

**THIAZOLE  
AND ITS DERIVATIVES**

**IN THREE PARTS**

**PART THREE**

*This is the thirty-fourth volume in the series*

**THE CHEMISTRY OF HETEROCYCLIC COMPOUNDS**

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**THE CHEMISTRY OF HETEROCYCLIC COMPOUNDS**  
A SERIES OF MONOGRAPHS

**ARNOLD WEISSBERGER and EDWARD C. TAYLOR**

*Editors*

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# THIAZOLE AND ITS DERIVATIVES

## PART THREE

*Edited by*

**Jacques V. Metzger**

UNIVERSITY OF AIX-MARSEILLES  
FRANCE

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## **The Chemistry of Heterocyclic Compounds**

The chemistry of heterocyclic compounds is one of the most complex branches of organic chemistry. It is equally interesting for its theoretical implications, for the diversity of its synthetic procedures, and for the physiological and industrial significance of heterocyclic compounds.

A field of such importance and intrinsic difficulty should be made as readily accessible as possible, and the lack of a modern detailed and comprehensive presentation of heterocyclic chemistry is therefore keenly felt. It is the intention of the present series to fill this gap by expert presentations of the various branches of heterocyclic chemistry. The subdivisions have been designed to cover the field in its entirety by monographs which reflect the importance and the interrelations of the various compounds, and accommodate the specific interests of the authors.

In order to continue to make heterocyclic chemistry as readily accessible as possible, new editions are planned for those areas where the respective volumes in the first edition have become obsolete by overwhelming progress. If, however, the changes are not too great so that the first editions can be brought up-to-date by supplementary volumes, supplements to the respective volumes will be published in the first edition.

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## Preface

Given their theoretical as well as practical interest, five-membered aromatic rings occupy a position of particular significance in the enormous field of heterocyclic chemistry. Thiazole is one of the important members of this family and thus merits a comprehensive study. The purpose of this book is to condense into a volume of reasonable size the chemistry of thiazole, covering the literature of approximately one century, up to December 1976. For technical reasons this work has been limited to the study of monocyclic thiazoles, excluding thiamine and partially reduced thiazoles, but including selenazoles. Though most of the important material has been published in the last twenty years, all the literature concerning thiazoles has been surveyed, and it is of special interest to see with what energy Arthur Hantzsch was obliged to defend his historical discovery of thiazole.

In the first chapter, devoted to thiazole itself, specific emphasis has been given to the structure and mechanistic aspects of the reactivity of the molecule: most of the theoretical methods and physical techniques available to date have been applied in the study of thiazole and its derivatives, and the results are discussed in detail. The chapter devoted to methods of synthesis is especially detailed and traces the way for the preparation of any monocyclic thiazole derivative. Three chapters concern the non-tautomeric functional derivatives, and two are devoted to amino-, hydroxy-, and mercaptothiazoles: these chapters constitute the core of the book. All discussion of chemical properties is complemented by tables in which all the known derivatives are inventoried and characterized by their usual physical properties. This information should be of particular value to organic chemists in identifying natural or synthetic thiazoles. Two brief chapters concern mesoionic thiazoles and selenazoles. Finally, an important chapter is devoted to cyanine dyes derived from thiazolium salts, completing some classical reviews on the subject and discussing recent developments in the studies of the reaction mechanisms involved in their synthesis.

The importance of this work, which was begun by Dr. J. M. Swan of Monash University, Melbourne, Australia, was very quickly recognized,

and in 1964 I joined him in his endeavor. Three years later, Dr. Swan was obliged to abandon it, and, for the last decade, 17 distinguished scientists have labored to realize this book. I acknowledge with sincere thanks the cooperation and perseverance of all of them, but I am especially indebted to Michel Chanon and his collaborator René Barone, for the untiring efficiency in management that they have exhibited throughout the preparation of this book. I acknowledge also the help of the numerous heterocyclic chemists of the world who sent so many of their valuable reprints to Marseilles. My thanks are also due to Mrs. J. de Caseneuve and Mrs. G. Formanek who carried out the tedious task of typing the manuscript, and to Thomas Murphy for his help in adjusting the poor original English of most of the manuscript to a hopefully acceptable one. Finally, grateful thanks are due to the University of Aix-Marseilles for financial support and library facilities.

JACQUES V. METZGER

*Marseilles, France  
December 1978*

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### **PART ONE**

#### **Introduction**

#### **I. Properties and Reactions of Thiazole**

J. V. METZGER and E. J. VINCENT, with the collaboration  
of J. CHOUTEAU and G. MILLE

#### **II. General Synthetic Methods for Thiazole and Thiazolium Salts**

G. VERNIN

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R. MEYER

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**Subject Index****PART TWO****General Introduction to Protomeric Thiazoles**

M. CHANON

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C. ROUSSEL, M. CHANON, and R. BARONE

**Subject Index**

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## VIII

# Mesoionic Thiazoles

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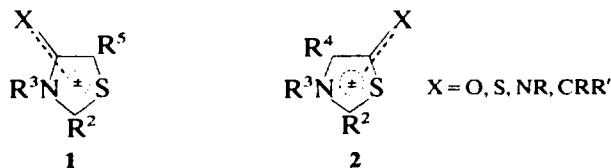
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## I. INTRODUCTION

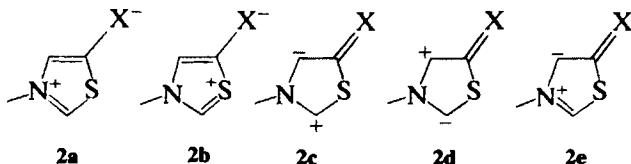
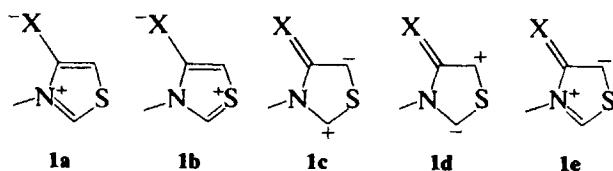
Mesoionic compounds are heterocyclic compounds that cannot be represented by any uncharged formula but only as a hybrid of dipolar structures (1, 2).

Only noncondensed thiazoles in which mesoionic charge delocalization involves atoms directly bonded to the thiazole ring are considered here. Two such systems, **1** and **2**, exist (Scheme 1). Structure **1** ( $X = O$ ) is

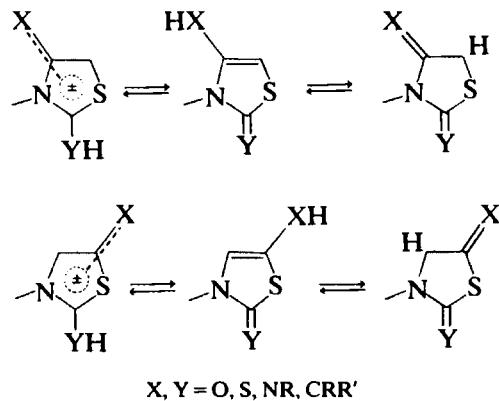


Scheme 1

denoted as a 4-hydroxythiazolium hydroxide inner salt (*Chem. Abstr.*, Index Guide, 1139G, 1977) but the terms *anhydro*-4-hydroxythiazolium hydroxide (**3**) or mesoionic thiazol-4-one (**4**) are also in current use. Structure **1** is a hybrid of the dipolar structures **1a** to **e** and **2** a hybrid of **2a** to **e** (Scheme 2). Prototypy may abolish the mesoionic structure if  $R^2$  in **1** or **2** possesses an  $\alpha$  hydrogen atom (Scheme 3). Observations indicate that the nondipolar tautomers prevail when  $Y = O$ ,  $S$ , or  $NR$ , (5–7), whereas both tautomers may be present when  $Y = CRR'$  (8).

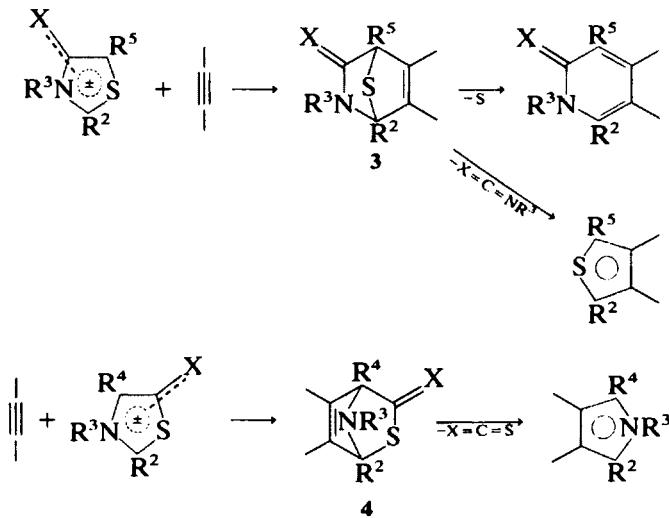


Scheme 2



Scheme 3

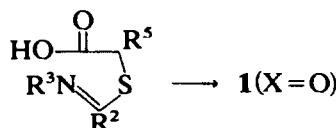
As apparent from the contributing resonance structures, both mesoionic systems contain an azomethinylide contribution, accounting for the reaction with representative dipolarenophiles to give cycloadducts such as **3** or **4** (Scheme 4). The cycloadditions and extrusion reactions of the adducts have been the main object of investigation since previous reviews on mesoionic thiazoles (2, 9). Results appearing since 1969 and before June 1976 are reported for each type of compound in this chapter. Tables VIII-1-5 contain all mesoionic thiazoles described before June 1976.



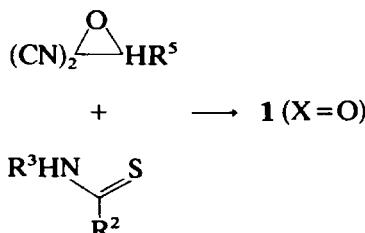
Scheme 4

## II. 4-HYDROXYTHIAZOLIUM HYDROXIDE INNER SALTS

The preparation of these [4-hydroxy-THISs, (**1**), X = O] by cyclization of  $\alpha$ -carboxy-*N*-arylthiobenzimides (**5**) by treatment with acetic anhydride and triethylamine has been investigated in detail, and the structure has been revised for the compound previously described as 2,3-diphenyl-4-hydroxythiazolium hydroxide inner salt (**1**, X = O, R<sup>2</sup> = R<sup>3</sup> = Ph, R<sup>5</sup> = H) (Scheme 5) (3, 10). 4-Hydroxy-THISs also arise by condensation of *gem*-dicyanoepoxides with thioamides (Scheme 6) (8).



Scheme 5

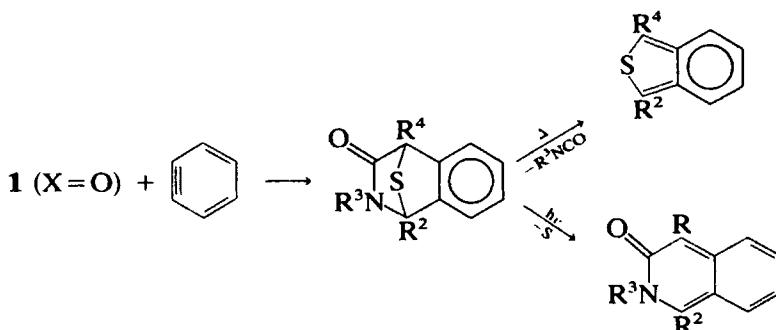


Scheme 6

The 4-Hydroxy-thiazoles are characterized by infrared absorption near 1610 cm<sup>-1</sup> (KBr) (3) or 1620 to 1650 cm<sup>-1</sup> (CCl<sub>4</sub>) (8), indicating a strongly polarized carbonyl group. H-5 resonates near 5.6 ppm in the <sup>1</sup>H NMR spectrum like similar protons in other mesoionic compounds (3). Two fragmentations of the molecular ion are observed in the mass spectra. The first involves rupture of the 1,2 and 3,4 bonds with loss of C<sub>2</sub>R<sup>5</sup>OS<sup>·</sup>. In the second, the 1,5 and 3,4 bonds are cleaved with elimination of C<sub>2</sub>R<sup>5</sup>O<sup>·</sup>.

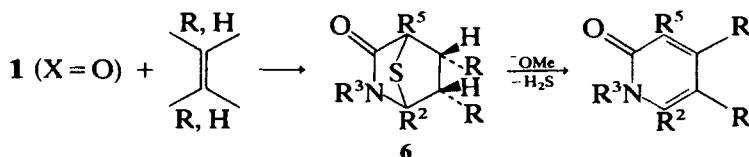
The 4-hydroxy-THISs are extremely hygroscopic; hydrolysis affords the parent thiobenzimide (3). When R<sup>2</sup> = Me and R<sup>5</sup> = *p*-ClPh or Ph, but not *p*-NO<sub>2</sub>Ph the nondipolar tautomer **3** (X = O, Y = CH<sub>2</sub>) is present according to NMR and infrared spectra, the latter exhibiting carbonyl absorption at 1710 to 1720 cm<sup>-1</sup>.

The 4-hydroxy-THISs react with electron-deficient alkynes to give cycloadducts (**3**) that spontaneously eliminate sulfur, producing 2-pyridones (**3**). Bulky 5-substituents lead to a decrease in the addition rate, and elimination of isocyanate with formation of thiophenes becomes favored (**3**, 12, 13). Benzyne yields an isolable adduct that exclusively extrudes isocyanate on thermolysis, but sulfur on irradiation (Scheme 7)

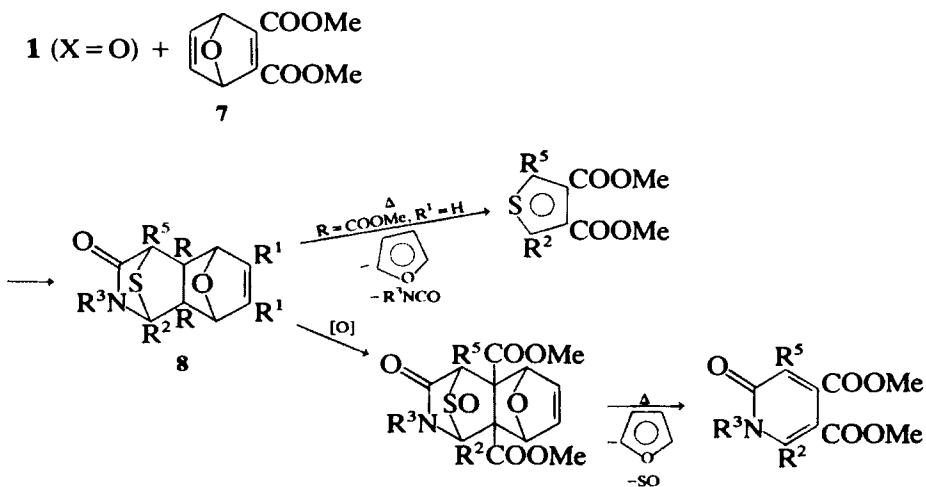


Scheme 7

(14, 15). Electron-deficient alkenes add stereospecifically to 4-hydroxy-THISs with formation of *endo*-cycloadducts. Only with methylvinylketone considerable amounts of the *exo* isomer are produced (Scheme 8) (16). The adducts (**6**) may extrude hydrogen sulfide on heating with methoxide producing 2-pyridones. The base is unnecessary with fumaronitrile adducts. The alternative elimination of isocyanate or sulfur may be controlled using **7** as the dipolarenophile. The cycloaddition produces two products, **8a** ( $R = H$ ,  $R^1 = COOMe$ ) and **8b** ( $R = COOMe$ ,  $R^1 = H$ ) (Scheme 9) (17). Pyrolysis of **8b** leads to extrusion of furan and isocyanate to give a thiophene. The alternative S-elimination can be effected by oxidation of the adduct and subsequent pyrolysis.

