alkoxycarbonylation

3,6-Diphenylpyridazine (**69**) gave a mixture of dimethyl 3,6-diphenyl-1,2,3,4-tetrahydro-1,2-pyridazinedicarboxylate (**70**), dimethyl 3,6-diphenyl-1,2,5,6-tetrahydro-1,4-pyridazinedicarboxylate (**71**), and dimethyl 3,6-diphenyl-1,2,3,4-tetrahydro-1,5-pyridazinedicarboxylate (**72**) (electrolysis, $CO_2$, MeI, see original paper for details: 12%, 35%, and 10%, respectively).[185]

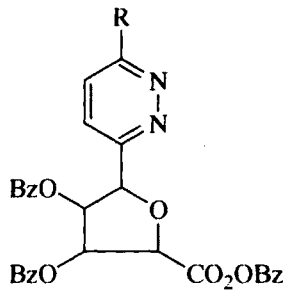
Diethyl 1,2,3,6-tetrahydro-1,2-pyridazinedicarboxylate (**73**) gave triethyl hexahydro-1,2,4-pyridazinetricarboxylate (**74**) [$PdCl_2$ $(PPh_3)_2$, EtOH–PhH, P(Co); 100 atm, 20°, 4 days; minimal details].[1646, cf. 2007]



(73) (74)

### From Pyridazinecarbaldehydes

6-(2′,3′,5′-Tri-*O*-Benzoyl-β-D-ribofuranosyl)-3-pyridazinecarbaldehyde (**75**, R = CHO) gave ethyl 6-(2′,3′,5′-tri-*O*-benzoyl-β-D-ribofuranosyl)-3-pyridazinecarboxylate (**75**, R = $CO_2Et$) (KOH, EtOH, $O_3\downarrow$, −78°, 7 h: 65%).[1539]

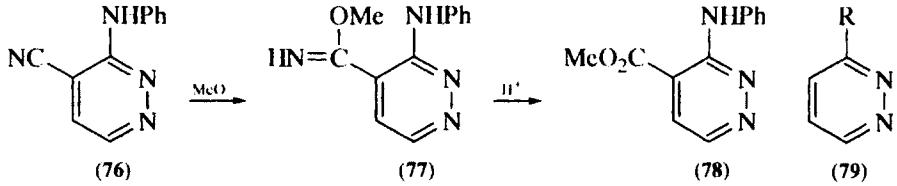


(75)

### From Pyridazinecarbonitriles or Pyridazinecarboxamides

3-Anilino-4-pyridazinecarbonitrile (**76**) gave methyl 3-anilino-4-pyridazinecarboximidate (**77**) (NaOH, MeOH, 5°, 24 h: 86%) and thence methyl 3-anilino-4-pyridazinecarboxylate (**78**) (dilute HCl, 25°, 10 min: 67%).[1079]

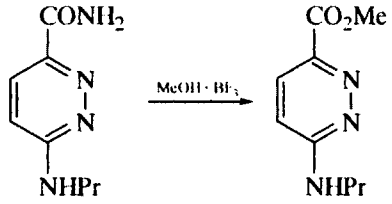
3-Pyridazinecarbonitrile (**79**, R = CN) gave ethyl 3-pyridazinecarboxylate (**79**, R = $CO_2Et$) (EtOH, 2M NaOH, to pH 8 25°, 3 h; then evaporate, HCl to pH 3, <5°, 5 h: 95%), a one-pot reaction, presumably via [**79**, R = C[ : NH]OEt].[1347]



(76) (77) (78) (79)

6-Propylamino-3-pyridazinecarboxamide (**80**) gave methyl 6-propylamino-3-pyridazinecarboxylate (**81**) ($BF_3 \cdot Et_2O$, MeOH, 105°, sealed, ~20 h: ~75%).[1592]

Also other examples.[1229, 1592, 1773]

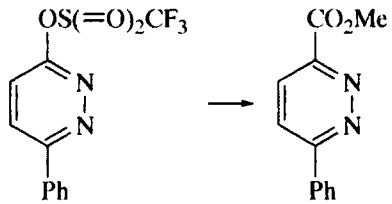


(80) (81)

## By Miscellaneous Procedures

*Note*: Some typical passenger introductions are included with these procedures.

3-Phenyl-6-trifluoromethanesulfonyloxypyridazine (**82**) gave methyl 6-phenyl-3-pyridazinecarboxylate (**83**) [$Pd(OAc)_2$, 1,1′-bis(diphenylphosphino)ferrocene, MeOH–$Me_2NCHO$, CO↓, 50°, 12 h: 86%];[1907] also analogues similarly.[1907]



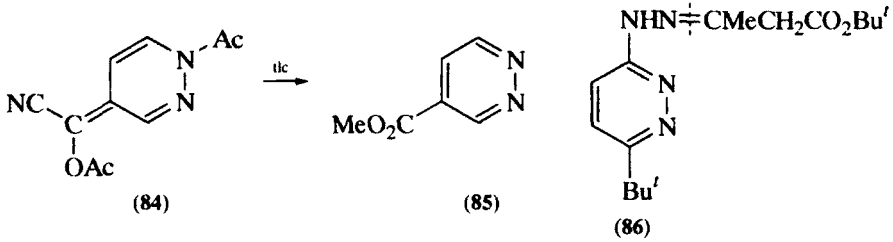
(82) (83)

4-(α-Acetoxy-α-cyanomethylene)-1-acetyl-1,4-dihydropyridazine (**84**) gave methyl 4-pyridazinecarboxylate (**85**) [by chromatography on a Kieselgel plate (Merck, Darmstadt): 56%; see original paper for details and discussion of mechanism].[959]

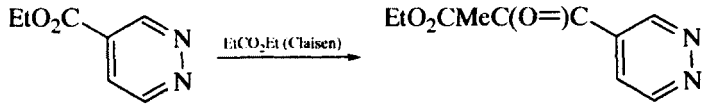
3-*t*-Butyl-6-hydrazinopyridazine gave 3-(β-*t*-butoxycarbonyl-α-methylethylidene)hydrazino-6-*t*-butylpyridazine (**86**) [$AcCH_2Co_2Bu^t$, 4Å molecular sieves, PhH, reflux with Dean–Stark trap, 13 h: 93%].[18]

Ethyl 4-pyridazinecarboxylate (**87**) underwent a Claisen reaction with ethyl propionate to give 4-(2-ethoxycarbonylpropionyl)pyridazine (**88**) (EtONa, PhH, reflux, 20 h: 43%);[79] see Section 8.2.2 for more such reactions.

Also other examples.[266,753,996]




(84) (85) (86)



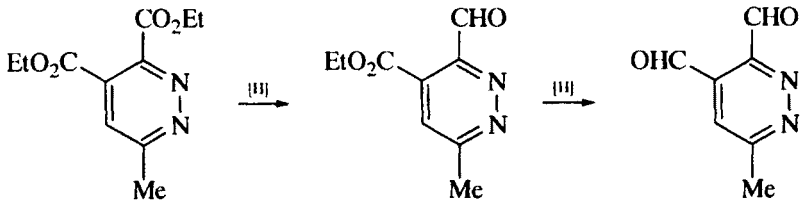

(87) (88)

## 8.2.2 Reactions of Pyridazinecarboxylic Esters (*H* 418)

Pyridazinecarboxylic esters undergo *hydrolysis to corresponding carboxylic acids* (see Section 8.1.1) as well as other expected reactions quite conveniently, as illustrated in the following classified examples:

### Reduction to Pyridazinecarbaldehydes, Hydroxymethylpyridazines, or Methylpyridazines

Diethyl 6-methyl-3,4-pyridazinedicarboxylate (**89**) gave only ethyl 3-formyl-6-methyl-4-pyridazinecarboxylate (**90**) ($Bu^i_2AlH$, THF, −70°: 66%)[996] or 6-methyl-3,4-pyridazinedicarbaldehyde (**91**) ($LiAlH_4$?, no details).[907]

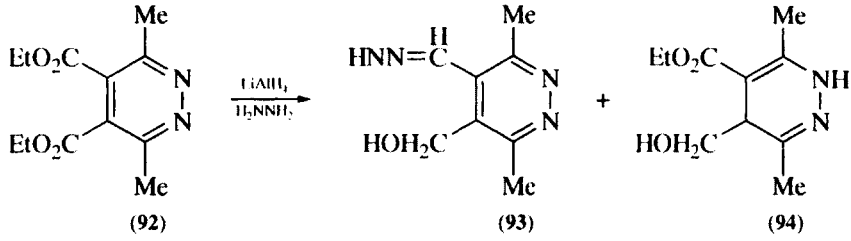


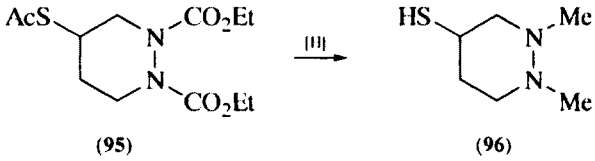

(89) (90) (91)

Diethyl 3,6-dimethyl-4,5-pyridazinecarboxylate (**92**) gave 5-hydroxymethyl-3,6-dimethyl-4-pyridazimecarbaldehyde hydrazone (**93**), ethyl 5-hydroxymethyl-3,6-dimethyl-4-pyridazinecarboxylate (**94**), and other products ($LiAlH_4$, THF, −70°, $N_2$, 90 min; hydrolysis; $H_2NNH_2$, MeOH, −70° → 20°, 14 h: ~4% and ~20%, respectively),[927]

Diethyl 4-acetylthiohexahydro-1,2-pyridazinedicarboxylate (**95**) gave 1,2-dimethylhexahydro-4-pyridazinethiol (**96**) ($LiAlH_4$, $Et_2O$, 20°, 2 h: 54%);[970] also analogues.[970]

Also other examples.[702] See also Section 5.3.1 for further data on the formation of hydroxymethylpyridazines.



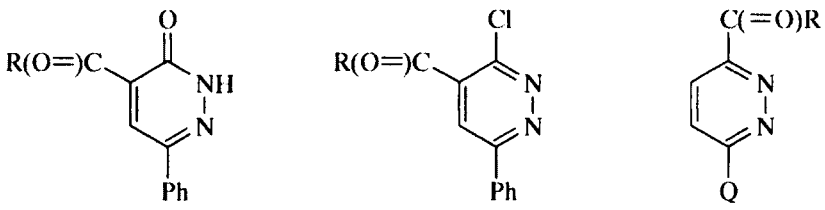




**To Pyridazinecarboxamides or Pyridazinecarbohydrazides**

*Note*: Pyridazine esters have been used extensively to prepare the corresponding amides or hydrazides, mainly by simple treatment with an appropriate amine.

Ethyl 3-oxo-6-phenyl-2,3-dihydro-4-pyridazinecarboxylate (**97**, R = OEt) gave 3-oxo-6-phenyl-2,3-dihydro-4-pyridazinecarboxamide (**97**, R = $NH_2$) ($NH_3$–MeOH, 20°, 12 h: 68%).[1041]

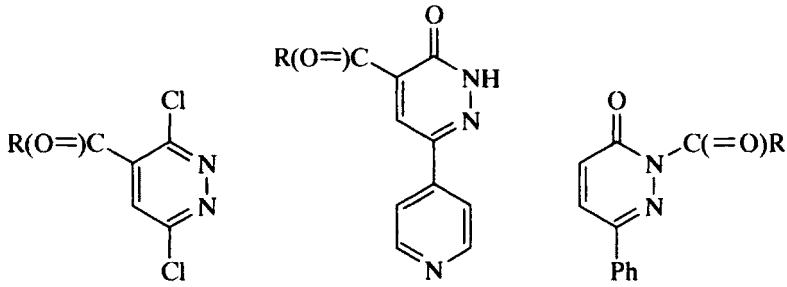
Ethyl 3-chloro-6-phenyl-4-pyridazinecarboxylate (**98**, R = OEt) gave 3-chloro-6-phenyl-4-pyridazinecarboxamide (**98**, R = $NH_2$) ($NH_4OH$, 20°, 12 h: 84%; note survival of the Cl substituent).[1319]




In contrast, pivaloylmethyl 6-chloro-3-pyridazinecarboxylate (**99**, Q = Cl, R = $OCH_2COBu^t$) gave 6-amino-3-pyridazinecarboxamide (**99**, Q = R = $NH_2$) (liquid $NH_3$, 100°, sealed, 22 h: 90%; note aminolysis of the Cl substituent).[1689]

Ethyl 3,6-dichoro-4-pyridazinecarboxylate (**100**, R = OEt) gave only 3,6-dichloro-4-pyridazinecarboxamide (**100**, R = $NH_2$) ($NH_4OH$, 5°, 12 h: ~85%; note survival of the Cl substituents).[1099]

Ethyl 3-oxo-6-pyridin-4′-yl-2,3-dihydro-4-pyridazinecarboxylate (**101**, R = OEt) gave 3-oxo-6-pyridin-4-′-yl-2,3,-dihydro-4-pyridazinecarbohydrazide (**101**, R = $NHNH_2$) ($H_2NNH_2 \cdot H_2O$, EtOH, reflux, 3.5 h: 84%).[1589]



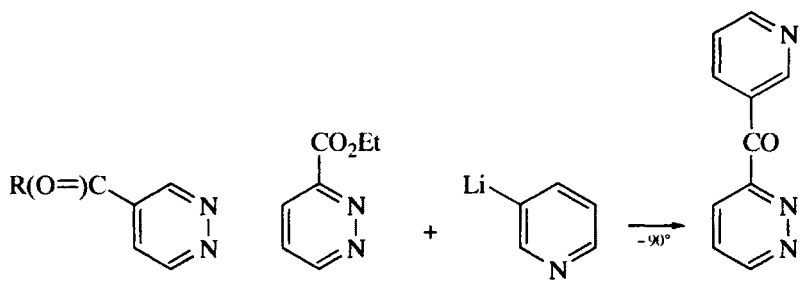

(**100**) (**101**) (**102**)

Ethyl 6-oxo-3-phenyl-1,6-dihydro-1-pyridazinecarboxylate (**102**, R = OEt) gave 6-oxo-3,*N*′-diphenyl-1,6-dihydro-1-pyridazinecarbohydrazide (**102**, R = NHNHPh) (PhNHNH$_2$, reflux, 3 h: 76%).[368]

Also many other examples.[46,96,119,686,736,775,927,947,962,974,996,1017,1056,1079,1090,1117,1164,1244 1251,1264,1268,1338,1343,1365,1434,1446,1458,1461,1539,1592,1773,1800,1801,1920,1966,2008,2054]

## To Pyridazine Ketones

Ethyl 4-pyridazinecarboxylate (**103**, R = OEt) gave 4-acetylpyridazine (**103**, R = Me) (MeLi, $Et_2O$, −30°, 1 h: 43%),[1347] 4-acetoacetylpyridazine (**103**, R = $CH_2Ac$) (AcMe, MeONa, PhH, reflux, 7 h: 30%),[1255] or 4-ethoxycarbonylacetylpyridazine (**103**, R = $CH_2CO_2Et$) (AcOEt, EtONa, PhMe, 80°, 2 h: 61% crude, 48% pure) and thence 4-acetylpyridazine (**103**, R = Me) (6M HCl, reflux, 2 h: 82%; by hydrolysis of the ester grouping and decarboxylation).[65]




(**103**) (**104**) (**105**) (**106**)

Ethyl 3-pyridazinecarboxylate (**104**) and 3-lithiopyridine (**105**) (made in situ) gave 3-nicotinoylpyridazine (**106**) ($Et_2O$–THF, $-90° \rightarrow 20°$, 12 h: 57%);[1948] analogues likewise.[1670,1679]

Also other examples.[807,1180]

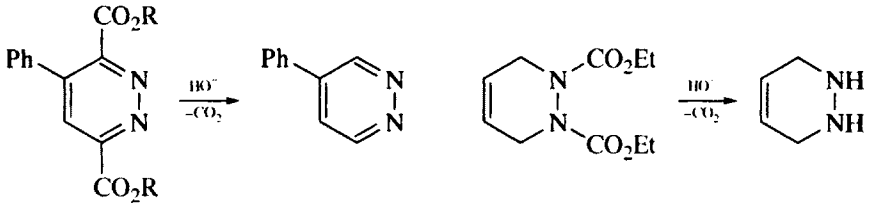
### Removal of the Ester Grouping

*Note*: The removal of an ester grouping from a pyridazine is done usually by hydrolysis and decarboxylation, sometimes in a one-pot process (cf. Sections 8.1.1 and 8.1.2, respectively).

Dimethyl 4-phenyl-3,6-pyridazinedicarboxylate (**107**, R = Me) gave 4-phenyl-3,6-pyridazinedicarboxylic acid (**107**, R = H) (NaOH, $H_2O$–THF, reflux, 12 h: 74%) and then 4-phenylpyridazine (**108**) (1,3,5-$Pr_3C_6H_3$, 200°, $N_2$, 15 min: 78%).[337]

Diethyl 1,2,3,6-tetrahydro-1,2-pyridazinedicarboxylate (**109**) gave 1,2,3,6-tetrahydropyridazine (**110**) (KOH, EtOH, reflux, 2 h: 20–50%).[629]

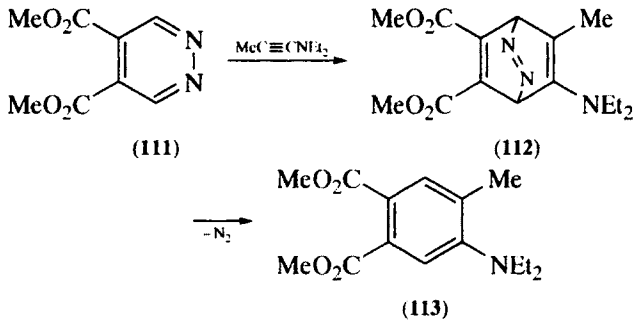
Also other examples.[612]




(**107**) (**108**) (**109**) (**110**)

### To Benzene Derivatives via Diels–Alder Adducts

Dimethyl 4,5-pyridazinedicarboxylate (**111**) gave dimethyl 4-diethylamino-5-methylphthalate (**113**) ($MeC{\equiv}CNEt_2$, MeCN, 60°, 15 min; then 20°, 3 days: 74%) by $N_2\uparrow$ from the adduct (**112**)[40]; also analogous reactions.[40,242]

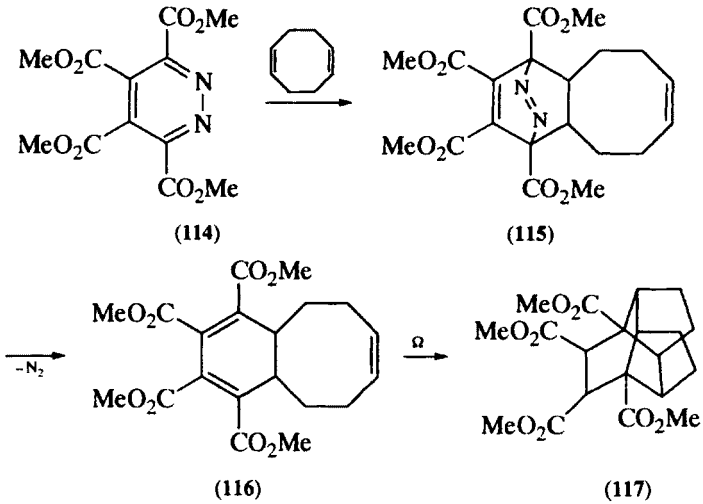



(**111**) (**112**) (**113**)

Tetramethyl 3,4,5,6-pyridazinetetracarboxylate (**114**) gave so-called tetramethyl tetracyclo[7.3.0.0$^{2,6}$0$^{5,10}$]dodec-7-ene-6,7,8,9-tetracarboxylate

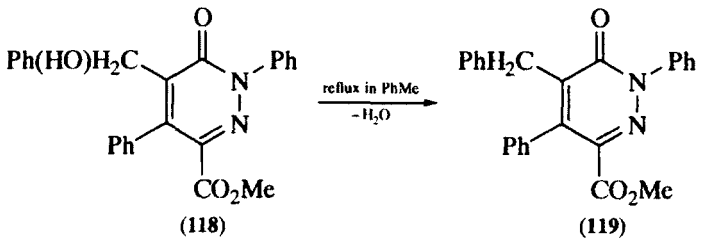
(**117**) via the unisolated intermediates **115** and **116** (cycloocta-1,5-diene, xylene, reflux, 20 h: 19%).[1537]

Also other examples.[41,1790]



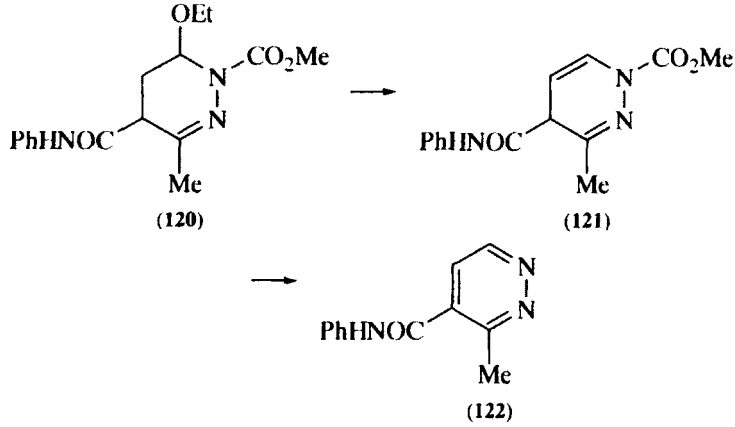

## Nuclear Oxidation or Reduction

Methyl 5-α-hydroxybenzyl-6-oxo-1,4-diphenyl-1,4,5,6-tetrahydro- (**118**) gave methyl 5-benzyl-6-oxo-1,4-diphenyl-1,6-dihydro-3-pyridazinecarboxylate (**119**) (PhMe, reflux, 12 h: ?%; by loss of $H_2O$).[391]



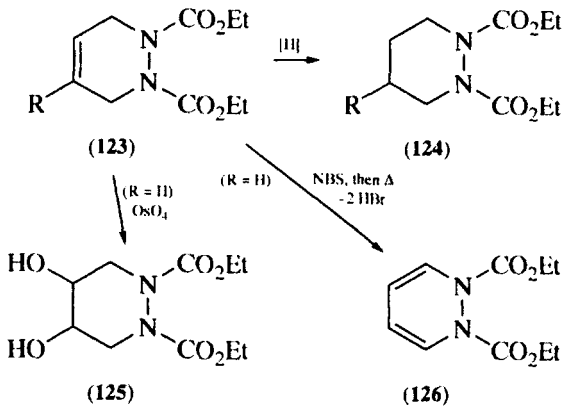

Methyl-6-ethoxy-3-methyl-4-phenylcarbamoyl-1,4,5,6-tetrahydro-1-pyridazinecarboxylate (**120**) gave mainly methyl 3-methyl-4-phenylcarbamoyl-1,4-dihydro-1-pyridazinecarboxylate (**121**) (trace $F_3CCO_2H$, $CH_2Cl_2$, 20°, 2 h: 61%; by loss of EtOH) and thence 3-methyl-*N*-phenyl-4-pyridazinecarboxamide (**122**) ($K_2CO_3$, MeOH, reflux, 12 h: 68%; presumably by hydrolysis, decarboxylation, and aerial oxidation).[1542]

Diethyl 1,2,3,6-tetrahydro- (**123**, R = H) gave diethyl hexahydro-1,2-pyridazinedicarboxylate (**124**, R = H) (Pd/C, EtOH, $H_2$: 89%);[91] diethyl 4-phenyl-1,2,3,6-tetrahydro- (**123**, R = Ph) gave diethyl 4-phenylhexahydro-1,2-pyridazinedicarboxylate (**124**, R = Ph) (Pt, EtOH, $H_2$, 45°, 43 h: 33%).[1146]




Diethyl 1,2,3,6-tetrahydro- (**123**, R = H) gave diethyl 4,5-dihydroxyhexahydro-1,2-pyridazinedicarboxylate (**125**) (4-methylmorpholine *N*-oxide, $OsO_4$, Bu'OH–AcMe–$H_2O$, 20°, 24 h: 80%)[1690] or diethyl 1,2-dihydro-1,2-pyridazinedicarboxylate (**126**) (*N*-bromosuccinimide, $CCl_4$, reflux 90 min; filtered; oil from filtrate, lutidine, reflux, 5 min: 50%; by $Br_2$ addition and subsequent loss of 2HBr).[622]

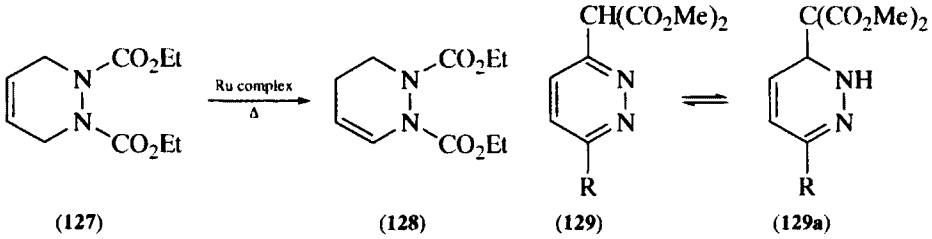
Also other examples.[440]




## Miscellaneous Reactions

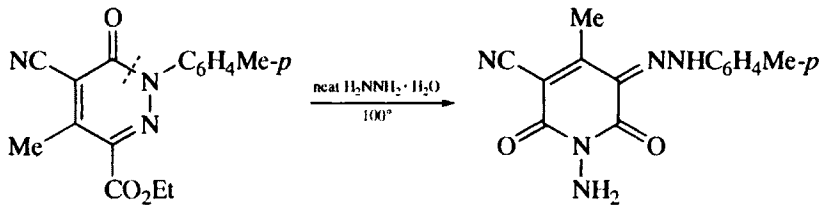
Diethyl 1,2,3,6-tetrahydro- (**127**) isomerized to diethyl 1,2,3,4-tetrahydro-1,2-pyridazinedicarboxylate (**128**) [$RuCl_2 \cdot (PPh_3)_4$, $N_2$, 100°, 7 h: 93%; other Ru and Rh complexes were less effective].[1211]

Kinetic studies have been reported for the tautomerism of 3-(dimethoxycarbonylmethyl)- (**129**, R = H) and 3-chloro-6-(dimethoxycarbonylmethyl)pyridazine (**129**, R = Cl) with their respective methylene forms (**129a**, R = H or Cl).[872]




(127) (128) (129) (129a)

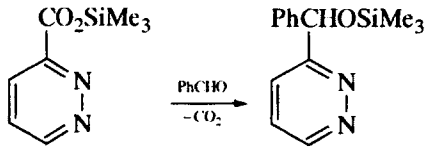
Ethyl 5-cyano-4-methyl-6-oxo-1-*p*-tolyl-1,6-dihdyro-3-pyridazinecarboxylate (**130**) reacted with ethanolic hydrazine hydrate to give the corresponding carbohydrazide, but with neat hydrazine hydrate, it has been reported to afford 1-amino-4-methyl-2,6-dioxo-5-*p*-tolylhydrazono-1,2,5,6-tetrahydro-3-pyridinecarbonitrile (**131**) (100°, 2 h: 40%).[1434]




(130) (131)

Trimethylsilyl 3-pyridazinecarboxylate (**132**) gave 3-α-(trimethylsiloxy)-benzylpyridazine (**133**) (neat PhCHO, 125°, $N_2$, 72 h: 67%; with $CO_2\uparrow$);[442] also other examples.[422]

Ethyl 3-oxo-5,6-diphenyl-2,3-dihydro-4-pyridazinecarboxylate gave 4-phenyl-1*H*-indeno[1,2-*d*]pyridazine-1,9(2*H*)-dione (Friedel–Crafts cyclization: ?%).[2053]




(132) (133)

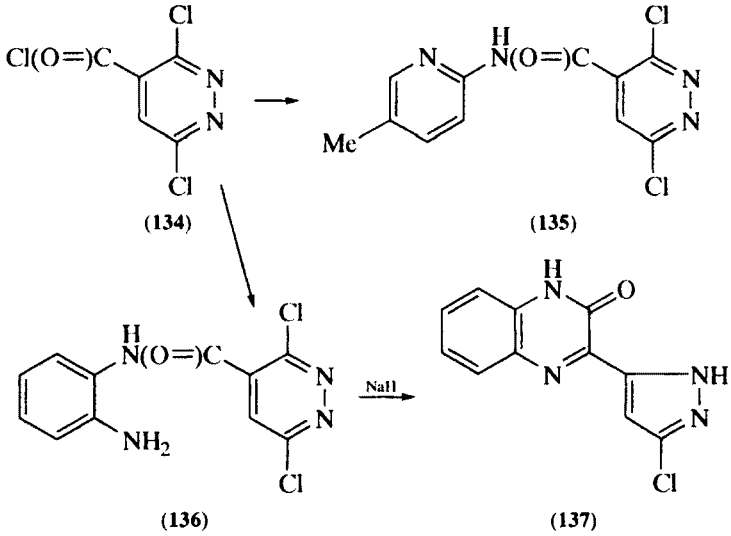
## 8.3 PREPARATION AND REACTIONS OF PYRIDAZINECARBONYL HALIDES

Pyridazinecarbonyl halides other than the chlorides are virtually unknown. The chlorides are useful intermediates, but they are seldom characterized prior to use.

PREPARATION Most such chlorides have been made from the corresponding carboxylic acids (see Section 8.1.2).

REACTIONS Pyridazinecarbonyl chlorides have been used as intermediates for the conversion of *pyridazinecarboxylic acids into the corresponding esters* (see Section 8.1.2). They have also been converted *into amides or into ketones*, examples of both reactions have been given (in Section 8.1.2) as ways to characterize unpurified pyridazinecarbonyl chlorides; other examples, using purified carbonyl chlorides, follow:

3,6-Dichloro-4-pyridazinecarbonyl chloride (**134**) with 5-methyl-2-pyridinamine gave 3,6-dichloro-*N*-(5-methylpyridin-2-yl)-4-pyridazinecarboxamide (**135**) (THF, 0° → 20°, 12 h: 69%)[25] or with *o*-fluoroaniline gave 3,6-dichloro-*N*-*o*-fluorophenyl-4-pyridazinecarboxamide ($Et_3N$, $CH_2Cl_2$, 0° → 20°, 4 h: 72%).[1954]




The same substrate (**134**) with *o*-phenylenediamine gave *N*-*o*-aminophenyl-3,6-dichloro-4-pyridazinecarboxamide (**136**) ($Et_3N$, $CH_2Cl_2$, $N_2$, 0° → 20°, 4 h: 77%) and then the unexpected 3-(3-chloro-1*H*-pyrazol-5-yl)-2(1*H*)-quinoxalinone (**137**) (NaH, $Me_2NCHO$, $N_2$, 100°, 10 min: 87%; structure confirmed by X-ray analysis).[1630]

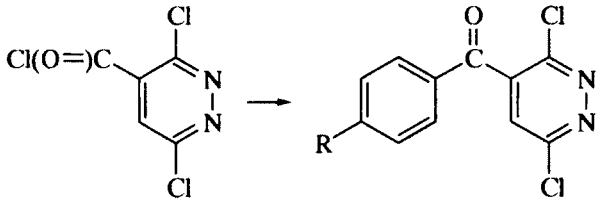
3,6-Dichloro-4-pyridazinecarbonyl chloride (**138**) gave 4-benzoyl-3,6-dichloropyridazine (**139**, R = H) (PhH, $SnCl_4$, $SnCl_4$, $AlCl_3$, $ClCH_2CH_2Cl$, reflux, 6 h: 63%), 3,6-dichloro-4-*p*-toluoylpyridazine (**139**, R = Me) (PhMe, likewise: 58%), or other such (substituted-benzoyl)pyridazine by appropriate Friedel–Crafts reactions.[443]

Also other examples.[2059, 2091, 2102, 2110]

## 8.4 PREPARATION AND REACTIONS OF PYRIDAZINECARBOXAMIDES (*H* 423)

PREPARATION Most pyridazinecarboxamides have been made *by primary syntheses* (see Chapters 1 and 2), *from pyridazinecarboxylic acids* (indirectly: see Section 8.1.2), or *from pyridazinecarboxylic esters* (see Section 8.2.2). In addition, some have been made *by hydrolysis (or thiolysis) of pyridazinecarbonitriles*, as illustrated in the following examples:

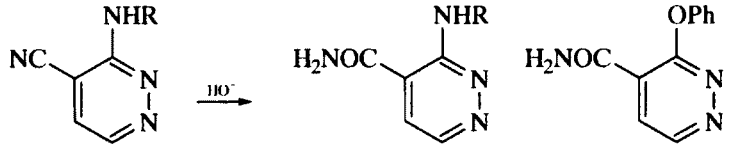
3-Amino-4-pyridazinecarbonitrile (**140**, R = H) gave 3-amino-4-pyridazinecarboxamide (**141**, R = H) (dilute $NH_4OH$, reflux, 24 h: 65%);[1681] 3-anilino-4-pyridazinecarbonitrile (**140**, R = Ph) likewise gave 3-anilino-4-pyridazinecarboxamide (**141**, R = Ph) (70%).[1079]



(**138**) (**139**)

3-Phenoxy-4-pyridazinecarbonitrile gave 3-phenoxy-4-pyridazinecarboxamide (**142**) (polyphosphoric acid, 120°, 3 h: 73%).[517]

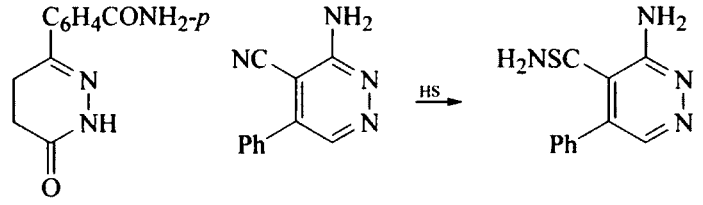
6-*p*-Cyanophenyl- gave 6-*p*-carbamoylphenyl-4,5-dihydro-3(2*H*)-pyridazinone (**143**) (NaOH, $H_2O_2$, EtOH–$H_2O$, 20°, 3 h: 37%; a controlled hydrolysis under Radziszewski conditions).[1256]



(**140**) (**141**) (**142**)

3-Amino-5-phenyl-4-pyridazinecarbonitrile (**144**) gave 3-amino-5-phenyl-4-pyridazinecarbothioamide (**145**) (pyridine, $Et_3N$, $H_2S\downarrow$, 20°, 12 h: 85%).[364]

Also other examples.[395, 820, 839, 1073, 1288, 1467, 1791]



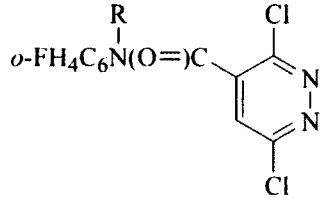
(**143**) (**144**) (**145**)

In addition, the formation of an *amide from the corresponding ketone* has been reported in the pyridazine series. Thus 3-chloro-6-2′-thenoylpyridazine in liquid ammonia at 100° for 6 h gave not only the expected 6-2′-thenoyl-3-pyridazinamine (65%) but also 6-amino-3-pyridazinecarboxamide (30%) by an unexplained mechanism.[1689]

REACTIONS Several important reactions of pyridazinecarboxamides have been covered already: *hydrolysis to pyridazinecarboxylic acids* (Section 8.1.1), *alcoholysis to pyridazinecarboxylic esters* (Section 8.2.1), *thiation to pyridazinecarbothioamides* (end of Section 4.2.4), and *conversion into pyridazinamines* by the Hofmann or related reactions (end of Section 7.2.1). The remaining reported reactions of such amides are illustrated by the following examples:

### *N*-Alkylation

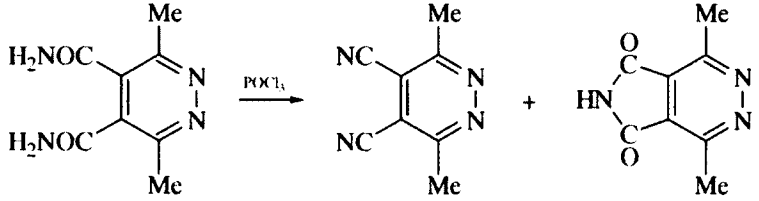
3,6-Dichloro-*N*-*o*-fluorophenyl-4-pyridazinecarboxamide (**146**, R = H) gave its *N*-methyl derivative (**146**, R = Me) (KOH, $Me_2SO$, $N_2$, 20°, 1 h; then MeI↓, 30 min: 85%);[1954] also analogous examples.[1984]



(**146**)

### Dehydration to Pyridazinecarbonitriles

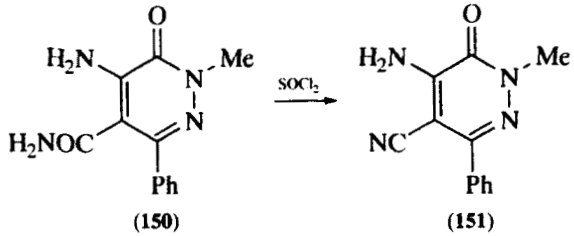
3,6-Dimethyl-4,5-pyridazinedicarboxamide (**147**) gave 3,6-dimethyl-4,5-pyridazinedicarbonitrile (**148**) [neat $POCl_3$, 20°, 5 h; then reflux, 24 h: 50%, after separation from 3,6-dimethyl-4,5-pyridazinedicarboximide (**149**) (~5% yield)].[927]



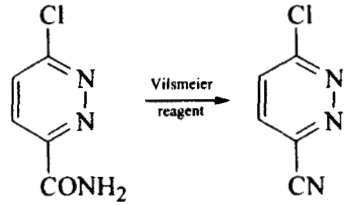
(**147**) (**148**) (**149**)

5-Amino-1-methyl-6-oxo-3-phenyl-1,6-dihydro-4-pyridazinecarboxamide (**150**) gave the corresponding pyridazinecarbonitrile (**151**) (neat $SOCl_2$, 60°, 4 h: 54%).[1832]

6-Chloro-3-pyridazinecarboxamide (**152**) gave 6-chloro-3-pyridazinecarbonitrile (**153**) [$SOCl_2$–$Me_2NCHO$ (Vilsmeier reagent), 0°, 5.5 h: 34%].[1338]



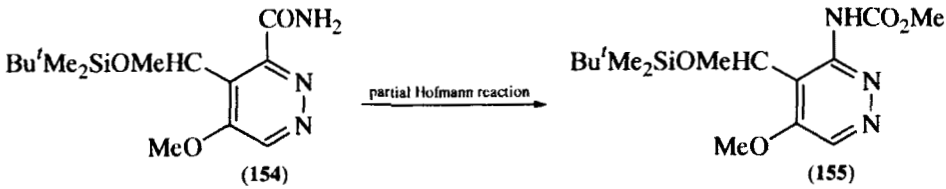

(**150**) (**151**)




(**152**) (**153**)

## Hofmann Reaction

4-α-(*t*-Butyldimethylsiloxy)ethyl-5-methoxy-3-pyridazinecarboxamide (**154**) gave 4-α-(*t*-butyldimethylsiloxy)ethyl-5-methoxy-3-methoxycarbonylaminopyridazine (**155**) (MeOBr, MeONa, MeOH, −43° → °, 30 min; then 60°, 30 min: ?%).[346]




(**154**) (**155**)

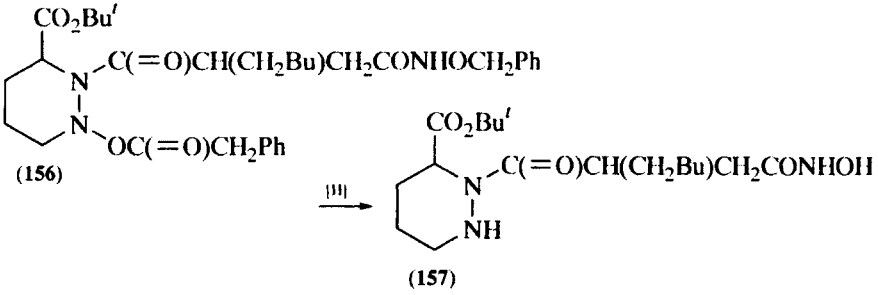
## Miscellaneous Reaction

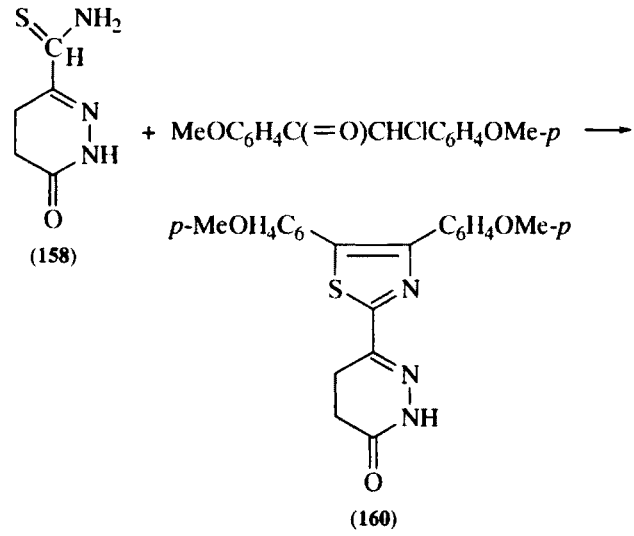
*t*-Butyl 1-benzylcarbonyloxy-2-[2-(*N*-benzyloxycarbamoylmethyl)heptanoyl]hexahydro-3-pyridazinecarboxylate (**156**) gave *t*-butyl 2-[2-(*N*-hydroxycarbamoylmethyl)heptanoyl]hexahydro-3-pyridazinecarboxylate (**157**) ($H_2$, Pd / C, MeOH, 20°, 5 h: 66%).[1814]

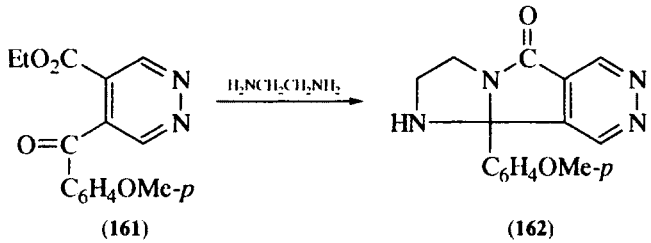
6-Oxo-1,4,5,6-tetrahydro-3-pyridazinecarbothioamide (**158**) and α-chloro-*p*-methoxybenzyl *p*-methoxyphenyl ketone (**159**) gave 6-(4,5-bis-*p*-methoxyphenylthiazol-2-yl)-4,5-dihydro-3(2*H*)-pyridazinone (**160**) [neat reactants (?), 85°, 14 h: 34%].[1750]

Ethyl 5-*p*-methoxybenzoyl-4-pyridazinecarboxylate (**161**) with ethylenediamine gave the corresponding amide, which cyclized at once to 9b-*p*-

methoxyphenyl-2,3-dihydro-1*H*-imidazo[1′,2′:1,2]-pyrrolo[3,4-*d*]pyridazin-5(9b*H*)-one (**162**), confirmed in structure by X-ray analysis (PhMe, trace TsOH, reflux with $H_2O$ removal, 40 h: 28%); also several analogues.[1920]




Several copper and/or nickel *complexes* of α,γ-bis(3-pyridazinecarboxamido)propane (**163**), some quite complicated, have been reported.[1861,2008] In addition, structural elucidation/partial syntheses of *antibiotics* containing hydropyridazinecarboxamide parts have been described,[1911,1942] and structure–

activity relationships of the potential *diuretics*, 3-methyl-6-phenyl-4-pyridazinecarboxamide (**164**) and related compounds have been summarized.[813]

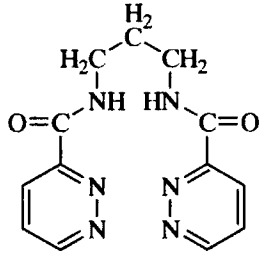
## 8.5 PREPARATION AND REACTION OF PYRIDAZINECARBOHYDRAZIDES (*H* 426)

PREPARATION Pyridazinecarbohydrazides have been made *by primary syntheses* (see Chapters 1 and 2), *from pyridazinecarboxylic acids* (indirectly: see Section 8.1.2), or *from pyridazinecarboxylic esters* (see Section 8.2.2).

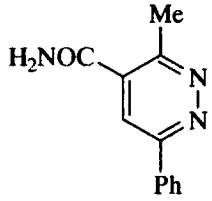
REACTIONS The *hydrolysis of hydrazides to carboxylic acids* has been covered in Section 8.1.1; the *degradation of hydrazides to pyridazinamines* has been exemplified in Section 7.2.1; and other reactions are illustrated in the following examples:

### *N*-Alkylidenation

*Note*: Pyridazinecarbohydrazides react with aldehydes (and ketones?) to afford their *N'*-alkylidene derivatives, akin to aldehyde hydrazones.

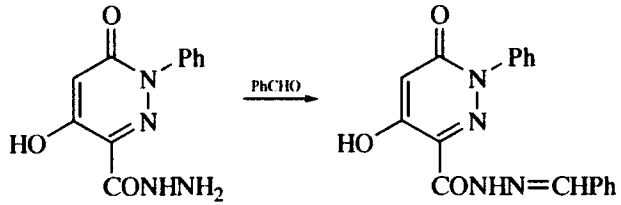


(163)



(164)

4-Hydroxy-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarbohydrazide (**165**) gave 6-benzylidenehydrazinocarbonyl-5-hydroxy-2-phenyl-3(2*H*)-pyridazinone (**166**) (PhCHO, EtOH, reflux, 30 min: 95%); also analogues.[1090]

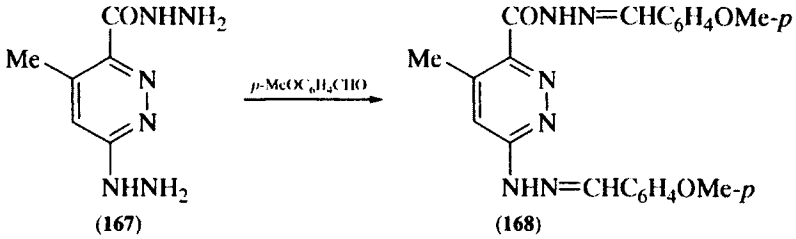



(165) (166)

6-Hydrazino-4-methyl-3-pyridazinecarbohydrazide (**167**) gave 3-*p*-methoxybenzylidenehydrazino-6-*p*-methoxybenzylidenehydrazinocarbonyl-5-

methylpyridazine (**168**) (*p*-$MeOC_6H_4CHO$, trace AcOH, EtOH, reflux, 10 min: 75%; note additional alkylidenation of the hydrazino substituent).[21]

Also other examples, some apparently catalyzed by a trace of piperidine.[1032, 1244, 1365]

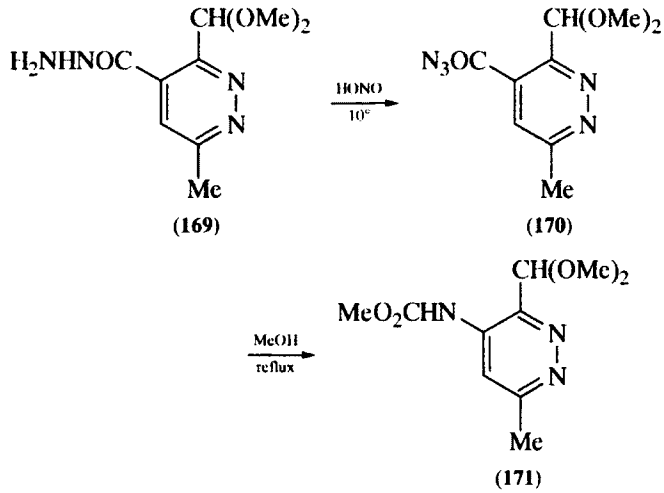



(**167**) (**168**)

## To Azidocarbonylpyridazines

*Note*: Some azidocarbonylpyridazines appear to be too unstable for satisfactory storage and are used immediately for further syntheses.

3-Dimethoxymethyl-6-methyl-4-pyridazinecarbohydrazide (**169**) gave 4-azidocarbonyl-6-methyl-3-pyridazinecarbaldehyde dimethyl acetal (**170**) ($NaNO_2$, $H_2O$–AcOH, 10°, 45 min: 74%, crude), which gave 4-methoxycarbonylamino-6-methyl-3-pyridazinecarbaldehyde dimethyl acetal (**171**) (MeOH, reflux, 4 h: 72%).[996]




(**169**) (**170**) (**171**)

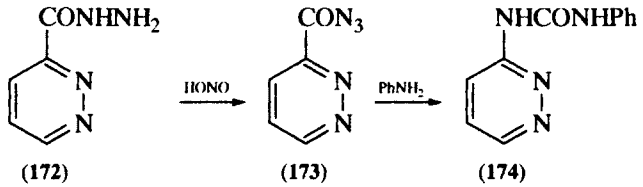
3-Pyridazinecarbohydrazide (**172**) gave 3-azidocarbonylpyridazine (**173**) ($NaNO_2$, HCl–$H_2O$, >5° → 20°, 2 h: ?%, crude), which gave not the expected anilide but *N*-phenyl-*N'*-(pyridazin-3-yl)urea (**174**) on warming with aniline ($PhNH_2$, $CHCl_3$, reflux, 2 h: 11%; presumably via 3-isocyanatopyridazine).[969]

Also other examples.[964, 1800, 1801, 1966]

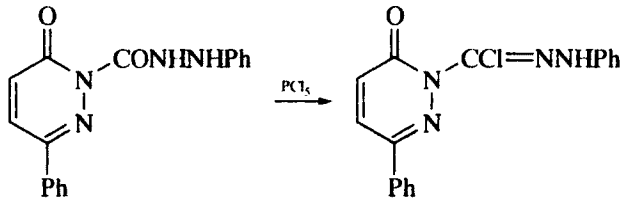
### Miscellaneous Reactions

6-Oxo-3,*N'*-diphenyl-1,6-dihydro-1-pyridazinecarbohydrazide (**175**) gave 2-(α-chloro-α-phenylhydrazonomethyl)-6-phenyl-3(2*H*)-pyridazinone (**176**) ($PCl_5$, $CH_2Cl_2$, 0°, 45 min: 52%); also analogues likewise.[368]

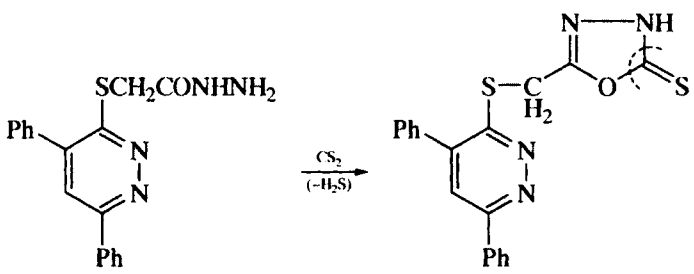
3-Hydrazinocarbonylmethylthio-4,6-diphenylpyridazine (**177**) gave 3,5-diphenyl-6-(5-thioxo-4,5-dihydro-1,3,4-oxadiazol-2-yl)pyridazine (**178**) ($CS_2$: ?%).[1870]




(172) (173) (174)




(175) (176)



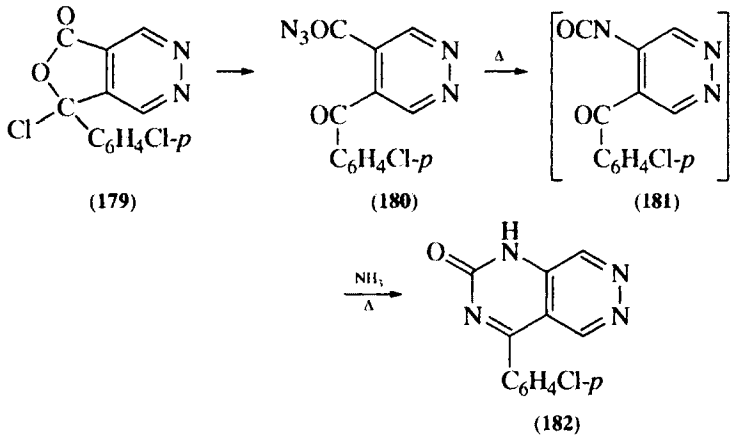

(177) (178)

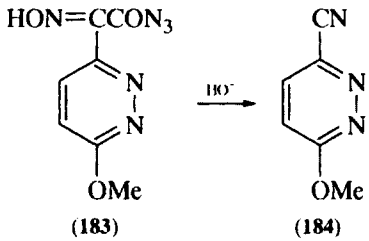
## 8.6 PREPARATION AND REACTIONS OF AZIDOCARBONYLPYRIDAZINES

PREPARATION Azidocarbonylpyridazines are usually made *from the corresponding pyridazinecarbohydrazides* by treatment with nitrous acid (see Section 8.5). Another interesting route, albeit of limited scope, is exemplified in the conversion of 5-*p*-chlorobenzoyl-4-pyridazinecarboxylic acid into the lactone (**179**) of 5-(α,*p*-dichloro-α-hydroxybenzyl)-4-pyridazinecarboxylic

acid (see Section 8.1.2) and thence into 4-azidocarbonyl-5-*p*-chlorobenzoylpyridazine (**180**) (41%) by treatment with cold acetonic sodium azide;[46] several analogues were made similarly.[46]

REACTIONS Few reactions of azidocarbonylpyridazines have been reported recently. Of these, *conversions into alkoxycarbonylaminopyridazines or pyrodazinylureas* (both via unisolated isocyanatopyridazines) were exemplified in Section 8.5. Other reactions include the cyclization of 4-azidocarbonyl-5-*p*-chlorobenzoylpyridazine (**180**) into 4-*p*-chlorophenylpyrimido[4,5-*d*]pyridazin-2(1*H*)-one (**182**) (85%), via the unisolated isocyanato intermediate (**181**), by first heating in refluxing toluene for 1 h, then bubbling ammonia through the cooled solution, and finally heating again at 190° in dimethylformamide;[516] also the conversion of 3-(α-azidocarbonyl-α-hydroxyiminomethyl)-6-methoxypyridazine (**183**) into 6-methoxy-3-pyridazinecarbonitrile (**184**) (84%) by simply stirring with aqueous sodium carbonate (mechanism?).[964]




## 8.7 PYRIDAZINECARBONITRILES (*H* 427)

Such carbonitriles are important, not only in their own right but also as convenient intermediates for a variety of other types of pyridazine derivative.

## 8.7.1 Preparation of Pyridazinecarbonitriles (*H* 427)

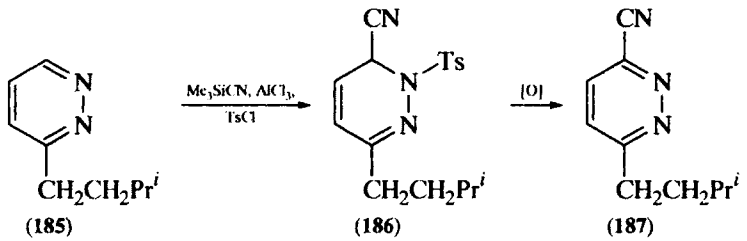
Many pyridazinecarbonitriles have been made *by primary syntheses* (see Chapters 1 and 2), *by cyanolysis of halogenopyridazines* (see Sections 4.2.8 and 4.4.6), *by indirect cine substitution of pyridazine N-oxides* (see Section 5.6.2), *by dehydration of pyridazinecarboxamides* (see Section 8.4), *from azidocarbonylpyridazines* (see Section 8.6), and also *by passenger insertion of a cyano group* in a variety of ways (e.g., see Section 3.2.1.3).

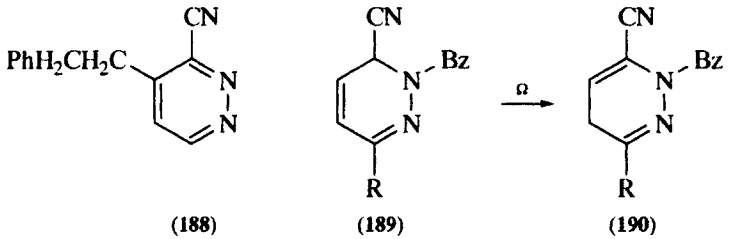
In addition, pyridazinecarbonitriles have been prepared by several other methods, exemplified as follows:

### By Reissert-like Reactions

*Note*: For such cyanations of unsubstituted pyridazine and two simple methylpyridazines, see Section 3.1.3. Other examples, mainly by modified procedures, follow.

3-Isopentylpyridazine (**185**) gave 6-isopentyl-2-tosyl-2,3-dihydro-3-pyridazinecarbonitrile (**186**) ($Me_3SiCN$, $AlCl_3$, $CH_2Cl_2$, $N_2$, 30 min; then TsCl↓, 3 h: 24%) and thence 6-isopentyl-3-pyridazinecarbonitrile (**187**) (1,8-diazabicyclo[5.4.0]undec-7-ene, THF, $N_2$, 1 h: 95%);[1660] 4-phenethyl-3-pyridazinecarbonitrile (**188**) (~17% yield) was made similarly.[1791]

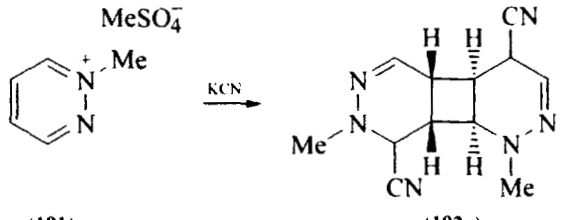


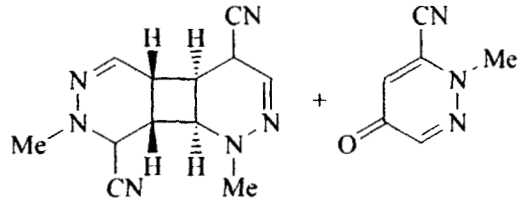

Pyridazine gave 2-benzoyl-2,3-dihydro-3-pyridazinecarbonitrile (**189**, R = H) ($Me_3SiCN$, BzCl, $AlCl_3$, $CH_2Cl_2$, 20°, ? h: 24%), which readily isomerized into 2-benzoyl-2,5-dihydro-3-pyridazinecarbonitrile (**190**, R = H) (60%) on column chromatography;[1066] 3-methylpyridazine likewise gave successively 2-benzoyl-6-methyl-2,3-dihydro-3-pyridazinecar-

bonitrile (**189**, R = Me)[976] and its 2,5-dihydro isomer (**190**, R = Me) (90%);[1066] and other such examples have been reported.[220]

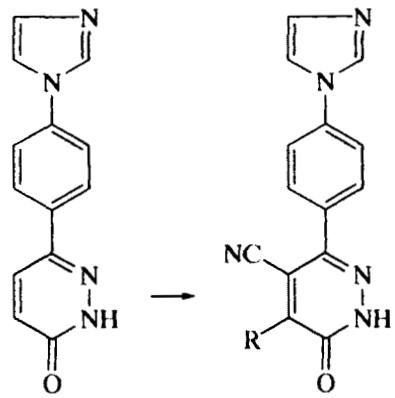
1-Methylpyridazinium methyl sulfate (**191**) (prepared from pyridazine in situ) with potassium cyanide gave a separable mixture of two cyanated "cyclobutane dimers," formulated as the "*anti* dimer" (**192a**) (3%) and the "*syn* dimer" (**192b**) (20%), and 3-methyl-5-oxo-2,5-dihydro-4-pyridazinecarbonitrile (**193**) (2%) (KCN, $H_2O$, 5°, 10 min: products separated by column chromatography);[772] 4-methyl-, 3,6-dimethyl-, and 3-methoxy-6-methylpyridazine were treated similarly to give a variety of cyanated products, each in < 5% yield.[772]




(**191**) (**192a**)




(**192b**) (**193**)




(**194**) (**195**)

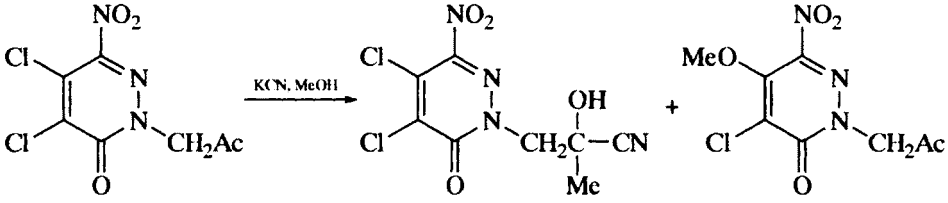
## By Addition of HCN

6-*p*-Imidazol-1′-ylphenyl-3(2*H*)-pyridazinone (**194**) gave 3-*p*-imidazol-1′-ylphenyl-6-oxo-1,6-dihydro-4-pyridazinecarbonitrile (**195**, R = H) (sub-

strate base, KCN, $Me_2SO$, 55°, 19 h: 10%, as perchlorate salt) or 3-*p*-imidazol-1′-ylphenyl-6-oxo-1,6-dihydro-4,5-pyridazinedicarbonitrile (**195**, R = CN) (substrate hydrochloride, likewise: 14%, as free base; see also Section 4.2.8).[1073] The mechanisms presumably involved addition with aerial (?) oxidation.

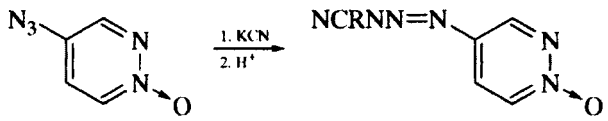
### By Miscellaneous Routes

2-Acetonyl-4,5-dichloro- (**196**) gave a separable mixture of 4,5-dichloro-2-(β-cyano-β-hydroxypropyl)- (**197**) and 2-acetonyl-4-chloro-5-methoxy-6-nitro-3(2*H*)-pyridazinone (**198**) (KCN, MeOH, $N_2$, 20°, 8 h: 22% and 15%, respectively).[1673]



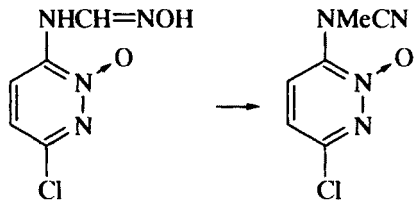

(196) (197) (197)

4-Azidopyridazine 1-oxide (**199**) gave the potassium salt (**200**, R = K) (KCN, EtOH–$H_2O$, 0°, 30 min: 91%) and then the free base, 4-(3-cyano-1-triazeno)pyridazine 1-oxide (**200**, R = H) (10% HCl, 20°, 30 min: ?%);[831] homologues were made similarly.[831]




(199) (200)

3-Chloro-6-formamidopyridazine 1-oxide oxime (**201**) gave 3-chloro-6-(*N*-cyano-*N*-methylamino)pyridazine 1-oxide (**202**) [neat $Me_2NCH(OMe)_2$, 25°, 5 days; then 80°, 1 h: 20%; see original for mechanism];[921] analogues likewise.[921,1010]




(201) (202)

### 8.7.2 Reactions of Pyridazinecarbonitriles (*H* 429)

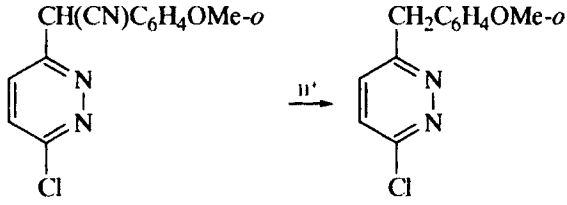
The following reactions of pyridazinecarbonitriles have been covered already: *conversion into arylpyridazines by Grignard reagents* (see Section 3.2.1.2), *reduction to aminomethylpyridazines* (see toward end of Section 7.2.1), *hydrolysis of pyridazinecarboxylic acids* (see Section 8.1.1), *conversion into pyridazinecarboxylic esters* (via carboximidates; see Section 8.2.1), and *controlled hydrolysis or thiolysis to pyridazinecarboxamides or pyridazinecarbothioamides* (see Section 8.4).

The remaining reactions of pyridazinecarbonitriles are illustrated in the following sets of examples:

#### Removal of the Cyano Group

*Note*: Cyano groups may often be removed quite easily by one-pot hydrolysis to the carboxylic acid and decarboxylation.

3-Chloro-6-(α-cyano-*o*-methoxybenzyl)pyridazine (**203**) gave 6-*o*-methoxybenzyl-3(2*H*)-pyridazinone (**204**) (10 M HCl–AcOH–$H_2O$, reflux, 6 h: 79%); note concomitant hydrolysis of Cl.[750]

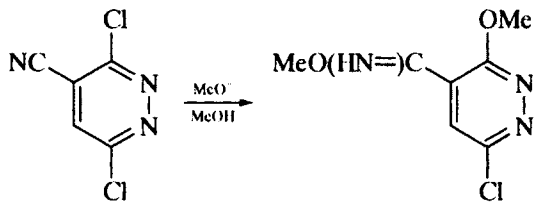



(**203**) (**204**)

#### Conversion into Alkyl Pyridazinecarboximidates

3,6-Dichloro-4-pyridazinecarbonitrile (**205**) gave methyl 6-chloro-3-methoxy-4-pyridazinecarboximidate (**206**) (MeONa, MeOH, 0° → 20°, 30 min: 63%; note concomitant alcoholysis of one Cl).[820]

Also other examples.[1365]



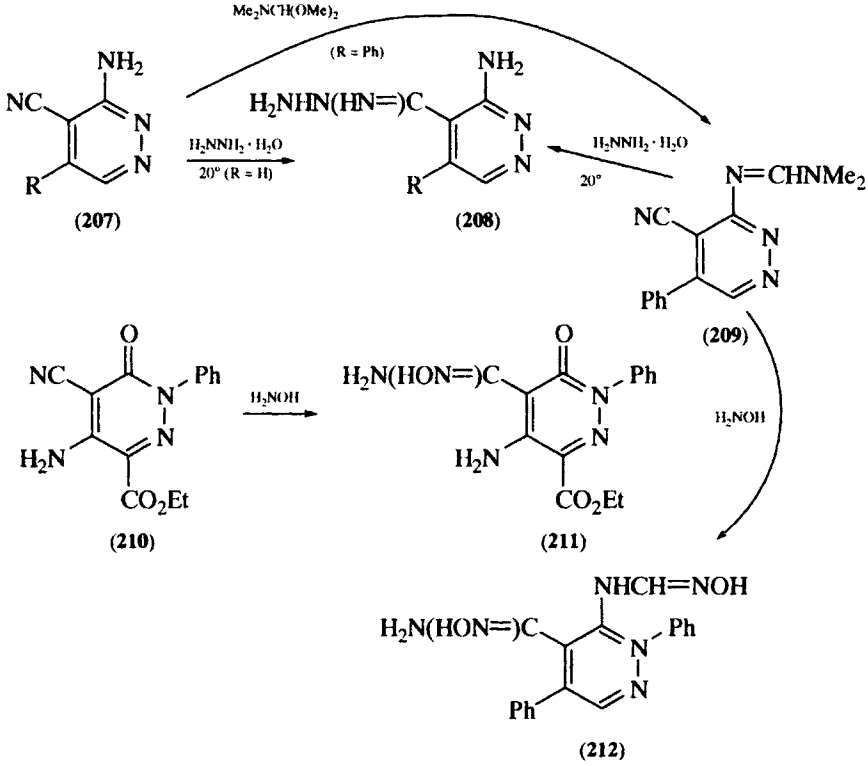

(**205**) (**206**)

## Conversion into Pyridazinecarboxamidrazones

3-Amino-4-pyridazinecarbonitrile (**207**, R = H) gave 3-amino-4-pyridazinecarboxamidrazone (**208**, R = H) (neat $H_2NNH_2 \cdot H_2O$, 20°, 5 days: 85%).[1681]

In contrast, 3-amino-5-phenyl-4-pyridazinecarbonitrile (**207**, R = Ph) did not give 3-amino-5-phenyl-4-pyridazinecarboxamidrazone (**208**, R = Ph) ($H_2NNH_2 \cdot H_2O$, reflux, 5 h). However, 3-dimethylaminomethyleneamino-5-phenyl-3-pyridazinecarbonitrile (**209**), derived from (**207**, R = Ph) [$Me_2NCH(OM)_2$, PhMe, reflux, 3 h: 85%], did give the required product (**208**, R = Ph) ($H_2NNH_2 \cdot H_2O$, 20°, 5 days: 64%).[130]

Also other examples.[55]




## Conversion into Pyridazinecarboxamide Oximes

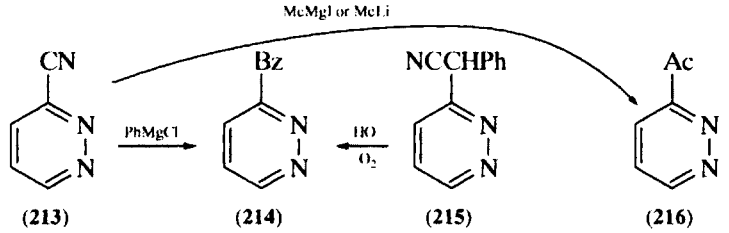
Ethyl 4-amino-5-cyano- (**210**) gave ethyl 4-amino-5-(*C*-amino-*C*-hydroxyiminomethyl)-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate (**211**) ($H_2NOH \cdot HCl$, AcONa, EtOH, reflux, 3 h: 58%).[119]

3-Dimethylaminomethyleneamino-5-phenyl-4-pyridazinecarbonitrile (**209**) gave 3-(hydroxyiminomethyl)amino-5-phenyl-4-pyridazinecarboxamide oxime (**212**) ($H_2NOH \cdot HCl$, EtOH, 20°, 16 h: 69%; note the additional transamination within the Schiff base substituent).[130]

## Conversion into *C*-Acylpyridazines (Pyridazine Ketones)

*Note*: Nuclear pyridazinecarbonitriles are converted into acylpyridazines by a Grignard or lithio reagent; extranuclear pyridazinecarbonitriles are best converted into corresponding acylpyridazines by oxidative decyanation.[2009] under alkaline conditions.

3-Pyridazinecarbonitrile (**213**) gave 3-benzoylpyridazine (**214**) (PhMgCl, $Et_2O$–THF, A, $-10° \rightarrow 20°$, 2 h: 59%);[1108] 3-(α-cyanobenzyl)pyridazine (**215**) gave the same product (**214**) (KOH, $PhCH_2Et_3NCl$, $H_2O$–MeOH, air↓ 45°, 90 min; 94%).[1354]




(**213**) (**214**) (**215**) (**216**)

Likewise, 3-pyridazinecarbonitrile (**213**) gave 3-*p*-chlorobenzoylpyridazine (*p*-$ClC_6H_4MgBr$, $Et_2O$, 0°, 12 h: 78%);[1692] and 3-(α-cyano-*o*-fluorobenzyl)pyridazine gave *o*-fluorobenzoylpyridazine (NaOH, $PhCH_2Et_3NCl$, $H_2O$–MeOH, air↓, 20°, until tlc showed no substrate: 75%).[1628]

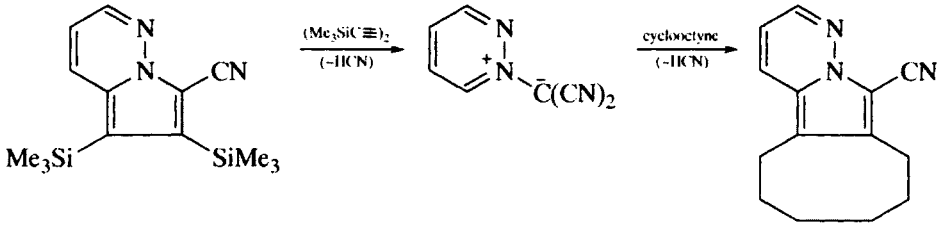
3-Pyridazinecarbonitrile (**213**) gave 3-acetylpyridazine (**216**) (MeMgI, $Et_2O$–PhH, A, $-15°$, 90 min: 70%; or MeLi, THF, A, $-70° \rightarrow 20°$, 30 min: 45%).[1347]

Also other examples of the Grignard procedure[1277, 1660, 1744] and of oxidative decyanation.[1586, 1698, 1798]

## Cyclization Reactions

*Note*: A few typical cyclizations involving pyridazinecarbonitriles are outlined here.

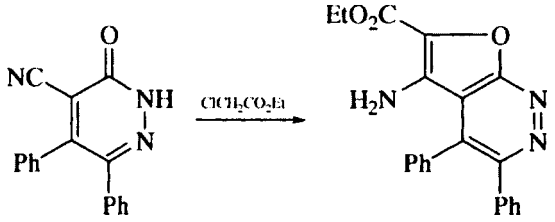
Pyridazinium-1-dicyanomethylide (**218**) with bis(trimethylsilyl)acetylene gave 5,6-bis(trimethylsilyl)pyrrolo[1,2-*b*]pyridazine-7-carbonitrile (**217**) (PhMe, reflux, 56 h: 92%)[36] or with cyclooctyne gave cycloocta[3,4]-pyrrolo[1,2-*b*]pyridazine-11-carbonitrile (**219**) (PhMe, reflux, >6 h: 97%).[1949] Also analogous condensations.[1505]




(**217**) (**218**) (**219**)

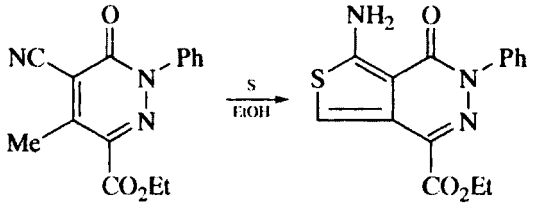
4,5-Pyridazinedicarbonitrile gave a variety of Diels–Alder adducts and/or derived products on treatment with appropriate dienophiles.[1428, 1817, 1893, 2027]

3-Oxo-5,6-diphenyl-2,3-dihydro-4-pyridazinecarbonitrile (**220**) gave ethyl 5-amino-3,4-diphenylfuro[2,3-*c*]pyridazine-6-carboxylate (**221**) ($Et_3CCH_2Cl$, $K_2CO_2$, AcMe, reflux, 24 h: 80%).[1717]



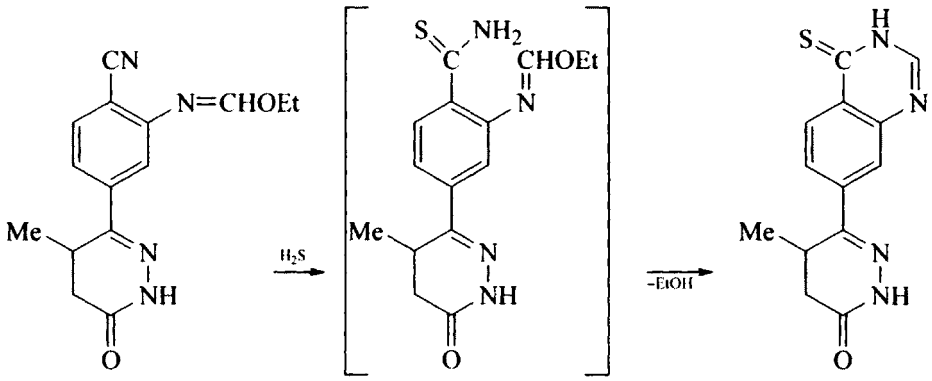
(**220**) (**221**)

Ethyl 5-cyano-4-methyl-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate (**222**) gave ethyl 5-amino-4-oxo-3-phenyl-3,4-dihydrothieno[3,4-*d*]pyridazine-1-carboxylate (**223**) (S, trace $Et_3N$, EtOH, reflux, 30 min: 79%);[54] also other such examples.[54, 1467]



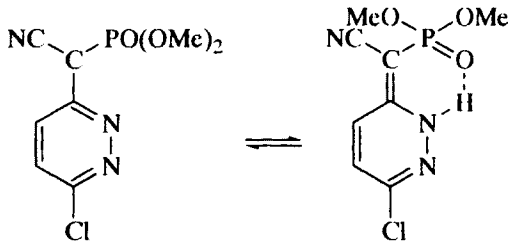
(**222**) (**223**)

6-(4-Cyano-3-ethoxymethyleneaminophenyl)-5-methyl-4,5-dihydro-3(2*H*)-pyridazinone (**224**) gave 7-(4-methyl-6-oxo-1,4,5,6-tetrahydropyridazin-3-yl)-4(3*H*)-quinazolinethione (**226**), presumably via the thioamide (**225**) ($Et_3N$, pyridine, $H_2S\downarrow$, 30 min: 70%).[1451]



(**224**) (**225**) (**226**)

Solid 3-chloro-6-[α-cyano-α-(dimethoxyphosphinyl)methyl]pyridazine (**227**) has been shown to exist in the hydrogen-bonded pseudobicyclic form (**227a**) by X-ray analysis.[1631]



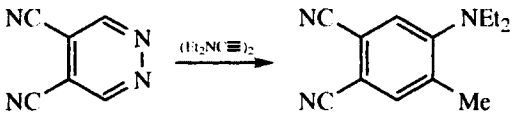
(**227**) (**227a**)

**To Benzene Derivatives via Diels-Alder Adducts**

*Note*: These conversions resemble those of pyridazine esters (see Section 8.2.2).

4,5-Pyridazinedicarbonitrile (**228**) with 1-diethylaminopropyne gave 4-diethylamino-5-methylphthalonitrile (**228a**) via a Diels–Alder adduct ($CH_2Cl_2$, 25°, 19 h: 85%); analogues likewise.[2065]

Also some related intramolecular Diels–Alder reactions.[2076]



(**228**) (**228a**)

## 8.8 PYRIDAZINECARBALDEHYDES (*H* 353)

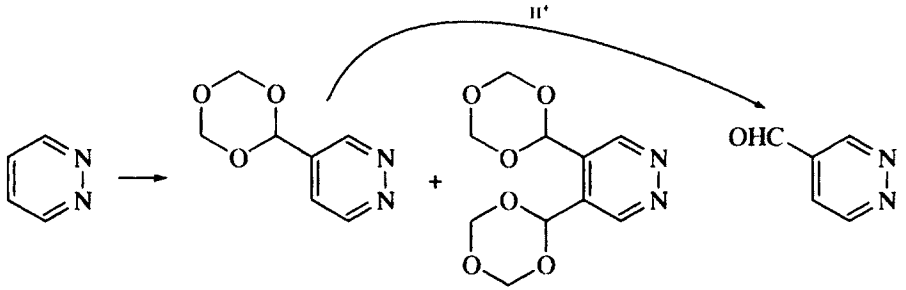
Although most pyridazinecarbaldehydes are reasonably stable, they are often characterized only as their oximes, hydrazones, simecarbazides, or acetals.

### 8.8.1 Preparation of Pyridazinecarbaldehydes (*H* 355, 383)

The usual methods for making such aldehydes or their derivatives have been outlined already: by *primary syntheses* (Chapters 1 and 2), *oxidation of alkylpyridazines* (Section 3.2.2.1), *an indirect oxidation of a bromomethylpyridazine* (neat end of Section 4.4.6), *by oxidation of hydroxymethylpyridazines* (Section 5.3.2), or *reduction of pyridazinecarboxylic esters* (Section 8.2.2).

The remaining method involves *direct C-formylation* by one means or another, as illustrated in the following examples.