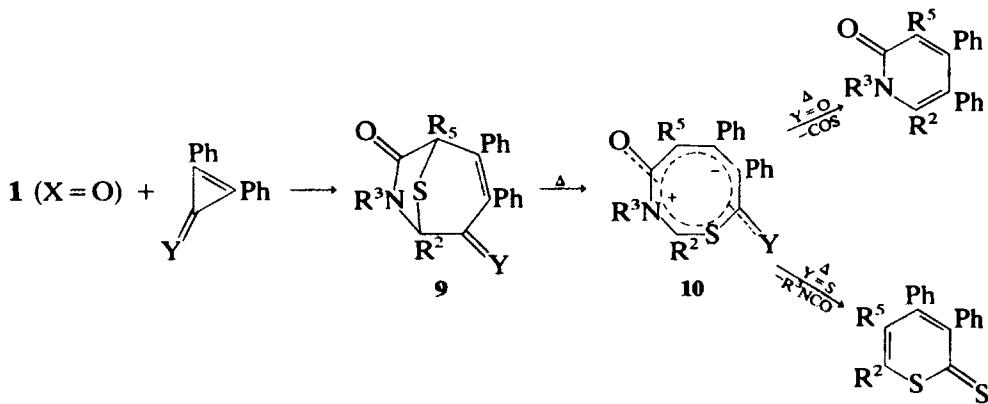


Scheme 8



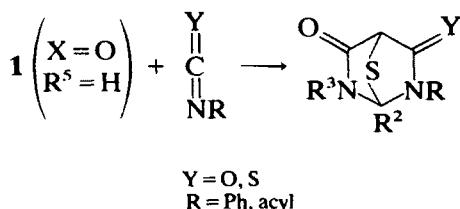
Scheme 9

4-Hydroxy-THISs add to the C–C bond of diphenylcyclopropenethione (18). Inner salts without substituents in 5-position react similarly with diphenylcyclopropenone (Scheme 10) (4, 18). Pyrolysis of the stable adducts (**9**) leads to rupture of the  $\text{R}^2\text{C}-\text{CY}$  bond. Subsequent ring closure yields **10**. When  $\text{Y}=\text{O}$ , **10** eliminates COS, producing 2-pyridone. When  $\text{Y}=\text{S}$ , **10** is isolated together with its isocyanate extrusion product, a thiopyran-2-thione (18).



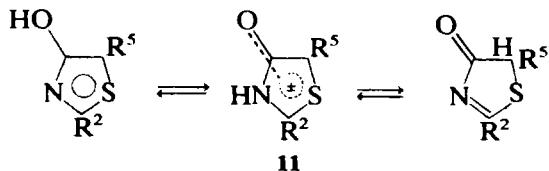
Scheme 10

4-Hydroxy-THISs add regioselectively to the C=N bonds of isocyanates and isothiocyanates producing stable adducts (Scheme 11) (19).



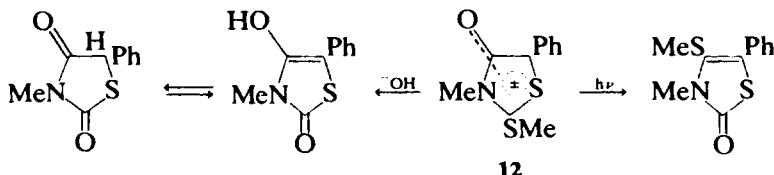
Scheme 11

Interestingly, 4-hydroxythiazoles (**11**) react like the 4-hydroxy-THISs with alkynes and alkenes (Scheme 12) (20), further demonstrating the usefulness of 4-hydroxythiazole derivatives for the preparation of 2-pyridones and thiophenes.



Scheme 12

Irradiation of a 2-methylthio-4-hydroxythiazolium hydroxide inner salt (**12**) leads to exchange of the carbon atoms located in the 2 and 4 positions, probably *via* a thiirenium ion (Scheme 13) (5).

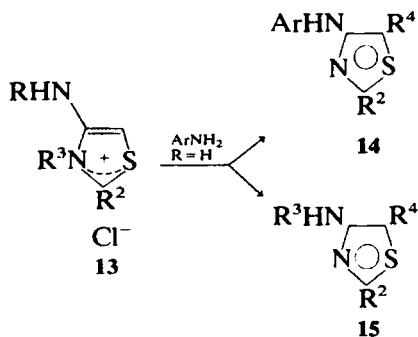


Scheme 13

The methylthio group in **12** can be replaced with OH. Subsequent deprotonation affords a 2,4-dioxothiazole (5).

### III. 4-AMINOTHIAZOLIUM HYDROXIDE INNER SALTS

4-Aminothiazolium hydroxide inner salts (**1**) ( $X = NR$ ) are only known as the derived hydrochlorides (**13**) or acylated hydrochlorides. The former react with arylamines to give 4-arylaminothiazoles. The ratio between **14** and **15** depends on the reaction conditions, a fact accounted for by a mechanism involving ring opening to a diaminocyanomethylthiomethane, followed by recyclization by nucleophilic attack of one of the amino nitrogens on the nitrile carbon. Subsequent ring opening and cyclization with elimination of amine yields the final product (Scheme 14) (21).



Scheme 14

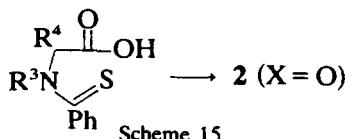
Photolysis of 4-aminothiazolium salts in aqueous solution may cause ring opening, probably via a thiirane, to give  $\alpha$ -cyano- $\beta$ -aminovinyl-disulfide or  $\alpha$ -acylacetamidine derivatives (22).

### IV. 4-MERCAPTO- OR 4-ALKYLIDENETHIAZOLIUM HYDROXIDE INNER SALTS

4-Mercapto- or 4-alkylidenethiazolium hydroxide inner salts (**1**) ( $X = S$  or  $CRR'$ ) have not been described.

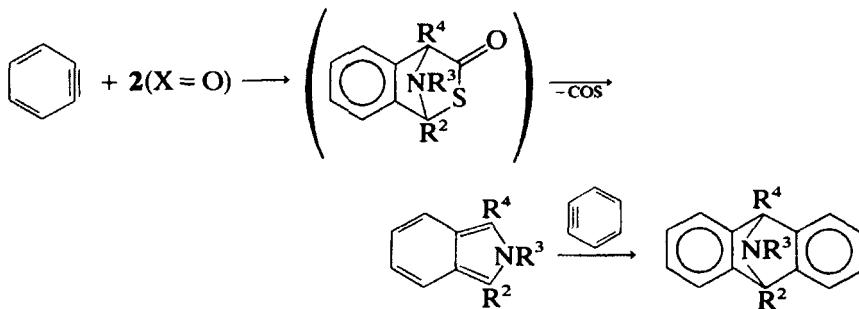
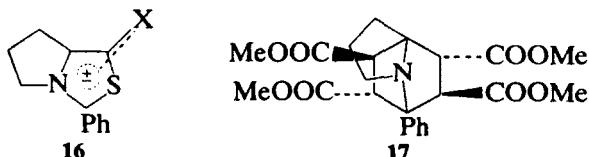
### V. 5-HYDROXYTHIAZOLIUM HYDROXIDE INNER SALTS

5-Hydroxythiazolium hydroxide inner salts (**2**) ( $X = O$ ) have been synthesized by an improved acetic-anhydride-triethylamine-catalyzed cyclization of *N*-substituted *N*-thiobenzoylalanines (Scheme 15) (23).

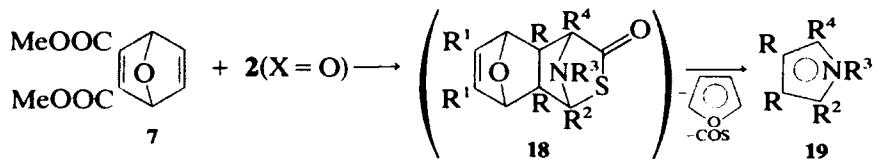


The reasonable stable products are characterized by an ir-absorption near  $1615\text{ cm}^{-1}$ . The 4-protons resonate near 6.2 ppm in the  $^1\text{H}$  NMR spectrum (23).  $^{13}\text{C}$  NMR spectra exhibit a carbonyl atom signal near 173 ppm, whereas C-4 resonates near  $\delta$  108; these positions are characteristic of other mesoionic ring carbon atoms (24). In the mass spectra, decomposition with loss of CO, rupture of the 1,5 and 2,3 bonds with elimination of  $\text{R}^3\text{NC}_2\text{R}^4\text{O}$ ; and cleavage of the 1,2 and 3,4 bonds with elimination of  $\text{C}_2\text{R}^4\text{OS}$  is observed (11)

5-Hydroxy-THISs react with electron-deficient alkynes to give nonisolable adducts that extrude carbonyl sulfide, affording pyrroles (23). Compound **16** ( $\text{X}=\text{O}$ ) seems particularly reactive (Scheme 16) (25). The cycloaddition to benzyne yields isoindoles in low yield. Further cycloaddition between isoindole and benzyne leads to an iminoanthracene as the main product (Scheme 17). The cycloadducts derived from electron-deficient alkenes are stable (23, 25) unless highly strained. Thus the two adducts, **18a** ( $\text{R}=\text{H}, \text{R}'=\text{COOMe}$ ) and **18b** ( $\text{R}=\text{COOMe}, \text{R}'=\text{H}$ ), formed from **7**, both extrude furan and COS under the reaction conditions producing the pyrroles (**19**,  $\text{R}=\text{H}$  or  $\text{COOMe}$ ) (Scheme 18). Similarly, the cycloadduct formed between **16** ( $\text{X}=\text{O}$ ) and dimethylfumarate

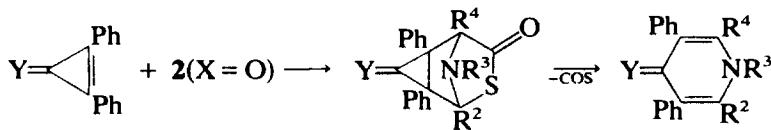


is unstable, eliminating COS and reacting with a second molecule of dimethylfumarate to give **17** as the final product (**17**).



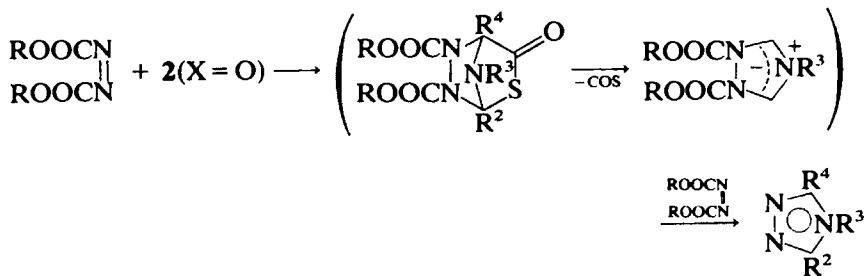
Scheme 18

In contrast to the 4-hydroxy isomers, the thermally stable 5-hydroxy-THISs add to the C=C bond of cyclopropenylidenes (4, 18, 27, 28). The adducts eliminate carbonyl sulfide, and the strained bond breaks resulting in ring-expansion with formation of pyridin-4-ones, -thiones, or -imines, or 4-alkylidenedihydropyridines (**20**, X=O, S, NR, or CRR') (Scheme 19).

 $\text{Y} = \text{O, S, NR, CRR'}$ 

Scheme 19

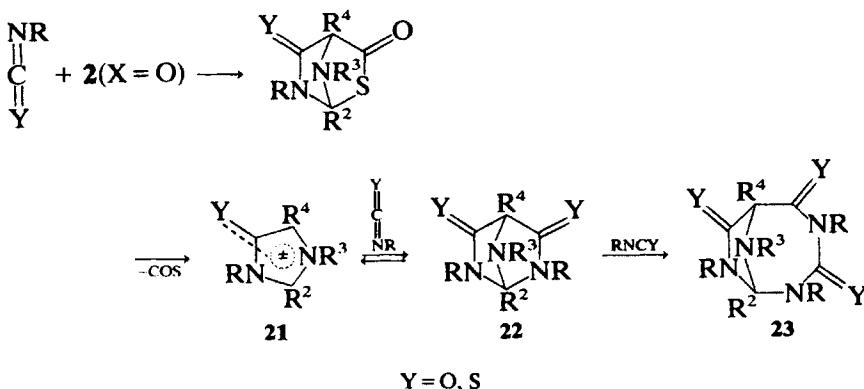
5-Hydroxy-THISs react with diethoxycarbonylazine producing a 1,2,4-triazole via addition, elimination of carbonyl sulfide (29), and subsequent loss of the ester groups (Scheme 20) (30).



Scheme 20

5-Hydroxy-THISs add regioselectively to the C=N bonds of isocyanates or isothiocyanates. The initially formed cycloadducts eliminate carbonyl sulfide with formation of 4-hydroxy- or 4-mercaptopimidazolium hydroxide inner salts (**21**) (Scheme 21). 4-Hydroxyimidazolium hydroxide

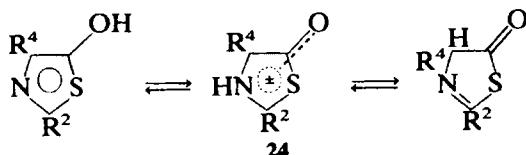
inner salts and not too highly substituted 4-mercapto analogs may undergo further reaction with excess of the heterocumulene, normally affording cycloadducts such as **22** as the final products (23). An equilibrium between **21** and **22** seems to exist, favoring **21** with increasing strain in **22** (26). The strained **22** ( $Y = O$ ,  $R_3R_4 = (\text{CH}_2)_3$ ) react differently, adding a further molecule of isocyanate to give **23** as the final product (26).



Scheme 21

Acylisocyanates or isothiocyanates undergo cycloaddition with 5-hydroxy-THISs under so mild conditions that isolation of the initial adducts becomes possible (23). In cycloaddition reactions the 5-hydroxy-THISs can be replaced by their precursors (23).

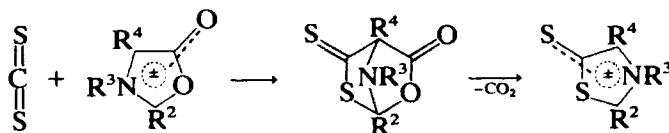
Again, it is noteworthy that 4-substituted 5-hydroxythiazoles (**24**) react like 5-hydroxy-THISs with alkynes to give pyrroles and sometimes with alkenes to give *exo*-cycloadducts (Scheme 22). In the latter case other processes compete with the cycloaddition, becoming dominant when **24** is treated with azo-compounds, enamines, or heterocumulenes (31).



Scheme 22

## VI. 5-MERCAPTO THIAZOLIUM HYDROXIDE INNER SALTS

5-Mercaptothiazolium hydroxide inner salts (**2**) ( $X = S$ ) are prepared from 5-hydroxy-oxazolium inner salts and  $CS_2$  (2, 25). The oxazolium inner salts may advantageously be replaced with their precursors, which are *N*-arylacetylalanins (Scheme 23).

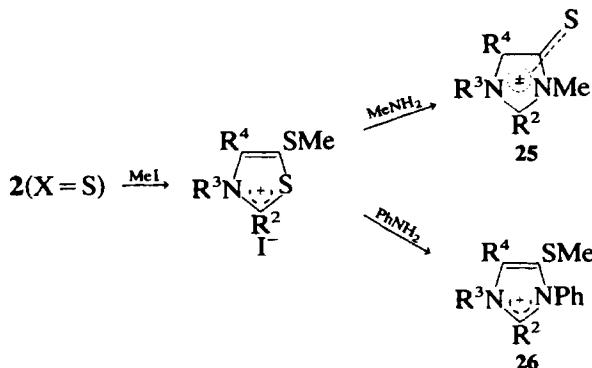


Scheme 23

1,3-Dipolar reactivity of 5-mercaptop-THISs has only been demonstrated for **16** ( $X = S$ ), which, like its oxygen analog, produces with dimethylfumarate, **17**, and with phenylisothiocyanate, **21** (25). Compound **16** ( $X = S$ ) does not react with other typical dipolarenophiles (25).

5-mercaptop-THISs are protonated or alkylated on the exocyclic sulfur atom with simultaneous disappearance of a characteristic long-wave absorption in the ultraviolet region (32, 33). These are stronger for **2** ( $X = S$ ) than for its 4-aza derivative (33).

The 5-methylthiothiazolium salts react with methylamine to give 4-mercaptopimidazolium hydroxide inner salts (**25**) and with aniline to give **26** (Scheme 24) (32).



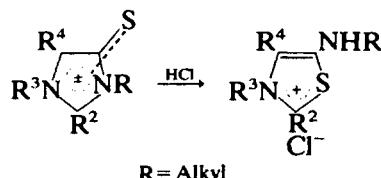
Scheme 24

5-Mercapto-THISs react with dimethoxycarbonylacetylene, producing tetramethoxycarbonylthiophene by an unknown mechanism (29).

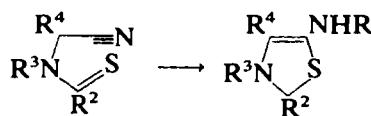
## VII. 5-AMINOTHIAZOLIUM HYDROXIDE INNER SALTS

5-Aminothiazolium hydroxide inner salts (**2**) ( $X = NR$ ) are only known as *N*-phenyl (29) or acyl derivatives, (6, 34) or as hydrochlorides (35).

4-Mercapto-imidazolium inner salts have been reported to rearrange under the influence of hydrochloric acid, producing 5-aminothiazolium chlorides (Scheme 25) (36). Their *N*-acylated derivatives are obtained by cyclization of *N*-thiobenzoyl alkylaminoacetonitriles, effected with acyl or sulfonyl halides (Scheme 26) (34, 35).



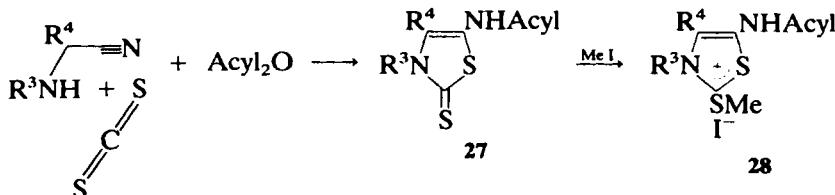
Scheme 25



$R = \text{H, Acyl, Sulfonyl}$

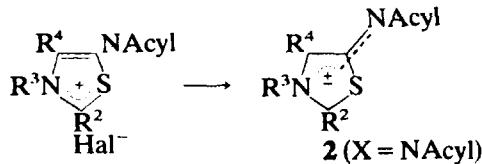
Scheme 26

2-Methylthio-5-aminothiazolium salts (**28**) are accessible through methylation of thiaolethiones (**27**), which are, in turn, obtained from alkylaminoacetonitriles and carbondisulfide (Scheme 27).



Scheme 27

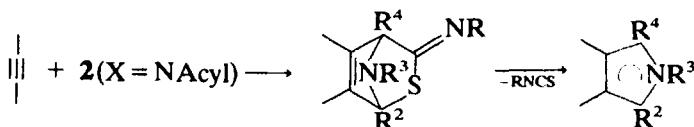
The 5-amino-THISs are very strong bases (35). The hydrochlorides, therefore, have not yet been deprotonized successfully. However, the decreased basicity of the *N*-acylated derivatives makes these readily accessible from their hydrochlorides (Scheme 28).



Scheme 28

The 5-acylamino-THISs are devoid of carbonyl absorptions above  $1600\text{ cm}^{-1}$ , suggesting that their oxygen atom adopt a considerable part of the negative charge of the system (6). A normal carbonyl band appears on protonation, also accompanied by a hypsochromic shift in ultraviolet absorption (6). H-4 of 5-acyl-amino-THISs resonates near 7.4 ppm in the  $^1\text{H}$  NMR spectrum (6). Three fragmentation modes of the molecular ion of these mesoions have been observed (6): rupture of the 2,3 and 1,5 bonds with formation of an  $\text{R}^2\text{CS}^+$  ion, rupture of 1,2 and 3,4 bonds with formation of  $\text{R}^2\text{CNR}^{3+}$ , and cleavage of the 2,3 and 4,5 bonds giving rise to  $\text{R}^3\text{NCR}^{4+}$ .

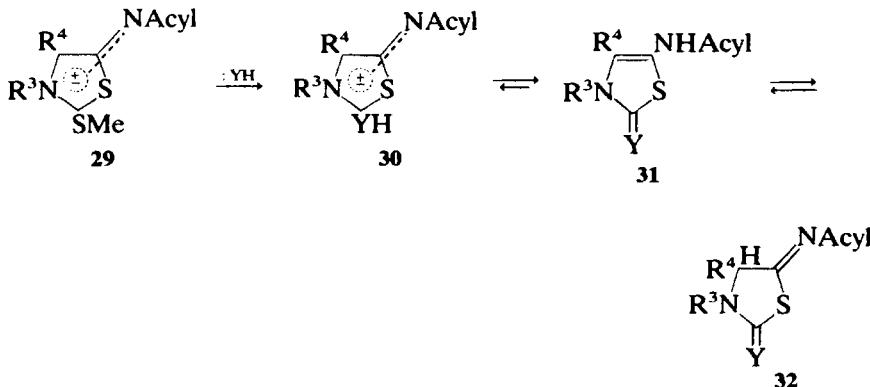
The 5-acylamino-THISs react with alkynes in a way already exemplified for 5-hydroxy-THISs. Pyrroles are formed under elimination of isothiocyanate (Scheme 29) (37). 5-Acylamino-THISs are readily brominated in the 4-position (21).



Scheme 29

2-methylthio-5-acylaminothiazolium hydroxide inner salts (**29**) and nucleophiles :YH react with replacement of the methylthio group

(Scheme 30) (6). Infrared spectra of the products possess a normal amide carbonyl absorption, indicating that the products are not present on the dipolar form (**30**) but rather as the neutral  $\Delta^4$ -thiazoline tautomer (**31** or **32**) (6).



:YH =  $\text{^{\circ}SH}$ ,  $\text{^{\circ}CH(CN)}_2$ , Piperidine in MeOH,  $\text{^{\circ}OH}$ ,  $\text{^{\circ}MeNH}_2$

Scheme 30

5-Benzoylamino-THISs seem to rearrange on photolysis (38).

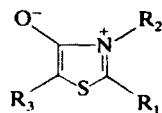
## VIII. 5-ALKYLIDENETHIAZOLIUM HYDROXIDE INNER SALTS

5-Alkylidenethiazolium hydroxide inner salts (**2**) ( $X = \text{CRR}'$ ) have not yet been described.

Besides being useful precursors to pyrroles; pyridine-2-ones; -4-ones, -4-thiones, and -4-imines; 4-alkylidene-dihydropyridines; thiophenes; 1,2,4-triazoles; thiapyrane-2-thiones, isoquinoline-3-ones; isobenzothiophenes; and 4-mercaptopimidazolium hydroxide inner salts, mesoionic thiazoles are potentially useful in the construction of molecules with herbicidic (39), central nerve stimulating, and antiinflammatory properties (40, 41). Application in dye synthesis has likewise been reported (42).

## IX. TABLES OF COMPOUNDS

TABLE VIII-1



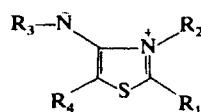
$R_1$	$R_2$	$R_3$	m.p.	Ref.*
H	$\text{Me}(\text{EtO})\text{CH}-$	$p\text{-ClC}_6\text{H}_4-$	—	20
H	$\text{MeO}_2\text{CCH}=$ $\text{C}(\text{CO}_2\text{Me})-$	$p\text{-ClC}_6\text{H}_4-$	—	20
Me	Ph	Ph	172–3	43, 44 u
Me	Ph	$p\text{-ClC}_6\text{H}_4-$	180	8, 45
Me	Ph	$p\text{-O}_2\text{NC}_6\text{H}_4-$	280	8, 45
Ph	Ph	H	113–5 (dec.)	3, 10, 11 sm, 19, 25, 44, 46
Ph	$\text{PhCH}_2-$	Ph	163	43, 44 u, 45
Ph	$\text{PhCH}_2-$	$p\text{-ClC}_6\text{H}_4-$	168–70	8, 45
Ph	$\text{PhCH}_2-$	$p\text{-O}_2\text{NC}_6\text{H}_4-$	210	8, 45
Ph	Ph	Ac	250–2	3, 10, 44
Ph	Ph	$\text{PHCO}-$	252–3	44
Ph	Ph	Ph	270 (free)	
			167–8	3, 8, 10, 11 m,
			( $\text{HClO}_4$ )	12, 14–17, 43–46
Ph	Ph	$p\text{-MeOC}_6\text{H}_4-$	250	45
Ph	Ph	$p\text{-ClC}_6\text{H}_4-$	300	8 i, 45
Ph	Ph	$p\text{-O}_2\text{NC}_6\text{H}_4-$	273	8, 45
Ph	<i>p</i> -Tolyl	Ac	252	43
Ph	<i>p</i> -Tolyl	Ph	248	43
Ph	$p\text{-MeOC}_6\text{H}_4-$	Ac	214	43
Ph	$p\text{-ClC}_6\text{H}_4-$	H	115–6	3, 10, 11, 43
Ph	$p\text{-ClC}_6\text{H}_4-$	Ac	240–2	3, 10
$p\text{-ClC}_6\text{H}_4-$	Ph	H	125–6 (dec)	3, 16, 19, 25
$p\text{-ClC}_6\text{H}_4-$	Ph	Ph	—	16
$p\text{-O}_2\text{NC}_6\text{H}_4-$	Ph	$p\text{-ClC}_6\text{H}_4-$	260	45
$p\text{-O}_2\text{NC}_6\text{H}_4-$	Ph	$p\text{-O}_2\text{NC}_6\text{H}_4-$	242	45
$(\text{Me})_2\text{N}-$	Ph	Ac	251–3	47
$(\text{Me})_2\text{N}-$	Ph	$p\text{-O}_2\text{NC}_6\text{H}_4-$	287	45
$(\text{Et})_2\text{N}-$	Ph	Ac	208	47
$(\text{Bu})_2\text{N}-$	Ph	Ac	132–3	411, 47
1-pyrrolidinyl	Ph	Ac	298–300	411, 47
1-Piperidino	Ph	Ac	214	411
1-Morpholino	Ph	H	244	411

TABLE VIII-1 (Continued)

R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	m.p.	Ref.
1-Morpholino	Ph	Ac	249	47
MeS-	Me	Ph	148	5, 49 x
MeS-	Et		—	42 a
MeS-	H <sub>2</sub> N-	Ph	179-80	49
MeS-	PhCH=N-	Ph	155-6	49
PhCH <sub>2</sub> S-	H <sub>2</sub> N-	Ph	163-4	49

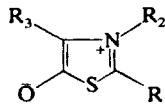
\* Reference code see p. 2 of Part One.

TABLE VIII-2



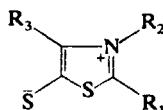
R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	R <sub>4</sub>	m.p.	Ref.
Me	Ph	H	H	173(HCl)	50
Me	Ph	Ac	H	228(HCl)	51
PhCH <sub>2</sub> -	Ph	H	H	190(HCl)	50
Ph	Me	H	H	201(HCl)	50
Ph	Me	Ac	H	215(HCl)	51
Ph	PhCH <sub>2</sub> -	H	H	192(HCl) 194(HBr)	50 51
Ph	PhCH <sub>2</sub> -	H	Ph	176(HCl)	50
Ph	PhCH <sub>2</sub> -	Ac	H	155(free) 151(HCl) 160(HBr)	51
Ph	Ph	H	H	207(HCl) 214-8(HCl)	21, 44, 50
Ph	Ph	H	Ph	155(HCl)	50
Ph	Ph	Ac	H	198(free) 290(HCl)	50, 51
Ph	Ph	PhCO-	H	211-2(free)	50, 37
Ph	Ph	2,4-diClC <sub>6</sub> H <sub>3</sub> OCH <sub>2</sub> CO-	H	—	39 a
Ph	<i>o</i> -Tolyl	H	H	(HCl)	21
Ph	<i>p</i> -MeO-C <sub>6</sub> H <sub>4</sub> -	H	H	(HCl)	21
Ph	<i>p</i> -ClC <sub>6</sub> H <sub>4</sub> -	H	H	(HCl)	21
Ph	<i>p</i> -ClC <sub>6</sub> H <sub>4</sub> -	Ac	H	279(HCl)	51
1-Pyrrolidinyl	Ph	Ac	H	295(HCl)	40 I
1-Piperidino	Ph	Ac	H	265	40 I
1-Morpholino	Ph	Ac	H	260(HCl)	52
MeS-	Ph	Ac	H	126-8(HI)	52
EtS-	Ph	Ac	H	170(HCl)	52
PhCH <sub>2</sub> S-	Ph	Ac	H	120(HCl)	52

TABLE VIII-3



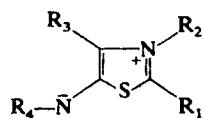
R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	m.p.	Ref.
Ph	Me	H	144-6	11 sm, 23 24 r, 25, 27, 48 53, 54
Ph	Me	Ac	133-5	54-56
Ph	Me	Ph	203-4	4, 15, 17, 28, 29 u, 36
Ph	Me	PhCO-	219-20	54
			220-21	57
Ph	Me	ClHg-	> 300	54
Ph	Me	AcOHg-	158-9	54
Ph	Me	(NC) <sub>2</sub> CHC(CN) <sub>2</sub> -	230-2	23
Ph	Me	(NC) <sub>2</sub> C=C(CN)-	275-7	23
Ph		-CH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> -	148	26, 58, 59, 60
Ph		-CH <sub>2</sub> -S-CH <sub>2</sub> -	145-50	60
Ph	Ph	H	130-1	54
Ph	Ph	Ac	192-3	54
Ph	Ph	Br	163(HBr)	54
Ph	Ph	I	168-9	54
Ph	Ph	ClHg-	187-9	54
p-MeOC <sub>6</sub> H <sub>4</sub> -	Me	H	155-6	11 sm, 23
p-ClC <sub>6</sub> H <sub>4</sub> -	Me	H	159-61	23, 25, 53
p-ClC <sub>6</sub> H <sub>4</sub> -	Me	(NC) <sub>2</sub> C=C(CN)-	283-5	23