Pyridazine (**229**) gave a separable mixture of 4-(1,3,5-trioxanyl)pyridazine (**230**) and 4,5-bis(1,3,5-trioxanyl)pyridazine (**231**) (1,3,5-trioxane, dilute $H_2SO_4$, $FeSO_4 \cdot 7H_2O$, 30% $H_2O_2$ ↓, 30°, 1 h: 27% and 2%, respectively), of which the first then gave 4-pyridazinecarbaldehyde (**232**) (6M HCl, reflux, 3 h: 65%).[971] 3-Methyl-4-pyridazinecarbaldehyde and isomers were made similarly.[227,971]

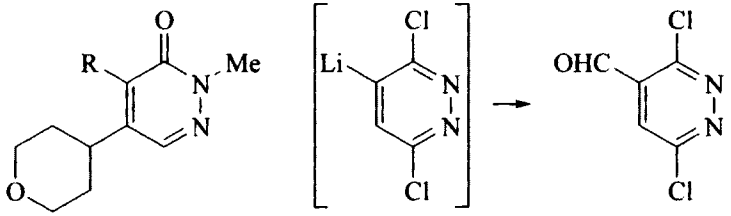



(229) (230) (231) (232)

2-Methyl-5-morpholino-3(2*H*)-pyridazinone (**233**, R = H) gave 2-methyl-5-morpholino-3-oxo-2,3-dihydro-4-pyridazinecarbaldehyde (**233**, R = CHO) ($POCl_3$, $Me_2NCHO$, 70°, 1 h: 85%; a Vilsmeier formylation).[1603]

3,6-Dichloropyridazine gave its 4-lithio derivative (**234**) (BuLi, $C_6H_{14}$–THF, 130°, A; then 2,2,6,6-$Me_4$–piperidine ↓, 0°, 30 min; then substrate ↓, −70°, 90 min: not isolated) and thence 3,6-dichloro-4-pyridazinecarbaldehyde (**235**) [$HCO_2Et$ ↓, −70°, 1 h: isolated initially as its 2,4-dinitrophenylhydrazone (20%) and thence as the free aldehyde (no details)].[1669]

Also other examples.[1646,2007]




(233) (234) (235)

## 8.8.2 Reactions of Pyridazinecarbaldehydes (*H* 378, 392)

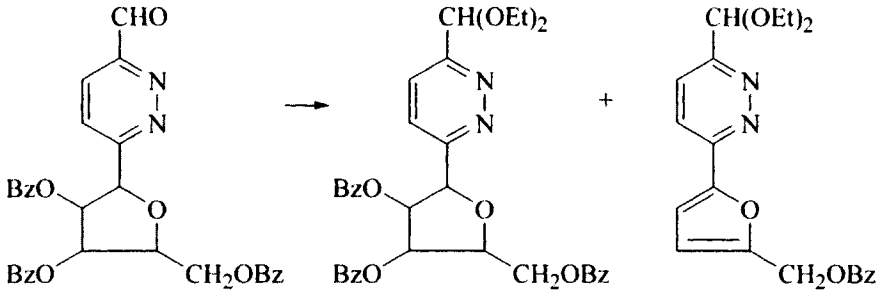
Some reactions of pyridazinecarbaldehydes have been discussed already: *condensation with methylene reagents to afford (substituted-vinyl)pyridazines* (Section 3.2.1.5), *reduction to hydroxyalkylpyridazines (including Cannizzaro*

*disproportionation*) (Section 5.3.1), and *oxidation to pyridazinecarboxylic acids (including Cannizzaro disproportionation)* (Section 8.1.1).

Other reactions are typified by the following classified examples:

### Formation of Acetals

6-(2,3,5-Tri-*O*-benzoyl-β-D-ribofuranosyl)-3-pyridazinecarbaldehyde (**236**) gave its diethyl acetal (**237**) and 6-(5-benzoyloxymethylfuran-2-yl)-3-pyridazinecarbaldehyde diethyl acetal (**238**) as a byproduct (EtOH, trace 10M HCl, 55°, 10 h: 41% and 17% after separation).[1539]

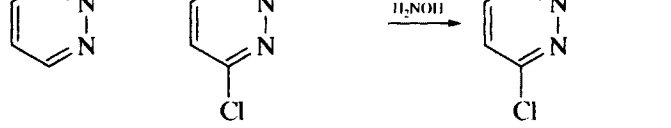



(236) (237) (238)

### Formation of Oximes

3-Pyridazinecarbaldehyde (**239**, X = O) gave its oxime (**239**, X = NOH) ($H_2NOH \cdot HCl$, $K_2CO_3$, $H_2O$–EtOH, 5°, 12 h: 89%).[1612]

3-Chloro-6-dimethylaminomethyleneaminopyridazine (**240**) gave 3-chloro-6-formamidopyridazine oxime (**241**) ($H_2NOH$, MeOH, 20°, 1 h: 89%; a virtual transamination).[1010]




(239) (240) (241)

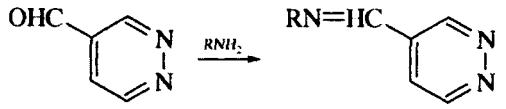
4-Pyridazinecarbaldehyde (**242**) gave its oxime (**243**, R = OH) ($H_2NOH \cdot HCl$, AcONa, $H_2O$, 100°, briefly: 82%),[67] shown to be the *E* isomer by nmr spectroscopy.[291]

### Formation of Schiff Bases

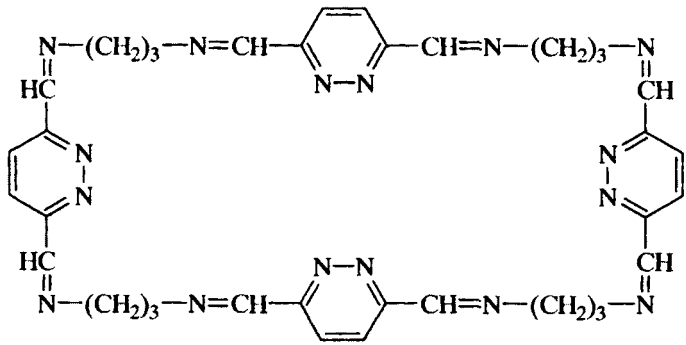
4-Pyridazinecarbaldehyde (**242**) gave 4-*p*-methoxyphenyliminomethyl- (**243**, $R = C_6H_4OMe$-*p*) or 4-benzyliminomethylpyridazine (**243**,

R = $CH_2Ph$) (*p*-$MeOC_6H_4NH_2$ or $PhCH_2NH_2$, respectively, $Na_2SO_4$, $CH_2Cl_2$, 0° → 20°, 4 h: each ~95%).[1906]

3,6-Pyridazinedicarbaldehyde and 1,3-propanediamine in the presence of lead (II) perchlorate gave the macrocyclic ligand (**244**) as its $Pb_2(ClO_4)_4$ complex (MeOH–MeCN, 20°: 47%, characterized by ultimate and X-ray analysis).[1565]



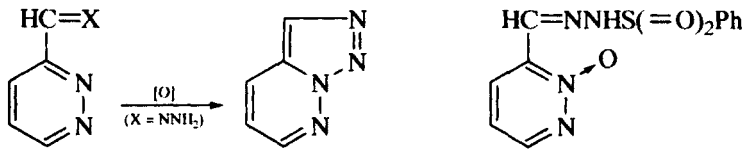
(242) (243)



(244)

## Formation of Hydrazones or Semicarbazones

3-Pyridazinecarbaldehyde (**245**, X = O) gave its hydrazone (**245**, X = $NNH_2$) ($H_2NNH_2 \cdot H_2O$, trace $H_2SO4$, EtOH, 60° 3 h: ~75%) and then [1,2,3]triazolo[1,5-*b*]pyridazine (**246**) [$Ph(OAc)_4$, PhH, 20°, 30 min: 70%].[439] The same substrate (**245**, X = O) gave its 2,4-dinitrophenylhydrazone [**245**, X = $NHHC_6H_3$ $(NO_2)_2$-2,4] (no details: >95%)[77] or its thiosemicarbazone [**245**, X = $NNHC(=S)NH_2$] [$H_2NNHC(=S)NH_2$, 20°, 7 days: >95%].[77]



(245) (246) (247)

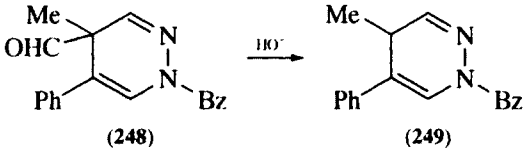
3-Pyridazinecarbaldehyde 2-oxide gave 3-benzenesulfonylhydrazonomethylpyridazine 2-oxide (**247**) ($PhSO_2NHNH_2$, MeOH, 3° → 20°, 30 min: >40%); also homologues similarly.[1261]

Also other examples.[35,1603]

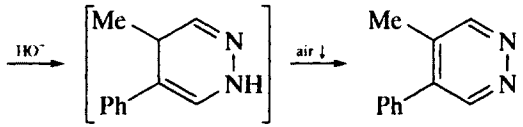
## Removal of Formyl Groups

*Note*: This is usually done by oxidation and subsequent decarboxylation but sometimes formyl groups may be removed from hydropyridazines by alkaline hydrolysis.

1-Benzoyl-4-methyl-5-phenyl-1,4-dihydro-4-pyridazinecarbaldehyde (**248**) gave 1-benzoyl-4-methyl-5-phenyl-1,4-dihydropyridazine (**249**) (KOH, MeOH–$H_2O$, 25°, 60 min; then air↓, 15 min: 45%).[297]



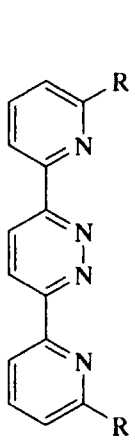

(248) (249)




(250) (251)

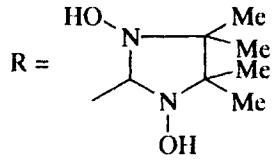
## Conversion into Imidazolylpyridazines

3,6-Bis(6-formylpyridin-2-yl)pyridazine (**252**) with 2,4-bishydroxyamino-2,4-dimethylbutane (2 mol) gave 3,6-bis[6-(1,3-dihydroxy-4,4,5,5-tetramethylimidazolidin-2-yl)pyridin-2-yl]pyridazine (**253**) (no details: good yield) and thence, on oxidation, a deep purple product formulated as the biradical (**254**) ($NaIO_3$ or $Ag_2O$, 0°, phase-transfer conditions: 94%).[1516]

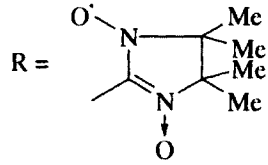



R = CHO

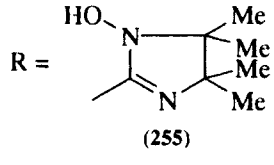
(**252**)



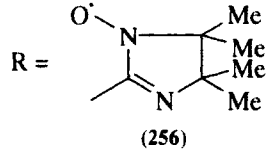

(**253**)




(**254**)




(**255**)




(**256**)

In contrast, the same substrate (**252**) with 2,4-bishydroxyamino-2,4-dimethylbutane (2 mol) in the presence of $SeO_2$ gave 3,6-bis[6-(3-

hydroxy-4,4,5,5-tetramethyl-1-imidazolin-2-yl)pyridin-2-yl]pyridazine (**255**) (MeOH: >40%) and then, on oxidation, an orange product formulated as the biradical (**256**) ($NaIO_3$, phase-transfer conditions: 45%).[1517]

## 8.9 PYRIDAZINE KETONES (*H* 353)

Ketones are well represented in the pyridazine series, but many of the better preparative routes and reactions have been covered earlier.

### 8.9.1 Preparation of Pyridazine Ketones (*H* 355, 383)

The following routes to pyridazine ketones have been discussed: *by primary syntheses* (Chapters 1 and 2), *by oxidation of aralkylpyridazines* (Section 3.2.2.1), *by oxidation of secondary hydroxyalkylpyridazines* (Section 5.3.2), *from pyridazinecarboxylic esters with alkyllithiums or carbanions* (Section 8.2.2), *from pyridazinecarbonyl chlorides by Friedel–Crafts reactions* (Sections 8.1.2 and 8.3), *by some Reissert reactions* (e.g., Section 8.7.1), and *from pyridazinecarbonitriles with a lithium or Grignard reagent* (Section 8.7.2).

The remaining routes to such ketones are illustrated by the following typical examples:

#### By Direct (Radical) *C*-Acylation

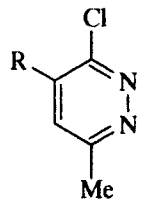
*Note*: The nuclear acylation of pyridazine, the extranuclear acylation of alkylpyridazines, and the *C*-formylation of pyridazines have been outlined in Sections 3.1.3, 3.2.2.3, and 8.8.1, respectively.

4-Methylpyridazine (**257**, R = H) gave 4-benzoyl-5-methylpyridazine (**257**, R = Bz) (PhCHO, $FeSO_4 \cdot 7H_2O$, $Bu^tHO_2$ / $Bu^t_2O_2$, $H_2SO_4$–AcOH–$H_2O$, 0°, 1 h: 27%).[1060]

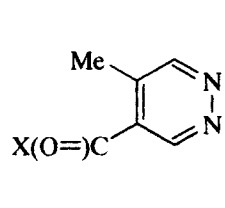
3-Chloro-6-methylpyridazine (**258**, R = H) gave 4-acetyl-3-chloro-6-methylpyridazine (**258**, R = Ac) (MeCHO, $FeSO_4 \cdot 7H_2O$, 34% $H_2O_2$, 25°, 15 min: 37%); also analogous acylations.[1514]



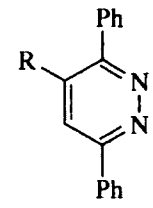

(**257**)




(**258**)




(**269**)




(**260**)

4-Propionylpyridazine (**259**, R = H, X = Et) gave 4-benzoyl-5-propionylpyridazine (**259**, R = Bz, X = Et) (PhCHO, $FeSO_4 \cdot 7H_2O$, $Bu^tHO_2/Bu^t_2O_2$, $H_2SO_4$–AcOH–$H_2O$, 0° → 20°, 1 h: 43%);[1095] 4-pyridazinecarboxamide (**259**, R = H, X = $NH_2$) likewise gave 5-benzoyl-4-pyridazinecarboxamide (**259**, R = Bz, X = $NH_2$) (30%).[46]

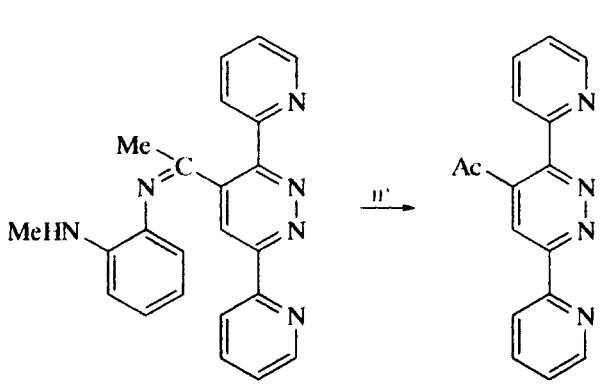
Also other examples.[513, 1095]

### From Tributylstannylpyridazines

3,6-Diphenyl-4-tributylstannylpyridazine (**260**, R = $SnBu_3$) gave 4-benzoyl-3,6-diphenylpyridazine (**260**, R = Bz) [BzCl, $Pd(PPh_3)_2Cl_2$, $Et_4NCl$, PhH, reflux, 41 h: 38%].[1591]

### From Corresponding Schiff Bases

5-α-(*o*-Methylaminophenylimino)ethyl- (**261**) (made by primary synthesis) gave 5-acetyl-3,6-dipyridin-2′-ylpyridazine (**262**) HCl, EtOH–$H_2O$, reflux, 10 min: 97%.[1083]




(**261**) (**262**)

## 8.9.2 Reactions of Pyridazine Ketones (*H* 378, 392)

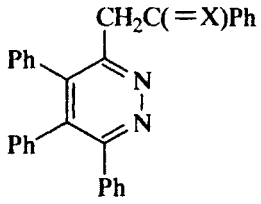
Some reactions of pyridazine ketones have been discussed elsewhere: *elimination of C-acyl groups* (Section 3.2.1.4), *conversion of C-aroyl- into C-α-arylstyrylpyridazines* (Section 3.2.1.5), *extranuclear halogenation of pyridazine ketones* (Section 4.3.2), *reduction of pyridazine ketones to alkyl- or hydroxyalkylpyridazines* (Section 3.2.1.5 or 5.3.1, respectively), and *conversion of pyridazine ketones into pyridazinecarboxamides* (Section 8.4).

Other reactions are illustrated briefly in the following typical examples:

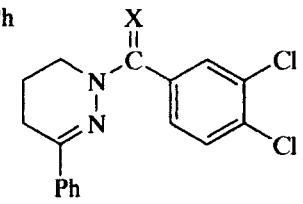
### Thiation

3-Phenacyl-4,5,6-triphenyl- (**263**, X = O) gave 3,4,5-triphenyl-6-thiophenacylpyridazine (**263**, X = S) ($P_2S_5$, xylene, reflux, 2 h: 45%).[1362]

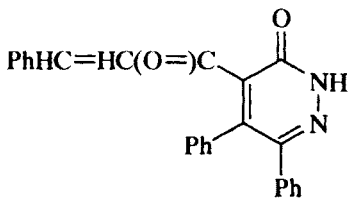
1-(3,4-Dichlorobenzoyl)- (**264**, X = O) gave 1-[3,4-dichloro(thiobenzoyl)]-3-phenyl-1,4,5,6-tetrahydropyridazine (**264**, X = S) ($P_2S_5$, PhMe, $N_2$, 60°, 12 h: ~25%).[1834]



(263)



(264)



(265)

### ω-Alkylidenation

4-Acetyl- gave 4-cinnamoyl-5,6-diphenyl-3(2*H*)-pyridazinone (**265**) (PhCHO; see original for details).[1447]

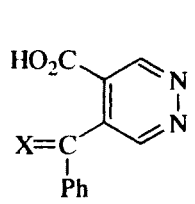
Also other examples.[974,1447,1616]

### Formation of Oximes

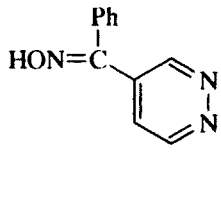
5-Benzoyl-4-pyridazinecarboxylic acid (**266**, X = O) gave its oxime (**266**, X = NOH) ($H_2NOH \cdot HCl$, AcONa, EtOH, 20°, 24 h: 80%);[46] 4-benzoylpyridazine likewise gave its oxime (**267**) (95%), separated chromatographically into the *Z* and *E* isomers (22% and 11%, respectively).[1798]

5-Acetyl-2-methyl-6-phenyl-3,4(1*H*,2*H*)-pyridazinedione (**268**, X = O) gave its oxime (**268**, X = NOH) ($H_2NOH \cdot HCl$, MeONa, MeOH–$H_2O$, 20°, 15 min: 87%).[1561]

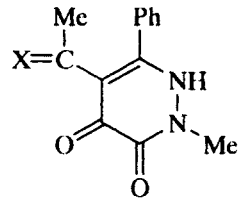
Also other examples.[65,859,1673,1716,2039]



(266)



(267)



(268)

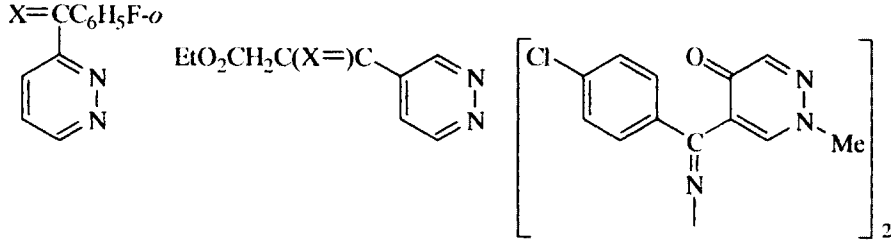
### Formation of Hydrazones or Semicarbazones

3-*o*-Fluorobenzoylpyridazine (**269**, X = O) gave its hydrazone (**269**, X = $NNH_2$) (excess neat 80% $H_2NNH_2 \cdot H_2O$ 120°, 30 min: 90%).[1628]

4-Ethoxymalonylpyridazine (**270**, X = O) gave its phenylhydrazone (**270**, X = NNHPh) ($PhHNNH_2$, EtOH, 20°, 24 h: 80%).[65]

5-*p*-Chlorobenzoyl-1-methyl-4(1*H*)-pyridazinone gave its azine (**271**) [$H_2NNH_2 \cdot HCl$ (2 equiv), EtOH, reflux, 6 h: 83%].[740]

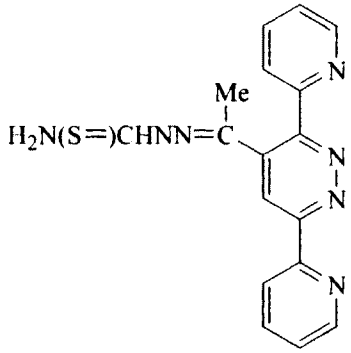
4-Acetyl-3,6-dipyridin-2′-ylpyridazine gave its thiosemicarbazone (**272**) ($H_2NHNC(=S)NH_2 \cdot HCl$, no details).[1083]



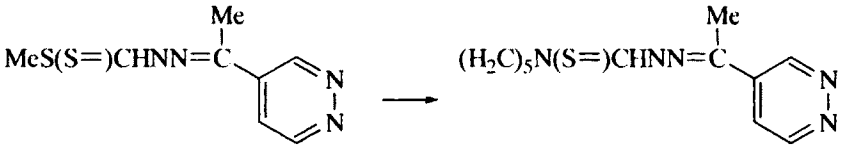
(269) (270) (271)

4-Acetylpyridazine gave 4-[α-methylthio(thiocarbonyl)hydrazono]ethylpyridazine (**273**) ($MeS_2CNHNH_2$, $Pr^iOH$, 65°, 1 h: 96%) and then 4-acetylpyridazine 4,4-pentamethylene(thiosemicarbazone) (**274**) [$(CH_2)_5NH$, EtOH, reflux until MeSH↑ ceased (< 18 h): 93%; a slow but effective route to such ω-substituted thiosemicarbazones].[1347]

Also other examples.[1272, 1692]



(272)

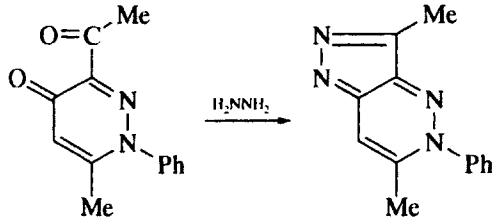


(273) (274)

## Cyclization Reactions

3-Acetyl-6-methyl-1-phenyl-4(1*H*)-pyridazinone (**275**) gave 3,6-dimethyl-5-phenyl-5*H*-pyrazolo[4,3-*c*]pyridazine (**276**) ($H_2NNH_2 \cdot H_2O$, EtOH, reflux, 1 h: 80%); also homologues.[1706]

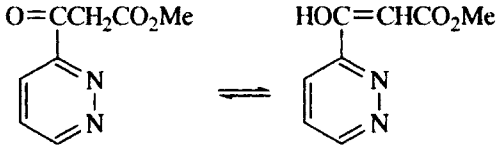
Also examples of other types.[597, 949]



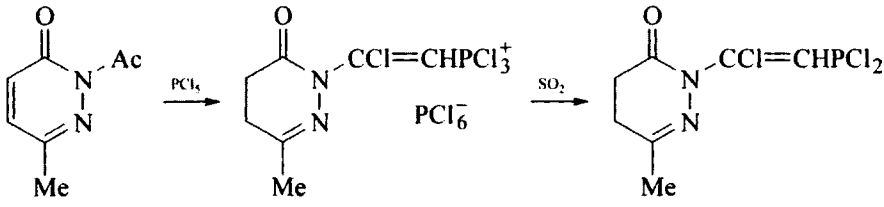

(275) (276)

## Keto Enol Tautomerism

3-Methoxymalonylpyridazine (**277**) has been shown by nmr measurements to exist as its enol (**277a**) to the extent of 21% in chloroform at $-35°$.[1180] Such studies have been extended to analogous derivatives of pyridine, pyrimidine, and pyrazine.[1180,1181]




(277) (277a)




(278) (279) (280)

## Phosphinylation

2-Acetyl-6-methyl-4,5-dihydro-3(2*H*)-pyridazinone (**278**) gave a reasonably stable intermediate (**279**) ($PCl_5$, PhH, 20°, 3 days: 56%) and then 2-(α-chloro-β-dichlorophosphinylvinyl)-6-methyl-4,5-dihydro-3(2*H*)-pyridazinone (**280**) (PhH, $SO_2\downarrow$, 20°, until clear: 18%).[2112]

*Chemistry of Heterocyclic Compounds, Volume 57*
D. J. Brown
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APPENDIX

# Table of Simple Pyridazines

This table is intended as a comprehensive alphabetical list of simple pyridazines described up to mid-1998. For each compound are recorded (1) melting and/or boiling point; (2) an indication of reported spectra or other physical properties; (3) any reported salts or simple derivatives, especially when the parent compound was poorly characterized; (4) an indication of whether any chelate complexes have been described; and (5) direct reference(s) to the original literature from 1972 onward, followed by indirect reference(s) to any previously published data of literature in the form of page number(s) [e.g., *H* 73, 400 (pages in the original *Hauptwerk* volume)] where such material appears in Castle's original book.[1839]

To keep the table within manageable proportions, the following categories of pyridazines have been *excluded* on the grounds that they are not simple:

Fused or nuclear-reduced pyridazines

Pyridazines with a cyclic substituent other than an unsubstituted cyclohexyl, morpholino, phenyl, or piperidino group

Pyridazines bearing a substituent with more than six carbon atoms, except for an unsubstituted benzoyl, benzyl, phenethyl, phenylethynyl, or styryl group

Pyridazines with two substituents at any one position

Pyridazines with two or more independent functional groups on a single substituent

The following conventions and abbreviations have been used in the table.

MELTING POINT The term *melting point* covers not only a regular melting point or melting range but also such variations as "decomposing at" or "melting with decomposition at." The use of the symbol > before a melting point indicates that the substance melts or decomposed above that temperature or that it does not melt or decompose below that temperature. When two differing melting points are given in the literature, they appear in the table as, for example, "88–89 or 94–95"; when more than two melting points are given, they are recorded in a form such as "218 to 225" in order to distinguish them from the single melting range "218–225."

BOILING POINT *Boiling points* are distinguished from melting points by the presence of a pressure in millimeters of mercury (mmHg) after the temperature: for instance, 125–128/0.8.

ABBREVIATIONS FOR PHYSICAL DATA

| | |
|---|---|
| anal | Analytical data (usually assumed) |
| crude | Compound not purified |
| dipole | Dipole moment |
| el den | Electron density |
| ir | Infrared spectrum |
| liq | Liquid |
| ms | Mass spectrum |
| nmr | Nuclear magnetic resonance spectrum (any nucleus) |
| pol | Polarographic data |
| st | Fine structure discussed |
| uv | Ultraviolet / visible spectrum |
| xl st | crystal structure (X-ray data) |

ABBREVIATIONS FOR SALTS, ASSOCIATED ANIONS, OR SOLVATES

| | |
|---|---|
| AcOH | Acetate salt |
| HBr, etc. | Appropriate hydrohalide salt |
| $H_2O$ | Hydrate |
| $HSO_4$ | Sulfate anion |
| $H_2SO_4$ | Sulfate salt |
| I, etc. | Appropriate halide anion |
| MeI | Quaternary methiodide |
| $NH_4$ | Ammonium salt |
| Na, etc. | Appropriate alkali metal salt |
| pic | Picrate (salt or anion) |
| TsOH | *p*-Toluenesulfonate salt |

ABBREVIATIONS FOR DERIVATIVES

| | |
|---|---|
| dnp | 2,4-Dinitrophenylhydrazone |
| $Et_2$acetal | diethyl acetal |
| $H_2NN=$ | Hydrazone |
| MeCH=, etc. | Appropriate alkylidene derivative |
| PhHNN= | Phenylhydrazone |
| PhCH= | Benzylidene derivative |
| sc | Semicarbazone |
| tsc | Thiosemicarbazone |

OTHER NOTES The use of "cf." before a reference usually indicates some inconsistent or mildly relevant information therein. A query mark (?) indicates some reasonable doubt associated with a datum or reference. A dash (—) in the data column indicates that no new physical data was obtained from original references covered for this supplement.

Alphabetical list of simple pyridazines, reported to the end of 1997

| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3-Acetamido-4-acetyl-5,6-diphenylpyridazine | — | *H* 555 |
| 4-Acetamido-5-acetyl-6-phenyl-3(2*H*)-pyridazinone | — | *H* 395 |
| 4-Acetamido-5-benzoyl-6-phenyl-3(2*H*)-pyridazinone | — | *H* 395 |
| 3-Acetamido-6-benzyloxypridazine | — | *H* 181 |
| 3-Acetamido-6-benzylthiopyridazine | — | *H* 553, 807 |
| 3-Acetamido-6-butoxy-5-nitropyridazine 2-oxide | — | *H* 198, 604 |
| 3-Acetamido-6-butoxypyridazine | — | *H* 178, 552 |
| 3-Acetamido-6-butoxypyridazine 2-oxide | — | *H* 603 |
| 4-Acetamido-6-chloro-3-hydrazinopyridazine | — | *H* 562 |
| 3-Acetamido-5-chloro-6-methoxypyridazine | — | *H* 182, 295 |
| 3-Acetamido-5-chloro-6-methoxypyridazine 2-oxide | — | *H* 197, 301 |
| 3-Acetamido-6-chloro-2-methyl-5-oxo-2,5-dihydro-4-pyridazinecarboxylic acid | 143–144, ir, nmr | 967 |
| 3-Acetamido-6-chloro-4-methylpyridazine | — | *H* 554 |
| 3-Acetamido-6-chloro-5-methylpyridazine | — | *H* 290, 554 |
| 4-Acetamido-6-chloro-2-methyl-3(2*H*)-pyridazinone | — | *H* 99, 323, 563 |
| 4-Acetamido-6-chloro-3-phenylpyridazine | 174–175, ir, nmr | 133 |
| 5-Acetamido-4-chloro-2-phenyl-3(2*H*)-pyridazinone | — | *H* 121, 313 |
| 5-Acetamido-6-chloro-2-phenyl-3(2*H*)-pyridazinone | — | *H* 93 |
| 3-Acatamido-6-chloropyridazine | oxime: 149–150, nmr | 1010; *H* 277, 303, 551 |
| 4-Acetamido-6-chloropyridazine | — | *H* 289, 558 |
| 3-Acetamido-6-chloropyridazine 2-oxide | — | *H* 299, 301, 603 |
| 4-Acetamido-6-chloro-3(2*H*)-pyridazinone | 260–265 or 262–264, nmr | 84, 1597; *H* 96, 293, 559 |
| 5-Acetamido-3-chloro-4(1*H*)-pyridazinone | — | *H* 132, 190 |
| 5-Acetamido-4-chloro-3(2*H*)-pyridazinone | — | *H* 116, 311, 559 |
| 4-Acetamido-5-cyano-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylic acid | 300, ir, nmr | 129 |
| 5-Acetamido-2-cyclohexyl-4-iodo-3(2*H*)-pyridazinone | — | *H* 124, 331 |
| 5-Acetamido-3,6-dimethoxy-4-pyridazinamine | — | *H* 193 |
| 4-Acetamido-3,6-dimethoxypyridazine | — | *H* 192, 560 |
| 4-Acetamido-2,6-dimethyl-3(2*H*)-pyridazinone | — | *H* 99, 563 |
| 5-Acetamido-2,6-dimethyl-3(2*H*)-pyridazinone | — | *H* 91, 564 |
| 3-Acetamido-5,6-diphenyl-4-pyridazinecarbonitrile | — | *H* 555 |
| 2-Acetamido-5,6-diphenyl-4-pyridazinecarboxamide | — | *H* 555 |
| 3-Acetamido-6-ethoxy-5-nitropyridazine 2-oxide | — | *H* 198, 604 |
| 3-Acetamido-6-ethoxypyridazine | — | *H* 552 |
| 3-Acetamido-6-ethoxypyridazine 2-oxide | — | *H* 603 |
| 3-Acetamido-6-hexyloxy-5-nitropyridazine 2-oxide | — | *H* 198, 604 |
| 3-Acetamido-6-hexyloxypyridazine | — | *H* 552 |
| 3-Acetamido-6-hexyloxypyridazine 2-oxide | — | *H* 604 |
| 6-Acetamido-4-imino-1-phenyl-1,4-dihydro-3,5-pyridazinedicarbonitrile | 210, ir, nmr | 55 |
| 5-Acetamido-4-iodo-2-phenyl-3(2*H*)-pyridazinone | — | *H* 124, 331 |
| 3-Acetamido-6-isopentyloxy-5-nitropyridazine 2-oxide | — | *H* 198, 604 |
| 3-Acetamido-6-isopentyloxypyridazine | — | *H* 552 |
| 3-Acetamido-6-isopentyloxypyridazine 2-oxide | — | *H* 604 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3-Acetamido-6-isopropoxypyridazine | — | *H* 552 |
| 3-Acetamido-6-isopropoxypyridazine 2-oxide | — | *H* 603 |
| 4-Acetamido-3-methoxy-5-methylpyridazine 1-oxide | 166–167, nmr | 902 |
| 4-Acetamido-6-methoxy-2-methyl-3(2*H*)-pyridazinone | — | *H* 99, 563 |
| 3-Acetamido-6-methoxy-4-methylthiopyridazine | — | *H* 809 |
| 3-Acetamido-6-methoxy-5-nitropyridazine 1-oxide | — | *H* 197 |
| 3-Acetamido-6-methoxy-5-nitropyridazine 2-oxide | — | *H* 604 |
| 4-Acetamido-6-methoxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 108 |
| 3-Acetamido-6-methoxypyridazine | — | *H* 176, 552 |
| 3-Acetamido-6-methoxypyridazine 2-oxide | — | *H* 603 |
| 4-Acetamido-6-methyl-2-phenyl-3(2*H*)-pyridazinone | — | *H* 106, 563 |
| 4-Acetamido-6-methyl-3(2*H*)-pyridazinone | — | *H* 97 |
| 3-Acetamido-6-methylthiopyridazine | — | *H* 803 |
| 3-Acetamido-6-methylthiopyridazine 2-oxide | — | *H* 821 |
| 3-Acetamido-5-nitro-6-pentyloxypyridazine 2-oxide | — | *H* 198, 604 |
| 3-Acetamido-5-nitro-6-propoxypyridazine 2-oxide | — | *H* 198, 604 |
| 3-Acetamido-6-pentyloxypyridazine | — | *H* 552 |
| 3-Acetamido-6-pentyloxypyridazine 2-oxide | — | *H* 604 |
| 3-Acetamido-6-propoxypyridazine | — | *H* 552 |
| 3-Acetamido-6-propoxypyridazine 2-oxide | — | *H* 604 |
| 3-Acetamidopyridazine | — | *H* 550 |
| 4-Acetamidopyridazine | — | *H* 557 |
| 3-Acetamidopyridazine 1-oxide | — | *H* 603 |
| 3-Acetamidopyridazine 2-oxide | — | *H* 603 |
| 4-Acetamidopyridazine 1-oxide | — | *H* 603 |
| 6-Acetamido-3-pyridazinesulfonamide | — | *H* 553, 823 |
| 6-Acetamido-3(2*H*)-pyridazinethione | — | *H* 553, 797 |
| 4-Acetamido-3(2*H*)-pyridazinone | — | *H* 560 |
| 5-Acetamido-3(2*H*)-pyridazinone | — | *H* 85, 560 |
| 2-Acetonyl-5-amino-4-bromo-3(2*H*)-pyridazinone | 192–193, ir, ms, nmr | 1884 |
| 2-Acetonyl-6-amino-4-chloro-5-methoxy-3(2*H*)-pyridazinone | 160–161, ir, nmr | 2039 |
| 2-Acetonyl-5-amino-4-chloro-3(2*H*)-pyridazinone | 194–195, ir, ms, nmr | 1884 |
| 2-Acetonyl-6-amino-4,5-dichloro-3(2*H*)-pyridazinone | 167–168, ir, nmr | 2039 |
| 2-Acetonyl-4-bromo-5-cyclopropylamino-3(2*H*)-pyridazinone | 132–133, ir, nmr | 2039 |
| 2-Acetonyl-4-bromo-5-hydroxy-3(2*H*)-pyridazinone | 190–191, ir, nmr | 1936 |
| 2-Acetonyl-4-bromo-5-methoxy-3(2*H*)-pyridazinone | 138–139, ir, nmr; oxime: 109–110, ir, nmr | 1936, 2039 |
| 2-Acetonyl-4-bromo-5-methylamino-3(2*H*)-pyridazinone | 167–168, ir, nmr | 2039 |
| 2-Acetonyl-4-chloro-4-hydroxy-3(2*H*)-pyridazinone | 215–216, ir, nmr | 1936 |
| 2-Acetonyl-4-chloro-5-methoxy-6-nitro-3(2*H*)-pyridazinone | 74–75, ir, nmr | 1673 |
| 2-Acetonyl-4-chloro-5-methoxy-3(2*H*)-pyridazinone | 121–122 or 131–132, ir, nmr | 1673, 1936 |
| 2-Acetonyl-6-chloro-5-methoxy-3(2*H*)-pyridazinone | — | *H* 92, 326, 400 |

*continued*

| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 2-Acetonyl-4,5-dibromo-6-hydroxyamino-3(2*H*)-pyridazinone | 165–166, ir, nmr | 2039 |
| 2-Acetonyl-4,5-dibromo-6-nitro-3(2*H*)-pyridazinone | 136–137, ir, nmr; oxime: 137–138, ir, nmr | 2039 |
| 2-Acetonyl-4,5-dibromo-3(2*H*)-pyridazinone | 167–168, ir, nmr; oxime: 124–125, ir, nmr | 1936, 2039 |
| 2-Acetonyl-4,5-dichloro-6-nitro-3(2*H*)-pyridazinone | 93–95, ir, nmr; oxime: 129–130, ir, nmr | 1055, 1673, 2039 |
| 1-Acetonyl-4,5-dichloro-3,6(1*H*,2*H*)-pyridazinedione | 203–204, ir, nmr | 2039 |
| 2-Acetonyl-4,5-dichloro-3(2*H*)-pyridazinone | 136–137 or 139–140, ir, nmr; oxime: 120–121, ir, nmr | 1055, 1673, 1936, 2039 |
| 2-Acetonyl-5,6-dichloro-3(2*H*)-pyridazinone | — | *H* 92, 327, 400 |
| 1-Acetonyl-6-hydroxyimino-3-phenyl-1,6-dihydropyridazine | oxime: 167–169, ir, nmr | 859 |
| 3-Acetonyl-6-methoxypyridazine | liq, anal, ir, nmr | 326 |
| 3-Acetonyl-6-methylpyridazine | 69–70, ir, nmr, uv | 825 |
| 2-Acetonyl-6-methyl-3(2*H*)-pyridazinone | — | *H* 73, 401 |
| 2-Acetonyl-6-phenyl-3(2*H*)-pyridazinone | dioxime: 167–169, nmr | 398; *H* 73, 400 |
| 3-Acetonylpyridazine | liq, ir, nmr, uv | 825 |
| 2-Acetonyl-3(2*H*)-pyridazinone | — | *H* 65, 401 |
| 5-Acetoxy-4-bromo-2-phenyl-3(2*H*)-pyridazinone | — | *H* 120, 319 |
| 6-Acetoxy-4-bromo-3(2*H*)-pyridazinone | 186–189, nmr | 163 |
| 2-δ-Acetoxybutyl-4,5-dichloro-3(2*H*)-pyridazinone | 83–84, ir, nmr | 1700 |
| 2-Acetoxy-5-chloro-6-methoxy-3(2*H*)-pyridazinone | — | *H* 90, 326 |
| 5-Acetoxy-4-chloro-2-phenyl-3(2*H*)-pyridazinone | — | *H* 123 |
| 2-Acetoxy-6-chloro-3(2*H*)-pyridazinone | — | *H* 76 |
| 6-Acetoxy-4,5-dibromo-3(2*H*)-pyridazinone | — | *H* 127, 330 |
| 6-Acetoxy-2,4 / 2,5-diphenyl-3(2*H*)-pyridazinone | 172 | 462 |
| 2-Acetoxy-6-ethoxy-5-methyl-3(2*H*)-pyridazinone | — | *H* 90 |
| 2-Acetoxy-6-ethoxy-3(2*H*)-pyridazinone | — | *H* 76 |
| 2-β-Acetoxyethyl-6-chloro-3(2*H*)-pyridazinone | — | *H* 73, 305 |
| 1-β-Acetoxyethyl-4,5-dichloro-2-isopropyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 167 |
| 6-Acetoxy-2-ethyl-3(2*H*)-pyridazinone | 76–77, ir, nmr | 292 |
| 2-Acetoxy-6-methoxy-5-methyl-3(2*H*)-pyridazinone | — | *H* 90 |
| 5-Acetoxy-4-methoxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 126 |
| 2-Acetoxy-6-methoxy-3(2*H*)-pyridazinone | — | *H* 76 |
| 2-Acetoxymethyl-4,5-dichloro-3(2*H*)-pyridazinone | — | *H* 113, 308 |
| 4-Acetoxymethyl-3,6-dimethoxypyridazine | — | *H* 192 |
| 3-Acetoxymethyl-6-ethoxypyridazine | — | *H* 177 |
| 3-Acetoxymethyl-6-methoxypyridazine | — | 913; *H* 176 |
| 3-Acetoxymethyl-6-methylpyridazine 1-oxide | 101–102, ir, nmr | 529 |
| 2-Acetoxymethyl-3-oxo-2,3-dihydro-4-pyridazinecarbonitrile | 47–49, ir, nmr | 1300 |
| 6-Acetoxymethyl-5-oxo-2,5-dihydro-3-pyridazinecarboxylic acid | 199, ir | 778 |

*continued*

| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 5-Acetoxymethyl-6-phenyl-3(2*H*)-pyridazinone | 129–132, ir, nmr | 1599, 2018 |
| 4-Acetoxy-6-methyl-3-pyridazinamine | — | *H* 556 |
| 3-Acetoxymethyl-4(1*H*)-pyridazinone | 207, nmr | 778 |
| 6-Acetoxymethyl-4(1*H*)-pyridazinone | 211, ir, nmr, uv | 778 |
| 6-Acetoxy-2-methyl-3(2*H*)-pyridazinone | 90–92 | 292 |
| 6-Acetoxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 81 |
| 2-Acetoxy-3(2*H*)-pyridazinimine | — | *H* 603 |
| 6-Acetoxy-3(2*H*)-pyridazinone | nmr | 163; *H* 68 |
| 5-Acetyl-2-allyl-4-amino-6-phenyl-3(2*H*)-pyridazinone | 93–95, ir, nmr | 1832 |
| 5-Acetyl-4-amino-2-β-cyanoethyl-6-phenyl-3(2*H*)-pyridazinone | 187–188, ir, nmr | 1769 |
| 5-Acetyl-4-amino-2-cyclohexyl-6-phenyl-3(2*H*)-pyridazinone | 214–215, ir, nmr | 1769 |
| 5-Acetyl-4-amino-2,6-diphenyl-3(2*H*)-pyridazinone | 167–169, ir, nmr | 1590 |
| 6-Acetyl-4-amino-5-hydroxy-2-phenyl-3(2*H*)-pyridazinone | 162–163, nmr | 1789 |
| 6-Acetyl-5-amino-3-imino-2-phenyl-2,3-dihydro-4-pyridazinecarbonitrile | 240, ir, nmr | 395 |
| 5-Acetyl-4-amino-2-β-methoxycarbonylethyl-6-phenyl-3(2*H*)-pyridazinone | 115, ir, nmr | 1769 |
| 5-Acetyl-4-amino-2-methyl-6-phenyl-3(2*H*)-pyridazinone | 199–200 or 200–203, ir, nmr | 223, 241, 1590 |
| 5-Acetyl-4-amino-6-methyl-5-phenyl-3(2*H*)-pyridazinone | 142–144, ir, nmr | 1769 |
| 6-Acetyl-4-amino-5-methyl-2-phenyl-3(2*H*)-pyridazinone | 188–190, ir, nmr | 1789 |
| 5-Acetyl-4-amino-6-methyl-3(2*H*)-pyridazinone | — | *H* 395, 561 |
| 5-Acetyl-4-amino-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarbonitrile | 280, ir, nmr | 1665 |
| 6-Acetyl-5-amino-3-oxo-2-phenyl-2,3-dihydro-4-pyridazinecarbonitrile | 300, ir, nmr | 395 |
| 6-Acetyl-5-amino-3-oxo-2-phenyl-2,3-dihydro-4-pyridazinecarboxamide | 234, ir, nmr | 395 |
| 4-Acetyl-5-amino-6-phenyl-3(2*H*)-pyridazinone | — | *H* 395, 561 |
| 5-Acetyl-4-amino-6-phenyl-3(2*H*)-pyridazinone | — | 1590; *H* 395, 561 |
| 4-Acetyl-5-amino-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 395, 561 |
| 4-Acetyl-3-anilino-5,6-diphenylpyridazine | 195–197, ir | 377 |
| 5-Acetyl-4-anilino-2-methyl-6-phenyl-3(2*H*)-pyridazinone | 185–186, ir, nmr | 1769 |
| 4-Acetyl-3-azido-5,6-diphenylpyridazine | 174, ir | 377 |
| 4-Acetyl-6-benzyl-5-hydroxy-3(2*H*)-pyridazinone | 198, nmr | 1122 |
| 5-Acetyl-2-benzyl-4-nitro-6-phenyl-3(2*H*)-pyridazinone | 140–142, ir, nmr | 1769 |
| 5-Acetyl-4-bromo-2-methyl-6-phenyl-3(2*H*)-pyridazinone | 128–130, ir, nmr | 1766 |
| 5-Acetyl-4-bromo-2-phenyl-3(2*H*)-pyridazinone | — | *H* 119 |
| 4-Acetyl-3-butylamino-5,6-diphenylpyridazine | 148, ir | 377 |
| 2-α-Acetylbutyl-6-methyl-3(2*H*)-pyridazinone | — | *H* 75, 400 |

*continued*

| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 4-Acetyl-2-γ-carboxypropyl-5,6-diphenyl-3(2*H*)-pyridazinone | 170–172 | 1272 |
| 4-Acetyl-3-chloro-5,6-diphenylpyridazine | 155, ir | 377, 1411 |
| 4-Acetyl-6-chloro-5-iodo-3-methoxypyridazine | 128–130, ir, nmr | 1610 |
| 4-Acetyl-6-chloro-3-methoxy-5-phenylpyridazine | 129–131, ir, nmr | 1610 |
| 5-Acetyl-4-chloro-2-methyl-6-phenyl-3(2*H*)-pyridazinone | 100–101, ir, nmr | 1561, 1766 |
| 6-Acetyl-4-chloro-5-methyl-2-phenyl-3(2*H*)-pyridazinone | 95, ir, nmr | 1620 |
| 4-Acetyl-3-chloro-6-methylpyridazine | liq, nmr | 1514 |
| 4-Acetyl-3-chloro-6-phenylpyridazine | 92–94, nmr | 1514 |
| 5-Acetyl-2-β-cyanoethyl-4-nitro-6-phenyl-3(2*H*)-pyridazinone | 141–142, ir, nmr | 1769 |
| 5-Acetyl-2-cyclohexyl-4-nitro-6-phenyl-3(2*H*)-pyridazinone | 144–146, ir, nmr | 132 |
| 5-Acetyl-4-diethylamino-2-methyl-6-phenyl-3(2*H*)-pyridazinone | liq, anal, ir, nmr | 1414, 1769 |
| 5-Acetyl-2,6-dimethyl-4-nitro-3(2*H*)-pyridazinone | 82, ir, nmr | 132 |
| 4-Acetyl-3,6-dimethylpyridazine | 97–100/20 | 125 |
| 4-Acetyl-5,6-diphenyl-3-piperidinopyridazine | 106, ir | 377 |
| 4-Acetyl-5,6-diphenyl-3-pyridazinamine | — | 1411; *H* 555 |
| 4-Acetyl-5,6-diphenyl-3(2*H*)-pyridazinethione | 212, ir | 377 |
| 4-Acetyl-5,6-diphenyl-3(2*H*)-pyridazinone | 232–234; $H_2NN{=}$: 264–266 | 982, 1056, 1272 |
| 3-Acetyl-4-ethoxy-1-phenyl-6-phenylimino-1,6-dihydropyridazine | 164, ir, ms, nmr | 17 |
| 2-β-Acetylethyl-6-chloro-3(2*H*)-pyridazinone | — | *H* 399 |
| 1-β-Acetylethyl-4,5-dichloro-2-isopropyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 336 |
| 2-β-Acetylethyl-4,5-dichloro-3(2*H*)-pyridazinone | — | *H* 399 |
| 2-α-Acetylethyl-6-methyl-3(2*H*)-pyridazinone | — | *H* 73, 401 |
| 2-β-Acetylethyl-6-methyl-3(2*H*)-pyridazinone | — | *H* 75, 400 |
| 5-Acetyl-2-ethyl-4-nitro-6-phenyl-3(2*H*)-pyridazinone | 107–108, ir, nmr | 1997 |
| 1-β-Acetylethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 155, 399 |
| 2-β-Acetylhydrazino-6-chloropyridazine | — | *H* 279, 668 |
| 4-Acetyl-2-β-hydroxyethyl-5,6-diphenyl-3(2*H*)-pyridazinone | 126–128 | 1272 |
| 4-Acetyl-5-hydroxy-6-methyl-3(2*H*)-pyridazinone | 242, nmr | 1122 |
| 4-Acetyl-5-hydroxy-6-morpholinomethyl-3(2*H*)-pyridazinone | 254–258, nmr | 1122 |
| 4-Acetyl-5-hydroxy-6-phenyl-3(2*H*)-pyridazinone | 228, nmr | 1122 |
| 6-Acetyl-2-hydroxy-3(2*H*)-pyridazinone | — | *H* 396 |
| 3-Acetylimino-6-chloro-2-methyl-2,3-dihydropyridazine | — | *H* 302 |
| 3-Acetyl-6-isopentylpyridazine | ir, nmr | 1660 |
| 5-Acetyl-2-β-methoxycarbonylethyl-4-nitro-6-phenyl-3(2*H*)-pyridazinone | 78–79, ir, nmr | 1769 |
| 3-Acetyl-4-methoxy-1-phenyl-6-phenylimino-1,6-dihydropyridazine | 164 | 269 |

*continued*

| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3-Acetyl-6-methoxypyridazine | — | *H* 396 |
| 3-Acetyl-6-methoxypyridazine 1-oxide | — | *H* 398 |
| 3-Acetyl-6-methoxypyridazine 2-oxide | — | *H* 398 |
| 4-Acetyl-3-methyl-5,6-diphenylpyridazine | 131–132, ir, ms, nmr | 314 |
| 4-Acetyl-2-methyl-5,6-diphenyl-3(2*H*)-pyridazinone | 151–152 | 1272; *H* 396 |
| 5-(β-Acetyl-α-methylhydrazino)-2-benzyl-3(2*H*)-pyridazinone | 204–205, ir, nmr | 873 |
| 5-(β-Acetyl-α-methylhydrazino)-2-methyl-3(2*H*)-pyridazinone | 186–187, ir, nmr | 873 |
| 5-(β-Acetyl-α-methylhydrazino)-2-phenyl-3(2*H*)-pyridazinone | 226–227, ir, nmr | 873 |
| 5-Acetyl-2-methyl-4-methylsulfinyl-6-phenyl-3(2*H*)-pyridazinone | 128–130, ir, nmr, xl st | 1766, 1974, 2075 |
| 5-Acetyl-2-methyl-4-methylsulfonyl-6-phenyl-3(2*H*)-pyridazinone | 182–183, ir, nmr | 1766 |
| 5-Acetyl-2-methyl-4-methylthio-6-phenyl-3(2*H*)-pyridazinone | 106–107, ir, nmr | 1766 |
| 5-Acetyl-2-methyl-4-morpholino-6-phenyl-3(2*H*)-pyridazinone | 134–135, ir, nmr | 1414, 1769 |
| 5-Acetyl-2-methyl-4-nitro-6-phenyl-3(2*H*)-pyridazinone | 140–141, ir, nmr | 132 |
| 5-Acetyl-3-methyl-6-oxo-1,6-dihydro-4-pyridazinecarboxylic acid | 285, ir, nmr | 128 |
| 5-Acetyl-4-methyl-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarbonitrile | >300, ir, nmr | 395 |
| 3-Acetyl-4-methyl-1-phenyl-6-phenylimino-1,6-dihydropyridazine | 152, ms, nmr | 17, 269 |
| 5-Acetyl-2-methyl-6-phenyl-4-piperidino-3(2*H*)-pyridazinone | — | 1414 |
| 5-Acetyl-2-methyl-6-phenyl-3,4(1*H*,2*H*)-pyridazinedione | 191–193, ir, nmr; oxime: 195, ir, nmr | 1561 |
| 3-Acetyl-6-methyl-1-phenyl-4(1*H*)-pyridazinone | 128–129, ir, nmr | 1706 |
| 5-Acetyl-2-methyl-6-phenyl-3(2*H*)-pyridazinone | 108, ir, nmr | 1769 |
| 6-Acetyl-5-methyl-2-phenyl-3(2*H*)-pyridazinone | 134, ir, ms, nmr | 17, 269, 1350, 1451, 2014 |
| 2-(α-Acetyl-β-methylpropyl)-6-methyl-3(2*H*)-pyridazinone | — | *H* 400 |
| 3-Acetyl-6-methylpyridazine | 46–48, 124–127/6, ir, ms, nmr | 125, 1638, 1744 |
| 2-Acetyl-4-methyl-3(2*H*)-pyridazinone | — | *H* 400 |
| 4-Acetyl-6-methyl-3(2*H*)-pyridazinone | 206–208 | 1514 |
| 4-Acetyl-3-morpholino-5,6-diphenylpyridazine | 133, ir | 377 |
| 3-Acetyl-6-morpholinopyridazine | — | 807 |
| 5-Acetyl-4-nitro-2,6-diphenyl-3(2*H*)-pyridazinone | 116, ir, nmr | 132 |
| 4-Acetyl-3-phenoxy-5,6-diphenylpyridazine | 101, ir | 377 |
| 4-Acetyl-6-phenyl-3(2*H*)-pyridazinone | >300 | 1514 |
| 4-Acetyl-3-phenyl-5-trimethylsilylpyridazine | 118, ms, nmr | 12 |
| 2-α-Acetylpropyl-6-methyl-3(2*H*)-pyridazinone | — | *H* 73, 400 |

*continued*

| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3-Acetylpyridazine | 89–90; $H_2NN$=: 67–69, ir, nmr; *p*-$BrC_6H_4NHN$=: 222–224, ir, nmr | 35, 1347; *H* 396 |
| 4-Acetylpyridazine | 69–70, ir, nmr; oxime: 169–172, nmr | 65, 73, 1347, 1668 |
| 1-Acetyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 153, 399 |
| 3-Acetylpyridazine 1-oxide | — | *H* 398 |
| 6-Acetyl-3(2*H*)-pyridazinone | — | *H* 396 |
| 4-Acetylthio-6-chloro-2-phenyl-3(2*H*)-pyridazinone | — | *H* 820 |
| 5-Acetylthio-4-methoxy-2-phenyl-3(2*H*)-pyridazinone | 101–102 | 1896 |
| 6-Acetylthio-3(2*H*)-pyridazinethione | — | *H* 820 |
| 6-Acryloyloxy-2-*β*-chloroethyl-3(2*H*)-pyridazinone | polymer | 1112 |
| 2-Allyl-6-allyloxy-3(2*H*)-pyridazinone | — | 855; *H* 73 |
| 4-Allylamino-6-chloro-3-hydrazinopyridazine | — | *H* 583 |
| 5-Allylamino-4-chloro-2-phenyl-3(2*H*)-pyridazinone | — | *H* 121, 313 |
| 3-Allylamino-6-chloropyridazine | — | *H* 574 |
| 4-Allylamino-3,6-dichloropyridazine | — | *H* 583 |
| 3-Allylamino-4-methoxy-6-methylpyridazine | — | *H* 574 |
| 3-Allylamino-6-methyl-4-(1*H*)-pyridazinone | — | *H* 574 |
| 2-Allyl-4-bromo-5-hydroxy-3(2*H*)-pyridazinone | 176–177, ir, nmr | 1936 |
| 2-Allyl-4-bromo-5-methoxy-3(2*H*)-pyridazinone | 68–69, ir, nmr | 1936 |
| 2-Allyl-4-chloro-5-hydroxy-3(2*H*)-pyridazinone | 176–177, ir, nmr | 1936 |
| 2-Allyl-4-chloro-5-methoxy-3(2*H*)-pyridazinone | 68–69, ir, nmr | 1936 |
| Allyl 5-chloro-1-methyl-6-oxo-1,6-dihydro-4-pyridazinecarboxylate | — | *H* 315 |
| 2-Allyl-4-chloro-5-morpholino-3(2*H*)-pyridazinone | 104–105 | 1269 |
| 2-Allyl-4,5-dibromo-3(2*H*)-pyridazinone | 66–67, ir, nmr | 1936 |
| 2-Allyl-4,5-dichloro-3(2*H*)-pyridazinone | 44–46, ir, nmr | 1936 |
| 2-Allyl-5,6-dichloro-3(2*H*)-pyridazinone | — | *H* 92, 327 |
| 1-Allyl-4-dimethylamino-2-phenyl-3,6(1*H*,3*H*)-pyridazinedione | — | *H* 595 |
| 2-Allyl-4-ethoxy-5-morpholino-3(2*H*)-pyridazinone | liq, anal | 1269 |
| 1-Allylideneamino-3,6(1*H*,2*H*)-pyridazinedione | — | 1050 |
| 2-Allyl-6-methyl-3(2*H*)-pyridazinone | — | 855; *H* 73 |
| 2-Allyl-5-morpholino-3(2*H*)-pyridazinone | 92–93 | 1269 |
| 5-Allyloxy-4-chloro-2-phenyl-3(2*H*)-pyridazinone | — | *H* 123 |
| 3-Allyloxy-6-chloropyridazine | — | *H* 178 |
| 6-Allyloxy-4-dimethylamino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 109, 589 |
| 3-Allyloxy-6-isopropylaminopyridazine | — | *H* 178, 573 |
| 6-Allyloxy-4-methylamino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 588 |
| 4-Allyloxy-2-methyl-5-morpholino-3(2*H*)-pyridazinone | 60–61 | 1269 |
| 4-Allyloxy-2-methyl-1-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 160 |
| 2-Allyl-4-phenethyl-6-phenyl-3(2*H*)-pyridazinone | 62–62, ir, nmr | 1959 |
| 2-Allyl-6-phenyl-3(2*H*)-pyridazinone | 163–165 / 1.5, nmr | 118, 855 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 1-Allyl-3,6(1*H*,2*H*)-pyridazinedione | — | 855 |
| 2-Allyl-3(2*H*)-pyridazinone | — | 855 |
| 3-Allylthio-6-chloro-4-methylpyridazine | — | *H* 806 |
| 3-Allylthio-6-chloro-5-methylpyridazine | — | *H* 806 |
| 4-Allylthio-6-chloro-2-phenyl-3(2*H*)-pyridazinone | — | *H* 811 |
| 5-Allylthio-4-chloro-2-phenyl-3(2*H*)-pyridazinone | — | *H* 812 |
| 5-Allylthio-6-chloro-2-phenyl-3(2*H*)-pyridazinone | — | *H* 812 |
| 3-Allylthio-6-chloropyridazine | 66 | 557; *H* 284, 804 |
| 6-Allylthio-3-pyridazinamine | 101–102, nmr | 1842 |
| 5-Amino-4-anilino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 602 |
| 5-Amino-3-azido-4(1*H*)-pyridazinethione | 150, ir | 1228 |
| 5-Amino-6-azido-3(2*H*)-pyridazinethione | >300, ir, nmr, uv | 1228 |
| 4-Amino-5-benzoyl-6-imino-1-phenyl-1,6-dihydro-3-pyridazinecarboxylic acid | >300, ir | 1665 |
| 4-Amino-5-benzoyl-2-methyl-3(2*H*)-pyridazinone | 141, ir, nmr | 1501 |
| 4-Amino-6-benzoyloxy-5-nitro-2-phenyl-3(2*H*)-pyridazinone | — | *H* 131 |
| 4-Amino-5-benzoyl-6-phenyl-3(2*H*)-pyridazinone | — | *H* 395, 561 |
| 4-Amino-2-benzyl-6-benzyloxy-5-nitro-3(2*H*)-pyridazinone | — | *H* 602 |
| 5-Amino-2-benzyl-6-benzyloxy-3,4(1*H*,2*H*)-pyridazinedione | — | *H* 169 |
| 3-Amino-5-benzyl-6-methyl-4(1*H*)-pyridazinone | — | *H* 133, 556 |
| 3-Amino-6-benzyl-4(1*H*)-pyridazinone | — | *H* 133, 556 |
| 5-Amino-4-benzylsulfonyl-2-methyl-3(2*H*)-pyridazinone | 248–251, nmr | 1903 |
| 5-Amino-4-benzylthio-3(2*H*)-pyridazinone | — | *H* 559 |
| 6-Amino-4-benzylthio-3(2*H*)-pyridazinone | — | *H* 811 |
| 5-Amino-4-bromo-2-$\beta$-cyanoethyl-3(2*H*)-pyridazinone | 219–220, ir, ms, nmr | 1884 |
| 5-Amino-4-bromo-2-cyclohexyl-3(2*H*)-pyridazinone | — | *H* 121, 321, 565 |
| 5-Amino-4-bromo-2-$\beta$-hydroxyiminopropyl-3(2*H*)-pyridazinone | 204–205, ir, ms, nmr | 1884 |
| 4-Amino-5-bromo-1-methyl-2-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 159 |
| 4-Amino-5-bromo-6-methyl-3-pyridazinecarbaldehyde | $Me_2$acetal: 74–75, nmr | 996 |
| 4-Amino-5-bromo-2-methyl-3(2*H*)-pyridazinone | 126–127, nmr | 956 |
| 5-Amino-4-bromo-2-methyl-3(2*H*)-pyridazinone | 215–217, nmr | 956 |
| 5-Amino-4-bromo-2-phenyl-3(2*H*)-pyridazinone | — | 580; *H* 120, 319 |
| 5-Amino-3-butylamino-6-chloro-4-pyridazinecarbaldehyde | 270–271, ir, nmr | 524 |
| 2-$\delta$-Aminobutyl-6-phenyl-3(2*H*)-pyridazinone | crude: liq, ir, nmr; HCl: 206–207 | 1289, 1337 |
| 5-Amino-2-carboxymethyl-4-chloro-3(2*H*)-pyridazinone | — | *H* 119, 312, 565 |
| 5-Amino-4-chloro-2-$\beta$-cyanoethyl-3(2*H*)-pyridazinone | — | *H* 119, 312, 565 |
| 5-Amino-4-chloro-2-cyclohexyl-3(2*H*)-pyridazinone | — | *H* 315, 564 |
| 4-Amino-5-chloro-2,6-dimethyl-3(2*H*)-pyridazinone | — | *H* 129, 328, 563 |
| 5-Amino-4-chloro-2,6-dimethyl-3(2*H*)-pyridazinone | — | *H* 129, 328, 564 |
| 5-Amino-4-chloro-2-ethyl-3(2*H*)-pyridazinone | — | *H* 119, 312, 564 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 5-Amino-4-chloro-2-δ-hydroxybutyl-3(2*H*)-pyridazinone | 170–171, ir, nmr | 1700 |
| 5-Amino-4-chloro-2-β-hydroxyethyl-3(2*H*)-pyridazinone | — | *H* 119, 312, 565 |
| 5-Amino-4-chloro-2-methoxycarbonylmethyl-3(2*H*)-pyridazinone | 222–223, ir, ms, nmr | 1884 |
| 5-Amino-4-chloro-2-γ-methoxypropyl-3(2*H*)-pyridazinone | — | *H* 119, 312, 565 |
| 4-Amino-5-chloro-6-methoxy-3-pyridazinecarbonitrile | — | *H* 184, 447, 562 |
| 3-Amino-6-chloro-5-methyl-4-pyridazinecarbonitrile | 220, nmr, uv | 821 |
| 4-Amino-5-chloro-2-methyl-3(2*H*)-pyridazinone | 108 or 113, ir, nmr, uv | 148, 1213, 1677 |
| 4-Amino-6-chloro-2-methyl-3(2*H*)-pyridazinone | — | *H* 98, 323, 563 |
| 5-Amino-4-chloro-2-methyl-3(2*H*)-pyridazinone | 203–205, ir, nmr, uv | 148, 1213, 1677; *H* 119, 312, 564 |
| 5-Amino-6-chloro-2-methyl-3(2*H*)-pyridazinone | p$K_a$, uv | 675; *H* 91, 326, 564 |
| 4-Amino-6-chloro-5-nitro-3(2*H*)-pyridazinone | — | *H* 561 |
| 5-Amino-3-chloro-4-nitro-3(2*H*)-pyridazinone | — | *H* 561 |
| 4-Amino-6-chloro-2-phenyl-3(2*H*)-pyridazinethione | — | *H* 333, 563, 798 |
| 4-Amino-5-chloro-2-phenyl-3(2*H*)-pyridazinone | 140–141 or 141–142, ir, nmr, uv | 148, 956, 1213; *H* 125, 328, 563 |
| 4-Amino-6-chloro-2-phenyl-3(2*H*)-pyridazinone | 198–200 | 402; *H* 104, 323, 563 |
| 5-Amino-4-chloro-2-phenyl-3(2*H*)-pyridazinone | 203 to 207, ir, nmr, p$K_a$, uv | 148, 464, 576, 865, 956, 1167, 1213, 1344; *H* 121, 313, 565 |
| 5-Amino-6-chloro-2-phenyl-3(2*H*)-pyridazinone | — | *H* 93, 327 |
| 3-Amino-6-chloro-4-pyridazinecarbonitrile | 192–195, uv | 820 |
| 4-Amino-6-chloro-3-pyridazinecarbonitrile | — | *H* 446, 562 |
| 3-Amino-6-chloro-4-pyridazinecarboxamide | 258–259 | 1099 |
| 6-Amino-3-chloro-4-pyridazinecarboxamide | 245–246 | 1099 |
| 3-Amino-6-chloro-4-pyridazinecarboxylic acid | — | *H* 555 |
| 4-Amino-6-chloro-3(2H)-pyridazinethione | 225, nmr | 654; *H* 293, 559, 798 |
| 4-Amino-5-chloro-3(2*H*)-pyridazinone | — | *H* 125, 328, 559 |
| 4-Amino-6-chloro-3-(2*H*)-pyridazinone | 283 or 293–294, ir, nmr | 654, 1597; *H* 96, 293, 559 |
| 5-Amino-3-chloro-4(1*H*)-pyridazinone | — | *H* 132, 190, 559 |
| 5-Amino-4-chloro-3(2*H*)-pyridazinone | 299–300, nmr, p$K_a$ | 1130, 1167, 1597; *H* 116, 310, 559 |
| 5-Amino-6-chloro-3(2*H*)-pyridazinone | — | *H* 87, 291, 559 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 5-Amino-6-cyano-3-imino-2-phenyl-2,3-dihydro-4-pyridazinecarboxamidrazone | >300, ir | 55 |
| 4-Amino-5-cyano-6-imino-1-phenyl-1,6-dihydro-3-pyridazinecarboxylic acid | 210 or >300, ir, nmr, xl st | 51, 151, 1665 |
| 5-Amino-6-cyano-3-imino-2-phenyl-2,3-dihydro-4-pyridazinecarboxylic acid | 218, ir | 151 |
| 4-Amino-5-cyano-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarbohydrazide | 258–261, ir, nmr | 55 |
| 4-Amino-5-cyano-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxamide | 276, ir, nmr | 87, 898 |
| 4-Amino-5-cyano-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylic acid | 198 or >300, ir, nmr | 55, 129, 229 |
| 4-Amino-5-cyano-1-phenyl-6-thioxo-1,6-dihydro-3-pyridazinecarbothioamide | 150–152, ir, nmr | 1214 |
| 5-Amino-2-cyclohexyl-3,4(1*H*,2*H*)-pyridazinedione | — | *H* 169 |
| 6-Amino-4,5-dichloro-2-ethyl-3(2*H*)-pyridazinone | — | *H* 130, 329, 571 |
| 6-Amino-4,5-dichloro-2-methyl-3(2*H*)-pyridazinone | 191–193 or 193–195, ir, nmr | 846, 1938; *H* 128, 329, 571 |
| 4-Amino-5,6-dichloro-3-pyridazinecarbonitrile | — | *H* 447, 562 |
| 5-Amino-3,6-dichloro-4-pyridazinecarbonitrile | 240–242, uv | 820 |
| 4-Amino-5,6-dichloro-3-pyridazinecarboxamide | — | *H* 562 |
| 4-Amino-5,6-dichloro-3-pyridazinecarboxylic acid | — | *H* 435 |
| 4-Amino-5-diethylamino-1,2-dimethyl-3,6(1*H*,2*H*)-pyridazinedione | 135–138, ir, nmr | 402 |
| 5-Amino-4-dimethylamino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 602 |
| 4-(γ-Amino-β,β-dimethylpropyl)-3,6-diphenylpyridazine | 116–117, ir, ms, nmr, uv | 512 |
| 3-Amino-5,6-dimethyl-4-pyridazinecarbonitrile | — | *H* 447, 555 |
| 3-Amino-5,6-dimethyl-4-pyridazinecarbothioamide | — | *H* 555 |
| 4-Amino-1,2-dimethyl-3,6(1*H*,2*H*)-pyridazinedione | 205–207 | 402 |
| 1-Amino-3,6-dimethylpyridazinium (+anion) | I: 134–136, nmr; mesitylenesulfonate: 78–80 | 782, 1359 |
| 3-Amino-5,6-dimethyl-4(1*H*)-pyridazinone | — | *H* 133, 556 |
| 4-Amino-2,6-dimethyl-3(2*H*)-pyridazinone | — | *H* 99, 563 |
| 5-Amino-2,6-dimethyl-3(2*H*)-pyridazinone | — | *H* 91, 564 |
| 5-Amino-3,6-dimorpholino-4-pyridazinecarbaldehyde | 194–195, ir, nmr | 524 |
| 5-Amino-3,6-dioxo-1-phenyl-1,2,3,6-tetrahydro-4-pyridazinecarbonitrile | 226, ir, nmr | 232 |
| 3-Amino-5,6-diphenyl-4-pyridazinecarbonitrile | 235–236, uv | 364, 1056; *H* 555 |
| 3-Amino-5,6-diphenyl-4-pyridazinecarbothioamide | 208 | 364 |
| 3-Amino-5,6-diphenyl-4-pyridazinecarboxamide | — | *H* 555 |
| 4-Amino-5,6-diphenyl-3(2*H*)-pyridazinone | — | 809 |
| 5-Amino-2-α-ethoxycarbonylethyl-3,4(1*H*,2*H*)-pyridazinedione | — | *H* 169 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 4-Amino-5-α-ethoxyethyl-2-methyl-6-phenyl-3(2*H*)-pyridazinone | 187–188, ir, nmr | 1832 |
| 2-β-Aminoethyl-4,5-dichloro-3(2*H*)-pyridazinone | — | *H* 113, 308 |
| 4-Amino-5-ethyl-2-methyl-6-phenyl-3(2*H*)-pyridazinone | 164–165, ir, nmr | 1832 |
| 2-β-Aminoethyl-6-phenyl-3(2*H*)-pyridazinone | 75–77, ir, nmr; HCl: 196–197 | 1289, 1337 |
| 3-β-Aminoethylpyridazine | — | *H* 609 |
| 6-α-Aminoethyl-3(2*H*)-pyridazinone | HCl: 233–237, ir, nmr, uv | 731 |
| 4-Amino-5-formamido-6-methoxy-3(2*H*)-pyridazinone | — | *H* 128, 599 |
| 2-ζ-Aminohexyl-6-phenyl-3(2*H*)-pyridazinone | crude: liq, ir, nmr, HCl: 75–78 | 1289, 1337 |
| 4-Amino-6-hydrazino-3(2*H*)-pyridazinone | 254–256, ir, nmr, uv; HCl: 248–249 | 1597 |
| 5-Amino-6-hydrazino-3(2*H*)-pyridazinone | 247–249 or 250–251, ir, nmr, uv | 636, 654, 1597 |
| 5-Amino-2-δ-hydroxybutyl-3(2*H*)-pyridazinone | liq, anal, ir, nmr | 1700 |
| 5-Amino-1-hydroxy-3,6-dimethyl-4(1*H*)-pyridazinone | 203–204 | 794 |
| 4-Amino-5-α-hydroxyethyl-2,6-diphenyl-3(2*H*)-pyridazinone | 178–180, ir, nmr | 1590 |
| 4-Amino-5-α-hydroxyethyl-2-methyl-6-phenyl-3(2*H*)-pyridazinone | 241–242, ir, nmr | 1590 |
| 4-Amino-5-α-hydroxyethyl-6-phenyl-3(2*H*)-pyridazinone | 277, ir, nmr | 1590 |
| 4-Amino-5-hydroxymethyl-2,6-diphenyl-3(2*H*)-pyridazinone | 206–208, ir, nmr | 1590 |
| 4-Amino-5-hydroxymethyl-2-methyl-6-phenyl-3(2*H*)-pyridazinone | 155–157, ir, nmr | 1590 |
| 4-Amino-5-hydroxymethyl-6-phenyl-3(2*H*)-pyridazinone | 275, ir, nmr | 1590 |
| 5-Amino-4-hydroxy-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarbonitrile | — | 1444 |
| 4-Amino-6-imino-1-phenyl-1,6-dihydro-3,5-pyridazinedicarbonitrile | 220–223 or >300, ir, nmr | 55, 1210, 1365 |
| 5-Amino-4-iodo-2-phenyl-3(2*H*)-pyridazinone | — | *H* 124, 331 |
| 4-Amino-5-mercapto-3(2*H*)-pyridazinone | >300, ir, uv | 923 |
| 3-Amino-4-methoxy-1,2-dimethyl-3,6(1*H*,2*H*),pyridazinedione | 158, ir, nmr | 402 |
| 5-Amino-1-methoxy-3,6-dimethyl-4(1*H*)-pyridazinone | HCl: 149–150 | 794 |
| 1-Amino-3-methoxy-6-methylpyridazinium (+anion) | I: 124–125, nmr; mesitylenesulfonate: 122–124 | 782, 877 |
| 4-Amino-6-methoxy-2-methyl-3(2*H*)-pyridazinone | — | *H* 99, 563 |
| 5-Amino-6-methoxy-2-methyl-3(2*H*)-pyridazinone | — | *H* 91, 564 |
| 4-Amino-6-methoxy-3-pyridazinecarbonitrile | — | *H* 184, 446, 562 |
| 4-Amino-6-methoxy-3-pyridazinecarboxylic acid | — | *H* 562 |
| 5-Amino-6-methoxy-3-pyridazinecarboxylic acid | — | *H* 182, 435 |