TABLE VIII-4



R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	m.p.	Ref.
Me	Me	Ph	184-6	29 i,u
Me	Ph	Me	223-6	29 i,u, 36
Ph	Me	Me	190-5	29 i,u
Ph	Me	Ph	184-5	15, 29, 32 33 k, 36
Ph	-CH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> -		193	26, 58
-o-C <sub>6</sub> H <sub>3</sub> CH <sub>2</sub> CH <sub>2</sub> -		p-O <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> -	—	29

TABLE VIII-5



R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	R <sub>4</sub>	m.p.	Ref.
Ph	Me	H	H	132-3 (HClO <sub>4</sub> )	35
Ph	Me	H	Ac	219-20 (free)	25 i,u,r,sm
				243-4 (HCl)	34, 35 i,u,r
Ph	Me	H	PhCO-	261-2 (free)	35
				237-8 (HCl)	35
Ph	Me	H	p-MeOC <sub>6</sub> H <sub>4</sub> CO-	—	34
Ph	Me	H	p-ClC <sub>6</sub> H <sub>4</sub> CO-	—	34
Ph	Me	H	p-O <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> CO-	—	34
Ph	Me	H	m-O <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> CO-	—	34
Ph	Me	H	PhSO <sub>2</sub> -	—	34
Ph	Me	H	p-MeOC <sub>6</sub> H <sub>4</sub> SO <sub>2</sub> -	—	34
Ph	Me	Me	H	243-4 (HCl)	6
Ph	Me	Me	Me	228-30 (HCl)	6
Ph	Me	Me	Ph	175-6	6
Ph	Me	Me	Ac-	218-9 (free)	6
Ph	Me	Me	PhCO-	250-1 (free)	6
Ph	Me	Ph	H	206-7(HCl) 302-3(HClO <sub>4</sub> )	35
Ph	Me	Ph	Ph	197-9	36
Ph	Me	Ph	Ac	229(free) 207-8(HCl)	35
Ph	Me	Ph	PhCO-	259(free) 187-8(HCl)	15, 35 i,u,r
Ph	Me	Ph	PhNHCO-	231(HCl) 246(free)	35
Ph	Me	Ph	PhNHCS-	241	35
Ph	Me	Ph	PhSO <sub>2</sub> -	242-3	35
Ph	Me	Ph	ON-	187	35
Ph	Me	Br	PhCO-	184(HBr) 236(free)	35
Ph	PhCH <sub>2</sub> -	H	PhCO-	—	34
MeS-	Me	Mc	Ac	176	6 u, 61
MeS-	Me	Ph	Ac	212-3	6 u
MeS-	Me	Ph	PhCO-	244-6	6 u
MeS-	i-Pr	H	Ac	179	6, 61

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## IX

# Cyanine Dyes Derived from Thiazolium Salts

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Hamer's book in this series (1), which reviewed the synthesis of sensitizing dyes, their physical characteristics, and general photographic properties up to 1958, remains the basic encyclopedic source for the study of methine dyes.

More recently, these dyes were the subject of an important chapter by Ficken in Venkataraman's volume published in 1971, "The Chemistry of Synthetic Dyes," which covers supplementary chemical knowledge (2).

Since then, the fundamental physicochemical aspects of the synthesis and properties of cyanines have been exhaustively reviewed by Heseltine and Sturmer in the fourth edition of Mee's treatise (3) and by Sturmer in Weissberger's edition of the "Chemistry of Heterocyclic Compounds" (4). So the purpose of this section dealing especially with thiazolomethine dyes is to give, apart from a complete and recent list of dyes and references, a description of the particularities of their chemistry and chiefly of the reaction mechanisms involved in their synthesis that have remained unknown or have not been discussed until now.

Another objective is to discuss briefly recent and major trends in the field of methine dyes color. Indeed, because of its relatively simple structure, the thiazole ring has been chosen in the past for studying color-structure relations. Using Brooker's "basicity" concepts (5), numerous valuable attempts in different countries succeeded in establishing semiempirical rules for explaining the effects of structural changes on color.

It appears now that, whatever its usefulness, the resonance theory is somewhat inadequate in explaining and predicting either chemical or physical characteristics of dyes compared to more or less sophisticated molecular orbital calculations.

Many patents and studies are still published in the field of thiazolo dyes because the photographic industry is always looking for new sensitizing dyes with improved efficiency and eager to know more about the mechanisms of their action on silver halide.

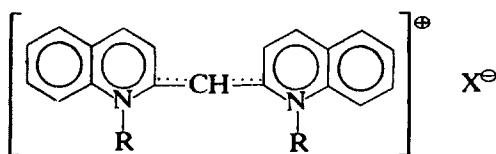
On the other hand, in spite of some interesting tinctorial properties, it does not seem that thiazolo dyes received wide application in the textile industry because they suffer from lack of stability to light and chemical agents.

Recently, however, new interest has developed due to their chemotherapeutic and bacteriostatic usefulness in animal husbandry. They also have been patented as herbicides and plant-growth-regulating agents.

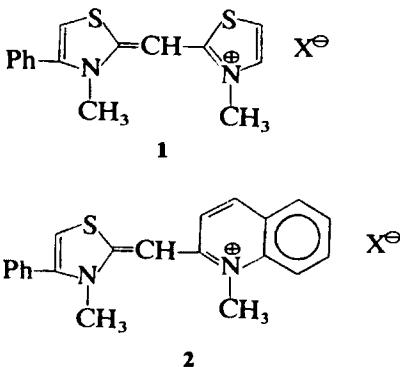
## I. NOMENCLATURE

According to general usage, the term "cyanine" designates any cationic dye in which two nuclei of different or same nature are linked by a mono or polymethine chain. When these groups ( $-\text{CH}=$ ) are replaced partially or totally by one or several nitrogen atoms, the cationic dye is called azacyanine.

The name found its origin in the fact that the first dyes known were all derived from quinoline as, for example, 2,2'-cyanine (Scheme 1). In the case of an asymmetrical or symmetrical dye involving one or two nuclei that are different from the quinoline ring, the name of the nucleus becomes the prefix in the name of the dye. So 3,3'-dimethyl-4-phenyl thiazolo cyanine is the dye of structure 1 and 3,1'-dimethyl 4-phenyl thiazolo 2'-cyanine is represented by the formula structure 2 (Scheme 2).



Scheme 1

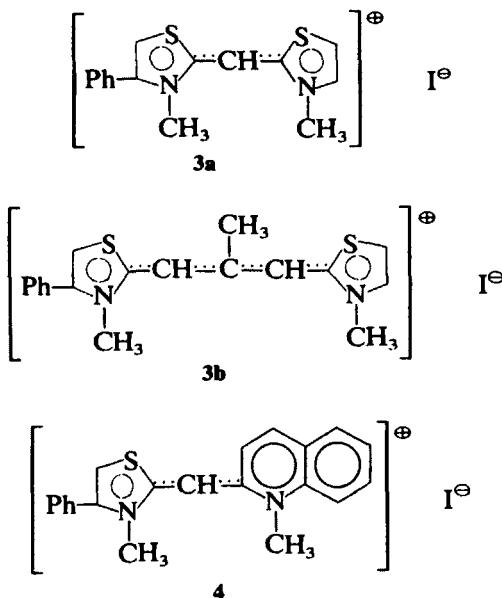


Scheme 2

It appears that this nomenclature is ambiguous to the extent that the basic name thiazolocyanine concerns two dyes of different natures that differ only by an index. Moreover, it implies that the positive charge is located on the nitrogen atom of the quinoline ring. In fact, the charge is entirely delocalized on the chromophoric chain (6–10): it cannot be attributed to one particular nitrogen atom of the component nuclei, which on the contrary possess both a relatively negative charge (11). As a consequence, it is difficult to represent the double bond in a given position rather than in the other one. The methine group is better represented by  $\text{--CH}=\text{}$  rather than by  $\text{--CH=}$ , and each bond possesses its particular value intermediate between a single and a double bond.

Some authors have suggested representing these two dyes as in (Scheme 3), using brackets (12, 13). Consequently, a cationic methine dye is better named in a more logical way by writing successively in brackets the names of the heterocycles involved (not under their dihydro forms) with the position of their attachment, then the length of the methine chain: mono-, tri-, pentamethine (rather than carbo for trimethine, di-carbo for pentamethine). The term cyanine corresponds to a dye of cationic character, since other methine dyes, negatively charged as oxonols, are not designated by the word cyanine.

Applying this rule, the preceding dyes are written (3-methyl-4-phenylthiazole-2)(3-methylthiazole-2)methine cyanine iodide (**3a**) and (3-methyl-4-phenylthiazole-2)(1-methylquinoline-2)methine cyanine iodide (**4**), respectively. Any substituent in the chain is named and its position designated by  $\alpha$ ,  $\beta$ , or  $\gamma$ , for example, (3-methyl-4-phenylthiazole-2)(3-methylthiazole-2)- $\beta$ -methyltrimethine cyanine iodide (**3b**).



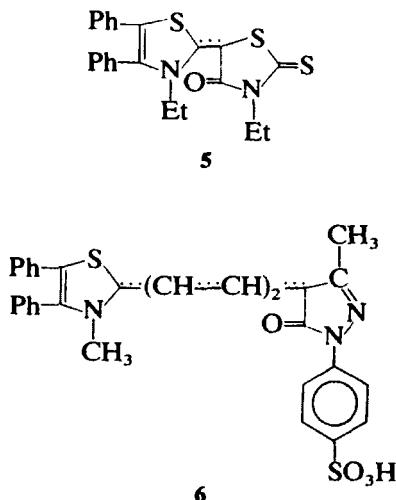
Scheme 3

Merocyanines belong to the class of nonionic methine dyes combining two nuclei, one of which is a ketomethylene of acidic nature such as pyrazolone, rhodanine, oxazolone, thiohydantoin, . . . .

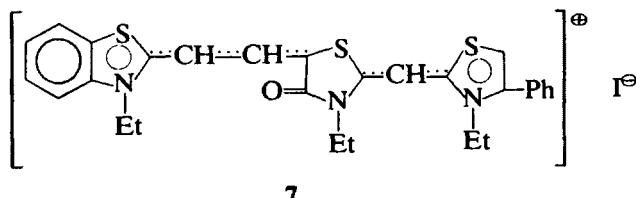
The linking methine chain includes an even number of methine groups (0, 2, 4). They are commonly named as derivatives of the ketomethylene ring, for example, 3-ethyl-5-(3-ethyl-4,5-diphenylthiazolin-2-ylidene)-rhodanine (**5**) and 4-[4-(3-methyl-4,5-diphenylthiazolin-2-ylidene)-2-butenyldiene]-3-methyl-1-*p*-sulfophenyl-2-pyrazolin-5-one (**6**) (Scheme 4).

German or Japanese authors name these dyes (3-ethyl-4,5-diphenylthiazole-2)(3-ethylrhodanine-5)-O-methine neutrocyanine and (3-methyl-4,5-diphenyl thiazole-2)(3-methyl-1-*p*-sulfophenyl-2-pyrazolin-5-one-4)tetramethineneutrocyanine, respectively.

Rhodacyanines possess two chromophoric systems. They are at the same time neutrocyanine derivatives, which involves position 5 of the ketomethylene, and methine cyanine, which involves position 2. Following IUPAC's standard nomenclature rules, structure **7** is named 3-ethyl-4-phenyl-2-{4-oxo-3-ethyl-5-[2-(3-ethyl-2,3-dihydro-benzo-1,3-thiazolylidene)ethylidene]-tetrahydro-1,3-thiazolylidene-methyl}-1,3-thiazolium iodide (Scheme 5). It implies that the 4-phenyl thiazole ring having the



Scheme 4



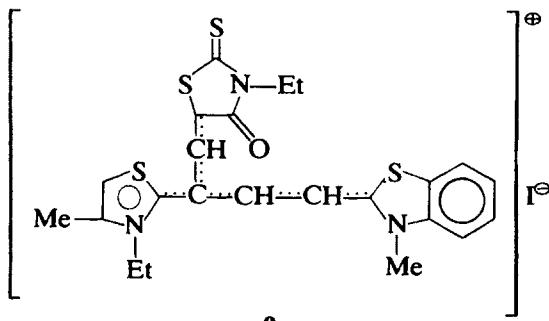
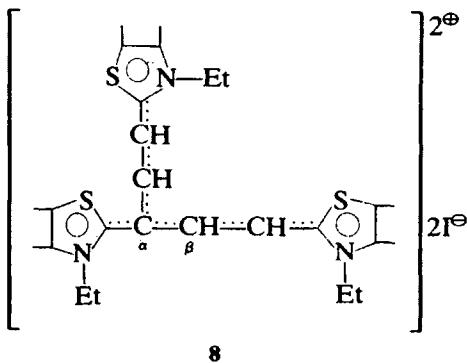
Scheme 5

positive charge plays a prominent part in the dye, although it is not actually possible to locate this charge. The name [(3-ethyl benzothiazole-2)(3-ethyl-4-onethiazole-5)dimethine][3-ethyl-4-phenylthiazole-2]mono-methine cyanine, would not possess such an ambiguity in describing the dye.

Likewise, the nomenclature of neocyanines would not present any more difficulties if these suggested rules were followed, but it is not our task to apply them until official nomenclature is modified.

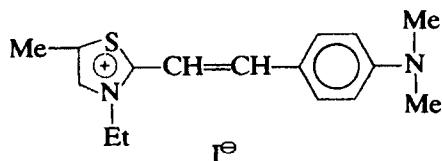
In this last case, the main skeleton considered could be the cationic trimethine cyanine and the third ring with its independent chain could be named as a substituent at the  $\alpha$  or  $\beta \cdots$  position. When this substituent is of an ionic nature, it could be named after the corresponding ring, for

example,  $\alpha$ -(2-vinyl-3-ethylthiazolium-2 iodide)bis-(3-ethyl thiazole-2)-trimethine iodide (**8**) and ( $\alpha$ -methylene-3-ethylrhodanine-5)(3-ethyl-4-methylthiazole-2)(3-methylbenzothiazole-2)trimethinecyanine iodide (**9**) (Scheme 6).



Scheme 6

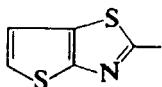
In styryl compounds, a part of the methine chain is replaced by a phenyl group. Their name is based on the nuclei from which they are issued: 3-ethyl-5-methyl-2-(*p*-dimethylaminostyryl)thiazolium iodide (Scheme 7).



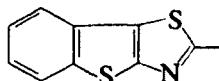
Scheme 7

All dyes of various types that are classified in the tables are derivatives of the thiazole ring with various substituents in positions 2, 3, 4, or 5.

The fused hetero rings of aromatic or pseudoaromatic character on the 4,5 bond as, for example, benzothiazole, naphthothiazole, thieno[2,3-d]-thiazole, benzthieno[2,3-d]thiazole, and so forth (Scheme 8), do not appear in the tables.