*continued*

| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 6-Amino-3-methoxy-4(1*H*)-pyridazinethione | — | *H* 800 |
| 1-Amino-3-methoxypyridazinium ( + anion) | Cl: 150; I: 93–94; pic: 139; mesitylenesulfonate: 138–140 | 756, 877 |
| 4-Amino-6-methoxy-3(2*H*)-pyridazinone | 286–289, nmr | 1597; *H* 98, 560 |
| 5-Amino-6-methoxy-3(2*H*)-pyridazinone | 265–267 or 270, ir, nmr, uv | 654, 1597; *H* 89, 560 |
| 6-Amino-3-methoxy-4(1*H*)-pyridazinone | — | *H* 133 |
| 4-Aminomethyl-3-anilinopyridazine | liq, ms, nmr | 1079 |
| 4-Aminomethyl-3-chloro-5,6-dimethylpyridazine | — | *H* 294, 610 |
| 4-Amino-2-methyl-5,6-diphenyl-3(2*H*)-pyridazinone | — | 814 |
| 6-Amino-3-methyl-1,5-diphenyl-4(1*H*)-pyridazinone | — | 713 |
| 5-Aminomethyl-4-hydroxy-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarbonitrile | — | 1444 |
| 4-Aminomethyl-3-methoxy-5,6-dimethylpyridazine | — | *H* 610 |
| 4-Amino-6-methyl-3(2*H*)-pyridazinone | — | *H* 610 |
| 4-Amino-2-methyl-5-methylsulfonyl-3(2*H*)-pyridazinone | 174–176, ir, nmr | 533 |
| 4-Amino-2-methyl-5-nitro-3(2*H*)-pyridazinone | — | *H* 125, 602 |
| 5-Amino-1-methyl-6-oxo-1,6-dihydro-4-pyridazinesulfonamide | — | *H* 125, 563, 825 |
| 5-Amino-2-methyl-3-oxo-2,3-dihydro-4-pyridazinesulfonamide | — | *H* 125, 564, 825 |
| 5-Amino-1-methyl-6-oxo-3-phenyl-1,6-dihydro-4-pyridazinecarbaldehyde | 149–150 or 157–159, ir, nmr | 1501, 1590 |
| 5-Amino-1-methyl-6-oxo-3-phenyl-1,6-dihydro-4-pyridazinecarbonitrile | 246–248, ir, nmr | 1832 |
| 5-Amino-1-methyl-6-oxo-3-phenyl-1,6-dihydro-4-pyridazinecarboxamide | 234–237, ir, nmr | 1832 |
| 4-Amino-2-methyl-1-phenyl-3,6(1*H*,3*H*)-pyridazinedione | — | *H* 159, 595 |
| 5-Amino-2-methyl-6-phenyl-3,4(1*H*,2*H*)-pyridazinedione | — | *H* 169 |
| 3-Amino-6-methyl-1-phenyl-4(1*H*)-pyridazinone | — | *H* 136, 594 |
| 4-Amino-2-methyl-6-phenyl-3(2*H*)-pyridazinone | 129–130, ir, nmr | 1585 |
| 4-Amino-5-methyl-6-phenyl-3(2*H*)-pyridazinone | 355–360, ir, nmr | 1585 |
| 4-Amino-6-methyl-2-phenyl-3(2*H*)-pyridazinone | — | *H* 106, 563 |
| 4-Amino-6-methyl-5-phenyl-3(2*H*)-pyridazinone | crude: 345, ir, nmr | 1585 |
| 5-Amino-2-methyl-6-phenyl-3(2*H*)-pyridazinone | 274, ir, nmr | 133 |
| 6-Amino-1-methyl-3-phenyl-4(1*H*)-pyridazinone | 274, ir, nmr | 133 |
| 6-Amino-5-methyl-2-phenyl-3(2*H*)-pyridazinone | — | *H* 93 |
| 4-Amino-2-methyl-6-phenyl-5-vinyl-3(2*H*)-pyridazinone | 122–123, ir, nmr | 1832 |
| 4-(β-Amino-β-methylpropyl)-3,6-diphenylpyridazine | 178, ir, ms, nmr, uv | 512 |
| 3-Aminomethylpyridazine | — | *H* 608 |
| 4-Amino-6-methyl-3-pyridazinecarbaldehyde | solid, anal, ir, ms, nmr; $Me_2$acetal: nmr | 996 |
| 3-Amino-6-methyl-4-pyridazinecarboxamide | — | *H* 436, 555 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3-Amino-6-methyl-4-pyridazinecarboxylic acid | — | *H* 436, 555 |
| 4-Amino-1-methyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 564 |
| 4-Amino-2-methyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 152, 563 |
| 5-Amino-2-methyl-3,4(1*H*,2*H*)-pyridazinedione | — | *H* 169 |
| 3-Amino-6-methyl-4(1*H*)-pyridazinethione | complex | 463; *H* 556, 800 |
| 1-Amino-6-methylpyridazinium ( + anion) | I: 129–130, nmr; pic: 164–166 | 782 |
| 3-Amino-1-methyl-4(1*H*)-pyridazinone | 181–182, nmr | 1132 |
| 3-Amino-6-methyl-4(1*H*)-pyridazinone | — | *H* 133, 555 |
| 4-Amino-2-methyl-3(2*H*)-pyridazinone | 173–175, nmr, $pK_a$, uv | 675, 1132; *H* 563 |
| 4-Amino-6-methyl-3(2*H*)-pyridazinone | 257–259 or 262–268, ir, nmr | 760, 1585; *H* 97, 558 |
| 5-Amino-2-methyl-3(2*H*)-pyridazinone | 194–196, $pK_a$, uv | 675; *H* 564 |
| 6-Aminomethyl-3(2*H*)-pyridazinone | HCl: >290, ir, nmr, uv | 731 |
| 6-Amino-2-methyl-3(2*H*)-pyridazinone | 222–224 | 675; *H* 571 |
| 6-Amino-4-methyl-3(2*H*)-pyridazinone | — | *H* 98 |
| 6-Amino-5-methyl-3(2*H*)-pyridazinone | — | *H* 89 |
| 4-Amino-5-methylsulfonyl-2-phenyl-3(2*H*)-pyridazinone | 212–213, ir, nmr | 533 |
| 3-Amino-6-methylthio-4,5-pyridazinedicarbonitrile | — | 1799 |
| 4-Amino-5-nitro-2-phenyl-3(2*H*)-pyridazinone | — | *H* 125, 602 |
| 4-Amino-5-nitro-3(2*H*)-pyridazinone | — | *H* 602 |
| 5-Amino-6-oxo-1,3-diphenyl-1,6-dihydro-4-pyridazinecarbaldehydre | 153–155, ir, nmr | 1590 |
| 6-Amino-4-oxo-1,5-diphenyl-1,4-dihydro-3-pyridazinecarbonitrile | — | 713 |
| 5-Amino-6-oxo-3-phenyl-1,6-dihydro-4-pyridazinecarbaldehyde | 251–253, ir, nmr | 1590 |
| 2-ε-Aminopentyl-6-phenyl-3(2*H*)-pyridazinone | crude: liq, ir, nmr; HCl: 196–197 | 1289, 1337 |
| 3-Amino-5-phenyl-4-pyridazinecarbonitrile | 243–244, uv | 364 |
| 3-Amino-6-phenyl-4-pyridazinecarbonitrile | 190–191 | 1885 |
| 5-Amino-6-phenyl-4-pyridazinecarbonitrile | 220–221, ir, nmr | 1080 |
| 3-Amino-5-phenyl-4-pyridazinecarbothioamide | 224, uv | 364 |
| 3-Amino-5-phenyl-4-pyridazinecarboxamidrazone | 196–198 | 130 |
| 4-Amino-1-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 156 |
| 5-Amino-2-phenyl-3,4(1*H*,2*H*)-pyridazinedione | — | *H* 169 |
| 3-Amino-6-phenyl-4(1*H*)-pyridazinone | — | *H* 133, 555 |
| 4-Amino-5-phenyl-3(2*H*)-pyridazinone | 299–302, ir, nmr | 1585 |
| 4-Amino-6-phenyl-3(2*H*)-pyridazinone | 322–323 or 328–331, nmr | 1041, 1585 |
| 5-Amino-2-phenyl-3(2*H*)-pyridazinone | 205–208 | 1344; *H* 565 |
| 5-Amino-6-phenyl-3(2*H*)-pyridazinone | 280–284, ir, nmr | 133 |
| 6-Amino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 80, 571 |
| 6-Amino-3-phenyl-4(1*H*)-pyridazinone | 247–248, ir, nmr | 133 |
| 5-Amino-2-phenyl-4-thioxo-1,4-dihydro-3(2*H*)-pyridazinone | >300, ir, nmr, uv | 923 |
| 2-γ-Aminopropyl-6-chloro-3(2*H*)-pyridazinone | HCl: 245–248 | 1760 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 4-γ-Aminopropyl-3,6-diphenylpyridazine | liq, ir, ms, nmr | 512 |
| 2-γ-Aminopropyl-6-phenyl-3(2*H*)-pyridazinone | 53–55, ir, nmr; HCl: 250–251 | 1289, 1337 |
| 3-Amino-4-pyridazinecarbonitrile | 193–194, ir, nmr | 1079 |
| 4-Amino-3-pyridazinecarbonitrile | — | *H* 562 |
| 6-Amino-3-pyridazinecarbonitrile | — | *H* 446 |
| 3-Amino-4-pyridazinecarbothioamide | HCl: 248–249 | 1099 |
| 3-Amino-4-pyridazinecarboxamide | 244–245, nmr | 1099, 1681 |
| 4-Amino-3-pyridazinecarboxamide | — | *H* 434, 562 |
| 5-Amino-4-pyridazinecarboxamide | — | *H* 562 |
| 6-Amino-3-pyridazinecarboxamide | 264, ir, nmr | 1689; *H* 435, 553 |
| 6-Amino-4-pyridazinecarboxamide | 308–310, nmr | 1099 |
| 3-Amino-pyridazinecarboxamidrazone | 199–203, nmr | 1681 |
| 3-Amino-4-pyridazinecarboxylic acid | 261–263 | 338; *H* 435, 555 |
| 4-Amino-3-pyridazinecarboxylic acid | — | *H* 434, 562 |
| 5-Amino-4-pyridazinecarboxylic acid | 310–312 | 958; *H* 562 |
| 6-Amino-3-pyridazinecarboxylic acid | — | *H* 434 |
| 1-Amino-3,6(1*H*,2*H*)-pyridazinedione | PhCH=:– | 903 |
| 4-Amino-3,6(1*H*,2*H*)-pyridazinedione | 325 or 330, nmr | 654, 1597 |
| 5-Amino-3,4(1*H*,2*H*)-pyridazinedione | — | *H* 169, 561 |
| 4-Amino-3,6(1*H*,2*H*)-pyridazinedithione | 325, ir | 654; *H* 559 |
| 5-Amino-3,4(1*H*,2*H*)-pyridazinedithione | — | *H* 802 |
| 4-Amino-3(2*H*)-pyridazinethione | 218–219, ir, nmr | 923 |
| 5-Amino-4(1*H*)-pyridazinethione | 230–231, ir, nmr | 923 |
| 6-Amino-3(2*H*)-pyridazinethione | — | 1152; *H* 553, 796, 797 |
| 6-Amino-4(1*H*)-pyridazinethione | 198–200, nmr | 930 |
| 1-Aminopyridazinium ( + anion) | Cl: 153–154; I: 176–177; pic: 169–179; $NO_3$: ir, nmr | 756, 1380 |
| 4-Amino-3(2*H*)-pyridazinone | 230–233 or 232–234, ir, nmr | 1585, 1597; *H* 84, 560 |
| 5-Amino-3(2*H*)-pyridazinone | 293–296, nmr | 1585; *H* 85, 560 |
| 6-Amino-3(2*H*)-pyridazinone | — | *H* 68, 551 |
| 3-Amino-6-thioxo-1,6-dihydro-4-pyridazinecarbothioamide | 228 | 1099 |
| 5-Amino-4-thioxo-1,4-dihydro-3(2*H*)-pyridazinone | >300, ir, uv | 923 |
| 3-Anilino-4-benzoyl-6-phenylpyridazine | PhN=: 200–202, ir | 1367 |
| 3-Anilino-6-benzylaminopyridazine | — | *H* 601 |
| 3-Anilino-4-benzyl-6-methylpyridazine | 149 | 1275 |
| 3-Anilino-6-benzyloxypyridazine | — | *H* 575 |
| 3-Anilino-6-bromopyridazine | — | *H* 286, 575 |
| 3-Anilino-6-butylaminopyridazine | — | *H* 601 |
| 4-Anilino-5-chloro-1,2-dimethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 163, 338, 596 |
| 4-Anilino-6-chloro-3-hydrazinopyridazine | — | *H* 587 |
| 5-Anilino-6-chloro-4-pyridazinamine | 196–197, ir nmr | 887 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3-Anilino-6-chloropyridazine | 185–187, p$K_a$, nmr | 94, 1161, 1330; *H* 277, 575 |
| 5-Anilino-4-chloro-3(2*H*)-pyridazinone | — | *H* 116, 310, 587 |
| 4-Anilino-3,6-dichloropyridazine | — | *H* 288, 587 |
| 4-Anilino-3,6-diphenylpyridazine | 176–178, nmr | 189 |
| 3-Anilino-5,6-diphenyl-4-pyridazinecarbonitrile | 222, ir, nmr | 1398, 1726 |
| 5-Anilino-4-ethoxy-2-methyl-3(2*H*)-pyridazinone | liq, anal | 1269 |
| 4-Anilino-2-ethyl-6-methyl-3(2*H*)-pyridazinone | — | *H* 100, 588 |
| 5-Anilino-2-ethyl-6-methyl-3(2*H*)-pyridazinone | — | *H* 588 |
| 4-Anilino-2-ethyl-1-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 162 |
| 3-Anilino-6-hydrazinopyridazine | 204–207, nmr | 1161 |
| 3-Anilino-4-methoxy-6-methylpyridazine | — | *H* 575 |
| 3-Anilino-6-methoxypyridazine | 112–115 | 1330 |
| 4-Anilino-2-methyl-5-nitro-6-phenyl-3(2*H*)-pyridazinone | — | *H* 602 |
| 4-Anilino-2-methyl-1-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 159, 595 |
| 3-Anilino-6-methylpyridazine | — | *H* 575 |
| 3-Anilino-6-methyl-4(1*H*)-pyridazinone | — | *H* 575 |
| 5-Anilino-6-methylsulfonyl-4-pyridazinamine | 160–162, ir, nmr | 887 |
| 3-Anilino-6-methylsulfonylpyridazine | — | *H* 575, 823 |
| 3-Anilino-6-methylthiopyridazine | — | *H* 575, 803 |
| 4-Anilino-5-nitro-2-phenyl-3(2*H*)-pyridazinone | — | *H* 602 |
| 3-Anilino-4-nitropyridazine | 93–95, nmr, uv | 259 |
| 3-Anilino-6-phenoxypyridazine | — | *H* 179, 575 |
| 6-Anilino-3-pyridazinamine | crude: nmr; pic: >250 | 1161 |
| 3-Anilinopyridazine | — | *H* 575 |
| 3-Anilino-4-pyridazinecarbonitrile | 123–124, ir, ms, nmr | 517 |
| 3-Anilino-4-pyridazinecarboxamide | 182–183, ir, nmr | 1079 |
| 3-Anilino-4-pyridazinecarboxylic acid | >200, ir, ms, nmr | 517 |
| 4-Anilino-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 151 |
| 6-Anilino-3(2*H*)-pyridazinethione | — | *H* 575, 797 |
| 6-Anilino-3(2*H*)-pyridazinone | p$K_a$ | 94; *H* 68, 575 |
| 3-Anilino-6-styrylpyridazine | — | 946 |
| 4-Anilino-3,5,6-triphenylpyridazine | — | *H* 587 |
| 3-Azido-4-benzyl-6-methylpyridazine | 95 | 1275 |
| 3-Azido-4-benzyl-6-phenylpyridazine | 172 | 1251 |
| 5-Azido-4-bromo-2-β-hydroxyethyl-3(2*H*)-pyridazinone | — | *H* 119, 319, 654 |
| 5-Azido-4-bromo-2-methyl-3(2*H*)-pyridazinone | — | *H* 119, 319, 654 |
| 5-Azido-4-bromo-2-phenyl-3(2*H*)-pyridazinone | — | *H* 120, 320, 654 |
| 5-Azido-4-bromo-3(2*H*)-pyridazinone | — | *H* 116, 319, 654 |
| 3-Azido-5-*t*-butylthio-6-phenylpyridazine | 134–136, ir nmr | 133 |
| 4-Azidocarbonyl-5,6-diphenyl-3(2*H*)-pyridazinone | — | 1966 |
| 4-Azidocarbonyl-6-methyl-3-pyridazinecarbaldehyde | $Me_2$acetal: nmr | 996 |
| 5-Azido-4-chloro-2-cyclohexyl-3(2*H*)-pyridazinone | — | *H* 124 |
| 3-Azido-6-chloro-4,5-dimethylpyridazine | 77–78, nmr | 558 |
| 5-Azido-4-chloro-2-δ-hydroxybutyl-3(2*H*)-pyridazinone | liq, anal, ir, nmr | 1700 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 6-Azido-3-chloro-5-(2-methoxycarbonylprop-1-enyl)-1-methyl-4(1*H*)-pyridazinone | *E*: 114–115, ir, nmr; *Z* (crude): 92–93, ir, nmr | 967 |
| 4-Azido-6-chloro-3-methoxypyridazine | 35, ir, nmr | 1900 |
| 4-Azido-6-chloro-3-methoxypyridazine 1-oxide | 93–94, ir, uv | 905 |
| 3-azidop-6-chloro-4-methylaminopyridazine | 244–246, ir | 1228 |
| 3-Azido-6-chloro-4-methylpyridazine | — | 408 |
| 3-Azido-6-chloro-5-methylpyridazine | — | 408 |
| 3-Azido-5-chloro-6-phenylpyridazine | 126–127, ir, nmr | 133 |
| 4-Azido-6-chloro-3-phenylpyridazine | 115–117, ir, nmr | 133, 134 |
| 4-Azido-6-chloro-2-phenyl-3(2*H*)-pyridazinone | 117, ir, nmr | 401, 402 |
| 3-Azido-6-chloropyridazine | — | 408, 1976 |
| 3-Azido-6-chloro-4-pyridazinecarboxamide | 226, ir | 1228 |
| 3-Azido-6-chloropyridazine 1-oxide | 100–102 | 480; *H* 654 |
| 5-Azido-4-chloro-3(2*H*)-pyridazinone | 150, ir | 1680 |
| 4-Azido-3,6-diethoxypyridazine 1-oxide | 80–81, ir | 831 |
| 4-Azido-3,6-dimethoxypyridazine | 35 or 74–75, ir, nmr | 885, 1443, 1900; *H* 192, 654 |
| 4-Azido-3,6-dimethoxypyridazine 1-oxide | 89–91 or 93–94, ir, nmr | 831, 1443; *H* 654 |
| 3-Azido-4,5-dimethylpyridazine | 130–131, nmr | 558 |
| 4-Azido-3,6-dimethylpyridazine | 86–87, ir, nmr | 1443 |
| 4-Azido-1,2-dimethyl-3,6(1*H*,2*H*)-pyridazinedione | 113–115, ir, nmr | 402 |
| 4-Azido-3,6-dimethylpyridazine 1-oxide | 140 or 143–144, ir, nmr, uv | 831, 1443 |
| 3-Azido-6-hydrazinopyridazine | PhCH=: 315–317 | 1008 |
| 3-Azido-5-isopropylthio-6-phenylpyridazine | 132–134, ir, nmr | 133 |
| 4-Azido-3-methoxy-6-methylpyridazine | 20, ir, nmr | 885, 1443 |
| 4-Azido-3-methoxy-6-methylpyridazine 1-oxide | 103–105, ir, nmr | 1443 |
| 4-Azido-3-methoxypyridazine | 70–71, ir, nmr | 885, 1443 |
| 4-Azido-3-methoxypyridazine 1-oxide | 140–141, ir, nmr | 1443 |
| 6-Azido-5-methylamino-3(2*H*)-pyridazinethione | 230, ir, nmr | 1228 |
| 5-Azido-2-methyl-6-phenyl-3(2*H*)-pyridazinone | 90, ir, nmr | 133 |
| 6-Azido-1-methyl-3-phenyl-4(1*H*)-pyridazinone | 160, ir, nmr | 133 |
| 3-Azido-5-methylpyridazine | 132, nmr | 558 |
| 4-Azido-6-methylpyridazine | 15, ir, nmr | 1443 |
| 4-Azido-6-methylpyridazine 1-oxide | 139–140, ir, nmr | 1443 |
| 6-Azido-5-methyl-3(2*H*)-pyridazinethione | 145, ir, nmr, uv | 1228 |
| 5-Azido-2-methyl-3(2*H*)-pyridazinone | — | 848 |
| 3-Azido-6-methylthiopyridazine | 169–170 | 1008 |
| 3-Azido-4-phenethyl-6-phenylpyridazine | 249, ir | 1959 |
| 6-Azido-3-phenyl-4-pyridazinamine | 230–231, ir, nmr | 133 |
| 3-Azido-4-phenylpyridazine | 199, nmr | 1408 |
| 3-Azido-5-phenylpyridazine | 193–194, nmr | 1408 |
| 3-Azido-6-phenylpyridazine | 210–212, ir, nmr | 133 |
| 5-Azido-6-phenyl-3(2*H*)-pyridazinone | 160, ir, nmr | 133, 134 |
| 6-Azido-3-phenyl-4(1*H*)-pyridazinone | 205, ir, nmr | 133 |
| 3-Azido-6-phenylthiopyridazine | 128–129 | 1008 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 1-Benzyl-4,5-dichloro-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 157, 334 |
| 2-Benzyl-4,5-dichloro-3(2*H*)-pyridazinone | — | *H* 113, 309 |
| 6-Benzyl-1,5-dihydroxy-4(1*H*)-pyridazinone | — | *H* 134 |
| 5-Benzyl-2-β-dimethylaminoethyl-6-methyl-3(2*H*)-pyridazinone | HCl: 226, ir, nmr | 884 |
| 5-Benzyl-4,6-dimethyl-3-pyridazinamine | — | *H* 556 |
| 4-Benzyl-2,6-dimethyl-3(2*H*)-pyridazinethione | 127 | 112 |
| 4-Benzyl-2,6-dimethyl-3(2*H*)-pyridazinone | 93 | 1268 |
| 4-Benzyl-3,6-diphenylpyridazine | 111–113 | 319 |
| 4/5-Benzyl-2,6-dipropyl-3(2*H*)-pyridazinethione | 160, ir, nmr, uv | 968 |
| 4-Benzyl-5,6-diphenyl-3(2*H*)-pyridazinone | — | 868 |
| 5-Benzyl-2-ethoxycarbonylmethyl-6-methyl-3(2*H*)-pyridazinone | 90, ir, nmr | 1446 |
| 4-Benzyl-2-ethoxycarbonylmethyl-6-phenyl-3(2*H*)-pyridazinone | 96 | 1251 |
| 2-Benzyl-4-ethoxy-5-morpholino-3(2*H*)-pyridazinone | 190–200/0.1 or 225–228/3.5, nmr | 532, 1269 |
| 3-Benzyl-4-ethoxypyridazine | liq, anal, ms, nmr | 1357 |
| 3-Benzyl-6-ethoxypyridazine | liq, anal, ms, nmr | 1357 |
| 4-Benzyl-3-ethylthio-6-phenylpyridazine | — | 811 |
| 5-Benzyl-2-hydrazinocarbonylmethyl-6-methyl-3(2*H*)-pyridazinone | 167, ir, nmr | 1446 |
| 4-Benzyl-2-hydrazinocarbonylmethyl-6-phenyl-3(2*H*)-pyridazinone | 149 | 1251 |
| 5-α-Benzylhydrazino-4-chloro-2-methyl-3(2*H*)-pyridazinone | 118–119, ir, nmr; PhCH=: 167–168, ir, nmr | 1054 |
| 5-α-Benzylhydrazino-4-chloro-2-phenyl-3(2*H*)-pyridazinone | PhCH=: 146–149, ir, nmr | 1054 |
| 3-β-Benzylhydrazino-4,5-dimethylpyridazine | — | *H* 669 |
| 4-Benzyl-3-hydrazino-6-methylpyridazine | 132; PhCH=: 149 | 1275 |
| 4-Benzyl-6-hydrazino-3-methylpyridazine | 100, nmr | 1067 |
| 4-Benzyl-3-hydrazino-6-phenylpyridazine | 144; RCH=: complexes | 432, 1251 |
| 3-Benzyl-6-hydrazinopyridazine | — | *H* 660 |
| 6-Benzyl-5-hydroxy-4-phenyl-3(2*H*)-pyridazinone | >270 | 1296 |
| 3-Benzyl-5-hydroxy-4(1*H*)-pyridazinone | — | *H* 133 |
| 4-Benzylimino-5,6-dichloro-1-phenyl-1,4-dihydropyridazine | — | *H* 608 |
| 4-Benzyl-6-β-isobutyrylhydrazino-3-methylpyridazine | 175 | 1751 |
| 2-Benzyl-5-mercapto-4-thioxo-1,4-dihydro-3(2*H*)-pyridazinone | — | *H* 802 |
| 4-Benzyl-5-methoxymethylpyridazine | 51–52, ms, nmr, uv | 82 |
| 4-Benzyl-3-methoxy-6-phenylpyridazine | 107 | 1251 |
| 3-Benzyl-4-methoxypyridazine | liq, anal, ms, nmr | 1357 |
| 3-Benzyl-6-methoxypyridazine | liq, anal, ms, nmr | 1357 |
| 4-Benzyl-5-methoxypyridazine | 75–78, ms, nmr | 82 |
| 2-Benzyl-6-methoxy-3(2*H*)-pyridazinone | — | *H* 82 |
| 4-Benzyl-6-methyl-2,5-diphenyl-3(2*H*)-pyridazinone | — | *H* 130 |

*continued*

| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3-Benzoyl-6-chloro-1,5-dihydroxy-4(1*H*)-pyridazinone | — | *H* 134, 333 |
| 6-Benzoyl-3-chloro-1,5-dihydroxy-4(1*H*)-pyridazinone | — | *H* 134, 333 |
| 4-Benzoyl-3-chloro-5,6-dimethylpyridazine | 130–131 | 1514 |
| 4-Benzoyl-3-chloro-6-methylpyridazine | 97–99 | 1514 |
| 4-Benzoyl-3-chloro-6-phenylpyridazine | 122–125, ir | 1716 |
| 4-Benzoyl-6-chloro-3-piperidinopyridazine | — | 1479 |
| 3-Benzoyl-6-chloropyridazine | 82, ir, nmr | 1689 |
| 4-Benzoyl-2-β-cyanoethyl-6-phenyl-3(2*H*)-pyridazinone | — | 1749 |
| 3-Benzoyl-4,6-di-*t*-butyl-5-diisopropylaminopyridazine | 110, ir, nmr | 1123 |
| 4-Benzoyl-3,6-dichloropyridazine | 97, ir, nmr | 443, 1669 |
| 5-Benzoyl-*N*,*N*-diethyl-4-pyridazinecarboxamide | 114–116, ir, nmr | 46 |
| 6-Benzoyl-1,5-dihydroxy-4(1*H*)-pyridazinone | — | *H* 134 |
| 4-Benzoyl-5-β-dimethylaminovinylpyridazine | 106–107, nmr | 1095 |
| 4-Benzoyl-5,6-dimethyl-3(2*H*)-pyridazinone | 206–208 | 1514 |
| 3-Benzoyl-1,4-diphenyl-6-phenylimino-1,6-dihydropyridazine | 239, ir, ms | 17, 269 |
| 3-Benzoyl-4,6-diphenylpyridazine | 141–143, ir, nmr, uv | 404, 582 |
| 3-Benzoyl-3,6-diphenylpyridazine | 183–184, nmr | 1591 |
| 1-Benzoyl-5,6-diphenyl-3,4(1*H*,2*H*)-pyridazinedione | 144–146, ir, nmr | 1496 |
| 5-Benzoyl-2,6-diphenyl-3,4(1*H*,2*H*)-pyridazinedione | 240–242, ir, ms, nmr, xl st | 85 |
| 4-Benzoyl-5,6-diphenyl-3(2*H*)-pyridazinone | 218–219, ir, nmr | 373; *H* 128, 396 |
| 6-Benzoyl-2,5-diphenyl-3(2*H*)-pyridazinone | 181–182, ir, nmr | 17, 269, 1350, 1351 |
| 3-β-Benzoylhydrazino-5-benzyl-6-methylpyridazine | 194 | 1751 |
| 3-β-Benzoylhydrazino-6-chloropyridazine | — | *H* 280, 668 |
| 3-β-Benzoylhydrazino-4-methyl-6-phenylpyridazine | 210–214 or 277–278 | 1279, 1333 |
| 3-β-Benzoylhydrazino-6-phenylpyridazine | 233–234 | 1279 |
| 4-Benzoyl-2-β-hydroxyethyl-5,6-diphenyl-3(2*H*)-pyridazinone | 176–177 | 1272 |
| 4-Benzoyl-5-hydroxy-6-methyl-3(2*H*)-pyridazinone | 207, nmr | 1122 |
| 6-Benzoyl-5-hydroxy-3-oxo-2-phenyl-2,3-dihydro-4-pyridazinecarbonitrile | 172–173, ir | 1702 |
| 3-Benzoyl-5-hydroxy-4(1*H*)-pyridazinone | — | *H* 133 |
| 5-Benzoyl-6-imino-1,4-diphenyl-1,6-dihydro-3-pyridazinecarbonitrile | 260–264, ir | 49 |
| 6-Benzoyl-3-imino-5-methyl-2-phenyl-2,3-dihydro-4-pyridazinecarbonitrile | 186–188, ir, nmr | 1663 |
| 3-Benzoyl-6-methoxypyridazine | 115–118, ir, ms, nmr | 1698 |
| 4-Benzoyl-3-methyl-6-phenylpyridazine | — | 585 |
| 4-Benzoyl-2-methyl-6-phenyl-3(3*H*)-pyridazinone | 145–147, ir | 1716 |
| 4-Benzoyl-5-methyl-2-phenyl-3(2*H*)-pyridazinone | — | 1448 |
| 4-Benzoyl-3-methylpyridazine | 170/0.2, ir, nmr | 1066 |
| 4-Benzoyl-5-methylpyridazine | 99–101, ms, nmr | 1060 |
| 5-Benzoyl-6-methyl-3-pyridazinecarboxylic acid | 136–141, ir, nmr | 1066 |
| 4-Benzoyl-6-methyl-3(2*H*)-pyridazinone | 177–179 | 1514 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 4-Benzoyl-3-morpholino-6-phenylpyridazine | 122–124, ir | 1367 |
| 5-Benzoyl-6-oxo-1,4-diphenyl-1,6-dihydro-3-pyridazinecarboxylic acid | 223 | 391 |
| 2-Benzoyloxy-4-chloro-6-methoxy-3(2*H*)-pyridazinone | — | *H* 111, 322 |
| 2-Benzoyloxy-5-chloro-6-methoxy-3(2*H*)-pyridazinone | — | *H* 90 |
| 6-Benzoyloxy-4-chloro-3(2*H*)-pyridazinone | — | 1042; *H* 98, 322 |
| 6-Benzoyloxy-5-chloro-3(2*H*)-pyridazinone | — | 1042 |
| 2-Benzoyloxy-6-ethoxy-5-methyl-3(2*H*)-pyridazinone | — | *H* 90 |
| 2-Benzoyloxy-6-ethoxy-3(2*H*)-pyridazinone | — | *H* 76 |
| 2-Benzoyloxy-6-methoxy-4-methyl-3(2*H*)-pyridazinone | — | *H* 111 |
| 2-Benzoyloxy-6-methoxy-5-methyl-3(2*H*)-pyridazinone | — | *H* 90 |
| 2-Benzoyloxy-6-methoxy-3(2*H*)-pyridazinone | — | *H* 76 |
| 6-Benzoyloxy-3(2*H*)-pyridazinone | — | *H* 68 |
| 4-Benzoyl-3-phenoxy-6-phenylpyridazine | 115–117, ir | 1367 |
| 4-Benzoyl-6-phenyl-3-piperidinopyridazine | 103–105, ir | 1367 |
| 3-Benzoyl-6-phenylpyridazine | — | *H* 394 |
| 4-Benzoyl-3-phenylpyridazine | 118–119, ms, nmr | 12 |
| 4-Benzoyl-6-phenyl-3(2*H*)-pyridazinethione | 248–250, ir | 1367 |
| 3-Benzoyl-1-phenyl-4(1*H*)-pyridazinone | 128–130, ir, nmr | 974 |
| 4-Benzoyl-2-phenyl-3(2*H*)-pyridazinone | 173, ms, nmr | 1877 |
| 4-Benzoyl-6-phenyl-3(2*H*)-pyridazinone | 225–226, ir; $H_2NN{=}$: 242–243, ir | 1716 |
| 4-Benzoyl-3-phenyl-5-trimethylsilylpyridazine | 103, ms, nmr | 12 |
| 4-Benzoyl-5-propionylpyridazine | 92–94, nmr | 1095 |
| 5-Benzoyl-4-pyridazinamine | 119–121, ir, nmr | 46 |
| 6-Benzoyl-3-pyridazinamine | 122, ir, nmr | 1689 |
| 3-Benzoylpyridazine | 68–69 or 69–70, ir, ms, nmr; *E*-oxime: 168, nmr; *Z*-oxime: 182, nmr | 1108, 1357, 1586, 1798 |
| 4-Benzoylpyridazine | 104 to 108, ir, nmr; *E*-oxime: 150, nmr; *Z*-oxime: 154, nmr | 46, 72, 73, 81, 1066, 1098, 1586, 1798 |
| 5-Benzoyl-4-pyridazinecarbonyl azide | 97–98, ir | 46 |
| 5-Benzoyl-4-pyridazinecarboxamide | 205–215, ir, nmr | 46 |
| 5-Benzoyl-3-pyridazinecarboxylic acid | 89–90, ir, nmr | 1066 |
| 5-Benzoyl-4-pyridazinecarboxylic acid | 168–170 or 218, ir, nmr; $NH_4$: ir, nmr | 46, 81, 513, 702 |
| 1-Benzoyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 154, 2057 |
| 4-Benzoyl-3(2*H*)-pyridazinone | 215–217, nmr | 1408 |
| 4-Benzoylthio-6-chloro-2-phenyl-3(2*H*)-pyridazinone | — | *H* 820 |
| 3-Benzoylthio-6-chloropyridazine | — | *H* 283, 819 |
| 6-Benzoylthio-3(2*H*)-pyridazinethione | 214–215 | 95; *H* 820 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3-Benzylamino-6-benzyloxypyridazine | — | *H* 181, 576 |
| 4-Benzylamino-5-bromo-2-methyl-3(2*H*)-pyridazinone | 83–84, nmr | 1601 |
| 5-Benzylamino-4-bromo-2-methyl-3(2*H*)-pyridazinone | 133–134, nmr | 1601 |
| 5-Benzylamino-4-bromo-2-phenyl-3(2*H*)-pyridazinone | — | *H* 120, 319, 593 |
| 5-Benzylamino-4-chloro-2-β-diethylaminoethyl-3(2*H*)-pyridazinone | — | *H* 119, 312, 591 |
| 4-Benzylamino-5-chloro-3,6-diphenylpyridazine | 160–161 | 29 |
| 4-Benzylamino-5-chloro-2-methyl-6-nitro-3(2*H*)-pyridazinone | 123–125, nmr | 1601 |
| 5-Benzylamino-4-chloro-2-methyl-6-nitro-3(2*H*)-pyridazinone | 123–125, nmr | 1601 |
| 4-Benzylamino-5-chloro-2-methyl-3(2*H*)-pyridazinone | 97–98, ir, nmr | 867, 1601 |
| 5-Benzylamino-4-chloro-2-methyl-3(2*H*)-pyridazinone | 183–185, ir, nmr | 867, 1601 |
| 4-Benzylamino-5-chloro-2-phenyl-3(2*H*)-pyridazinone | 109–110 or 110–112, ir, nmr | 867, 1601 |
| 5-Benzylamino-4-chloro-2-phenyl-3(2*H*)-pyridazinone | 210–212 or 213–215, ir, nmr | 867, 1601 |
| 3-Benzylamino-6-chloropyridazine | 150–156 or 163–164 | 675, 1330; *H* 277, 576 |
| 5-Benzylamino-4-chloro-3(2*H*)-pyridazinone | — | *H* 116, 310, 583 |
| 5-Benzylamino-3,6-dichloro-4-pyridazinecarbonitrile | 126–127, uv | 820 |
| 3-Benzylamino-4,6-dinitropyridazine 1-oxide | 158–159 or 162–163, uv | 760, 905 |
| 4-Benzylamino-3,6-diphenylpyridazine | 137–138, ir, nmr | 29, 252; *H* 584 |
| 3-Benzylamino-6-ethoxypyridazine | — | *H* 177, 576 |
| 4-Benzylamino-5-mercapto-2-methyl-3(2*H*)-pyridazinone | 157–158, ir | 867 |
| 4-Benzylamino-5-mercapto-2-phenyl-3(2*H*)-pyridazinone | 176–177, ir | 867 |
| 3-Benzylamino-4-methoxy-6-methylpyridazine | — | *H* 576 |
| 3-Benzylamino-6-methoxy-5-nitropyridazine 2-oxide | HCl: 186–188, uv | 905 |
| 3-Benzylamino-6-methoxypyridazine | 99–100 | 1330; *H* 176, 576 |
| 3-Benzylamino-6-methyl-4-nitropyridazine 1-oxide | 217–218 | 760; *H* 604 |
| 3-Benzylamino-6-methylpyridazine | — | *H* 576 |
| 3-Benzylamino-6-methyl-4(1*H*)-pyridazinone | — | *H* 576 |
| 4-Benzylamino-2-methyl-3(2*H*)-pyridazinone | 86–87, ir, nmr | 867 |
| 6-Benzylamino-2-methyl-3(2*H*)-pyridazinone | — | *H* 70 |
| 4-Benzylamino-5-nitro-2-phenyl-3(2*H*)-pyridazinone | — | 601 |
| 3-Benzylamino-4-nitropyridazine | 157–160, nmr, uv | 259 |
| 3-Benzylamino-4-nitropyridazine 1-oxide | 200 | 760 |
| 3-Benzylamino-6-phenylpyridazine | 166 | 1248 |
| 3-Benzylamino-6-propoxypyridazine | — | *H* 178, 575 |
| 6-Benzylamino-3-pyridazinamine 2-oxide | 189–191, nmr | 1161 |
| 3-Benzylaminopyridazine | — | *H* 576 |
| 3-Benzylamino-4-pyridazinecarbonitrile | 132–133, ir, nmr | 1079 |
| 3-Benzylamino-4-pyridazinecarboxamide | 182–183, ir, nmr | 1079 |
| 6-Benzylamino-3-pyridazinecarboxamide | 192–194 | 1592, 1773 |
| 6-Benzylamino-3(2*H*)-pyridazinone | — | *H* 68, 576 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3-Benzylamino-6-styrylpyridazine | — | 946 |
| 2-Benzyl-4-benzylamino-5-chloro-3(2*H*)-pyridazinone | 124–126, ir | 867 |
| 2-Benzyl-5-benzylamino-4-chloro-3(2*H*)-pyridazinone | 130–131, ir | 867 |
| 2-Benzyl-4-benzylamino-5-mercapto-3(2*H*)-pyridazinone | 150–151, ir | 867 |
| 2-Benzyl-5-α-benzylhydrazino-4-chloro-3(2*H*)-pyridazinone | $H_2C$=: 114–115, ir, nmr; MeCH=: 116–117, ir, nmr; PhCH=: 100–102, ir, nmr | 1054 |
| 2-Benzyl-6-benzyloxy-4-chloro-5-nitro-3(2*H*)-pyridazinone | — | *H* 131, 648 |
| 2-Benzyl-4-benzyloxy-6-chloro-3(2*H*)-pyridazinone | 128–129, nmr, uv | 665 |
| 2-Benzyl–6-benzyloxy-4,5-dimethyl-3(2*H*)-pyridazinone | — | *H* 131 |
| 2-Benzyl-6-benzyloxy-3(2*H*)-pyridazinone | 62–64, ir, nmr | 1498; *H* 82 |
| 2-Benzyl-4-benzylthio-5-chloro-3(2*H*)-pyridazinone | — | *H* 811 |
| 2-Benzyl-5-benzylthio-4-chloro-3(2*H*)-pyridazinone | — | *H* 812 |
| 2-Benzyl-4-benzylthio-5-hydroxy-3(2*H*)-pyridazinone | — | *H* 811 |
| 4-Benzyl-3-benzylthio-6-phenylpyridazine | — | 811 |
| 2-Benzyl-5-benzylthio-3,4(1*H*,2*H*)-pyridazinedione | — | *H* 812 |
| 2-Benzyl-4,5-bisbenzylthio-3(2*H*)-pyridazinone | — | *H* 815 |
| 2-Benzyl-4-bromo-5-α-methylhydrazino-3(2*H*)-pyridazinone | 103, ir, nmr | 1540 |
| 5-Benzyl-2-carbamoylmethyl-6-methyl-3(2*H*)-pyridazinone | 160, ir, nmr | 1446 |
| 4-Benzyl-2-carbamoylmethyl-6-phenyl-3(2*H*)-pyridazinone | 196 | 1251 |
| 2-Benzyl-4-chloro-5-hydrazino-3(2*H*)-pyridazinone | 159–161, ir, nmr | 1054 |
| 2-Benzyl-4-chloro-5-hydroxy-3(2*H*)-pyridazinone | — | 1044 |
| 3-Benzyl-6-chloro-5-hydroxy-4(1*H*)-pyridazinone | — | *H* 132, 294 |
| 2-Benzyl-4-chloro-5-mercapto-3(2*H*)-pyridazinone | — | *H* 316, 802 |
| 2-Benzyl-4-chloro-5-α-methylhydrazino-3(2*H*)-pyridazinone | 130, ir, nmr; PhCH=: 160–162, ir, nmr | 873, 1054 |
| 3-Benzyl-6-chloro-5-methylpyridazine | 300 (? HCl) | 1319 |
| 4-Benzyl-3-chloro-6-methylpyridazine | 75 | 1268 |
| 4-Benzyl-6-chloro-3-methylpyridazine | 124, nmr | 1067 |
| 2-Benzyl-4-chloro-5-morpholino-3(2*H*)-pyridazinone | 98–101, ir, nmr | 532 |
| 4-Benzyl-3-chloro-6-phenylpyridazine | 114–116, nmr | 238, 1251, 1319 |
| 3-Benzyl-6-chloropyridazine | — | *H* 276 |
| 2-Benzyl-6-chloro-3(2*H*)-pyridazinone | — | *H* 82 |
| 2-Benzyl-5-chloro-4-thioxo-1,4-dihydro-3(2*H*)-pyridazinone | — | *H* 329, 801 |
| 4-Benzyl-2-β-cyanoethyl-6-methyl-3(2*H*)-pyridazinone | 118, ir | 112 |
| 2-Benzyl-4,5-dibromo-3(2*H*)-pyridazinone | 102–103, ir, nmr | 1540 |
| 1-Benzyl-4,5-dichloro-2-methyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 166, 335 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 1-Benzyl-4,5-dichloro-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 157, 334 |
| 2-Benzyl-4,5-dichloro-3(2*H*)-pyridazinone | — | *H* 113, 309 |
| 6-Benzyl-1,5-dihydroxy-4(1*H*)-pyridazinone | — | *H* 134 |
| 5-Benzyl-2-β-dimethylaminoethyl-6-methyl-3(2*H*)-pyridazinone | HCl: 226, ir, nmr | 884 |
| 5-Benzyl-4,6-dimethyl-3-pyridazinamine | — | *H* 556 |
| 4-Benzyl-2,6-dimethyl-3(2*H*)-pyridazinethione | 127 | 112 |
| 4-Benzyl-2,6-dimethyl-3(2*H*)-pyridazinone | 93 | 1268 |
| 4-Benzyl-3,6-diphenylpyridazine | 111–113 | 319 |
| 4/5-Benzyl-2,6-dipropyl-3(2*H*)-pyridazinethione | 160, ir, nmr, uv | 968 |
| 4-Benzyl-5,6-diphenyl-3(2*H*)-pyridazinone | — | 868 |
| 5-Benzyl-2-ethoxycarbonylmethyl-6-methyl-3(2*H*)-pyridazinone | 90, ir, nmr | 1446 |
| 4-Benzyl-2-ethoxycarbonylmethyl-6-phenyl-3(2*H*)-pyridazinone | 96 | 1251 |
| 2-Benzyl-4-ethoxy-5-morpholino-3(2*H*)-pyridazinone | 190–200/0.1 or 225–228/3.5, nmr | 532, 1269 |
| 3-Benzyl-4-ethoxypyridazine | liq, anal, ms, nmr | 1357 |
| 3-Benzyl-6-ethoxypyridazine | liq, anal, ms, nmr | 1357 |
| 4-Benzyl-3-ethylthio-6-phenylpyridazine | — | 811 |
| 5-Benzyl-2-hydrazinocarbonylmethyl-6-methyl-3(2*H*)-pyridazinone | 167, ir, nmr | 1446 |
| 4-Benzyl-2-hydrazinocarbonylmethyl-6-phenyl-3(2*H*)-pyridazinone | 149 | 1251 |
| 5-α-Benzylhydrazino-4-chloro-2-methyl-3(2*H*)-pyridazinone | 118–119, ir, nmr; PhCH=: 167–168, ir, nmr | 1054 |
| 5-α-Benzylhydrazino-4-chloro-2-phenyl-3(2*H*)-pyridazinone | PhCH=: 146–149, ir, nmr | 1054 |
| 3-β-Benzylhydrazino-4,5-dimethylpyridazine | — | *H* 669 |
| 4-Benzyl-3-hydrazino-6-methylpyridazine | 132; PhCH=: 149 | 1275 |
| 4-Benzyl-6-hydrazino-3-methylpyridazine | 100, nmr | 1067 |
| 4-Benzyl-3-hydrazino-6-phenylpyridazine | 144; RCH=: complexes | 432, 1251 |
| 3-Benzyl-6-hydrazinopyridazine | — | *H* 660 |
| 6-Benzyl-5-hydroxy-4-phenyl-3(2*H*)-pyridazinone | >270 | 1296 |
| 3-Benzyl-5-hydroxy-4(1*H*)-pyridazinone | — | *H* 133 |
| 4-Benzylimino-5,6-dichloro-1-phenyl-1,4-dihydropyridazine | — | *H* 608 |
| 4-Benzyl-6-β-isobutyrylhydrazino-3-methylpyridazine | 175 | 1751 |
| 2-Benzyl-5-mercapto-4-thioxo-1,4-dihydro-3(2*H*)-pyridazinone | — | *H* 802 |
| 4-Benzyl-5-methoxymethylpyridazine | 51–52, ms, nmr, uv | 82 |
| 4-Benzyl-3-methoxy-6-phenylpyridazine | 107 | 1251 |
| 3-Benzyl-4-methoxypyridazine | liq, anal, ms, nmr | 1357 |
| 3-Benzyl-6-methoxypyridazine | liq, anal, ms, nmr | 1357 |
| 4-Benzyl-5-methoxypyridazine | 75–78, ms, nmr | 82 |
| 2-Benzyl-6-methoxy-3(2*H*)-pyridazinone | — | *H* 82 |
| 4-Benzyl-6-methyl-2,5-diphenyl-3(2*H*)-pyridazinone | — | *H* 130 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 2-Benzyl-5-α-methylhydrazino-3(2*H*)-pyridazinone | 146–147, ir, nmr | 873 |
| 4-Benzyl-6-methyl-3-methylthiopyridazine | 89 | 112 |
| 5-Benzyl-6-methyl-2-β-morpholinoethyl-3(2*H*)-pyridazinone | HCl: 200, ir, nmr | 884 |
| 5-Benzyl-6-methyl-2-morpholinomethyl-3(2*H*)-pyridazinone | 80, ir, nmr | 884 |
| 5-Benzyl-6-methyl-2-phenethyl-3(2*H*)-pyridazinone | 86, ir, nmr | 884 |
| 4-Benzyl-2-methyl-6-phenyl-3(2*H*)-pyridazinone | 100 or 115 | 1251, 1253 |
| 2-Benzyl-6-methyl-3(2*H*)-pyridazinethione | 74–75, nmr | 538 |
| 4-Benzyl-6-methyl-3(2*H*)-pyridazinethione | 148 | 112 |
| 2-Benzyl-6-methyl-3(2*H*)-pyridazinone | — | *H* 82 |
| 4-Benzyl-6-methyl-3(2*H*)-pyridazinone | 165–166, uv | 634, 1190, 1268, 1275 |
| 5-Benzyl-6-methyl-3(2*H*)-pyridazinone | 172 or 176, ms, nmr, tautomerism | 884, 1667, 1827 |
| 6-Benzyl-4-methyl-3(2*H*)-pyridazinone | 160 | 1319 |
| 4-Benzyl-3-methyl-6-ureidopyridazine | 240, ir, nmr | 1751 |
| 4-Benzyl-3-morpholino-6-phenylpyridazine | 121, ir, uv; HCl: 146–149, ir, uv | 651 |
| 4-Benzyl-5-morpholinopyridazine | 92–93, ms, nmr | 82 |
| 4-Benzyl-6-oxo-1-phenyl-1,6-dihydro-3,5-pyridazinedicarbonitrile | 212, ir, ms, nmr | 1881 |
| 3-Benzyloxy-6-chloro-4-dimethylaminopyridazine | — | *H* 182, 293, 584 |
| 5-Benzyloxy-4-chloro-2-phenyl-3(2*H*)-pyridazinone | — | *H* 123 |
| 3-Benzyloxy-6-chloropyridazine | — | *H* 181, 281 |
| 6-Benzyloxy-4-dimethylamino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 589 |
| 3-Benzyloxy-6-dimethylaminopyridazine | 65–67, $pK_a$ | 94 |
| 1-Benzyloxy-3,6-dimethyl-5-nitro-4(1*H*)-pyridazinone | 125–126, uv | 794 |
| 4-Benzyloxy-3,6-dimethylpyridazine 1,2-dioxide | 203, ir, nmr | 796 |
| 2-Benzyloxy-6-ethoxy-5-methyl-3(2*H*)-pyridazinone | — | *H* 90 |
| 2-Benzyloxy-6-ethoxy-3(2*H*)-pyridazinone | — | *H* 76 |
| 2-Benzyloxy-6-methoxy-5-methyl-3(2*H*)-pyridazinone | — | *H* 90 |
| 2-Benzyloxy-6-methoxy-3(2*H*)-pyridazinone | — | *H* 76 |
| 4-Benzyloxy-2-methyl-1-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 160 |
| 4-Benzyloxy-6-methyl-3-pyridazinamine | — | *H* 187, 554 |
| 3-Benzyloxy-6-methylpyridazine | — | *H* 181 |
| 3-Benzyloxy-6-methylpyridazine 1-oxide | — | *H* 195 |
| 2-Benzyloxy-6-methyl-3(2*H*)-pyridazinone | — | *H* 76 |
| 6-Benzyloxy-2-methyl-3(2*H*)-pyridazinone | — | *H* 70 |
| 6-Benzyloxy-3-pyridazinamine | — | *H* 181, 553 |
| 3-Benzyloxypyridazine | — | *H* 174 |
| 4-Benzyloxypyridazine | — | *H* 187 |
| 6-Benzyloxy-3-pyridazinecarbonitrile | — | *H* 181, 446 |
| 3-Benzyloxypyridazine 1-oxide | — | *H* 194 |
| 4-Benzyloxypyridazine 1-oxide | — | *H* 194 |
| 6-Benzyloxy-3(2*H*)-pyridazinone | 148–153, ir, nmr | 1498; *H* 68, 76 |
| 4-Benzyl-6-phenyl-3-pyridazinamine | 210, nmr | 1381 |

*continued*

| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3-Benzyl-6-phenyl-4-pyridazinecarboxylic acid | — | *H* 436 |
| 2-Benzyl-6-phenyl-3(2*H*)-pyridazinethione | 113–115 or 120, nmr | 413, 1674 |
| 4/5-Benzyl-6-phenyl-3(2*H*)-pyridazinethione | 195, ir, nmr, uv | 811, 968 |
| 2-Benzyl-6-phenyl-3(2*H*)-pyridazinone | 90–91, nmr | 118 |
| 3-Benzyl-6-phenyl-4(1*H*)-pyridazinone | — | 1439, 1468 |
| 4-Benzyl-6-phenyl-3(2*H*)-pyridazinone | 175 to 190, ir, nmr | 238, 1243, 1253, 1319 |
| 6-Benzyl-4-phenyl-3(2*H*)-pyridazinone | — | *H* 98 |
| 6-Benzyl-5-phenyl-3(2*H*)-pyridazinone | — | *H* 89 |
| 3-Benzylpyridazine | 40 to 67, 170/0.5, ms, nmr | 123, 1108, 1357, 1547, 1666 |
| 4-Benzylpyridazine | 125/0.001, ms, nmr; HCl: 129 | 72, 73, 1098, 1666 |
| 1-Benzyl-3,6(1*H*,2*H*)-pyridazinedione | 198–201, ir, nmr | 1496; *H* 157 |
| 3-Benzylpyridazine 2-oxide | 83–84 | 123 |
| 4-Benzyl-6-styryl-3(2*H*)-pyridazinone | 139 | 1282 |
| 4-Benzylsulfonyl-5-chloro-2-methyl-3(2*H*)-pyridazinone | 99–101, ms, nmr | 1903 |
| 3-Benzylsulfonyl-6-chloropyridazine | — | *H* 823 |
| 6-Benzylsulfonyl-3(2*H*)-pyridazinone | — | *H* 824 |
| 5-Benzylthio-4-bromo-3(2*H*)-pyridazinone | — | *H* 812 |
| 4-Benzylthio-5-chloro-2-methyl-3(2*H*)-pyridazinone | liq, anal, ir, nmr | 1903; *H* 811 |
| 5-Benzylthio-4-chloro-2-methyl-3(2*H*)-pyridazinone | — | *H* 119, 812 |
| 4-Benzylthio-5-chloro-6-morpholinopyridazine | — | *H* 579 |
| 4-Benzylthio-5-chloro-2-phenyl-3(2*H*)-pyridazinone | — | *H* 126, 328 |
| 4-Benzylthio-6-chloro-2-phenyl-3(2*H*)-pyridazinone | — | *H* 811 |
| 5-Benzylthio-4-chloro-2-phenyl-3(2*H*)-pyridazinone | — | *H* 316, 812 |
| 3-Benzylthio-6-chloropyridazine | — | *H* 284 |
| 4-Benzylthio-6-chloropyridazine 2-oxide | 180–184 | 679 |
| 5-Benzylthio-4-chloro-3(2*H*)-pyridazinone | — | *H* 812 |
| 4-Benzylthio-5-ethoxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 811 |
| 3-Benzylthio-6-ethoxypyridazine | — | *H* 807 |
| 4-Benzylthio-5-hydroxy-2-methyl-3(2*H*)-pyridazinone | — | *H* 811 |
| 4-Benzylthio-5-methoxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 811 |
| 3-Benzylthio-6-methoxypyridazine | — | *H* 807 |
| 3-Benzylthio-6-methylpyridazine | — | *H* 807 |
| 4-Benzylthio-5-morpholino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 592, 810 |
| 4-Benzylthio-5-morpholino-3(2*H*)-pyridazinone | — | *H* 586, 810, 811 |
| 5-Benzylthio-4-morpholino-3(2*H*)-pyridazinone | — | *H* 812 |
| 4-Benzylthio-2-phenyl-5-piperidino-3(2*H*)-pyridazinone | — | *H* 592, 810 |
| 3-Benzylthio-6-phenylpyridazine | — | 811 |
| 3-Benzylthio-6-propoxypyridazine | — | *H* 807 |
| 6-Benzylthio-3-pyridazinamine | 109–110, nmr | 1152; *H* 553, 807 |
| 6-Benzylthio-3(2*H*)-pyridazinethione | — | *H* 809 |
| 6-Benzylthio-3(2*H*)-pyridazinone | — | *H* 808 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 5-Benzylthio-4-thioxo-1,4-dihydro-3(2H)-pyridazinone | — | *H* 812 |
| 3,6-Bisacetoxymethylpyridazine | 62–63, ir, nmr | 529 |
| 3,6-Bisacetylthiopyridazine | — | *H* 820 |
| 3,6-Bisbenzoylthiopyridazine | 196 | 95; *H* 820 |
| 3,6-Bisbenzylaminopyridazine | 118–120, nmr; pic: 218–220 | 16, 675 |
| 4,5-Bisbenzylthio-3-chloropyridazine | — | *H* 290, 814 |
| 4,5-Bisbenzylthio-2-methyl-3(2*H*)-pyridazinone | — | *H* 815 |
| 4,5-Bisbenzylthio-3-morpholinopyridazine | — | *H* 579 |
| 4,5-Bisbenzylthio-2-phenyl-3(2*H*)-pyridazinone | — | *H* 815 |
| 3,6-Bisbenzylthiopyridazine | — | *H* 813 |
| 4,5-Bisbenzylthio-3(2*H*)-pyridazinone | — | *H* 815 |
| 3,4-Bisbutylthio-5-chloropyridazine | 87–89, ms, nmr | 1219 |
| 3,5-Bis-*t*-butylthio-6-phenylpyridazine | 104–105, ir, nmr | 1087 |
| 3,6-Bis-*t*-butylthiopyridazine | 104–107, ms, nmr | 1219 |
| 3,6-Bischloromethyl-4(1*H*)-pyridazinone | 206 | 446; *H* 133 |
| 3,4-Bisdiisopropylamino-6-methylpyridazine | 1-$EtClO_4$: 108–109, ir, nmr | 31 |
| 3,4-Bisdiisopropylamino-6-pivaloylpyridazine | 65, ir, nmr | 1070 |
| 3,4-Bisdiisopropylaminopyridazine | 1-$MeClO_4$: 133–134, ir, nmr | 31 |
| 4,5-Bisdimethylamino-6-ethoxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 130, 602 |
| 4,5-Bisdimethylamino-6-isopropoxy-2-phenyl 3(2*H*)-pyridazinone | — | *H* 602 |
| 3,6-Bisdimethylamino-4-methylpyridazine | — | *H* 600 |
| 4,5-Bisdimethylamino-2-phenyl-6-propoxy-3(2*H*)-pyridazinone | — | *H* 130 |
| 4,5-Bisdimethylamino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 602 |
| 4,6-Bisdimethylamino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 108, 602 |
| 5,6-Bisdimethylamino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 93, 602 |
| 3,5-Bisdimethylaminopyridazine | HCl: 259–260, nmr | 637 |
| 3,6-Bisdimethylaminopyridazine | p$K_a$; HCl: 253 | 94, 1796; *H* 600 |
| 3,6-Bisdimethylamino-4(1*H*)-pyridazinone | 247–250, nmr | 640 |
| 4,5-Bisethylthio-2-phenyl-3(2*H*)-pyridazinone | — | *H* 815 |
| 4,5-Bisethylthio-3(2*H*)-pyridazinone | — | *H* 815 |
| 3,6-Bishydroxymethylpyridazine | ms, nmr, uv, xl st | 173, 1164, 1939 |
| 3,6-Bishydroxymethyl-4(1*H*)-pyridazinone | 221, nmr | 778; *H* 133, 397 |
| 3,6-Bisisopropylaminopyridazine | — | *H* 600 |
| 3,6-Bismethoxymethylpyridazine | 28–32, nmr | 529 |
| 3,4-Bismethylaminopyridazine | pic: 207–208 | 1138 |
| 3,6-Bismethylsulfinylpyridazine | 203–204, ir, ms, nmr | 304 |
| 3,6-Bismethylsulfinylpyridazine 1-oxide | 201–203, nmr | 304 |
| 3,5-Bismethylsulfonyl-4-methylthiopyridazine (or isomer) | 173–175, p$K_a$, uv | 689 |
| 3,6-Bismethylsulfonylpyridazine | 278–279, nmr | 304 |
| 3,6-Bismethylsulfonylpyridazine 1-oxide | 262, nmr | 304 |
| 3,5-Bismethylsulfonyl-4(1*H*)-pyridazinone | 312–314 | 689 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3,6-Bismethylthio-4-phenylpyridazine | 68–69, ir, ms, nmr | 353 |
| 4,5-Bismethylthio-2-phenyl-3(2*H*)-pyridazinone | nmr | *H* 815, 1213 |
| 3,6-Bismethylthiopyridazine | 125 to 131, ir, ms, nmr, p$K_a$, uv; Etl: 136; MeI: 163–165, uv | 95, 111, 353, 636; *H* 796, 813, 818 |
| 3,6-Bismethylthiopyridazine 1-oxide | 163, nmr | 304 |
| 4,6-Bismethylthio-3(2*H*)-pyridazinone | 229–230 | 1129 |
| 3,5-Bismorpholinomethylpyridazine | 60–62, 210–214/2, nmr; oxalate: 136–137 | 769 |
| 3,6-Bisphenylethynylpyridazine | 183, ir, nmr | 834 |
| 3,6-Bisphenylthiopyridazine | — | *H* 813 |
| 4,5-Bisphenylthio-3(2*H*)-pyridazinone | — | *H* 815 |
| 3,6-Bispiperidinocarbonyl-4-pyridazinecarbonitrile | 135–136, nmr | 215 |
| 3,6-Bispropoxymethyl-4(1*H*)-pyridazinone | — | *H* 133 |
| 3,6-Bistrimethylsiloxypyridazine | 60–62, 88–90/2, nmr, uv | 1168, 1172 |
| 4-Bromo-6-butanesulfonyloxy-3(2*H*)-pyridazinone | — | *H* 330 |
| 3-Bromo-6-butoxypyridazine | — | *H* 178, 286 |
| 4-Bromo-5-butylamino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 320, 592 |
| 2-δ-Bromobutyl-4,5-dichloro-3(2*H*)-pyridazinone | 72–74, ir, nmr | 1950, 2037 |
| 4-Bromo-2-butyl-5-hydroxy-3(2*H*)-pyridazinone | 177–178, ir, nmr | 1936 |
| 4-Bromo-2-butyl-5-methoxy-3(2*H*)-pyridazinone | 52–53, ir, nmr | 1936 |
| 2-δ-Bromobutyl-6-phenyl-3(2*H*)-pyridazinone | 42–43, 210–225/2, ir, nmr | 118, 1289, 1337 |
| 2-δ-Bromobutyl-3(2*H*)-pyridazinimine | — | *H* 551 |
| 4-Bromo-2-β-carbamoylethyl-5-chloro-3(2*H*)-pyridazinone | — | 587 |
| 4-Bromo-2-β-carboxyethyl-5-chloro-3(2*H*)-pyridazinone | — | 587 |
| 5-Bromo-4-chloro-2-chloromethyl-3(2*H*)-pyridazinone | — | *H* 112, 119, 312 |
| 4-Bromo-5-chloro-1,2-dimethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 163, 337 |
| 5-Bromo-6-chloro-3-ethoxy-4-pyridazinamine | — | *H* 559 |
| 3-Bromo-4-chloro-5-ethylimino-2-phenyl-2,5-dihydropyridazine | — | *H* 608 |
| 5-Bromo-4-chloro-2-hydroxymethyl-3(2*H*)-pyridazinone | — | *H* 112 |
| 3-Bromo-6-chloro-4-methylpyridazine | — | *H* 292 |
| 3-Bromo-6-chloro-5-methylpyridazine | — | *H* 290 |
| 4-Bromo-5-chloro-2-methyl-3(2*H*)-pyridazinone | 104–105, ir, nmr | 542 |
| 5-Bromo-4-chloro-2-methyl-3(2*H*)-pyridazinone | — | *H* 112 |
| 6-Bromo-2-chloromethyl-3(2*H*)-pyridazinone | 101–102 | 441 |
| 4-Bromo-5-chloro-2-phenyl-3(2*H*)-pyridazinone | 147–148 or 152, ir, nmr | 542, 956; *H* 320 |
| 5-Bromo-4-chloro-2-phenyl-3(2*H*)-pyridazinone | — | *H* 112, 121, 312 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 4-Bromo-2-γ-chloropropyl-6-phenyl-3(2*H*)-pyridazinone | — | *H* 331 |
| 3-Bromo-4-chloropyridazine | crude: unstable | 447 |
| 3-Bromo-6-chloropyridazine | — | *H* 276 |
| 4-Bromo-5-chloro-3(2*H*)-pyridazinone | 230, ir, nmr | 542; *H* 319 |
| 5-Bromo-4-chloro-3(2*H*)-pyridazinone | — | *H* 112, 310 |
| 4-Bromo-2-cyclohexyl-5-methoxy-3(2*H*)-pyridazinone | — | *H* 121, 319 |
| 5-Bromo-3,6-dichloro-4-pyridazinamine | — | *H* 559 |
| 4-Bromo-3,5-dichloropyridazine | — | *H* 289 |
| 4-Bromo-3,6-dichloropyridazine | 80, nmr | 654, *H* 287 |
| 2-Bromo-4,5-dichloro-3(2*H*)-pyridazinone | — | 916; *H* 112 |
| 4-Bromo-5-diethylamino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 120, 319, 593 |
| 4-Bromo-5-diethylamino-3(2*H*)-pyridazinone | — | *H* 319 |
| 6-Bromo-2-(diethylcarbamoyl)methyl-3(2*H*)-pyridazinone | — | *H* 79, 307 |
| 3-Bromo-6-dimethylamino-4-methylpyridazine | — | *H* 295, 577 |
| 4-Bromo-5-dimethylamino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 593 |
| 4-Bromo-5-dimethylamino-6-phenyl-3(2*H*)-pyridazinone | — | *H* 120, 319 |
| 6-Bromo-5-dimethylamino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 92, 330, 593 |
| 4-Bromo-2-γ-dimethylaminopropyl-6-phenyl-3(2*H*)-pyridazinone | — | *H* 331 |
| 3-Bromo-6-dimethylaminopyridazine | — | *H* 286 302, 303 |
| 4-(β-Bromo-α,α-dimethylethyl)-3,6-dichloropyridazine | 86–88, ir, nmr | 354 |
| 4-Bromo-1,2-dimethyl-3,6(1*H*,2*H*)-pyridazinedione | 151–152 | 1129 |
| 4-Bromo-3,6-dimethylpyridazine 1,2-dioxide | 223, ir, nmr | 796 |
| 5-Bromo-3,6-dimethyl-4(1*H*)-pyridazinone 2-oxide | 200–201 | 791 |
| 5-Bromo-2,6-diphenylpyridazinium-4-olate | 227, ms, nmr, xl st | 273 |
| 4-Bromo-5-ethoxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 120, 319 |
| 3-Bromo-6-ethoxypyridazine | — | *H* 177, 286 |
| 2-β-Bromoethyl-4,5-dichloro-3(2*H*)-pyridazinone | 76–77, ir, nmr | 1950, 2037 |
| 4-Bromo-2-ethyl-5-hydroxy-3(2*H*)-pyridazinone | 224–225, ir, nmr | 1936 |
| 4-Bromo-2-ethyl-5-methoxy-3(2*H*)-pyridazinone | 127–128, ir, nmr | 1936 |
| 4-Bromo-1-ethyl-2-phenyl-3,6-(1*H*,2*H*)-pyridazinedione | — | *H* 162, 339 |
| 2-β-Bromoethyl-6-phenyl-3(2*H*)-pyridazinone | 188–190/0.6, ir, nmr | 118, 1337 |
| 2-ζ-Bromohexyl-4,5-dichloro-3(2*H*)-pyridazinone | liq, ir, nmr | 1950, 2037 |
| 2-ζ-Bromohexyl-6-phenyl-3(2*H*)-pyridazinone | — | 1337 |
| 3-Bromo-6-hydrazino-4-methylpyridazine | — | *H* 295 |
| 3-Bromo-6-hydrazino-5-methylpyridazine | — | *H* 295, 660 |
| 4-Bromo-5-hydrazino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 120, 319–661 |
| 4-Bromo-5-hydrazino-3(2*H*)-pyridazinone | — | *H* 177, 318, 661–663 |
| 5-Bromo-1-hydroxy-3,6-dimethyl-4(1*H*)-pyridazinone | 192–194, uv | 794 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 5-Bromo-2-β-hydroxyethyl-4-phenoxy-3(2*H*)-pyridazinone | — | 591 |
| 4-Bromo-5-hydroxy-2-isopropyl-3(2*H*)-pyridazinone | 250–251, ir, nmr | 1936 |
| 4-Bromo-5-hydroxy-2-methyl-3(2*H*)-pyridazinone | 285–286, ir, nmr | 1936 |
| 6-Bromo-2-hydroxymethyl-3(2*H*)-pyridazinone | anal | 441 |
| 4-Bromo-5-hydroxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 120, 319 |
| 4-Bromo-5-hydroxy-2-propyl-3(2*H*)-pyridazinone | 199–200, ir, nmr | 1936 |
| 4-Bromo-5-hydroxy-3(2*H*)-pyridazinone | — | *H* 319 |
| 4-Bromo-5-isocyanato-2-phenyl-3(2*H*)-pyridazinone | — | *H* 120 |
| 4-Bromo-6-isopentanesulfonyloxy-3(2*H*)-pyridazinone | — | *H* 331 |
| 3-Bromo-6-isopentyloxypyridazine | — | *H* 178 |
| 3-Bromo-6-isopropoxypyridazine | — | *H* 178, 286 |
| 4-Bromo-2-isopropyl-5-methoxy-3(2*H*)-pyridazinone | 124–124, ir, nmr | 1936 |
| 4-Bromo-5-mercapto-2-phenyl-3(2*H*)-pyridazinone | — | *H* 120, 319, 802 |
| 4-Bromo-5-mercapto-3(2*H*)-pyridazinone | — | *H* 802 |
| 4-Bromo-5-methoxy-3,6-dimethylpyridazine | liq, nmr | 529 |
| 5-Bromo-1-methoxy-3,6-dimethyl-4(1*H*)-pyridazinone | 125–126 | 794 |
| 4-Bromo-3-methoxymethyl-6-methylpyridazine | 58–60, nmr | 446 |
| 4-Bromo-5-methoxy-2-methyl-3(2*H*)-pyridazinone | 159–160, ir, nmr | 1936 |
| 3-Bromo-6-methoxy-5-nitropyridazine 2-oxide | 135–137, nmr, uv | 905 |
| 4-Bromo-5-methoxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 120, 319 |
| 4-Bromo-5-methoxy-2-propyl-3(2*H*)-pyridazinone | 75–76, ir, nmr | 1936 |
| 3-Bromo-6-methoxypyridazine | — | *H* 176, 286 |
| 4-Bromo-5-methylamino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 120, 319, 592 |
| 3-Bromo-6-methylaminopyridazine | — | *H* 303 |
| 5-Bromomethyl-4-cyano-6-ethoxycarbonyl-1-phenylpyridazinium-3-olate | 110, ir, nmr | 1645 |
| 2-Bromomethyl-4,5-dichloro-3(2*H*)-pyridazinone | 74–76, ir, nmr | 1950, 2037 |
| 4-Bromo-5-α-methylhydrazino-2-phenyl-3(2*H*)-pyridazinone | 107–108, ir, nmr | 1540 |
| 4-Bromo-5-α-methylhydrazino-3(2*H*)-pyridazinone | 141–142, ir, nmr | 1540 |
| 4-Bromo-2-methyl-5-α-methylhydrazino-3(2*H*)-pyridazinone | 111–112, ir, nmr | 1540 |
| 5-Bromo-2-methyl-4-α-methylhydrazino-3(2*H*)-pyridazinone | 64–65, ir, nmr | 1540 |
| 3-Bromo-1-methyl-6-methylimino-1,6-dihydropyridazine | — | *H* 302 |
| 4-Bromo-2-methyl-5-morpholino-3(2*H*)-pyridazinone | 148–149, ir, nmr | 542 |
| 5-Bromo-6-methyl-4-oxo-1-phenyl-1,4-dihydro-3-pyridazinecarboxylic acid | — | *H* 134, 333, 443 |
| 5-Bromo-2-methyl-4-phenoxy-3(2*H*)-pyridazinone | — | 591 |
| 4-Bromo-1-methyl-2-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 159, 338 |
| 4-Bromo-2-methyl-1-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 159 |
| 5-Bromomethyl-6-phenyl-3(2*H*)-pyridazinone | 181–184, ir, nmr | 1588 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 5-Bromo-6-methyl-1-phenyl-4(1*H*)-pyridazinone | — | *H* 134, 333 |
| 3-Bromo-6-methylpyridazine | nmr; 2-MeBr: 138–139 | 115, 1037, 1638; *H* 286, 302, 303 |
| 6-Bromo-2-methyl-3(2*H*)-pyridazinimine | — | *H* 302 |
| 4-Bromo-6-methyl-3(2*H*)-pyridazinone 1-oxide | 205–206 | 833; *H* 300 |
| 6-Bromo-4-methyl-3(2*H*)-pyridazinone 1-oxide | 158 | 833; *H* 300 |
| 4-Bromo-2-$\beta$-morpholinoethyl-6-phenyl-3(2*H*)-pyridazinone | — | *H* 101 |
| 4-Bromo-5-morpholino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 120, 320, 592 |
| 6-Bromo-5-morpholino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 92, 330, 593 |
| 4-Bromo-5-morpholino-3(2*H*)-pyridazinone | 185, ir, nmr | 542; *H* 318, 586 |
| 4-Bromo-5-nitro-2-phenyl-3(2*H*)-pyridazinone | — | *H* 120, 321, 648 |
| 6-Bromo-4-nitro-3(2*H*)-pyridazinone 1-oxide | 230, nmr | 905 |
| 5-Bromo-6-oxo-1-phenyl-1,6-dihydro-4-pyridazinecarboxylic acid | — | *H* 119, 320, 439 |
| 3-Bromo-6-*s*-pentyloxypyridazine | — | *H* 286 |
| 2-$\varepsilon$-Bromopentyl-6-phenyl-3(2*H*)-pyridazinone | 191–195/1.5, ir, nmr | 118, 1288, 1289, 1337 |
| 5-Bromo-4-phenoxy-2-phenyl-3(2*H*)-pyridazinone | — | 591; *H* 126 |
| 3-Bromo-6-phenoxypyridazine | — | *H* 179, 286 |
| 5-Bromo-4-phenoxy-3(2*H*)-pyridazinone | — | 591 |
| 4-Bromo-6-phenylmethanesulfonyloxy-3(2*H*)-pyridazinone | — | *H* 331 |
| 4-Bromo-2-phenyl-5-piperidino-3(2*H*)-pyridazinone | — | *H* 120, 592 |
| 4-Bromo-2-phenyl-5-propylamino-3(2*H*)-pyridazinone | — | *H* 120, 320, 592 |
| 3-Bromo-6-phenylpyridazine | — | *H* 286, 302, 303 |
| 4-Bromo-6-phenylpyridazine | 120, ir, ms. nmr | 2093 |
| 4-Bromo-1-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 155 337 |
| 5-Bromo-6-phenyl-3(2*H*)-pyridazinone | — | *H* 87, 330 |
| 5-Bromo-6-phenyl-4(1*H*)-pyridazinone | 2-PhBr: 222 | 273 |
| 6-Bromo-2-phenyl-3(2*H*)-pyridazinone | — | *H* 80, 307, 330 |
| 4-Bromo-2-phenyl-5-thiocyanato-3(2*H*)-pyridazinone | — | *H* 320 |
| 5-Bromo-3-piperidinomethyl-4(1*H*)-pyridazinone 2-oxide | — | *H* 299, 609 |
| 3-Bromo-6-propoxypyridazine | — | *H* 178, 286 |
| 2-$\gamma$-Bromopropyl-4,5-dichloro-3(2*H*)-pyridazinone | 74–76, ir, nmr | 1950, 2037 |
| 2-$\gamma$-Bromopropyl-6-phenyl-3(2*H*)-pyridazinone | 210–213/0.8, nmr | 118, 1337 |
| 3-Bromo-4-pyridazinamine | 114–118, nmr | 447 |
| 5-Bromo-4-pyridazinamine | 134–136, nmr; pic: 216–217 | 447 |
| 6-Bromo-3-pyridazinamine | — | *H* 286, 303, 551 |
| 3-Bromopyridazine | — | *H* 286 |
| 6-Bromo-3-pyridazinecarboxylic acid | 149–150 | 1127 |
| 4-Bromo-3,6(1*H*,2*H*)-pyridazinedione | 257, nmr | 614, 654; *H* 150, 339 |
| 3-Bromopyridazine 1-oxide | — | *H* 299 |
| 3-Bromopyridazine 2-oxide | — | *H* 300 |