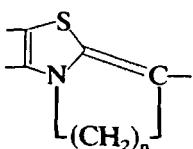
Thieno[2,3-d]thiazole



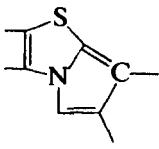
Benzothieno[2,3-d]thiazole

Scheme 8

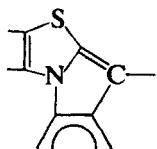
Only the rings shown in Scheme 9 have been indexed.



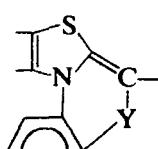
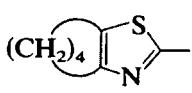
2,3-Polymethylene-thiazole



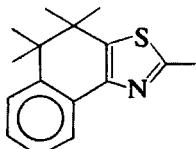
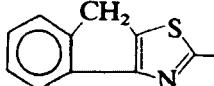
Pyrrolo[2,1-b]-thiazole



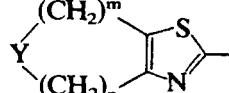
Thiazolo[3,2-a]-indole

Thiazolo[2,3-c]-1,4-benzoxazine  
(Y = O)

4,5,6,7-Tetrahydro-benzothiazole

4,5-Dihydro- $\beta$ -naphtho-thiazole

Indeno[1,2-d]-thiazole

Dihydropyrano-[4,3-d]thiazole  
(Y = O)

Scheme 9

## II. THIAZOLIUM QUATERNARY SALTS\*

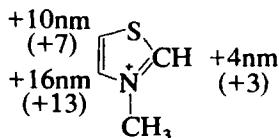
One may find many publications in the literature on the theoretical aspects of thiazolium quaternary salts, because of the biological importance of thiamine and their use as catalysts for benzoin condensation.

\* The authors are indebted to Mrs. J. Vitry for her contribution to the compilation of the references for this section.

This section is restricted to the general properties and chemical behavior of quaternary salts chiefly when they are used as dye intermediates (Tables IX-1-3, IX-4abc, IX-5).

Thiazolium salts can be obtained either directly by slight modifications of ring-closure methods, already described for the parent bases, or by classical quaternization of the bases, the detailed mechanism of which have been reported in Chapter III; the quaternization is best represented by a classical  $SN_2$  mechanism, the solvent playing an important part (14) unless the reaction is run without any solvent.

Nishimoto's fractional core charge model introduced in the Pariser-Parr-Pople method allows good correlation between experimental data, calculated characteristics of electronic absorption of thiazolium salts, and oxidation and reduction potentials (15). Quaternization of the nitrogen atom of a thiazole molecule gives a shift of the first band (662, 663). The importance of the bathochromic effect produced by a methyl substituent in different positions of the nucleus depends on the position of the methyl group as shown by the comparison of theoretical increments (in brackets) to the measured ones (Scheme 10) (15). The important hypsochromic shift [4.92 eV (252 nm) to 5.44 eV (229 nm)] observed between absorption of 4-phenylthiazole and 2,3-dimethyl-4-phenyl-thiazolium has been related to the absence of conjugation between the two cycles as the methyl group on the nitrogen atom prevents the phenyl ring from being coplanar with the thiazole (15, 660).

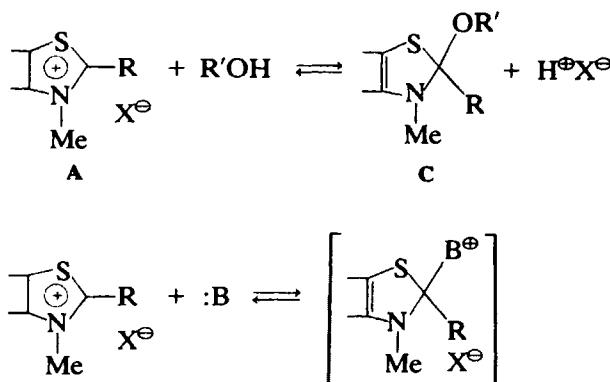


Scheme 10

The mobility of the proton in position 2 of a quaternized molecule and the kinetics of exchange with deuterium has been studied extensively (18-20); it is increased in a basic medium (21-23). The rate of exchange is close to that obtained with the base itself, and the protonated form is supposed to be the active intermediate (236, 664). The remarkable lability of 2-H has been ascribed to a number of factors, including a possible stabilizing resonance effect with contributions of both carbene and ylid structure. This latter may result from the interaction of a  $\sigma$  orbital at the sulfur atom with the  $\sigma$  orbital out of the ring at C-2 (21).

### 1. Electrophilic Properties of Thiazolium Salts

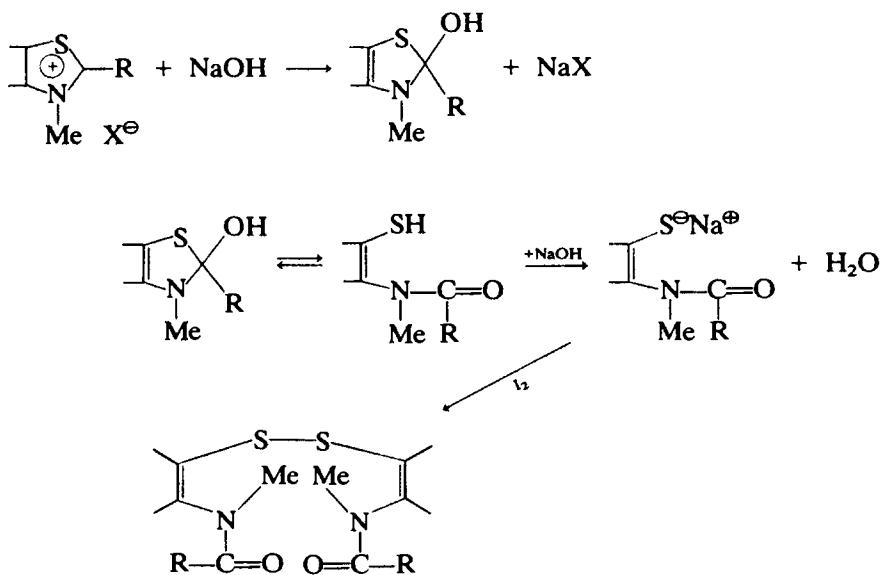
The thiazolium ring, as most heterocycloammoniums, is a Lewis acid conferring to the carbon atom in the 2-position the carbocationic property of adding the free pair of a base either organic or mineral that may be the molecule of solvent as ROH (Scheme 11). For many nuclei of suitable acidity, these equilibria can be observed in dilute solution by means of absorption spectra when species **A** and **C** possess different characteristics (24). For example, benzothiazolium and benzoxazolium in methanol and ethanol give at  $10^{-4}$  mole liter $^{-1}$  8 and 54% of the alkoxy derivatives for the former and 29 and 90% for the latter respectively.



Besides the well-known lower basicity of ethanol, these data illustrate the greater acidity of benzoxazolium compared with benzothiazolium. The relative  $pK_a$  values of the quaternary salts obtained in acetonitrile when treated with tetrabutylammonium hydroxide are 18.3 and 17.6, respectively (25). Those of 2-methyl 4-phenyl thiazolium and 2,4-dimethyl thiazolium are 20.5 and 21.8 under the same conditions (25).

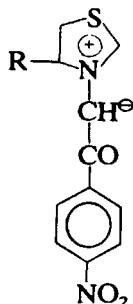
The thiazolium is not acidic enough for observing directly solvation of the molecule (or an hydrolysis and subsequent cleavage of the ring) (24) without adding a base, as it is the case for benzoxazolium or benzothiazolium. With the same dilution ( $10^{-4}$  mole liter $^{-1}$ ), it is necessary to add sodium ethylate to the solution of 2-methyl-4,5-diphenylthiazolium to observe the equilibrium described above. A new band appears in the UV spectrum at 320 nm that is attributed to the ethoxy derivative by analogy to what has been observed with other benzothiazoliums (26).

In aqueous solution whatever the nature of substituent in position 2 ( $R = H$ , alkyl or aryl), a sufficient concentration of NaOH is necessary to cleave the ring after formation of an unstable carbinol in a first step (Scheme 12) (27). The total consumption of alkali amounts to 2 moles per mole (28). The cleavage of the molecule is reversible, since ring closure occurs when adding an acid. The thiol formed can be titrated by an oxidizing agent (29). The kinetics of reversible cleavage were automatically followed by anodic waves measurements of thiols issued from various substituted thiazoliums. Electron-donating groups seem to stabilize the ring for OH attack (30), confirming that cleavage of the unsubstituted ring is easier (31).



Scheme 12

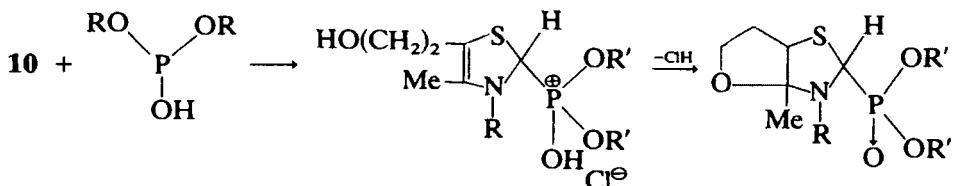
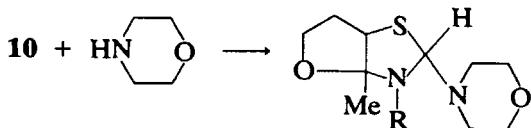
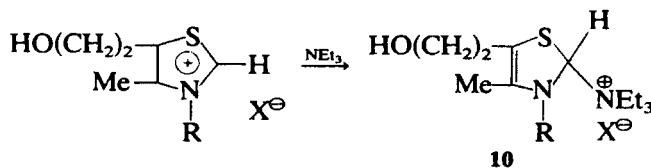
When the nitrogen atom is substituted by a nitrophenacyl group, OH attack gives the betainic zwitterion (Scheme 13), which is soluble in organic solvents (32). The stability of the C-betainic or ylid structure has been explained as an effect of resonance of the negative charge in the molecule (33, 34).



Scheme 13

Chiefly in an hydrophobic medium, a base can extract the proton on position 2 leading to a reactive intermediate (able to give subsequent condensation) that could be an ylid (35, 36) or a carbene (37), though no dimer has ever been isolated as is the case with benzothiazolium (32, 38). Two mechanisms have been proposed for explaining the particular reactivity of thiazolium:

1. Electrophilic reaction can account for the additions of morpholine or phosphonyl (Scheme 14), or

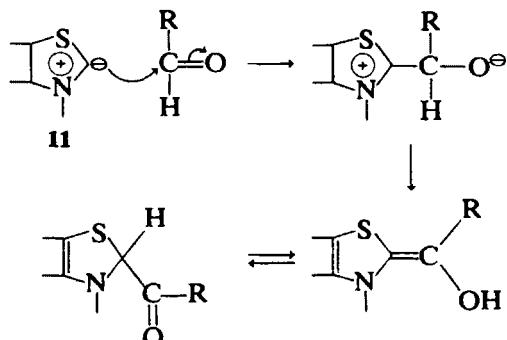


Scheme 14

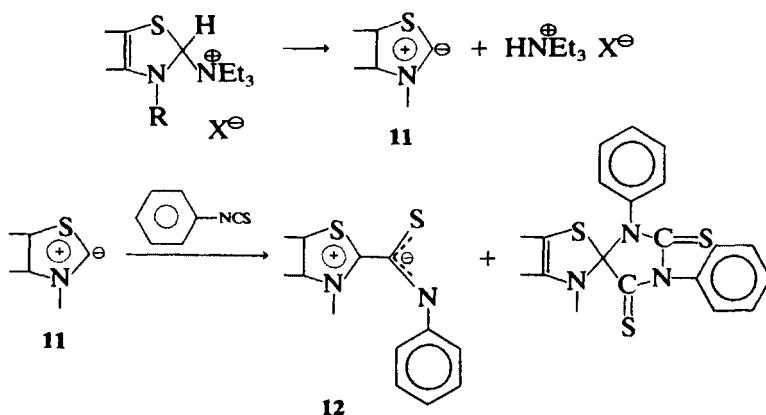
## 2. Nucleophilic reaction of an ylid, under hydrophobic conditions.

This has been the subject of many investigations starting from thiamine itself or derivatives, with the aim of explaining the role of pyrimidine group in coenzymatic activity.

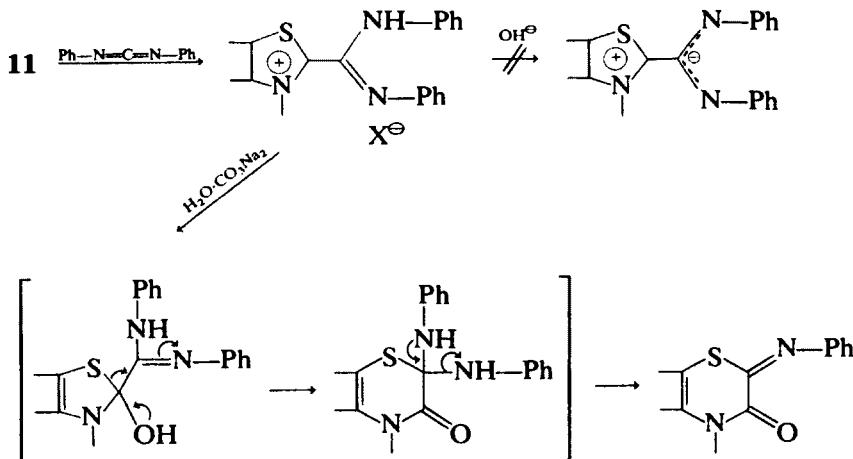
Adducts from various quaternary salts have been isolated, in reactions with aldehydes,  $\alpha$ -ketoaldehydes, dialkylacylphosphonates and dialkylphosphonates, isocyanates, isothiocyanates, and so forth (Scheme 15) (36). The ylid (**11**) resulting from removal of a C<sub>2</sub> proton from 3,4-dimethyl-5- $\beta$ -hydroxyethylthiazolium iodide by NEt<sub>3</sub> in DMF gives with phenylisothiocyanate the stable dipolar adduct (**12**) that has been identified by its NMR spectrum and reactional product, such as acid addition and thiazolidine obtention via NaBH<sub>4</sub> reduction (Scheme 16) (35). It must be mentioned that the adduct issued from di-*p*-tolylcarbodiimide is separated in its halohydrogenated form. An alkaline treatment occasions an easy ring expansion into a 1,4-thiazine derivative (Scheme 17) (35).