*continued*

| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 4-Bromopyridazine 1-oxide | — | *H* 299 |
| 4-Bromopyridazine 2-oxide | — | *H* 300 |
| 6-Bromo-3(2*H*)-pyridazinethione | — | 315; *H* 286, 797 |
| 6-Bromo-3(2*H*)-pyridazinone | — | *H* 66, 286 |
| 3-Bromo-6-thiocyanatopyridazine | — | *H* 286, 825 |
| 4-Bromo-3-trimethylsiloxy-5-trimethylsilylaminopyridazine | 84–86, 132–133/1, nmr, uv | 1172 |
| 4-Bromo-3,5,6-triphenylpyridazine | — | *H* 296 |
| 6-Butanesulfonyloxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 81 |
| 6-Butanesulfonyloxy-3(2*H*)-pyridazinone | — | *H* 69 |
| 4-But-2′-enylamino-6-chloro-3-hydrazinopyridazine | — | *H* 583 |
| 4-But-2′-enylamino-3,6-dichloropyridazine | — | *H* 583 |
| 3-Butoxy-4,6-bisdimethylaminopyridazine | HCl: 194–195, nmr | 637 |
| 2-Butoxycarbonylmethyl-6-chloro-3(2*H*)-pyridazinone | — | *H* 78, 306 |
| 6-*t*-Butoxycarbonylmethyl-4,5-dimethyl-3(2*H*)-pyridazinone | 201 | 1742 |
| 4-Butoxycarbonylmethyl-1-methyl-3,6(1*H*,2*H*)-pyridazinedione | 113 | 207 |
| 4-Butoxycarbonylmethyl-2-methyl-3,6(1*H*,2*H*)-pyridazinedione | 105 | 207 |
| 6-*t*-Butoxycarbonylmethyl-5-methyl-3(2*H*)-pyridazinone | 114–116 | 1742 |
| 4-Butoxycarbonylmethyl-3,6-(1*H*,2*H*)-pyridazinedione | — | *H* 151 |
| 6-*t*-Butoxycarbonylmethyl-3(2*H*)-pyridazinone | 135 | 1742 |
| 3-Butoxy-6-chloro-4-diethylaminopyridazine | — | *H* 182, 293, 585 |
| 3-Butoxy-6-chloro-4-dimethylaminopyridazine | 39 | 1796 |
| 3-Butoxy-6-chloro-5-dimethylaminopyridazine | nmr; pic: 99–100 | 640 |
| 4-Butoxy-3-chloro-6-dimethylaminopyridazine | 58–59 | 640, 1796 |
| 4-Butoxy-6-chloro-3-dimethylaminopyridazine | 45–49, nmr | 640 |
| 3-Butoxy-6-chloro-4-ethylaminopyridazine | — | *H* 182, 293, 582 |
| 3-Butoxy-6-chloro-4-methylaminopyridazine | — | *H* 182, 293, 582 |
| 3-Butoxy-4-chloro-5-morpholinopyridazine | 83–85 | 637 |
| 3-Butoxy-6-chloro-4-morpholinopyridazine | 65–68 | 637, 641 |
| 4-Butoxy-3-chloro-6-morpholinopyridazine | 134–135 | 640 |
| 4-Butoxy-5-chloro-6-morpholinopyridazine | 73–74 | 640 |
| 4-Butoxy-6-chloro-3-morpholinopyridazine | 77–78 | 640 |
| 5-Butoxy-4-chloro-2-phenyl-3(2*H*)-pyridazinone | — | *H* 123 |
| 3-Butoxy-6-chloro-4-pyridazinamine | — | *H* 182, 293, 559 |
| 6-Butoxy-5-chloro-4-pyridazinamine | — | *H* 183, 295, 559 |
| 6-Butoxy-5-chloro-3-pyridazinamine 2-oxide | — | *H* 198, 301 |
| 3-Butoxy-6-chloropyridazine | — | *H* 178, 280 |
| 3-*s*-Butoxy-6-chloropyridazine | — | 688; *H* 178 |
| 3-*t*-Butoxy-6-chloropyridazine | — | *H* 178, 280 |
| 5-Butoxy-4-chloro-3(2*H*)-pyridazinone | 203 | 640 |
| 6-Butoxy-4-chloro-3(2*H*)-pyridazinone | 107, nmr | 641 |
| 6-Butoxy-5-chloro-3(2*H*)-pyridazinone | 153 or 155, nmr | 640, 641 |
| 3-Butoxy-6-cyclohexyloxypyridazine | — | 677 |
| 3-Butoxy-5,6-dichloro-4-morpholinopyridazine | 77–78 | 637 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 4-Butoxy-3,6-dichloropyridazine | — | *H* 186, 288 |
| 4-Butoxy-5,6-dichloropyridazine | 49–51 | 640 |
| 3-Butoxy-4-dimethylamino-6-methoxypyridazine | nmr; HCl: 207–208 | 637 |
| 6-Butoxy-4-dimethylamino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 589 |
| 3-Butoxy-4-dimethylaminopyridazine | HCl: 181–182, nmr | 1796 |
| 3-Butoxy-5-dimethylaminopyridazine | HCl: 145 | 1796 |
| 4-Butoxy-6-dimethylaminopyridazine | HCl: 160 | 1796 |
| 4-*t*-Butoxy-5,6-diphenyl-3(2*H*)-pyridazinone | — | 809 |
| 3-Butoxy-6-ethoxy-4-morpholinopyridazine | crude: liq, nmr; HCl: 125–126 | 641 |
| 3-Butoxy-6-ethoxy-5-morpholinopyridazine | crude: liq, nmr; HCl: 126 | 641 |
| 4-Butoxy-2-ethyl-1-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 162 |
| 6-Butoxy-2-hydroxy-3(2*H*)-pyridazinone | — | *H* 76 |
| 3-Butoxy-6-iodopyridazine | — | *H* 178, 287 |
| 4-Butoxy-2-methyl-5-morpholino-3(2*H*)-pyridazinone | liq, anal | 1269 |
| 6-Butoxy-2-methyl-5-morpholino-3(2*H*)-pyridazinone | — | 1725 |
| 3-Butoxy-4-methyl-6-phenylpyridazine | 87–88 | 1279 |
| 4-Butoxy-2-methyl-1-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 160 |
| 5-Butoxy-2-methyl-3,4(1*H*,2*H*)-pyridazinedione | 105–106 | 533 |
| 3-Butoxy-4-morpholinopyridazine | HCl: 167 | 637 |
| 3-Butoxy-5-morpholinopyridazine | HCl: 122 | 637 |
| 4-Butoxy-5-morpholinopyridazine | HCl: 154–156, nmr | 640 |
| 5-Butoxy-6-morpholino-3(2*H*)-pyridazinone | 168–170, ir, nmr | 640 |
| 6-Butoxy-4-morpholino-3(2*H*)-pyridazinone | 183, ir, nmr | 641 |
| 6-Butoxy-5-morpholino-3(2*H*)-pyridazinone | 221–222, ir, nmr | 641 |
| 6-Butoxy-5-nitro-3-pyridazinamine 2-oxide | — | *H* 198, 604 |
| 3-Butoxy-6-phenoxypyridazine | — | 688 |
| 3-*s*-Butoxy-6-phenoxypyridazine | — | 688 |
| 3-Butoxy-6-phenylpyridazine | 75–76 | 1279 |
| 6-Butoxy-3-pyridazinamine | — | *H* 178, 552 |
| 6-Butoxy-4-pyridazinamine | — | *H* 183, 561 |
| 3-Butoxypyridazine | — | *H* 174 |
| 5-Butoxy-3(2*H*)-pyridazinone | 158, nmr | 640 |
| 6-Butoxy-3(2*H*)-pyridazinone | — | *H* 68 |
| 4-Butylamino-3-chloro-6-phenylpyridazine | liq anal | 1041 |
| 4-Butylamino-5-chloro-2-phenyl-3(2*H*)-pyridazinone | 55–56, ir, uv | 148 |
| 4-*t*-Butylamino-5-chloro-2-phenyl-3(2*H*)-pyridazinone | 50–51, nmr | 956 |
| 5-Butylamino-4-chloro-2-phenyl-3(2*H*)-pyridazinone | 81–85, ir, uv | 148; *H* 121, 313 |
| 5-*s*-Butylamino-4-chloro-2-phenyl-3(2*H*)-pyridazinone | — | *H* 121, 313 |
| 5-*t*-Butylamino-4-chloro-2-phenyl-3(2*H*)-pyridazinone | 161–162, nmr | 956 |
| 3-Butylamino-6-chloropyridazine | 99–101 | 1330; *H* 277, 573 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 4-*t*-Butylamino-2,6-diphenylpyridazine | 142, nmr | 56 |
| 3-Butylamino-4-methyl-6-phenylpyridazine | $pK_a$ | 1720 |
| 4-Butylamino-2-methyl-1-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 159, 595 |
| 3-Butylamino-6-methylsulfonylpyridazine | — | *H* 573, 823 |
| 4-Butylamino-6-phenyl-3(2*H*)-pyridazinone | 150–151 | 1041 |
| 6-Butylamino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 80, 594 |
| 3-Butylaminopyridazine | — | *H* 573 |
| 4-Butylaminopyridazine | — | *H* 583 |
| 6-Butylamino-3-pyridazinecarboxamide | 166–168 | 1592, 1773 |
| 6-Butylamino-3-pyridazinesulfonamide | — | *H* 573 |
| *t*-Butyl-5,6-bisdipropylamino-3-pyridazinecarboxylate | 250/0.7, ir, ms, nmr | 1070 |
| *N*-*t*-Butyl-3,5-bistrimethylsilyl-4-pyridazinecarboxamide | 184, nmr | 1609 |
| *N*-Butyl-6-butylamino-3-pyridazinecarboxamide | 121–123 | 1592 |
| 2-(Butylcarbamoyl)methyl-6-phenyl-3(2*H*)-pyridazinone | — | 775 |
| 4-*t*-Butyl-3-chloro-6-dimethylaminopyridazine | 96/0.15 | 137; *H* 292 |
| 1-*t*-Butyl-5-chloro-3-ethyl-4(1*H*)-pyridazinone | 67, nmr | 135 |
| *t*-Butyl-6-chloro-3-hydrazino-4-pyridazinecarboxylate | — | 945 |
| 2-Butyl-4-chloro-5-hydroxy-3(2*H*)-pyridazinone | 169–170, ir, nmr | 1044, 1936 |
| 2-*t*-Butyl-4-chloro-5-hydroxy-3(2*H*)-pyridazinone | — | 1044 |
| 2-Butyl-4-chloro-5-methoxy-3(2*H*)-pyridazinone | 544–55. 162–165/2, ir, nmr | 118, 1936 |
| 4-*t*-Butyl-3-chloro-6-methylpyridazine | 59–62, nmr | 1676 |
| 4-*t*-Butyl-6-chloro-3-methylpyridazine | nmr | 1676; *H* 293 |
| 2-Butyl-4-chloro-5-morpholino-3(2*H*)-pyridazinone | 215–220/2, nmr | 118 |
| 2-Butyl-6-chloro-4-nitro-3(2*H*)-pyridazinone | — | *H* 325 |
| Butyl 3-chloro-6-oxo-1,6-dihydro-1-pyridazinecarboxylate | — | *H* 71, 305 |
| 4-*t*-Butyl-3-chloro-6-phenylpyridazine | 60–61, nmr | 1676 |
| 4-*t*-Butyl-5-chloro-3-phenyl-4(1*H*)-pyridazinone | 151, hmr | 135 |
| 3-*t*-Butyl-6-chloropyridazine | 70 | 1114 |
| *N*-Butyl-6-chloro-3-pyridazinecarboxamide | — | 1592 |
| Butyl 6-chloro-3-pyridazinecarboxylate | — | *H* 276, 434 |
| *N*-Butyl-6-chloro-3-pyridazinesulfonamide | — | *H* 284, 823 |
| 2-Butyl-6-chloro-3(2*H*)-pyridazinone | — | *H* 75, 306 |
| *t*-Butyl 4,6-di-t-butyl-5-diisopropylamino-3-pyridazinecarboxylate | 136, ir, nmr | 1123 |
| 4-*s*-Butyl-3,6-dichloropyridazine | liq, ms, nmr | 1223 |
| 4-*t*-Butyl-3,6-dichloropyridazine | 37–40, ms, nmr | 1223; *H* 288 |
| *t*-Butyl 3,6-dichloro-4-pyridazinecarboxylate | — | 945 |
| 2-Butyl-4,5-dichloro-3(2*H*)-pyridazinone | 125–128/0.8, ir, nmr | 118, 916, 1901, 1936; *H* 114, 309 |
| 2-*s*-Butyl-4,5-dichloro–3(2*H*)-pyridazinone | — | *H* 114, 309 |
| 4-*t*-Butyl-3,6-dimethoxypyridazine | — | 136; *H* 192 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 4-*t*-Butyl-6-dimethylamino-3-methylaminopyridazine | — | *H* 600 |
| 3-Butoxy-5-dimethylaminopyridazine | HCl: 143 | 640 |
| 3-*t*-Butyl-6-dimethylaminopyridazine | 95–97 | 138 |
| 1-Butyl-*N*,*N*-dimethyl-6-oxo-1,6-dihydro-3-pyridazinecarboxamide | 163–164/2, nmr | 118 |
| 6-Butyl-2,4-dimethyl-3(2*H*)-pyridazinone | HCl; 141, nmr | 503 |
| 4-(*t*-Butyldimethylsilyl)pyridazine | liq, anal, ir, nmr | 1387 |
| 3-*t*-Butyl-5,6-diphenylpyridazine | 185–186, nmr, uv | 499 |
| 4-*t*-Butyl-3,6-diphenylpyridazine | 104–105, nmr, uv | 499 |
| 2-Butyl-4-ethoxy-5-morpholino-3(2*H*)-pyridazinone | 164–166/0.15 | 1269 |
| *N*-Butyl-6-ethylsulfonyl-3-pyridazinecarboxamide | 133–134 | 1592 |
| 3-*t*-Butyl-6-hydrazinopyridazine | 100–102, ir, nmr; $Me_2C$=:120–122, nmr | 18 |
| *N*-*t*-Butyl-5-α-hydroxyethyl-4-pyridazinecarboxamide | liq, anal, nmr | 1609 |
| 3-*t*-Butyl-6-isopropylpyridazine | 136–137 | 138 |
| 4-*t*-Butyl-6-methoxy-3-phenylpyridazine | — | *H* 184 |
| 4-*t*-Butyl-6-methoxy-3(2*H*)-pyridazinone | — | *H* 98 |
| 2-*t*-Butyl-5-methyl-3-oxo-2,3-dihydro-4-pyridazinecarboxylic acid | 123–125, ms, nmr | 715 |
| 2-*t*-Butyl-6-methyl-3-oxo-2,3-dihydro-4-pyridazinecarboxylic acid | 86–87, ms, nmr | 715 |
| 3-Butyl-6-methylpyridazine | liq, ir, ms, nmr | 1659 |
| 4-*t*-Butyl-6-methylpyridazine | 68–71, 140/20, nmr | 880 |
| 2-*t*-Butyl-5-methyl-3(2*H*)-pyridazinone | 148–150/16, ms, nmr | 715, 719 |
| 2-Butyl-6-methyl-3(2*H*)-pyridazinone | 105–107/1.5, nmr | 118 |
| 2-*t*-Butyl-6-methyl-3(2*H*)-pyridazinone | 138–140/16, ms, nmr | 715, 719 |
| 1-Butyl-5-morpholino-3,6(1*H*,2*H*)-pyridazinedione | 130–131, ir, nmr | 641 |
| 1-Butyl-6-oxo-1,6-dihydro-3-pyridazinecarboxylic acid | — | *H* 75, 438 |
| 2-*t*-Butyl-3-oxo-2,3-dihydro-4-pyridazinecarboxylic acid | 62–64, ms, nmr | 715 |
| Butyl 6-oxo-3-phenoxy-1,6-dihydro-1-pyridazinecarboxylate | — | 876 |
| Butyl 6-oxo-1-vinyl-1,6-dihydro-3-pyridazinecarboxylate | — | 693 |
| Butyl 3-phenethylamino-6-phenyl-4-pyridazinecarboxylate | xl st | 172 |
| 4-Butyl-6-phenylpyridazine | 97, ms, nmr | 1676 |
| 4-*t*-Butyl-6-phenylpyridazine | 94, ir, ms, nmr | 2093 |
| 2-Butyl-6-phenyl-3(2*H*)-pyridazinone | 166–168/3 | 118 |
| 2-*s*-Butyl-6-phenyl-3(2*H*)-pyridazinone | 166–168/2 | 118 |
| 4-*t*-Butyl-6-phenyl-3(2*H*)-pyridazinone | — | *H* 97 |
| 4-Butyl-3-phenyl-5-trimethylsilylpyridazine | 61, ms, nmr | 12 |
| 4-*t*-Butylpyridazine | — | 2046 |
| *N*-Butyl-4-pyridazinecarboxamide | 153–154, ir, nmr | 1609 |
| 1-Butyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 155 |
| 2-Butyl-3(2*H*)-pyridazinone | 89–90/3, nmr | 118 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 2-*t*-Butyl-3(2*H*)-pyridazinone | 134–136/16, ms, nmr | 715, 719 |
| 6-Butyl-3(2*H*)-pyridazinone | 95, ir, nmr | 1562 |
| 5-*t*-Butylthio-3-chloro-6-phenylpyridazine | 100–102, ir, nmr | 1087 |
| 3-*t*-Butylthio-5-ethoxy-3-phenylpyridazine | 88–90, ir, nmr | 1087 |
| 5-*t*-Butylthio-3-ethoxy-6-phenylpyridazine | 88–90, ir, nmr | 1087 |
| 5-*t*-Butylthio-3-ethoxy-6-phenylpyridazine | 116–117, ir, nmr | 1087 |
| 3-*t*-Butylthio-5-methoxy-6-phenylpyridazine | 90–93, ir | 1087 |
| 5-*t*-Butylthio-3-methoxy-6-phenylpyridazine | 79–80, ir, nmr | 1087 |
| 3-*t*-Butylthio-6-methoxypyridazine | liq, ms, nmr | 1219 |
| 5-*t*-Butylthio-2-methyl-6-phenyl-3(2*H*)-pyridazinone | 109–110, ir, nmr | 1087 |
| 6-*t*-Butylthio-1-methyl-3-phenyl-4(1*H*)-pyridazinone | 103–104, ir, nmr | 1087 |
| 5-*t*-Butylthio-6-phenyl-3-pyridazinamine | 229–230, ir, nmr; HCl: 198, ir, nmr | 133 |
| 5-*t*-Butylthio-6-phenyl-3(2*H*)-pyridazinethione | 248–250, ir, nmr | 1087 |
| 5-*t*-Butylthio-6-phenyl-3(2*H*)-pyridazinone | 211–213, ir, nmr | 1087 |
| 6-Butylthio-3-pyridazinamine | — | *H* 553, 804 |
| *N*-*t*-Butyl-5-trimethylsilyl-4-pyridazinecarboxamide | 194, nmr | 1609 |
| 3-But-3′-ynylamino-4-pyridazinecarbonitrile | — | 2076 |
| 3-But-3′-ynyloxy-6-chloropyridazine | 74–75, ir, ms, nmr | 336 |
| 3-But-3′-ynyloxy-4-pyridazinecarbonitrile | — | 2076 |
| 4-But-1′-ynyl-3-phenyl-5-trimethylsilylpyridazine | 79, ms, nmr | 12 |
| 2-Butyrylmethyl-6-methyl-3(2*H*)-pyridazinone | — | *H* 75, 401 |
| 6-Butyryloxy-3(2*H*)-pyridazinone | — | *H* 68 |
| 3-Butyrylpyridazine | liq, anal, ir, ms, nmr | 1744 |
| 2-*β*-Carbamoylethyl-4,5-dichloro-3(2*H*)-pyridazinone | — | *H* 114, 309 |
| 2-*β*-Carbamoylethyl-6-methyl-3(2*H*)-pyridazinone | 148–150 | 1288 |
| 2-*β*-Carbamoylethyl-6-phenyl-3(2*H*)-pyridazinone | 177–179 | 1288 |
| 6-*β*-Carbamoylethyl-3(2*H*)-pyridazinone | — | *H* 67 |
| 2-Carbamoylmethyl-6-chloro-3(2*H*)-pyridazinone | — | *H* 78, 306 |
| 1-Carbamoylmethyl-4,5-dichloro-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 154, 334 |
| 2-Carbamoylmethyl-4,5-dichloro-3(2*H*)-pyridazinone | — | *H* 114, 309 |
| 2-Carbamoylmethyl-4,6-diphenyl-3(2*H*)-pyridazinone | 174, ir, nmr | 1343 |
| 2-Carbamoylmethyl-6-methyl-3(2*H*)-pyridazinone | — | *H* 78 |
| 2-Carbamoylmethyl-6-phenyl-3(2*H*)-pyridazinone | — | *H* 78 |
| 1-Carbamoylmethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 154 |
| 2-*γ*-Carbamoylpropyl-4-methyl-6-phenyl-3(2*H*)-pyridazinimine | $H_2O$: 119 | 1309 |
| 5-Carbamoyl-4-pyridazinecarboxylic acid | 180–195, ir, nmr | 958 |
| Carbazoyl-, *see* Hydrazinocarbonyl- | | |
| 2-*γ*-Carboxybutyl-4-methyl-6-phenyl-3(2*H*)-pyridazinimine | HBr: 175 | 1309 |
| 2-*δ*-Carboxybutyl-4-methyl-6-phenyl-3(2*H*)-pyridazinimine | HCl: 214 | 1309 |
| 2-*α*-Carboxybutyl-6-methyl-3(2*H*)-pyridazinone | — | *H* 78 |
| 2-*α*-Carboxyethyl-4-chloro-5-morpholino-3(2*H*)-pyridazinone | 217–220, ir, nmr | 534 |
| 2-*β*-Carboxyethyl-6-chloro-3(2*H*)-pyridazinone | — | *H* 73, 306 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 1-β-Carboxyethyl-4,5-dichloro-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 155, 334 |
| 2-β-Carboxyethyl-4,5-dichloro-3(2*H*)-pyridazinone | — | *H* 114, 309 |
| 2-β-Carboxyethyl-4,6-diphenyl-3(2*H*)-pyridazinone | — | 1419 |
| 2-β-Carboxyethyl-4-ethoxy-5-morpholino-3(2*H*)-pyridazinone | anal, ir, nmr | 534 |
| 2-β-Carboxyethyl-4-methyl-6-phenyl-3(2*H*)-pyridazinone | HBr: >210 | 1309 |
| 2-α-Carboxyethyl-6-methyl-3(2*H*)-pyridazinone | — | *H* 78 |
| 2-β-Carboxyethyl-6-methyl-3(2*H*)-pyridazinone | 142–144 | 1288; *H* 73 |
| 2-α-Carboxyethyl-5-morpholino-3(2*H*)-pyridazinone | 225–228, ir, nmr | 534 |
| 2-β-Carboxyethyl-6-phenyl-3(2*H*)-pyridazinone | — | 775 |
| 1-β-Carboxyethyl-3,6 (1*H*,2*H*)-pyridazinedione | — | *H* 155 |
| 6-β-Carboxyethyl-3(2*H*)-pyridazinone | — | *H* 67 |
| 2-Carboxymethyl-4-chloro-5-hydroxy-3(2*H*)-pyridazinone | — | *H* 119, 312 |
| 2-Carboxymethyl-6-chloro-5-hydroxy-3(2*H*)-pyridazinone | — | *H* 92, 326 |
| 2-Carboxymethyl-4-chloro-5-methoxy-3(2*H*)-pyridazinone | — | *H* 119, 312 |
| 4-Carboxymethyl-5-chloro-2-methyl-3(2*H*)-pyridazinone | 93–95, ir, nmr | 534 |
| 2-Carboxymethyl-6-chloro-4-methylthio-3(2*H*)-pyridazinone | — | *H* 101, 323, 811 |
| 2-Carboxymethyl-4-chloro-5-morpholino-3(2*H*)-pyridazinone | 193–194, ir, nmr | 534 |
| 5-Carboxymethyl-6-chloro-2-phenyl-3(2*H*)-pyridazinone | — | *H* 93, 326 |
| 2-Carboxymethyl-6-chloro-3,4(1*H*,2*H*)-pyridazinedione | — | *H* 323 |
| 2-Carboxymethyl-6-chloro-3(2*H*)-pyridazinone | — | *H* 77, 305 |
| 4-Carboxymethyl-6-chloro-3(2*H*)-pyridazinone | 160–162, ir, uv | 947 |
| 5-Carboxymethyl-6-chloro-3(2*H*)-pyridazinone | 169–171, ir, uv | 947 |
| 1-Carboxymethyl-4,5-dichloro-2-methyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 164, 334 |
| 4-Carboxymethyl-3,6-dichloropyridazine | 109–111, ir, nmr | 947 |
| 1-Carboxymethyl-4,5-dichloro-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 153, 334 |
| 2-Carboxymethyl-4,5-dichloro-3(2*H*)-pyridazinone | — | *H* 114, 309 |
| 2-Carboxymethyl-4,6-dichloro-3(2*H*)-pyridazinone | — | *H* 101, 324 |
| 2-Carboxymethyl-5,6-dichloro-3(2*H*)-pyridazinone | — | *H* 92, 327 |
| 2-Carboxymethyl-4,6-dimethyl-3(2*H*)-pyridazinone | — | *H* 101 |
| 2-Carboxymethyl-5,6-dimethyl-3(2*H*)-pyridazinone | — | *H* 92 |
| 2-Carboxymethyl-4,6-diphenyl-3(2*H*)-pyridazinone | — | 1419 |
| 2-(α-Carboxy-α-methylethyl)-6-methyl-3(2*H*)-pyridazinone | — | *H* 78 |
| 1-(β-Carboxy-α-methylethyl)-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 155 |
| 2-Carboxymethyl-6-ethyl-3(2*H*)-pyridazinone | — | *H* 77 |

*continued*

| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 2-Carboxymethyl-6-ethylsulfonyl-3(2*H*)-pyridazinone | — | *H* 78 |
| 4-Carboxymethyl-6-hydrazino-3(2*H*)-pyridazinone | HCl: 223–226, ir | 947 |
| 2-Carboxymethyl-6-isopropylsulfonyl-3(2*H*)-pyridazinone | — | *H* 78 |
| 2-Carboxymethyl-4-methyl-6-phenyl-3(2*H*)-pyridazinone | 240 | 1309 |
| 4-Carboxymethyl-1-methyl-3,6(1*H*,2*H*)-pyridazinedione | 252 | 207 |
| 4-Carboxymethyl-2-methyl-3,6(1*H*,2*H*)-pyridazinedione | 210–215 | 207 |
| 2-Carboxymethyl-4-methyl-3(2*H*)-pyridazinone | — | *H* 84 |
| 2-Carboxymethyl-6-methyl-3(2*H*)-pyridazinone | — | *H* 77 |
| 4-Carboxymethyl-2-methyl-3(2*H*)-pyridazinone | 89–91, ir, nmr | 534 |
| 2-Carboxymethyl-6-methylsulfonyl-3(2*H*)-pyridazinone | — | *H* 78 |
| 2-Carboxymethyl-5-morpholino-3(2*H*)-pyridazinone | 231–236, ir, nmr | 534 |
| 1-Carboxymethyl-6-oxo-1,6-dihydro-3-pyridazinecarboxylic acid | — | *H* 77, 437 |
| 2-Carboxymethyl-3-oxo-6-phenyl-2,3-dihydro-4-pyridazinecarboxylic acid | 240, ir, nmr | 1768 |
| 3-Carboxymethyl-6-phenylpyridazine | — | 92 |
| 4-Carboxymethyl-1-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 156 |
| 2-Carboxymethyl-6-phenyl-3(2*H*)-pyridazinone | — | 775; *H* 77 |
| 3-Carboxymethyl-6-phenyl-4(1*H*)-pyridazinone | 282–283, ir, nmr | 1489 |
| 2-(γ-Carboxy-α-methylpropyl)-4-methyl-6-phenyl-3(2*H*)-pyridazinimine | HBr: 180 | 1309 |
| 2-(α-Carboxy-β-methylpropyl)-6-methyl-3(2*H*)-pyridazinone | — | *H* 78 |
| 1-Carboxymethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 153 |
| 4-Carboxymethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 151 |
| 2-Carboxymethyl-3(2*H*)-pyridazinone | 165–170 | 147; *H* 65 |
| 4-Carboxymethyl-3(2*H*)-pyridazinone | 168–170, ir | 947 |
| 2-Carboxymethyl-4,5,6-trichloro-3(2*H*)-pyridazinone | — | *H* 129, 329 |
| 2-ε-Carboxypentyl-4-methyl-6-phenyl-3(2*H*)-pyridazinimine | HBr: 197 | 1309 |
| 2-α-Carboxypentyl-6-methyl-3(2*H*)-pyridazinone | — | *H* 78 |
| 5-(2-Carboxyprop-1-enyl)-3,6-dichloro-4(1*H*)-pyridazinone | crude: nmr | 963 |
| 2-γ-Carboxypropyl-6-chloro-3(2*H*)-pyridazinone | $H_2O$: 182 | 1309 |
| 2-γ-Carboxypropyl-6-cyclohexyl-3(2*H*)-pyridazinimine | HBr: 170, xl st | 1192, 1309 |
| 2-γ-Carboxypropyl-4,6-diphenyl-3(2*H*)-pyridazinimine | HCl: 204 | 1309 |
| 2-γ-Carboxypropyl-6-isobutyl-3(2*H*)-pyridazinimine | HBr: 195 | 1309 |
| 2-γ-Carboxypropyl-4-methyl-6-phenyl-3(2*H*)-pyridazinimine | HCl: 238 or 248–249 | 1118, 1309 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 2-γ-Carboxypropyl-5-methyl-6-phenyl-3(2*H*)-pyridazinimine | HBr: anal | 1309 |
| 2-γ-Carboxypropyl-6-methyl-5-phenyl-3(2*H*)-pyridazinimine | HBr: 186 | 1309 |
| 2-γ-Carboxypropyl-4-methyl-3(2*H*)-pyridazinimine | HBr: 146 | 1309 |
| 2-α-Carboxypropyl-6-methyl-3(2*H*)-pyridazinone | — | *H* 78 |
| 2-γ-Carboxypropyl-5-phenyl-3(2*H*)-pyridazinimine | HCl: 238 | 1309 |
| 2-γ-Carboxypropyl-6-phenyl-3(2*H*)-pyridazinimine | HBr: 240, xl st | 171, 1309 |
| 1-β-Carboxypropyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 155 |
| 2-γ-Carboxypropyl-4,5,6-triphenyl-3(2*H*)-pyridazinone | 171–173 | 1272 |
| 3-β-Carboxyvinylpyridazine | 245, ir, nmr | 1668 |
| 4-β-Carboxyvinylpyridazine | 224–226, ir, nmr | 67 |
| 4-Chloro-3,6-bischloromethylpyridazine | xl st | 174 |
| 6-Chloro-4,5-bisdimethylamino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 130, 602 |
| 3-Chloro-4,6-bisdimethylaminopyridazine | HCl: 218–220 | 637 |
| 3-Chloro-4,6-bismorpholinomethylpyridazine | 100–101, nmr | 769 |
| 6-Chloro-4-(β-chloro-α,α-dimethylethyl)-3(2*H*)-pyridazinone | 189–192, nmr | 354 |
| 6-Chloro-2-β-chloroethyl-3(2*H*)-pyridazinone | 55–57, ir, nmr, uv | 237; *H* 71, 305 |
| 3-Chloro-4-chloromethyl-5,6-dimethylpyridazine | — | *H* 294 |
| 3-Chloro-5-chloromethyl-6-methoxypyridazine | — | *H* 181, 292 |
| 3-Chloro-4-chloromethyl-6-methylpyridazine | — | *H* 291 |
| 6-Chloro-2-chloromethyl-3(2*H*)-pyridazinone | 88–89 | 441; *H* 70, 305 |
| 6-Chloro-2-γ-chloropropyl-3(2*H*)-pyridazinone | liq, ir, nmr, uv | 237 |
| 3-Chloro-6-cyanoaminopyridazine | 180–181, nmr | 1010 |
| 6-Chloro-4-cyanoamino-3(2*H*)-pyridazinone | $NH_4$: 250, nmr; $H_2NNH_3$: 165, nmr | 84 |
| 3-Chloro-6-(*N*-cyano-*N*-ethylamino)pyridazine | 103–112 (!), nmr | 1010 |
| 4-Chloro-2-β-cyanoethyl-5-isopropylamino-3(2*H*)-pyridazinone | — | *H* 119, 312, 592 |
| 4-Chloro-2-β-cyanoethyl-5-morpholino-3(2*H*)-pyridazinone | 101–102, nmr | 118 |
| 6-Chloro-2-β-cyanoethyl-3(2*H*)-pyridazinone | — | *H* 71, 306 |
| 3-Chloro-6-(*N*-cyano-*N*-methylamino)pyridazine | 160–161, nmr | 1010 |
| 3-Chloro-6-(*N*-cyano-*N*-methylamino)pyridazine 1-oxide | — | 921 |
| 4-Chloro-5-cyclohexylamino-2-methyl-3(2*H*)-pyridazinone | 87–88, ir, uv | 148 |
| 5-Chloro-4-cyclohexylamino-2-methyl-3(2*H*)-pyridazinone | 33–35, ir, uv | 148 |
| 4-Chloro-5-cyclohexylamino-2-phenyl-3(2*H*)-pyridazinone | 56–67 (!), ir, uv | 148 |
| 5-Chloro-4-cyclohexylamino-2-phenyl-3(2*H*)-pyridazinone | 42–44, ir, uv | 148 |
| 3-Chloro-6-cyclohexylaminopyridazine | — | *H* 277, 574 |
| 4-Chloro-5-cyclohexylamino-3(2*H*)-pyridazinone | — | *H* 116, 310, 583 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 4-Chloro-2-cyclohexyl-5-hydrazino-3(2*H*)-pyridazinone | — | *H* 124, 315 |
| 4-Chloro-2-cyclohexyl-5-hydroxy-3(2*H*)-pyridazinone | — | 1044; *H* 224 |
| 4-Chloro-2-cyclohexyl-5-isocyanato-3(2*H*)-pyridazinone | — | *H* 124, 316, 564 |
| 4-Chloro-2-cyclohexyl-5-methoxy-3(2*H*)-pyridazinone | — | *H* 124 |
| 4-Chloro-2-cyclohexyl-5-methylamino-3(2*H*)-pyridazinone | 197–198, ir, uv | 148 |
| 5-Chloro-2-cyclohexyl-4-methylamino-3(2*H*)-pyridazinone | 113–115, ir, uv | 148 |
| 3-Chloro-4-cyclohexyl-6-methylpyridazine | 79–80, nmr | 1676 |
| 3-Chloro-5-cyclohexyl-6-methylpyridazine | liq, ms, nmr | 1676 |
| 3-Chloro-6-cyclohexyloxypyridazine | — | 677; *H* 181, 281 |
| 4-Chloro-1-cyclohexyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 158, 337 |
| 4-Chloro-2-cyclohexyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 158, 337 |
| 3-Chloro-4-diallylamino-6-hydrazinopyridazine | — | *H* 292, 585 |
| 3-Chloro-6-diallylaminopyridazine | 138–140/0.5 | 1258; *H* 284, 579 |
| 3-Chloro-4,5-dicyclohexyl-6-methylpyridazine | 154–159, ms, nmr | 1676 |
| 4-Chloro-3,6-diethoxypyridazine | 49 | 640 |
| 4-Chloro-2-$\beta$-diethylaminoethyl-5-methoxy-3(2*H*)-pyridazinone | — | *H* 119 |
| 4-Chloro-2-$\beta$-diethylaminoethyl-5-morpholino-3(2*H*)-pyridazinone | — | *H* 119, 312, 591 |
| 4-Chloro-2-$\beta$-diethylaminoethyl-5-piperidino-3(2*H*)-pyridazinone | — | *H* 119, 312, 591 |
| 3-Chloro-5-diethylamino-6-methoxypyridazine | — | *H* 182, 293, 585 |
| 6-Chloro-2-diethylaminomethyl-3(2*H*)-pyridazinone | — | *H* 17, 305 |
| 4-Chloro-5-diethylamino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 121, 313, 316, 591 |
| 6-Chloro-4-diethylamino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 104, 489 |
| 5-Chloro-4-diethylamino-2-propyl-3(2*H*)-pyridazinone | 112/2.6, ir, uv | 148 |
| 3-Chloro-6-diethylaminopyridazine | 49–50 or 51–53; HCl: 133–134 | 924, 1258; *H* 278, 578 |
| 4-Chloro-5-diethylamino-3(2*H*)-pyridazinone | — | *H* 116, 311, 585 |
| 6-Chloro-2-(diethylcarbamoyl)methyl-3(2*H*)-pyridazinone | — | *H* 79, 306 |
| 3-Chloro-4,5-diethyl-6-methylpyridazine | 66–71, ms, nmr | 1676 |
| 3-Chloro-*N*,*N*-diethyl-6-oxo-1,6-dihydro-1-pyridazinecarboxamide | — | *H* 71, 305 |
| 4-Chloro-3,6-diisopropoxypyridazine | 19–20, nmr | 640 |
| 3-Chloro-4,5-diisopropyl-6-methylpyridazine | 103–107, ms, nmr | 1676 |
| 6-Chloro-4,5-dimethoxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 130, 328 |
| 3-Chloro-4,5-dimethoxypyridazine | 95–97, nmr | 882; *H* 185, 290 |
| 3-Chloro-4,6-dimethoxypyridazine | 110–111, nmr | 640, 881; *H* 184, 291 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3-Chloro-5,6-dimethoxypyridazine | 127–128 or 128–129, nmr, uv | 665, 881, 917; *H* 186, 293 |
| 4-Chloro-3,5-dimethoxypyridazine | 161–163 or 163–164, nmr; 1-MeI; 145–150 then 203–205 | 689, 882; *H* 185, 295 |
| 4-Chloro-3,6-dimethoxypyridazine | — | *H* 192, 295 |
| 4-Chloro-5,6-dimethoxypyridazine | 59–60, nmr | 882 |
| 3-Chloro-5,6-dimethoxypyridazine 2-oxide | 187–188 | 905; *H* 197, 300, 301 |
| 4-Chloro-3,6-dimethoxypyridazine 1-oxide | — | *H* 197, 300 |
| 3-Chloro-5-dimethylamino-6-ethoxypyridazine | — | *H* 182, 293, 584 |
| 3-Chloro-6-dimethylamino-4-ethoxypyridazine | 102–103 | 637 |
| 3-Chloro-6-dimethylamino-5-ethylpyridazine | — | *H* 293 |
| 3-Chloro-6-dimethylamino-4-hexyloxypyridazine | 101 | 637 |
| 3-Chloro-5-dimethylamino-6-isopropoxypyridazine | — | *H* 182, 293, 584 |
| 3-Chloro-6-dimethylamino-4-isopropylpyridazine | — | *H* 292 |
| 3-Chloro-6-dimethylamino-5-isopropylpyridazine | — | *H* 293 |
| 3-Chloro-5-dimethylamino-6-methoxypyridazine | — | *H* 181, 293, 584 |
| 3-Chloro-6-dimethylamino-4-methoxypyridazine | 158–159, or 159–160, nmr | 637, 640 |
| 3-Chloro-6-dimethylamino-5-methoxypyridazine | 70–71, nmr | 640 |
| 3-Chloro-6-dimethylamino-4-methylpyridazine | — | *H* 291, 302, 303, 577 |
| 3-Chloro-6-dimethylamino-5-methylpyridazine | — | *H* 302, 303, 577 |
| 4-Chloro-5-dimethylamino-2-methyl-3(2*H*)-pyridazinone | 74–75, ir, nmr, uv | 148, 641, 1213; *H* 591 |
| 5-Chloro-4-dimethylamino-2-methyl-3(2*H*)-pyridazinone | 28–29 or 31–32, ir, nmr, uv | 148, 641, 1213 |
| 6-Chloro-2-dimethylaminomethyl-3(2*H*)-pyridazinone | — | *H* 70, 305 |
| 6-Chloro-4-dimethylamino-2-methyl-3(2*H*)-pyridazinone | — | *H* 99, 323, 588 |
| 6-Chloro-5-dimethylamino-2-methyl-3(2*H*)-pyridazinone | — | *H* 91, 326, 591 |
| 3-Chloro-6-dimethylamino-4-pentyloxypyridazine | 106 | 637 |
| 3-Chloro-5-dimethylamino-6-phenoxypyridazine | — | *H* 182, 293, 584 |
| 3-Chloro-4-dimethylamino-6-phenylpyridazine | 89–90 | 1041 |
| 3-Chloro-6-dimethylamino-5-phenylpyridazine | — | *H* 293 |
| 4-Chloro-5-dimethylamino-2-phenyl-3(2*H*)-pyridazinone | 98–100, ir, nmr, uv | 148, 1213; *H* 121, 313, 591 |
| 5-Chloro-4-dimethylamino-2-phenyl-3(2*H*)-pyridazinone | 79–80, ir, nmr, uv | 148, 1213 |
| 6-Chloro-4-dimethylamino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 104, 323, 589 |
| 6-Chloro-5-dimethylamino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 93, 327, 593 |
| 3-Chloro-5-dimethylamino-6-propoxypyridazine | — | *H* 182, 293, 584 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3-Chloro-6-dimethylamino-4-propoxypyridazine | 102–103 | 637 |
| 6-Chloro-2-γ-dimethylaminopropyl-3(2*H*)-pyridazinone | HCl: 170–172 | 1760 |
| 3-Chloro-5-dimethylaminopyridazine | — | *H* 584 |
| 3-Chloro-6-dimethylaminopyridazine | 100–101 or 103–104, dipole, p$K_a$, nmr, uv; HCl: 230–231 | 94, 924, 1183, 1258, 1352, 1796; *H* 278, 302, 303, 577 |
| 4-Chloro-5-dimethylamino-3(2*H*)-pyridazinone | 202–203, nmr | 641, 1794; *H* 116, 311, 584 |
| 5-Chloro-4-dimethylamino-3(2*H*)-pyridazinone | 151–152, nmr | 1794 |
| 6-Chloro-3-dimethylamino-4(1*H*)-pyridazinone | 239–244 | 640 |
| 6-Chloro-4-dimethylamino-3(2*H*)-pyridazinone | — | *H* 96, 293, 584 |
| 6-Chloro-5-dimethylamino-3(2*H*)-pyridazinone | — | *H* 87 |
| 6-Chloro-2-(dimethylcarbamoyl)methyl-3(2*H*)-pyridazinone | — | *H* 79, 306 |
| 3-Chloro-4,5-dimethyl-6-morpholinopyridazine | 98–99 | 1293 |
| 3-Chloro-5,6-dimethyl-4-nitropyridazine 1-oxide | — | *H* 301, 649 |
| 3-Chloro-4,5-dimethyl-6-phenylpyridazine | 166, nmr | 761, 762 |
| 6-Chloro-4,5-dimethyl-3-pyridazinamine | 199–200 | 116; *H* 294 |
| 3-Chloro-4,5-dimethylpyridazine | 63, nmr | 762; *H* 289 |
| 3-Chloro-4,6-dimethylpyridazine | nmr | 762, 1514; *H* 290 |
| 3-Chloro-5,6-dimethylpyridazine | — | *H* 292 |
| 4-Chloro-3,6-dimethylpyridazine | — | *H* 295 |
| 3-Chloro-5,6-dimethyl-4-pyridazinecarbonitrile | — | *H* 294, 447 |
| 4-Chloro-1,2-dimethyl-3,6(1*H*,2*H*)-pyridazinedione | — | 980, 997 |
| 4-Chloro-3,6-dimethylpyridazine 1,2-dioxide | 199–200, nmr | 796 |
| 3-Chloro-5,6-dimethylpyridazine 1-oxide | — | *H* 301 |
| 3-Chloro-5,6-dimethylpyridazine 2-oxide | — | *H* 300 |
| 4-Chloro-3,6-dimethylpyridazine 1-oxide | 132–133 | 794 |
| 4-Chloro-3,6-dimethylpyridazine 2-oxide | — | *H* 300 |
| 4-Chloro-2,6-dimethyl-3(2*H*)-pyridazinone | — | *H* 99 |
| 5-Chloro-2,6-dimethyl-3(2*H*)-pyridazinone | — | *H* 91, 326 |
| 6-Chloro-4,5-dimethyl-3(2*H*)-pyridazinone | 200, nmr | 755 |
| 3-Chloro-4,6-dimorpholinopyridazine | 175–176 or 180–183 | 544, 637 |
| 4-Chloro-3,5-dimorpholinopyridazine | 149–150 or 152–153, nmr | 637, 1058 |
| 3-Chloro-4,6-diphenylpyridazine | 100, ir, nmr | 1082; *H* 291 |
| 3-Chloro-5,6-diphenylpyridazine | — | *H* 293 |
| 4-Chloro-3,5-diphenylpyridazine | — | 1802; *H* 295 |
| 4-Chloro-3,6-diphenylpyridazine | 133–134 or 135–136, nmr | 451, 1071; *H* 295 |
| 3-Chloro-5,6-diphenyl-4-pyridazinecarbonitrile | 201, ir, nmr | 1046, 1056, 1398, 1411, 1726 |
| 4-Chloro-2,6-diphenyl-3(2*H*)-pyridazinone | — | *H* 107, 322 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3-Chloro-4,6-dipiperidinopyridazine | 105–107 or 108–110, ir, nmr | 544, 640 |
| 4-Chloro-3,5-dipiperidinopyridazine | 81–83, ms, nmr | 1058 |
| 4-Chloro-2-α-ethoxycarbonylethyl-5-morpholino-3(2*H*)-pyridazinone | anal, ir, nmr | 534 |
| 4-Chloro-5-ethoxycarbonylmethyl-3,6-diphenylpyridazine | 99–100, ir, nmr | 222 |
| 4-Chloro-2-ethoxycarbonylmethyl-5-methoxy-3(2*H*)-pyridazinone | — | *H* 119, 312 |
| 6-Chloro-2-ethoxycarbonylmethyl-5-methoxy-3(2*H*)-pyridazinone | — | *H* 92, 326 |
| 4-Chloro-2-ethoxycarbonylmethyl-5-morpholino-3(2*H*)-pyridazinone | 134, ir, nmr | 534 |
| 6-Chloro-2-ethoxycarbonylmethyl-3(2*H*)-pyridazinone | — | *H* 77, 305 |
| 3-Chloro-4-ethoxy-5-formamidopyridazine | 240–241, nmr | 83 |
| 3-Chloro-6-ethoxy-4-methyl-5-nitropyridazine 2-oxide | 111–112, nmr | 902 |
| 3-Chloro-6-ethoxy-4-methylpyridazine | — | *H* 184, 291 |
| 3-Chloro-6-ethoxy-5-methylpyridazine | — | *H* 182, 292 |
| 4-Chloro-5-ethoxy-2-methyl-3(2*H*)-pyridazinone | 100–101, ir, nmr | 1089 |
| 5-Chloro-4-ethoxy-2-methyl-3(2*H*)-pyridazinone | 33–34 or 34–35, ir, nmr | 1089, 1269 |
| 6-Chloro-5-ethoxy-2-methyl-3(2*H*)-pyridazinone | — | *H* 91, 326 |
| 4-Chloro-5-ethoxy-2-phenyl-3(2*H*)-pyridazinone | 135–136 or 136–137, ir, nmr | 764, 1089; *H* 123, 315 |
| 5-Chloro-4-ethoxy-2-phenyl-3(2*H*)-pyridazinone | 85–86, ir, nmr | 1089 |
| 6-Chloro-4-ethoxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 105 |
| 6-Chloro-5-ethoxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 93, 327 |
| 5-Chloro-6-ethoxy-4-pyridazinamine | — | *H* 183, 295, 559 |
| 6-Chloro-3-ethoxy-4-pyridazinamine | — | *H* 182, 293, 559 |
| 6-Chloro-5-ethoxy-4-pyridazinamine | 129–130, nmr | 83 |
| 5-Chloro-6-ethoxy-3-pyridazinamine 2-oxide | — | *H* 198, 301 |
| 3-Chloro-4-ethoxypyridazine | — | *H* 186, 290 |
| 3-Chloro-5-ethoxypyridazine | — | *H* 186, 289 |
| 3-Chloro-6-ethoxypyridazine | 59–60 | 99, 1258; *H* 177, 280 |
| 6-Chloro-3-ethoxy-4-pyridazinecarboxamide | 155–156 | 1099 |
| 3-Chloro-6-ethoxypyridazine 1-oxide | — | *H* 195, 299 |
| 3-Chloro-6-ethoxypyridazine 2-oxide | 115–116 | 103; *H* 195, 300 |
| 3-Chloro-5-ethoxy-4(1*H*)-pyridazinethione | — | *H* 290 |
| 6-Chloro-5-ethoxy-3(2*H*)-pyridazinone | — | *H* 87 |
| 3-Chloro-5-ethylamino-6-hydrazinopyridazine | — | *H* 582 |
| 3-Chloro-5-ethylamino-6-methoxypyridazine | — | *H* 181, 293, 582 |
| 6-Chloro-3-ethylamino-2-methyl-5-oxo-2,5-dihydro-4-pyridazinecarboxylic acid | 146–147, ir, nmr | 967 |
| 4-Chloro-5-ethylamino-2-phenyl-3(2*H*)-pyridazinone | 154–156, nmr | 956; *H* 121, 313 |
| 5-Chloro-4-ethylamino-2-phenyl-3(2*H*)-pyridazinone | 77–78, nmr | 956; *H* 125 |

*continued*

| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3-Chloro-6-ethylaminopyridazine | — | *H* 276, 572 |
| 6-Chloro-3-ethylamino-4-pyridazinecarbonitrile | 221–222, uv | 820 |
| 6-Chloro-3-ethylamino-4-pyridazinecarboxamide | 185–186, uv | 820 |
| 3-Chloro-6-ethylaminopyridazine 1-oxide | — | *H* 299, 604 |
| 3-Chloro-6-ethylaminopyridazine 2-oxide | — | *H* 300, 604 |
| 4-Chloro-5-ethylamino-3(2*H*)-pyridazinone | — | *H* 116, 310, 582 |
| 2-$\beta$-Chloroethyl-4-ethoxy-5-morpholino-3(2*H*)-pyridazinone | 105–107, ir | 53 |
| 4-Chloro-2-ethyl-5-hydroxy-3(2*H*)-pyridazinone | 233–235, ir, nmr | 1936 |
| 6-Chloro-2-ethyl-5-hydroxy-3(2*H*)-pyridazinone | — | *H* 91, 326 |
| 4-Chloro-2-ethyl-5-methoxy-3(2*H*)-pyridazinone | 99–100, ir, nmr | 1936 |
| 4-Chloro-2-ethyl-5-methylamino-3(2*H*)-pyridazinone | 166–167, ir, uv | 148 |
| 5-Chloro-2-ethyl-4-methylamino-3(2*H*)-pyridazinone | 74–76, ir, uv | 148 |
| 3-Chloro-4-ethyl-6-methylpyridazine | liq, ms, nmr | 1676; *H* 291 |
| 3-Chloro-5-ethyl-6-methylpyridazine | crude: ms | 1676 |
| 2-$\beta$-Chloroethyl-6-methyl-3(2*H*)-pyridazinone | 58 | 433; *H* 71 |
| 4-Chloro-2-ethyl-6-methyl-3(2*H*)-pyridazinone | — | *H* 100 |
| 6-Chloro-2-ethyl-4-nitro-3(2*H*)-pyridazinone | — | *H* 100, 325 |
| 3-Chloro-4-ethyl-6-phenylpyridazine | 61–63, nmr | 1676 |
| 4-Chloro-5-ethyl-3-phenylpyridazine | — | 1802 |
| 2-$\beta$-Chloroethyl-6-phenyl-3(2*H*)-pyridazinone | 57 to 70, nmr | 237 |
| 4-Chloro-6-ethyl-2-phenyl-3(2*H*)-pyridazinone | — | *H* 106, 322 |
| 3-Chloro-6-ethylpyridazine | — | *H* 276, 302, 303 |
| 4-$\alpha$-Chloroethylpyridazine | liq, ms, nmr | 82 |
| 4-Chloro-1-ethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 153, 339 |
| 2-$\beta$-Chloroethyl-3(2*H*)-pyridazinone | 114–115/1, nmr | 1179 |
| 6-Chloro-4-ethyl-3(2*H*)-pyridazinone | 138–141, nmr, uv | 30, 821 |
| 6-Chloro-5-ethyl-3(2*H*)-pyridazinone | 190–193 or 205, nmr, uv | 30, 821 |
| 3-Chloro-5-ethylthio-6-methoxypyridazine 2-oxide | 103–104, uv | 905 |
| 3-Chloro-6-ethylthio-4-methylpyridazine | — | *H* 806 |
| 3-Chloro-6-ethylthio-5-methylpyridazine | — | *H* 806 |
| 5-Chloro-4-ethylthio-2-methyl-3(2*H*)-pyridazinone | liq, anal, ir, nmr | 1089 |
| 4-Chloro-5-ethylthio-2-phenyl-3(2*H*)-pyridazinone | 101–102, ir, nmr | 1089; *H* 812 |
| 5-Chloro-4-ethylthio-2-phenyl-3(2*H*)-pyridazinone | 64–65, ir, nmr | 1089 |
| 6-Chloro-4-ethylthio-2-phenyl-3(2*H*)-pyridazinone | — | *H* 105, 323, 810 |
| 6-Chloro-5-ethylthio-2-phenyl-3(2*H*)-pyridazinone | — | *H* 812 |
| 3-Chloro-6-ethylthiopyridazine | — | *H* 283, 804 |
| 3-Chloro-6-ethylthiopyridazine 1-oxide | — | *H* 299 |
| 4-Chloro-5-ethylthio-3(2*H*)-pyridazinone | — | *H* 118, 312, 812 |
| 3-Chloro-5-fluoro-6-methoxypyridazine | — | 917 |
| 4-Chloro-5-fluoro-2-phenyl-3(2*H*)-pyridazinone | — | 1018 |
| 3-Chloro-6-fluoropyridazine | — | 1650 |
| 3-Chloro-5-formamido-4-methoxypyridazine | 250–253, nmr | 83 |
| 3-Chloro-6-formamidopyridazine | 176–178 or 208–210, ir, ms, nmr; oxime: 195–199 | 198, 295, 1010 |
| 3-Chloro-6-formamidopyridazine 1-oxide | oxime: 175–177 | 921 |
| 3-Chloro-6-$\beta$-formylhydrazino-4-methylpyridazine | — | *H* 668 |

*continued*

| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3-Chloro-6-β-formylhydrazino-5-methylpyridazine | — | *H* 668 |
| 3-Chloro-6-β-formylhydrazinopyridazine | — | *H* 279, 668 |
| 3-Chloro-5-guanidino-4(1*H*)-pyridazinone | 230, nmr | 83 |
| 3-Chloro-6-hexyloxypyridazine | — | *H* 179, 281 |
| 3-Chloro-6-hex-5′-ynyloxypyridazine | 31–32, ir, ms, nmr | 336 |
| 3-Chloro-4-hydrazinocarbonylmethylpyridazine | 125–126, ir | 947 |
| 6-Chloro-2-hydrazinocarbonylmethyl-3(2*H*)-pyridazinone | — | *H* 78, 306 |
| 6-Chloro-4-hydrazinocarbonylmethyl-3(2*H*)-pyridazinone | 194–196, ir, uv | 947 |
| 6-Chloro-5-hydrazinocarbonylmethyl-3(2*H*)-pyridazinone | 242–245, ir | 947 |
| 3-Chloro-6-hydrazino-4,5-dimethylpyridazine | — | *H* 660 |
| 3-Chloro-6-hydrazino-4-hydrazinocarbonylpyridazine | 194–196, ir | 947 |
| 4-Chloro-5-hydrazino-2-δ-hydroxybutyl-3(2*H*)-pyridazinone | liq, anal, ir, nmr | 1700 |
| 3-Chloro-6-hydrazino-5-methylaminopyridazine | — | *H* 293, 582 |
| 3-Chloro-6-hydrazino-4-methylpyridazine | — | *H* 291, 660 |
| 3-Chloro-6-hydrazino-5-methylpyridazine | — | *H* 292, 660 |
| 4-Chloro-5-hydrazino-2-methyl-3(2*H*)-pyridazinone | $H_2C$=: 213–214; PhCH=:261–262 | 1054; *H* 119, 312 |
| 3-Chloro-6-hydrazino-4/5-morpholinopyridazine | 180, ir, nmr; EtMeC=: 145–147 | 544 |
| 3-Chloro-6-hydrazino-5-nitro-4-pyridazinamine | 188, ir, nmr | 1001 |
| 4-Chloro-5-hydrazino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 123, 315, 661 |
| 6-Chloro-4-hydrazino-2-phenyl-3(2*H*)-pyridazinone | $H_2C$=: –; $Me_2NCH$=: 206, ir, nmr; $Me_2NCH$=: HCl: 189, ir, nmr | 1429, 1471; *H* 104, 323, 661 |
| 5-Chloro-3-hydrazino-4-pyridazinamine | — | *H* 295, 561 |
| 6-Chloro-3-hydrazino-4-pyridazinamine | 210, nmr, uv; $Me_2C$=: 160–162 | 654; *H* 293, 561, 562 |
| 3-Chloro-6-hydrazinopyridazine | 138–139 or 140–141, dipole, ir, nmr, uv; PhCH=: 257–258, uv; $Me_2C$=: – | 284, 636, 1183, 1224; *H* 279, 280, 659, 666, 667 |
| 6-Chloro-3-hydrazino-4-pyridazinecarboxamide | 184 | 765; *H* 660 |
| 6-Chloro-3-hydrazino-4-pyridazinecarboxylic acid | 198–201, nmr | 1228 |
| 3-Chloro-6-hydrazinopyridazine 2-oxide | 180–181 or 185–186; $Me_2C$=: 198–200; EtMeC=: 133–134; etc. | 546, 891 |
| 4-Chloro-5-hydrazino-3(2*H*)-pyridazinone | MeCH=: 246–248 | 1054: *H* 118, 311, 661, 662, 663 |
| 6-Chloro-5-hydrazino-3(2*H*)-pyridazinone | $H_2C$=: 235–237 | 1054; *H* 87, 326 |
| 3-Chloro-6-hydrazono-1-methyl-1,6-dihydropyridazine | 90–92, dipole, nmr, uv; PhCH=: 108–110, uv | 1183 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3-Chloro-6-hydroxyaminopyridazine | 142–143, nmr | 191, 966 |
| 4-Chloro-2-δ-hydroxybutyl-5-methoxy-3(2*H*)-pyridazinone | 74–76, ir, nmr | 1700 |
| 4-Chloro-2-δ-hydroxybutyl-5-methylamino-3(2*H*)-pyridazinone | 148–149, ir, nmr | 1700 |
| 3-Chloro-5-α-hydroxyethyl-4-iodo-3-methoxypyridazine | 83–85, ir, nmr | 1610 |
| 3-Chloro-4-α-hydroxyethyl-6-methoxypyridazine | nmr | 1880 |
| 3-Chloro-5-α-hydroxyethyl-6-methoxypyridazine | 105–107, ir, nmr | 1610, 1880 |
| 4-Chloro-2-β-hydroxyethyl-5-morpholino-3(2*H*)-pyridazinone | 126–127, ir | 534 |
| 5-Chloro-2-β-hydroxyethyl-4-phenoxy-3(2*H*)-pyridazinone | — | 591 |
| 3-Chloro-4-α-hydroxyethyl-6-pivalamidopyridazine | 120, nmr | 1421 |
| 3-Chloro-5-α-hydroxyethyl-6-pivalamidopyridazine | 160, nmr | 1421 |
| 6-Chloro-2-β-hydroxyethyl-3(2*H*)-pyridazinone | — | *H* 73, 305, 402 |
| 4-Chloro-5-hydroxy-2-δ-hydroxybutyl-3(2*H*)-pyridazinone | 132–133, ir, nmr | 1700 |
| 4-Chloro-5-hydroxy-2-isopropyl-3(2*H*)-pyridazinone | 248–250, ir, nmr | 1044, 1936 |
| 6-Chloro-1-hydroxy-3-methoxy-4(1*H*)-pyridazinethione | 244–245, uv | 905 |
| 5-Chloro-2-hydroxy-6-methoxy-3(2*H*)-pyridazinone | — | *H* 89, 326 |
| 3-Chloro-5-hydroxymethyl-6-methoxypyridazine | — | *H* 181, 292 |
| 3-Chloro-4-hydroxymethyl-5-methyl-6-phenylpyridazine | 180 | 761 |
| 3-Chloro-4-hydroxymethyl-6-methylpyridazine | — | *H* 291, 397 |
| 4-Chloro-5-hydroxy-2-methyl-3(2*H*)-pyridazinone | 243 to 261, ir, nmr | 224, 1044, 1213, 1269, 1936 |
| 6-Chloro-2-hydroxymethyl-3(2*H*)-pyridazinone | — | *H* 71, 305, 402, 441 |
| 6-Chloro-4-hydroxy-2-methyl-3(2*H*)-pyridazinone | — | *H* 99 |
| 6-Chloro-5-hydroxy-2-methyl-3(2*H*)-pyridazinone | — | *H* 91, 326 |
| 4-Chloro-5-hydroxy-2-pentyl-3(2*H*)-pyridazinone | — | 1044 |
| 6-Chloro-5-hydroxy-2-phenyl-3,4(1*H*,2*H*)-pyridazinedione | — | *H* 169 |
| 4-Chloro-5-hydroxy-2-phenyl-3(2*H*)-pyridazinone | 273–274, nmr, $pK_a$ | 764, 1044, 1167, 1213; *H* 123, 315 |
| 3-Chloro-5-α-hydroxypropyl-6-methoxypyridazine | 79–81, ir, nmr | 1610 |
| 4-Chloro-5-hydroxy-2-propyl-3(2*H*)-pyridazinone | 201–203, ir, nmr | 1044, 1936 |
| 6-Chloro-2-hydroxy-3(2*H*)-pyridazinethione | 74–76, nmr | 679 |
| 4-Chloro-5-hydroxy-3(2*H*)-pyridazinone | 267–272, ir, nmr | 90, 224 |
| 6-Chloro-2-hydroxy-3(2*H*)-pyridazinone | — | *H* 76, 307 |
| 6-Chloro-5-hydroxy-3(2*H*)-pyridazinone | — | *H* 87, 291 |
| 6-Chloro-3-imino-2,3-dihydro-2-pyridazinamine | mesitylenesulfonate: 249–253, nmr | 412 |
| 6-Chloro-4-imino-1-methyl-1,4-dihydro-3(2*H*)-pyridazinone | — | *H* 303, 608 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 4-Chloro-6-iminomethyl-3-methoxypyridazine 1-oxide | — | *H* 197 |
| 3 / 4-Chloro-4 / 3-iodo-5-methoxypyridazine | l-MeI: 150–155 then 228–230 | 689 |
| 3-Chloro-4 + 5-iodo-6-methoxypyridazine | anal (mixture), nmr (each) | 1880 |
| 5/6-Chloro-6/5-iodo-2-methylpyridazinium-4-olate | 230–231 | 689 |
| 3-Chloro-6-iodopyridazine | crude: ms, nmr | 1638 |
| 3-Chloro-6-isobutoxypyridazine | — | 688; *H* 178 |
| 4-Chloro-5-isobutylamino-2-methyl-3(2*H*)-pyridazinone | 123–125, ir, uv | 148 |
| 5-Chloro-4-isobutylamino-2-methyl-3(2*H*)-pyridazinone | 85–87, ir, uv | 148 |
| 4-Chloro-5-isobutylamino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 121, 313 |
| 4-Chloro-5-isocyanato-2-phenyl-3(2*H*)-pyridazinone | — | *H* 316, 566 |
| 3-Chloro-6-isopentyloxypyridazine | — | 688; *H* 178, 280 |
| 4-Chloro-5-isopropoxy-2-phenyl-3(2*H*)-pyridazinone | 118, ir, nmr | 1089; *H* 123 |
| 5-Chloro-4-isopropoxy-2-phenyl-3(2*H*)-pyridazinone | 165 / 1.4, ir, nmr | 1089 |
| 3-Chloro-6-isopropoxypyridazine | — | *H* 178, 280 |
| 4-Chloro-5-isopropylamino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 121, 313 |
| 6-Chloro-4-isopropylamino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 588 |
| 3-Chloro-6-isopropylaminopyridazine | 103–105 or 114–116 | 1258, 1330; *H* 277, 573 |
| 6-Chloro-2-*N'*-isopropylhydrazinocarbonylmethyl-3(2*H*)-pyridazinone | — | *H* 78 |
| 4-Chloro-2-isopropyl-5-methoxy-3(2*H*)-pyridazinone | 109–110, ir, nmr | 1936 |
| 3-Chloro-6-(*N*-isopropyl-*N*-methylamino)pyridazine | — | 1352 |
| 3-Chloro-4-isopropyl-6-methylpyridazine | liq, anal, nmr | 1676; *H* 291 |
| 3-Chloro-5-isopropyl-6-methylpyridazine | liq, ms, nmr | 1676 |
| 3-Chloro-4-isopropyl-6-phenylpyridazine | 71–74, nmr | 1676 |
| 3-Chloro-5-isopropyl-6-phenylpyridazine | 65–68, nmr | 1676 |
| 4-Chloro-5-isopropyl-3-phenylpyridazine | 80–82, nmr | 1802 |
| 6-Chloro-4-isopropylthio-2-phenyl-3(2*H*)-pyridazinone | — | *H* 811 |
| 3-Chloro-6-isopropylthiopyridazine | — | *H* 284, 804 |
| 3-Chloro-6-isopropylthiopyridazine 1-oxide | — | *H* 299 |
| 4-Chloro-5-isothiocyanato-2-phenyl-3(2*H*)-pyridazinone | — | *H* 566 |
| 4-Chloro-5-mercapto-2-methyl-3(2*H*)-pyridazinone | — | *H* 802 |
| 4-Chloro-5-mercapto-2-phenyl-3(2*H*)-pyridazinone | — | *H* 123, 315, 802 |
| 6-Chloro-5-mercapto-2-phenyl-3(2*H*)-pyridazinone | — | *H* 802 |
| 4-Chloro-5-mercapto-3(2*H*)-pyridazinone | — | *H* 802 |
| 3-Chloro-6-methanesulfonylaminopyridazine | 145–146, ir, nmr | 89 |
| 3-Chloro-4-methoxycarbonylmethylpyridazine | liq, anal, ir, nmr | 947 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 6-Chloro-2-methoxycarbonylmethyl-3(2*H*)-pyridazinone | — | *H* 77, 305 |
| 6-Chloro-4-methoxycarbonylmethyl-3(2*H*)-pyridazinone | 150–152, ir, nmr, uv | 947 |
| 6-Chloro-5-methoxycarbonylmethyl-3(2*H*)-pyridazinone | 145–147, ir, nmr, uv | 947 |
| 4-Chloro-5-methoxy-3,6-dimethylpyridazine | 44–49, nmr; pic: 114–115 | 529 |
| 4-Chloro-5-methoxy-1,2-dimethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 163, 338 |
| 3-Chloro-6-methoxy-5-methylaminopyridazine | — | *H* 181, 293, 582 |
| 4-Chloro-3-methoxymethyl-6-methylpyridazine | 47–48, nmr | 446 |
| 3-Chloro-6-methoxy-4-methyl-5-nitropyridazine 2-oxide | 136–137, nmr | 902 |
| 3-Chloro-4-methoxy-6-methylpyridazine | — | *H* 292 |
| 3-Chloro-6-methoxy-4-methylpyridazine | nmr | 1569; *H* 183, 184, 291 |
| 3-Chloro-6-methoxy-5-methylpyridazine | 117–119, nmr | 1386, 1569; *H* 181, 292 |
| 4-Chloro-3-methoxy-6-methylpyridazine | — | *H* 181, 295 |
| 3-Chloro-6-methoxy-4-methylpyridazine 2-oxide | 151–152 | 833; *H* 197, 300 |
| 3-Chloro-6-methoxy-5-methylpyridazine 2-oxide | — | *H* 300 |
| 4-Chloro-3-methoxy-5-methylpyridazine 1-oxide | 156–157 | 833 |
| 4-Chloro-3-methoxy-6-methylpyridazine 1-oxide | — | *H* 197, 299, 301 |
| 5-Chloro-6-methoxy-2-methylpyridazinium-4-olate | 206–207 | 689 |
| 6-Chloro-5-methoxy-2-methylpyridazinium-4-olate | 221, uv | 699 |
| 3-Chloro-5-methoxy-1-methyl-4(1*H*)-pyridazinone | 235–237 | 689 |
| 3-Chloro-6-methoxy-1-methyl-4(1*H*)-pyridazinone | — | *H* 133, 333 |
| 4-Chloro-5-methoxy-2-methyl-3(2*H*)-pyridazinone | 153–155 or 159, ir, nmr | 90, 689, 1213, 1936; *H* 119 |
| 4-Chloro-6-methoxy-2-methyl-3(2*H*)-pyridazinone | — | 980, 997 |
| 5-Chloro-4-methoxy-2-methyl-3(2*H*)-pyridazinone | nmr | 1213 |
| 5-Chloro-6-methoxy-2-methyl-3(2*H*)-pyridazinone | — | 980, 997; *H* 91 |
| 6-Chloro-2-methoxymethyl-3(2*H*)-pyridazinone | — | *H* 71, 305 |
| 6-Chloro-4-methoxy-2-methyl-3(2*H*)-pyridazinone | — | *H* 99, 323 |
| 6-Chloro-5-methoxy-2-methyl-3(2*H*)-pyridazinone | — | *H* 91, 326 |
| 3-Chloro-6-methoxy-5-methylthiopyridazine 2-oxide | 133–135, uv | 905 |
| 3-Chloro-6-methoxy-5-morpholinopyridazine | — | *H* 184 |
| 6-Chloro-3-methoxy-5-nitro-4-pyridazinamine | — | *H* 561 |
| 3-Chloro-6-methoxy-5-nitro-4-pyridazinamine 2-oxide | 205–206, ir | 1072 |
| 3-Chloro-6-methoxy-4-nitropyridazine 1-oxide | — | *H* 197 |
| 3-Chloro-6-methoxy-5-nitropyridazine 2-oxide | 143–144, nmr | 450, 905; *H* 300, 649 |
| 3-Chloro-6-methoxy-5-nitro-4-styrylpyridazine 2-oxide | 187–188, ir, nmr | 902 |
| 4-Chloro-5-methoxy-2-phenyl-3(2*H*)-pyridazinone | 149–150, ir, nmr | 1089, 1213; *H* 123, 315 |
| 4-Chloro-6-methoxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 108, 322 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 5-Chloro-4-methoxy-2-phenyl-3(2*H*)-pyridazinone | nmr | 1213 |
| 5-Chloro-6-methoxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 94, 326 |
| 6-Chloro-4-methoxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 105, 323 |
| 6-Chloro-5-methoxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 93, 326 |
| 4-Chloro-5-methoxy-2-propyl-3(2*H*)-pyridazinone | 50–51, ir, nmr | 1936 |
| 3-Chloro-6-methoxy-4-pyridazinamine | 161–162 | 1099 |
| 5-Chloro-3-methoxy-4-pyridazinamine | — | *H* 295, 559 |
| 5-Chloro-6-methoxy-3-pyridazinamine | — | *H* 182, 295 |
| 5-Chloro-6-methoxy-4-pyridazinamine | — | *H* 183, 295, 559 |
| 6-Chloro-3-methoxy-4-pyridazinamine | 197 or 206, nmr, ur | 654, 1900; *H* 181, 293 559 |
| 5-Chloro-6-methoxy-3-pyridazinamine 2-oxide | nmr | 450; *H* 197, 301 |
| 5-Chloro-6-methoxy-4-pyridazinamine 2-oxide | — | *H* 604 |
| 6-Chloro-3-methoxy-4-pyridazinamine 1-oxide | 160–161 | 905 |
| 3-Chloro-4-methoxypyridazine | — | *H* 185, 290 |
| 3-Chloro-5-methoxypyridazine | 99–100, ir, nmr | 90, 1821 |
| 3-Chloro-6-methoxypyridazine | 89–90, dipole, nmr, uv | 450, 644, 1183, 1349, 1386, 1638; *H* 176, 280, 302, 303 |
| 5-Chloro-6-methoxy-3-pyridazinecarbaldehyde 2-oxide | — | *H* 197, 299 |
| 5-Chloro-6-methoxy-3-pyridazinecarbonitrile 2-oxide | — | *H* 197, 229, 446 |
| 3-Chloro-6-methoxy-4-pyridazinecarboxamide | 210–211 | 1099 |
| 6-Chloro-3-methoxy-4-pyridazinecarboxamide | 142–144 or 145–147 | 820, 1099 |
| 3-Chloro-6-methoxy-4-pyridazinecarboxylic acid | — | *H* 184, 436 |
| 6-Chloro-3-methoxy-4-pyridazinecarboxylic acid | — | *H* 181, 293 |
| 3-Chloro-6-methoxy-4,5-pyridazinediamine | — | *H* 599 |
| 3-Chloro-6-methoxypyridazine 1-oxide | — | *H* 195, 299 |
| 3-Chloro-6-methoxypyridazine 2-oxide | 159–160 or 160–161, nmr | 103, 449; *H* 195, 300 |
| 4-Chloro-3-methoxypyridazine 1-oxide | 120–121 | 833 |
| 3-Chloro-5-methoxy-4(1*H*)-pyridazinone | 248–249 | 224, 882 |
| 3-Chloro-6-methoxy-4(1*H*)-pyridazinone | 225–228 | 881; *H* 291 |
| 4-Chloro-5-methoxy-3(2*H*)-pyridazinone | 235 or 245–246, ir, nmr | 90, 689 |
| 4-Chloro-6-methoxy-3(2*H*)-pyridazinone | — | 997 |
| 5-Chloro-3-methoxy-4(1*H*)-pyridazinone | 240–241 | 224 |
| 5-Chloro-6-methoxy-3(2*H*)-pyridazinone | — | 997 |
| 5-Chloro-6-methoxy-4(1*H*)-pyridazinone | 188 | 882 |
| 6-Chloro-3-methoxy-4(1*H*)-pyridazinone | — | *H* 133, 293 |
| 6-Chloro-4-methoxy-3(2*H*)-pyridazinone | — | *H* 96, 293 |
| 6-Chloro-5-methoxy-3(2*H*)-pyridazinone | — | *H* 87, 291 |
| 3-Chloro-6-methoxy-5-trimethylsilylpyridazine | <35, nmr | 1610 |
| 3-Chloro-6-(*N*-methylacetamido)pyridazine | 123–124, nmr | 1085 |
| 3-Chloro-5-methylamino-6-α-methylhydrazinopyridazine | 172 | 1138 |

*continued*

| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 4-Chloro-5-methylamino-2-phenyl-3(2*H*)-pyridazinone | 213–215, ir, nmr, uv | 148, 1213; *H* 121, 313, 592 |
| 5-Chloro-4-methylamino-2-phenyl-3(2*H*)-pyridazinone | 115–117, ir, nmr, uv | 148, 1213 |
| 6-Chloro-4-methylamino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 104, 323, 588 |
| 5-Chloro-3-methylamino-4-pyridazinamine | 203–204, ir, nmr, uv | 908 |
| 6-Chloro-3-methylamino-4-pyridazinamine | — | *H* 293, 597 |
| 6-Chloro-4-methylamino-3-pyridazinamine | — | *H* 293, 597 |
| 6-Chloro-5-methylamino-3-pyridazinamine | 190–191, nmr | 447 |
| 3-Chloro-5-methylaminopyridazine | — | *H* 289, 582 |
| 3-Chloro-6-methylaminopyridazine | 195–196 | 1136, 1330; *H* 276, 303, 572 |
| 6-Chloro-3-methylamino-4-pyridazinecarbonitrile | 259–260, uv | 820 |
| 6-Chloro-3-methylamino-4-pyridazinecarboxamide | 209–210, uv | 820 |
| 4-Chloro-5-methylamino-3(2*H*)-pyridazinone | — | *H* 116, 310, 582 |
| 6-Chloro-2-(methylcarbamoyl)methyl-3(2*H*)-pyridazinone | — | *H* 78, 306 |
| 1-Chloromethyl-4,5-dimethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 152 |
| 4-Chloro-6-methyl-3,5-diphenylpyridazine | 127–128, ir, nmr | 311 |
| 3-Chloro-6-methyl-5,*N*-diphenyl-4-pyridazinecarboxamide 1-oxide | — | 2054 |
| 2-Chloromethyl-6-ethyl-3(2*H*)-pyridazinone | 74–76 | 441 |
| 4-Chloro-5-α-methylhydrazino-2-phenyl-3(2*H*)-pyridazinone | 153–154, ir, nmr; PhCH=: 181–182, ir, nmr | 873, 1054 |
| 6-Chloro-4-α-methylhydrazino-2-phenyl-3(2*H*)-pyridazinone | –; $H_2C$=: – | 1471 |
| 5-Chloro-3-α-methylhydrazino-4-pyridazinamine | 137–139, ir, nmr, uv | 908 |
| 6-Chloro-3-α-methylhydrazino-4-pyridazinamine | — | *H* 562 |
| 3-Chloro-6-α-methylhydrazinopyridazine | 108–109. dipole, ir, nmr, pol, uv; PhCH=: 131, uv | 1183, 1983 |
| 4-Chloro-5-α-methylhydrazino-3(2*H*)-pyridazinone | 174–175, ir, nmr; $H_2C$=: 243–245, ir, nmr; MeCH=: 205–206, ir, nmr; PhCH: 199–200, ir, nmr | 1054 |
| 5-Chloromethyl-6-methoxy-3(2*H*)-pyridazinone | — | *H* 89 |
| 4-Chloro-6-methyl-3-methylaminopyridazine 1-oxide | 168–169 | 760 |
| 4-Chloro-2-methyl-5-methylamino-3(2*H*)-pyridazinone | 154–155 or 161–162, ir, nmr, uv | 148, 1213, 1677 |
| 5-Chloro-2-methyl-4-methylamino-3(2*H*)-pyridazinone | 134–135 or 142, ir, nmr, uv | 148, 1213, 1677 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 4-Chloro-2-methyl-5-α-methylhydrazino-3(2*H*)-pyridazinone | 131–133, ir, nmr; $H_2C$=: 137–138, ir, nmr; PhCH=: 119–120, ir, nmr | 1054 |
| 5-Chloro-2-methyl-4-α-methylhydrazino-3(2*H*)-pyridazinone | 52–53, ir,nmr | 1685 |
| 3-Chloro-1-methyl-6-methylimino-1,6-dihydropyridazine | — | *H* 302 |
| 3-Chloro-2-methyl-5-methylimino-6-phenyl-2,5-dihydropyridazine | — | *H* 608 |
| 3-Chloromethyl-6-methyl-4-nitropyridazine 1-oxide | 94–95, ir, nmr | 796 |
| 2-Chloromethyl-6-methyl-3(2*H*)-pyridazinone | 88–89 or 94–95, ir, nmr | 441, 789; *H* 70 |
| 6-Chloromethyl-3-methyl-4(1*H*)-pyridazinone | — | *H* 132 |
| 4-Chloro-2-methyl-5-methylsulfonyl-3(2*H*)-pyridazinone | 175–177, ir, nmr | 533 |
| 3-Chloro-4-methyl-6-methylthiopyridazine | — | *H* 806 |
| 3-Chloro-5-methyl-6-methylthiopyridazine | — | *H* 806 |
| 4-Chloro-2-methyl-5-methylthio-3(2*H*)-pyridazinone | 124–126, ir, nmr | 533, 1213 |
| 5-Chloro-2-methyl-4-methylthio-3(2*H*)-pyridazinone | nmr | 1213 |
| 3-Chloro-4-methyl-6-morpholinopyridazine | 149–151 | 544 |
| 4-Chloro-2-methyl-5-morpholino-3(2*H*)-pyridazinone | 133–134 or 134–135, ir, nmr | 542, 1269; *H* 119, 312, 591 |
| 5-Chloro-2-methyl-4-morpholino-3(2*H*)-pyridazinone | 101–102 | 1269 |
| 6-Chloro-2-methyl-5-morpholino-3(2*H*)-pyridazinone | 127–128 | 1269 |
| 3-Chloro-6-methyl-5-nitro-4-pyridazinamine | — | *H* 561 |
| 3-Chloro-6-methyl-4-nitropyridazine 1-oxide | 103, nmr | 904, 952; *H* 299, 301, 649 |
| 3-Chloro-6-methyl-5-nitropyridazine 1-oxide | 81–82, ir, nmr | 904 |
| 4-Chloro-2-methyl-5-nitro-3(2*H*)-pyridazinone | — | *H* 119, 648 |
| 4-Chloro-2-methyl-6-nitro-3(2*H*)-pyridazinone | — | *H* 99 |
| 6-Chloro-2-methyl-4-nitro-3(2*H*)-pyridazinone | — | *H* 99, 325 |
| 4-Chloro-1-methyl-6-oxo-1,6-dihydro-3-pyridazinecarboxylic acid | — | *H* 91, 326, 438 |
| 3-Chloro-4-methyl-6-phenoxypyridazine | — | *H* 184 |
| 5-Chloro-2-methyl-4-phenoxy-3(2*H*)-pyridazinone | — | 591 |
| 3-Chloro-4-methyl-6-phenylpyridazine | 139 to 145, nmr | 152, 761, 762, 961, 1104, 1514, 1632 |
| 3-Chloro-5-methyl-6-phenylpyridazine | 123–124 | 1557 |
| 3-Chloro-6-methyl-4-phenylpyridazine | — | *H* 291, 302, 303 |
| 4-Chloro-1-methyl-2-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 159, 338 |
| 4-Chloro-2-methyl-1-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 159, 338 |
| 3-Chloro-4-methyl-6-phenylpyridazine 2-oxide | 150, nmr | 1104 |
| 2-Chloromethyl-6-phenyl-3(2*H*)-pyridazinone | 132–133 | 441 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 4-Chloro-6-methyl-2-phenyl-3(2*H*)-pyridazinone | — | *H* 105, 322 |
| 6-Chloro-4-methyl-2-phenyl-3(2*H*)-pyridazinone | — | *H* 104, 323, 328 |
| 6-Chloro-5-methyl-2-phenyl-3(2*H*)-pyridazinone | — | *H* 93, 326, 328 |
| 3-Chloro-4-methyl-6-piperidinopyridazine | 101–102 | 544 |
| 4-Chloro-2-methyl-5-piperidino-3(2*H*)-pyridazinone | 57–59, ir, uv | 148; *H* 119, 312, 591 |
| 5-Chloro-2-methyl-4-piperidino-3(2*H*)-pyridazinone | 133/136, ir, uv | 148 |
| 3-Chloro-6-methyl-4-pyridazinamine | — | *H* 558 |
| 6-Chloro-4-methyl-3-pyridazinamine | — | *H* 292, 554 |
| 6-Chloro-5-methyl-3-pyridazinamine | 185–187 or 190–191, nmr | 116, 1145; *H* 290, 554 |
| 3-Chloro-6-methyl-5-pyridazinamine 1-oxide | 210–212, ir, nmr | 904 |
| 3-Chloro-4-methylpyridazine | — | *H* 289 |
| 3-Chloro-5-methylpyridazine | — | *H* 289 |
| 3-Chloro-6-methylpyridazine | nmr, $pK_a$; 2-MeCl: 133–134 | 94, 115, 1638; *H* 276, 301, 302, 303 |
| 4-Chloromethylpyridazine | HCl: 119–120, ir, ms, nmr | 66 |
| 5-Chloro-6-methyl-3-pyridazinecarbaldehyde 2-oxide | — | *H* 299, 398 |
| 3-Chloro-6-methyl-4-pyridazinecarbohydrazide | — | *H* 291, 436 |
| 3-Chloro-6-methyl-4-pyridazinecarbonitrile | — | 447; *H* 291 |
| 4-Chloro-6-methyl-3-pyridazinecarbonitrile 2-oxide | — | *H* 300, 446 |
| 5-Chloro-6-methyl-3-pyridazinecarbonitrile 2-oxide | — | *H* 299, 446 |
| 3-Chloro-6-methyl-4-pyridazinecarboxamide | — | *H* 291, 436 |
| 3-Chloro-6-methyl-4-pyridazinecarboxylic acid | — | *H* 291 |
| 5-Chloro-6-methyl-3-pyridazinecarboxylic acid 2-oxide | — | *H* 299, 435 |
| 3-Chloro-6-methyl-4,5-pyridazinediamine | — | *H* 599 |
| 1-Chloromethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 152 |
| 4-Chloro-1-methyl-3,6(1*H*,2*H*)-pyridazinedione | 262–263, $pK_a$, uv | 675, 980, 997, 1129; *H* 152, 337 |
| 4-Chloro-2-methyl-3,6(1*H*,2*H*)-pyridazinedione | 189–190 or 193–196, $pK_a$, uv | 675, 980, 997, 1129; *H* 152, 337 |
| 5-Chloro-2-methyl-3,4(1*H*,2*H*)-pyridazinedione | 216, nmr | 224, 1213 |
| 6-Chloro-2-methyl-3,4(1*H*,2*H*)-pyridazinedione | — | *H* 323 |
| 3-Chloro-3-methylpyridazine 1-oxide | — | *H* 299 |
| 3-Chloro-5-methylpyridazine 1-oxide | — | *H* 299 |
| 3-Chloro-6-methylpyridazine 1-oxide | 163–164 | 103, 913; *H* 299, 301 |
| 4-Chloro-3-methylpyridazine 1-oxide | — | *H* 299 |
| 4-Chloro-5-methylpyridazine 1-oxide | — | *H* 299 |
| 4-Chloro-6-methylpyridazine 1-oxide | — | *H* 299, 301 |
| 6-Chloro-4-methyl-3(2*H*)-pyridazinethione | — | *H* 798 |
| 6-Chloro-5-methyl-3(2*H*)-pyridazinethione | — | *H* 798 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 6-Chloro-2-methyl-3(2*H*)-pyridazinimine | — | *H* 302 |
| 2-Chloromethyl-3(2*H*)-pyridazinone | 67–68 | 441 |
| 3-Chloro-6-methyl-4(1*H*)-pyridazinone | — | *H* 292 |
| 4-Chloro-2-methyl-3(2*H*)-pyridazinone | 82–84, ir, nmr | 533; *H* 315 |
| 5-Chloro-2-methyl-3(2*H*)-pyridazinone | nmr | 1213; *H* 91, 326 |
| 6-Chloro-2-methyl-3(2*H*)-pyridazinone | 89 or 90–92, dipole, nmr, uv | 789, 1183; *H* 70, 305 |
| 6-Chloro-4-methyl-3(2*H*)-pyridazinone | 147 to 171, ir, nmr, uv | 30, 755, 821, 947; *H* 96, 292, 322, 328 |
| 6-Chloro-5-methyl-3(2*H*)-pyridazinone | 219 to 232, nmr, uv | 30, 821, 947; *H* 87, 291, 326, 328 |
| 4-Chloro-5-methyl-3(2*H*)-pyridazinone 1-oxide | 232 | 833 |
| 4-Chloro-6-methyl-3(2*H*)-pyridazinone 1-oxide | 219 or 227–228 | 760, 833 |
| 6-Chloro-4-methyl-3(2*H*)-pyridazinone 1-oxide | 204 | 833 |
| 6-Chloro-5-methyl-3(2*H*)-pyridazinone 1-oxide | 230 | 833 |
| 6-Chloro-4-methylsulfinyl-2-phenyl-3(2*H*)-pyridazinone | — | *H* 325, 823 |
| 6-Chloro-5-methylsulfinyl-2-phenyl-3(2*H*)-pyridazinone | — | *H* 327, 823 |
| 3-Chloro-6-methylsulfinylpyridazine | 74–79, ir, nmr | 304; *H* 282, 822 |
| 3-Chloro-6-methylsulfinylpyridazine 1-oxide | 131–132, nmr | 304, cf. 679 |
| 4-Chloro-5-methylsulfonyl-2-phenyl-3(2*H*)-pyridazinone | — | *H* 124, 315, 824 |
| 5-Chloro-4-methylsulfonyl-2-phenyl-3(2*H*)-pyridazinone | — | *H* 824 |
| 5-Chloro-6-methylsulfonyl-2-phenyl-3(2*H*)-pyridazinone | — | *H* 824 |
| 6-Chloro-4-methylsulfonyl-2-phenyl-3(2*H*)-pyridazinone | — | *H* 105, 323, 824 |
| 6-Chloro-5-methylsulfonyl-2-phenyl-3(2*H*)-pyridazinone | — | *H* 824 |
| 3-Chloro-6-methylsulfonylpyridazine | 122–123, nmr | 304; *H* 283, 823 |
| 3-Chloro-6-methylsulfonylpyridazine 1-oxide | 152 or 155–157, nmr | 679, cf. 304 |
| 4-Chloro-5-methylthio-2-phenyl-3(2*H*)-pyridazinone | nmr | 1213; *H* 123, 315, 812 |
| 6-Chloro-4-methylthio-2-phenyl-3(2*H*)-pyridazinone | — | *H* 105, 323, 810 |
| 6-Chloro-5-methylthio-2-phenyl-3(2*H*)-pyridazinone | — | *H* 812 |
| 6-Chloro-3-methylthio-4-pyridazinamine | 213–214, nmr | 654 |
| 3-Chloro-6-methylthiopyridazine | 96–99 or 102 | 111, 1135; *H* 282, 803 |
| 3-Chloro-6-methylthiopyridazine 1-oxide | 192–193 or 194–197, nmr | 679, cf. 304; *H* 299 |
| 3-Chloro-6-methylthiopyridazine 2-oxide | 169–172, nmr | 679, cf. 304 |
| 6-Chloro-4-methylthio-3(2*H*)-pyridazinone | — | *H* 96, 293, 810 |
| 5-Chloro-2-methyl-4-thioxo-1,4-dihydro-3(2*H*)-pyridazinone | — | *H* 801 |
| 6-Chloro-2-methyl-4-ureido-3(2*H*)-pyridazinone | 295, nmr | 84 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3-Chloro-5-morpholinomethyl-6-phenylpyridazine | crude: solid | 1339 |
| 3-Chloro-4-morpholinomethylpyridazine | 102–104, nmr; oxalate: 121–123 | 769 |
| 3-Chloro-6-morpholinomethylpyridazine | 115–116 | 769 |
| 6-Chloro-2-morpholinomethyl-3(2*H*)-pyridazinethione | — | *H* 798 |
| 6-Chloro-2-morpholinomethyl-3(2*H*)-pyridazinone | — | *H* 71, 305 |
| 6-Chloro-4-morpholinomethyl-3(2*H*)-pyridazinone 1-oxide | — | *H* 197, 300, 610 |
| 3-Chloro-4-morpholino-6-phenylpyridazine | 148–150 | 1041 |
| 4-Chloro-5-morpholino-2-phenyl-3(2*H*)-pyridazinone | — | 687; *H* 122, 313, 592 |
| 6-Chloro-4-morpholino-2-phenyl-3(2*H*)-pyridazinone | — | 878; *H* 105, 323, 590 |
| 6-Chloro-5-morpholino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 93, 327, 593 |
| 4-Chloro-5-morpholino-2-prop-1′-enyl-3(2*H*)-pyridazinone | 111–112 | 1269 |
| 3-Chloro-5-morpholinopyridazine | — | *H* 586 |
| 3-Chloro-6-morpholinopyridazine | — | *H* 279, 574 |
| 3-Chloro-6-morpholino-4-pyridazinecarbonitrile | 195–196, uv | 820 |
| 6-Chloro-3-morpholino-4-pyridazinecarbonitrile | 134–135, uv | 820 |
| 3-Chloro-6-morpholino-4-pyridazinecarboxamide | 255–257 | 820 |
| 6-Chloro-3-morpholino-4-pyridazinecarboxamide | 172–175 | 820 |
| 3-Chloro-5-morpholino-4(1*H*)-pyridazinone | 244–245 | 224 |
| 4-Chloro-5-morpholino-3(2*H*)-pyridazinone | 223 to 232, ir, nmr | 224, 542, 641; *H* 117, 311, 586 |
| 5-Chloro-6-morpholino-4(1*H*)-pyridazinone | 250 | 224 |
| 3-Chloro-6-nitroaminopyridazine | — | *H* 279, 607 |
| 4-Chloro-5-nitro-2-phenyl-3(2*H*)-pyridazinone | — | *H* 123, 648 |
| 6-Chloro-4-nitro-3-pyridazinamine 1-oxide | 221–222, nmr | 449, 450 |
| 6-Chloro-4-nitro-3,5-pyridazinediamine | — | *H* 599 |
| 6-Chloro-5-nitro-3,4-pyridazinediamine | — | *H* 599 |
| 6-Chloro-4-nitro-3,5-pyridazinediamine 1-oxide | 285-287 | 449 |
| 6-Chloro-4-nitro-3(2*H*)-pyridazinone 1-oxide | nmr | 450; *H* 197, 300, 649 |
| 6-Chloro-3-oxo-2,3-dihydro-4-pyridazinecarbohydrazide | — | *H* 96, 322, 440 |
| 5-Chloro-6-oxo-1,6-dihydro-3-pyridazinecarbonitrile 2-oxide | — | *H* 196, 299 |
| 3-Chloro-6-oxo-1,6-dihydro-4-pyridazinecarboxamide | — | *H* 87, 291, 439 |
| 6-Chloro-3-oxo-2,3-dihydro-4-pyridazinecarboxamide | 253–254 | 1099; *H* 96, 322, 440 |
| 3-Chloro-6-oxo-1,6-dihydro-4-pyridazinecarboxylic acid | — | *H* 87, 291, 326, 439 |
| 6-Chloro-3-oxo-2,3-dihydro-4-pyridazinecarboxylic acid | 213–215 | 820; *H* 96, 292, 322, 440 |
| 5-Chloro-6-oxo-1,6-dihydro-3-pyridazinecarboxylic acid 2-oxide | — | *H* 196, 299, 438 |
| 3-Chloro-6-pentyloxypyridazine | — | 688; *H* 178, 280 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 5-Chloro-6-pentyloxy-4-pyridazinecarbaldehyde 2-oxide | — | *H* 198, 300, 398 |
| 3-Chloro-4-phenyl-6-phenylpyridazine | 40–41, nmr | 1632 |
| 3-Chloro-6-pentylpyridazine | — | *H* 276 |
| 3-Chloro-6-pent-4′-ynyloxypyridazine | 71–73, ir, ms, nmr | 336 |
| 4-Chloro-5-phenethylamino-3(2*H*)-pyridazinone | — | *H* 116, 310, 584 |
| 3-Chloro-4-phenethyl-6-phenylpyridazine | 104, ms, nmr | 1959 |
| 4-Chloro-5-phenoxy-2-phenyl-3(2*H*)-pyridazinone | 99–100, ir, nmr | 1089 |
| 5-Chloro-4-phenoxy-2-phenyl-3(2*H*)-pyridazinone | 101–102, ir, nmr | 591 |
| 3-Chloro-6-phenoxypyridazine | — | *H* 179, 281 |
| 5-Chloro-4-phenoxy-3(2*H*)-pyridazinone | — | 591 |
| 3-Chloro-6-phenylazopyridazine | 119–120, ir, nmr | 854 |
| 3-Chloro-6-phenylethynylpyridazine | 107–108, ir, nmr | 834 |
| 3-Chloro-6-$\beta$-phenylhydrazinopyridazine | 153–154, ir, nmr | 854 |
| 6-Chloro-2-phenyl-4-$\beta$-phenylhydrazino-3(2*H*)-pyridazinone | — | 1471 |
| 4-Chloro-2-phenyl-5-phenylsulfonyl-3(2*H*)-pyridazinone | anal, ir, ms | 1774 |
| 4-Chloro-2-phenyl-5-phenylthio-3(2*H*)-pyridazinone | 114–116, or 122, ir, nmr | 1089, 1774; *H* 123, 315, 812 |
| 6-Chloro-2-phenyl-4-phenylthio-3(2*H*)-pyridazinone | — | 1409 |
| 6-Chloro-2-phenyl-5-phenylthio-3(2*H*)-pyridazinone | — | 1409 |
| 4-Chloro-2-phenyl-5-piperidino-3(2*H*)-pyridazinone | — | 687; *H* 122, 313, 592 |
| 6-Chloro-2-phenyl-4-piperidino-3(2*H*)-pyridazinone | — | *H* 589 |
| 6-Chloro-2-phenyl-5-piperidino-3(2*H*)-pyridazinone | — | *H* 93, 327, 593 |
| 4-Chloro-2-phenyl-5-propionamido-3(2*H*)-pyridazinone | — | *H* 121, 313 |
| 4-Chloro-2-phenyl-5-propoxy-3(2*H*)-pyridazinone | — | *H* 123 |
| 4-Chloro-2-phenyl-5-propylamino-3(2*H*)-pyridazinone | — | *H* 121, 313, 591 |
| 6-Chloro-2-phenyl-4-propylamino-3(2*H*)-pyridazinone | — | *H* 104 |
| 6-Chloro-2-phenyl-4-propylthio-3(2*H*)-pyridazinone | — | *H* 811 |
| 6-Chloro-2-phenyl-5-propylthio-3(2*H*)-pyridazinone | — | *H* 812 |
| 5-Chloro-6-phenyl-3-pyridazinamine | 156–157, ir, nmr; HCl: 182–186, ir, nmr | 133 |
| 6-Chloro-3-phenyl-4-pyridazinamine | 133–135, ir, nmr | 133 |
| 3-Chloro-4-phenylpyridazine | 97–109 or 110–112, nmr | 1186, 1408 |
| 3-Chloro-5-phenylpyridazine | 181, nmr | 2069 |
| 3-Chloro-6-phenylpyridazine | 159–160 or 162–163, nmr, p$K_a$ | 59, 94, 936, 1114, 1145, 1222, 1569, 2069; *H* 276, 302, 303 |
| 3-Chloro-6-phenyl-4-pyridazinecarbonitrile | 206, ir, nmr | 1319 |
| 3-Chloro-6-phenyl-4-pyridazinecarboxamide | 204, ir, nmr | 1319, 2040 |
| 4-Chloro-1-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 156, 337 |

*continued*

| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 4-Chloro-2-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 155, 337 |
| 5-Chloro-2-phenyl-3,4(1*H*,2*H*)-pyridazinedione | nmr | 1213 |
| 6-Chloro-2-phenyl-3,4(1*H*,2*H*)-pyridazinedione | — | *H* 169, 323 |
| 3-Chloro-6-phenylpyridazine 1-oxide | — | *H* 301 |
| 3-Chloro-6-phenylpyridazine 2-oxide | — | *H* 300 |
| 6-Chloro-2-phenyl-3(2*H*)-pyridazinethione | — | *H* 333, 798 |
| 3-Chloro-6-phenyl-4(1*H*)-pyridazine | 233–234, or 283–284, ir, nmr | 1041 |
| 4-Chloro-2-phenyl-3(2*H*)-pyridazinone | — | *H* 84, 315 |
| 4-Chloro-6-phenyl-3(2*H*)-pyridazinone | 220–221, ir, nmr | 1041 |
| 5-Chloro-2-phenyl-3(2*H*)-pyridazinone | — | *H* 85, 92, 326 |
| 5-Chloro-6-phenyl-3(2*H*)-pyridazinone | 230–231, ir, nmr | 941, 1588; *H* 87, 326 |
| 6-Chloro-2-phenyl-3(2*H*)-pyridazinone | — | 878; *H* 80, 306 |
| 6-Chloro-3-phenyl-4(1*H*)-pyridazinone | 214–216 | 1087 |
| 5-Chloro-2-phenyl-6-thiocyanato-3(2*H*)-pyridazinone | — | *H* 826 |
| 6-Chloro-2-phenyl-4-thiocyanato-3(2*H*)-pyridazinone | — | *H* 826 |
| 6-Chloro-2-phenyl-5-thiocyanato-3(2*H*)-pyridazinone | — | *H* 826 |
| 3-Chloro-6-phenylthiopyridazine | — | *H* 284, 807 |
| 3-Chloro-6-phenylthiopyridazine 1-oxide | — | *H* 299 |
| 4-Chloro-5-phenylthio-3(2*H*-pyridazinone | — | *H* 118, 312, 812 |
| 5-Chloro-2-phenyl-4-thioxo-1,4-dihydro-3(2*H*)-pyridazinone | — | *H* 329 |
| 6-Chloro-2-phenyl-4-thioxo-1,4-dihydro-3(2*H*)-pyridazinone | — | *H* 105, 323, 801 |
| 3-Chloro-4-piperidinomethylpyridazine | 58–60, 150/7, nmr; oxalate: 139 | 769 |
| 6-Chloro-2-piperidinomethyl-3(2*H*)-pyridazinone | — | *H* 798 |
| 6-Chloro-2-piperidinomethyl-3(2*H*)-pyridazinone | — | *H* 71, 305 |
| 6-Chloro-4-piperidinomethyl-3(2*H*)-pyridazinone 1-oxide | — | *H* 196, 610 |
| 3-Chloro-6-piperidinopyridazine | — | *H* 278, 578 |
| 3-Chloro-6-piperidino-4-pyridazinecarbonitrile | 126–127, uv | 820 |
| 6-Chloro-3-piperidino-4-pyridazinecarbonitrile | 57–59, uv | 820 |
| 6-Chloro-3-piperidino-4-pyridazinecarboxamide | 144–145 | 820 |
| 3-Chloro-6-piperidinopyridazine 2-oxide | — | *H* 300, 604 |
| 4-Chloro-5-piperidino-3(2*H*)-pyridazinone | — | *H* 116, 311, 586 |
| 3-Chloro-6-pivalamidopyridazine | 159, ir, nmr | 1421 |
| 6-Chloro-2-propoxycarbonylmethyl-3(2*H*)-pyridazinone | — | *H* 78, 305 |
| 5-Chloro-6-propoxy-3-pyridazinamine 2-oxide | — | *H* 198, 301 |
| 3-Chloro-6-propoxypyridazine | — | *H* 178, 280 |
| 3-Chloro-6-propoxypyridazine 2-oxide | — | *H* 300 |
| 2-γ-Chloropropyl-6-methyl-3(2*H*)-pyridazinone | liq, ir, nmr, uv | 237 |
| 2-γ-Chloropropyl-6-phenyl-3(2*H*)-pyridazinone | liq, ir, nmr, uv | 237 |
| 6-Chloro-*N*-propyl-3-pyridazinesulfonamide | — | *H* 284, 823 |
| 2-γ-Chloropropyl-3(2*H*)-pyridazinone | liq, ir, nmr, uv | 237 |
| 3-Chloro-4-pyridazinamine | 140–142, nmr | 447; *H* 290, 558 |
| 5-Chloro-4-pyridazinamine | 120–122 | 675; *H* 295, 558 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 6-Chloro-3-pyridazinamine | 209 to 236, nmr, $pK_a$, uv | 98, 116, 450, 675, 1176, 1283; *H* 276, 303, 551 |
| 6-Chloro-4-pyridazinamine | 161–162, nmr | 654; *H* 289, 558 |
| 6-Chloro-3-pyridazinamine 1-oxide | 225–226 | 891 |
| 6-Chloro-3-pyridazinamine 2-oxide | nmr | 450; *H* 299, 301, 603 |
| 6-Chloro-4-pyridazinamine 2-oxide | — | *H* 604 |
| 3-Chloropyridazine | nmr, $pK_a$ | 94, 450; *H* 275 |
| 4-Chloropyridazine | — | *H* 275 |
| 5-Chloro-3-pyridazinecarbaldehyde 2-oxide | — | *H* 299, 398 |
| 6-Chloro-3-pyridazinecarbohydrazide | 254–255, ir, ms | 1459 |
| 3-Chloro-4-pyridazinecarbonitrile | 41–43 | 820; *H* 289, 447 |
| 6-Chloro-3-pyridazinecarbonitrile | crude: 85–88 | 1338; *H* 276, 446 |
| 5-Chloro-3-pyridazinecarbonitrile 2-oxide | — | *H* 299, 446 |
| 3-Chloro-4-pyridazinecarboxamide | — | 1684; *H* 290, 435 |
| 6-Chloro-3-pyridazinecarboxamide | 243–244, ir, nmr | 1592, 1773; *H* 276, 434 |
| 6-Chloro-3-pyridazinecarboxylic acid | nmr | 1638; *H* 276, 434 |
| 3-Chloro-4,5-pyridazinediamine | 197–198, nmr, uv | 973; *H* 599 |
| 4-Chloro-3,5-pyridazinediamine | — | *H* 598 |
| 5-Chloro-3,4-pyridazinediamine | — | *H* 295, 597 |
| 6-Chloro-3,4-pyridazinediamine | $pK_a$, uv | 675; *H* 293, 597 |
| 4-Chloro-3,6(1*H*,2*H*)-pyridazinedione | $pK_a$, uv | 675, 802; *H* 150, 337 |
| 6-Chloro-3,4(1*H*,2*H*)-pyridazinedione | 111–112, ir, nmr, uv | 665; *H* 169, 293 |
| 3-Chloropyridazine 1-oxide | 93–94, nmr | 103, 450; *H* 299 |
| 3-Chloropyridazine 2-oxide | nmr | 450; *H* 300 |
| 4-Chloropyridazine 1-oxide | — | *H* 299 |
| 4-Chloropyridazine 2-oxide | — | *H* 300 |
| 6-Chloro-3-pyridazinesulfonic acid | — | *H* 284, 825 |
| 6-Chloro-3-sulfonohydrazide | — | *H* 284, 823 |
| 6-Chloro-3-pyridazinesulfonyl chloride | — | *H* 284, 825 |
| 6-Chloro-3(2*H*)-pyridazinethione | — | 315, 1724; *H* 282, 797 |
| 6-Chloro-3,4,5-pyridazinetriamine | — | *H* 601 |
| 4-Chloro-3(2*H*)-pyridazinone | — | *H* 84, 96, 322 |
| 6-Chloro-3(2*H*)-pyridazinone | 136 to 148, dipole, nmr, pol, uv | 94, 98, 644, 755, 1116, 1167, 1169, 1183, 1638, 1983; *H* 66, 280 |
| 4-Chloro-3(2*H*)-pyridazinone 1-oxide | 212–213 | 833; *H* 299 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 6-Chloro-3(2*H*)-pyridazinone 1-oxide | — | *H* 195, 300 |
| 3-Chloro-6-styrylpyridazine | 176 or 180–182, ir, ms, nmr | 26, 1282 |
| 6-Chloro-2-β-thiocyanatoethyl-3(2*H*)-pyridazinone | — | *H* 73, 305, 826 |
| 6-Chloro-2-thiocyanatomethyl-3(2*H*)-pyridazinone | — | *H* 71, 305, 826 |
| 3-Chloro-6-thiocyanatopyridazine | — | *H* 283, 825 |
| 5-Chloro-4-thioxo-1,4-dihydro-3(2*H*)-pyridazinone | — | *H* 801 |
| 3-Chloro-4,5,6-trifluoropyridazine | nmr | 459 |
| 4-Chloro-3,5,6-trifluoropyridazine | 155/760, $n_D^{20°}$ 1.4562, nmr | 459 |
| 3-Chloro-4,5,6-trimethoxypyridazine | — | *H* 184, 294 |
| 3-Chloro-6-trimethylammoniopyridazine ( + anion) | Cl; nmr | 1796 |
| 3-Chloro-4,5,6-trimethylpyridazine | 93–95, nmr | 762 |
| 4-Chloro-3,5,6-trimethylpyridazine | 76–77 | 105, cf. 1793 |
| 3-Chloro-6-trimethylsiloxypyridazine | 74–76, 85–87/1, nmr, uv | 1172 |
| 4-Chloro-3-trimethylsiloxy-5-trimethylsilylaminopyridazine | 120–123, 133–135/1, uv | 1172 |
| 3-Chloro-6-trimethylsilylaminopyridazine | 127–130, 143–144/1, ir, nmr | 1172 |
| 3-Chloro-4,5,6-triphenylpyridazine | — | 1426 |
| 3-Chloro-4,5,6-trismethylthiopyridazine | — | *H* 294, 814 |
| 3-Chloro-5-ureido-4(1*H*)-pyridazinone | 270, nmr | 83 |
| 6-Chloro-4-ureido-3(2*H*)-pyridazinone | 295, nmr | 84 |
| 2-δ-Cyanobutyl-6-phenyl-3(2*H*)-pyridazinone | 196–203/0.7, nmr | 118 |
| 4-Cyano-6-ethoxycarbonyl-5-methyl-1-phenylpyridazinium-3-olate | 90, ir, nmr | 1645 |
| 4-Cyano-6-ethoxycarbonyl-1-phenyl-5-styrylpyridazinium-3-olate | 260–263, ir, nmr | 1645 |
| 2-β-Cyanoethyl-4-ethoxy-5-morpholino-3(2*H*)-pyridazinone | anal, ir, nmr | 534 |
| 2-β-Cyanoethyl-3-methoxy-6-methylthiopyridazine | — | 2024 |
| 2-β-Cyanoethyl-6-methyl-3(2*H*)-pyridazinone | 96–97, nmr | 118, 1288 |
| 5-Cyano-4-ethyl-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxamide | 187–189, ir, nmr | 1808 |
| 5-Cyano-4-ethyl-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylic acid | 230–231, ir, nmr | 1808 |
| 2-β-Cyanoethyl-6-phenyl-3(2*H*)-pyridazinethione | 127–128, ir, nmr | 1288 |
| 2-β-Cyanoethyl-6-phenyl-3(2*H*)-pyridazinone | 103–104, nmr | 118, 1288; *H* 71 |
| 4-β-Cyanoethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 155 |
| 2-β-Cyanoethyl-3(2*H*)-pyridazinethione | 63–64, ir, nmr | 1288 |
| 2-β-Cyanoethyl-3(2*H*)-pyridazinone | 75–76, nmr | 118 |
| 5-Cyano-6-imino-1,4-diphenyl-1,6-dihydro-3-pyridazinecarboxanilide | 155 | 219 |
| 5-Cyano-4-imino-1,6-diphenyl-1,4-dihydro-3-pyridazinecarboxylic acid | 198, ir | 376 |
| 5-Cyano-6-imino-4-methyl-1-phenyl-1,6-dihydro-3-pyridazinecarboxanilide | 160 | 219 |

*continued*

| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 5-Cyano-6-imino-4-methyl-1-phenyl-1,6-dihydro-3-pyridazinecarboxylic acid | 210 or >270, ir | 1216, 1864 |
| 3-(*N*-Cyano-*N*-methylamino)pyridazine | 151–154, nmr | 1010 |
| 3-(*N*-Cyano-*N*-methylamino)pyridazine 2-oxide | — | 921 |
| 4-Cyanomethyl-3,6-diphenylpyridazine | 156–157, ir, nmr | 222 |
| 5-Cyano-4-methyl-1,6-diphenylpyridazinium-3-carboxylate | 210, ir | 1216 |
| 4-Cyanomethyl-6-imino-1-phenyl-1,6-dihydro-3,5-pyridazinedicarbonitrile | >300, ir, nmr | 1364 |
| 5-Cyano-3-methyl-6-oxo-1,6-dihydro-4-pyridazinecarboxylic acid | 230–231, ir, nmr | 128 |
| 5-Cyano-4-methyl-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarbohydrazide | 230–233, complexes, ir, nmr; PhCH=: 190–194, ir, nmr | 1365, 1714, 1800, 1801 |
| 5-Cyano-4-methyl-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxanilide | 230 | 219 |
| 5-Cyano-4-methyl-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylic acid | 238 or 250, ir, nmr, $pK_a$ | 52, 1240, 1365, 1368 |
| 6-Cyano-5-methyl-3-oxo-2-phenyl-2,3-dihydro-4-pyridazinecarboxylic acid | 204, ir | 1217 |
| 3-Cyanomethyl-6-phenyl-4(1*H*)-pyridazinone | 273–274, ir, nmr | 1489 |
| 3-Cyanomethylpyridazine | uv | 1164 |
| 4-Cyano-1-methylpyridazinium-3-olate | 200–203, ir, nmr, uv | 1300 |
| 2-ε-Cyanopentyl-6-phenyl-3(2*H*)-pyridazinone | 200–207/0.7, ir, nmr | 118, 1288 |
| 2-γ-Cyanopropyl-4,6-diphenyl-3(2*H*)-pyridazinimine | HBr: hygroscopic | 1309 |
| 2-γ-Cyanopropyl-6-methoxy-3-methylthiopyridazine | — | 2024 |
| 2-γ-Cyanopropyl-4-methyl-6-phenyl-3(2*H*)-pyridazinimine | HBr: hygroscopic | 1309 |
| 2-γ-Cyanopropyl-6-phenyl-3(2*H*)-pyridazinimine | HBr: anal, hygroscopic | 1309 |
| 2-γ-Cyanopropyl-6-phenyl-3(2*H*)-pyridazinone | 185–190/0.3, ir, nmr | 118, 1288 |
| 4-Cyclohexylamino-3,6-diphenylpyridazine | — | 252 |
| 4-Cyclohexylamino-2-methyl-1-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 159, 595 |
| 3-Cyclohexylamino-6-methylsulfonylpyridazine | — | *H* 823 |
| 4-Cyclohexylamino-5-nitro-2-phenyl-3(2*H*)-pyridazinone | — | 601 |
| 2-Cyclohexyl-4,5-bismethylthio-3(2*H*)-pyridazinone | — | *H* 815 |
| 2-Cyclohexyl-4,5-dimethoxy-3(2*H*)-pyridazinone | — | *H* 126 |
| 2-Cyclohexyl-4-dimethylamino-6-ethoxy-3(2*H*)-pyridazinone | — | *H* 588 |
| 2-Cyclohexyl-5-dimethylamino-6-ethoxy-3(2*H*)-pyridazinone | — | *H* 591 |
| 1-Cyclohexyl-4-dimethylamino-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 591 |
| 1-Cyclohexyl-5-dimethylamino-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 588 |
| 6-Cyclohexyl-2-methyl-3-oxo-2,3-dihydro-4-pyridazinecarbonitrile | — | *H* 99 |
| 4-Cyclohexyl-6-methylpyridazine | 57–59, ms, nmr | 1676 |

*continued*

| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 6-Cyclohexyl-4-methyl-3(2*H*)-pyridazinone | 173 | 1319 |
| 2-Cyclohexyl-5-nitro-3,4(1*H*,2*H*)-pyridazinedione | — | *H* 169, 648 |
| 6-Cyclohexyl-3-oxo-2,3-dihydro-4-pyridazinecarbonitrile | — | *H* 98 |
| 3-Cyclohexyloxy-6-dimethylaminopyridazine | — | 707 |
| 3-Cyclohexyloxy-6-ethoxypyridazine | — | 677 |
| 3-Cyclohexyloxy-6-isobutoxypyridazine | — | 677 |
| 3-Cyclohexyloxy-6-isopentyloxypyridazine | — | 677 |
| 3-Cyclohexyloxy-6-isopropoxypyridazine | — | 677 |
| 3-Cyclohexyloxy-6-methoxypyridazine | — | 677 |
| 3-Cyclohexyloxy-6-pentyloxypyridazine | — | 677 |
| 3-Cyclohexyloxy-6-phenoxypyridazine | 2-MeI: – | 690 |
| 3-Cyclohexyloxy-6-propoxypyridazine | — | 677 |
| 6-Cyclohexyl-3(2*H*)-pyridazinone | 156, nmr | 1309 |
| 6-Cyclohexylthio-3-pyridazinamine | 124–125, nmr | 1147 |
| 4,5-Diacetamido-3,6-dimethoxypyridazine | — | *H* 599 |
| 4,5-Diacetamido-6-methoxy-3(2*H*)-pyridazinone | — | *H* 128, 599 |
| 4,5-Diacetamido-2-methyl-3(2*H*)-pyridazinone | 195–196, nmr | 325 |
| 4,5-Diacetamido-6-methyl-3(2*H*)-pyridazinone | — | *H* 601 |
| 3,4-Diacetamidopyridazine | — | *H* 597 |
| 1,2-Diacetonyl-4,5-dichloro-3,6(1*H*,2*H*)-pyridazinedione | 137–138, ir, nmr | 2039 |
| 3,6-Diacetonylpyridazine | 68, ir, nmr | 825 |
| 3,6-Diacetyl-4,5-dihydro-4,5-pyridazinedione 1,2-dioxide | 95–97 | 506 |
| 3,6-Diacetylpyridazine | dioxime: anal, complexes, ir, nmr | 1545 |
| 4,5-Diacetylpyridazine | 52–53, ir, nmr | 81 |
| 4-Diallylamino-3,6-dichloropyridazine | — | *H* 585 |
| 5-Diallylamino-4-ethoxy-2-methyl-3(2*H*)-pyridazinone | 130/2 | 1269 |
| 3-Diallylamino-6-hydrazinopyridazine | 2 HCl: 214–217; PhCH=: 173–175 | 1258; *H* 579 |
| 3,6-Diallyloxypyridazine | — | *H* 189 |
| 1,2-Diallyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 163 |
| 4,5-Diamino-2-benzyl-6-benzyloxy-3(2*H*)-pyridazinone | — | *H* 602 |
| 2,3-Diamino-6-chloropyridazinium ( + anion) | mesitylenesulfonate: 244–245 | 929 |
| 4,5-Diamino-6-chloro-3(2*H*)-pyridazinone | — | *H* 599 |
| 4,5-Diamino-2-cyclohexyl-3(2*H*)-pyridazinone | — | *H* 602 |
| 4,5-Diamino-2,6-dimethyl-3(2*H*)-pyridazinone | — | *H* 602 |
| 4,5-Diamino-6-methoxy-3(2*H*)-pyridazinone | — | *H* 128, 599 |
| 4,5-Diamino-2-methyl-3(2*H*)-pyridazinethione | 229–231, nmr, uv | 197, 325 |
| 4,5-Diamino-2-methyl-3(2*H*)-pyridazinone | 212 | 1206; *H* 602 |
| 4,5-Diamino-6-methyl-3(2*H*)-pyridazinone | — | *H* 599 |
| 4,5-Diamino-6-morpholinocarbonyl-2-phenyl-3(2*H*)-pyridazinone | 196–198, nmr | 1789 |
| 4,5-Diamino-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarbohydrazide | 265–268, ir, nmr | 1789 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 4,5-Diamino-2-phenyl-3(2*H*)-pyridazinone | 200, p$K_a$ | 601, 1167, 1206; *H* 602 |
| 5,6-Diamino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 93, 602 |
| 4,5-Diamino-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 601 |
| 4,5-Diamino-3(2*H*)-pyridazinethione | 275–276, nmr, uv | 197, 793; *H* 599, 798 |
| 5,6-Diamino-3(2*H*)-pyridazinethione | — | *H* 597, 798 |
| 5,6-Diamino-4(1*H*)-pyridazinethione | — | *H* 597, 800 |
| 2,3-Diaminopyridazinium ( + anion), *see* 3-Imino-2,3-dihydro-2-pyridazinamine (salt) | — | |
| 4,5-Diamino-3(2*H*)-pyridazinone | 231–232, nmr, uv | 650, 973; *H* 599, 601 |
| 3,6-Dianilinopyridazine | — | *H* 601 |
| 4,5-Diazido-6-nitro-3(2*H*)-pyridazinone | 115–118, ir | 1680 |
| 3,5-Diazido-6-phenylpyridazine | 190, ir, nmr | 133, 134 |
| 4,5-Diazido-2-phenyl-3(2*H*)-pyridazinone | — | *H* 654 |
| 3,6-Diazidopyridazine | 128–129 | 408, 1355; *H* 654 |
| 5-Diazonio-2-ethyl-3-oxo-2,3-dihydro-4-pyridazinolate | — | *H* 653 |
| 4-Diazonio-5-oxo-2-phenyl-2,5-dihydro-3-pyridazinolate | — | 909 |
| 5-Diazonio-3-oxo-2-phenyl-2,3-dihydro-4-pyridazinolate | — | 909; *H* 653 |
| 6-Diazonio-3-pyridazinolate 1-oxide | — | *H* 653 |
| 3,6-Dibenzoyl-4,5-dihydro-4,5-pyridazinedione 1,2-dioxide | 180–182, nmr | 250, 506; *H* 198 |
| 3,6-Dibenzoyl-1,2-dihydroxy-4,5(1*H*,2*H*)-pyridazinedione | — | *H* 198 |
| 3,6-Dibenzoyl-1,5-dihydroxy-4(1*H*)-pyridazinone | — | *H* 134 |
| 3,6-Dibenzoylpyridazine | 127–129, ir, ms, nmr; dioxime: 228, complexes, ir, ms, nmr | 1545, 1638, 1698, 1939 |
| 4,5-Dibenzoylpyridazine | 119–120, ir, ms, nmr; mono-sc: 200–202, ir, nmr | 72, 79, 81 |
| 3,6-Dibenzyl-5-hydroxy-4(1*H*)-pyridazinone | — | *H* 132, 133 |
| 2,5-Dibenzyl-6-methyl-3(2*H*)-pyridazinone | 88, ir, nmr | 884 |
| 3,4-Dibenzyloxy-6-chloropyridazine | 106 then 126–127, nmr, uv | 665 |
| 3,6-Dibenzyloxypyridazine | — | *H* 191 |
| 2,4-Dibenzyl-6-phenyl-3(2*H*)-pyridazinone | 102 | 1251 |
| 4,5-Dibromo-3,6-bistrimethylsiloxypyridazine | 71–72, 125–127/2, nmr, uv | 1168, 1172 |
| 4,5-Dibromo-6-butanesulfonyloxy-3(2*H*)-pyridazinone | — | *H* 331 |
| 4,5-Dibromo-2-butyl-3(2*H*)-pyridazinone | liq, ir, nmr | 1936 |
| 4,5-Dibromo-2-$\beta$-carboxyethyl-3(2*H*)-pyridazinone | — | 587; *H* 112, 317 |
| 4,5-Dibromo-6-$\beta$-carboxyethyl-3(2*H*)-pyridazinone | — | *H* 331 |

*continued*

| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 4,5-Dibromo-2-(chloroformyl)ethyl-3(2*H*)-pyridazinone | — | *H* 112, 317 |
| 3,6-Dibromo-4-chloropyridazine | 62–64, 140/3, nmr | 447 |
| 4,5-Dibromo-2-β-cyanoethyl-3(2*H*)-pyridazinone | — | 587; *H* 112, 317 |
| 4,5-Dibromo-1,2-dimethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 163, 337 |
| 4,5-Dibromo-6-ethoxy-3(2*H*)-pyridazinone | — | *H* 127, 184, 296, 330 |
| 4,5-Dibromo-1-ethyl-2-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 162, 339 |
| 4,5-Dibromo-2-ethyl-3(2*H*)-pyridazinone | 47–48, ir, nmr | 1936 |
| 4,5-Dibromo-3-β-hydroxyethyl-3(2*H*)-pyridazinone | — | *H* 401 |
| 4,5-Dibromo-2-hydroxymethyl-3(2*H*)-pyridazinone | — | 587 |
| 4,5-Dibromo-6-isopentanesulfonyloxy-3(2*H*)-pyridazinone | — | *H* 331 |
| 4,5-Dibromo-1-isopropyl-2-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 167, 339 |
| 4,5-Dibromo-2-isopropyl-3(2*H*)-pyridazinone | 85–86, ir, nmr | 1936 |
| 4,5-Dibromo-6-methoxy-2-methyl-3(2*H*)-pyridazinone | — | *H* 128, 330 |
| 4,5-Dibromo-6-methoxy-2-phenyl-3(2*H*)-pyridazinone | 149–150, ir, nmr, uv | 547 |
| 4,5-Dibromo-1-methyl-2-β-morpholinoethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 164, 339 |
| 4,5-Dibromo-2-methyl-6-nitro-3(2*H*)-pyridazinone | — | *H* 128, 330, 648 |
| 4,5-Dibromo-1-methyl-2-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 159, 339 |
| 3,6-Dibromo-4-methylpyridazine | — | *H* 295 |
| 4,5-Dibromo-1-methyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 152, 337 |
| 4,5-Dibromo-2-methyl-3(2*H*)-pyridazinone | 113 to 119, ir, nmr | 542, 956, 1936; *H* 112, 317 |
| 4,6-Dibromo-5-methyl-3(2*H*)-pyridazinone 1-oxide | 240 | 833 |
| 4,5-Dibromo-6-nitro-3(2*H*)-pyridazinone | — | *H* 127, 330, 648 |
| 4,5-Dibromo-6-phenylmethanesulfonyloxy-3(2*H*)-pyridazinone | — | *H* 331 |
| 4,5-Dibromo-1-phenyl-3,6(1*H*,2*H*)-pyridazinedione | 234–235, ir | 547 |
| 4,5-Dibromo-2-phenyl-3(2*H*)-pyridazinone | 145–146, ir, nmr | 542; *H* 317 |
| 5,6-Dibromo-2-phenyl-3(2*H*)-pyridazinone | — | *H* 92, 330 |
| 4,5-Dibromo-2-propyl-3(2*H*)-pyridazinone | 58–59, ir, nmr | 1936 |
| 3,6-Dibromo-4-pyridazinamine | 225–226, nmr | 447 |
| 3,6-Dibromopyridazine | nmr | 1638; *H* 286 |
| 4,5-Dibromo-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 127, 150, 296, 330, 337 |
| 4,5-Dibromo-3(2*H*)-pyridazinone | 224–225, ir, nmr | 542; *H* 112, 317 |
| 3,5-Dibromo-4(1*H*)-pyridazinone 2-oxide | — | *H* 196, 300 |
| 4,6-Dibromo-3(2*H*)-pyridazinone 1-oxide | — | *H* 196 |
| 4,5-Dibromo-3-trimethylsiloxypyridazine | 48–50, 112–113/2, nmr, uv | 1172 |
| 3,5-Dibutoxy-4-chloropyridazine | crude: liq; HCl: 114–118 | 640 |
| 3,5-Dibutoxy-6-chloropyridazine | 48–50, nmr | 640 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3,6-Dibutoxy-4-chloropyridazine | 13–14, nmr | 640 |
| 3,4-Dibutoxy-5-dimethylaminopyridazine | HCl: 97 | 637 |
| 3,4-Dibutoxy-6-dimethylaminopyridazine | HCl: 156–157 | 637 |
| 3,5-Dibutoxy-4-dimethylaminopyridazine | liq, nmr; pic: 77–80 | 641 |
| 3,5-Dibutoxy-6-dimethylaminopyridazine | nmr; pic: 75–76 | 640 |
| 3,6-Dibutoxy-4-dimethylaminopyridazine | HCl: 145–146 or 149–150 | 637, 640 |
| 3,5-Dibutoxy-6-morpholinopyridazine | 80–82, nmr; HCl: 122–124 | 640 |
| 3,6-Dibutoxy-4-morpholinopyridazine | HCl: 134–135 | 637 |
| 3,6-Dibutoxy-4-nitropyridazine 1-oxide | — | *H* 198, 649 |
| 3,6-Dibutoxypyridazine | — | *H* 190 |
| 3,6-Di-*t*-butoxypyridazine | — | *H* 190 |
| 3,6-Dibutoxypyridazine 1-oxide | — | *H* 195 |
| 4,5-Di-*s*-butyl-3,6-dichloropyridazine | 97–100, ms, nmr | 1223 |
| 3,6-Di-*t*-butylpyridazine | 179–180, ir, nmr | 38 |
| 4,5-Di-*t*-butylpyridazine | 108–109, ir, nmr | 239, 628, 2046 |
| 1,2-Dibutyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 163 |
| 3,5-Dichloro-4,6-bisdimethylaminopyridazine | 58 | 637 |
| 4,5-Dichloro-3,6-bisdimethylaminopyridazine | crude: liq, ms; HCl: 137–140 | 1796 |
| 4,5-Dichloro-1,2-bis(α-ethylpropyl)-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 163, 336 |
| 3,6-Dichloro-4,5-bisethylthiopyridazine | — | *H* 289, 814 |
| 3,6-Dichloro-4,5-bismethylthiopyridazine | — | *H* 289, 814 |
| 4,5-Dichloro-1,2-bis(β-morpholinoethyl)-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 163, 336 |
| 4,5-Dichloro-3,6-bistrimethylsiloxypyridazine | 55–56, 112–113/3, nmr, uv | 1168, 1172 |
| 3,6-Dichloro-4-(β-chloro-α,α-dimethylethyl)pyridazine | 64–65, ir, nmr | 354 |
| 4,5-Dichloro-2-β-chloroethyl-6-methoxy-3(2*H*)-pyridazinone | — | *H* 130, 329 |
| 4,5-Dichloro-1-β-chloroethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 153, 334 |
| 5,6-Dichloro-2-β-chloroethyl-3(2*H*)-pyridazinone | — | *H* 92, 327 |
| 4,5-Dichloro-2-β-(chloroformyl)ethyl-3(2*H*)-pyridazinone | — | *H* 114, 309 |
| 4,5-Dichloro-2-(β-chloro-β-methylbutyl)-3(2*H*)-pyridazinone | — | *H* 114 |
| 4,5-Dichloro-2-chloromethyl-6-methoxy-3(2*H*)-pyridazinone | — | *H* 129, 329 |
| 4,5-Dichloro-2-(β-chloromethyl-β-methylbutyl)-3(2*H*)-pyridazinone | — | *H* 114 |
| 4,5-Dichloro-2-(β-chloro-β-methylpentyl)-3(2*H*)-pyridazinone | — | *H* 114 |
| 4,5-Dichloro-2-(β-chloro-β-methylpropyl)-3(2*H*)-pyridazinone | — | *H* 114 |
| 4,5-Dichloro-1-chloromethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 151, 334 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 4,5-Dichloro-2-chloromethyl-3(2*H*)-pyridazinone | 69–70 | 441, 916; *H* 112, 308 |
| 4,6-Dichloro-2-chloromethyl-3(2*H*)-pyridazinone | — | *H* 99, 324 |
| 5,6-Dichloro-2-chloromethyl-3(2*H*)-pyridazinone | — | *H* 91, 327 |
| 4,5-Dichloro-2-β-cyanoethyl-6-methoxy-3(2*H*)-pyridazinone | — | *H* 130, 329 |
| 4,5-Dichloro-1-β-cyanoethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 155, 334 |
| 4,5-Dichloro-2-β-cyanoethyl-3(2*H*)-pyridazinone | 100–101, nmr | 118; *H* 114, 309 |
| 3,6-Dichloro-5-β-cyanovinyl-4(1*H*)-pyridazinone | 226–227 | 963 |
| 3,6-Dichloro-4-cyclohexylaminopyridazine | — | *H* 288, 583 |
| 3,6-Dichloro-5-cyclohexylamino-4-pyridazinecarbonitrile | 117–118, uv | 820 |
| 4,5-Dichloro-1-cyclohexyl-2-methyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 167, 335 |
| 4,5-Dichloro-1-cyclohexyl-2-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 168, 336 |
| 3,6-Dichloro-4-cyclohexylpyridazine | 72–73, ms, nmr | 1223 |
| 4,5-Dichloro-2-cyclohexyl-3(2*H*)-pyridazinone | — | *H* 115 |
| 4,6-Dichloro-2-cyclohexyl-3(2*H*)-pyridazinone | — | *H* 110, 325 |
| 5,6-Dichloro-2-cyclohexyl-3(2*H*)-pyridazinone | — | *H* 326 |
| 4,5-Dichloro-1,2-dicyclohexyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 163, 336 |
| 4,5-Dichloro-1-β-diethylaminoethyl-2-methyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 164, 334 |
| 4,5-Dichloro-1-β-diethylaminoethyl-2-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 167, 336 |
| 4,5-Dichloro-2-β-diethylaminoethyl-3(2*H*)-pyridazinone | 193–194 | 553; *H* 113, 308 |
| 5,6-Dichloro-2-β-diethylaminoethyl-3(2*H*)-pyridazinone | — | *H* 92, 327 |
| 4,5-Dichloro-1-diethylaminomethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 334 |
| 4,5-Dichloro-2-diethylaminomethyl-3(2*H*)-pyridazinone | — | *H* 113, 308 |
| 5,6-Dichloro-2-diethylaminomethyl-3(2*H*)-pyridazinone | — | *H* 91, 327 |
| 3,6-Dichloro-4-diethylaminopyridazine | — | *H* 288, 585 |
| 3,6-Dichloro-1-(diethylcarbamoyl)methyl-4(1*H*)-pyridazinone | — | *H* 134, 333 |
| 4,5-Dichloro-2-(diethylcarbamoyl)methyl-3(2*H*)-pyridazinone | — | *H* 114, 309 |
| 4,6-Dichloro-2-(diethylcarbamoyl)methyl-3(2*H*)-pyridazinone | — | *H* 101, 324 |
| 5,6-Dichloro-2-(diethylcarbamoyl)methyl-3(2*H*)-pyridazinone | — | *H* 92, 327 |
| 3,6-Dichloro-4,5-diethylpyridazine | 108–109, ms, nmr | 1223 |
| 3,4-Dichloro-5,6-difluoropyridazine | nmr | 459 |
| 3,5-Dichloro-4,6-difluoropyridazine | nmr | 459 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 4,5-Dichloro-3,6-difluoropyridazine | 48–49, 190/760, nmr | 459; *H* 297 |
| 3,6-Dichloro-4-diisopropylaminopyridazine | — | *H* 288, 585 |
| 3,6-Dichloro-4,5-diisopropylpyridazine | 138–141, ms, nmr | 1223 |
| 4,5-Dichloro-1,2-diisopropyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 163, 336 |
| 3,6-Dichloro-4,5-dimethoxypyridazine | — | *H* 185, 289 |
| 4,5-Dichloro-1-γ-dimethylaminobutyl-2-methyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 166, 335 |
| 4,5-Dichloro-1-β-dimethylaminoethyl-2-methyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 164, 334 |
| 4,5-Dichloro-2-β-dimethylaminoethyl-3(2*H*)-pyridazinone | 248–250 | 553 |
| 4,5-Dichloro-1-(γ-dimethylamino-β-methylpropyl)-2-methyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 166, 335 |
| 4,5-Dichloro-6-dimethylamino-2-methyl-3(2*H*)-pyridazinone | 96–97, ir, nmr | 1938 |
| 4,5-Dichloro-1-β-dimethylaminopropyl-2-methyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 166 |
| 4,5-Dichloro-1-γ-dimethylaminopropyl-2-methyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 166 |
| 4,5-Dichloro-2-γ-dimethylaminopropyl-3(2*H*)-pyridazinone | 237–238 | 553 |
| 3,4-Dichloro-5-dimethylaminopyridazine | 89–90 | 1796; *H* 584 |
| 3,4-Dichloro-6-dimethylaminopyridazine | 144–145, nmr | 1796 |
| 3,5-Dichloro-4-dimethylaminopyridazine | 43–45, nmr | 641 |
| 3,6-Dichloro-4-dimethylaminopyridazine | 66–67 | 1796; *H* 287, 584 |
| 3,5-Dichloro-6-dimethylamino-4(1*H*)-pyridazinone | 228–230 | 637 |
| 4,5-Dichloro-6-dimethylamino-3(2*H*)-pyridazinone | 193–194, ir, nmr | 1938 |
| 4,6-Dichloro-5-dimethylamino-3(2*H*)-pyridazinone | 205 | 461 |
| 5,6-Dichloro-4-dimethylamino-3(2*H*)-pyridazinone | 200 | 641 |
| 3,6-Dichloro-4-α,α-dimethylbutylpyridazine | liq, ms, nmr | 1223 |
| 3,6-Dichloro-4,5-dimethylpyridazine | 115–116, nmr | 755, 762; *H* 289, 294 |
| 4,5-Dichloro-3,6-dimethylpyridazine | 62–65, nmr; pic: 93–94 | 529 |
| 4,5-Dichloro-1,2-dimethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 163, 334 |
| 3,6-Dichloro-4,5-dimethylpyridazine 1-oxide | 138–139, ir | 103 |
| 4,5-Dichloro-3,6-dimethylpyridazine 1-oxide | 105–109, nmr | 796 |
| 4,5-Dichloro-2,6-dimethyl-3(2*H*)-pyridazinone | — | *H* 128, 328 |
| 4,5-Dichloro-2-α-ethoxycarbonylethyl-3(2*H*)-pyridazinone | — | *H* 114, 309 |
| 4,5-Dichloro-1-ethoxycarbonylmethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 154, 334 |
| 3,6-Dichloro-1-ethoxycarbonylmethyl-4(1*H*)-pyridazinone | — | *H* 134, 333 |
| 4,5-Dichloro-2-ethoxycarbonylmethyl-3(2*H*)-pyridazinone | — | *H* 114, 309 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 4,6-Dichloro-2-ethoxycarbonylmethyl-3(2*H*)-pyridazinone | — | *H* 101, 324 |
| 5,6-Dichloro-2-ethoxycarbonylmethyl-3(2*H*)-pyridazinone | — | *H* 92, 327 |
| 3,6-Dichloro-4-ethoxypyridazine | — | *H* 288 |
| 3,6-Dichloro-4-ethylaminopyridazine | — | *H* 287, 582 |
| 3,6-Dichloro-5-ethylamino-4-pyridazinecarbonitrile | 135–137, uv | 820 |
| 3,4-Dichloro-5-ethylimino-2-phenyl-2,5-dihydropyridazine | — | *H* 608 |
| 3,6-Dichloro-4-ethyl-5-methylpyridazine | — | *H* 289 |
| 4,5-Dichloro-2-ethyl-6-nitro-3(2*H*)-pyridazinone | — | *H* 130, 329, 648 |
| 3,6-Dichloro-4-ethylpyridazine | liq, ms, nmr | 1223 |
| 3,6-Dichloro-1-ethyl-4(1*H*)-pyridazinone | — | *H* 134, 333 |
| 4,5-Dichloro-2-ethyl-3(2*H*)-pyridazinone | 49–50, ir, nmr | 916, 1901, 1936; *H* 113, 308 |
| 4,6-Dichloro-2-ethyl-3(2*H*)-pyridazinone | — | *H* 100, 324 |
| 5,6-Dichloro-2-ethyl-3(2*H*)-pyridazinone | — | *H* 91, 327 |
| 3,6-Dichloro-4-ethylthiopyridazine | — | *H* 288, 810 |
| 3,6-Dichloro-4-fluoropyridazine | — | *H* 917, 1053 |
| 3,6-Dichloro-4-hydrazinocarbonylmethylpyridazine | 137–140, ir | 947 |
| 3,6-Dichloro-4-hydrazinopyridazine | 213 | 640; *H* 288, 661 |
| 4,5-Dichloro-2-δ-hydroxybutyl-3(2*H*)-pyridazinone | 92–93, ir, nmr | 1700 |
| 3,6-Dichloro-4-(β-hydroxy-α,α-dimethylethyl)pyridazinone | 134–136, ir, nmr | 354 |
| 4,5-Dichloro-1-β-hydroxyethyl-2-isopropyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 167, 336 |
| 4,5-Dichloro-2-β-hydroxyethyl-6-methoxy-3(2*H*)-pyridazinone | — | *H* 130, 329, 402 |
| 3,6-Dichloro-4-α-hydroxyethylpyridazine | 80, ir, nmr | 1669 |
| 4,5-Dichloro-1-β-hydroxyethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 153, 334, 402 |
| 4,5-Dichloro-2-β-hydroxyethyl-3(2*H*)-pyridazinone | — | *H* 114, 309, 402 |
| 5,6-Dichloro-2-β-hydroxyethyl-3(2*H*)-pyridazinone | — | *H* 92, 327, 403 |
| 4,5-Dichloro-2-hydroxymethyl-6-nitro-3(2*H*)-pyridazinone | 85–86, ir, nmr | 1700 |
| 3,6-Dichloro-5-(3-hydroxy-2-methylprop-1-enyl)-4(1*H*)-pyridazinone | 146–147, nmr | 963 |
| 3,6-Dichloro-4-hydroxymethylpyridazine | 128–130, nmr | 755; *H* 288 |
| 4,5-Dichloro-1-hydroxymethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 153, 334, 402 |
| 4,5-Dichloro-2-hydroxymethyl-3(2*H*)-pyridazinone | 111–113, ir, nmr | 441, 1700; *H* 113, 308, 402 |
| 4,6-Dichloro-2-hydroxymethyl-3(2*H*)-pyridazinone | — | *H* 100, 324, 402 |
| 5,6-Dichloro-2-hydroxymethyl-3(2*H*)-pyridazinone | — | *H* 91, 327, 403 |
| 4,6-Dichloro-5-hydroxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 130, 329 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3,6-Dichloro-5-hydroxy-4(1*H*)-pyridazinone | — | *H* 132, 289 |
| 3,6-Dichloro-4-iodopyridazine | 135, ir, nmr | 1669 |
| 4,5-Dichloro-2-isobutyl-3(2*H*)-pyridazinone | — | *H* 114, 309 |
| 3,4-Dichloro-5-isopropylaminopyridazine | nmr | 1917 |
| 3,5-Dichloro-4-isopropylaminopyridazine | — | 1917 |
| 3,6-Dichloro-4-isopropylaminopyridazine | — | *H* 583 |
| 4,5-Dichloro-1-isopropyl-2-phenyl-3,6(1*H*, 2*H*)-pyridazinedione | — | *H* 167, 336 |
| 3,6-Dichloro-4-isopropylpyridazine | liq, ms, nmr | 1223 |
| 4,5-Dichloro-2-isopropyl-3(2*H*)-pyridazinone | 68–69, ir, nmr | 1936; *H* 114, 309 |
| 4,5-Dichloro-1-methoxycarbonylmethyl-2-methyl-3,6(1*H*, 2*H*)-pyridazinedione | — | *H* 164, 334 |
| 3,6-Dichloro-4-methoxycarbonylmethylpyridazine | 32–33, ir, uv | 947 |
| 3,5-Dichloro-6-methoxy-4-methylpyridazine 2-oxide | 138–139 | 833 |
| 4,5-Dichloro-6-methoxy-2-methyl-3(2*H*)-pyridazinone | — | *H* 129, 328 |
| 4,6-Dichloro-5-methoxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 130, 329 |
| 3,4-Dichloro-5-methoxypyridazine | 100–102, ir, nmr | 90, 689, 882; *H* 185, 289, 295 |
| 3,4-Dichloro-6-methoxypyridazine | 68–69, nmr | 881; *H* 183, 290 |
| 3,5-Dichloro-4-methoxypyridazine | 61, nmr | 882; *H* 185 |
| 3,5-Dichloro-6-methoxypyridazine | 80–81, nmr | 881; *H* 181 |
| 3,6-Dichloro-4-methoxypyridazine | 130, nmr | 640, 881; *H* 185, 288 |
| 4,5-Dichloro-3-methoxypyridazine | 96–97, nmr | 882 |
| 3,4-Dichloro-6-methoxypyridazine 1-oxide | — | *H* 196, 300 |
| 3,5-Dichloro-6-methoxypyridazine 2-oxide | 139–141 or 148–149, uv | 833, 905; *H* 300 |
| 3,6-Dichloro-4-methoxypyridazine 1-oxide | — | *H* 196, 301 |
| 3,6-Dichloro-4-methoxypyridazine 2-oxide | — | *H* 196, 300 |
| 2,4-Dichloro-5-methoxy-3(2*H*)-pyridazinone | — | *H* 119 |
| 3,6-Dichloro-4-methylaminopyridazine | — | *H* 287, 582 |
| 3,6-Dichloro-5-methylamino-4-pyridazinecarbonitrile | 164–165, uv | 820 |
| 4,5-Dichloro-2-β-(methylcarbamoyl)ethyl-3(2*H*)-pyridazinone | — | *H* 114, 309 |
| 3,4-Dichloro-5-α-methylhydrazinopyridazine | 132–134, ir, nmr | 1061 |
| 3,4-Dichloro-5-methylimino-2-phenyl-2,5-dihydropyridazine | — | *H* 608 |
| 4,5-Dichloro-1-methyl-2-β-morpholinoethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 165, 334 |
| 4,5-Dichloro-2-methyl-6-nitro-3(2*H*)-pyridazinone | 98–99, ir, nmr | 1938; *H* 129, 328, 648 |
| 4,5-Dichloro-1-methyl-6-oxo-1,6-dihydro-3-pyridazinecarboxylic acid | — | *H* 128, 328, 438 |
| 4,5-Dichloro-1-methyl-2-phenethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 164, 335 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3,6-Dichloro-4-methyl-5-phenylpyridazine | — | *H* 289 |
| 4,5-Dichloro-1-methyl-2-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 159, 335 |
| 3,6-Dichloro-4-methylpyridazine | 63–65, nmr | 755, 762; *H* 287 |
| 3,6-Dichloro-5-methyl-4-pyridazinecarbonitrile | 99, nmr, uv | 821 |
| 4,5-Dichloro-6-methyl-3-pyridazinecarbonitrile 2-oxide | 178–179, ir, nmr | 796 |
| 3,6-Dichloro-*N*-methyl-4-pyridazinecarboxamide | — | 2040 |
| 4,5-Dichloro-1-methyl-3,6-(1*H*,2*H*)-pyridazinedione | | *H* 152, 334 |
| 3,4-Dichloro-6-methylpyridazine 1-oxide | | *H* 300 |
| 4,5-Dichloro-1-methylpyridazinium-3-olate | 230, nmr, uv | 1300 |
| 5,6-Dichloro-2-methylpyridazinium-4-olate | 217, ir, nmr, uv | 699 |
| 3,6-Dichloro-1-methyl-4(1*H*)-pyridazinone | — | *H* 133, 333 |
| 4,5-Dichloro-2-methyl-3(2*H*)-pyridazinone | 87 to 92, ir, nmr | 689, 956, 1213, 1269, 1901, 1936, 2048; *H* 112, 308 |
| 4,6-Dichloro-2-methyl-3(2*H*)-pyridazinone | — | *H* 98, 324 |
| 5,6-Dichloro-1-methyl-4(1*H*)-pyridazinone | 173–174 | 141 |
| 5,6-Dichloro-2-methyl-3(2*H*)-pyridazinone | 97–98, p$K_a$, uv | 675, 881; *H* 91, 327 |
| 4,6-Dichloro-5-methyl-3(2*H*)-pyridazinone 1-oxide | 234 | 833 |
| 3,6-Dichloro-4-methylthiopyridazine | — | *H* 208, 810 |
| 4,5-Dichloro-1-β-morpholinoethyl-2-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 167, 338, 336 |
| 4,5-Dichloro-2-β-morpholinoethyl-3(2*H*)-pyridazinone | 250–252 | 553 |
| 4,5-Dichloro-2-morpholinomethyl-3(2*H*)-pyridazinone | — | *H* 113, 308 |
| 2,4-Dichloro-5-morpholinopyridazine | 105–107 or 109, ms, nmr | 637, 1058 |
| 3,6-Dichloro-4-morpholinopyridazine | 113–115, ir, nmr | 544, 637 |
| 3,6-Dichloro-5-nitro-4-pyridazinamine | — | *H* 561 |
| 4,5-Dichloro-6-nitro-3(2*H*)-pyridazinone | — | 1673; *H* 127, 328, 648 |
| 4,5-Dichloro-6-oxo-1,6-dihydro-1-pyridazinecarbonitrile | — | *H* 113, 308 |
| 3,6-Dichloro-4-*t*-pentylpyridazine | liq, ms, nmr | 1223 |
| 4,5-Dichloro-2-phenethyl-3(2*H*)-pyridazinone | — | *H* 113, 309 |
| 3,4-Dichloro-5-phenoxypyridazine | — | *H* 186, 290 |
| 3,6-Dichloro-4-phenoxypyridazine | — | *H* 186, 288 |
| 3,4-Dichloro-6-phenylpyridazine | 164–165 | 1041 |
| 3,6-Dichloro-4-phenylpyridazine | 92–93 | 461; *H* 287 |
| 4,5-Dichloro-1-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 156, 334 |
| 3,6-Dichloro-1-phenyl-4(1*H*)-pyridazinimine | — | *H* 608 |
| 5,6-Dichloro-1-phenyl-4(1*H*)-pyridazinimine | — | *H* 608 |
| 4,5-Dichloro-2-phenyl-3(2*H*)-pyridazinone | 163–164, nmr | 464, 916, 956, 1213; *H* 114, 309 |
| 4,6-Dichloro-2-phenyl-3(2*H*)-pyridazinone | — | *H* 104, 325 |
| 5,6-Dichloro-2-phenyl-3(2*H*)-pyridazinone | nmr, xl st | 1126; *H* 92, 327 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 4,5-Dichloro-2-piperidinomethyl-3(2*H*)-pyridazinone | — | *H* 113, 308 |
| 3,4-Dichloro-5-piperidinopyridazine | 88–89, ms, nmr | 1058 |
| 3,6-Dichloro-4-piperidinopyridazine | 80 | 641 |
| 4,5-Dichloro-1-propyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 154, 334 |
| 4,5-Dichloro-2-propyl-3(2*H*)-pyridazinone | liq, anal, ir, nmr | 1901, 1936 |
| 5,6-Dichloro-2-propyl-3(2*H*)-pyridazinone | — | *H* 92, 327 |
| 3,5-Dichloro-4-pyridazinamine | 149–150, nmr, uv | 1820; *H* 588 |
| 3,6-Dichloro-4-pyridazinamine | 204–205 or 206–207, ir, nmr, $K_a$, uv | 640, 654, 675, 1228; *H* 287, 558 |
| 5,6-Dichloro-4-pyridazinamine | 177–178 or 183–185, nmr, uv | 83, 1821; *H* 259, 559 |
| 3,5-Dichloro-4-pyridazinamine 1-oxide | — | *H* 300, 603 |
| 5,6-Dichloro-4-pyridazinamine 2-oxide | — | *H* 300, 603 |
| 3,4-Dichloropyridazine | — | *H* 275 |
| 3,5-Dichloropyridazine | — | *H* 275 |
| 3,6-Dichloropyridazine | 60 to 68, dipole, nmr, p$K_a$, pol, st, uv | 94, 98, 99, 163, 177, 450, 1098, 1183, 1196,1345, 1504, 1638, 1983, 2020; *H* 275, 302, 303 |
| 4,5-Dichloropyridazine | 67–68, nmr, uv | 923; *H* 275 |
| 3,6-Dichloro-4-pyridazinecarbaldehyde | nmr; dnp: 259, ir, nmr | 1669 |
| 5,6-Dichloro-3-pyridazinecarbaldehyde 2-oxide | — | *H* 300, 398 |
| 3,6-Dichloro-4-pyridazinecarbonitrile | 105–106, ir, nmr, uv | 820 |
| 5,6-Dichloro-3-pyridazinecarbonitrile 2-oxide | — | *H* 300, 446 |
| 3,6-Dichloro-4-pyridazinecarbonyl chloride | 110/10 | 519 |
| 3,6-Dichloro-4-pyridazinecarboxamide | 177 or 178–179 | 25, 1099 |
| 3,6-Dichloro-4-pyridazinecarboxylic acid | 139–142, ir, nmr | 65, 1748; *H* 287, 436 |
| 3,6-Dichloro-4,5-pyridazinediamine | — | *H* 599 |
| 4,5-Dichloro-3,6(1*H*,2*H*)-pyridazinedione | 303–305, xl st | 139, 802; *H* 150, 334 |
| 3,6-Dichloropyridazine 1-oxide | 118–119 | 103, 546, 891; *H* 300 |
| 5,6-Dichloro-4(1*H*)-pyridazinethione | — | *H* 290 |
| 2,6-Dichloro-3(2*H*)-pyridazinone | — | *H* 307 |
| 3,6-Dichloro-4(1*H*)-pyridazinone | — | *H* 132, 288 |
| 4,5-Dichloro-3(2*H*)-pyridazinone | 199–200 or 200–201, arom, ir, nmr, p$K_a$, uv, xl st | 170, 675, 916, 973, 1014, 1024, 1415, 1673, 1901, 2048; *H* 112, 308 |
| 4,6-Dichloro-3(2*H*)-pyridazinone | — | *H* 96, 324 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 5,6-Dichloro-3(2*H*)-pyridazinone | 200–203 | 1796; *H* 87, 327 |
| 5,6-Dichloro-4(1*H*)-pyridazinone | 210 | 224 |
| 4,6-Dichloro-3(2*H*)-pyridazinone 1-oxide | 223 | 833 |
| 4,5-Dichloro-2-thiocyanatomethyl-3(2*H*)-pyridazinone | — | *H* 113, 308, 826 |
| 4,6-Dichloro-2-thiocyanatomethyl-3(2*H*)-pyridazinone | — | *H* 100, 324, 826 |
| 5,6-Dichloro-2-thiocyanatomethyl-3(2*H*)-pyridazinone | — | *H* 91, 327, 826 |
| 3,6-Dichloro-4-thiocyanatopyridazine | — | *H* 288, 825 |
| 3,4-Dichloro-6-trimethylammoniopyridazine (+ anion) | Cl: nmr | 1796 |
| 3,6-Dichloro-4-trimethylammoniopyridazine (+ anion) | Cl: nmr | 1796 |
| 4,5-Dichloro-3-trimethylsiloxypyridazine | 81–82/2, nmr, uv | 932, 1285 |
| 3,6-Dichloro-4-trimethylsilylpyridazine | liq, anal, nmr | 1669 |
| 3,5-Dicyano-6-oxo-1-phenyl-1,6-dihydro-4-pyridazinecarbaldehyde | PhNHN=: 260, ir, nmr | 395 |
| 1,2-Dicyclohexyl-5,6-diphenyl-3,4(1*H*,2*H*)-pyridazinedione | 170–171, ir, nmr | 1496 |
| 2,4-Dicyclohexyl-6-methyl-3(2*H*)-pyridazinone | liq, anal, ms, nmr | 1676 |
| 3,6-Dicyclohexyloxypyridazine | — | 673; *H* 191 |
| 2,5-Diethoxy-6-methoxy-3(2*H*)-pyridazinone | — | *H* 90 |
| 3,6-Diethoxy-4-methylpyridazine 1-oxide | — | *H* 198 |
| 3,6-Diethoxy-4-methylpyridazine 2-oxide | — | *H* 198 |
| 2,6-Diethoxy-5-methyl-3(2*H*)-pyridazinone | — | *H* 90 |
| 4,5-Diethoxy-2-methyl-3(2*H*)-pyridazinone | 28–30, 102–105/0.5, ir, nmr | 531 |
| 3,6-Diethoxy-4-morpholinopyridazine | 62–63, nmr; HCl: 144–145 | 640 |
| 3,6-Diethoxy-5-nitro-4-pyridazinamine | — | *H* 561 |
| 3,6-Diethoxy-4-nitropyridazine 1-oxide | — | *H* 198, 649 |
| 4,5-Diethoxy-2-phenyl-3(2*H*)-pyridazinone | 80 | 764; *H* 126 |
| 4,6-Diethoxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 109 |
| 3,6-Diethoxy-4-pyridazinamine | — | *H* 560 |
| 3,6-Diethoxypyridazine | — | *H* 188 |
| 3,6-Diethoxy-4,5-pyridazinediamine | — | *H* 599 |
| 3,6-Diethoxypyridazine 1-oxide | — | *H* 195 |
| 2,6-Diethoxy-3(2*H*)-pyridazinone | — | *H* 76 |
| Diethyl 6-acetamido-4-imino-1-phenyl-1,4-dihydro-3,5-pyridazinedicarboxylate | 196, ir, nmr | 151 |
| 4-Diethylamino-3,5-dimethyl-6-phenylpyridazine 2-oxide | HCl: 135–137 | 311 |
| 4-Diethylamino-5,6-dimethylpyridazine | — | *H* 585 |
| 3-Diethylamino-4,6-dinitropyridazine 1-oxide | 153–155 | 760 |
| 4-Diethylamino-3,6-diphenylpyridazine | — | 226 |
| 5-Diethylamino-4-ethoxy-2-methyl-3(2*H*)-pyridazinone | 144–149/5 | 1269 |
| 4-Diethylamino-6-ethoxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 109, 499 |
| 2-β-Diethylaminoethyl-4-ethyl-6-phenyl-3(2*H*)-pyridazinone | — | *H* 101 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 2-β-Diethylaminoethyl-4-methyl-6-phenyl-3(2*H*)-pyridazinone | — | *H* 101 |
| 2-β-Diethylaminoethyl-6-methyl-3(2*H*)-pyridazinone | — | *H* 72 |
| 2-β-Diethylaminoethyl-3-oxo-5,6-diphenyl-2,3-dihydro-4-pyridazinecarbonitrile | — | *H* 130, 449 |
| 2-β-Diethylaminoethyl-6-phenyl-3(2*H*)-pyridazinone | — | *H* 72 |
| 4-Diethylamino-6-ethyl-2-phenyl-3(2*H*)-pyridazinone | — | *H* 589 |
| 1-β-Diethylaminoethyl-2,4(1*H*,2*H*)-pyridazinedione | ir, nmr, uv | 1975 |
| 3-Diethylamino-6-hydrazinopyridazine | 2HCl: 234–236; PhCH=: 202–205 | 1258; *H* 579 |
| Diethyl 4-amino-6-imino-1-phenyl-1,6-dihydro-3,5-pyridazinedicarboxylate | 220, ir | 119, 151 |
| 4-Diethylamino-5-methyl-3,6-bismethylthiopyridazine | liq, ir, ms, nmr | 353 |
| 4-Diethylamino-5-methyl-3,6-diphenylpyridazine | 120–122, nmr | 311; *H* 585 |
| 4-Diethylamino-5-methyl-3,6-diphenylpyridazine 2-oxide | 153–156, ms | 311 |
| 2-Diethylaminomethyl-3-oxo-2,3-dihydro-4-pyridazinecarbonitrile | — | *H* 84, 448 |
| 3-Diethylamino-4-methyl-6-phenylpyridazine | liq, ms, nmr | 1442 |
| 4-Diethylamino-5-methyl-6-phenylpyridazine | — | *H* 585 |
| 4-Diethylamino-2-methyl-1-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 159, 595 |
| 4-Diethylamino-6-methyl-2-phenyl-3(2*H*)-pyridazinone | — | *H* 106, 588 |
| 3-Diethylaminomethylpyridazine | — | *H* 608 |
| 4-Diethylamino-5-methylpyridazine | — | *H* 585 |
| 1-Diethylaminomethyl-3,6(1*H*,2*H*)-pyridazinedione | — | 674; *H* 152 |
| 3-Diethylamino-6-phenylethynylpyridazine | 106–107, ir, nmr | 834 |
| 6-Diethylamino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 80, 594 |
| 3-Diethylaminopyridazine | 68/0.03 | 924 |
| 4-Diethylamino-3,6(1*H*,2*H*)-pyridazinedione | 237–239 | 641 |
| 6-Diethylamino-3(2*H*)-pyridazinethione | — | *H* 797 |
| 4-Diethylamino-3,5,6-trifluoropyridazine | — | *H* 297, 585 |
| 6-Diethylamino-5,*N*,*N*-trimethyl-4-oxo-3-phenyl-1,4-dihydro-1-pyridazinecarbothioamide | xl st | 169 |
| 2-(Diethylcarbamoyl)methyl-6-methyl-3(2*H*)-pyridazinone | — | *H* 79 |
| Diethyl 3-chloro-6-methyl-4,5-pyridazinedicarboxylate | liq, purified | 1514 |
| Diethyl 3,6-dimethyl-4,5-pyridazinedicarboxylate 3,6-pyridazinedicarboxylate | — | *H* 445 |
| Diethyl 4,5-dioxo-4,5-dihydro-1,3-dioxide (?) | — | *H* 196, 455 |
| Diethyl 3,6-diphenyl-4,5-pyridazinedicarboxylate | — | *H* 445 |
| *N*,*N*-Diethyl-1-methyl-6-oxo-1,6-dihydro-3-pyridazinecarboxamide | — | *H* 437 |
| Diethyl 3-methyl-6-oxo-1,6-dihydro-4,5-pyridazinedicarboxylate | 112–114 | 1514 |
| Diethyl 3-methyl-4,5-pyridazinedicarboxylate | liq, ir, ms, nmr | 416 |
| Diethyl 4-methyl-3,5-pyridazinedicarboxylate | 160/0.1, ir, ms, nmr | 416 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| Diethyl 6-methyl-3,4-pyridazinedicarboxylate | — | *H* 445 |
| Diethyl 6-oxo-1,6-dihydro-3,4-pyridazinedicarboxylate | — | *H* 88, 445 |
| Diethyl 4-phenethyl-3,5-pyridazinedicarboxylate | 79–85, ir, ms, nmr | 416 |
| Diethyl 5-phenethyl-3,4-pyridazinedicarboxylate | liq, ir, ms, nmr | 416 |
| 4,6-Diethyl-2-phenyl-3(2*H*)-pyridazinone | — | *H* 107 |
| 3,6-Diethylpyridazine | liq, ms, nmr | 186 |
| Diethyl 3,4-pyridazinedicarboxylate | 163–165/0.3, ms, nmr | 1120; *H* 445 |
| Diethyl 3,6-pyridazinedicarboxylate | 114, ir, nmr | 1077, 1509 |
| Diethyl 4,5-pyridazinedicarboxylate | 130/0.1 | 416, 1378 |
| 1-β-(Diethylsulfamoyl)ethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 829 |
| 3,6-Difluoro-4,5-bisphenylthiopyridazine | — | *H* 297, 814 |
| 3,4-Difluoro-5,6-dimethoxypyridazine | — | *H* 297 |
| 3,5-Difluoro-4,6-dimethoxypyridazine | — | *H* 297 |
| 3,6-Difluoro-4,5-dimethoxypyridazine | — | *H* 185, 297 |
| 4,5-Difluoro-3,6-dimethoxypyridazine | — | *H* 184, 298 |
| 4,5-Difluoro-1,2-dimethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 163, 337 |
| 5,6-Difluoro-4-methoxy-2-methyl-3(2*H*)-pyridazinone | — | *H* 129, 332 |
| 4,6-Difluoro-5-methoxy-3(2*H*)-pyridazinone | — | *H* 127, 332 |
| 5,6-Difluoro-4-methoxy-3(2*H*)-pyridazinone | — | *H* 127, 332 |
| 4,5-Difluoro-1-methyl-2-β-morpholinoethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 165, 338 |
| 3,6-Difluoropyridazine | — | 448, 1650 |
| 4,5-Difluoro-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 150, 337 |
| 3,6-Dihexyloxypyridazine | — | *H* 190 |
| 3,6-Dihydrazinopyridazine | 195–196, ir; di-$Me_2C$=: 250, ir | 636; *H* 660, 667 |
| 5,6-Dihydroxy-1-phenyl-3,4(1*H*,2*H*)-pyridazinedione | — | *H* 169 |
| 4,5-Diiodo-1,2-dimethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 163, 337 |
| 4,5-Diiodo-2-phenyl-3(2*H*)-pyridazinone | — | *H* 116, 331 |
| 3,6-Diiodopyridazine | nmr | 1638; *H* 287 |
| 3,6-Diisobutoxy-4-morpholinopyridazine | HCl: 147–148 | 637 |
| 4,5-Diisobutyrylpyridazine | liq, ir, ms, nmr | 79 |
| 3,6-Diisopropoxy-4-methylpyridazine | — | *H* 193 |
| 3,6-Diisopropoxy-4-morpholinopyridazine | 102–104, nmr | 640 |
| 3,6-Diisopropoxypyridazine | — | *H* 189 |
| 3,6-Diisopropoxypyridazine 1-oxide | liq, anal | 640 |
| 2,4-Diisopropyl-6-methyl-3(2*H*)-pyridazinone | liq, anal, nmr | 1676 |
| 4,5-Dimethoxy-3,6-dimethylpyridazine | 139/10, nmr; pic: 104–105 | 529 |
| 3,4-Dimethoxy-5,6-diphenylpyridazine | — | 814 |
| 3,6-Dimethoxy-4-methylpyridazine | 85–86, ir, ms, nmr | 358; *H* 192 |
| 3,4-Dimethoxy-6-methylpyridazine 1-oxide | — | *H* 197 |
| 3,5-Dimethoxy-6-methylpyridazine 1-oxide | — | *H* 196 |
| 3,6-Dimethoxy-4-methylpyridazine 1-oxide | — | *H* 197 |
| 3,6-Dimethoxy-4-methylpyridazine 2-oxide | — | *H* 197 |
| 2,6-Dimethoxy-4-methyl-3(2*H*)-pyridazinone | — | *H* 111 |
| 2,6-Dimethoxy-5-methyl-3(2*H*)-pyridazinone | — | *H* 90 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3,5-Dimethoxy-1-methyl-4(1*H*)-pyridazinone | 153–154 | 689 |
| 4,5-Dimethoxy-2-methyl-3(2*H*)-pyridazinone | 70–71, nmr | 689, 1213 |
| 3,6-Dimethoxy-5-nitro-4-pyridazinamine | — | *H* 193, 561 |
| 3,6-Dimethoxy-5-nitro-4-pyridazinamine 2-oxide | 187–188, ir, nmr | 1072 |
| 3,4-Dimethoxy-6-nitropyridazine 1-oxide | 155–156 | 757; *H* 197, 649 |
| 3,6-Dimethoxy-4-nitropyridazine 1-oxide | nmr | 952; *H* 197, 649 |
| 3,6-Dimethoxy-4-nitropyridazine 2-oxide | — | *H* 197 |
| 3,6-Dimethoxy-4-phenylpyridazine | — | *H* 192 |
| 4,5-Dimethoxy-2-phenyl-3(2*H*)-pyridazinone | nmr | 764, 1213; *H* 126 |
| 4,6-Dimethoxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 109 |
| 5,6-Dimethoxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 94 |
| 3,5-Dimethoxy-4-pyridazinamine | — | 1873 |
| 3,6-Dimethoxy-4-pyridazinamine | 178–180, nmr | 1072, 1900; *H* 192, 559 |
| 3,6-Dimethoxy-4-pyridazinamine 1-oxide | — | *H* 603 |
| 3,4-Dimethoxypyridazine | 55–57, nmr | 882; *H* 185 |
| 3,5-Dimethoxpyridazine | 70–71, nmr | 882; *H* 185 |
| 3,6-Dimethoxypyridazine | 104 or 105–106, nmr, $pK_a$, uv | 95, 450, 644, 673, 1128, 1183, 1548, 1638; *H* 176, 188 |
| 4,5-Dimethoxypyridazine | — | *H* 185 |
| 5,6-Dimethoxy-3-pyridazinecarbonitrile | — | *H* 186, 446 |
| 5,6-Dimethoxy-3-pyridazinecarbonitrile 2-oxide | — | *H* 197, 446 |
| 3,6-Dimethoxy-4-pyridazinecarboxylic acid | — | *H* 184, 436 |
| 3,6-Dimethoxy-4,5-pyridazinediamine | — | *H* 192, 599 |
| 3,4-Dimethoxypyridazine 1-oxide | 137–138 | 757; *H* 194 |
| 3,5-Dimethoxypyridazine 1-oxide | — | *H* 197 |
| 3,6-Dimethoxypyridazine 1-oxide | — | *H* 195 |
| 2,5-Dimethoxy-3(2*H*)-pyridazinone | — | *H* 85 |
| 2,6-Dimethoxy-3(2*H*)-pyridazinone | — | *H* 76 |
| 3,5-Dimethoxy-4(1*H*)-pyridazinone | 240–241 | 689 |
| 3,6-Dimethoxy-4-trimethylsilylpyridazine | liq, ir, ms, nmr | 358 |
| Dimethyl 4-acetyl-5-methoxy-3,6-pyridazinedicarboxylate | 76–77, ir, ms, nmr | 337, 346, 624 |
| Dimethyl 4-acetyl-5-trimethylsilyl-3,6-pyridazinedicarboxylate | 128, ir, ms, nmr | 11 |
| 1-γ-Dimethylaminobutyl-4,5-difluoro-2-methyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 338 |
| 4-Dimethylamino-2,6-dimethyl-3(2*H*)-pyridazinone | — | *H* 99 |
| 3-Dimethylamino-4,6-dinitropyridazine 1-oxide | 123–124 | 760 |
| 3-Dimethylamino-3,6-diphenylpyridazine | 108, ir, nmr | 1082 |
| 4-Dimethylamino-3,6-diphenylpyridazine | — | 226 |
| 4-Dimethylamino-2,6-diphenyl-3(2*H*)-pyridazinone | — | *H* 107, 589 |
| 4-Dimethylamino-6-ethoxy-2-methyl-3(2*H*)-pyridazinone | — | *H* 588 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 5-Dimethylamino-4-ethoxy-2-methyl-3(2*H*)-pyridazinone | 36–38 | 1269 |
| 5-Dimethylamino-6-ethoxy-2-methyl-3(2*H*)-pyridazinone | — | *H* 591 |
| 4-Dimethylamino-3-ethoxy-6-nitropyridazine 1-oxide | 108–109 | 760 |
| 4-Dimethylamino-6-ethoxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 109, 589 |
| 3-Dimethylamino-5-ethoxypyridazine | HCl: 181 | 637 |
| 4-Dimethylamino-3-ethyl-5,6-diphenylpyridazine 2-oxide | 147–148, ms | 311 |
| 2-β-Dimethylaminoethyl-4,6-diphenyl-3(2*H*)-pyridazinone | HCl: 155, ir, nmr | 888 |
| 2-β-Dimethylaminoethyl-5-ethyl-4-methyl-3(2*H*)-pyridazinone | — | *H* 125 |
| 2-β-Dimethylaminoethyl-4-ethyl-6-phenyl-3(2*H*)-pyridazinone | — | *H* 101 |
| 2-β-Dimethylaminoethyl-4-methyl-6-phenyl-3(2*H*)-pyridazinone | — | *H* 101 |
| 2-β-Dimethylaminoethyl-6-methyl-3(2*H*)-pyridazinone | — | *H* 71 |
| 4-Dimethylamino-2-ethyl-1-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 162, 595 |
| 2-β-Dimethylaminoethyl-6-phenyl-3(2*H*)-pyridazinone | — | *H* 72 |
| 4-Dimethylamino-6-ethyl-2-phenyl-3(2*H*)-pyridazinone | — | *H* 107 |
| 1-β-Dimethylaminoethyl-3,6(1*H*,2*H*)-pyridazinedione | ir, nmr, uv | 1975 |
| 3-Dimethylamino-5-hexyloxypyridazine | HCl: 155 | 637 |
| 3-Dimethylamino-6-hydrazinopyridazine | 2HCl: 233–235 or 238–240; PhCH=: 229–232 | 57, 1258 |
| 3-Dimethylamino-6-(3-hydroxyprop-1-ynyl)pyridazine | 108–110, nmr | 1385 |
| 3-Dimethylamino-6-iodopyridazine | 132–134, nmr | 1385; *H* 302, 303 |
| 4-Dimethylamino-6-isobutoxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 589 |
| 4-Dimethylamino-6-isopentyloxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 589 |
| 4-Dimethylamino-6-isopropoxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 589 |
| 3-Dimethylamino-4-methoxy-6-methylpyridazine | — | *H* 577 |
| 4-Dimethylamino-6-methoxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 108, 589 |
| 5-Dimethylamino-6-methoxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 94 |
| 3-Dimethylamino-5-methoxypyridazine | HCl: 165–166 or 167–168, nmr | 637 |
| 3-Dimethylamino-6-methoxypyridazine | 25, 88/0.4 | 137 |