Scheme 15



Scheme 16



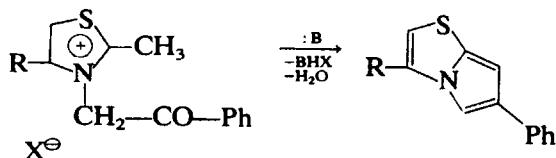
Scheme 17

## 2. Specific Behavior of 2-Methylthiazolium Salts in Basic Medium



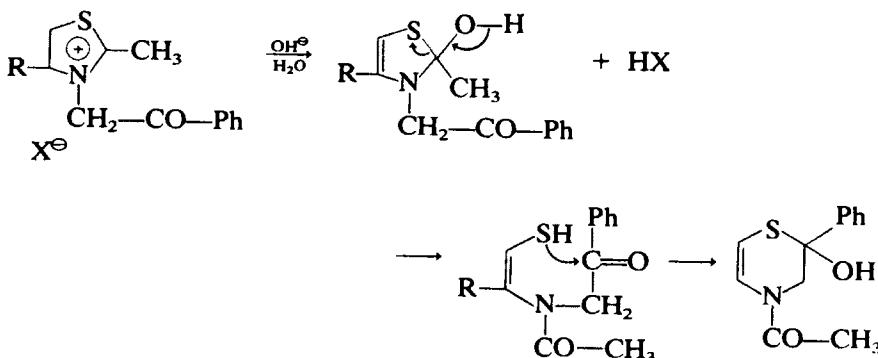
When the substituent on the nitrogen atom is  $-\text{CH}-\text{C}(=\text{O})-\text{R}_2$ ,  $\text{R} = \text{H}, \text{Me}$ ,  $\text{R}_2 = \text{Ph}, \text{Me}$ , two types of reactions can occur, according to the nature of the medium in which the base is allowed to react:

1. In aprotic conditions acetic anhydride sodium acetate induces formation of a fused ring through an intra molecular condensation. It results in a pyrrolo[2,1-*b*]thiazole (39), which constitutes an interesting intermediate for the synthesis of dyes (Scheme 18) (40).



Scheme 18

2. But when the reaction is carried out in an aqueous solution of sodium hydrogen carbonate, extension of the ring occurs with formation of dihydro-1,4-thiazine derivative (Scheme 19), the structure of which has been established by mean of NMR and infrared spectra (41).



Scheme 19

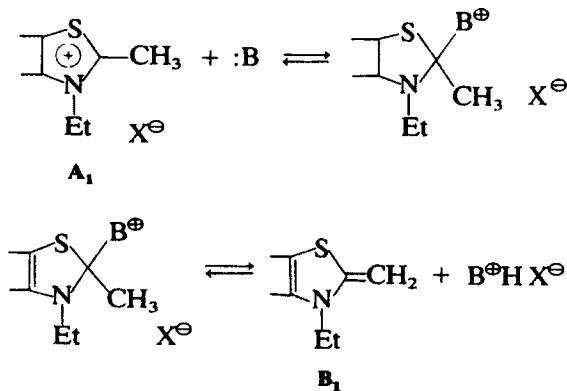
### A. Anhydrobases

In 1923, Mills introduced thiazole for the first time in the synthesis of methine dyes through a somewhat indirect route. In order to demonstrate the 2,4'-cyanine mechanism of formation by quinoline and quinaldine quaternary salts reacting together, Mills used other pairs of quaternary salts as 2-methylthiazolium with either quinolinium or benzothiazolium (42, 43).

Even if the specific role attributed to benzothiazolium was not confirmed later (24), all these syntheses account for the significant and common behavior of quaternary salts, carbocations giving either symmetrical or asymmetrical reactive anhydrobases. They constitute the main step in cyanine dye formation.

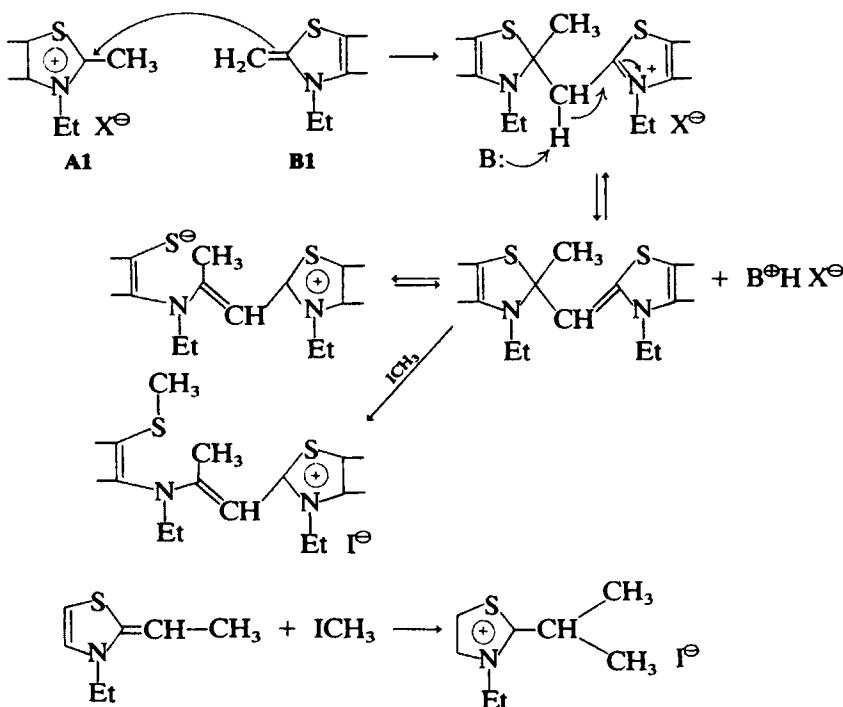
A sufficient concentration of base B is necessary for the removal of a proton of the  $\text{CH}_3$  group. In a first step, the equilibrium in Scheme 20 results, in which the monomeric anhydrobase  $B_1$  constitutes the conjugated base of the quaternary salt  $A_1$ . As has been shown for other rings (24), the equilibrium depends upon the concentration of the different species and the relative strength of the bases B and  $B_1$ , and depends also upon the nature of X.

Many monomeric heterocyclic anhydrobases can be isolated now using specific methods (44), but application of these methods to thiazole ring did not succeed; however, appropriate conditions lead to the separation of a dimer, the structure of which has been established by its NMR spectra and chemical reactivity (26). The most probable mechanism of its formation appears identical with the one previously described in the benzothiazolium series (24). A second molecule of quaternary salt  $A_1$



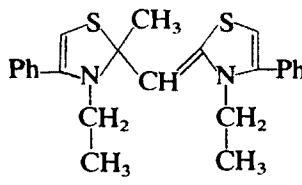
Scheme 20

condenses with the anhydrobase **B<sub>1</sub>**, giving the conjugated acid of the dimer; afterwards, this dimer leads to the anhydrobase through removal of a proton (Scheme 21). This dimer is more stable than **B<sub>1</sub>** itself.

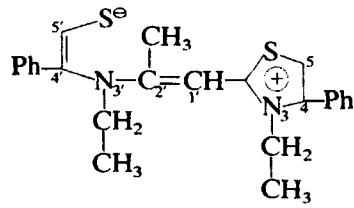


Scheme 21

At first, the dimeric nature of the base isolated from 3-ethyl-2-methyl-4-phenylthiazolium was postulated via a chemical route. Indeed the adduct of  $\text{ICH}_3$  on a similar 2-ethylidene base is a 2-isopropylthiazolium salt; in the case of methylene base it is an anilinovinyl compound identified by its absorption spectrum and chemical reactivity (45–47). This dimeric structure of the molecule has been definitively established by its NMR spectrum. It is very similar to the base issued from 2,3-dimethylbenzothiazolium (48). It corresponds to 2-(3'-ethyl-4'-phenyl-2'-methyleneethiazolinilydene)2-methyl-3-ethyl-4-phenylthiazoline (13). There is only one methyl signal ( $\delta_2 = 2.59$ ), and two series of signals ( $\delta_3 = 1.36\text{--}3.90$ ,  $\delta_3 = 1.12\text{--}3.78$ ) correspond to ethyl groups. Three protons attributed to positions 1',5,5' are shifted to a lower field: 5.93, 6.58, and 8.36 ppm. The bulk of the ten phenyl protons is at 7.3 ppm (Scheme 22).



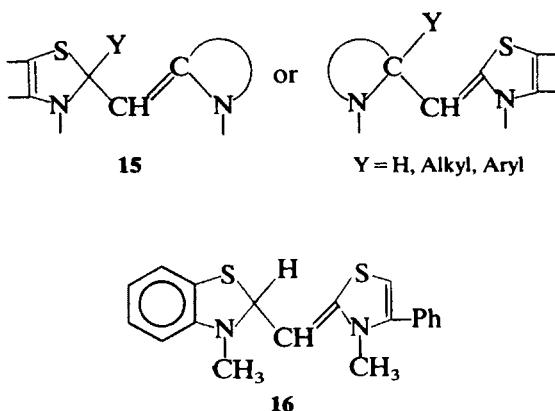
13



Scheme 22

By comparison with the anhydروبase of 2,3-dimethyl benzothiazolium (48), the following facts can be noted. The shift of the proton 5' signal seems abnormal, the proton signals of 2'-methyl group (2.59 ppm in  $\text{CDCl}_3$  and  $\text{CD}_3\text{CN}/\text{Tetramethylsilane}$  are shifted upfield with respect to the corresponding methyl group (1.66 ppm), and it is the same for the signal of the methine proton 1': 5.93 versus 4.42 ppm. The NMR spectrum accounts better for a zwitterion vinylic structure such as 14, which also possesses the same absorption spectrum as the identified anilinovinyl quaternary adduct, resulting from  $\text{ICH}_3$  action ( $\lambda_{\max} = 380 \text{ nm}$ ) (26).

The zwitterionic opened form, and the closed form, could exist in equilibrium. Anyway, such a molecule is characterized by a methine group joining two rings, one of the starting rings still possessing its methyl-substituted group. Such dimeric structures issued from one same ring may be considered as symmetrical, contrary to asymmetrical ones, where the methine bridge joins two heterocyclic ring of different nature (**15**) such as described for benzothiazole series (Scheme 23) (49). These last derivatives are much more difficult to separate. However they appear to be at the origin of the dyes obtained either in Mill's reaction (42), for example, **16**, or resulting from the action of a basic agent on 2-methylthiazolium.

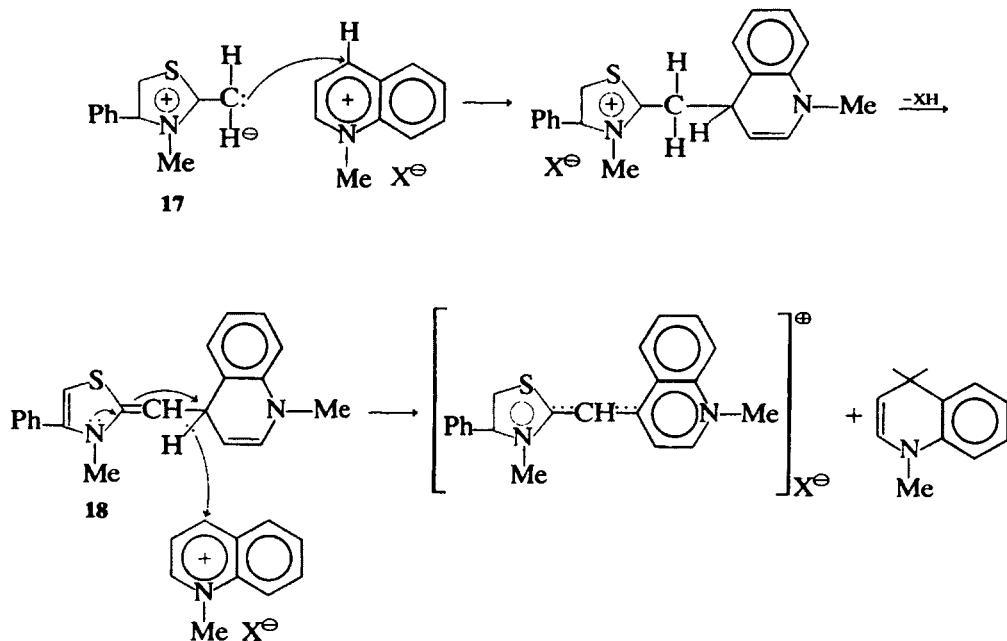


Scheme 23

### B. Mechanism of the Thiazolocyanine Formation from Anhydrides

This mechanism undoubtedly has a close analogy with the ones described previously in quinoline series, which have received experimental support (54). They can be classified as oxidative synthesis. The dye (3-methyl-4-phenyl thiazole-2)(1-methyl quinoline-4)monomethine cyanine iodide was obtained by condensing the corresponding quaternary salts in alcoholic solution with sodium hydroxide as basic agent (method H) (Scheme 24) In a first step, a proton is extracted from the methyl group by the basic agent B, giving the conjugated base (**17**). It is followed by a nucleophilic attack of the monomeric anhydride on carbon-4 of a quinolinium ion, which is the most electrophilic substrate (quinolinium is

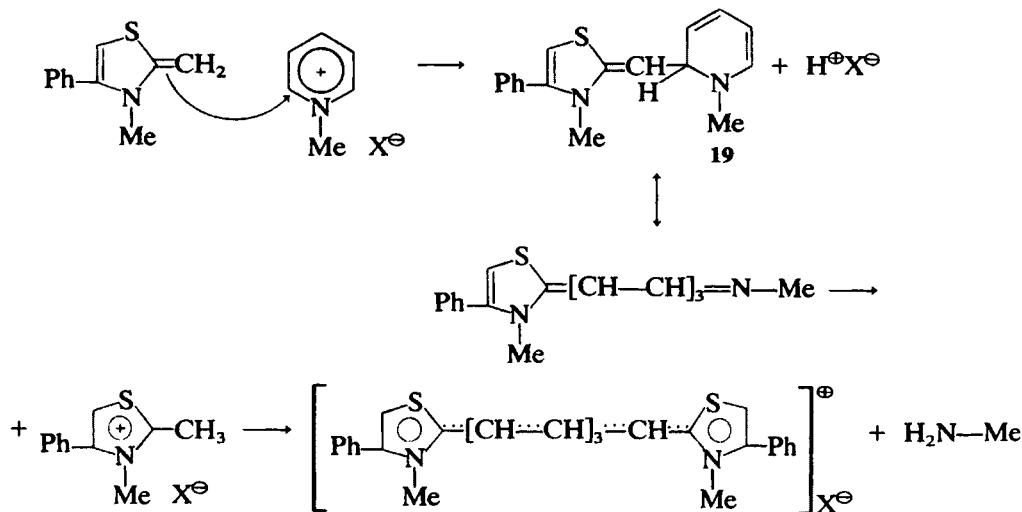
known to undergo nucleophilic attack in position 2 and chiefly in position 4). The conjugated acid loses one proton by action of B to give the reactive unsymmetrical anhydrobase (**18**). In a second step, the hydrogen atom in position 4 of this molecule is transferred in the form of a hydride ion to a second molecule of quinolinium. The former molecule being oxidized, a charge appears on it while the latter is reduced.