*continued*

| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 4-Dimethylamino-3-methoxypyridazine | HCl: 163–164 | 637 |
| 3-Dimethylamino-6-methylaminopyridazine | — | *H* 600 |
| 4-Dimethylamino-3-methyl-5,6-diphenylpyridazine | 153–154, nmr | 311 |
| 4-Dimethylamino-6-methyl-3,5-diphenylpyridazine | 127–128, nmr | 311 |
| 2-(β-Dimethylamino-α-methylethyl)-5,6-dimethyl-3-oxo-2,3-dihydro-4-pyridazinecarbonitrile | — | *H* 130, 449 |
| 4-Dimethylaminomethyl-6-ethyl-2-phenyl-3(2*H*)-pyridazinone | — | *H* 106, 612 |
| 3-Dimethylaminomethyl-6-ethylpyridazine | uv | 1164 |
| 2-Dimethylaminomethyl-6-iodo-3(2*H*)-pyridazinone | — | *H* 71, 307 |
| 3-Dimethylamino-5-methyl-6-methylaminopyridazine | — | *H* 600 |
| 4-Dimethylaminomethyl-6-methyl-2-phenyl-3(2*H*)-pyridazinone | — | *H* 105, 612 |
| 3-Dimethylaminomethyl-6-methylpyridazine | uv | 1164 |
| 5-Dimethylamino-2-methyl-3-oxo-2,3-dihydro-4-pyridazinecarbaldehyde | 141–142; $H_2$ NN=: 127–128 | 1603 |
| 4-Dimethylamino-2-methyl-1-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 159, 959 |
| 4-Dimethylamino-6-methyl-2-phenyl-3(2*H*)-pyridazinone | — | *H* 106, 588 |
| 6-Dimethylamino-5-methyl-2-phenyl-3(2*H*)-pyridazinone | — | *H* 594 |
| 1-(γ-Dimethylamino-β-methylpropyl)-4,5-difluoro-2-methyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 166 |
| 3-Dimethylaminomethylpyridazine | 76–78/2, nmr; oxalate: 158–159; pic: 133–135 | 769, 1164 |
| 3-Dimethylamino-4-methylpyridazine | — | *H* 577 |
| 3-Dimethylamino-5-methylpyridazine | — | *H* 577 |
| 3-Dimethylamino-6-methylpyridazine | — | *H* 577, 578 |
| 1-Dimethylaminomethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 152 |
| 4-Dimethylamino-1-methyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 591 |
| 4-Dimethylamino-2-methyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 588 |
| 3-Dimethylamino-6-methyl-4(1*H*)-pyridazinone | — | *H* 577 |
| 5-Dimethylamino-2-methyl-3(2*H*)-pyridazinone | crude HCl: nmr | 641; *H* 571 |
| 6-Dimethylaminomethyl-3(2*H*)-pyridazinone | 104–105; HCl: 259–261 | 769; *H* 609 |
| 3-Dimethylaminomethyl-4(1*H*)-pyridazinone 2-oxide | — | *H* 610 |
| 6-Dimethylaminomethyl-3(2*H*)-pyridazinone 1-oxide | HCl: 229–230 | 769; *H* 195, 609 |
| 3-Dimethylamino-6-methylsulfonylpyridazine | — | *H* 577, 823 |
| 6-Dimethylamino-5-methylthio-2-phenyl-3(2*H*)-pyridazinone | — | *H* 594 |
| 4-(β-Dimethylamino-α-methylvinyl)-6-oxo-1-phenyl-1,6-dihydro-3,5-pyridazinedicarbonitrile | 205, ir, nmr | 1808 |
| 4-Dimethylamino-5-nitro-2-phenyl-3(2*H*)-pyridazinone | — | *H* 602 |
| 3-Dimethylamino-5-pentyloxypyridazine | HCl: 163–164 | 637 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3-Dimethylamino-6-phenoxypyridazine | — | 707 |
| 3-Dimethylamino-6-phenylazopyridazine | 174–176, nmr | 150 |
| 3-Dimethylamino-6-phenylethynylpyridazine | 146–148, nmr | 1385 |
| 4-Dimethylamino-2-phenyl-6-prop-1′-enyloxy-3(2*H*)-pyridazinone | — | *H* 589 |
| 4-Dimethylamino-2-phenyl-6-propoxy-3(2*H*)-pyridazinone | — | *H* 589 |
| 3-Dimethylamino-6-phenylpyridazine | 115–116, nmr, p$K_a$ | 94, 1385; *H* 577 |
| 4-Dimethylamino-3-phenylpyridazine | 2-$MeClO_4$: 121, ir, nmr; 2-$PhClO_4$: 180–182, ir, nmr | 109, 277 |
| 4-Dimethylamino-6-phenylpyridazine | — | 277 |
| 4-Dimethylamino-1-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 156 |
| 4-Dimethylamino-2-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 589 |
| 3-Dimethylamino-6-phenyl-4(1*H*)-pyridazinone | 231–232, nmr | 1041 |
| 4-Dimethylamino-2-phenyl-3(2*H*)-pyridazinone | 49–50, ir, nmr | 109; *H* 84, 588 |
| 4-Dimethylamino-6-phenyl-3(2*H*)-pyridazinone | 186–188 | 1041 |
| 5-Dimethylamino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 85, 592 |
| 6-Dimethylamino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 80, 594 |
| 3-Dimethylamino-5-propoxypyridazine | HCl: 156–157 | 637 |
| 2-γ-Dimethylaminopropyl-4-methyl-6-phenyl-3(2*H*)-pyridazinone | — | *H* 101 |
| 2-γ-Dimethylaminopropyl-6-methyl-3(2*H*)-pyridazinone | — | *H* 72 |
| 2-γ-Dimethylaminopropyl-6-phenyl-3(2*H*)-pyridazinone | — | *H* 72 |
| 2-γ-Dimethylaminopropyl-3(2*H*)-pyridazinone | HCl: 145–146 | 1760 |
| 3-Dimethylaminopyridazine | p$K_a$; pic: 181–182 | 94, 1796; *H* 577 |
| 4-Dimethylaminopyridazine | pic: 219–220 | 1796; *H* 584 |
| 3-Dimethylamino-4(1*H*)-pyridazinone | HCl: 140–143 | 640 |
| 4-Dimethylamino-3(2*H*)-pyridazinone | 147–148 or 150–151, ir, nmr | 109, 641, 1794 |
| 5-Dimethylamino-3(2*H*)-pyridazinone | HCl: 235 | 641 |
| 6-Dimethylamino-3(2*H*)-pyridazinone | 192–194, p$K_a$ | 94 |
| 6-Dimethylamino-4(1*H*)-pyridazinone | HCl: 255, nmr | 637 |
| 4-Dimethylamino-3,5,6-triphenylpyridazine | 163–165, nmr | 311 |
| 4-Dimethylamino-3,5,6-triphenylpyridazine 2-oxide | 241–243, ms | 311 |
| 4-β-Dimethylaminovinyl-3,6-diphenylpyridazine | 183–184 | 43 |
| 4-β-Dimethylaminovinyl-2-methoxy-6-methylpyridazine | liq, nmr | 1642 |
| 4-β-Dimethylaminovinyl-6-oxo-1-phenyl-1,6-dihydro-3,5-pyridazinedicarbonitrile | 198, ir, nmr | 1830 |
| 3-β-Dimethylaminovinylpyridazine | 52–54, nmr | 1642 |
| Dimethyl 4-anilino-3,6-pyridazinedicarboxylate | 191–192, ir, ms, nmr | 507 |
| Dimethyl 4-benzoyl-5-trimethylsilyl-3,6-pyridazinedicarboxylate | 108, ir, ms, nmr | 11 |
| Dimethyl 4-benzyloxy-3,6-pyridazinedicarboxylate | 126–127, ir, ms, nmr | 337 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| Dimethyl 4,5-bistrimethylsilyl-3,6-pyridazinedicarboxylate | 149, ms, nmr | 1208 |
| Dimethyl 4-butyl-3,6-pyridazinedicarboxylate | — | *H* 445 |
| Dimethyl 6-butyl-3,5-pyridazinedicarboxylate | — | 440 |
| Dimethyl 4-butyl-5-trimethylsilyl-3,6-pyridazinedicarboxylate | $n_D^{20°}$ 1.5045, ms, nmr | 11 |
| 2-(Dimethylcarbamoyl)methyl-5,6-dimethyl-3-oxo-2,3-dihydro-4-pyridazinecarbonitrile | — | *H* 130, 449 |
| 2-(Dimethylcarbamoyl)methyl-5,6-dimethyl-3(2*H*)-pyridazinone | — | *H* 92 |
| 2-(Dimethylcarbamoyl)methyl-6-methyl-3(2*H*)-pyridazinone | — | *H* 79 |
| 2-(Dimethylcarbamoyl)methyl-6-phenyl-3(2*H*)-pyridazinone | — | *H* 79 |
| 1-(Dimethylcarbamoyl)methyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 154 |
| Dimethyl 4-diethylamino-5-methyl-3,6-pyridazinedicarboxylate | — | *H* 585 |
| Dimethyl 4,5-dimethyl-3,6-pyridazinedicarboxylate | 101–102, ir, ms, nmr | 337 |
| *N*,*N*-Dimethyl-3,6-dioxo-1,2,3,6-tetrahydro-1-pyridazinesulfonamide | — | *H* 158 |
| 4,5-Dimethyl-3,6-diphenylpyridazine | 208–210 | 319 |
| Dimethyl 3,6-diphenyl-4,5-pyridazinedicarboxylate | 214–216, nmr | 41 |
| Dimethyl 4,5-diphenyl-3,6-pyridazinedicarboxylate | 196–197, nmr | 41 |
| Dimethyl 5,6-diphenyl-3,4-pyridazinedicarboxylate | 108, ir, nmr, uv | 1242 |
| Dimethyl 4-ethoxy-5-formamido-3,6-pyridazinedicarboxylate | — | 1031 |
| Dimethyl 4-ethoxy-3,6-pyridazinedicarboxylate | — | *H* 186, 445 |
| Dimethyl 4-ethyl-5-methyl-3,6-pyridazinedicarboxylate | 50–52, ir, ms, nmr | 337 |
| Dimethyl 4-ethyl-5-trimethylsilyl-3,6-pyridazinedicarboxylate | $n_D^{20°}$ 1.5081, ms, nmr | 11 |
| Dimethyl 4-formamido-3,6-pyridazinedicarboxylate | — | 1031 |
| Dimethyl 4-hexyl-5-trimethylsilyl-3,6-pyridazinedicarboxylate | ms, $n_D^{20°}$ 1.5003, nmr | 11 |
| 4-$\beta$,$\beta$-Dimethylhydrazino-2-methyl-5-methylsulfonyl-3(2*H*)-pyridazinone | 173–175 | 533 |
| Dimethyl 4-hydroxy-5-oxo-2,5-dihydro-3,6-pyridazinedicarboxylate | 218–219, ir, nmr, uv | 406 |
| Dimethyl 4-$\gamma$-hydroxypropyl-3,6-pyridazinedicarboxylate | liq (?), ir, ms, nmr | 352 |
| Dimethyl 4-methoxy-2-methyl-2,5-dihydro-3,6-pyridazinedicarboxylate | 98–99, ir, nmr, uv | 406 |
| Dimethyl 4-methoxy-3,6-pyridazinedicarboxylate | 104–105, ir, ms, nmr | 283, 337, 1509 |
| Dimethyl 4-methylamino-3,6-pyridazinedicarboxylate | 164, ir, ms, nmr | 507 |
| Dimethyl 4-methyl-3,6-pyridazinedicarboxylate | — | 508, 875; *H* 445 |
| 5,6-Dimethyl-3-methylthio-4-pyridazinecarbonitrile | — | *H* 447, 806 |
| Dimethyl 4-methyl-5-trimethylsilylethynyl-3,6-pyridazinedicarboxylate | 72, ir, ms, nmr | 11 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| Dimethyl 4-methyl-5-trimethylsilyl-3,6-pyridazinedicarboxylate | ms, $n_D^{20}$ 1.5152, nmr | 11 |
| 4,5-Dimethyl-2-(β-morpholinoethyl-6-phenyl-3(2*H*)-pyridazinone | — | *H* 130 |
| 3,6-Dimethyl-5-morpholinomethyl-4(1*H*)-pyridazinone 2-oxide | 208–209, nmr | 791 |
| 3,6-Dimethyl-4-nitropyridazine 1,2-dioxide | 158–159, ir, nmr | 527 |
| 3,4-Dimethyl-5-nitropyridazine 2-oxide | — | *H* 649 |
| 3,4-Dimethyl-6-nitropyridazine 1-oxide | — | *H* 649 |
| 3,6-Dimethyl-4-nitropyridazine 1-oxide | 117–118 | 527, 794; *H* 649 |
| 3,6-Dimethyl-4-nitropyridazine 2-oxide | 81–83 | 527, 791; *H* 649 |
| 2,6-Dimethyl-4-nitro-3(2*H*)-pyridazinone | — | *H* 99 |
| 3,6-Dimethyl-1-nitroso-4(1*H*)-pyridazinone | — | *H* 134 |
| 1,5-Dimethyl-6-oxo-1,6-dihydro-4-pyridazinecarbonitrile | 85–86, ir, ms, nmr | 231 |
| 2,4-Dimethyl-5-oxo-2,5-dihydro-3-pyridazinecarbonitrile | 80–81, ir | 772 |
| 2,5-Dimethyl-3-oxo-2,3-dihydro-4-pyridazinecarbonitrile | 136–137, ir, ms, nmr | 231 |
| 5,6-Dimethyl-3-oxo-2,3-dihydro-4-pyridazinecarbonitrile | — | *H* 127, 448 |
| 1,*N*-Dimethyl-6-oxo-1,6-dihydro-3-pyridazinecarboxamide | — | *H* 70, 437 |
| 1,3-Dimethyl-6-oxo-1,6-dihydro-4-pyridazinecarboxylic acid | — | *H* 90, 440 |
| 2,6-Dimethyl-3-oxo-2,3-dihydro-4-pyridazinecarboxylic acid | — | *H* 99, 441 |
| 5,6-Dimethyl-3-oxo-2,3-dihydro-4-pyridazinecarboxylic acid | — | *H* 127, 441 |
| Dimethyl 4-oxo-1,4-dihydro-3,6-pyridazinedicarboxylate | — | 1509 |
| 2,3-Dimethyl-5-oxo-6-phenyl-2,5-dihydro-4-pyridazinecarbonitrile | 180, ir | 772 |
| 2,4-Dimethyl-5-oxo-6-phenyl-2,5-dihydro-3-pyridazinecarbonitrile | 180, ir | 772 |
| 2,6-Dimethyl-3-oxo-5-phenyl-2,3-dihydro-4-pyridazinecarbonitrile | — | *H* 129, 449 |
| 5,6-Dimethyl-3-oxo-2-phenyl-2,3-dihydro-4-pyridazinecarbonitrile | 131–132, ir, nmr | 1731 |
| 6,*N*-Dimethyl-4-oxo-1-phenyl-1,4-dihydro-3-pyridazinecarboxamide | — | *H* 135, 442 |
| *N*,*N*-Dimethyl-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxamide | — | *H* 80, 438 |
| 3,6-Dimethyl-5-oxo-2-phenyl-2,5-dihydro-4-pyridazinecarboxylic acid | — | *H* 136, 443 |
| Dimethyl 4-pentyl-5-trimethylsilyl-3,6-pyridazinedicarboxylate | ms, $n_D^{20}$ 1.5029, nmr | 11 |
| Dimethyl 3-phenethyl-4,5-pyridazinedicarboxylate | 35–39, ir, ms, nmr | 1791 |
| 3,4-Dimethyl-6-phenylpyridazine | 103–104, nmr | 394 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3,6-Dimethyl-4-phenylpyridazine | 165–170/2, nmr; pic: 136–137 | 823 |
| Dimethyl 4-phenyl-3,6-pyridazinedicarboxylate | 92–93 or 95–96, ir, ms, nmr | 337, 1568; *H* 445 |
| Dimethyl 5-phenyl-3,4-pyridazinedicarboxylate | — | *H* 445 |
| 1,4-Dimethyl-2-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 159 |
| 1,5-Dimethyl-2-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 159 |
| 4,5-Dimethyl-1-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 156 |
| 2,4-Dimethyl-6-phenyl-3(2*H*)-pyridazinethione | 94–95 or 104, ir, nmr, uv | 371, 413 |
| 1,6-Dimethyl-3-phenyl-4(1*H*)-pyridazinone | 143–144, ir | 772 |
| 2,4-Dimethyl-6-phenyl-3(2*H*)-pyridazinone | 102 | 1104, 1253; *H* 99 |
| 3,6-Dimethyl-1-phenyl-4(1*H*)-pyridazinone | 245, ir, nmr | 110; *H* 136 |
| 4,6-Dimethyl-2-phenyl-3(2*H*)-pyridazinone | — | *H* 105 |
| 1,2-Dimethyl-4-phenylsulfinyl-3,6(1*H*,2*H*)-pyridazinedione | 150–151, nmr | 1672 |
| 5,6-Dimethyl-3-phenylthio-4-pyridazinecarbonitrile | 123–125, ir, nmr, uv | 146 |
| 1,2-Dimethyl-4-phenylthio-3,6(1*H*,2*H*)-pyridazindione | 128–129, nmr | 1672 |
| 3,6-Dimethyl-5-piperidinomethyl-4(1*H*)-pyridazinone 2-oxide | HCl: 187–188, ir, nmr | 791 |
| 5,6-Dimethyl-3-piperidino-4-pyridazinecarbonitrile | — | *H* 447 |
| 3,6-Dimethyl-4-piperidinopyridazine 1-oxide | — | *H* 604 |
| 3,6-Dimethyl-4-piperidinopyridazine 2-oxide | — | *H* 604 |
| Dimethyl 4-propyl-5-trimethylsilyl-3,6-pyridazinedicarboxylate | ms, $n_D^{20°}$ 1.5061, nmr | 11 |
| 3,6-Dimethyl-4-pyridazinamine | — | 796; *H* 558 |
| 4,6-Dimethyl-3-pyridazinamine | — | *H* 556 |
| 5,6-Dimethyl-3-pyridazinamine | 220–221, ir | 560, 1160; *H* 554 |
| 5,6-Dimethyl-4-pyridazinamine | — | *H* 558 |
| 3,6-Dimethyl-4-pyridazinamine 1-oxide | 281 | 796; *H* 603 |
| 3,6-Dimethyl-4-pyridazinamine 2-oxide | 190–191, ir, nmr | 796 |
| 3,4-Dimethylpyridazine | 43–44, ms, nmr; pic: 174–175 | 211, 762, 823 |
| 3,5-Dimethylpyridazine | ms, nmr | 211, 762 |
| 3,6-Dimethylpyridazine | 77/2 or 104–107/11, complexes, ir, ms, nmr | 211, 230, 823, 847, 894, 1133 |
| 4,5-Dimethylpyridazine | 53–55, ms, nmr; 1-$PhPF_6$: — | 211, 583, 762 |
| 3,6-Dimethyl-4-pyridazinecarbonitrile | 102–103, ir | 772 |
| 3,6-Dimethyl-4,5-pyridazinedicarbohydrazide | 160–180 then >300, ir, nmr | 927 |
| 3,6-Dimethyl-4,5-pyridazinedicarbonitrile | 118–120 or 178–179, ir, nmr, uv | 772, 927 |
| 3,6-Dimethyl-4,5-pyridazinedicarboxamide | — | *H* 445 |
| 3,6-Dimethyl-4,5-pyridazinedicarboximide | 303 | 927; *H* 445 |
| Dimethyl 3,6-pyridazinedicarboxylate | 202, ir, nmr | 1077; *H* 445 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| Dimethyl 4,5-pyridazinedicarboxylate | 52, ir, nmr | 736 |
| 3,6-Dimethyl-4,5-pyridazinedicarboxylic acid | — | *H* 445 |
| 3,6-Dimethyl-4,5-pyridazinedicarboxylic anhydride | 233–235, ir, uv | 927 |
| 1,2-Dimethyl-3,6(1*H*,2*H*)-pyridazinedione | 136–137 or 140–142, ir, nmr, $pK_a$, uv, xl st | 95, 140, 332, 417, 1128, 1548; *H* 163 |
| 1,4-Dimethyl-3,6(1*H*,2*H*)-pyridazinedione | 262–263, ir, nmr | 789 |
| 4,5-Dimethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 151 |
| 3,6-Dimethylpyridazine 1,2-dioxide | 215–216, ir, ms, nmr | 106, 190, 321 |
| 1,2-Dimethyl-3,6(1*H*,2*H*)-pyridazinedithione | 209–210, $pK_A$, uv | 95, 672 |
| 3,6-Dimethylpyridazine 1-oxide | complex | 260, 527 |
| 2,6-Dimethyl-3(2*H*)-pyridazinethione | 102, nmr | 413; *H* 797 |
| 1,6-Dimethylpyridazinium 3-olate | 171–172, ir, ms, nmr | 668 |
| 2,6-Dimethyl-3(2*H*)-pyridazinone | — | 397, 693, 1594; *H* 70 |
| 3,6-Dimethyl-4(1*H*)-pyridazinone | nmr | 446; *H* 132 |
| 4,5-Dimethyl-3(2*H*)-pyridazinone | — | *H* 125 |
| 4,6-Dimethyl-3(2*H*)-pyridazinone | 129; HCl: 172, nmr | 503, 1319; *H* 97 |
| 5,6-Dimethyl-3(2*H*)-pyridazinone | — | *H* 88 |
| 2,5-Dimethyl-3(2*H*)-pyridazinone 1-oxide | 195–196, ir, nmr | 789 |
| 2,6-Dimethyl-3(2*H*)-pyridazinone 1-oxide | 110–111 | 789 |
| 3,6-Dimethyl-4(1*H*)-pyridazinone 2-oxide | 257–258, nmr | 791; *H* 196 |
| 5,6-Dimethyl-3(2*H*)-pyridazinone 1-oxide | — | *H* 196 |
| 2-*β*-[*N*,*N*-Dimethyl (thiocarbamoyl)]ethyl-6-phenyl-3(2*H*)-pyridazinone | 132–134, ir, nmr | 1288 |
| 5,6-Dimethyl-3-thioxo-2,3-dihydro-4-pyridazinecarbonitrile | — | *H* 447, 798 |
| 1,2-Dimethyl-6-thioxo-1,6-dihydro-3(2*H*)-pyridazinone | 143–145, $pK_a$, uv | 95, 672 |
| Dimethyl 4-trimethylsilyl-3,6-pyridazinedicarboxylate | 52–53, ms, nmr | 1208 |
| Dimethyl 4-trimethylsilyl-5-trimethylsilylethynyl-3,6-pyridazinedicarboxylate | 72, ir, nmr | 11 |
| Dimethyl 4-valeryl-3,6-pyridazinedicarboxylate | 68–69, ir, ms, nmr | 352 |
| Dimethyl 4-vinyl-3,6-pyridazinedicarboxylate | liq (?), ir, ms, nmr | 352 |
| 5,6-Dimorpholino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 194, 602 |
| 3,5-Dimorpholinopyridazine | HCl: 258–260, nmr | 637; *H* 601 |
| 4,5-Dineopentylpyridazine | anal, nmr | 1518 |
| 3,5-Dinitro-6-piperidinopyridazine 2-oxide | 141–142 | 760 |
| 4,6-Dinitro-3-pyridazinamine 1-oxide | 209–210 | 760 |
| 3,6-Dioxo-1-phenyl-1,2,3,6-tetrahydro-4-pyridazinecarbaldehyde | — | *H* 156, 396 |
| 3,6-Dioxo-1,2,3,6-tetrahydro-4-pyridazinecarbaldehyde | — | *H* 396 |
| 3,6-Diphenethylpyridazine | 96–97, ms, nmr | 822 |
| 3,6-Diphenoxypyridazine | 140–141, uv; 1-MeI: — | 690, 1183; *H* 190 |
| 3,6-Diphenyl-4,5-didehydropyridazine | unstable | 476 |
| 5,6-Diphenyl-3-phenylhydrazino-4-pyridazinecarbonitrile | 230–231, ir | 1398, 1726 |
| 5,6-Diphenyl-4-phenylsulfonyl-3(2*H*)-pyridazinone | 254–256 | 1272 |

*continued*

| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 5,6-Diphenyl-3-phenylthio-4-pyridazinecarbonitrile | 220, ir | 1398, 1726 |
| 5,6-Diphenyl-4-phenylthio-3(2*H*)-pyridazinone | — | 809 |
| 2,6-Diphenyl-4-piperidinomethyl-3(2*H*)-pyridazinone | — | *H* 106 |
| 4,6-Diphenyl-3-piperidinopyridazine | 139, ir, nmr | 1081 |
| 2,6-Diphenyl-4-piperidino-3(2*H*)-pyridazinone | — | *H* 107, 590 |
| 5,6-Diphenyl-4-piperidino-3(2*H*)-pyridazinone | — | 809 |
| 5,6-Diphenyl-4-propionyl-3(2*H*)-pyridazinone | 163–165, ir, uv | 373 |
| 3,5-Diphenyl-1-propyl-4(1*H*)-pyridazinone | 90–93, nmr | 249 |
| 3,6-Diphenyl-4-pyridazinamine | 292–295, nmr | 451, 588; *H* 558 |
| 4,6-Diphenyl-3-pyridazinamine | 184–186 | 1885; *H* 556 |
| 3,4-Diphenylpyridazine | 106–108 or 108–109, nmr | 12, 473, 634 |
| 3,5-Diphenylpyridazine | 137 to 145, ir, nmr | 404, 563, 1150, 1153, 1795, 2052, 2093 |
| 3,6-Diphenylpyridazine | 212 to 225, complexes, ir, ms, nmr | 29, 38, 58, 226, 230, 268, 278, 319, 321, 345, 409, 472, 568, 581, 607, 722, 823, 896, 918, 1071, 1148, 1154, 1174, 1491, 1492, 1569, 1883, 1922, 2073 |
| 3,6-Diphenyl-4-pyridazinecarbaldehyde | $Me_2$acetal: 79–81 | 319 |
| 3,6-Diphenyl-4-pyridazinecarbonitrile | — | *H* 447 |
| 3,6-Diphenyl-4-pyridazinecarboxylic acid | — | *H* 436 |
| 3,6-Diphenyl-4,5-pyridazinediamine | 259–362, ir, ms, nmr | 1358; *H* 598 |
| 3,6-Diphenyl-4,5-pyridazinedicarboxylic acid | — | *H* 445 |
| 1,4/1,5-Diphenyl-3,6(1*H*,2*H*)-pyridazinedione | 310–313 | 462 |
| 5,6-Diphenyl-3,4(1*H*,2*H*)-pyridazinedione | 234–235 or 239–240, ir, nmr | 795, 942, 1496 |
| 3,6-Diphenylpyridazine 1,2-dioxide | 176–177 or 258, ir, ms, nmr | 106, 190, 321, 411 |
| 3,6-Diphenylpyridazine 1-oxide | 198–199, complex | 260, 321, 581 |
| 2,6-Diphenyl-3(2*H*)-pyridazinethione | 190 | 413; *H* 798 |
| 3,5-Diphenyl-4(1*H*)-pyridazinethione | 222–224, nmr | 249 |
| 4,6-Diphenyl-3(2*H*)-pyridazinethione | 208 | 1264 |
| 5,6-Diphenyl-3(2*H*)-pyridazinethione | 240 | 659, 1264; *H* 798 |
| 2,6-Diphenyl-3(2*H*)-pyridazinone | 147–149 | 566; *H* 80 |
| 3,5-Diphenyl-4(1*H*)-pyridazinone | 325 to 339, ir, nmr | 109, 208, 249, 451; *H* 133 |
| 3,6-Diphenyl-4(1*H*)-pyridazinone | — | 588; *H* 133 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 4,5-Diphenyl-3(2*H*)-pyridazinone | 183 or 184, ir, nmr | 1082, 1264; *H* 97 |
| 5,6-Diphenyl-3(2*H*)-pyridazinone | 178 | 1264; *H* 89 |
| 3,4-Diphenyl-6-styrylpyridazine | 185–186 | 634 |
| 5,6-Diphenyl-3-thioxo-2,3-dihydro-4-pyridazinecarbonitrile | 250 to >300, ir, nmr | 1056, 1390, 1398, 1726, 1913 |
| 5,6-Diphenyl-3-thioxo-2,3-dihydro-4-pyridazinecarboxylic acid | 213–215, ir | 389 |
| 3,4-Diphenyl-5-trimethylsilylpyridazine | 152, ms, nmr | 12 |
| 3,5-Dipiperidinopyridazine | HCl: 215–217, nmr | 640 |
| 3,6-Dipiperidinopyridazine | — | *H* 600 |
| 4,6-Dipiperidinopyridazine | 92–94, ir, nmr | 544 |
| 3,6-Dipiperidinopyridazine 1-oxide | — | *H* 604 |
| 3,6-Dipiperidino-4(1*H*)-pyridazinone | 303, nmr | 640 |
| 3,6-Dipropionyl-4,5-dihydro-4,5-pyridazinedione 1,2-dioxide | 89–91 | 505 |
| 4,5-Dipropionylpyridazine | ms | 79 |
| 3,6-Dipropoxypyridazine | — | *H* 189 |
| 3,6-Dipropoxypyridazine 1-oxide | — | *H* 195 |
| 3,5-Dipropyl-4(1*H*)-pyridazinone | — | *H* 133 |
| 3,6-Distyrylpyridazine 1,2-dioxide | 254 | 527 |
| 1,2-Divinyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 163 |
| 6-Ethanesulfonyloxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 81 |
| 6-Ethanesulfonyloxy-3(2*H*)-pyridazinone | — | *H* 69 |
| 4-Ethoxy-3,6-bismethylthiopyridazine | — | 353 |
| 2-γ-Ethoxycarbonylallyl-4-methyl-6-phenyl-3(2*H*)-pyridazinimine | HBr: 187, nmr | 160 |
| 5-Ethoxycarbonyl-3,6-dimethyl-4-pyridazinecarboxylic acid | — | *H* 445 |
| 2-β-Ethoxycarbonylethyl-4,6-diphenyl-3(2*H*)-pyridazinone | 98, ir, nmr | 1343 |
| 2-α-Ethoxycarbonylethyl-6-methyl-3(2*H*)-pyridazinone | — | *H* 78 |
| 2-β-Ethoxycarbonylethyl-6-methyl-3(2*H*)-pyridazinone | — | *H* 73 |
| 2-α-Ethoxycarbonylethyl-5-morpholino-3(2*H*)-pyridazinone | anal, ir, nmr | 534 |
| 2-β-Ethoxycarbonylethyl-6-phenyl-3(2*H*)-pyridazinone | 199–202/0.9, nmr | 118, 1288 |
| 2-β-Ethoxycarbonylethyl-3(2*H*)-pyridazinimine | — | *H* 551 |
| 6-β-Ethoxycarbonylethyl-3(2*H*)-pyridazinone | — | *H* 67 |
| 2-Ethoxycarbonylmethyl-4,6-dimethyl-3(2*H*)-pyridazinone | — | *H* 101 |
| 2-Ethoxycarbonylmethyl-5,6-dimethyl-3(2*H*)-pyridazinone | — | *H* 92 |
| 4-Ethoxycarbonylmethyl-3,6-diphenylpyridazine | 139–140, nmr | 34, 222 |
| 2-Ethoxycarbonylmethyl-4,6-diphenyl-3(2*H*)-pyridazinone | 160, ir, nmr | 1343 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 2-(α-Ethoxycarbonyl-α-methylethyl)-6-methyl-3(2*H*)-pyridazinone | — | *H* 78 |
| 2-Ethoxycarbonylmethyl-6-ethyl-3(2*H*)-pyridazinone | — | *H* 77 |
| 2-Ethoxycarbonylmethyl-6-ethylsulfonyl-3(2*H*)-pyridazinone | — | *H* 78, 824 |
| 2-Ethoxycarbonylmethyl-6-isopropylsulfonyl-3(2*H*)-pyridazinone | — | *H* 78, 824 |
| 2-Ethoxycarbonylmethyl-4-methyl-3(2*H*)-pyridazinone | — | *H* 84 |
| 2-Ethoxycarbonylmethyl-6-methyl-3(2*H*)-pyridazinone | — | *H* 77 |
| 2-Ethoxycarbonylmethyl-6-methylsulfonyl-3(2*H*)-pyridazinone | — | *H* 78, 824 |
| 2-Ethoxycarbonylmethyl-5-morpholino-4-nitro-3(2*H*)-pyridazinone | anal, ir, nmr | 534 |
| 2-Ethoxycarbonylmethyl-5-morpholino-3(2*H*)-pyridazinone | 144, ir, nmr | 534 |
| 4-Ethoxycarbonylmethyl-1-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 156 |
| 5-Ethoxycarbonyl-3-methyl-2-phenylpyridazinium-4-olate | 168, nmr | 500 |
| 2-Ethoxycarbonylmethyl-6-phenyl-3(2*H*)-pyridazinone | 99–100, nmr | 118, 775; *H* 78 |
| 3-Ethoxycarbonylmethyl-6-phenyl-4(1*H*)-pyridazinone | 200–201, ir, nmr | 1489 |
| 1-Ethoxycarbonylmethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 153 |
| 4-Ethoxycarbonylmethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 151 |
| 2-Ethoxycarbonylmethyl-3(2*H*)-pyridazinimine | — | *H* 551 |
| 2-Ethoxycarbonylmethyl-3(2*H*)-pyridazinone | — | *H* 65 |
| 2-Ethoxycarbonylmethyl-4,5,6-trimethyl-3(2*H*)-pyridazinone | — | *H* 130 |
| 2-γ-Ethoxycarbonylpropyl-4,6-diphenyl-3(2*H*)-pyridazinone | 54, ir, nmr | 1343 |
| 3-γ-Ethoxycarbonylpropyl-4-methyl-6-phenyl-3(2*H*)-pyridazinone | crude: liq, ir, nmr; HBr: 260 | 1309 |
| 3-β-Ethoxycarbonylvinylpyridazine | 47–48, ir, nmr | 1668 |
| 4-β-Ethoxycarbonylvinylpyridazine | 85, ir, nmr | 1668 |
| 4-β-Ethoxycarbonylvinylpyridazine 1-oxide | 107–109, ir, nmr | 1906 |
| 4-β-Ethoxycarbonylvinylpyridazine 2-oxide | 142–144, ir, nmr | 1906 |
| 3-Ethoxy-6-chloro-5-nitro-4-pyridazinamine | — | *H* 561 |
| 2-Ethoxy-5,6-dimethoxy-3(2*H*)-pyridazinone | — | *H* 90 |
| 3-Ethoxy-5,6-dimethyl-4-pyridazinecarbonitrile | — | *H* 184, 447 |
| 4-Ethoxy-2,6-dimethyl-3(2*H*)-pyridazinone | — | *H* 99 |
| 3-Ethoxy-4,6-dinitropyridazine 1-oxide | 113–115 | 757 |
| 3-Ethoxy-5,6-diphenylpyridazine | — | *H* 184 |
| 4-Ethoxy-3,6-diphenylpyridazine | 102–104 | 451; *H* 186 |
| 3-Ethoxy-6-ethylaminopyridazine | — | *H* 177 |
| 3-Ethoxy-6-ethylaminopyridazine 1-oxide | — | *H* 604 |
| 3-Ethoxy-6-ethylaminopyridazine 2-oxide | — | *H* 604 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 6-β-Ethoxyethyl-4-methylamino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 107 |
| 4-Ethoxy-2-ethyl-5-morpholino-3(2*H*)-pyridazinone | 64–66 | 1269 |
| 6-Ethoxy-2-ethyl-5-morpholino-3(2*H*)-pyridazinone | — | 1725 |
| 4-Ethoxy-2-ethyl-1-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 162 |
| 6-Ethoxy-2-ethyl-3(2*H*)-pyridazinone | — | *H* 71 |
| 3-Ethoxy-6-hydrazinopyridazine | 94; $Me_2C$=: 150–153, ir | 636, 1258; *H* 667 |
| 4-Ethoxy-2-β-hydroxyethyl-5-morpholino-3(2*H*)-pyridazinone | 101–102, ir | 534 |
| 3-Ethoxy-6-hydroxymethylpyridazine | — | *H* 177 |
| 4-Ethoxy-5-hydroxy-2-methyl-3(2*H*)-pyridazinone | 146–147, ir, nmr | 531 |
| 6-Ethoxy-2-hydroxy-5-methyl-3(2*H*)-pyridazinone | — | *H* 90 |
| 4-Ethoxy-5-hydroxy-2-phenyl-3(2*H*)-pyridazinone | 167–168, ir, nmr | 764 |
| 6-Ethoxy-2-hydroxy-3(2*H*)-pyridazinone | — | *H* 76 |
| 3-Ethoxy-6-iodopyridazine | — | *H* 177, 287 |
| 6-Ethoxy-4-isopropylamino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 588 |
| 4-Ethoxy-2-isopropyl-5-morpholino-3(2*H*)-pyridazinone | 147–149/0.1 | 1269 |
| 4-Ethoxy-5-mercapto-2-methyl-3(2*H*)-pyridazinone | 67–69, ir, uv | 1896 |
| 4-Ethoxy-5-mercapto-2-phenyl-3(2*H*)-pyridazinone | 104–106, ir, uv | 1896 |
| 5-Ethoxy-4-methanesulfonyloxy-2-methyl-3(2*H*)-pyridazinone | 85–86, ir, ms | 531 |
| 5-Ethoxy-4-methanesulfonyloxy-2-phenyl-3(2*H*)-pyridazinone | 146–147, ir, ms | 531 |
| 3-Ethoxy-6-methoxy-4-methylpyridazine 1-oxide | — | *H* 198 |
| 2-Ethoxy-6-methoxy-5-methyl-3(2*H*)-pyridazinone | — | *H* 90 |
| 6-Ethoxy-2-methoxy-5-methyl-3(2*H*)-pyridazinone | — | *H* 90 |
| 5-Ethoxy-4-methoxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 126 |
| 6-Ethoxy-4-methoxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 109 |
| 3-Ethoxy-6-methoxypyridazine | — | 673 |
| 3-Ethoxy-6-methoxypyridazine 1-oxide | — | *H* 195 |
| 2-Ethoxy-6-methoxy-3(2*H*)-pyridazinone | — | *H* 76 |
| 6-Ethoxy-2-methoxy-3(2*H*)-pyridazinone | — | *H* 76 |
| 6-Ethoxy-4-methylamino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 109, 588 |
| 4-Ethoxy-2-methyl-5-methylamino-3(2*H*)-pyridazinone | 80–81 | 1269 |
| 4-Ethoxy-2-methyl-5-morpholino-3(2*H*)-pyridazinone | 89–91 | 1269 |
| 5-Ethoxy-2-methyl-4-morpholino-3(2*H*)-pyridazinone | — | 1725 |
| 6-Ethoxy-2-methyl-4-morpholino-3(2*H*)-pyridazinone | — | 1725 |
| 6-Ethoxy-2-methyl-5-morpholino-3(2*H*)-pyridazinone | — | 1725 |
| 3-Ethoxy-6-methyl-5-nitro-4-pyridazinamine | — | *H* 561 |
| 3-Ethoxy-4-methyl-6-nitropyridazine 1-oxide | — | *H* 198, 649 |
| 3-Ethoxy-6-methyl-4-nitropyridazine 1-oxide | — | *H* 198 |
| 3-Ethoxy-4-methyl-6-phenylpyridazine | — | *H* 182 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 4-Ethoxy-2-methyl-1-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 160 |
| 4-Ethoxy-6-methyl-2-phenyl-3(2*H*)-pyridazinone | — | *H* 106 |
| 4-Ethoxy-2-methyl-5-piperidino-3(2*H*)-pyridazinone | 140–141/0.05 | 1269 |
| 3-Ethoxy-6-methyl-4-pyridazinamine | — | *H* 558 |
| 3-Ethoxy-6-methylpyridazine | — | *H* 177 |
| 3-Ethoxy-6-methyl-4,5-pyridazinediamine | — | *H* 599 |
| 1-Ethoxymethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 153 |
| 5-Ethoxy-2-methyl-3,4(1*H*,2*H*)-pyridazinedione | 138–139, ir, nmr | 531 |
| 3-Ethoxy-4-methylpyridazine 1-oxide | — | *H* 194 |
| 3-Ethoxy-6-methylpyridazine 1-oxide | — | *H* 195 |
| 5-Ethoxy-2-methyl-3(2*H*)-pyridazinone | 103–104, ir, nmr | 531 |
| 6-Ethoxy-2-methyl-3(2*H*)-pyridazinone | — | *H* 70 |
| 6-Ethoxy-5-methyl-3(2*H*)-pyridazinone | — | *H* 89 |
| 4-Ethoxy-5-morpholino-2-prop-1′-enyl-3(2*H*)-pyridazinone | 91–92 | 1269 |
| 4-Ethoxy-5-morpholino-2-propyl-3(2*H*)-pyridazinone | 150–160/0.5 | 1269 |
| 4-Ethoxy-5-morpholino-3(2*H*)-pyridazinone | 185–187, nmr | 532, 1269 |
| 6-Ethoxy-5-nitro-3-pyridazinamine 2-oxide | — | *H* 198, 604 |
| 3-Ethoxy-4-nitropyridazine 1-oxide | 105–106 | 757; *H* 194, 649 |
| 3-Ethoxy-6-phenoxypyridazine | — | 688 |
| 6-Ethoxy-2-phenyl-4-propylamino-3(2*H*)-pyridazinone | — | *H* 109 |
| 3-Ethoxy-6-phenylpyridazine | 100, nmr; 2-$MeClO_4$: 140–141; 2-$PhClO_4$: 136–141 | 413; *H* 177 |
| 4-Ethoxy-2-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 157 |
| 5-Ethoxy-2-phenyl-3,4(1*H*,2*H*)-pyridazinedione | 164–165, ir, nmr | 531 |
| 6-Ethoxy-2-phenyl-3,4(1*H*,2*H*)-pyridazinedione | — | *H* 169 |
| 5-Ethoxy-6-phenyl-3(2*H*)-pyridazinethione | 218–220, ir, nmr | 1087 |
| 5-Ethoxy-2-phenyl-3(2*H*)-pyridazinone | 126–127, ir, nmr | 531; *H* 85 |
| 6-Ethoxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 80 |
| 3-Ethoxy-4-pyridazinamine | — | *H* 183, 560 |
| 6-Ethoxy-3-pyridazinamine | — | *H* 177, 552 |
| 6-Ethoxy-4-pyridazinamine | — | *H* 183, 560 |
| 3-Ethoxypyridazine | 34, 75/4, nmr | 99; *H* 174 |
| 4-Ethoxypyridazine | — | *H* 186 |
| 3-Ethoxy-4-pyridazinecarboxamide | 154–155, nmr | 1099 |
| 6-Ethoxy-3-pyridazinecarboxylic acid | 130–132 | 1592 |
| 3-Ethoxy-4,5-pyridazinediamine | — | *H* 599 |
| 3-Ethoxypyridazine 1-oxide | — | *H* 194 |
| 4-Ethoxypyridazine 1-oxide | — | *H* 194 |
| 4-Ethoxypyridazine 2-oxide | — | *H* 196 |
| 6-Ethoxy-3(2*H*)-pyridazinethione | 190, ir | 636; *H* 797 |
| 1-Ethoxy-4(1*H*)-pyridazinone | — | *H* 134 |
| 4-Ethoxy-3(2*H*)-pyridazinone | 77–78 | 1269 |
| 5-Ethoxy-3(2*H*)-pyridazinone | — | *H* 85 |
| 6-Ethoxy-3(2*H*)-pyridazinone | — | *H* 68 |
| 4-Ethoxy-3,5,6-triphenylpyridazine | — | *H* 186 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| Ethyl 4-acetamido-5-cyano-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate | 185, ir | 119 |
| Ethyl 3-acetamido-5,6-diphenyl-4-pyridazinecarboxylate | — | *H* 555 |
| Ethyl 5-acetamido-4-pyridazinecarboxylate | — | *H* 562 |
| Ethyl 4-acetoxy-6-phenyl-3-pyridazinecarboxylate | 83–84 or 237 (?), ir, nmr | 1969 |
| Ethyl 5-acetyl-4-pyridazinecarboxylate | 39–40, ir, nmr | 81 |
| Ethyl 1-allyl-6-oxo-1,6-dihydro-3-pyridazinecarboxylate | — | 855 |
| Ethyl 4-amino-5-cyano-6-imino-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate | 197 or >300, ir | 51, 229 |
| Ethyl 4-amino-5-cyano-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate | 228–230 or 235 (or > 300?), ir, nmr | 55, 1665 |
| 4-Ethylamino-2,6-dimethylpyridazine | — | *H* 582 |
| 4-Ethylamino-3,6-dimethylpyridazine 2-oxide | — | *H* 604 |
| 3-Ethylamino-4,6-dinitropyridazine 1-oxide | 149–150 | 760 |
| Ethyl 3-amino-5,6-diphenyl-4-pyridazinecarboxylate | — | *H* 555 |
| 4-Ethylamino-2-methyl-1-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | 678 |
| 3-Ethylaminomethyl-4(1*H*)-pyridazinone 2-oxide | — | *H* 196, 610 |
| Ethyl 5-amino-3-oxo-2,3-dihydro-4-pyridazinecarboxylate | — | *H* 125, 440 |
| Ethyl 3-amino-6-phenyl-4-pyridazinecarboxylate | 123–124 | 1185 |
| 3-Ethylaminopyridazine | — | 1176; *H* 572 |
| Ethyl 5-amino-4-pyridazinecarboxylate | — | *H* 562 |
| Ethyl 6-amino-3-pyridazinecarboxylate | — | *H* 435, 553 |
| 4-Ethylamino-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 151 |
| 3-Ethylaminopyridazine 1-oxide | — | *H* 604 |
| 3-Ethylaminopyridazine 2-oxide | — | *H* 604 |
| Ethyl 6-amino-3-pyridazinesulfenate | — | *H* 553 |
| Ethyl 6-benzoyl-5-hydroxy-3-oxo-2-phenyl-2,3-dihydro-4-pyridazinecarboxylate | 195–197, ir | 1702 |
| Ethyl 6-benzoyl-3-oxo-2-phenyl-2,3-dihydro-4-pyridazinecarboxylate | 114–116, ir, nmr | 1960 |
| Ethyl 5-benzoyl-4-pyridazinecarboxylate | 67–68 or 71, ir, nmr | 46, 81 |
| Ethyl 3-benzyl-6-phenyl-4-pyridazinecarboxylate | — | *H* 436 |
| Ethyl 5,6-bisdiisopropylamino-3-pyridazinecarboxylate | 230/4 × $10^{-6}$, ir, ms, nmr | 1070 |
| Ethyl 1-butyl-6-oxo-1,6-dihydro-3-pyridazinecarboxylate | 138–139/2, nmr | 118 |
| Ethyl 3-chloro-5,6-diphenyl-4-pyridazinecarboxylate | — | *H* 436 |
| Ethyl 3-chloro-5-ethyl-6-oxo-1,6-dihydro-4-pyridazinecarboxylate | 104–106 or 111–113, nmr, uv | 30, 821 |
| Ethyl 6-chloro-5-ethyl-3-oxo-2,3-dihydro-4-pyridazinecarboxylate | 131–132, nmr, uv | 30, 821 |
| Ethyl 3-chloro-5-methyl-6-oxo-1,6-dihydro-4-pyridazinecarboxylate | 112–113 or 122–123, nmr, uv | 30, 821 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| Ethyl 6-chloro-5-methyl-3-oxo-2,3-dihydro-4-pyridazinecarboxylate | 173–175, ms, nmr, uv | 30, 821 |
| Ethyl 3-chloro-6-methyl-4-pyridazinecarboxylate | liq, nmr | 1514; *H* 291, 292, 436 |
| Ethyl 3-chloro-6-oxo-1,6-dihydro-1-pyridazinecarboxylate | — | *H* 71, 305 |
| Ethyl 3-chloro-6-oxo-1,6-dihydro-4-pyridazinecarboxylate | — | *H* 87, 291, 439 |
| Ethyl 6-chloro-3-oxo-2,3-dihydro-4-pyridazinecarboxylate | — | *H* 96, 322, 440 |
| Ethyl 3-chloro-6-phenyl-4-pyridazinecarboxylate | 45–46 or 60 | 759, 1319 |
| Ethyl 3-chloro-1-pyridazinecarboxylate | — | *H* 276, 434 |
| Ethyl 3-chloro-4-pyridazinecarboxylate | — | *H* 290, 435 |
| Ethyl 6-chloro-3-pyridazinecarboxylate | 154–155, nmr | 1459, 1638 |
| Ethyl 5-cyano-4-β-dimethylaminovinyl-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate | 160–161, ir, nmr | 1830 |
| Ethyl 5-cyano-6-imino-1,4-diphenyl-1,6-dihydro-3-pyridazinecarboxylate | 193, ir | 219, 988 |
| Ethyl 5-cyano-6-imino-4-methyl-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate | 136–138; HBr: 195–197 | 124, 234, 988 |
| Ethyl 5-cyano-3-methyl-6-oxo-1,6-dihydro-4-pyridazinecarboxylate | 240, ir, nmr | 1487 |
| Ethyl 5-cyano-4-methyl-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate | 150 to 162, ir, ms, nmr, uv | 37, 52, 54, 221, 379, 1216, 1240, 2043 |
| Ethyl 6-cyano-5-methyl-3-oxo-2-phenyl-2,3-dihydro-4-pyridazinecarboxylate | 168, ir | 1217 |
| Ethyl 5-cyano-6-oxo-1-phenyl-4-styryl-1,6-dihydro-3-pyridazinecarboxylate | 212 or 220, ir | 52, 379 |
| Ethyl 3,4-dichloro-6-oxo-1,6-dihydro-1-pyridazinecarboxylate | — | *H* 91, 327 |
| Ethyl 4,5-dichloro-6-oxo-1,6-dihydro-1-pyridazinecarboxylate | — | *H* 113, 308 |
| Ethyl 3,6-dichloro-4-pyridazinecarboxylate | 116–118/1.3 | 1099, 1748 |
| 4-Ethyl-3,6-dimethoxypyridazine | — | *H* 192 |
| Ethyl 1-β-dimethylaminoethyl-6-oxo-1,6-dihydro-3-pyridazinecarboxylate | — | *H* 438 |
| Ethyl 3-β-dimethylaminovinyl-4-pyridazinecarboxylate | 64, ir, ms, nmr | 1678 |
| Ethyl 5-β-dimethylaminovinyl-4-pyridazinecarboxylate | 122–123, nmr | 1095 |
| 2-Ethyl-5,6-dimethyl-3-oxo-2,3-dihydro-4-pyridazinecarbonitrile | — | *H* 130, 449 |
| Ethyl 3,6-dimethyl-5-oxo-2,5-dihydro-4-pyridazinecarboxylate | 165, nmr | 948 |
| Ethyl 5,6-dimethyl-3-oxo-2,3-dihydro-4-pyridazinecarboxylate | — | *H* 128, 441 |
| 5-Ethyl-4,6-dimethyl-3-pyridazinamine | — | *H* 556 |
| 4-Ethyl-3,6-dimethylpyridazine | 87–89/4, anal | 122 |

*continued*

| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| Ethyl 3,6-dimethyl-4-pyridazinecarboxylate | — | *H* 436 |
| 3-Ethyl-4,6-diphenylpyridazine | 84–85, nmr | 394 |
| 4-Ethyl-3,6-diphenylpyridazine | 77 to 83, nmr, uv | 319, 394, 499 |
| Ethyl 3,6-diphenyl-4-pyridazinecarboxylate | 98, nmr | 40; *H* 436 |
| 1-Ethyl-3,5-diphenyl-4(1*H*)-pyridazinone | 124–125, nmr | 249 |
| Ethyl 3-ethoxycarbonylmethyl-5,6-diphenyl-4-pyridazinecarboxylate | < 185 / 0.5, ms | 339 |
| Ethyl 1-ethoxycarbonylmethyl-6-oxo-1,6-dihydro-3-pyridazinecarboxylate | — | *H* 77, 437 |
| Ethyl 2-ethoxycarbonylmethyl-3-oxo-6-phenyl-2,3-dihydro-4-pyridazinecarboxylate | liq, anal | 1768 |
| 1-Ethyl-5-ethylamino-2-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 595 |
| Ethyl 3-ethyl-6-methyl-4-pyridazinecarboxylate | 100/0.4, ir, nmr | 10 |
| Ethyl 5-ethyl-3-oxo-2,3-dihydro-4-pyridazinecarboxylate | 77–79, nmr, uv | 821 |
| Ethyl 5-ethyl-6-oxo-1,6-dihydro-4-pyridazinedcarboxylate | 112–115, nmr, uv | 821 |
| 2-Ethyl-6-ethylsulfonyl-3(2*H*)-pyridazinone | — | *H* 824 |
| 3-(β-Ethyl-β-formylhydrazino)-6-methyl-4-phenylpyridazine | — | *H* 669 |
| Ethyl 3-formyl-6-methyl-4-pyridazinecarboxylate | 90, nmr; $Et_2$acetal: nmr | 907, 996, 1111 |
| Ethyl 5-formyl-4-oxo-1-phenyl-1,4-dihydro-3-pyridazinecarboxylate | 34, ir, nmr; PhNHN=: 173, ir, nmr | 1302 |
| Ethyl 3-formyl-4-pyridazinecarboxylate | $Et_2$acetal: anal, nmr | 1111 |
| 6-Ethyl-4-hexylamino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 107 |
| Ethyl 5-hydrazinocarbonyl-6-imino-4-methyl-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate | PhCH=: 230, ir | 1215 |
| 6-Ethyl-2-hydrazinocarbonylmethyl-3(2*H*)-pyridazinone | — | *H* 79 |
| Ethyl 6-hydrazino-3-pyridazinecarboxylate | anal, ms, nmr | 1459 |
| Ethyl 1-β-hydroxyethyl-6-oxo-1,6-dihydro-3-pyridazinecarboxylate | — | *H* 71 |
| Ethyl 1-β-hydroxyethyl-6-oxo-1,6-dihydro-4-pyridazinecarboxylate | — | *H* 86, 439 |
| Ethyl 1-hydroxymethyl-6-oxo-1,6-dihydro-3-pyridazinecarboxylate | — | *H* 71, 402, 437 |
| Ethyl 6-hydroxymethyl-5-oxo-2,5-dihydro-3-pyridazinecarboxylate | 196, ir, nmr, uv | 778 |
| Ethyl 4-hydroxy-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate | — | *H* 136, 444 |
| Ethyl 4-hydroxy-6-oxo-1-phenyl-5-phenylazo-1,6-dihydro-3-pyridazinecarboxylate | — | *H* 136, 444, 651 |
| 4-(α-Ethyl-α-hydroxypropyl)-5,6-diphenyl-3(2*H*)-pyridazinetione | 211–213, ir, uv | 373 |
| 4-(α-Ethyl-α-hydroxypropyl)-5,6-diphenyl-3(2*H*)-pyridazinone | 225–227, ir, uv | 373 |
| 2-Ethyl-4-isopropoxy-6-methyl-3(2*H*)-pyridazinone | — | *H* 101 |

*continued*

| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 2-Ethyl-6-isopropoxy-3(2*H*)-pyridazinone | — | *H* 71 |
| 2-Ethyl-6-isopropylsulfonyl-3(2*H*)-pyridazinone | — | *H* 824 |
| 2-Ethyl-6-methoxy-5-oxo-2,5-dihydro-3-pyridazinecarbonitrile | — | *H* 134, 355 |
| Ethyl 5-methoxy-6-oxo-1,6-dihydro-4-pyridazinecarboxylate | — | *H* 125, 440 |
| 1-Ethyl-5-methoxy-2-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 162 |
| 4-Ethyl-5-methoxypyridazine | liq, ms, nmr | 82 |
| 2-Ethyl-6-methoxy-3(2*H*)-pyridazinone | — | *H* 71 |
| 4-Ethyl-5-methyl-3,6-bismethylthiopyridazine | liq?, ir, ms, nmr | 353 |
| 4-Ethyl-5-methyl-3,6-diphenylpyridazine | 119–121 | 319 |
| Ethyl 3-methyl-5,6-diphenyl-4-pyridazinecarboxylate | 85–86, ir, ms, nmr | 314 |
| Ethyl 3-α-methylhydrazino-6-morpholino-4-pyridazinecarboxylate | $Me_2C$=: 121–122, ir, nmr, uv | 753 |
| 5-(β-Ethyl-α-methylhydrazino)-2-phenyl-3(2*H*)-pyridazinone | 109–110, ir, nmr | 1540 |
| 5-Ethyl-4-methyl-2-β-morpholinoethyl-3(2*H*)-pyridazinone | — | *H* 125 |
| 2-Ethyl-6-methyl-4-nitro-3(2*H*)-pyridazinone | — | *H* 100 |
| Ethyl 1-methyl-6-oxo-1,6-dihydro-3-pyridazinecarboxylate | — | *H* 70, 437 |
| Ethyl 3-methyl-6-oxo-1,6-dihydro-4-pyridazinecarboxylate | — | *H* 88, 439 |
| Ethyl 5-methyl-3-oxo-2,3-dihydro-4-pyridazinecarboxylate | 100–101, nmr, uv | 821 |
| Ethyl 5-methyl-6-oxo-1,6-dihydro-4-pyridazinecarboxylate | 144–145, nmr, uv | 821 |
| Ethyl 6-methyl-3-oxo-2,3-dihydro-4-pyridazinecarboxylate | — | *H* 97, 441 |
| Ethyl 6-methyl-5-oxo-2,5-dihydro-4-pyridazinecarboxylate | 260 | 500 |
| Ethyl 2-methyl-3-oxo-5,6-diphenyl-2,3-dihydro-4-pyridazinecarboxylate | — | *H* 129, 441 |
| Ethyl 3-methyl-5-oxo-6-phenyl-2,5-dihydro-4-pyridazinecarboxylate | 168, nmr | 948 |
| Ethyl 4-methyl-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate | 102–103, nmr | 1350, 1351, 2014; *H* 93, 438 |
| Ethyl 5-methyl-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate | — | *H* 106, 439 |
| Ethyl 6-methyl-4-oxo-1-phenyl-1,4-dihydro-3-pyridazinecarboxylate | — | *H* 135, 442 |
| Ethyl 6-methyl-5-oxo-3-phenyl-2,5-dihydro-4-pyridazinecarboxylate | 195, nmr | 948 |
| Ethyl 6-methyl-5-oxo-3-propyl-2,5-dihydro-4-pyridazinecarboxylate | 136, nmr | 948 |
| 3-Ethyl-4-methyl-6-phenylpyridazine | 44–45 or 51–53, nmr | 394, 1952 |
| 4-Ethyl-3-methyl-6-phenylpyridazine | — | 1611 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| Ethyl 3-methyl-6-phenyl-4-pyridazinecarboxylate | — | *H* 436 |
| 4-Ethyl-6-methyl-2-phenyl-3(2*H*)-pyridazinone | — | *H* 106 |
| 6-Ethyl-4-methyl-2-phenyl-3(2*H*)-pyridazinone | — | *H* 106 |
| Ethyl 6-methyl-3-propyl-4-pyridazinecarboxylate | 102/0.3, ir, nmr | 10 |
| 3-Ethyl-4-methylpyridazine | 30, nmr; pic: 100–101 | 823 |
| 3-Ethyl-6-methylpyridazine | 105–110/4; pic: 107–109 | 230, 823 |
| 4-Ethyl-5-methylpyridazine | ms, nmr | 337 |
| 4-Ethyl-6-methylpyridazine | liq, ms, nmr | 1676 |
| Ethyl 3-methyl-4-pyridazinecarboxylate | 60, ir, ms, nmr | 416, 1678 |
| Ethyl 3-methyl-5-pyridazinecarboxylate | crude: nmr | 416 |
| Ethyl 4-methyl-3-pyridazinecarboxylate | 75–80, ms, nmr | 416 |
| Ethyl 5-methyl-4-pyridazinecarboxylate | < 30, ms, nmr | 211, 416 |
| Ethyl 6-methyl-4-pyridazinecarboxylate | nmr | 1111 |
| Ethyl methyl 4,5-pyridazinedicarboxylate | liq, ir, nmr | 228 |
| 4-Ethyl-5-methyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 151 |
| 2-Ethyl-6-methyl-3(2*H*)-pyridazinethione | liq, nmr | 538 |
| 2-Ethyl-6-methyl-3(2*H*)-pyridazinone | 68–70, ir, nmr | 1134; *H* 71 |
| 4-Ethyl-6-methyl-3(2*H*)-pyridazinone | — | *H* 97 |
| 5-Ethyl-4-methyl-3(2*H*)-pyridazinone | — | *H* 125 |
| 6-Ethyl-4-methyl-3(2*H*)-pyridazinone | 83, ir, nmr | 1562 |
| 2-Ethyl-6-methylsulfonyl-3(2*H*)-pyridazinone | — | *H* 824 |
| 2-Ethyl-6-methylthio-3(2*H*)-pyridazinethione | 45–46 | 95 |
| 5-Ethyl-2-$\beta$-morpholinoethyl-6-phenyl-3(2*H*)-pyridazinone | — | *H* 104 |
| 6-Ethyl-4-morpholinomethyl-2-phenyl-3(2*H*)-pyridazinone | — | *H* 612 |
| 1-Ethyl-5-morpholino-2-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 162, 595 |
| 6-Ethyl-4-morpholino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 107, 589 |
| Ethyl 6-morpholino-3-pyridazinecarboxylate | 125–126 | 1338 |
| 5-Ethyl-3-oxo-2,3-dihydro-4-pyridazinecarbonitrile | 155–158, nmr, uv | 821 |
| Ethyl 3-oxo-2,3-dihydro-4-pyridazinecarboxylate | 92–94 | 821; *H* 84, 440 |
| Ethyl 6-oxo-1,6-dihydro-3-pyridazinecarboxylate | 130–131 | 1459; *H* 67, 437 |
| Ethyl 6-oxo-1,6-dihydro-4-pyridazinecarboxylate | 125–127 | 821; *H* 85, 439(?) |
| Ethyl 3-oxo-5,6-diphenyl-2,3-dihydro-4-pyridazinecarboxylate | — | 1407; *H* 128, 441 |
| Ethyl 4-oxo-1,6-diphenyl-1,4-dihydro-3-pyridazinecarboxylate | — | *H* 135, 443 |
| Ethyl 6-oxo-3-phenoxy-1,6-dihydro-1-pyridazinecarboxylate | — | 876 |
| 2-Ethyl-3-oxo-5-phenyl-2,3-dihydro-4-pyridazinecarbonitrile | — | *H* 125, 448 |
| Ethyl 3-oxo-6-phenyl-2,3-dihydro-4-pyridazinecarboxylate | 145 to 156, ir, nmr | 759, 1319, 1768 |
| Ethyl 4-oxo-1-phenyl-1,4-dihydro-3-pyridazinecarboxylate | 99–100, nmr | 316 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| Ethyl 4-oxo-6-phenyl-1,4-dihydro-3-pyridazinecarboxylate | 196–198, ir, nmr | 1969, 2111 |
| Ethyl 6-oxo-3-phenyl-1,6-dihydro-1-pyridazinecarboxylate | 118–119, ir | 368 |
| 4-Ethyl-6-oxo-1-phenyl-1,6-dihydro-3,5-pyridazinedicarbonitrile | 145–147, ir, nmr | 1808 |
| Ethyl 6-oxo-1-phenyl-5-phenylazo-1,6-dihydro-3-pyridazinecarboxylate | — | *H* 106, 441 |
| Ethyl 3-oxo-2,5,6-triphenyl-2,3-dihydro-4-pyridazinecarboxylate | — | *H* 131, 441 |
| Ethyl 6-oxo-1-vinyl-1,6-dihydro-3-pyridazine carboxylate | — | 693 |
| Ethyl 5-phenethyl-4-pyridazinecarboxylate | 77–82, ir, ms, nmr | 416 |
| 6-Ethyl-2-phenyl-4-piperidinomethyl-3(2*H*)-pyridazinone | — | *H* 612 |
| 1-Ethyl-2-phenyl-5-piperidino-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 162, 595 |
| 4-Ethyl-6-phenyl-3-pyridazinamine | 149, nmr | 1381 |
| 3-Ethyl-6-phenylpyridazine | 68–69, nmr | 823 |
| 4-Ethyl-3-phenylpyridazine | 61, ms, nmr | 12 |
| 4-Ethyl-6-phenylpyridazine | 56–58 or 58–60, ms, nmr | 1676, 1952 |
| 1-Ethyl-2-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 162 |
| 2-Ethyl-6-phenyl-3(2*H*)-pyridazinethione | 70–71, nmr | 538 |
| 4-Ethyl-3-phenyl-5-trimethylsilylpyridazine | 74, ms, nmr | 12 |
| 3-Ethyl-6-piperidinopyridazine | 150/0.01, nmr; pic: 163–164 | 823 |
| Ethyl 5-propionyl-4-pyridazinecarboxylate | 48–49, ir, nmr | 81 |
| 6-Ethyl-3-pyridazinamine | — | *H* 551 |
| 3-Ethylpyridazine | liq, nmr; pic: 134–135 | 230, 822 |
| 4-Ethylpyridazine | ms | 73, 79 |
| Ethyl 3-pyridazinecarboxylate | 68 | 1347; *H* 434 |
| Ethyl 4-pyridazinecarboxylate | 76/1, ir, nmr | 65, 416, 1231; *H* 435 |
| Ethyl 3-pyridazinecarboxylate 1-oxide | — | *H* 434 |
| 1-Ethyl-3,6(1*H*,2*H*)-pyridazinedione | 143–145, ir, nmr | 292, 943 |
| 5-Ethyl-3(2*H*)-pyridazinone | 99–101, nmr, uv | 30, 821 |
| 6-Ethyl-3(2*H*)-pyridazinone | liq, ms, nmr | 186; *H* 67 |
| Ethyl 5-styryl-4-pyridazinecarboxylate | 115–117, ms, nmr | 211 |
| 5-Ethylsulfinyl-2,4-dimethyl-3(2*H*)-pyridazinone | 129–130, ir, ms, nmr | 231 |
| 3-Ethylsulfinyl-6-methoxy-4-methylpyridazine | liq, nmr | 2035 |
| 3-Ethylsulfinyl-6-methoxypyridazine | liq, nmr | 2035 |
| 4-Ethylsulfinyl-2-methyl-3(2*H*)-pyridazinone | — | 1028 |
| 5-Ethylsulfinyl-2-methyl-3(2*H*)-pyridazinone | — | *H* 1029 |
| 6-Ethylsulfinyl-3-pyridazinamine | — | *H* 822 |
| 5-Ethylsulfonyl-2,4-dimethyl-3(2*H*)-pyridazinone | 133–134, ir, ms, nmr | 231 |
| 3-Ethylsulfonyl-6-hydrazinopyridazine | — | 1723 |
| 3-Ethylsulfonyl-6-methylpyridazine | — | *H* 823 |
| 4-Ethylsulfonyl-2-methyl-3(2*H*)-pyridazinone | — | 1028 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 5-Ethylsulfonyl-2-methyl-3(2*H*)-pyridazinone | — | 1028, 1602 |
| 6-Ethylsulfonyl-2-methyl-3(2*H*)-pyridazinone | — | *H* 824 |
| 3-Ethylsulfonyl-6-phenylpyridazine | — | 733 |
| 6-Ethylsulfonyl-3(2*H*)-pyridazinone | — | 1723; *H* 824 |
| 2-β-[*N*-Ethyl(thiocarbamoyl)]ethyl-6-phenyl-3(2*H*)-pyridazinone | 140–145 | 1288 |
| 4-Ethylthio-5,6-diphenyl-3(2*H*)-pyridazinone | — | 809 |
| 4-Ethylthio-2-methyl-5-methylamino-3(2*H*)-pyridazinone | 104–105 | 2089 |
| 6-Ethylthio-5-methyl-3-oxo-2-phenyl-2,3-dihydro-4-pyridazinecarbonitrile | — | 1618 |
| 4-Ethylthio-2-methyl-1-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 830 |
| 3-Ethylthio-6-methylpyridazine | — | *H* 804 |
| 4-Ethylthio-1-methyl-3,6(1*H*,2*H*)-pyridazinedione | — | 1007 |
| 4-Ethylthio-2-methyl-3,6(1*H*,2*H*)-pyridazinedione | — | 1007 |
| 5-Ethylthio-2-methyl-3,4(1*H*,2*H*)-pyridazinedione | 143–145, ir, uv | 1896 |
| 4-Ethylthio-2-methyl-3(2*H*)-pyridazinone | — | 1028 |
| 5-Ethylthio-2-methyl-3(2*H*)-pyridazinone | — | 1028 |
| 3-Ethylthio-6-phenylpyridazine | — | 733, 811 |
| 5-Ethylthio-1-phenyl-3,4(1*H*,2*H*)-pyridazinedione | 132–134, ir, uv | 1896 |
| 6-Ethylthio-2-phenyl-3,4(1*H*,2*H*)-pyridazinedione | — | *H* 831 |
| 6-Ethylthio-3-pyridazinamine | 46–48, nmr | 1147; *H* 553, 804 |
| 6-Ethylthio-3,4(1*H*,2*H*)-pyridazinedione | — | *H* 169 |
| 4-Ethylthio-3,5,6-triphenylpyridazine | — | *H* 810 |
| Ethyl 3,4,5-trichloro-6-oxo-1,6-dihydro-1-pyridazinecarboxylate | — | *H* 129, 329 |
| Ethyl 4,5,6-trichloro-3-pyridazinecarboxylate | 27, 110/0.3, ir, ms, nmr | 1232 |
| 2-Ethyl-4,5,6-trifluoro-3(2*H*)-pyridazinone | — | *H* 130, 332 |
| Ethyl 3,5,6-triphenyl-4-pyridazinecarboxylate | 124–125, ir, nmr | 314 |
| 1-Ethyl-3,5,6-triphenyl-4(1*H*)-pyridazinone | — | *H* 134 |
| Ethyl 4,5,6-trisdimethylamino-3-pyridazinecarboxylate | liq, anal, ir, nmr | 8 |
| 4-Ethynyl-3,6-dimethylpyridazine | crude: 82–84, ir, nmr | 122 |
| 3-Ethynyl-6-methylpyridazine | 113–114 or 135–137, ir, nmr | 122, 834 |
| 6-Fluoro-4,5-dimethoxy-3(2*H*)-pyridazinone | — | *H* 127, 332 |
| 3-Fluoro-6-(3-hydroxyprop-1-ynyl)pyridazine | 86–88, nmr | 1385 |
| 3-Fluoro-6-iodopyridazine | 118–120, nmr | 1385 |
| 3-Fluoro-6-methylpyridazine | 15, anal, unstable | 1127 |
| 3-Fluoro-6-phenylpyridazine | anal | 1593 |
| 4-Fluoro-1-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | 917 |
| 6-Fluoro-3-pyridazinamine | 177–179, nmr | 1145 |
| 4-Fluoro-3,6(1*H*,2*H*)-pyridazinedione | — | 917 |
| 3-Fluoro-4,5,6-trimethoxypyridazine | — | *H* 184, 297 |
| 4-Fluoro-3,5,6-trimethoxypyridazine | — | *H* 184, 298 |
| 3-Fluoro-4,5,6-trisphenylthiopyridazine | — | *H* 297, 814 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3-Formamidopyridazine | oxime: 185 | 1010 |
| 3-Formamidopyridazine 2-oxide | oxime: 178–180 | 921 |
| 6-Formamido-3(2*H*)-pyridazinone | 285–286, nmr | 295 |
| 1-β-Formylethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 402 |
| 3-Formylethynyl-4-methyl-6-phenylpyridazine | $Et_2$acetal: 83, ir, ms, nmr | 1629 |
| 3-Formylethynyl-6-methylpyridazine | $Et_2$acetal: 56, ir, ms, nmr | 1629 |
| 3-Formylethynyl-6-phenylpyridazine | $Me_2$acetal: 117, ir, ms, nmr | 1629 |
| 6-Formyl-3-imino-2,5-diphenyl-2,3-dihydro-4-pyridazinecarbonitrile | 206–208, ir, nmr | 1960 |
| 4-β-Formylmethyl-3,6-dimethylpyridazine | $Me_2$acetal: 140–143/3, nmr | 125 |
| 3-β-Formylmethyl-6-methylpyridazine | $Me_2$acetal: 131–132/4, nmr | 125 |
| 6-Formyl-4-methyl-1-phenylpyridazinium-3-sulfonate | oxime: 255–260, xl st | 1536, 1785 |
| 3-Formyl-6-methyl-4-pyridazinecarbohydrazide | $Me_2$acetal: nmr | 996 |
| 3-Formyl-6-methyl-4-pyridazinecarboxamide | $Et_2$acetal: 112, ir, nmr; $Me_2$acetal: 141, ir, nmr | 996 |
| 3-β-Formylvinylpyridazine | 147–149, ir, nmr, uv | 511 |
| 4-β-Formylvinylpyridazine | 159–161, ir, nmr, uv | 511 |
| 1-Hexanoyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 155 |
| 4-Hexyl-3,6-bismethylthiopyridazine | liq(?), ir, ms, nmr | 353 |
| 6-Hexyloxy-2-hydroxy-3(2*H*)-pyridazinone | — | *H* 76 |
| 3-Hexyloxy-6-iodopyridazine | — | *H* 179, 287 |
| 4-Hexyloxy-2-methyl-1-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 160 |
| 6-Hexyloxy-5-nitro-3-pyridazinamine 2-oxide | — | *H* 198, 604 |
| 3-Hexyloxy-6-phenoxypyridazine | — | 688 |
| 6-Hexyloxy-3-pyridazinamine | — | *H* 179, 552 |
| 6-Hexyloxy-3(2*H*)-pyridazinethione | 105, ir | 365 |
| 3-Hexylpyridazine | nmr | 414 |
| 6-Hexyl-3(2*H*)-pyridazinone | — | *H* 67 |
| 6-Hexylthio-3-pyridazinamine | 68–69, nmr | 1147 |
| 3-Hex-1′-ynyl-6-methylpyridazine | 100/5, ir, nmr | 191, 834 |
| 3-Hex-1′-ynyl-6-methylpyridazine 1-oxide | 58–60 or 60–61, ir, nmr | 191, 834 |
| 2-α-Hydrazinocarbonylbutyl-6-methyl-3(2*H*)-pyridazinone | — | *H* 79 |
| 2-β-Hydrazinocarbonylethyl-4,6-diphenyl-3(2*H*)-pyridazinone | 169, ir, nmr | 1343 |
| 2-α-Hydrazinocarbonylethyl-6-methyl-3(2*H*)-pyridazinone | — | *H* 79 |
| 2-β-Hydrazinocarbonylethyl-6-methyl-3(2*H*)-pyridazinone | — | *H* 73 |
| 2-Hydrazinocarbonylmethyl-4,6-dimethyl-3(2*H*)-pyridazinone | — | *H* 101 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 2-Hydrazinocarbonylmethyl-5,6-dimethyl-3(2*H*)-pyridazinone | — | *H* 92 |
| 2-Hydrazinocarbonylmethyl-4,6-diphenyl-3(2*H*)-pyridazinone | 170, ir, nmr | 1343 |
| 2-(α-Hydrazinocarbonyl-α-methylethyl)-6-methyl-3(2*H*)-pyridazinone | — | *H* 79 |
| 3-Hydrazinocarbonylmethyl-6-methoxypyridazine | 149, ir | 962 |
| 2-Hydrazinocarbonylmethyl-4-methyl-3(2*H*)-pyridazinone | — | *H* 84 |
| 2-Hydrazinocarbonylmethyl-6-methyl-3(2*H*)-pyridazinone | — | *H* 78 |
| 1-Hydrazinocarbonylmethyl-6-oxo-1,6-dihydro-3-pyridazinecarbohydrazide | — | *H* 77, 437 |
| 2-Hydrazinocarbonylmethyl-6-phenyl-3(2*H*)-pyridazinone | — | *H* 79 |
| 3-Hydrazinocarbonylmethyl-6-phenyl-4(1*H*)-pyridazinone | 319–320, ir, nmr | 1489 |
| 3-Hydrazinocarbonylmethylpyridazine | uv | 1164 |
| 2-Hydrazinocarbonylmethyl-3(2*H*)-pyridazinone | — | *H* 65 |
| 2-Hydrazinocarbonylmethyl-4,5,6-trimethyl-3(2*H*)-pyridazinone | — | *H* 130 |
| 2-α-Hydrazinocarbonylpentyl-6-methyl-3(2*H*)-pyridazinone | — | *H* 79 |
| 2-γ-Hydrazinocarbonylpropyl-4,6-diphenyl-3(2*H*)-pyridazinone | 136, ir, nmr | 1343 |
| 2-α-Hydrazinocarbonylpropyl-6-methyl-3(2*H*)-pyridazinone | — | *H* 79 |
| 4-Hydrazino-3,6-dimethoxypyridazine | — | *H* 192, 661 |
| 3-Hydrazino-4,5-dimethyl-6-morpholinopyridazine | 172–176 | 1293 |
| 3-Hydrazino-4,6-dimethylpyridazine | — | *H* 660, 667 |
| 3-Hydrazino-4,6-diphenylpyridazine | — | *H* 660 |
| 6-Hydrazino-4-hydrazinocarbonylmethyl-3(2*H*)-pyridazinone | 249–251, ir, uv | 947 |
| 5-Hydrazino-2-δ-hydroxybutyl-3(2*H*)-pyridazinone | 157–158, ir, nmr | 1700 |
| 3-Hydrazino-4-isobutylaminopyridazina | — | *H* 583 |
| 3-Hydrazino-6-isopropylaminopyridazine | 2HCl: 225 | 554, 1258 |
| 3-Hydrazino-6-methoxypyridazine | — | *H* 660 |
| 3-Hydrazino-4-methylaminopyridazine | — | *H* 582 |
| 4-Hydrazino-2-methyl-5-methylsulfonyl-3(2*H*)-pyridazinone | 147–149 | 533 |
| 3-Hydrazino-4-methyl-6-morpholinopyridazine | HCl: 222–224; PhCH=: 193–197 or 197–199 | 544, 1293 |
| 3-Hydrazino-6-methyl-4-nitropyridazine 1-oxide | 169 | 760 |
| 3-Hydrazino-4-methyl-6-phenylpyridazine | 159 or 160–161, ir, nmr; HCl: 168–170; $Me_2C$=, maleate salt: 155 | 1104, 1279, 1333 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3-Hydrazino-5-methyl-6-phenylpyridazine | $Me_2C$=: 171–173, ir, nmr | 1585 |
| 3-Hydrazino-6-methyl-5-phenylpyridazine | $Me_2C$=: 192–194, ir, nmr | 1585 |
| 3-Hydrazino-4-methyl-6-piperidinopyridazine | HCl: 217–220; PhCH=: 181–185 | 544 |
| 3-Hydrazino-6-methylpyridazine | 118 or 119, nmr; $Et_2C$=: 130–131/0.3, ir, nmr; $PhCH_2CH$=: 149–150, ir, nmr, and the like | 671, 1037, 1127; *H* 659, 666 |
| 6-Hydrazino-4-methyl-3-pyridazinecarbohydrazide | 169–172, nmr | 21 |
| 4-Hydrazino-2-methyl-3(2*H*)-pyridazinone | 138–140, ir, nmr | 533 |
| 3-Hydrazino-6-methylsulfonylpyridazine | 178–179, nmr | 304; *H* 660 |
| 3-Hydrazino-6-methylsulfonylpyridazine 1-oxide | 190–194 | 304 |
| 3-Hydrazino-6-methylthiopyridazine | 120, ir | 636 |
| 3-Hydrazino-5-morpholinomethyl-6-phenylpyridazine | 144–145, ir | 1339 |
| 3-Hydrazino-5-morpholinopyridazine | — | *H* 586 |
| 3-Hydrazino-6-morpholinopyridazine | 2HCl: 235–236; MeCH=: 181–183; PhCH=: 281 | 57, 1293; *H* 579 |
| 3-Hydrazino-4-nitropyridazine 1-oxide | 185–186 | 760 |
| 4-Hydrazino-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarbohydrazide | 212–213, ir, nmr; bis(MeCH =): 176–178, ir, nmr | 1117 |
| 3-Hydrazino-6-phenethylpyridazine | — | *H* 660 |
| 3-Hydrazino-6-phenoxypyridazine | 128, ir | 636; *H* 179, 660 |
| 3-Hydrazino-6-phenyl-5-piperidinomethylpyridazine | 125–128 | 1339 |
| 3-Hydrazino-5-phenylpyridazine | $Me_2C$=: 162–164, ir, nmr | 1585 |
| 3-Hydrazino-6-phenylpyridazine | 160–161; $Me_2C$=: 162–164; PhCH=: 272, ms | 1246, 1279; *H* 660, 667 |
| 3-Hydrazino-5-piperidinopyridazine | — | *H* 585, 586 |
| 3-Hydrazino-6-piperidinopyridazine | 206–208 | 57; *H* 578 |
| 3-Hydrazino-4-pyridazinamine | — | *H* 561 |
| 5-Hydrazino-4-pyridazinamine | — | *H* 562 |
| 6-Hydrazino-4-pyridazinamine | — | *H* 562 |
| 3-Hydrazinopyridazine | PhCH=: 210–213 | 1037, 1176; *H* 659, 666 |
| 4-Hydrazinopyridazine | — | *H* 661 |
| 6-Hydrazino-3-pyridazinecarbohydrazide | anal, ms | 1459; *H* 435, 660 |
| 6-Hydrazino-3-pyridazinecarbonitrile | crude: 173–175 | 1338 |
| 6-Hydrazino-3-pyridazinecarboxamide | — | *H* 435, 660 |
| 6-Hydrazino-3-pyridazinecarboxylic acid | anal, ir, ms, nmr | 1459 |
| 4-Hydrazino-3,6(1*H*,2*H*)-pyridazinedione | 225–229 or >300, ir, ms, nmr, uv; $Me_2C$=: <350, nmr | 213, 1512, 1597 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3-Hydrazinopyridazine 1-oxide | 152–154 or 158–159 | 546, 891; *H* 660, 666 |
| 3-Hydrazinopyridazine 2-oxide | — | *H* 660, 666 |
| 4-Hydrazinopyridazine 1-oxide | — | *H* 661, 667 |
| 4-Hydrazinopyridazine 2-oxide | — | *H* 661, 667 |
| 5-Hydrazino-3(2*H*)-pyridazinone | — | *H* 85, 661 |
| 6-Hydrazino-3(2*H*)-pyridazinone | 235–237; $Me_2C$=: 272–275, nmr | 1338 |
| 3-Hydrazino-4,5,6-triphenylpyridazine | 235–236, ir, nmr | 1362 |
| 4-Hydroxyamino-3,6-dimethylpyridazine | — | *H* 650 |
| 4-Hydroxyamino-3,6-dimethylpyridazine 2-oxide | 185, ir, nmr | 796 |
| 4-Hydroxyamino-3-methoxy-6-methylpyridazine 1-oxide | — | *H* 650 |
| 3-Hydroxyamino-3-methoxy-6-methylpyridazine 1-oxide | — | *H* 650 |
| 3-Hydroxyamino-6-methylpyridazine | 133–137, nmr | 966 |
| 4-Hydroxyamino-6-methylpyridazine 1-oxide | — | *H* 650 |
| 3-Hydroxyamino-6-phenylpyridazine | 185–186, ir, nmr | 193 |
| 3-Hydroxyaminopyridazine 1-oxide | — | *H* 650 |
| 4-α-Hydroxybenzyl-6-methoxy-3-phenylsulfinylpyridazine | liq, nmr | 2035 |
| 4-α-Hydroxybenzyl-3-methoxy-6-phenylsulfonylpyridazine | solid, nmr | 2035 |
| 4-α-Hydroxybenzyl-6-methoxy-3-phenylsulfonylpyridazine | solid, nmr | 2035 |
| 2-δ-Hydroxybutyl-4,5-dimethoxy-3(2*H*)-pyridazinone | 63–64, ir, nmr | 1700 |
| 2-δ-Hydroxybutyl-5-methoxy-3(2*H*)-pyridazinone | 85–86, ir, nmr | 1700 |
| 2-δ-Hydroxybutyl-5-methylamino-3(2*H*)-pyridazinone | 142–143, ir, nmr | 1700 |
| 1-δ-Hydroxybutyl-3,6(1*H*,2*H*)-pyridazinedione | 140–141, ir, nmr | 1700 |
| 2-Hydroxy-5,6-dimethoxy-3(2*H*)-pyridazinone | — | *H* 90 |
| 4-(β-Hydroxy-α,α-dimethylethyl)-3,6(1*H*,2*H*)-pyridazinedione | 231, nmr | 354 |
| 1-Hydroxy-3,6-dimethyl-5-nitro-4(1*H*)-pyridazinone | 184–185, uv | 794; *H* 196 |
| 2-(β-Hydroxy-α,β-dimethylpropyl)-6-methyl-3(2*H*)-pyridazinone | — | *H* 73, 403 |
| 1-Hydroxy-3,6-dimethyl-4(1*H*)-pyridazinone | 255 | 794; *H* 196 |
| 5-Hydroxy-4,6-diphenyl-3(2*H*)-pyridazinone | — | *H* 128 |
| 4-α-Hydroxyethyl-2-β-hydroxyethyl-4,5-diphenyl-3(2*H*)-pyridazinone | 130–133 | 1272 |
| 5-α-Hydroxyethyl-4-methoxy-2-methyl-6-phenyl-3(2*H*)-pyridazinone | 112–115, ir, nmr | 1832 |
| 4-α-Hydroxyethyl-6-methoxy-3-phenylsulfinylpyridazine | liq, nmr | 2035 |
| 4-α-Hydroxyethyl-3-methoxy-6-phenylsulfonylpyridazine | liq, nmr | 2035 |
| 4-α-Hydroxyethyl-6-methoxy-3-phenylsulfonylpyridazine | liq, nmr | 2035 |
| 4-α-Hydroxyethyl-3-methoxy-4-phenylthiopyridazine | liq, nmr | 2035 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 2-β-Hydroxyethyl-4-methyl-6-phenyl-3(2*H*)-pyridazinone | — | *H* 402 |
| 2-β-Hydroxyethyl-6-methyl-3(2*H*)-pyridazinone | 146–147 | 433 |
| 2-β-Hydroxyethyl-5-nitro-3,4(1*H*,2*H*)-pyridazinedione | — | *H* 169, 402, 648 |
| 1-β-Hydroxyethyl-6-oxo-1,6-dihydro-4-pyridazine-carboxylic acid | — | *H* 86 |
| 2-β-Hydroxyethyl-6-phenyl-3(2*H*)-pyridazinone | 98–99 or 105–106, nmr | 1179, 1292; *H* 402 |
| 5-α-Hydroxyethyl-6-phenyl-3(2*H*)-pyridazinone | 131–132, ir, uv | 370 |
| 4-α-Hydroxyethyl-3-pivalamidopyridazine | 138, nmr | 1421 |
| 4-α-Hydroxyethyl-5-pivalamidopyridazine | 181, nmr | 1609 |
| 4-α-Hydroxyethyl-6-pivalamidopyridazine | crude: nmr | 1421 |
| 3-α-Hydroxyethylpyridazine | liq, anal, ir, nmr | 1387 |
| 3-β-Hydroxyethylpyridazine | 110/1, nmr | 494, 1581 |
| 4-α-Hydroxyethylpyridazine | 60, ir, nmr | 66 |
| 4-β-Hydroxyethylpyridazine | 160/1, nmr | 1581 |
| 1-β-Hydroxyethyl-3,6(1*H*,2*H*)-pyridazinedione | 149–150 | 569 |
| 2-β-Hydroxyethyl-3(2*H*)-pyridazinone | 121–122/1, nmr | 1179 |
| 2-β-Hydroxyethyl-4,5,6-triphenyl-3(2*H*)-pyridazinone | 189 | 1272 |
| 5-Hydroxy-2-δ-hydroxybutyl-3(2*H*)-pyridazinone | 140–141, ir, nmr | 1700 |
| 2-Hydroxy-6-isopentyloxy-3(2*H*)-pyridazinone | — | *H* 76 |
| 1-Hydroxy-6-mercapto-5-methyl-4-thioxo-1,4-dihydro-3(2*H*)-pyridazinone | 182 | 833; *H* 821 |
| 1-Hydroxy-6-mercapto-4-thioxo-1,4-dihydro-3(2*H*)-pyridazinone | — | *H* 821 |
| 2-Hydroxy-6-methoxy-4-methyl-3(2*H*)-pyridazinone | — | *H* 111 |
| 2-Hydroxy-6-methoxy-5-methyl-3(2*H*)-pyridazinone | — | *H* 90 |
| 5-Hydroxy-4-methoxy-2-phenyl-3(2*H*)-pyridazinone | 182–184, ir, nmr | 764, 1213; *H* 126 |
| 2-Hydroxy-6-methoxy-3(2*H*)-pyridazinethione | 138–140 | 679; *H* 821 |
| 2-Hydroxy-6-methoxy-3(2*H*)-pyridazinone | — | *H* 76, 195 |
| 3-(3-Hydroxy-3-methylbut-1-ynyl)-4-methyl-6-phenylpyridazine | 166–167, ir, ms, nmr | 1629 |
| 3-(3-Hydroxy-3-methylbut-1-ynyl)-6-methylpyridazine | 113–114, ir, ms, nmr | 191 |
| 3-(3-Hydroxy-3-methylbut-1-ynyl)-6-methylpyridazine 1-oxide | 158–159, ir, nmr | 191, 834 |
| 3-(3-Hydroxy-3-methylbut-1-ynyl)-6-phenylpyridazine | 120–121, ir, ms, nmr | 1629 |
| 4-Hydroxymethyl-3,6-dimethoxypyridazine | — | *H* 192 |
| 4-Hydroxymethyl-3,6-dimethylpyridazine | — | *H* 397 |
| 5-Hydroxymethyl-3,6-dimethyl-4-pyridazinecarbaldehyde | $H_2NN$=: 190, ir, nmr | 927 |
| 5-Hydroxy-6-methyl-2,4-diphenyl-3(2*H*)-pyridazinone | — | 713 |
| 4-(α-Hydroxy-α-methylethyl)-6-methoxy-3-phenylsulfinylpyridazine | liq, nmr | 2035 |
| 2-Hydroxymethyl-6-iodo-3(2*H*)-pyridazinone | — | *H* 71, 307 |
| 3-Hydroxymethyl-5-methoxy-6-methylpyridazine | — | *H* 397 |
| 4-Hydroxymethyl-3-methoxy-6-methylpyridazine | — | *H* 397 |
| 3-Hydroxymethyl-6-methoxypyridazine | — | 913; *H* 176, 397 |
| 2-Hydroxymethyl-6-methoxy-3(2*H*)-pyridazinone | — | *H* 71 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 2-Hydroxymethyl-6-methyl-4-phenyl-3(2*H*)-pyridazinone | — | 874 |
| 5-Hydroxymethyl-4-methyl-6-phenyl-3(2*H*)-pyridazinone | 187–188 | 370 |
| 4-Hydroxymethyl-6-methylpyridazine | — | *H* 397 |
| 3-Hydroxymethyl-6-methylpyridazine 1-oxide | 123–124, ir, nmr | 527, 529 |
| 6-Hydroxymethyl-2-methylpyridazinium-4-olate | 188, ir, uv; HCl: 176, ir, nmr, uv | 475, 781 |
| 2-Hydroxymethyl-6-methyl-3(2*H*)-pyridazinone | — | *H* 71, 402 |
| 3-Hydroxymethyl-6-methyl-4(1*H*)-pyridazinone | 131 | 441; *H* 396 |
| 6-Hydroxymethyl-3-methyl-4(1*H*)-pyridazinone | — | *H* 132 |
| 5-Hydroxy-2-methyl-4-methylthio-3(2*H*)-pyridazinone | nmr | 1213 |
| 2-Hydroxymethyl-3-oxo-2,3-dihydro-4-pyridazinecarbonitrile | 114–116, ir, nmr | 1300 |
| 6-Hydroxymethyl-4-oxo-1,4-dihydro-3-pyridazinecarboxylic acid | 206, ir, nmr, uv | 475, 778 |
| 6-Hydroxymethyl-5-oxo-2,5-dihydro-3-pyridazinecarboxylic acid | 219, ir, nmr, uv | 475, 778 |
| 4-Hydroxy-2-methyl-1-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 160 |
| 6-Hydroxymethyl-2-phenylpyridazinium-4-olate | 211, ir, nmr, uv; HCl: 176, ir, nmr, uv | 475, 781 |
| 2-Hydroxymethyl-6-phenyl-3(2*H*)-pyridazinone | 158–160, ir, nmr | 441, 1292; *H* 71, 402 |
| 5-Hydroxymethyl-6-phenyl-3(2*H*)-pyridazinone | 174–175 or 198–201, ir, nmr, uv | 370, 1588, 2018 |
| 3-(β-Hydroxy-β-methylpropyl)-6-methylpyridazine | liq, anal, nmr | 827 |
| 2-(β-Hydroxy-α-methylpropyl)-6-methyl-3(2*H*)-pyridazinone | — | *H* 73, 403 |
| 3-(β-Hydroxy-β-methylpropyl)pyridazine | 65–66, nmr | 827 |
| 4-(β-Hydroxy-β-methylpropyl)pyridazine | 93–94, nmr | 827 |
| 3-Hydroxymethylpyridazine | 66, uv | 77, 1040, 1164; *H* 397 |
| 4-Hydroxymethylpyridazine | 83, ir, nmr; HCl: 135–137, ir | 66, 67 |
| 1-Hydroxymethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 153 |
| 5-Hydroxy-2-methyl-3,4(1*H*,2*H*)-pyridazinedione | 265–267, ir, nmr | 531 |
| 2-Hydroxymethyl-3(2*H*)-pyridazinone | — | 441; *H* 65, 402 |
| 3-Hydroxymethyl-4(1*H*)-pyridazinone | 212, nmr | 778; *H* 397 |
| 5-Hydroxy-2-methyl-3(2*H*)-pyridazinone | 176, ir, nmr | 90 |
| 5-Hydroxy-3-methyl-4(1*H*)-pyridazinone | — | 1040 |
| 6-Hydroxymethyl-4(1*H*)-pyridazinone | — | *H* 132 |
| 5-Hydroxy-4-methylthio-2-phenyl-3(2*H*)-pyridazinone | — | 1213 |
| 1-Hydroxy-4-methyl-6-thioxo-1,6-dihydro-3(2*H*)-pyridazinone | 213 | 833; *H* 821 |
| 1-Hydroxy-6-methyl-4-thioxo-1,4-dihydro-3(2*H*)-pyridazinone | 233 | 833; *H* 821 |
| 4-Hydroxymethyl-3,5,6-triphenylpyridazine | 143–145, nmr | 758 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 4-Hydroxy-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarbohydrazide | 224, ir, nmr; $H_2N-\dot{N}H_3$: 190–191, ir, nmr; PhCh=: 184–186, ir, nmr | 1090 |
| 4-Hydroxy-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarbonitrile | 233–237, ir, nmr | 1364 |
| 4-Hydroxy-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylic acid | 251–253, ir | 1090; *H* 136, 444 |
| 4-Hydroxy-6-oxo-1-phenyl-5-phenylazo-1,6-dihydro-3-pyridazinecarboxylic acid | — | *H* 136, 444, 651 |
| 2-Hydroxy-6-phenoxy-3(2*H*)-pyridazinethione | — | *H* 821 |
| 4-Hydroxy-1-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 156 |
| 5-Hydroxy-2-phenyl-3,4(1*H*,2*H*)-pyridazinedione | 202–203 | 531; *H* 169 |
| 5-Hydroxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 85 |
| 2-Hydroxy-6-propoxy-3(2*H*)-pyridazinone | — | *H* 76 |
| 3-β-Hydroxypropyl-6-phenylpyridazine | 103–105, ir, nmr | 836 |
| 3-γ-Hydroxypropylpyridazine | anal, nmr | 1778 |
| 3-(3-Hydroxyprop-1-ynyl)-6-methoxypyridazine | 140, nmr | 1385 |
| 3-(3-Hydroxyprop-1-ynyl)-4-methyl-6-phenylpyridazine | 139, ir, ms, nmr | 1629 |
| 3-(3-Hydroxyprop-1-ynyl)-6-methylpyridazine | 122–123 or 165, ir, nmr | 834, 1629 |
| 3-(3-Hydroxyprop-1-ynyl)-6-phenylpyridazine | 115, ir, ms, nmr | 1629 |
| 1-Hydroxy-4(1*H*)-pyridazinone | st | 2079; *H* 194 |
| 2-Hydroxy-3(2*H*)-pyridazinone | — | *H* 65 |
| 5-Hydroxy-3(2*H*)-pyridazinone | 274–276, ir, nmr, xl st | 90 |
| 5-Hydroxy-4(1*H*)-pyridazinone | 178–179, ir, nmr | 923, 1040; *H* 132 |
| 3-Imino-2,3-dihydro-2-pyridazinamine | mesitylenesulfonate (salt): 138–140, nmr | 412, 929 |
| 4-Imino-1,6-dimethyl-1,4-dihydro-3(2*H*)-pyridazinone | — | *H* 608 |
| 3-Imino-2,5-diphenyl-2,3-dihydro-4-pyridazinecarbonitrile | 173, ir, nmr | 37, 221 |
| 6-Imino-1,4-diphenyl-1,6-dihydro-3,5-pyridazinedicarbonitrile | 218–220 or 250, ir, nmr | 151, 216, 1665 |
| 3-Imino-2,5-diphenyl-6-thioxo-1,2,3,6-tetrahydro-4-pyridazinecarbonitrile | 250 | 1374 |
| 4-Imino-6-methoxy-1-methyl-1,4-dihydro-3(2*H*)-pyridazinone | — | *H* 608 |
| 4-Imino-1-methyl-1,4-dihydro-3-pyridazinamine | HI: 208–210 or 213–214, nmr, $pK_a$, uv | 675, 1132 |
| 5-Imino-2-methyl-2,5-dihydro-3-pyridazinamine | HI: 254–256, nmr, $pK_a$, uv; pic: 228 | 675 |
| 5-Imino-2-methyl-2,5-dihydro-4-pyridazinamine | HI: 229–231, nmr, $pK_a$, uv; pic: 225–226 | 675 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 6-Imino-1-methyl-1,6-dihydro-3-pyridazinamine | HI: 271–272, nmr, p$K_a$, uv; pic: 215–216 | 675 |
| 4-Imino-1-methyl-1,4-dihydro-3(2*H*)-pyridazinone | — | *H* 608 |
| 4-Imino-1-methyl-5,6-diphenyl-1,4-dihydro-3(2*H*)-pyridazinone | — | 814 |
| 5-Imino-2-methyl-3-methylthio-2,5-dihydro-4-pyridazinamine | HI: 199–203, nmr | 325 |
| 5-Imino-2-methyl-6-methylthio-2,5-dihydro-4-pyridazinamine | HI: 261–262, nmr, xl st | 165, 325 |
| 3-Imino-5-methyl-2-phenyl-2,3-dihydro-4-pyridazinecarbonitrile | 117 | 37, 221 |
| 3-Imino-6-methyl-2-phenyl-2,3-dihydro-4,5-pyridazinecarbonitrile | 108, ir | 3 |
| 6-Imino-4-methyl-1-phenyl-1,6-dihydro-3,5-pyridazinedicarbonitrile | 190 or 218, ir, nmr | 395, 1718 |
| 4-Iodo-3,6-diphenylpyridazine | 167–168, nmr | 1591 |
| 4-Iodo-5-isocyanato-2-phenyl-3(2*H*)-pyridazinone | — | *H* 124, 331 |
| 3-Iodo-6-iospropoxypyridazine | — | *H* 178, 267 |
| 4-Iodo-3-methoxymethyl-6-methylpyridazine | 62–63, nmr | 446 |
| 3-Iodo-6-methoxypyridazine | 99–100, 1-MeI: – | 690, 1385; *H* 176, 287 |
| 3-Iodo-6-methyl-4-nitropyridazine 1-oxide | — | *H* 299 |
| 4-Iodo-5-methyl-2-phenyl-3(2*H*)-pyridazinone | — | *H* 124, 331 |
| 3-Iodo-6-methylpyridazine | — | *H* 287, 301, 302, 303 |
| 3-Iodo-6-methylpyridazine 2-oxide | — | *H* 300 |
| 5-Iodo-2-methyl-3(2*H*)-pyridazinone | 182–183 | 1602 |
| 4-Iodo-5-methylthio-2-phenyl-3(2*H*)-pyridazinone | — | *H* 124, 331 |
| 3-Iodo-6-methylthiopyridazine | 116–118, nmr | 1385 |
| 3-Iodo-6-pentyloxypyridazine | 1-MeI: – | 690; *H* 178, 287 |
| 3-Iodo-6-phenylpyridazine | — | *H* 302, 303 |
| 4-Iodo-6-phenylpyridazine | 132, ir, ms, nmr | 2093 |
| 6-Iodo-2-piperidinomethyl-3(2*H*)-pyridazinone | — | *H* 71, 307 |
| 3-Iodo-5-piperidinopyridazine | — | *H* 287, 585 |
| 3-Iodo-6-propoxypyridazine | — | *H* 178, 287 |
| 6-Iodo-3-pyridazinamine | — | *H* 287, 551 |
| 3-Iodopyridazine | 125 or 152, ir, nmr, uv | 1127, 1387 |
| 6-Iodo-3(2*H*)-pyridazinone | — | *H* 66, 307 |
| Isobutanesulfonyloxy-3(2*H*)-pyridazinone | — | *H* 69 |
| 4-Isobutoxy-2-methyl-1-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 160 |
| 3-Isobutoxy-6-phenoxypyridazine | — | 688 |
| 6-Isobutylamino-3-pyridazinesulfonamide | — | *H* 573 |
| Isobutyl 6-oxo-3-phenoxy-1,6-dihydro-1-pyridazinecarboxylate | — | 876 |
| 3-Isobutyl-6-phenylpyridazine | 82–83, nmr | 394 |
| 4-Isobutyl-6-phenylpyridazine | 62, ms, nmr, uv | 1797 |

*continued*

| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 2-Isobutyl-6-phenyl-3(2*H*)-pyridazinone | 166–169/2.5, nmr | 118 |
| 6-Isobutyl-3(2*H*)-pyridazinone | 63, nmr | 1309 |
| 5-(β-Isobutyryl-α-methylhydrazino)-2-methyl-3(2*H*)-pyridazinone | 204–205, ir, nmr | 873 |
| 2-Isobutyrylmethyl-6-methyl-3(2*H*)-pyridazinone | — | *H* 75, 401 |
| 4-Isobutyryl-2-phenyl-3(2*H*)-pyridazinone | 67, ms, nmr | 1877 |
| 4-Isobutyrylpyridazine | ms | 79 |
| 6-Isopentanesulfonyloxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 81 |
| 6-Isopentanesulfonyloxy-3(2*H*)-pyridazinone | — | *H* 69 |
| 6-Isopentyloxy-5-nitro-3-pyridazinamine 2-oxide | — | *H* 198, 604 |
| 3-Isopentyloxy-6-phenoxypyridazine | — | 688 |
| 6-Isopentyloxy-3-pyridazinamine | — | *H* 179 |
| 3-Isopentyloxypyridazine | — | *H* 174 |
| 3-Isopentylpyridazine | ir, nmr | 1660 |
| 6-Isopentyl-3-pyridazinecarbonitrile | 33–35, ir, nmr | 1660 |
| 6-Isopentyl-3(2*H*)-pyridazinone | — | *H* 67 |
| 4-Isopropenyl-3,6-dimethylpyridazine | — | 1567 |
| 3-Isopropoxy-5,6-dimethyl-4-pyridazinecarbonitrile | — | *H* 184, 447 |
| 4-Isopropoxy-5-mercapto-2-phenyl-3(2*H*)-pyridazinone | 78–80, ir, uv | 1896 |
| 3-Isopropoxy-6-methoxypyridazine | — | 673 |
| 4-Isopropoxy-2-methyl-5-morpholino-3(2*H*)-pyridazinone | liq, anal | 1269 |
| 4-Isopropoxy-2-methyl-1-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 160 |
| 5-Isopropoxy-2-methyl-3,4(1*H*,2*H*)-pyridazinedione | 112–114 | 533 |
| 6-Isopropoxy-2-methyl-3(2*H*)-pyridazinone | — | *H* 70 |
| 3-Isopropoxy-6-phenoxypyridazine | 2-MeI: – | 688, 690 |
| 3-Isopropoxy-6-phenylpyridazine | 98–100, nmr | 1674 |
| 6-Isopropoxy-3-pyridazinamine | — | *H* 552 |
| 3-Isopropoxypyridazine | — | *H* 174 |
| 6-Isopropoxy-3(2*H*)-pyridazinone | 140–142 | 640 |
| Isopropyl 1-allyl-6-oxo-1,6-dihydro-3-pyridazinecarboxylate | — | 855 |
| 4-Isopropylamino-3,6-diphenylpyridazine | 113, nmr | 56 |
| 3-Isopropylamino-6-methyl-4-nitropyridazine 1-oxide | — | *H* 604 |
| 3-Isopropylamino-6-morpholinopyridazine | 135–137 | 1330 |
| 3-Isopropylamino-6-phenylthiopyridazine | — | *H* 573, 807 |
| 3-Isopropylamino-4-pyridazinecarbonitrile | 76–77, ir, nmr | 1079 |
| 3-Isopropylamino-4-pyridazinecarboxamide | 208–209, ir, nmr | 1079 |
| Isopropyl 6-amino-3-pyridazinecarboxylate | 168–170, ir, nmr | 1689 |
| 4-Isopropyl-3,6-dimethoxypyridazine | — | *H* 192 |
| 4-Isopropyl-3,6-diphenylpyridazine | 96 to 112, nmr, uv | 407, 438, 499 |
| 3-β-Isopropylhydrazino-4-methyl-6-phenylpyridazine | 167 | 1333 |
| 4-Isopropyl-3-methoxy-6-phenylpyridazine | — | *H* 182 |
| 5-Isopropyl-6-methoxy-3(2*H*)-pyridazinone | — | *H* 89 |
| 6-Isopropyl-5-methoxy-3(2*H*)-pyridazinone | — | *H* 88 |
| 4-Isopropyl-3-methyl-6-phenylpyridazine | liq, nmr | 394 |

*continued*

| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 4-Isopropyl-6-methyl-2-phenyl-3(2*H*)-pyridazinone | — | *H* 106 |
| 4-Isopropyl-6-methylpyridazine | liq, anal, ms, nmr | 1676 |
| 5-Isopropyl-2-methyl-3(2*H*)-pyridazinone | crude: nmr | 327 |
| 4-Isopropyl-2-$\beta$-morpholinoethyl-6-phenyl-3(2*H*)-pyridazinone | — | *H* 104 |
| Isopropyl 6-oxo-3-phenoxy-1,6-dihydro-1-pyridazinecarboxylate | — | 876 |
| Isopropyl 6-oxo-1-vinyl-1,6-dihydro-3-pyridazinecarboxylate | — | 693 |
| 4-Isopropyl-6-phenyl-3-pyridazinamine | 82, nmr | 1381 |
| 4-Isopropyl-6-phenylpyridazine | 62–64, ms, nmr | 1676 |
| 4-Isopropyl-6-phenyl-3(2*H*)-pyridazinone | 161, ir, nmr | 104 |
| 3-Isopropylpyridazine | liq, nmr; pic: 117–119 | 822 |
| 4-Isopropylpyridazine | ms | 79 |
| 6-Isopropylsulfonyl-2-methyl-3(2*H*)-pyridazinone | — | *H* 824 |
| 6-Isopropylsulfonyl-3(2*H*)-pyridazinone | — | *H* 824 |
| 5-Isopropylthio-6-phenyl-3-pyridazinamine | 217–219, ir, nmr; HCl: 172, ir, nmr | 133 |
| 5-Isopropylthio-2-phenyl-3,4(1*H*,2*H*)-pyridazinedione | 101–103, ir, uv | 1896 |
| 2-Isovalerylmethyl-6-methyl-3(2*H*)-pyridazinone | — | *H* 75, 401 |
| 6-Isovaleryloxy-3(2*H*)-pyridazinone | — | *H* 68 |
| 3-Isovalerylpyridazine | ir, nmr | 1660 |
| Maleic hydrazide, *see* 3,6(1*H*,2*H*)-Pyridazinedione | | |
| 5-Mercapto-4-methoxy-2-methyl-3(2*H*)-pyridazinone | 88–90, ir, uv | 1896 |
| 5-Mercapto-4-methoxy-2-phenyl-3(2*H*)-pyridazinone | 140–142, ir, uv | 1896 |
| 5-Mercapto-2-methyl-6-phenyl-3(2*H*)-pyridazinethione | 134–136, ir, nmr | 1087 |
| 5-Mercapto-2-methyl-6-phenyl-3(2*H*)-pyridazinone | 152–154, ir, nmr | 1087 |
| 6-Mercapto-1-methyl-3-phenyl-4(1*H*)-pyridazinone | 239–240, ir, nmr | 1087 |
| 5-Mercapto-2-methyl-4-propoxy-3(2*H*)-pyridazinone | 52–53, ir, uv | 1896 |
| 3-Mercaptomethylpyridazine | — | *H* 830 |
| 4-Mercapto-1-methyl-3,6(1*H*,2*H*)-pyridazinedione | crude: 245–247 | 1129 |
| 5-Mercapto-1-methyl-3,4(1*H*,2*H*)-pyridazinedithione | 151–152, p$K_a$, uv | 689 |
| 5-Mercapto-2-methyl-3,4(1*H*,2*H*)-pyridazinedithione | 118–119, p$K_a$, uv | 689 |
| 5-Mercapto-2-methyl-4-thioxo-1,4-dihydro-3(2*H*)-pyridazinone | — | *H* 802 |
| 5-Mercapto-6-phenyl-3(2*H*)-pyridazinethione | 207–208, ir, nmr | 1087 |
| 5-Mercapto-6-phenyl-3(2*H*)-pyridazinone | 298–304, ir, nmr | 1087 |
| 6-Mercapto-3-phenyl-4(1*H*)-pyridazinone | 261–263, ir, nmr | 1087 |
| 5-Mercapto-2-phenyl-4-thioxo-1,4-dihydro-3(2*H*)-pyridazinone | — | *H* 802 |
| 5-Mercapto-3,4(1*H*,2*H*)-pyridazinedithione | >360, complexes, p$K_a$, uv | 689, 1260; *H* 802 |
| 6-Mercapto-3,4(1*H*,2*H*)-pyridazinedithione | 175 then 360, p$K_a$, uv | 1129 |
| 4-Mercapto-6-thioxo-1,6-dihydro-3(2*H*)-pyridazinone | crude: 150–155 | 1129 |
| 5-Mercapto-4-thioxo-1,4-dihydro-3(2*H*)-pyridazinone | — | *H* 802 |
| 1-Methacryloyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 154 |
| 6-Methanesulfonyloxy-3(2*H*)-pyridazinone | — | *H* 69 |
| 4-Methoxy-3,6-bismethylthiopyridazine | 103–105, ir, ms, nmr | 353 |

*continued*

| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 4-Methoxycarbonyl-5,6-diphenyl-3-pyridazinecarboxylic acid | 134–135, ir, nmr, uv | 1242 |
| 3-β-Methoxycarbonylethylpyridazine | 119–121 | 1778 |
| 6-β-Methoxycarbonylethyl-3(2*H*)-pyridazinone | — | *H* 67 |
| 4-Methoxycarbonylmethyl-1-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 156 |
| 3-Methoxycarbonylmethyl-6-phenyl-4(1*H*)-pyridazinone | 217–218, ir, nmr | 1489 |
| 3-Methoxycarbonylmethylpyridazine | uv | 1164 |
| 4-Methoxycarbonylmethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 151 |
| 2-Methoxycarbonylmethyl-3(2*H*)-pyridazinone | HCl: 154–156 | 147 |
| 4-Methoxycarbonylmethyl-3(2*H*)-pyridazinone | 121–123, ir, nmr, uv | 947 |
| 3-Methoxy-5,6-dimethyl-4-nitropyridazine | — | *H* 184 |
| 3-Methoxy-5,6-dimethyl-4-nitropyridazine 1-oxide | — | *H* 649 |
| 1-Methoxy-3,6-dimethyl-5-nitro-4(1*H*)-pyridazinone | 78–79, uv | 794 |
| 6-Methoxy-2,3-dimethyl-5-oxo-2,5-dihydro-4-pyridazinecarbonitrile | 230–231, ir | 772 |
| 3-Methoxy-4,5-dimethyl-6-phenylpyridazine | 77–78 | 761 |
| 3-Methoxy-4,5-dimethyl-6-phenylsulfonylpyridazine | liq, nmr | 2035 |
| 3-Methoxy-5,6-dimethyl-4-pyridazinamine | — | *H* 561 |
| 3-Methoxy-4,6-dimethylpyridazine | liq, nmr; pic: 121–122 | 762 |
| 3-Methoxy-5,6-dimethylpyridazine | — | *H* 184 |
| 4-Methoxy-3,6-dimethylpyridazine | 73–75 or 78–80, nmr | 529, 1443; *H* 186 |
| 3-Methoxy-5,6-dimethyl-4-pyridazinecarbonitrile | — | *H* 184, 447 |
| 4-Methoxy-3,6-dimethylpyridazine 1,2-dioxide | 221, nmr | 716 |
| 4-Methoxy-3,6-dimethylpyridazine 1-oxide | 149 | 695, 794; *H* 196 |
| 4-Methoxy-3,6-dimethylpyridazine 2-oxide | 136–137 | 791; *H* 196 |
| 1-Methoxy-3,6-dimethylpyridazinium (+anion) | $HSO_4$: – | 479 |
| 1-Methoxy-3,6-dimethyl-4(1*H*)-pyridazinone | — | *H* 134 |
| 3-Methoxy-4,6-dinitropyridazine | — | *H* 182 |
| 3-Methoxy-4,6-dinitropyridazine 1-oxide | 125–126 | 757; *H* 197, 649 |
| 4-Methoxy-3,5-diphenylpyridazine | — | *H* 185 |
| 3-Methoxy-5,6-diphenyl-4-pyridazinecarbonitrile | — | 1056 |
| 4-Methoxy-5,6-diphenyl-3(2*H*)-pyridazinone | — | 809 |
| 6-β-Methoxyethyl-4-methylamino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 107 |
| 4-α-Methoxyethylpyridazine | liq, ms, nmr | 82 |
| 3-Methoxy-6-methoxycarbonylmethylpyridazine | 127–129/2.5, ir, nmr | 962 |
| 4-Methoxy-3-methoxymethyl-6-methylpyridazine | 32–34, nmr; pic: 134–135 | 529 |
| 4-Methoxy-6-methoxymethyl-3-methylpyridazine | liq, nmr; pic: 115 | 529 |
| 6-Methoxy-4-*N*-methylacetamido-2-phenyl-3(2*H*)-pyridazinone | — | *H* 588 |
| 6-Methoxy-4-methylamino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 108, 588 |
| 3-Methoxy-6-methylaminopyridazine | 82–85 | 1330 |
| 3-Methoxy-5-methyl-4,6-dinitropyridazine 1-oxide | 175–176 | 833; *H* 649 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 4-Methoxy-2-methyl-5,6-diphenyl-3(2*H*)-pyridazinone | — | 814 |
| 4-Methoxy-6-methyl-3-methylaminopyridazine | — | *H* 572 |
| 3-Methoxy-6-methyl-4-(*N*-methyl-*N*-nitroamino)pyridazine | — | *H* 182, 607 |
| 3-Methoxymethyl-6-methyl-4-pyridazinamine | 133–135, ir, nmr | 446 |
| 6-Methoxymethyl-3-methyl-4-pyridazinamine | 85–87, ir, nmr | 446 |
| 3-Methoxymethyl-6-methylpyridazine | liq, nmr; pic: 101 | 446, 529 |
| 3-Methoxymethyl-6-methyl-4(1*H*)-pyridazinone | 180, ir, nmr | 446 |
| 3-Methoxy-4-methyl-6-methylthiopyridazine | — | *H* 182, 806 |
| 3-Methoxy-5-methyl-6-methylthiopyridazine | — | *H* 184, 806 |
| 6-Methoxy-2-methyl-4-methylthio-3(2*H*)-pyridazinone | — | 1007 |
| 6-Methoxy-2-methyl-5-methylthio-3(2*H*)-pyridazinone | — | 1007 |
| 4-Methoxy-6-methyl-3-morpholinopyridazine | — | *H* 579 |
| 4-Methoxy-2-methyl-5-morpholino-3(2*H*)-pyridazinone | 72–73 | 1269 |
| 3-Methoxy-6-methyl-4-nitroaminopyridazine | — | *H* 182, 607 |
| 3-Methoxy-6-methyl-5-nitro-4-pyridazinamine | — | *H* 184, 561 |
| 6-Methoxy-2-methyl-5-nitro-3,4(1*H*,2*H*)-pyridazinedione | — | *H* 169, 648 |
| 3-Methoxy-4-methyl-6-nitropyridazine 1-oxide | — | *H* 197, 649 |
| 3-Methoxy-5-methyl-4-nitropyridazine 1-oxide | 148–149 | 833; *H* 649 |
| 3-Methoxy-5-methyl-6-nitropyridazine 1-oxide | 113–115 | 833; *H* 649 |
| 3-Methoxy-6-methyl-4-nitropyridazine 1-oxide | 98–99, nmr | 952; *H* 197, 649 |
| 3-Methoxy-6-methyl-5-nitropyridazine 1-oxide | 105–106, ir, nmr | 904 |
| 6-Methoxy-2-methyl-5-oxo-2,5-dihydro-3-pyridazinecarbonitrile | — | *H* 133, 455 |
| 6-Methoxy-2-methyl-5-oxo-3-phenyl-2,5-dihydro-4-pyridazinecarbonitrile | 228, ir | 772 |
| 4-Methoxy-6-methyl-3-phenethylaminopyridazine | — | *H* 576 |
| 3-Methoxy-4-methyl-6-phenylpyridazine | 53 to 61, nmr | 761, 1569, 1632; *H* 182 |
| 4-Methoxy-1-methyl-2-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 160 |
| 4-Methoxy-2-methyl-1-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 160 |
| 3-Methoxy-4-methyl-6-phenylpyridazine 2-oxide | 110 | 1104 |
| 1-Methoxy-6-methyl-3-phenylpyridazinium (+ anion) | $HSO_4$: – | 479 |
| 2-Methoxy-4-methyl-6-phenyl-3(2*H*)-pyridazinone | 128, ir, nmr, uv | 1104 |
| 6-Methoxy-5-methyl-2-phenyl-3(2*H*)-pyridazinone | — | *H* 94 |
| 3-Methoxy-5-methyl-6-phenylsulfinylpyridazine | liq, nmr | 2035 |
| 6-Methoxy-2-methyl-4-phenylsulfinyl-3(2*H*)-pyridazinone | 162–167, nmr | 1672 |
| 6-Methoxy-2-methyl-5-phenylsulfinyl-3(2*H*)-pyridazinone | 140–142, nmr | 1672 |
| 3-Methoxy-4-methyl-6-phenylsulfonylpyridazine | liq, nmr | 2035 |
| 3-Methoxy-5-methyl-6-phenylsulfonylpyridazine | liq, nmr | 2035 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 6-Methoxy-2-methyl-5-phenylsulfonyl-3(2*H*)-pyridazinone | 167–169 | 1672 |
| 4-Methoxy-6-methyl-3-phenylthiopyridazine | — | *H* 808 |
| 6-Methoxy-2-methyl-4-phenylthio-3(2*H*)-pyridazinone | 131–1332, nmr | 1672 |
| 6-Methoxy-2-methyl-5-phenylthio-3(2*H*)-pyridazinone | 147–148, nmr | 1672 |
| 4-Methoxy-2-methyl-6-phenyl-5-vinyl-3(2*H*)-pyridazinone | 78–80, ir, nmr | 1832 |
| 4-Methoxy-6-methyl-3-piperidinopyridazine | — | *H* 578 |
| 3-Methoxy-6-methyl-4-pyridazinamine | — | *H* 181, 458 |
| 4-Methoxy-6-methyl-3-pyridazinamine | — | *H* 186, 554 |
| 6-Methoxy-5-methyl-3-pyridazinamine | — | *H* 182, 555 |
| 3-Methoxy-5-methyl-4-pyridazinamine 1-oxide | 203–204, ir, nmr | 902 |
| 3-Methoxy-6-methyl-4-pyridazinamine 1-oxide | — | *H* 197, 603 |
| 6-Methoxy-3-methyl-4-pyridazinamine 2-oxide | 180–181, ir, nmr | 904 |
| 6-Methoxy-5-methyl-3-pyridazinamine 2-oxide | — | *H* 197, 604 |
| 3-Methoxy-4-methylpyridazine | nmr | 762; *H* 183 |
| 3-Methoxy-5-methylpyridazine | — | *H* 183 |
| 3-Methoxy-6-methylpyridazine | 102–104/18 | 823 |
| 4-Methoxymethylpyridazine | liq, ms, nmr | 82 |
| 4-Methoxy-5-methylpyridazine | 40–42, ms, nmr | 82 |
| 4-Methoxy-6-methylpyridazine | liq, anal, nmr | 1443 |
| 4-Methoxy-6-methyl-3-pyridazinecarbonitrile 2-oxide | — | *H* 196, 447 |
| 3-Methoxy-6-methyl-4-pyridazinecarboxamide | — | *H* 181, 436 |
| 5-Methoxy-6-methyl-3-pyridazinecarboxylic acid | — | *H* 196, 435 |
| 3-Methoxy-6-methyl-4,5-pyridazinediamine | — | *H* 184, 599 |
| 1-Methoxymethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 153 |
| 5-Methoxy-2-methyl-3,4(1*H*,2*H*)-pyridazinedione | 162–164, nmr | 224, 533, 1213 |
| 3-Methoxy-4-methylpyridazine 1-oxide | — | *H* 194 |
| 3-Methoxy-5-methylpyridazine 1-oxide | 61–62 | 833 |
| 3-Methoxy-6-methylpyridazine 1-oxide | — | 913 |
| 4-Methoxy-3-methylpyridazine 1-oxide | — | *H* 194 |
| 4-Methoxy-6-methylpyridazine 1-oxide | — | *H* 196 |
| 4-Methoxy-6-methylpyridazine 2-oxide | — | *H* 196 |
| 6-Methoxy-2-methyl-3(2*H*)-pyridazinethione | — | *H* 797 |
| 6-Methoxy-5-methyl-3(2*H*)-pyridazinethione | — | *H* 798 |
| 5-Methoxy-2-methyl-3(2*H*)-pyridazinone | 107–108 or 113, ir, nmr, xl st | 90, 1821 |
| 6-Methoxy-2-methyl-3(2*H*)-pyridazinone | 63–65 or 68, ir, nmr, p$K_a$, uv, xl st | 95, 175, 332, 417, 1128, 1167, 1548; *H* 70 |
| 6-Methoxy-4-methyl-3(2*H*)-pyridazinone | — | *H* 98 |
| 6-Methoxy-5-methyl-3(2*H*)-pyridazinone | — | *H* 89 |
| 3-Methoxy-6-methylsulfinylpyridazine | 85–87, nmr | 304 |
| 3-Methoxy-6-methylsulfonylpyridazine | 99–102, nmr | 304 |
| 3-Methoxy-6-methylsulfonypyridazine 1-oxide | 193–195, nmr | 304 |
| 3/5-Methoxy-5/3-methylsulfonyl-4(1*H*)-pyridazinone | 268–270 | 689 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3-Methoxy-6-methylthio-5-phenylpyridazine | — | 2024 |
| 6-Methoxy-5-methylthio-3-pyridazinamine | — | *H* 810 |
| 3-Methoxy-6-methylthiopyridazine | 88–89, nmr | 304, 2024; *H* 796, 803, 818 |
| 6-Methoxy-4-methylthio-3(2*H*)-pyridazinone | — | 1007 |
| 6-Methoxy-5-methylthio-3(2*H*)-pyridazinone | — | 1007 |
| 3-Methoxy-4-nitro-5,6-diphenylpyridazine | — | 795 |
| 4-Methoxy-5-nitro-2-phenyl-3(2*H*)-pyridazinone | — | *H* 125, 648 |
| 6-Methoxy-5-nitro-3-pyridazinamine 2-oxide | 183–184 | 952; *H* 197, 604 |
| 3-Methoxy-6-nitropyridazine | — | *H* 176, 648 |
| 3-Methoxy-4-nitropyridazine 1-oxide | — | *H* 194, 649 |
| 3-Methoxy-4-nitropyridazine 2-oxide | — | *H* 196, 649 |
| 3-Methoxy-5-nitropyridazine 1-oxide | — | *H* 197 |
| 3-Methoxy-6-nitropyridazine 1-oxide | — | *H* 195, 649 |
| 3-Methoxy-6-oxo-1,6-dihydro-4-pyridazinecarboxamide | 250–252 | 820; *H* 89, 439 |
| 3-Methoxy-6-oxo-1,6-dihydro-4-pyridazinecarboxylic acid | — | *H* 89, 439 |
| 5-Methoxy-6-oxo-1,6-dihydro-4-pyridazinecarboxylic acid | — | *H* 125, 440 |
| 4-Methoxy-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarbonitrile | 216, ir, ms, nmr | 17 |
| 3-Methoxy-6-phenoxypyridazine | — | *H* 191 |
| 3-Methoxy-6-phenylethynylpyridazine | 96–98, ir, nmr | 834, 1385 |
| 3-Methoxy-6-phenylpyridazine | 110 to 116, nmr | 823, 1385, 1569; *H* 176 |
| 3-Methoxy-6-phenyl-4-pyridazinecarbonitrile | 136–137, ir | 772 |
| 4-Methoxy-1-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 157 |
| 4-Methoxy-2-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 157 |
| 5-Methoxy-2-phenyl-3,4(1*H*,2*H*)-pyridazinedione | nmr | 1213 |
| 6-Methoxy-2-phenyl-3,4(1*H*,2*H*)-pyridazinedione | — | *H* 169 |
| 5-Methoxy-6-phenyl-3(2*H*)-pyridazinethione | 175–177, ir, nmr | 1087 |
| 6-Methoxy-2-phenyl-3(2*H*)-pyridazinethione | — | *H* 798 |
| 6-Methoxy-1-phenyl-4(1*H*)-pyridazinimine | HCl: 133–135; $MeHSO_4$: 176 | 1344 |
| 1-Methoxy-3-phenylpyridazinium (+anion) | $HSO_4$: – | 479 |
| 4-Methoxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 84 |
| 5-Methoxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 85 |
| 6-Methoxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 81 |
| 6-Methoxy-5-phenyl-3(2*H*)-pyridazinone | — | *H* 89 |
| 5-Methoxy-2-phenyl-4-thioxo-1,4-dihydro-3(2*H*)-pyridazinone | — | *H* 801 |
| 3-Methoxy-6-piperidinopyridazine | — | *H* 176 |
| 3-Methoxy-6-piperidinopyridazine 1-oxide | — | *H* 604 |
| 3-Methoxy-6-piperidinopyridazine 2-oxide | — | *H* 604 |
| 3-Methoxy-6-pivaloylmethylpyridazine | 48–49, ir, nmr | 326 |
| 3-Methoxy-6-propoxypyridazine | — | 673 |

*continued*

| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3-Methoxy-4-pyridazinamine | 126–127, ir, nmr | 1072, 1873, 1900; *H* 183, 560 |
| 6-Methoxy-3-pyridazinamine | $pK_a$ | 98; *H* 176, 551 |
| 6-Methoxy-4-pyridazinamine | — | *H* 183, 560 |
| 3-Methoxy-4-pyridazinamine 1-oxide | — | *H* 194, 603 |
| 3-Methoxy-4-pyridazinamine 2-oxide | — | *H* 196 |
| 6-Methoxy-3-pyridazinamine 1-oxide | — | *H* 195 |
| 6-Methoxy-3-pyridazinamine 2-oxide | — | *H* 195, 603 |
| 6-Methoxy-4-pyridazinamine 2-oxide | — | *H* 604 |
| 3-Methoxypyridazine | nmr, $pK_a$, uv | 95, 403, 450, 1128; *H* 174 |
| 4-Methoxypyridazine | 42–44, nmr | 1128, 1443; *H* 185 |
| 6-Methoxy-3-pyridazinecarbaldehyde | — | 913; *H* 396 |
| 3-Methoxy-4-pyridazinecarbonitrile | 85–87, ir, nmr, uv | 1300 |
| 6-Methoxy-3-pyridazinecarbonitrile | 92–93 | 964; *H* 176, 446 |
| 6-Methoxy-3-pyridazinecarboxamide oxime | 200–202 | 946 |
| 6-Methoxy-3-pyridazinecarboxylic acid | 160–163, nmr | 74 |
| 3-Methoxy-4,5-pyridazinediamine | — | *H* 599 |
| 6-Methoxy-3,5-pyridazinediamine | — | *H* 182 |
| 3-Methoxypyridazine 1-oxide | nmr | 450; *H* 194 |
| 3-Methoxypyridazine 2-oxide | — | *H* 196 |
| 4-Methoxypyridazine 1-oxide | — | *H* 194 |
| 4-Methoxypyridazine 2-oxide | — | *H* 196 |
| 6-Methoxy-3(2*H*)-pyridazinone | 133 | 365; *H* 796, 797 |
| 3-Methoxypyridazinium 1-acetamidate | 136–137; pic: 145–146 | 756 |
| 3-Methoxypyridazinium-1-benzamidate | 169; pic: 176–177 | 756 |
| 1-Methoxypyridazinium ( + anion) | $HSO_4$: – | 479 |
| 1-Methoxy-4(1*H*)-pyridazinone | $pK_a$ | 1167; *H* 134 |
| 4-Methoxy-3(2*H*)-pyridazinone | 226, nmr | 766 |
| 5-Methoxy-3(2*H*)-pyridazinone | 246–249 or 253-255, ir, nmr, xl st | 90, 1821 |
| 6-Methoxy-3(2*H*)-pyridazinone | 162–164, ir, nmr | 332, 1128, 1597; *H* 68 |
| 3-Methoxy-6-trimethylsiloxypyridazine | 54–56, 82–83/2, nmr, uv | 1172 |
| 3-(*N*-Methylacetamido)-6-morpholinopyridazine | 96–98, nmr | 1085 |
| Methyl 4-acetamido-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate | 175–177, ir, nmr | 1117 |
| Methyl 4-acetoxy-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate | 121–122, ir, nmr | 1090 |
| 2-δ-Methylaminobutyl-6-phenyl-3(2*H*)-pyridazinone | crude: liq, ir, nmr | 1289, 1337 |
| Methyl 3-amino-6-chloro-4-pyridazinecarboximidate | 186–188, nmr | 820 |
| Methyl 4-amino-5-cyano-6-imino-1-phenyl-1,6-dihydro-3-pyridazinecarboximidate | 205–207, ir | 1365 |
| Methyl 4-amino-5-cyano-6-imino-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate | 244 | 51 |

*continued*

| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3-Methylamino-4,6-dinitropyridazine 1-oxide | 154–155 | 760 |
| 2-*β*-Methylaminoethyl-6-morpholino-3(2*H*)-pyridazinone | HCl: 174–178 | 1760 |
| Methyl 5-amino-2-methyl-3,6-dioxo-1,2,3,6-tetrahydro-1-pyridazinecarbodithioate | — | 984 |
| Methyl 5-amino-1-methyl-6-oxo-3-phenyl-1,6-dihydro-4-pyridazinecarboxylate | 184–186, ir, nmr | 1832 |
| 4-Methylaminomethylpyridazine | liq, anal, ir, nmr | 1752 |
| Methyl 3-amino-6-methyl-4-pyridazinecarboxylate | — | *H* 436, 555 |
| 3-Methylaminomethyl-4(1*H*)-pyridazinone 2-oxide | — | *H* 196, 610 |
| 4-Methylamino-5-methylsulfonyl-2-phenyl-3(2*H*)-pyridazinone | 170–172 | 533 |
| 3-Methylamino-6-methylthiopyridazine | — | *H* 572, 796, 803 |
| 3-Methylamino-6-morpholinopyridazine | 195–197 | 1330 |
| Methyl 4-amino-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate | 232–234, ir, nmr | 1117 |
| 4-Methylamino-2-phenyl-6-propylamino-3(2*H*)-pyridazinone | — | *H* 109 |
| 6-Methylamino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 80, 594 |
| 3-Methylamino-4-pyridazinamine | — | *H* 597 |
| 4-Methylamino-3-pyridazinamine | — | *H* 497 |
| 5-Methylamino-3-pyridazinamine | 205–206, nmr | 447 |
| 3-Methylaminopyridazine | — | *H* 572 |
| 4-Methylaminopyridazine | — | *H* 582 |
| Methyl 6-amino-3-pyridazinecarboxylate | — | *H* 434, 553 |
| Methyl 6-amino-3-pyridazinesulfenate | — | *H* 553 |
| 6-Methylamino-3(2*H*)-pyridazinethione | — | *H* 572, 796, 797 |
| Methyl 4-anilino-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate | 157–158, ir, nmr | 1117 |
| Methyl 3-anilino-4-pyridazinecarboximidate | 130, nmr | 1079 |
| Methyl 3-anilino-4-pyridazinecarboxylate | 91–92, nmr | 1079 |
| Methyl 4-azido-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate | 129–130, ir | 1117 |
| Methyl 4-benzenesulfonyloxy-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate | 131–133, ir, nmr | 1090 |
| Methyl 5-benzoyl-6-oxo-1,4-diphenyl-1,6-dihydro-3-pyridazinecarboxylate | 196 | 391 |
| Methyl 5-benzoyl-4-pyridazinecarboxylate | 87–88, ir, nmr, uv | 46, 702 |
| Methyl 4-benzylamino-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate | 150–152, ir, nmr | 1117 |
| Methyl 6-benzylamino-3-pyridazinecarboxylate | 155–157 | 1592, 1773 |
| Methyl 5-benzyl-6-oxo-1,4-diphenyl-1,6-dihydro-3-pyridazinecarboxylate | 120–121 | 391 |
| Methyl 4-benzylthio-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate | 206–207, ir, nmr | 1117 |
| Methyl 5,6-bis(diisopropylamino)-3-pyridazinecarboxylate | 40–42, 210/0.7, ir, ms, nmr | 1070 |
| Methyl 3,6-bismethylthio-4-pyridazinecarboxylate | — | 353 |
| 1-Methyl-3,5-bismethylthio-4(1*H*)-pyridazinethione | 317–318, uv | 689 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 2-Methyl-4,5-bismethylthio-3(2*H*)-pyridazinethione | 139–140, p$K_a$, uv | 689 |
| 2-Methyl-4,6-bismethylthio-3(2*H*)-pyridazinethione | 135–137, ir, nmr | 1129 |
| 2-Methyl-5,6-bismethylthio-3(2*H*)-pyridazinethione | 150–151, p$K_a$, nmr, uv | 1129 |
| 2-Methyl-5,6-bismethylthiopyridazinium-4-olate | 153–155 | 689 |
| 1-Methyl-3,5-bismethylthio-4(1*H*)-pyridazinone | 150–152 | 689 |
| 2-Methyl-4,5-bismethylthio-3(2*H*)-pyridazinone | 99–100, nmr | 689, 1213; *H* 125, 815 |
| 2-Methyl-4,6-bismethylthio-3(2*H*)-pyridazinone | 131–132, nmr | 1129 |
| 2-Methyl-5,6-bismethylthio-3(2*H*)-pyridazinone | 151–152, nmr | 1129 |
| Methyl 4-bromo-2,5-dimethyl-3,6-dioxo-1,2,3,6-tetrahydro-1-pyridazinecarbodithioate | — | 1460 |
| Methyl 5-bromo-4-hydroxy-6-oxo-1-phenyl-1,6-dihydro-3-pyridzinecarboxylate | 162–163, ir, nmr | 1549 |
| Methyl 5-bromo-2-methyl-3,6-dioxo-1,2,3,6-tetrahydro-1-pyridazinecarbodithioate | — | 984 |
| Methyl 5-bromo-6-methyl-4-oxo-1-phenyl-1,4-dihydro-3-pyridazinecarboxylate | — | *H* 134, 333 |
| Methyl 6-bromo-3-pyridazinecarboxylate | 159–160, nmr, uv | 1127 |
| 1-(3-Methylbut-2-enylidene)amino-3,6(1*H*,2*H*)-pyridazinedione | — | 1050 |
| Methyl 6-butylamino-3-pyridazinecarboxylate | 119–121 | 1592, 1773 |
| Methyl 1-butyl-6-oxo-1,6-dihydro-3-pyridazinecarboxylate | — | *H* 73, 438 |
| Methyl 4-butylthio-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate | 125–127, ir, nmr | 1117 |
| Methyl 4-*t*-butylthio-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate | 128–130, ir, nmr | 1117 |
| 2-$\beta$-(*N*-Methylcarbamoyl)ethyl-6-phenyl-3(2*H*)-pyridazinone | 165–167 | 1288 |
| Methyl 5-carbamoyl-4-pyridazinecarboxylate | 137–138, ir, nmr | 1231 |
| Methyl 2-$\beta$-chloroethyl-5-methyl-3,6-dioxo-1,2,3,6-tetrahydro-1-pyridazinecarbodithioate | — | 1460 |
| Methyl 5-chloro-4-hydroxy-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate | 137–138, ir, nmr | 1549 |
| Methyl 6-chloro-3-methoxy-4-pyridazinecarboximidate | 97–98, nmr | 820 |
| Methyl 5-chloro-1-methyl-6-oxo-1,6-dihydro-4-pyridazinecarboxylate | — | *H* 315 |
| Methyl 3-chloro-6-oxo-1,6-dihydro-4-pyridazinecarboxylate | — | *H* 87, 326, 439 |
| Methyl 6-chloro-3-oxo-2,3-dihydro-4-pyridazinecarboxylate | — | *H* 96, 323, 440 |
| Methyl 4-chloro-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate | 105–106, ir, nmr | 1117 |
| Methyl 6-chloro-3-pyridazinecarboxylate | 145 or 147–149 | 1127, 1592; *H* 276 |
| Methyl 5-cyano-4-ethyl-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate | 139, ir, nmr | 1808 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| Methyl 4-cyclohexylthio-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate | 141–143, ir, nmr | 1117 |
| Methyl 4,6-di-*t*-butyl-5-diisopropylamino-6-pyridazinecarboxylate | 96, ir, nmr | 1123 |
| Methyl 2,5-dimethyl-3,6-dioxo-1,2,3,6-tetrahydro-1-pyridazinecarbodithioate | — | 1460 |
| Methyl 1,5-dimethyl-6-oxo-1,6-dihydro-4-pyridazinecarboxylate | 102–103, ir, ms, nmr | 231 |
| Methyl 2,5-dimethyl-3-oxo-2,3-dihydro-4-pyridazinecarboxylate | 81, ir, ms, nmr | 231 |
| Methyl 5,6-dimethyl-3-oxo-2,3-dihydro-4-pyridazinecarboxylate | — | *H* 128, 441 |
| 4-Methyl-3,6-dimorpholinopyridazine | 136–138, ir, nmr | 544 |
| Methyl 3,6-dioxo-1,2,3,6-tetrahydro-1-pyridazinecarbodithioate | — | 984 |
| 3-Methyl-4,6-diphenylpyridazine | 91–92 or 92–93, nmr, uv | 394, 499 |
| 3-Methyl-5,6-diphenylpyridazine | 121–123, ir, ms, nmr | 314, 499 |
| 4-Methyl-3,6-diphenylpyridazine | 132 to 136, ms, nmr | 28, 29, 43, 278, 319, 722 |
| Methyl 3,6-diphenyl-4-pyridazinecarboxylate | 122, nmr | 41 |
| Methyl 5,6-diphenyl-4-pyridazinecarboxylate | 141, ir, nmr, uv | 1242 |
| 3-Methyl-5,6-diphenyl-4-pyridazinecarboxylic acid | 180–181, ir, ms, nmr | 314 |
| 2-Methyl-5,6-diphenyl-3,4(1*H*,2*H*)-pyridazinedione | — | 814 |
| 1-Methyl-3,5-diphenyl-4(1*H*)-pyridazinethione | 122–123, nmr | 249 |
| 1-Methyl-3,5-diphenyl-4(1*H*)-pyridazinone | 165–167, nmr | 249 |
| 2-Methyl-4,6-diphenyl-3(2*H*)-pyridazinone | 120, ir, nmr | 1081 |
| 2-Methyl-5,6-diphenyl-3(2*H*)-pyridazinone | — | *H* 91 |
| 3-Methyl-1,6-diphenyl-4(1*H*)-pyridazinone | — | *H* 136 |
| 4-Methyl-2,6-diphenyl-3(2*H*)-pyridazinone | — | *H* 107 |
| 4-Methyl-5,6-diphenyl-3(2*H*)-pyridazinone | — | 868 |
| 6-Methyl-1,3-diphenyl-4(1*H*)-pyridazinone | — | *H* 136 |
| 6-Methyl-3,5-diphenyl-4(1*H*)-pyridazinone | 304–305, ir, nmr | 311 |
| 3-Methyl-5,6-dipropylpyridazine | liq, anal, ir, nmr, uv | 298 |
| Methyl 5-ethoxycarbonyl-3,6-dimethyl-4-pyridazinecarboxylate | 94–95, ms, nmr | 2107 |
| Methyl 2-ethoxycarbonylmethyl-5-methyl-3,6-dioxo-1,2,3,6-tetrahydro-1-pyridazinecarbodithioate | — | 1460 |
| Methyl 2-ethyl-5-methyl-3,6-dioxo-1,2,3,6-tetrahydro-1-pyridazinecarbodithioate | — | 1460 |
| Methyl 6-ethylsulfonyl-3-pyridazinecarboxylate | 148–149 | 1592 |
| Methyl 4-ethylthio-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate | 140–142, ir, nmr | 1117 |
| Methyl 6-ethylthio-3-pyridazinecarboxylate | 129–131 | 1592 |
| Methyl 6-fluoro-3-pyridazinecarboxylate | 125, nmr, uv | 1127 |
| Methyl 3-formyl-6-methyl-4-pyridazinecarboxylate | $Me_2$acetal: 135–137/0.4, nmr | 996 |
| Methyl 6-hydrazinocarbonyl-3-pyridazinecarboxylate | 242, ir, nmr | 1077 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3-α-Methylhydrazino-6-morpholinopyridazine | 2HCl: 171–174 or 183–190 | 57, 1293 |
| Methyl 4-hydrazino-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate | 205, ir, nmr | 1117 |
| 5-α-Methylhydrazino-2-phenyl-3(2*H*)-pyridazinone | 192–194, ir, nmr | 873 |
| 5-α-Methylhydrazino-4-pyridazinamine | crude: liq | 908 |
| 5-α-Methylhydrazino-3(2*H*)-pyridazinone | 230–231, ir, nmr | 873 |
| Methyl 4-hydroxy-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate | 129–130, ir, nmr | 1090; *H* 136, 444 |
| Methyl 6-iodo-3-pyridazinecarboxylate | 165, nmr, uv | 1127 |
| Methyl 4-isopropylthio-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate | 118–120, ir, nmr | 1117 |
| Methyl 3-methoxy-6-oxo-1,6-dihydro-4-pyridazinecarboxylate | — | *H* 89, 439 |
| Methyl 4-methoxy-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate | 151, ir, ms, nmr | 17, 269 |
| Methyl 6-methoxy-4-oxo-1-phenyl-1,4-dihydro-3-pyridazinecarboxylate | — | *H* 136, 442 |
| Methyl-4-methoxy-1-phenyl-6-phenylimino-1,6-dihydro-3-pyridazinecarboxylate | 104, ir, ms, nmr | 17, 269 |
| Methyl 6-methoxy-3-pyridazinecarboxylate | — | *H* 176, 434 |
| 1-Methyl-5-β-methylallyl-2-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 160 |
| *N*-Methyl-3-methylamino-5,6-diphenyl-4-pyridazinecarboxamide | — | *H* 436, 572 |
| 6-Methyl-4-methylaminomethyl-3(2*H*)-pyridazinone 1-oxide | — | *H* 611 |
| 2-Methyl-4-methylamino-5-methylsulfonyl-3(2*H*)-pyridazinone | 135–136 | 533 |
| 3-Methyl-6-methylamino-5-nitropyridazine 2-oxide | 144–145 | 760 |
| 4-Methyl-3-methylamino-6-phenylpyridazine | 132, nmr | 1333 |
| 1-Methyl-5-methylamino-2-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 159, 595 |
| 6-Methyl-4-methylamino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 106, 588 |
| 6-Methyl-3-methylamino-4(1*H*)-pyridazinone | — | *H* 572 |
| 6-Methyl-2-(2-methylbutyryl)methyl-3(2*H*)-pyridazinone | — | *H* 75, 401 |
| Methyl 2-methyl-3,6-dioxo-1,2,3,6-tetrahydro-1-pyridazinecarbodithioate | — | 984 |
| Methyl 4-methyl-3,6-dioxo-1,2,3,6-tetrahydro-1-pyridazinecarbodithioate | — | 1457 |
| Methyl 5-methyl-3,6-dioxo-1,2,3,6-tetrahydro-1-pyridazinecarbodithioate | — | 1460 |
| 2-Methyl-4-α-methylhydrazino-5-methylsulfonyl-3(2*H*)-pyridazinone | 172–174 | 533 |
| 4-Methyl-3-α-methylhydrazino-6-phenylpyridazine | crude: 98; HCl: 167, nmr; $H_2C$=: 77 | 1333 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 2-Methyl-4-α-methylhydrazino-3(2*H*)-pyridazinone | 109–110, ir, nmr | 533 |
| 2-Methyl-5-α-methylhydrazino-3(2*H*)-pyridazinone | 185–187, ir, nmr | 1061 |
| Methyl 2-methyl-5-morpholino-3,6-dioxo-1,2,3,6-tetrahydro-1-pyridazinecarbodithioate | — | 984 |
| 3-Methyl-6-(*N*-methyl-*N*-nitroamino)pyridazine | — | *H* 607 |
| 4-Methyl-3-(*N*-methyl-*N*-nitrosoamino)-6-phenylpyridazine | 110 | 1333 |
| Methyl 1-methyl-6-oxo-1,6-dihydro-3-pyridazinecarboxylate | — | *H* 70, 437 |
| Methyl 6-methyl-3-oxo-2,3-dihydro-4-pyridazinecarboxylate | — | *H* 97, 441 |
| Methyl 4-methyl-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylate | 126–127, nmr | 1350, 1351; *H* 92, 438 |
| Methyl 5-methyl-4-oxo-1-phenyl-1,4-dihydro-3-pyridazinecarboxylate | 161, ir nmr | 1808 |
| Methyl 6-methyl-4-oxo-1-phenyl-1,4-dihydro-3-pyridazinecarboxylate | — | *H* 135, 442 |
| Methyl 4-methyl-6-oxo-1-phenyl-5-phenylazo-1,6-dihydro-3-pyridazinecarboxylate | — | *H* 652 |
| Methyl 4-methyl-6-phenyl-3-pyridazinecarboxylate | 128, ir, nmr | 1907 |
| Methyl 5-methyl-6-phenyl-3-pyridazinecarboxylate | 100, ir, nmr | 1907 |
| 2-Methyl-5-(α-methyl-β-propionylhydrazino)-3(2*H*)-pyridazinone | 181–182, ir, nmr | 873 |
| Methyl 6-methyl-3-pyridazinecarboxylate | 92, ir, nmr | 1066, 1907 |
| 4-Methyl-3-methylsulfonyl-6-phenylpyridazine | 210, ir | 371 |
| 2-Methyl-5-methylsulfonyl-3,4(1*H*, 2*H*)-pyridazinedione | 257–259 | 533 |
| 3-Methyl-6-methylsulfonylpyridazine 2-oxide | — | *H* 821 |
| 2-Methyl-6-methylsulfonyl-3(2*H*)-pyridazinone | — | *H* 824 |
| 2-{α-Methyl-β-[*N*-methyl(thiocarbamoyl)]ethyl}-3(2*H*)-pyridazinone | 155–157 | 1288 |
| 2-{β-Methyl-β-[*N*-methyl(thiocarbamoyl)]ethyl}-3(2*H*)-pyridazinone | 172–175 | 1288 |
| 6-Methyl-2-(α-methyl-β-thiocarbamoylethyl)-3(2*H*)-pyridazinone | 101–104 | 1288 |
| 6-Methyl-2-(β-methyl-β-thiocarbamoylethyl)-3(2*H*)-pyridazinone | 106–107 | 1288 |
| 3-Methyl-6-methylthio-5-nitropyridazine | 92–94, nmr, uv | 259 |
| 4-Methyl-6-methylthio-5-nitropyridazine | 94–96, nmr, uv | 259 |
| 4-Methyl-3-methylthio-6-phenylpyridazine | 67–68, ir, uv | 371 |
| 3-Methyl-6-methylthiopyridazine | — | *H* 803, 818, 819 |
| 1-Methyl-4-methylthio-3,6(1*H*,2*H*)-pyridazinedione | 297–299 or 301–302, nmr | 1007, 1129 |
| 1-Methyl-5-methylthio-3,6(1*H*,2*H*)-pyridazinedione | — | 1007 |
| 2-Methyl-5-methylthio-3,4(1*H*,2*H*)-pyridazinedione | 155–157, ir, nmr, uv | 1213, 1896 |
| 3-Methyl-6-methylthiopyridazine 2-oxide | — | *H* 821 |
| 2-Methyl-6-methylthio-3(2*H*)-pyridazinethione | 86–87, $pK_a$, uv | 95; *H* 809 |
| 4-Methyl-6-methylthio-3(2*H*)-pyridazinone | — | *H* 98, 808 |
| 5-Methyl-6-methylthio-3(2*H*)-pyridazinone | — | *H* 808 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 4-Methyl-2-β-morpholinoethyl-6-phenyl-3(2*H*)-pyridazinone | — | *H* 102 |
| 6-Methyl-2-β-morpholinoethyl-3(2*H*)-pyridazinone | — | *H* 73 |
| 4-Methyl-2-morpholinomethyl-6-phenyl-3(2*H*)-pyridazinone | 166–167, ir | 371 |
| 6-Methyl-2-morpholinomethyl-4-phenyl-3(2*H*)-pyridazinone | — | 874 |
| 6-Methyl-4-morpholinomethyl-2-phenyl-3(2*H*)-pyridazinone | — | *H* 612 |
| 6-Methyl-2-morpholinomethyl-3(2*H*)-pyridazinone | — | *H* 72 |
| 6-Methyl-4-morpholinomethyl-3(2*H*)-pyridazinone | — | *H* 610 |
| 6-Methyl-4-morpholinomethyl-3(2*H*)-pyridazinone 1-oxide | — | *H* 197, 610 |
| 2-Methyl-5-morpholino-4-nitro-3(2*H*)-pyridazinone | 140–141, ir, nmr | 534 |
| 2-Methyl-5-morpholino-3-oxo-2,3-dihydro-4-pyridazinecarbaldehyde | 140–141; $H_2NN{=}$: 167–168 | 1603 |
| 2-Methyl-5-morpholino-4-pentyloxy-3(2*H*)-pyridazinone | liq, anal | 1269 |
| 1-Methyl-5-morpholino-2-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 160, 595 |
| 1-Methyl-4-morpholino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 106, 590 |
| 2-Methyl-5-morpholino-4-propoxy-3(2*H*)-pyridazinone | 87–89 | 1269 |
| 2-Methyl-5-morpholino-6-propoxy-3(2*H*)-pyridazinone | — | 1725 |
| 4-Methyl-6-morpholinopyridazine | 124–126 | 544 |
| 2-Methyl-5-morpholino-3,4(1*H*,2*H*)-pyridazinedione | 229–231 | 1269 |
| 2-Methyl-4-morpholino-3(2*H*)-pyridazinone | 138–139 | 1269 |
| 2-Methyl-5-morpholino-3(2*H*)-pyridazinone | 150–151 or 154–155, nmr | 542, 1269, 1603 |
| 6-Methyl-3-morpholino-4(1*H*)-pyridazinone | — | *H* 579 |
| 3-Methyl-6-nitroaminopyridazine | — | *H* 607 |
| 2-Methyl-4-nitro-5,6-diphenyl-3(2*H*)-pyridazinone | — | 795, 814 |
| Methyl 6-nitro-3-oxo-2-phenyl-2,3-dihydro-4-pyridazinecarboxylate | — | *H* 108, 440, 648 |
| 2-Methyl-4-nitro-6-phenyl-5-propionyl-3(2*H*)-pyridazinone | 122–124, ir, nmr | 132 |
| 3-Methyl-5-nitro-6-piperidinopyridazine 2-oxide | — | *H* 604 |
| 4-Methyl-6-nitro-3-propoxypyridazine 1-oxide | — | *H* 198, 649 |
| 6-Methyl-4-nitro-3-pyridazinamine 1-oxide | 203–204 or 213, nmr | 760, 952 |
| 2-Methyl-5-nitro-3,4(1*H*,2*H*)-pyridazinedione | — | *H* 169, 648 |
| 3-Methyl-4-nitropyridazine 1-oxide | — | *H* 649 |
| 3-Methyl-4-nitropyridazine 2-oxide | — | *H* 649 |
| 3-Methyl-5-nitropyridazine 2-oxide | — | *H* 649 |
| 4-Methyl-5-nitropyridazine 2-oxide | — | *H* 649 |
| 4-Methyl-6-nitro-3(2*H*)-pyridazinone 1-oxide | 179 | 833; *H* 649 |
| 5-Methyl-4-nitro-3(2*H*)-pyridazinone 1-oxide | 191–192 | 833; *H* 649 |