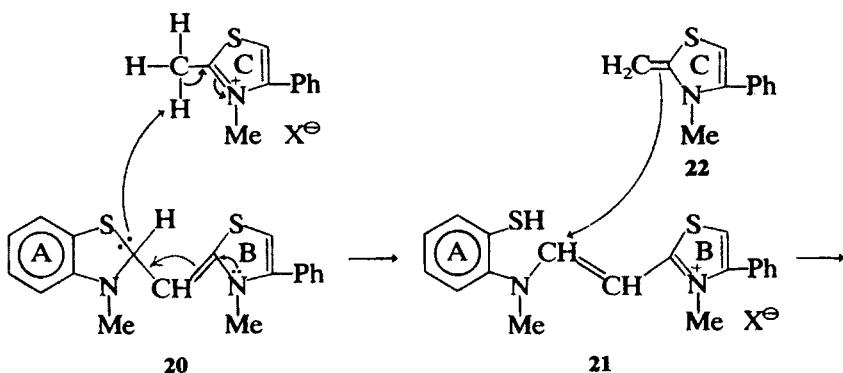
Scheme 24

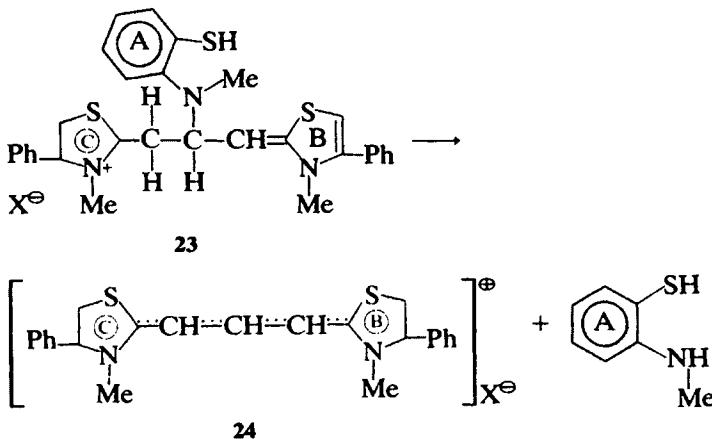
As a whole, 2 moles of quinolinium react with one of thiazolium together with 2 moles of base. This explains the traditional interest of making the condensation with an excess of quinolinium salt in order to get the maximum yield (50, 51).

In the case of a pyridinium salt, an indirect proof of the correctness of this mechanism can be found in the presence of a heptamethine thiazolo-cyanine, together with monomethine, which results unambiguously from **19** by the known opening of the pyridine ring (Scheme 25) (52).



The asymmetrical anhydrobase (**20**) constitutes the first step of the formation of trimethine thiazolocyanine when a 2-methylthiazolium salt reacts either with a benzothiazolium or its opened form [which is bis-*o*-(formylmethylamino)(diphenyl disulfide] (Scheme 26). In a second step, **20** is protonated by a second molecule of 2-methylthiazolium. It results in cleavage of the benzothiazoline ring, which gives **21** together with the formation of the monomeric anhydrobase (**22**). Cleavage of the C-S bond of **20** can be explained by the important electronic desaturation of the C atom observed in NMR spectrum and the great polarizability of the C-S bond in this type of ring (48).





Scheme 26

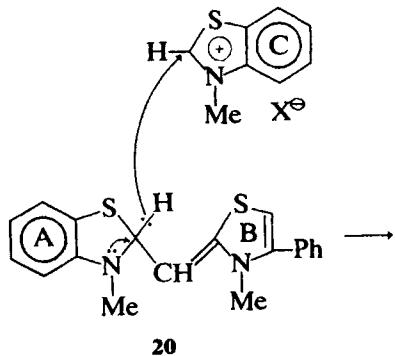
In a third step, the anhydrobase reacts on the carbon which is the most sensitive to nucleophilic attack. The unstable intermediate **23** splits into aminothiophenol and trimethine thiazolocyanine (**24**).

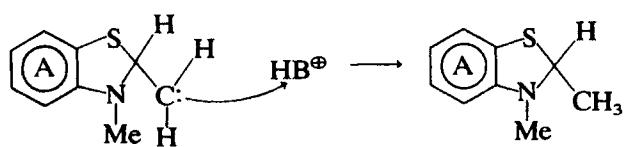
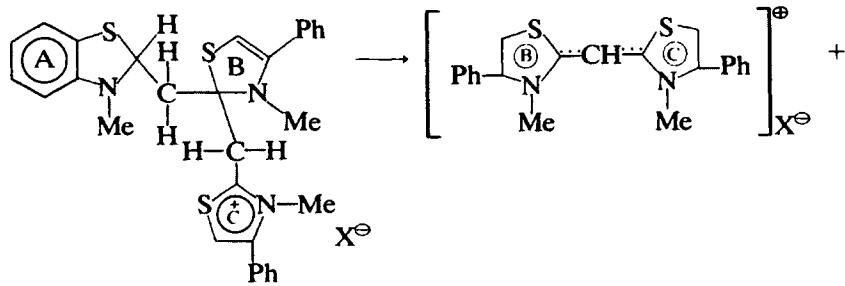
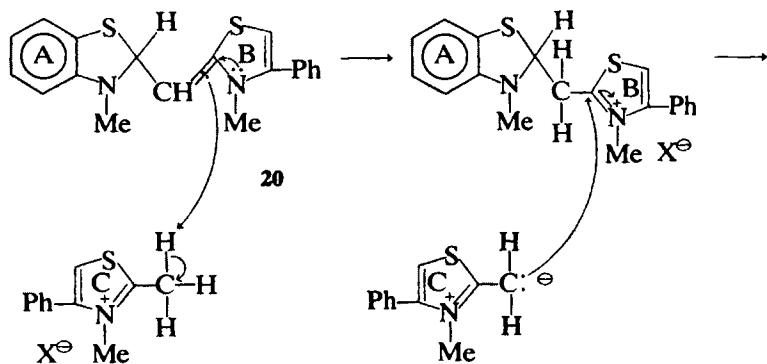
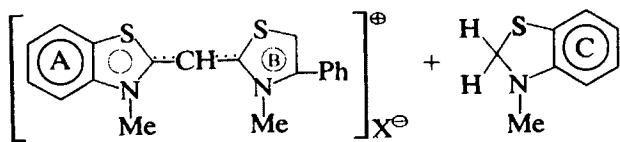
The structure of **20** also explains why the Mill's reaction (43) leads to (3-methylbenzothiazole-2)(3-methyl-4-phenylthiazole)monomethine iodide.

Two probable mechanisms may be involved:

- Hydride ion transfer as in the case of isocyanine (49),
- Possibility of making 2-methyl benzothiazolium either by reaction of the benzothiazolium quaternary salt on the anhydrobase (**24**) or by reaction of *o*-methylaminothiophenol on the same anhydrobase (**26**).

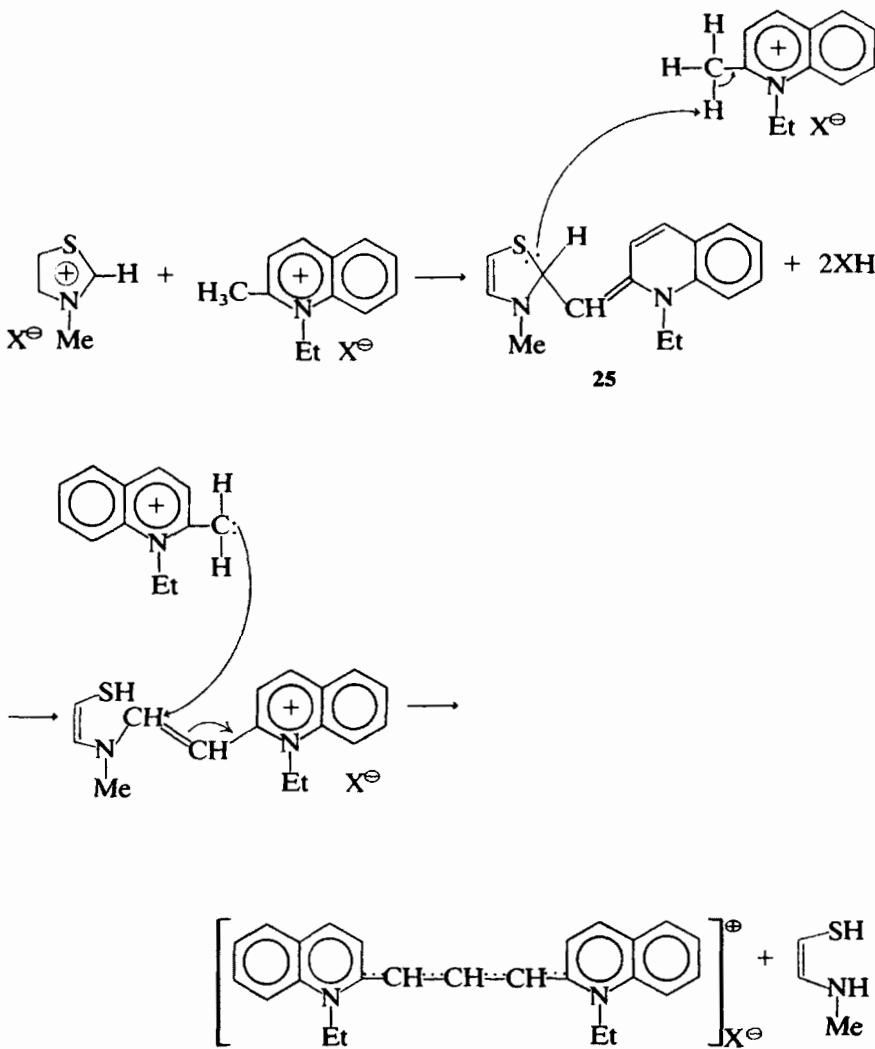
The symmetrical dye, bis-(3-methyl-4-phenylthiazole)monomethine cyanine (not mentioned by Mills), has also been identified, and its formation is explained by the enamine character of **20** (Scheme 27).



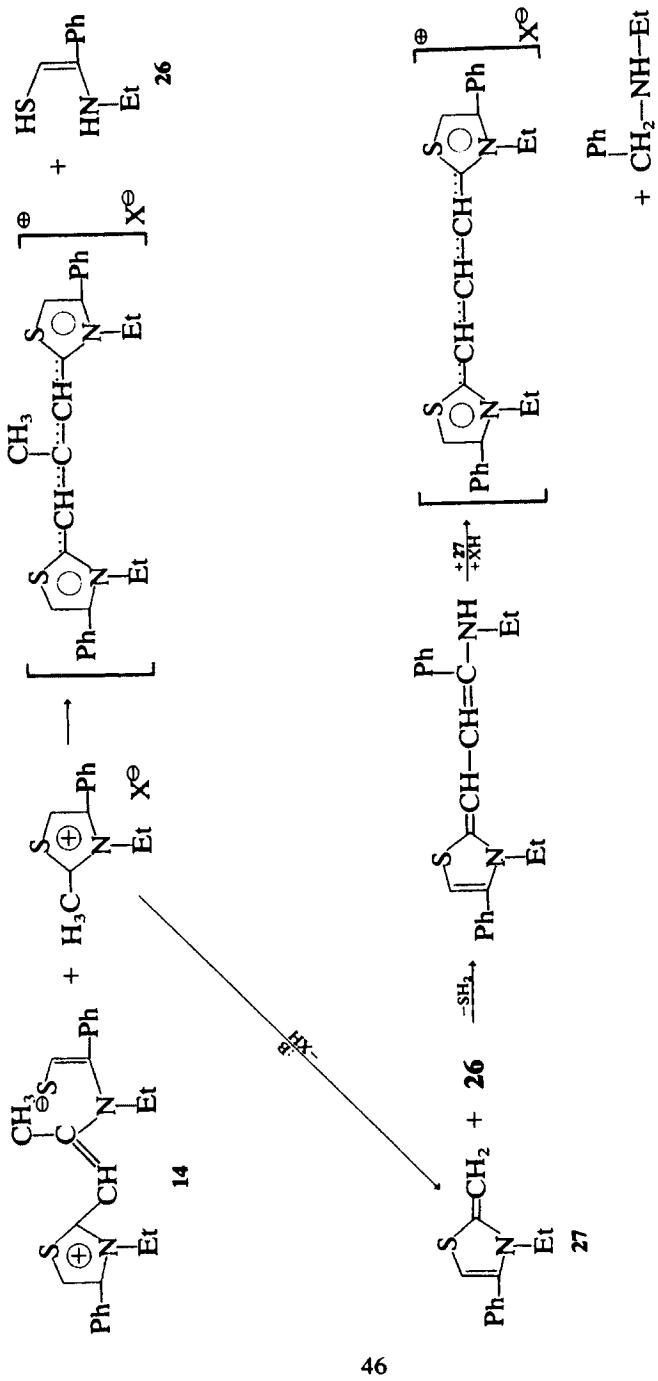


Scheme 27

When a quinaldinium salt and 2-unsubstituted thiazolium are condensed together in the presence of a basic agent, the resulting bis-(methylquinoline-2)trimethine cyanine is issued from the cleavage of the thiazolium ring of the anhydروبase (25). It is induced by the  $-CH_3$  attack of quinaldinium according to a process already described (Scheme 28).



Scheme 28



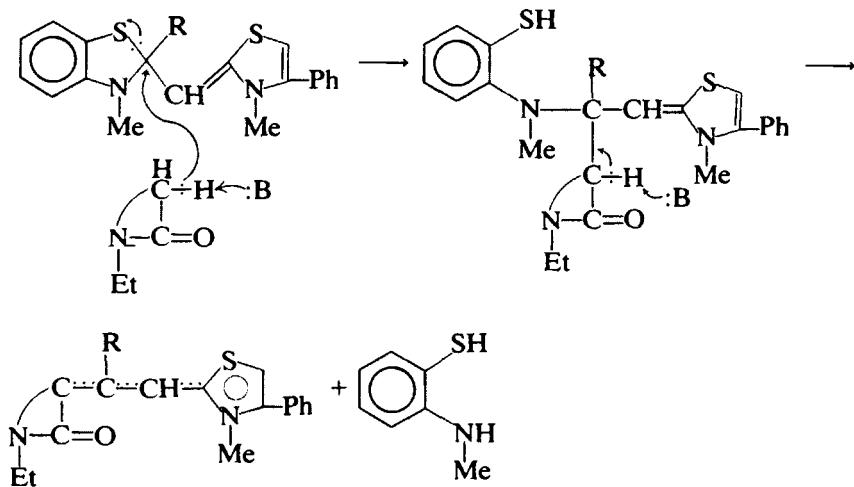
Scheme 29

Due to the structure of the symmetrical anhydrobase, every time a 2-methylthiazolium undergoes the attack of a base, theoretically it can result in two trimethine thiazolocyanines: the mesomethylsubstituted one and the unsubstituted one. For an unexplained reason, it seems that when position 5 of the starting molecule is substituted, only the mesomethyl dye is produced according to the absorption spectrum, 530 nm for the methylmeso and 569 nm for the 4-phenyl substituted derivative (Scheme 29).

As an illustration of the real complexity of Mill's reaction, when two molecules of heterocycloammoniums of different nature, one of them being thiazolium (2-substituted or not), are put together in a basic medium, nine dyes theoretically can be produced (depending on the nature of the substituent in the ring): three thiazolomonomethine cyanines (two symmetrical, one unsymmetrical) and six trimethine cyanines (two symmetrical, two symmetrical mesosubstituted, one unsymmetrical, one unsymmetrical mesosubstituted). One cannot separate such a mixture by usual chromatographic means.

### C. Obtention of Neutrocyanine from Anhydrobases

The nucleophilic carbon of ketomethylene compounds can react with anhydrobases of different species in a basic medium. This reaction presents a narrow similitude with  $-\text{CH}_3$  attack. The resulting dye, neutrodimethine cyanine either mesomethyl-substituted or not, varies with the nature of the anhydro base (Scheme 30) (53, 54).