*continued*

| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 6-Methyl-4-nitro-3(2*H*)-pyridazinone 1-oxide | 200 or 203–204, uv; $NH_4$: 265; $Me_2NH_2$: 197–198 | 760, 833; *H* 649 |
| 5-Methyl-6-oxo-1,6-dihydro-3-pyridazinecarbohydrazide | 235, ms, nmr | 503 |
| 6-Methyl-3-oxo-2,3-dihydro-4-pyridazinecarbohydrazide | — | *H* 97, 441 |
| 2-Methyl-3-oxo-2,3-dihydro-4-pyridazinecarbonitrile | 130–131, ir, nmr, uv | 1300; *H* 84, 448 |
| 2-Methyl-5-oxo-2,5-dihydro-3-pyridazinecarbonitrile | 131, ir | 772 |
| 5-Methyl-3-oxo-2,3-dihydro-4-pyridazinecarbonitrile | 228–230, nmr, uv | 821; *H* 125, 448 |
| 6-Methyl-3-oxo-2,3-dihydro-4-pyridazinecarbonitrile | — | *H* 97, 448 |
| 1-Methyl-6-oxo-1,6-dihydro-3-pyridazinecarbonyl chloride | — | *H* 70, 437 |
| 1-Methyl-6-oxo-1,6-dihydro-3-pyridazinecarboxamide | — | *H* 70, 437 |
| 6-Methyl-3-oxo-2,3-dihydro-4-pyridazinecarboxamide | — | *H* 97, 441 |
| Methyl 3-oxo-2,3-dihydro-4-pyridazinecarboxylate | — | *H* 84, 440 |
| Methyl 6-oxo-1,6-dihydro-3-pyridazinecarboxylate | 189 | 1127; *H* 66, 437 |
| Methyl 6-oxo-1,6-dihydro-4-pyridazinecarboxylate | — | *H* 85, 439 (?) |
| 1-Methyl-6-oxo-1,6-dihydro-3-pyridazinecarboxylic acid | — | *H* 70, 437 |
| 2-Methyl-3-oxo-2,3-dihydro-4-pyridazinecarboxylic acid | — | *H* 84, 440 |
| 3-Methyl-6-oxo-1,6-dihydro-4-pyridazinecarboxylic acid | — | *H* 88, 439 |
| 5-Methyl-3-oxo-2,3-dihydro-4-pyridazinecarboxylic acid | — | *H* 125, 441 |
| 5-Methyl-6-oxo-1,6-dihydro-3-pyridazinecarboxylic acid | — | *H* 97, 439 |
| 6-Methyl-3-oxo-2,3-dihydro-4-pyridazinecarboxylic acid | — | *H* 97, 441 |
| 3-Methyl-6-oxo-1,6-dihydro-4,5-pyridazinedicarbonitrile | 210, ir, nmr | 1487 |
| 1-Methyl-6-oxo-1,6-dihydro-4,5-pyridazinedisulfonic acid | — | *H* 125, 825 |
| 2-Methyl-3-oxo-5,6-diphenyl-2,3-dihydro-4-pyridazinecarbonitrile | 219–220 | 1272; *H* 129, 449 |
| 2-Methyl-3-oxo-5,6-diphenyl-2,3-dihydro-4-pyridazinecarboxylic acid | — | *H* 129, 441 |
| Methyl 6-oxo-3-phenoxy-1,6-dihydro-1-pyridazinecarboxylate | — | 876 |
| 5-Methyl-6-oxo-3-phenyl-1,6-dihydro-4-pyridazinecarbaldehyde | 101–102 | 370 |
| 6-Methyl-4-oxo-1-phenyl-1,4-dihydro-3-pyridazinecarbohydrazide | — | *H* 135, 442 |
| 2-Methyl-3-oxo-5-phenyl-2,3-dihydro-4-pyridazinecarbonitrile | — | *H* 125, 448 |
| 5-Methyl-3-oxo-2-phenyl-2,3-dihydro-4-pyridazinecarbonitrile | 130 or 153–154, ir, nmr | 1240, 1731 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 5-Methyl-4-oxo-1-phenyl-1,4-dihydro-3-pyridazinecarbonitrile | 201, ir, nmr | 1808 |
| 6-Methyl-3-oxo-5-phenyl-2,3-dihydro-4-pyridazinecarbonitrile | — | *H* 128 |
| 4-Methyl-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarbonyl chloride | — | *H* 93, 438 |
| 6-Methyl-4-oxo-1-phenyl-1,4-dihydro-3-pyridazinecarboxamide | — | *H* 135, 442 |
| 6-Methyl-4-oxo-1-phenyl-1,4-dihydro-3-pyridazinecarboxamide 2-oxide | — | *H* 442 |
| Methyl 4-oxo-1-phenyl-1,4-dihydro-3-pyridazinecarboxylate | 122–124, ir, nmr | 974 |
| 4-Methyl-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylic acid | — | *H* 93, 438 |
| 5-Methyl-3-oxo-2-phenyl-2,3-dihydro-4-pyridazinecarboxylic acid | 111–114, ms, nmr | 715 |
| 5-Methyl-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylic acid | — | *H* 106, 438 |
| 6-Methyl-3-oxo-2-phenyl-2,3-dihydro-4-pyridazinecarboxylic acid | 102–103, ms, nmr | 715 |
| 6-Methyl-4-oxo-1-phenyl-1,4-dihydro-3-pyridazinecarboxylic acid | — | *H* 134, 442 |
| 6-Methyl-4-oxo-1-phenyl-1,4-dihydro-3-pyridazinecarboxylic acid 2-oxide | — | *H* 196 |
| 4-Methyl-6-oxo-1-phenyl-1,6-dihydro-3,5-pyridazinedicarbonitrile | 142 or 148, ir, nmr | 395, 1217 |
| 4-Methyl-6-oxo-1-phenyl-5-phenylazo-1,6-dihydro-3-pyridazinecarboxylic acid | — | *H* 130, 439 |
| Methyl 6-oxo-1-phenyl-4-phenylthio-1,6-dihydro-3-pyridazinecarboxylate | 170–172, ir, nmr | 1117 |
| Methyl 6-oxo-1-phenyl-4-propylthio-1,6-dihydro-3-pyridazinecarboxylate | 146–148, ir, nmr | 1117 |
| Methyl 6-oxo-1-vinyl-1,6-dihydro-3-pyridazinecarboxylate | — | 693 |
| Methyl 6-pentylamino-3-pyridazinecarboxylate | 124–125 | 1773 |
| 1-Methyl-5-pentyloxy-2-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 160 |
| Methyl 6-pentyl-3-pyridazinecarboxylate | 83–84, ir, nmr | 864 |
| 6-Methyl-2-pentyl-3(2*H*)-pyridazinone | — | *H* 75 |
| Methyl 6-pent-1′-ynyl-3-pyridazinecarboxylate | 104–105, ir, nmr | 864 |
| Methyl 6-phenethylamino-3-pyridazinecarboxylate | 168–169 | 1773 |
| 6-Methyl-3-phenethylamino-4(1*H*)-pyridazinone | — | *H* 576 |
| 3-Methyl-6-phenethylpyridazine | 41, ms, nmr; pic: 119–120 | 822, 834 |
| Methyl 3-phenethyl-4-pyridazinecarboxylate | crude: ms, nmr | 1791 |
| Methyl 6-phenethyl-4-pyridazinecarboxylate | 102–112, ir, ms, nmr | 1791 |
| 3-Methyl-6-phenethylpyridazine 1-oxide | 110–111, nmr | 537 |
| 6-Methyl-4-phenethyl-3(2*H*)-pyridazinone | 118–120, ms, nmr | 1959 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3-Methyl-6-phenoxy-5-phenylpyridazine | — | *H* 182 |
| 3-Methyl-6-phenylazopyridazine | 119–120, ir, nmr | 854 |
| 3-Methyl-6-phenylethynylpyridazine | 109–110, ir, nmr | 191, 834 |
| 3-Methyl-6-phenylethynylpyridazine 2-oxide | 207–208, ir, nmr | 834 |
| 6-Methyl-1-phenyl-5-phenylazo-4(1*H*)-pyridazinone | 225–226, nmr | 1139 |
| 4-Methyl-6-phenyl-3-phenylhydrazinopyridazine | 146–148 | 1279 |
| 1-Methyl-2-phenyl-4-phenylthio-3,6(1*H*,2*H*)-pyridazinedione | — | 1418, 1436 |
| 1-Methyl-2-phenyl-5-phenylthio-3,6(1*H*,2*H*)-pyridazinedione | — | 1418, 1436 |
| 4-Methyl-6-phenyl-2-piperidinomethyl-3(2*H*)-pyridazinethione | 88–89, ir | 371 |
| 6-Methyl-2-phenyl-4-piperidinomethyl-3(2*H*)-pyridazinone | — | *H* 105 |
| 1-Methyl-2-phenyl-4-piperidino-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 595 |
| 1-Methyl-2-phenyl-5-piperidino-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 159, 595 |
| 6-Methyl-2-phenyl-4-piperidino-3(2*H*)-pyridazinone | — | *H* 106, 590 |
| 4-Methyl-6-phenyl-3-pivalamidopyridazine | 203–204, nmr | 1632 |
| 1-Methyl-2-phenyl-5-propoxy-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 160 |
| 4-Methyl-2-phenyl-6-propyl-3(2*H*)-pyridazinone | — | *H* 107 |
| 6-Methyl-2-phenyl-4-propyl-3(2*H*)-pyridazinone | — | *H* 106 |
| 1-Methyl-2-phenyl-5-prop-2′-ynyloxy-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 160 |
| 3-Methyl-6-phenyl-4-pyridazinamine | — | 588; *H* 558 |
| 4-Methyl-6-phenyl-3-pyridazinamine | 130 or 131–132 [or 180–181 (?)], nmr, $pK_a$ | 1118, 1279, 1381, 1720 |
| 5-Methyl-6-phenyl-3-pyridazinamine | 152, nmr | 1381 |
| 6-Methyl-4-phenyl-3-pyridazinamine | — | *H* 555 |
| 6-Methyl-5-phenyl-3-pyridazinamine | 169, nmr | 1381 |
| 3-Methyl-5-phenylpyridazine | 102–104, nmr, uv | 1982 |
| 3-Methyl-6-phenylpyridazine | 98 to 105, nmr | 282, 823, 1237, 1611, 1952 |
| 4-Methyl-3-phenylpyridazine | liq, nmr | 823 |
| 4-Methyl-5-phenylpyridazine | 82–83 | 297 |
| 4-Methyl-6-phenylpyridazine | 85–87 or 89–91, nmr | 1104, 1237, 1611, 1952 |
| 4-Methyl-5-phenyl-3-pyridazinecarbaldehyde | — | *H* 395 |
| 6-Methyl-3-phenyl-4-pyridazinecarbaldehyde | 201–202, ir, uv | 370 |
| 4-Methyl-6-phenyl-3-pyridazinecarbonitrile | 177, ir, nmr | 1104 |
| 6-Methyl-3-phenyl-4-pyridazinecarbonitrile | 174–175, ir | 772 |
| 4-Methyl-6-phenyl-3-pyridazinecarboxamide | crude: ir | 1104 |
| Methyl 5-phenyl-4-pyridazinecarboxylate | — | *H* 435 |
| Methyl 6-phenyl-3-pyridazinecarboxylate | 69, ir, nmr | 1907 |
| 3-Methyl-6-phenyl-4-pyridazinecarboxylic acid | — | *H* 436 |
| 4-Methyl-5-phenyl-3-pyridazinecarboxylic acid | — | *H* 435 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 4-Methyl-6-phenyl-3-pyridazinecarboxylic acid | 125, ir, nmr | 1104 |
| 6-Methyl-5-phenyl-3,4-pyridazinedicarboxylic acid | — | *H* 445 |
| 1-Methyl-2-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 159 |
| 2-Methyl-6-phenyl-3,4(1*H*,2*H*)-pyridazinedione | 216 | 842 |
| 4-Methyl-1-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 156 |
| 4-Methyl-2-phenyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 156 |
| 6-Methyl-1-phenyl-3,4-(1*H*,2*H*)-pyridazinedione | — | *H* 170 |
| 6-Methyl-2-phenyl-3,4(1*H*,2*H*)-pyridazinedione | nmr | 719; *H* 169 |
| 3-Methyl-6-phenylpyridazine 1,2-dioxide | 181–182, ir, ms, nmr | 190, 321, 411 |
| 4-Methyl-6-phenylpyridazine 2-oxide | 100, nmr | 1104 |
| 2-Methyl-6-phenyl-3(2*H*)-pyridazinethione | 150–152 or 151–153, nmr | 202, 413, 538; *H* 797 |
| 3-Methyl-6-phenyl-3(2*H*)-pyridazinethione | 202, ir, uv | 375 |
| 6-Methyl-2-phenyl-3(2*H*)-pyridazinethione | 176, nmr | 413; *H* 798 |
| 2-Methyl-6-phenyl-3(2*H*)-pyridazinone | 111–113 | 566, 919; *H* 70 |
| 3-Methyl-5-phenyl-4(1*H*)-pyridazinone | 309, ir, nmr | 1713 |
| 3-Methyl-6-phenyl-4(1*H*)-pyridazinone | — | 588; *H* 132 |
| 4-Methyl-2-phenyl-3(2*H*)-pyridazinone | — | *H* 84 |
| 4-Methyl-6-phenyl-3(2*H*)-pyridazinone | 185 or 190, nmr | 104, 152, 1104, 1569; *H* 97 |
| 5-Methyl-2-phenyl-3(2*H*)-pyridazinone | 84, nmr | 719, 1350, 1351; *H* 85 |
| 5-Methyl-3-phenyl-4(1*H*)-pyridazinone | 241–244, ir, nmr | 109 |
| 5-Methyl-6-phenyl-3(2*H*)-pyridazinone | 210 to 219, ir, nmr | 961, 1113, 1334, 1557, 1588 |
| 6-Methyl-1-phenyl-4(1*H*)-pyridazinone | — | *H* 134 |
| 6-Methyl-2-phenyl-3(2*H*)-pyridazinone | 75–77 or 77–78, ir, ms, nmr, uv | 715, 1200, 1350, 1351; *H* 80 |
| 6-Methyl-3-phenyl-4(1*H*)-pyridazinone | 280–283, ir, nmr | 109 |
| 6-Methyl-4-phenyl-3(2*H*)-pyridazinone | 170, ir, uv | 634, 1190; *H* 97 |
| 6-Methyl-5-phenyl-3(2*H*)-pyridazinone | 220–222, nmr | 1107, 1309, 1557; *H* 88 |
| 4-Methyl-6-phenyl-2-$\beta$-thiocarbamoylethyl-3(2*H*)-pyridazinone | 132–134 | 1288 |
| 5-Methyl-6-phenyl-2-$\beta$-thiocarbamoylethyl-3(2*H*)-pyridazinone | 105–106 | 1288 |
| 1-Methyl-5-phenylthio-3,6(1*H*,2*H*)-pyridazinedione | — | 1007 |
| 6-Methyl-3-phenylthio-4(1*H*)-pyridazinone | — | *H* 813 |
| 4-Methyl-1-piperidinomethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 153 |
| 6-Methyl-2-piperidinomethyl-3(2*H*)-pyridazinone | — | *H* 72 |
| 6-Methyl-4-piperidinomethyl-3(2*H*)-pyridazinone | — | *H* 610 |
| 6-Methyl-4-piperidinomethyl-3(2*H*)-pyridazinone 1-oxide | — | *H* 196, 610 |
| 3-Methyl-6-piperidinopyridazine | 61–62, nmr | 823; *H* 578 |
| 4-Methyl-6-piperidinopyridazine | 72–73 | 544 |
| 6-Methyl-3-piperidino-4(1*H*)-pyridazinone | — | *H* 578 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3-(2-Methylprop-1-enyl)pyridazine | liq, nmr; pic: 150–151 | 827 |
| 4-(2-Methylprop-1-enyl)pyridazine | 38–40; pic: 153–154 | 827 |
| 6-Methyl-2-propionylmethyl-3(2*H*)-pyridazinone | — | *H* 75, 401 |
| 2-Methyl-5-propoxy-3,4(1*H*,2*H*)-pyridazinedione | 129–130 | 533 |
| 2-Methyl-6-propoxy-3(2*H*)-pyridazinone | — | *H* 70 |
| Methyl 6-propylamino-3-pyridazinecarboxylate | 138–140 | 1592, 1773 |
| 5-(α-Methyl-β-propylhydrazino)-2-phenyl-3(2*H*)-pyridazinone | 98, ir, nmr | 1540 |
| 5-Methyl-6-propyl-3(2*H*)-pyridazinone | — | *H* 88 |
| 2-Methyl-5-propylthio-3,4(1*H*,2*H*)-pyridazinedione | 118–120, ir, uv | 1896 |
| 3-Methyl-4-pyridazinamine | — | *H* 557 |
| 4-Methyl-3-pyridazinamine | — | *H* 550 |
| 5-Methyl-3-pyridazinamine | — | *H* 550 |
| 5-Methyl-4-pyridazinamine | — | *H* 557 |
| 6-Methyl-3-pyridazinamine | 223 or 225–226, ir, nmr, $pK_a$ | 94, 560, 1127, 1160; *H* 550 |
| 6-Methyl-4-pyridazinamine | — | *H* 557 |
| 3-Methyl-4-pyridazinamine 1-oxide | 251–253, ir, nmr | 904 |
| 6-Methyl-4-pyridazinamine 1-oxide | — | *H* 603 |
| 3-Methylpyridazine | 98/7, aromaticity, ir, nmr | 211, 230, 299, 426, 894, 1163, 1538, 1686 |
| 4-Methylpyridazine | aromaticity, nmr; oxalate: 114–116 | 211, 762, 769, 1686 |
| 3-Methyl-4-pyridazinecarbaldehyde | liq, nmr; 0.5 $H_2O$: 70–72, ir, nmr | 971 |
| 4-Methyl-3-pyridazinecarbaldehyde | liq, ir, nmr | 971 |
| 5-Methyl-4-pyridazinecarbaldehyde | liq, ir, nmr | 971 |
| 6-Methyl-3-pyridazinecarbaldehyde | $H_2NN{=}$: 180–182, ir, nmr | 35 |
| 4-Methyl-3-pyridazinecarbonitrile | 43–44, ir, nmr | 220 |
| 6-Methyl-3-pyridazinecarbonitrile | 86–87 or 89–90, ir, nmr | 220, 798, 1638; *H* 446 |
| 6-Methyl-3-pyridazinecarbonitrile 2-oxide | — | *H* 446 |
| 6-Methyl-3-pyridazinecarboxamide | — | 1500 |
| 6-Methyl-3-pyridazinecarboxamide 2-oxide | — | *H* 435 |
| Methyl 3-pyridazinecarboxylate | 138–139 or 140, ir, ms, nmr | 76, 101, 1347; *H* 434 |
| Methyl 4-pyridazinecarboxylate | 63–64, ir, ms, nmr | 101, 959, 1229, 1347, 1668; *H* 435 |
| Methyl 3-pyridazinecarboxylate 1-oxide | — | *H* 434 |
| 6-Methyl-3-pyridazinecarboxylic acid | — | 1500, 1684 |
| 6-Methyl-3,4-pyridazinediamine | — | *H* 597 |
| 6-Methyl-3,4-pyridazinedicarbaldehyde | anal, nmr; bis-$Et_2$acetal: anal, nmr | 907, 1111 |
| 6-Methyl-3,4-pyridazinedicarbonitrile | — | *H* 447 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 6-Methyl-3,4-pyridazinedicarboxamide | — | *H* 445 |
| 6-Methyl-3,4-pyridazinedicarboxylic acid | — | *H* 445 |
| 1-Methyl-3,6(1*H*,2*H*)-pyridazinedione | 210 to 216, ir, nmr, p$K_a$, xl st | 176, 292, 332, 417, 789, 943, 1128; *H* 152 |
| 2-Methyl-3,4(1*H*,2*H*)-pyridazinedione | 173–174 | 224 |
| 4-Methyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 150 |
| 6-Methyl-3,4(1*H*,2*H*)-pyridazinedione | — | *H* 169 |
| 4-Methyl-3,6(1*H*,2*H*)-pyridazinedithione | — | *H* 800 |
| 3-Methylpyridazine 1-oxide | complex | 260 |
| 3-Methylpyridazine 2-oxide | 82–83, complex | 97, 260 |
| 4-Methylpyridazine 1-oxide | complex | 260 |
| 1-Methyl-4(1*H*)-pyridazinethione | — | *H* 796, 800 |
| 2-Methyl-3(2*H*)-pyridazinethione | — | *H* 796, 797 |
| 6-Methyl-3(2*H*)-pyridazinethione | 197 to 204 | 819, 1116, 1127; *H* 797 |
| 1-Methyl-4(1*H*)-pyridazinimine | HI: 123–124, nmr, p$K_a$, uv | 675 |
| 2-Methyl-3(2*H*)-pyridazinimine | HI: 223–224, nmr, p$K_a$, uv; pic: 231–232 | 675 |
| 1-Methylpyridazinium-3-olate | 137–138, ir, nmr | 668 |
| 1-Methyl-4(1*H*)-pyridazinone | nmr | 675, 1128; *H* 133 |
| 2-Methyl-3(2*H*)-pyridazinone | nmr, p$K_a$, uv | 95, 675, 1128, 1548; *H* 65 |
| 3-Methyl-4(1*H*)-pyridazinone | — | *H* 132 |
| 4-Methyl-3(2*H*)-pyridazinone | 159 or 167, ms, nmr, st | 503, 719, 766, 1309, 1890; *H* 84 |
| 5-Methyl-3(2*H*)-pyridazinone | 151–153 or 157–158, ms, nmr, st, uv | 30, 327, 715, 719, 821, 1890; *H* 85 |
| 6-Methyl-3(2*H*)-pyridazinone | 105 to 144, ir, ms, nmr, p$K_a$, st; HBr: – ; *O*-Ts: 105, nmr, uv | 94 (cf. 1792), 433, 437, 715, 719, 847, 1116, 1128, 1167, 1405, 1459, 1562, 1638, 1721, 1809, 1890; *H* 66 |
| 2-Methyl-3(2*H*)-pyridazinone 1-oxide | 124–125, ir, nmr | 789 |
| 4-Methyl-3(2*H*)-pyridazinone 1-oxide | — | *H* 194 |
| 5-Methyl-3(2*H*)-pyridazinone 1-oxide | 206 | 833 |
| 6-Methyl-3(2*H*)-pyridazinone 1-oxide | — | *H* 195 |
| 2-Methyl-6-styryl-3(2*H*)-pyridazinone | 157 | 1282 |
| 3-Methylsulfinyl-6-methylsulfonylpyridazine | 184–185, ir, ms | 304 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3-Methylsulfinyl-6-methylsulfonylpyridazine 2-oxide | 193–195, ir, ms | 304 |
| 3-Methylsulfinyl-6-methylthiopyridazine | 118–199, nmr | 304 |
| 3-Methylsulfinyl-6-methylthiopyridazine 1-oxide | 148–151, nmr | 304 |
| 3-Methylsulfinyl-6-methylthiopyridazine 2-oxide | 143–145, nmr | 304 |
| 6-Methylsulfinyl-3-pyridazinamine | — | *H* 822 |
| 3-Methylsulfinylpyridazine | — | *H* 822 |
| 4-Methylsulfinylpyridazine | — | *H* 822 |
| 3-Methylsulfonyl-6-methylthiopyridazine | 92–95, nmr | 304 |
| 3-Methylsulfonyl-6-piperidinopyridazine | 124–125, nmr | 304; *H* 578 |
| 6-Methylsulfonyl-3-piperidinopyridazine 1-oxide | 163–165, nmr | 304 |
| 6-Methylsulfonyl-3-pyridazinamine | 183–185; pic: 204–206 | 675 |
| 3-Methylsulfonylpyridazine | — | *H* 823 |
| 4-Methylsulfonylpyridazine | — | *H* 824 |
| 6-Methylsulfonyl-3(2*H*)-pyridazinone | — | *H* 824 |
| 6-Methyl-2-δ-thiocarbamoylbutyl-3(2*H*)-pyridazinone | 113–115 | 1288 |
| 2-(α-Methyl-β-thiocarbamoylethyl)-6-phenyl-3(2*H*)-pyridazinone | 248–250 | 1288 |
| 2-(β-Methyl-β-thiocarbamoylethyl)-6-phenyl-3(2*H*)-pyridazinone | 172–174, ir, nmr | 1288 |
| 2-β-[*N*-Methyl(thiocarbamoyl)]ethyl-6-phenyl-3(2*H*)-pyridazinone | 149–150, ir, nmr | 1288 |
| 2-(α-Methyl-β-thiocarbamoylethyl)-3(2*H*)-pyridazinone | 140–143 | 1288 |
| 2-(β-Methyl-β-thiocarbamoylethyl)-3(2*H*)-pyridazinone | 149–152 | 1288 |
| 2-β-[*N*-Methyl(thiocarbamoyl)]ethyl-3(2*H*)-pyridazinone | 142–144 | 1288 |
| 6-Methyl-2-β-thiocarbamoylethyl-3(2*H*)-pyridazinone | 156–158 | 1288 |
| 6-Methyl-2-thiocarbamoylmethyl-3(2*H*)-pyridazinone | 209–212 | 1288 |
| 6-Methyl-2-ε-thiocarbamoylpentyl-3(2*H*)-pyridazinone | 69–72 | 1288 |
| 6-Methyl-2-γ-thiocarbamoylpropyl-3(2*H*)-pyridazinone | 86–87 | 1288 |
| 3-Methylthio-5,6-diphenyl-4-pyridazinecarbonitrile | 161, ir, nmr | 1390 |
| 4-Methylthio-5,6-diphenyl-3(2*H*)-pyridazinone | — | 809 |
| 6-Methylthio-2-morpholinomethyl-3(2*H*)-pyridazinethione | — | *H* 809 |
| 3-Methylthio-4-nitropyridazine | 117–120, nmr, uv | 259 |
| 6-Methylthio-3-oxo-2,5-diphenyl-2,3-dihydro-4-pyridazinecarbonitrile | 229–231, nmr, uv | 88 |
| 3-Methylthio-6-phenoxypyridazine | — | *H* 179, 803 |
| 3-Methylthio-6-phenylpyridazine | 130–132, ir, uv | 371; *H* 819 |
| 5-Methylthio-2-phenyl-3,4(1*H*,2*H*)-pyridazinedione | 111–117, ir, uv | 1896 |
| 6-Methylthio-2-piperidinomethyl-3(2*H*)-pyridazinethione | — | *H* 809 |
| 3-Methylthio-6-piperidinopyridazine | — | *H* 579, 796, 803 |
| 5-Methylthio-3-pyridazinamine | 192–193, nmr | 930 |
| 6-Methylthio-3-pyridazinamine | 117–118 | 675; *H* 553, 796, 803 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 6-Methylthio-3-pyridazinamine 2-oxide | HCl: 150–153, nmr | 679; *H* 821 |
| 3-Methylthiopyridazine | — | *H* 796, 803, 818 |
| 4-Methylthiopyridazine | — | *H* 796, 809 |
| 3-Methylthio-4,5-pyridazinediamine | 195–196, nmr, uv; 1-MeI: uv, xl st; 2-MeI: uv | 197, 973; *H* 599, 803 |
| 4-Methylthio-3,6(1*H*,2*H*)-pyridazinedione | — | 939 |
| 6-Methylthio-3(2*H*)pyridazinethione | 150–152, $pK_a$, uv | 95; *H* 808 |
| 6-Methylthio-3(2*H*)-pyridazinone | — | *H* 796, 808 |
| 3-Methylthio-4,5,6-triphenylpyridazine | 230–231, ir, nmr | 1362 |
| 2-Methyl-6-thioxo-1,6-dihydro-3(2*H*)-pyridazinone | 102–106 | 1223 |
| 4-Methyl-6-thioxo-1,6-dihydro-3(2*H*)-pyridazinone | — | *H* 799 |
| Methyl 2,5,6-trimethyl-3-oxo-2,3-dihydro-4-pyridazinecarboxylate | — | *H* 129, 441 |
| 4-Methyl-3,5,6-triphenylpyridazine | 177–178, nmr | 758 |
| Methyl 4,5,6-trisdimethylamino-3-pyridazinecarboxylate | 57, ir, nmr | 1123 |
| 6-Methyl-2-valerylmethyl-3(2*H*)-pyridazinone | — | *H* 75, 401 |
| 6-Methyl-2-vinyl-3(2*H*)-pyridazinone | 57, ir, nmr | 433, 693 |
| 3-Morpholino-4,6-dinitropyridazine 1-oxide | 230–231 | 760 |
| 3-Morpholino-4,6-diphenylpyridazine | 136, ir, nmr | 1081 |
| 4-Morpholino-2,6-diphenyl-3(2*H*)-pyridazinone | — | *H* 107, 590 |
| 4-Morpholino-5,6,-diphenyl-3(2*H*)-pyridazinone | — | *H* 809 |
| 4-Morpholino-3,6-dipropoxypyridazine | HCl: 139 | 637 |
| 2-$\beta$-Morpholinoethyl-4,6-diphenyl-3(2*H*)-pyridazinone | 144, ir, nmr | 888 |
| 2-$\beta$-Morpholinoethyl-6-phenyl-4-propyl-3(2*H*)-pyridazinone | — | *H* 104 |
| 2-$\beta$-Morpholinoethyl-6-phenyl-3(2*H*)-pyridazinone | — | *H* 73 |
| 2-Morpholinomethyl-6-phenyl-3(2*H*)-pyridazinethione | 114–115, ir | 371, 733 |
| 4-Morpholinomethyl-2-phenyl-3(2*H*)-pyridazinone | — | *H* 84 |
| 5-Morpholinomethyl-6-phenyl-3(2*H*)-pyridazinone | 177–178, ir, nmr | 1339 |
| 3-Morpholinomethylpyridazine | 52–54; pic: 167 | 769; *H* 608 |
| 4-Morpholinomethylpyridazine | liq; oxalate: 147; pic: 168–169 | 769 |
| 1-Morpholinomethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 153 |
| 1-Morpholinomethyl-3,6(1*H*,2*H*)-pyridazinedithione | — | *H* 800 |
| 2-Morpholinomethyl-3(2*H*)-pyridazinone | — | *H* 65 |
| 4-Morpholinomethyl-3(2*H*)-pyridazinone | — | *H* 610, 611 |
| 6-Morpholinomethyl-3(2*H*)-pyridazinone | 147–148 | 769; *H* 609, 611 |
| 4-Morpholinomethyl-3(2*H*)-pyridazinone 1-oxide | — | *H* 611 |
| 6-Morpholinomethyl-3(2*H*)-pyridazinone 1-oxide | HCl: 211 | 769; *H* 195, 608, 611 |
| 2-Morpholinomethyl-6-styryl-3(2*H*)-pyridazinone | — | 990 |
| 4-Morpholino-5-nitro-2-phenyl-3(2*H*)-pyridazinone | — | 601 |
| 3-Morpholino-6-phenyl-4-$\beta$-phenylhydrazinopyridazine | 205–206, ir, uv | 651 |
| 4-Morpholino-1-phenyl-2,6(1*H*,2*H*)-pyridazinedione | — | *H* 156, 593 |
| 4-Morpholino-6-phenyl-3(2*H*)-pyridazinone | 187–188 | 1041 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 5-Morpholino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 85, 592 |
| 6-Morpholino-2-phenyl-3(2*H*)-pyridazinone | — | *H* 81, 594 |
| 5-Morpholino-2-prop-1′-enyl-3(2*H*)-pyridazinone | 149–151 | 1269 |
| 5-Morpholino-6-propoxy-3(2*H*)-pyridazinone | 164 | 641 |
| 4-Morpholinopyridazine | 86–89, ir, nmr; pic: 194–195 | 544, 637; *H* 586 |
| 6-Morpholino-3-pyridazinecarboxyhydrazide | crude: 186–187 | 1338 |
| 3-Morpholino-4-pyridazinecarbonitrile | 114–115, uv | 820 |
| 3-Morpholino-4-pyridazinecarboxamide | 156–158, nmr | 820 |
| 6-Morpholino-4-pyridazinecarboxamide | 216–217, nmr | 820 |
| 4-Morpholino-3,6(1*H*,2*H*)-pyridazinedione | 298–299 | 641 |
| 6-Morpholino-3,4(1*H*,2*H*)-pyridazinedione | 232–235, ir, nmr | 524 |
| 5-Morpholino-3(2*H*)-pyridazinone | 267–270 | 542, 1269; *H* 586 |
| 5-Morpholino-4-thioxo-1,4-dihydro-3(2*H*)-pyridazinone | — | *H* 801 |
| 5-Nitroamino-4-pyridazinamine | — | *H* 562, 607 |
| 6-Nitroamino-4-pyridazinamine | — | *H* 562, 607 |
| 3-Nitroaminopyridazine | — | *H* 607 |
| 4-Nitroaminopyridazine | — | *H* 607 |
| 4-Nitro-3,6-bispiperidinocarbonylpyridazine | liq, ms, nmr | 215 |
| 1-β-Nitrobutyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 155 |
| 4-Nitro-5,6-diphenyl-3(2*H*)-pyridazinone | — | 795 |
| 4-Nitro-3,6-dipropoxypyridazine 1-oxide | — | *H* 198, 649 |
| 5-Nitro-6-pentyloxy-3-pyridazinamine 2-oxide | — | *H* 198, 604 |
| 5-Nitro-3-phenyl-4-pyridazinamine | 233–234, nmr | 1080 |
| 4-Nitro-6-phenylpyridazine | 166–167, nmr | 215 |
| 5-Nitro-2-phenyl-3,4(1*H*,2*H*)-pyridazinedione | — | 601; *H* 169, 648 |
| 5-Nitro-6-propoxy-3-pyridazinamine 2-oxide | — | *H* 198, 604 |
| 5-Nitro-4-pyridazinamine | 227, nmr | 1080 |
| 5-Nitro-3-pyridazinamine 1-oxide | 246–247 | 760 |
| 5-Nitro-4-pyridazinamine 2-oxide | 287–288, ir, nmr | 1072 |
| 4-Nitropyridazine | crude | 1080 |
| 4-Nitro-3,5-pyridazinediamine | — | *H* 598 |
| 5-Nitro-3,4(1*H*,2*H*)-pyridazinedione | — | *H* 169, 648 |
| 3-Nitropyridazine 1-oxide | — | *H* 649 |
| 4-Nitropyridazine 1-oxide | nmr | 450, 952; *H* 649 |
| 4-Nitropyridazine 2-oxide | — | *H* 649 |
| 4-Nitro-3(2*H*)-pyridazinone 1-oxide | 124–126 | 833; *H* 649 |
| 4-Nitro-3-styrylpyridazine 1-oxide | 209–210, ir, nmr | 904 |
| 3-Oxo-2,3-dihydro-4-pyridazinecarbohydrazide | — | *H* 84 |
| 6-Oxo-1,6-dihydro-3-pyridazinecarbohydrazide | 234–235, ms | 1459; *H* 67, 437 |
| 3-Oxo-2,3-dihydro-4-pyridazinecarbonitrile | 184–186 | 1300; *H* 84, 448 |
| 3-Oxo-2,3-dihydro-4-pyridazinecarbothioamide | — | *H* 84, 440, 827 |
| 3-Oxo-2,3-dihydro-4-pyridazinecarboxamide | — | *H* 84, 440 |
| 6-Oxo-1,6-dihydro-3-pyridazinecarboxamide | — | *H* 67, 437 |
| 3-Oxo-2,3-dihydro-4-pyridazinecarboxylic acid | — | *H* 84, 440 |
| 6-Oxo-1,6-dihydro-3-pyridazinecarboxylic acid | 193–195 or 255–256, ms, $pK_a$, uv | 94, 157, 675, 1459; *H* 66, 437 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 6-Oxo-1,6-dihydro-4-pyridazinecarboxylic acid | — | *H* 85, 439(?) |
| 6-Oxo-1,6-dihydro-3-pyridazinecarboxylic acid 2-oxide | — | *H* 195, 437 |
| 4-Oxo-1,4-dihydro-3,6-pyridazinedicarbaldehyde | — | *H* 133 |
| 4-Oxo-1,4-dihydro-3,6-pyridazinedicarboxylic acid | 207 | 778; *H* 133, 445 |
| 6-Oxo-1,6-dihydro-3,4-pyridazinedicarboxylic acid | — | *H* 88, 445 |
| 3-Oxo-5,6-diphenyl-2,3-dihydro-4-pyridazinecarbohydrazide | — | 1966 |
| 3-Oxo-5,6-diphenyl-2,3-dihydro-4-pyridazinecarbonitrile | 260–261, ir, nmr | 926, 1390, 1398, 1411, 1726; *H* 128, 449 |
| 4-Oxo-1,5-diphenyl-1,4-dihydro-3-pyridazinecarbonitrile | 218 | 2015 |
| 3-Oxo-5,6-diphenyl-2,3-dihydro-4-pyridazinecarboxylic acid | 234–235, ir | 389; *H* 128, 441 |
| 4-Oxo-1,6-diphenyl-1,4-dihydro-3-pyridazinecarboxylic acid | 350, ir, nmr | 1036, 2090; *H* 134, 443 |
| 3-Oxo-2,5-diphenyl-6-phenylazo-2,3-dihydro-4-pyridazinecarbonitrile | 240, ir, nmr | 37, 221 |
| 3-Oxo-2,5-diphenyl-6-thioxo-1,2,3,6-tetrahydro-4-pyridazinecarbonitrile | 195–197, ir, uv | 88 |
| 3-Oxo-6-phenyl-2,3-dihydro-4-pyridazinecarbaldehyde | dimethyl dithioacetal: 220–222, nmr | 2114 |
| 6-Oxo-3-phenyl-1,6-dihydro-4-pyridazinecarbaldehyde | 230–231, ir, nmr | 2018 |
| 4-Oxo-1-phenyl-1,4-dihydro-3-pyridazinecarbohydrazide | 179–181, ir, nmr | 974 |
| 6-Oxo-3-phenyl-1,6-dihydro-4-pyridazinecarbothialdehyde | $Me_2$dithioacetal: 153–154, ir, nmr | 131 |
| 3-Oxo-6-phenyl-2,3-dihydro-4-pyridazinecarboxamide | 300 or 312–313, ir, nmr | 1041, 1319 |
| 6-Oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxamide | — | *H* 80, 438 |
| 3-Oxo-2-phenyl-2,3-dihydro-4-pyridazinecarboxylic acid | 95–96, ms, nmr | 715 |
| 6-Oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxylic acid | — | *H* 80, 438 |
| 6-Oxo-1-phenyl-1,6-dihydro-4-pyridazinecarboxylic acid | — | *H* 85, 439 |
| 6-Oxo-3-phenyl-1,6-dihydro-4-pyridazinecarboxylic acid | 243–245, ir, nmr | 2018 |
| 4-Oxo-1-phenyl-5-phenylazo-1,4-dihydro-3-pyridazinecarbaldehyde | — | *H* 395 |
| 4-Oxo-1-phenyl-5-phenylazo-1,4-dihydro-3-pyridazinecarboxylic acid | — | *H* 136, 442 |
| 6-Oxo-1-phenyl-4-styryl-1,6-dihydro-3,5-pyridazinedicarbonitrile | 225 or 246, ir, nmr | 395, 1217 |
| 3-Oxo-2,5,6-triphenyl-2,3-dihydro-4-pyridazinecarbonitrile | — | 926 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3-Oxo-2,5,6-triphenyl-2,3-dihydro-4-pyridazinecarboxylic acid | — | *H* 130, 441 |
| 6-Pentylamino-3-pyridazinecarboxamide | 164–166 | 1773 |
| 3-Pentyloxy-3-phenoxypyridazine | — | 688 |
| 3-*s*-Pentyloxy-6-phenoxypyridazine | — | 688 |
| 6-Pentyloxy-3-pyridazinamine | — | *H* 178 |
| 6-Pentyloxy-3(2*H*)-pyridazinethione | 110 | 365 |
| 6-Pentyl-3(2*H*)-pyridazinone | — | *H* 67 |
| 3-Pent-3′-ynyloxy-4-pyridazinecarbonitrile | — | 2076 |
| 3-Phenethylamino-6-phenylpyridazine | 132 | 1248 |
| 6-Phenethylamino-3-pyridazinamine 2-oxide | 170–173, nmr | 1161 |
| 6-Phenethylamino-3-pyridazinecarboxamide | 153–155 | 1773 |
| 3-Phenethyl-5,6-diphenylpyridazine | 114–115, nmr | 330 |
| 3-Phenethyl-5,6-diphenyl-4-pyridazinecarboxylic acid | 215–216, nmr | 330 |
| 2-Phenethyl-4,6-diphenyl-3(2*H*)-pyridazinone | 104, ir, nmr | 888 |
| 6-Phenethyloxy-3-pyridazinamine | — | *H* 553 |
| 4-Phenethyl-6-phenyl-3-pyridazinamine | 158, nmr | 1381 |
| 2-Phenethyl-6-phenyl-3(2*H*)-pyridazinone | 214–215/1, nmr | 118 |
| 4-Phenethyl-6-phenyl-3(2*H*)-pyridazinone | 165–166, ir, nmr | 1959 |
| 3-Phenethylpyridazine | 31–33, ms, nmr; pic: 157–159 | 822, 1666, 1791 |
| 4-Phenethylpyridazine | 66–68, ms, nmr | 67, 822, 1666 |
| 4-Phenethyl-3-pyridazinecarbonitrile | 64–67, ir, ms, nmr | 1791 |
| 4-Phenethyl-3-pyridazinecarboxamide | 119–122, ir, ms, nmr | 1791 |
| 4-Phenethyl-3-pyridazinecarboxylic acid | 116–119, ir, ms, nmr | 1791 |
| 5-Phenethyl-4-pyridazinecarboxylic acid | 194–200, ir, ms, nmr | 1791 |
| 3-Phenethylpyridazine 2-oxide | 92–93, nmr | 537 |
| 4-Phenoxy-5,6-diphenyl-3(2*H*)-pyridazinone | — | 809 |
| 3-Phenoxy-6-propoxypyridazine | — | 688 |
| 6-Phenoxy-3-pyridazinamine | 155–156, nmr | 1150; *H* 552 |
| 3-Phenoxypyridazine | — | *H* 174 |
| 4-Phenoxypyridazine | — | *H* 186 |
| 3-Phenoxy-4-pyridazinecarbonitrile | 132–133, ir, ms, nmr | 517 |
| 3-Phenoxy-4-pyridazinecarboxamide | 153–154 ir, nmr | 517 |
| 3-Phenoxy-4-pyridazinecarboxylic acid | 203–204, ir, nmr | 517 |
| 6-Phenoxy-3-pyridazinecarboxylic acid | — | 1592 |
| 3-Phenoxypyridazine 1-oxide | — | *H* 194 |
| 6-Phenoxy-3(2*H*)-pyridazinone | — | *H* 68 |
| 2-Phenyl-4,5-bisphenylthio-3(2*H*)-pyridazinone | 168–170, ir, nmr | 1089; *H* 815 |
| 2-Phenyl-5,6-dipiperidino-3(2*H*)-pyridazinone | — | *H* 93, 602 |
| 3-Phenylethynyl-6-piperidinopyridazine | 163, ir, nmr | 834 |
| 3-Phenylethynyl-4-pyridazinecarbonitrile | 162–165, ir, ms, nmr | 1791 |
| 3-Phenylethynylpyridazine 1-oxide | 153–155, ir, nmr | 834 |
| 6-Phenylmethanesulfonyloxy-3(2*H*)-pyridazinone | — | *H* 69 |
| 3-Phenyl-6-phenylazopyridazine | 181–182, ir, nmr | 854 |
| 3-Phenyl-6-phenylethynylpyridazine | 170, ir, nmr | 834 |
| 3-Phenyl-6-phenylhydrazinopyridazine | 138–139 or 162–164, ir, nmr | 854, 1279 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 6-Phenyl-2-piperidinomethyl-3(2*H*)-pyridazinone | — | 733 |
| 6-Phenyl-5-piperidinomethyl-3(2*H*)-pyridazinone | 180–181 | 1339 |
| 3-Phenyl-6-piperidinopyridazine | 131–132, nmr | 823 |
| 1-Phenyl-4-piperidino-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 156, 593 |
| 2-Phenyl-5-piperidino-3(2*H*)-pyridazinone | — | *H* 592 |
| 2-Phenyl-6-piperidino-3(2*H*)-pyridazinone | — | *H* 80, 594 |
| 2-Phenyl-6-propanesulfonyloxy-3(2*H*)-pyridazinone | — | *H* 81 |
| 3-Phenyl-4-propionyl-5-trimethylsilylpyridazine | 99, ms, nmr | 12 |
| 6-Phenyl-5-propyl-3-pyridazinamine | 137, nmr | 1381 |
| 3-Phenyl-5-propylpyridazine | 69–70, ms, nmr, uv | 1797 |
| 3-Phenyl-4-pyridazinamine | 146–147, nmr | 1012 |
| 5-Phenyl-3-pyridazinamine | 203, nmr | 1381 |
| 5-Phenyl-4-pyridazinamine | — | 557 |
| 6-Phenyl-3-pyridazinamine | 151 to 157, ir, nmr | 133, 1012, 1145, 1279; *H* 551 |
| 6-Phenyl-4-pyridazinamine | 149–150, nmr | 1012 |
| 3-Phenylpyridazine | 101 to 104, 160/3, nmr, uv | 230, 282, 403, 450, 452, 1153, 1186, 1237, 1611, 1858 |
| 4-Phenylpyridazine | 84–85 or 85–87, ms, nmr, uv | 337, 1186, 2046 |
| 6-Phenyl-3-pyridazinecarbaldehyde | — | *H* 396 |
| 6-Phenyl-3-pyridazinecarbonitrile | 184–185 | 798; *H* 446 |
| 6-Phenyl-4-pyridazinecarbonitrile | 193–194, nmr | 215 |
| 5-Phenyl-4-pyridazinecarboxamide | — | *H* 435 |
| 5-Phenyl-4-pyridazinecarboxylic acid | — | *H* 435 |
| 6-Phenyl-3-pyridazinecarboxylic acid | — | *H* 435 |
| 3-Phenyl-4,5-pyridazinediamine | 212–214, nmr | 1080 |
| 6-Phenyl-3,5-pyridazinediamine | 176–178, ir, ms, nmr | 133 |
| 5-Phenyl-3,4-pyridazinedicarboxylic acid | — | *H* 445 |
| 5-Phenyl-3,4-pyridazinedicarboxylic anhydride | — | *H* 445 |
| 1-Phenyl-3,6(1*H*,2*H*)-pyridazinedione | 262–263 or 267–268, ir, nmr | 292, 313, 943; *H* 155 |
| 2-Phenyl-3,4(1*H*,2*H*)-pyridazinedione | — | *H* 169 |
| 4-Phenyl-3,6(1*H*,2*H*)-pyridazinedione | 275–276 | 461; *H* 151 |
| 6-Phenyl-3,4(1*H*,2*H*)-pyridazinedione | 266–268, ir, ms | 942 |
| 3-Phenylpyridazine 1-oxide | complex | 260 |
| 2-Phenyl-3(2*H*)-pyridazinethione | — | *H* 799 |
| 6-Phenyl-3(2*H*)-pyridazinethione | 158–159 or 160 | 413, 659, 811, 819; *H* 797 |
| 2-Phenyl-3(2*H*)-pyridazinone | 107–108 or 109–110, nmr | 290, 719, 1350, 1351; *H* 65 |
| 3-Phenyl-4(1*H*)-pyridazinone | 198–200, ir, nmr | 109 |
| 4-Phenyl-3(2*H*)-pyridazinone | 217–220, ir, nmr | 738, 1408 |
| 5-Phenyl-3(2*H*)-pyridazinone | 194–196, ir, nmr | 327, 738, 1408, 2069 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 6-Phenyl-3(2*H*)-pyridazinone | 194 to 205, ir, p$K_a$, nmr; AcOH: xl st | 94, 497, 504, 738, 1041, 1535, 1721, 1823, 2069; *H* 67 |
| 6-Phenyl-4(1*H*)-pyridazinone | 256–257, ir, nmr | 1041 |
| 4-Phenyl-6-styryl-3(2*H*)-pyridazinone | 235–236, ir, uv | 369 |
| 6-Phenylsulfinyl-3-pyridazinamine | 213, ir, nmr | 1693 |
| 6-Phenylsulfonyl-3-pyridazinamine | 243, ir, nmr | 1693 |
| 6-Phenyl-2-δ-thiocarbamoylbutyl-3(2*H*)-pyridazinone | 154–156 | 1288 |
| 6-Phenyl-2-β-thiocarbamoylethyl-3(2*H*)-pyridazinone | 173–175, ir, nmr | 1288 |
| 6-Phenyl-2-thiocarbamoylmethyl-3(2*H*)-pyridazinone | 245 | 1288 |
| 6-Phenyl-2-ε-thiocarbamoylpentyl-3(2*H*)-pyridazinone | 102–103, ir, nmr | 1288 |
| 6-Phenyl-2-γ-thiocarbamoylpropyl-3(2*H*)-pyridazinone | 129–130, ir, nmr | 1288 |
| 6-Phenylthio-3-pyridazinamine | 139–140 | 1147; *H* 553, 807 |
| 3-Phenylthiopyridazine | liq, anal, nmr | 1387 |
| 3-Phenylthio-4-pyridazinecarbonitrile | 164–168, ir, nmr | 517 |
| 4-Phenylthio-3,6(1*H*,2*H*)-pyridazinedione | — | 939 |
| 6-Phenyl-2-δ-thioureidobutyl-3(2*H*)-pyridazinone | 65–68, ir, nmr | 1289 |
| 1-Phenyl-6-thioxo-1,6-dihydro-3(2*H*)-pyridazinone | — | *H* 799 |
| 3-Phenyl-4-trimethylsilylpyridazine | 67, ir, nmr | 2093 |
| 3-Phenyl-5-trimethylsilylpyridazine | 77, ms, nmr | 12 |
| 3-Phenyl-5-trimethylsilyl-4-trimethylsilylethynylpyridazine | 78, ms, nmr | 12 |
| 6-Phenyl-2-δ-ureidobutyl-3(2*H*)-pyridazinone | 163–166, ir, nmr | 1337 |
| 6-Phenyl-2-β-ureidoethyl-3(2*H*)-pyridazinone | 208–210 | 1337 |
| 6-Phenyl-2-ε-ureidopentyl-3(2*H*)-pyridazinone | 163–165 | 1337 |
| 6-Phenyl-2-γ-ureidopropyl-3(2*H*)-pyridazinone | 208–210 | 1337 |
| 6-Phenyl-2-vinyl-3(2*H*)-pyridazinone | 75–76, nmr | 693, 1179 |
| 3-Piperidinomethylpyridazine | — | *H* 609 |
| 4-Piperidinomethylpyridazine | <130/1, anal | 769 |
| 1-Piperidinomethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 152 |
| 1-Piperidinomethyl-3,6(1*H*,2*H*)-pyridazinedithione | — | *H* 800 |
| 2-Piperidinomethyl-3(2*H*)-pyridazinone | — | *H* 65 |
| 4-Piperidinomethyl-3(2*H*)-pyridazinone | — | *H* 610 |
| 6-Piperidinomethyl-3(2*H*)-pyridazinone | — | *H* 609 |
| 3-Piperidinomethyl-4(1*H*)-pyridazinone 2-oxide | — | *H* 609, 611 |
| 5-Piperidinomethyl-4(1*H*)-pyridazinone 2-oxide | — | *H* 611 |
| 6-Piperidinomethyl-3(2*H*)-pyridazinone 1-oxide | — | *H* 195, 609 |
| 3-Piperidinopyridazine | — | *H* 578 |
| 4-Piperidinopyridazine | HCl: 181 | 641 |
| 3-Piperidino-4-pyridazinecarbonitrile | 71–72, uv | 820 |
| 3-Piperidino-4-pyridazinecarboxamide | 133–134, nmr | 820 |
| 4-Piperidino-3,6(1*H*,2*H*)-pyridazinedione | 279–280 | 641 |
| 3-Piperidinopyridazine 1-oxide | — | *H* 604 |
| 3-Piperidinopyridazine 2-oxide | — | *H* 604 |
| 4-Piperidinopyridazine 1-oxide | — | *H* 604 |
| 4-Piperidinopyridazine 2-oxide | — | *H* 604 |

*continued*

| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 6-Piperidino-3(2*H*)-pyridazinone | — | *H* 578, 796 |
| 3-Pivalamidopyridazine | 126, ir, nmr | 1421 |
| 4-Pivalamidopyridazine | 166, ir, nmr | 1609 |
| 3-Pivaloylpyridazine | ir, nmr | 1660 |
| 6-Propanesulfonyloxy-3(2*H*)-pyridazinone | — | *H* 69 |
| 6-Prop-1′-enylthio-3(2*H*)-pyridazinone | 105, nmr | 557 |
| 6-Propionyloxy-3(2*H*)-pyridazinone | — | *H* 68 |
| 3-Propionylpyridazine | 58, ir, nmr | 1347, 1660 |
| 4-Propionylpyridazine | <25, <90/0.01, ir, ms, nmr | 79, 81 |
| 5-Propionyl-4-pyridazinecarboxylic acid | 164–165, ir, nmr | 81, 1095 |
| 4-Propoxycarbonylmethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 151 |
| 6-Propoxy-3-pyridazinamine | liq, anal, ms, nmr | 1841; *H* 178, 552 |
| 6-Propoxy-3-pyridazinecarboxylic acid | — | 1592 |
| 3-Propoxypyridazine 1-oxide | — | *H* 194 |
| 6-Propoxy-3(2*H*)-pyridazinethione | — | *H* 797 |
| 6-Propoxy-3(2*H*)-pyridazinone | — | *H* 68 |
| 3-Propylaminopyridazine | — | *H* 573 |
| 4-Propylaminopyridazine | — | *H* 583 |
| 6-Propylamino-3-pyridazinecarboxamide | 188–190 | 1592, 1773 |
| Propyl 6-amino-3-pyridazinecarboxylate | — | *H* 435, 553 |
| 6-Propylamino-3-pyridazinesulfonamide | — | *H* 573 |
| Propyl 6-chloro-3-pyridazinecarboxylate | — | *H* 276, 434 |
| Propyl 6-oxo-1-vinyl-1,6-dihydro-3-pyridazinecarboxylate | — | 693 |
| 6-Propylthio-3-pyridazinamine | 77–78, nmr | 1147 |
| 3-Pyridazinamine | 168–169 or 174–175, p$K_a$, uv; 1-MeI: 182–183, nmr, p$K_a$, uv; 1-Me pic: 162–163 | 675, 1037, 1176, 1421; *H* 550 |
| 4-Pyridazinamine | 127–129, p$K_a$, uv; 2-MeI: 186–187, nmr, p$K_a$, uv | 675, 1012; *H* 557 |
| 3-Pyridazinamine 1-oxide | 136–138 | 891; *H* 603 |
| 3-Pyridazinamine 2-oxide | — | *H* 603 |
| 4-Pyridazinamine 1-oxide | — | *H* 603 |
| 4-Pyridazinamine 2-oxide | — | *H* 603, 604 |
| Pyridazine | 207/760, aromaticity, complexes, dipole, ms, nmr, p$K_a$, pol, st (gas), uv, xl st; HCl: xl st; 1,2-di($MeBF_4$): 171–172, nmr; 1-Et$BF_4$, 2-Me$BF_4$: 153–154, nmr; | 177, 180, 211, 230, 262, 294, 299, 414, 450, 460, 490, 498, 575, 626, 675, 773, 900, 925, 1091, |

*continued*

| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| | 1-$EtBF_4$: 26–27, nmr; 1-MeI: –; 1-$PhCH_2Cl$: 180–182; 1-$BrCH_2CH_2$-$CONH_2$: 148, nmr | 1093, 1098, 1133, 1163, 1183, 1191, 1202, 1231, 1249, 1433, 1502, 1532, 1546, 1548, 1571, 1638, 1658, 1666, 1860, 1891, 1926, 1967, 2013, 2049, 2077, 2078, 2082; *H* 5, 6 |
| 3-Pyridazinecarbaldehyde | 47, ir, ms, nmr; dnp: 250–355, ir, nmr; tsc: 237, ir, nmr; PhN=: 85, nmr; $H_2NN$=: 107–109, nmr; oxime: 142–143, nmr; oxime, MeI: 194–196, nmr | 77, 439, 1612; *H* 395 |
| 4-Pyridazinecarbaldehyde | 90–91, ir, nmr; dnp: 296–299, ir; tsc: 239–242, nmr; PhN=: 81–82, nmr; oxime: 173–175, ir, nmr, st | 67, 291, 971 |
| 3-Pyridazinecarbaldehyde 1-oxide | — | *H* 398 |
| 3-Pyridazinecarbaldehyde 2-oxide | 167–169, ir; PhNHN=: 277–279 | 767, 1261; *H* 398 |
| 4-Pyridazinecarbaldehyde 1-oxide | — | *H* 398 |
| 4-Pyridazinecarbaldehyde 2-oxide | — | *H* 398 |
| 3-Pyridazinecarbohydrazide | — | *H* 434 |
| 4-Pyridazinecarbohydrazide | — | *H* 435 |
| 4-Pyridazinecarbohydroxamic acid | 159–163, ir, nmr | 46 |
| 3-Pyridazinecarbonitrile | 43–44, ir, nmr | 220; *H* 446 |
| 4-Pyridazinecarbonitrile | — | *H* 447 |
| 3-Pyridazinecarbothioamide | — | *H* 434, 828 |
| 4-Pyridazinecarbothioamide | — | *H* 435, 826 |
| 3-Pyridazinecarboxamide | — | *H* 434 |
| 4-Pyridazinecarboxamide | 190, ir, ms, nmr; 1-[$I(CH_2)_4CO_2H$]: 150–153, nmr; 2-isomer: 161–165, nmr | 1229, 1779; *H* 435 |

*continued*

| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3-Pyridazinecarboxylic acid | 199–201, ir, nmr | 76, 77, 101, 925, 935, 1066; *H* 434 |
| 4-Pyridazinecarboxylic acid | 233 to 240, ir, nmr | 65, 67, 76, 101, 1120; *H* 435 |
| 3,4-Pyridazinediamine | p$K_a$, uv; HCl: 198–200; pic: 253–254 | 449, 675; *H* 597 |
| 3,5-Pyridazinediamine | p$K_a$, uv; HCl: –; pic: 303–305; 1-MeI: 208–210, nmr, p$K_a$, uv; 1-Me pic: 205 | 675; *H* 598 |
| 3,6-Pyridazinediamine | 235–237, ir, nmr, p$K_a$, uv; HCl: 240; pic: 277–279 | 675, 1355; *H* 599 |
| 4,5-Pyridazinediamine | nmr, p$K_a$, uv; pic: 240–241 | 675, 1080; *H* 598 |
| 3,4-Pyridazinedicarbaldehyde | unstable: anal, ir, nmr; bis-Et$_2$acetal: anal, nmr; complexes | 1111, 1894 |
| 3,6-Pyridazinedicarbaldehyde | dioxime: >200, ms, nmr | 1939 |
| 4,5-Pyridazinedicarbaldehyde | bis(Et$_2$acetal): anal, nmr; dioxime: complexes | 1111, 1872 |
| 3,6-Pyridazinedicarbaldehyde 1-oxide | — | *H* 398 |
| 3,6-Pyridazinedicarbohydrazide | >200, ir, nmr | 1077 |
| 3,4-Pyridazinedicarbonitrile | — | *H* 447 |
| 4,5-Pyridazinedicarbonitrile | — | *H* 447 |
| 3,6-Pyridazinedicarbonyl dichloride | 138 or 208–210, ir, ms, nmr | 435, 1939, 2091 |
| 3,4-Pyridazinedicarboxamide | — | *H* 445 |
| 4,5-Pyridazinedicarboxamide | — | *H* 445 |
| 3,4-Pyridazinedicarboxylic acid | 170, ir, ms, nmr | 1120 |
| 3,6-Pyridazinedicarboxylic acid | 230, complexes, ir, nmr, xl st | 1077, 1914, 1963 |
| 4,5-Pyridazinedicarboxylic acid | 210–213, nmr | 101, 656, 1378, 1697; *H* 445 |
| 4,5-Pyridazinedicarboxylic anhydride | 170–190, ir, nmr | 958 |
| 3,6(1*H*,2*H*)-Pyridazinedione (maleic hydrazide) | 290 to 312, ir, nmr, p$K_a$, pol, uv, xl st | 94, 95, 98, 99, 153, 163, 292 417, 802, 860, 1098, 1128, 1167, 1345, 1348, 1533, 1548, 1638, 2047; *H* 150 |

*continued*

| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| Pyridazine 1,2-dioxide | st | 2079 |
| 3,6(1*H*,2*H*)-Pyridazinedithione | 230 to 262, ir, $pK_a$, uv | 95, 365, 636, 1348; *H* 796, 800 |
| Pyridazine 1-imide | unstable | 477 |
| Pyridazine 1-oxide | 38–39, 138–140/4, complex, ir, nmr | 260, 450, 1944 |
| 3,4,5,6-Pyridazinetetracarboxylic acid | — | 680; *H* 445 |
| 3(2*H*)-Pyridazinethione | aromaticity, ir, st | 315, 1415, 1655; *H* 796, 797 |
| 4(1*H*)-Pyridazinethione | — | *H* 796, 800 |
| 3,4,5-Pyridazinetriamine | HCl: 212–214 | 449; *H* 601 |
| 3,4,6-Pyridazinetricarbaldehyde | 3,4-Bis($Et_2$acetal): nmr; tris($Et_2$acetal): nmr | 1111 |
| Pyridazinium-1-acetamidate | 125–127; pic: 128–129 | 756 |
| Pyridazinium-1-benzamidate | 156–157; pic; 135–136 | 756 |
| 3(2*H*)-Pyridazinone | 103–104 or 107–108, ir, ms, nmr, $pK_a$, st, uv | 95, 403, 715, 719, 1128, 1167, 1392, 1548, 1656, 1809, 1878; *H* 65 |
| 4(1*H*)-Pyridazinone | 250, nmr; 2-MeCl: 234, ir, nmr | 781, 1128, 1392; *H* 132 |
| 3(2*H*)-Pyridazinone 1-oxide | st | 2079; *H* 194 |
| 4(1*H*)-Pyridazinone 2-oxide | st | 2079 |
| 3-Styrylpyridazine | 83–85, ir, ms, nmr | 26, 77 |
| 4-Styrylpyridazine | 64, ir, nmr | 67, 509 |
| 6-Styryl-3(2*H*)-pyridazinethione | — | 946 |
| 6-Styryl-3(2*H*)-pyridazinone | 185 | 1282 |
| 1-$\beta$-Sulfoethyl-3,6(1*H*,2*H*)-pyridazinedione | — | *H* 829 |
| 3,4,5,6-Tetra-*t*-butylpyridazine | 79, ir, ms, nmr | 1438, 1613 |
| 3,4,5,6-Tetrachloro-1-methoxypyridazinium ( + anion) | crude $MeSO_4$: – | 893 |
| 3,4,5,6-Tetrachloropyridazine | 85–86, ir, nmr | 1938; *H* 275 |
| 3,4,5,6-Tetrachloropyridazine 1-oxide | 131–132 or 133 | 103, 893 |
| 2,4,5,6-Tetrachloro-3(2*H*)-pyridazinone | — | *H* 128, 329 |
| Tetraethyl 3,4,5,6-pyridazinetetracarboxylate | < 30, ir, ms, nmr | 416 |
| 3,4,5,6-Tetrafluoropyridazine | — | 460, 706; *H* 297 |
| 3,4,5,6-Tetrakisbromomethylpyridazine | crude: liq, nmr | 747 |
| 3,4,5,6-Tetrakisphenylthiopyridazine | — | *H* 814 |
| 3,4,5,6-Tetramethoxypyridazine | — | *H* 184 |
| 3,4,5,6-Tetramethylpyridazine | 69, ms, nmr | 211, 762, 1088 |
| Tetramethyl 3,4,5,6-pyridazinetetracarboxylate | 143–144, ir, ms, nmr | 40, 357 |
| 3,4,5,6-Tetraphenylpyridazine | 194 or 196–197, nmr, uv | 499, 632, 763 |

*continued*

| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3,4,5,6-Tetraphenylpyridazine 1-oxide | 185–186 | 763 |
| 1,3,5,6-Tetraphenyl-4(1*H*)-pyridazinone | — | *H* 136 |
| 1-β-Thiocarbamoylethyl-3,6(1*H*,2*H*)-pyridazinedione | 163–164 | 1288 |
| 2-β-Thiocarbamoylethyl-3(2*H*)-pyridazinone | 145–147 | 1288 |
| 6-Thioxo-1,6-dihydro-3(2*H*)-pyridazinone | — | *H* 796, 799 |
| 3,4,6-Tribromopyridazine | 95–96 | 447 |
| 3,4,5-Tri-*t*-butyl-6-isopropylpyridazine | 90, ir, ms, nmr, uv | 1614 |
| 4,5,6-Trichloro-2-chloromethyl-3(2*H*)-pyridazinone | 97–99 | 893; *H* 129, 329 |
| 3,4,6-Trichloro-5-β-cyanovinylpyridazine | 136–137, nmr | 963 |
| 3,4,6-Trichloro-5-cyclohexylaminopyridazine | — | *H* 289, 583 |
| 3,4,6-Trichloro-5-diethylaminopyridazine | — | *H* 289, 585 |
| 3,4,5-Trichloro-6-dimethylaminopyridazine | 63 | 1796 |
| 3,4,6-Trichloro-5-dimethylaminopyridazine | — | *H* 289, 584 |
| 3,4,6-Trichloro-5-ethoxypyridazine | — | *H* 289 |
| 3,4,5-Trichloro-6-fluoropyridazine | 51, 226/760, nmr | 459 |
| 4,5,6-Trichloro-2-hydroxymethyl-3(2*H*)-pyridazinone | — | *H* 129, 329, 403 |
| 3,4,6-Trichloro-5-isopropylaminopyridazine | — | *H* 289, 583 |
| 3,4,6-Trichloro-5-methoxypyridazine | — | *H* 289 |
| 4,5,6-Trichloro-2-methoxy-3(2*H*)-pyridazinone | 104–106 | 893 |
| 3,4,6-Trichloro-5-methylaminopyridazine | — | *H* 289, 582 |
| 3,4,5-Trichloro-6-methylpyridazine | 73, ms; HCl: 170–172 | 1232 |
| 4,5,6-Trichloro-2-methyl-3(2*H*)-pyridazinone | 99 | 893; *H* 128, 329 |
| 4,5,6-Trichloro-2-methyl-3(2*H*)-pyridazinone 1-oxide | 184 | 893 |
| 3,4,6-Trichloro-5-morpholinopyridazine | 119 | 637 |
| 4,5,6-Trichloro-2-phenyl-3(2*H*)-pyridazinone | — | *H* 130, 329 |
| 3,4,6-Trichloro-5-propoxypyridazine | — | *H* 289 |
| 3,5,6-Trichloro-4-pyridazinamine | — | *H* 289, 562 |
| 3,4,5-Trichloropyridazine | 54, ms, nmr | 573, 641, 727, 1003, 1005; *H* 275 |
| 3,4,6-Trichloropyridazine | — | 614; *H* 275 |
| 4,5,6-Trichloro-3-pyridazinecarboxylic acid | 84–85 | 1232 |
| 3,4,5-Trichloropyridazine 1-oxide | — | *H* 300 |
| 3,4,6-Trichloropyridazine 1- + 2-oxide | — | *H* 893 |
| 2,4,5-Trichloro-3(2*H*)-pyridazinone | — | 916; *H* 112 |
| 2,4,6-Trichloro-3(2*H*)-pyridazinone | — | *H* 98, 325 |
| 3,5,6-Trichloro-4(1*H*)-pyridazinone | — | *H* 132, 289 |
| 4,5,6-Trichloro-3(2*H*)-pyridazinone | 226–227, ir, nmr | 1938; *H* 127, 329 |
| 4,5,6-Trichloro-2-thiocyanatomethyl-3(2*H*)-pyridazinone | — | *H* 129, 329, 826 |
| 2,4,5-Tricyclohexyl-6-methyl-3(2*H*)-pyridazinone | 135–136, ms, nmr | 1676 |
| Triethyl 5-methyl-3,4,6-pyridazinetricarboxylate | 90/0.1, ir, ms, nmr | 416 |
| Triethyl 6-methyl-3,4,5-pyridazinetricarboxylate | <30, ir, ms, nmr | 416 |
| Triethyl 5-phenethyl-3,4,6-pyridazinetricarboxylate | <30, ir, ms, nmr | 416 |
| Triethyl 3,4,5-pyridazinetricarboxylate | <30, 150/0.1, ms, nmr | 416 |
| 3,4,5-Trifluoro-6-methoxypyridazine | — | *H* 184, 298 |
| 3,4,6-Trifluoro-5-methoxypyridazine | — | *H* 185, 297 |
| 4,5,6-Trifluoro-2-methyl-3(2*H*)-pyridazinone | — | *H* 129, 332 |

*continued*

| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3,5,6-Trifluoro-4-pyridazinamine | — | *H* 297, 559 |
| 4,5,6-Trifluoro-3(2*H*)-pyridazinone | — | *H* 127, 298, 332 |
| 2,4,5-Triisopropyl-6-methyl-3(2*H*)-pyridazinone | 97–98, ms, nmr | 1676 |
| 4,5,6-Trimethoxy-2-phenyl-3(2*H*)-pyridazinone | — | *H* 131 |
| 3,4,5-Trimethoxypyridazine | 76 to 80, nmr; 1-MeI: 98–100 then 148–150 | 689, 882 |
| 3,4,6-Trimethoxypyridazine | 121, nmr | 640, 881; *H* 186 |
| 3,4,6-Trimethoxypyridazine 1-oxide | 117 | 757; *H* 197 |
| 2,5,6-Trimethoxy-3(2*H*)-pyridazinone | — | *H* 90 |
| 2,5,6-Trimethyl-3-oxo-2,3-dihydro-4-pyridazinecarbonitrile | — | *H* 129, 449 |
| 2,5,6-Trimethyl-3-oxo-2,3-dihydro-4-pyridazinecarbothioamide | — | *H* 129, 441, 827 |
| 1,*N*,*N*-Trimethyl-6-oxo-1,6-dihydro-3-pyridazinecarboxamide | — | *H* 70 |
| 2,5,6-Trimethyl-3-oxo-2,3-dihydro-4-pyridazinecarboxamide | — | *H* 129, 441 |
| 2,5,6-Trimethyl-3-oxo-2,3-dihydro-4-pyridazinecarboxylic acid | — | *H* 129, 441 |
| 4,*N*,*N*-Trimethyl-6-oxo-1-phenyl-1,6-dihydro-3-pyridazinecarboxamide | 53–54 | 1351; *H* 93, 438 |
| 2,5,6-Trimethyl-4-phenyl-3(2*H*)-pyridazinone | — | *H* 129 |
| 4,5,6-Trimethyl-3-pyridazinamine | 186–187, ir | 560, 1160; *H* 556 |
| 3,4,5-Trimethylpyridazine | 63–64, ms, nmr | 211, 762 |
| 3,4,6-Trimethylpyridazine | 90–91, ms, nmr | 211, 762, 823 |
| Trimethyl 3,4,6-pyridazinetricarboxylate | 95–96, nmr | 40 |
| 2,5,6-Trimethyl-3(2*H*)-pyridazinone | — | *H* 90 |
| 3,5,6-Trimethyl-4(1*H*)-pyridazinone | — | *H* 132 |
| 4,5,6-Trimethyl-3(2*H*)-pyridazinone | — | *H* 127 |
| 3-Trimethylsiloxy-4-pyridazinecarbonitrile | crude | 1300 |
| 6-Trimethylsiloxy-3(2*H*)-pyridazinone | — | *H* 69 |
| 3-Trimethylsiloxy-6-trimethylsilylaminopyridazine | 123–125, uv | 1172 |
| 4-Trimethylsilylpyridazine | — | 2046 |
| 3,4,6-Trimorpholinopyridazine | 199–201 | 637 |
| 3,5,6-Triphenyl-4-pyridazinamine | — | 588; *H* 561 |
| 3,4,5-Triphenylpyridazine | 254–255, ir | 632 |
| 3,4,6-Triphenylpyridazine | 168 to 176, nmr | 32, 314, 319, 763, 1591, 1922 |
| 3,5,6-Triphenyl-4-pyridazinecarboxylic acid | 215, ir, ms, nmr | 314 |
| 3,4,6-Triphenylpyridazine 1,2-dioxide | 208–209, ir, nmr | 106 |
| 3,4,6-Triphenylpyridazine 1-oxide | 170–171 | 763 |
| 3,5,6-Triphenyl-4(1*H*)-pyridazinethione | — | *H* 800 |
| 4,5,6-Triphenyl-3(2*H*)-pyridazinethione | 250–251 | 1362; *H* 798 |
| 1,3,6-Triphenyl-4(1*H*)-pyridazinone | — | *H* 136 |
| 2,5,6-Triphenyl-3(2*H*)-pyridazinone | 234 to 247, ir, nmr | 931, 1350, 1351, 1376; *H* 93 |

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| Pyridazine | Melting Point (°C) etc. | Reference(s) |
|---|---|---|
| 3,5,6-Triphenyl-4(1*H*)-pyridazinone | — | 588; *H* 133 |
| 4,5,6-Triphenyl-3(2*H*)-pyridazinone | 282 or 290–292 | 843, 1273, 1277; *H* 128 |
| 3,4,5-Trisbenzylthiopyridazine | — | *H* 814 |
| 3,4,5-Tris-*t*-butylthiopyridazine | 99–100, ms, nmr, unstable | 1219 |
| 3,4,5-Trismethylthiopyridazine | 105–106, p$K_a$, uv; 1-MeI: 148–149, uv; 2-MeI: 115–138, uv | 689 |
| 3,4,6-Trismethylthiopyridazine | 119–120, ir, nmr, uv | 1129 |
| 6-Valeryloxy-3(2*H*)-pyridazinone | — | *H* 68 |
| 6-Vinyloxy-3(2*H*)-pyridazinone | nmr | 1897 |
| 3-Vinylpyridazine | liq, nmr | 494 |
| 2-Vinyl-3(2*H*)-pyridazinone | 59–61 / 0.7, nmr | 693, 1179 |

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# Index

This index covers the text but neither the Appendix (Table of Simple Pyridazines) nor the Glance Indices (appended to Chapters 1 and 2).

The page number(s) immediately following each primary entry refer to synthesis or general information. Although each number indicates that the subject is treated on that (and possibly subsequent pages), the actual word(s) of the primary entry may appear there only in part, as a synonym, as a formula number, or even simply by inference.

Several unusual terms have been employed extensively as succinct secondary entries. For example, the terms chlorolysis and alkanelysis were coined to indicate the direct replacement of appropriate functional groups by a chloro or an alkyl substituent, respectively, so mimicing conventional terms such as aminolysis, thiolysis, and so on.

The few authors mentioned by name in the text, usually for specific contributions or as pioneers of named reactions, are included in this index.

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