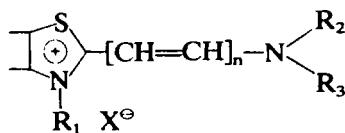
Scheme 30

### III. SYNTHESSES OF THIAZOLOCYANINES

#### 1. Mononuclear Cyanines

##### A. Hemicyanines (Tables 1111, 1112A,B,C,D, 1113)

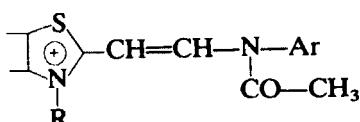
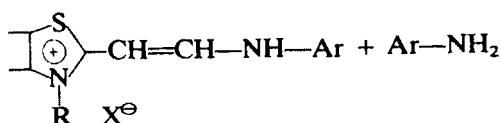
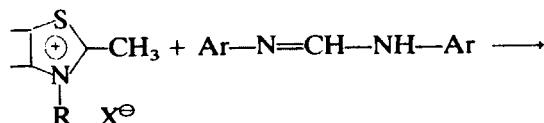
The hemicyanines constitute a particular class among streptocyanines in which one of the two nitrogen atoms belongs to a heterocycloammonium. Thiazolohemicyanine corresponds to the general formula in Scheme 31. When  $R_2 = R_3 = H$ , the dyes are of aminovinyl type; when  $R_2 = H$ ,  $R_3 = Ph$ , they are anilinovinyl compounds, and when ( $-CH=CH-$ ) is replaced by a benzene ring, they are aminophenyl.



Scheme 31

Anilinovinyl derivatives condense with methyl or methylene reactive groups of heterocycloammonium or ketomethylene, and are useful intermediates in the syntheses of thiazolotrimethine cyanines and thiazolodimethine neutrocyanine. They are prepared according to the following methods:

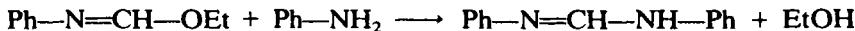
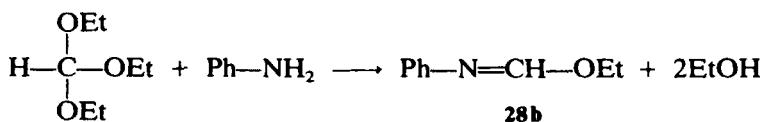
- Melting of a quaternary salt with an arylformamidine (method A, Scheme 32).



28a

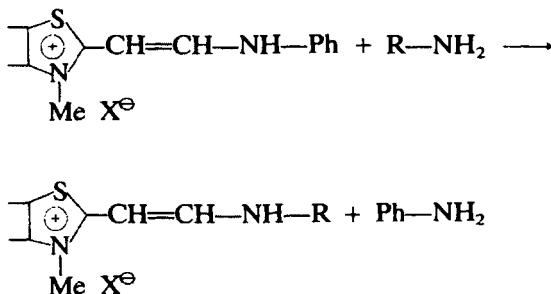
Scheme 32

- Mixing of quaternary salt, aniline, and ethylorthoformate (method B, Scheme 33).



Scheme 33

- Use of ethylisoformanilide (**28b**) (method C).
- Transamination (method D, Scheme 34).



Scheme 34

- Condensation in acetic anhydride involving acetanilidovinyl derivatives (**28a**) (method E, Scheme 32).

The aminophenyl compounds were obtained only by a direct heterocyclization.

### B. Styryl Dyes (Tables 1121A,B,C,D,E,F,G)

Styryl Dyes result from the condensation of benzaldehyde or *p*-dialkylaminobenzaldehyde with quaternary salts of 2-methylthiazole, either in acetic anhydride or in a solvent with a base as catalyst.

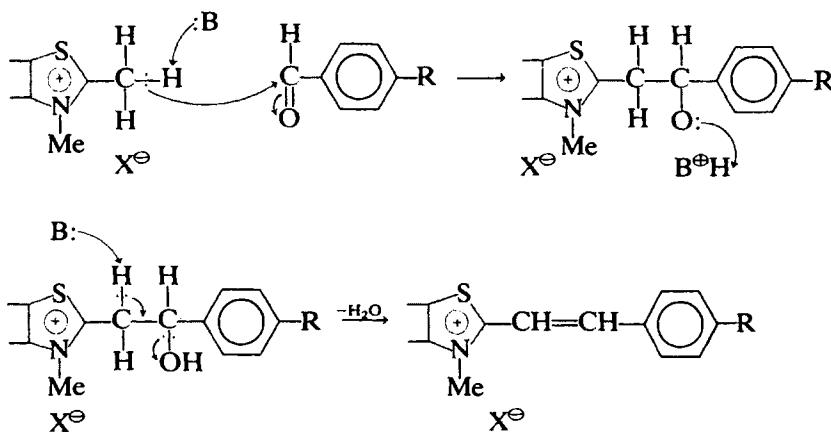
This class of dye has offered in the past two matters of interest. It has been chosen by Mills as the material for demonstrating the mechanism of

the condensation and the role of the anhydrobases issued from quaternary salts by loss of a proton.

It has been used to establish the "basicity" scale of nuclei by means of the deviations calculated from  $\lambda_{\max}$ . This "basicity" has in fact a concrete physical sense, corresponding to the  $pK_a$  of the heterocycloammonium (25, 55); regarding Mills' views about the role of an active allenic intermediate (56), such compound could never be isolated or even identified (49).

Moreover, the specific role of anhydrobase in this condensation is not confirmed, since separated anhydrobases are not reactive toward aldehydes and the condensation can be made in an acid medium.

The reaction probably must involve two steps, aldolization followed by crotonisation, and depends among other factors on the specific reactivity of the aldehyde and of the methyl, which is influenced both by the nature of the substituents on the ring and by the nature of the anion (Scheme 35) (57, 58, 657)



Scheme 35

The stereo aspect of the condensation has been studied for pyridine derivatives, and according to the nature of solvent, either ethanol or acetic anhydride, a *cis* or a *trans* dye could be obtained (59).

A mesophenyl-substituted trimethine cyanine has been identified as a by-product in syntheses leading to styryl dyes; it is also obtained when the anhydrobase reacts with the styryl dye (26).

C. *Dimethine Cyanines* (Tables 1122A,B)

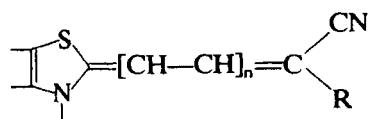
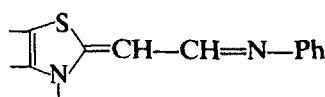
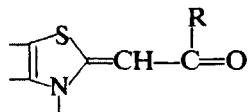
Except in the case of formaldehyde, the electrophilic character of carbon atom of an aliphatic aldehyde is not strong enough to allow its condensation on a CH<sub>3</sub> reactive group. However, such a condensation can occur with an aromatic or pseudoaromatic substance such as benzaldehyde or pyrroloaldehyde, and the  $\lambda_{\text{max}}$  of the resulting dimethine dyes have been used in this last case to obtain the “basicity” scale of various rings (16).

D. *Azastyryl Dyes* (Table 1123)

The azastyryl dyes result from the condensation of 2-formylthiazolium salt either on *N,N*-dimethyl-*p*-phenylene diamine or aromatic nitroso derivative.

E. *Nonionic Mononuclear Compounds* (Tables 121, 122, 123)

Very few compounds of this type have been described and appear in the tables of ketomethylene, anilinovinyl bases, and semicyanines (Scheme 36).



Scheme 36

## 2. Dinuclear Cyanines

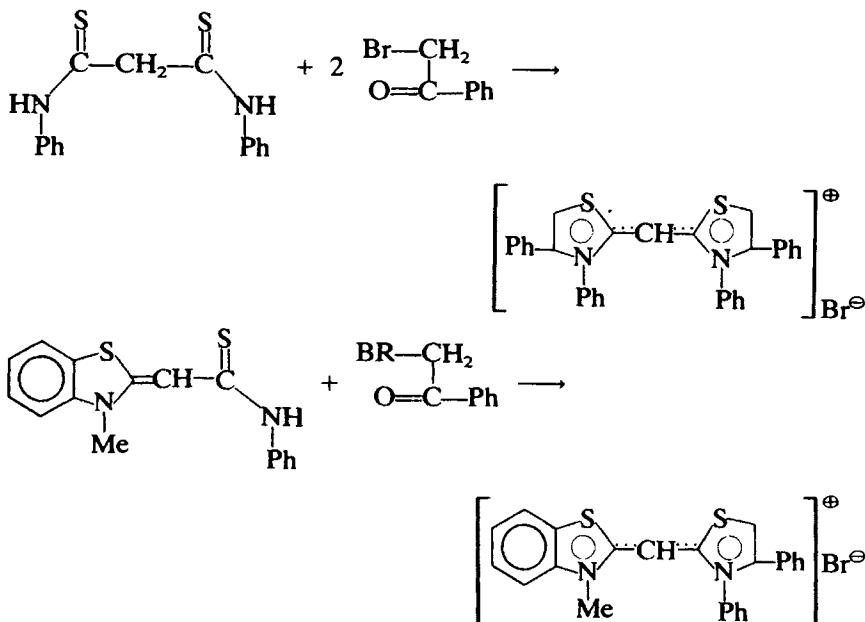
### A. Cationic Dyes

#### a. MONOMETHINE THIAZOLOCYANINES (Table 2111)

The general and historical method of formation of monomethine cyanine, which consists of condensing amyl nitrite on a 2-methyl reactive group in acetic anhydride (method A) does not give any result in the case of thiazolium (60). However, sodium nitrite in acetic acid was used to synthesize symmetrical thiazolocyanines (method B). For an unexplained reason, a 2-substituted halogen in a thiazole ring does not possess the reactivity exhibited in the case of other heterocycloammoniums or in the case of other electrophilic reagents such as  $-SR$ ,  $-SO_3^-$ ,  $-SO_3Me$ , whatever the nature of the ring may be.

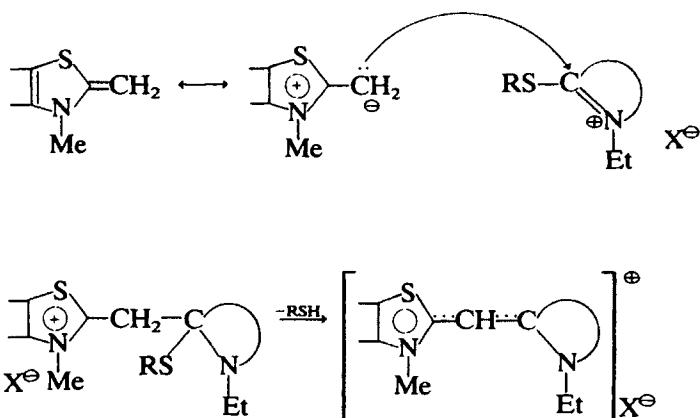
Thiazolomonomethine cyanines result from the condensation of 2 moles of 2-alkylmercaptothiazolium on 1 mole of malonic acid in pyridine (method C) but could not be obtained from this intermediate in acetic anhydride as is the case for other rings (26).

The one-step condensation of phenacyl bromide on dithiomalonanilide (method D) leads to *N*-phenyl thiazolocyanines (Scheme 37).

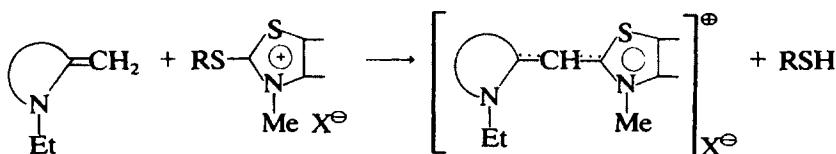


Scheme 37

The unsymmetrical thiazolocyanines (Tables 2112A,B,C,D,E) result from the condensation of an electrophilic reagent such as an heterocyclo-ammonium possessing a substituent leaving group  $-\text{SR}$ ,  $\text{SO}_3^-$ ,  $-\text{SO}_3\text{Me}$ , OR on a reactive methyl group. The reaction can be carried out in the two ways (Schemes 38 and 39). The formation of an anhydروبase constitutes the first step of the reaction. It adds onto the positive carbon atom of the quaternary salt bearing the leaving group, which is eliminated afterwards under the effect of the basic condensation agent. The yields appear to be the same when the anhydروبases are actually isolated, and used in place of the methyl quaternary salts. Any substituents that as carbethoxy are electron withdrawing, and enhance the acidity of thiazolium, facilitate the obtention of anhydروبases. The yields are consequently increased by comparison with an unsubstituted molecule (61, 62).

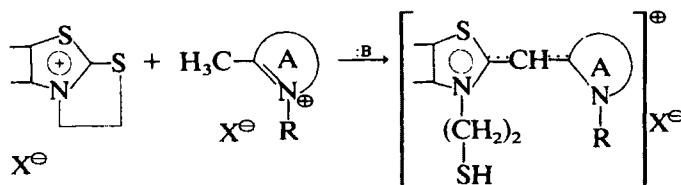


Scheme 38



Scheme 39

The interesting reactions where a free mercapto group is linked to the nitrogen atom of the thiazole (63), after the cleavage of a fused ring, is another illustration of the additive properties of the carbocation (Scheme 40).



Scheme 40

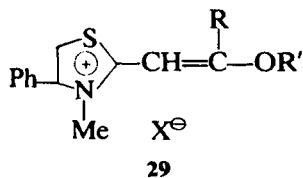
The monomethine cyanines with a methyl group on the chain (Table 2113) are prepared in a basic medium from a 2-alkyl-substituted thiazolium by condensation of an electrophilic reagent.

Few representative dyes of cyanine base (Table 2114) resulting from the condensation of alkylmercapto indolenine on 2-methylthiazolium have been described.

b. TRIMETHINE THIAZOLOCYANINES (Tables 2121A,B,C, 2122A,B,C, 2123A,B, 2124A,B)

**SYNTHESSES WITH O-ESTERS (SYMMETRICAL).** Such functionally symmetrical reagents reacting on quaternary salts in pyridine give generally good results for rings for which the  $pK_a$  is the more acidic; for the less acidic rings of thiazolium, pyridinium, and quinolinium, except in the case of the very reactive ethyl *o*-formate, the yields are much lower. That is why the literature gives very few results furnishing at the same time purity, yield, and precise  $\lambda_{\text{max}}$ , as is the case with alkyl *o*-acetate or *o*-propionate.

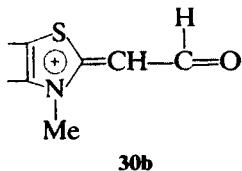
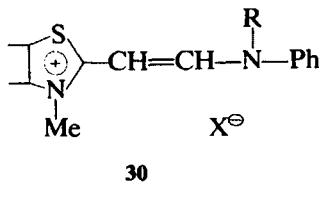
The reason for this relative lack of reactivity of 2-methylthiazolium is probably due to the too-weak nucleophilic character of its carbon-2. For example, any  $\beta$ -alkoxyalcene (**29**) derivatives resulting from the condensation of *o*-ester could never have been isolated, whereas they constitute the essential intermediate step in trimethine syntheses for rings of acidic character (64). However, even if a negative 5-substituent such as ethoxy-carbonyl increases the yield (61) by promoting independently the possible formation of the methylene base, it may be stressed that the presence of this base is not the essential condition of the reaction, since the isolated anhydrobase itself is not reactive toward the *o*-ester (Scheme 41).



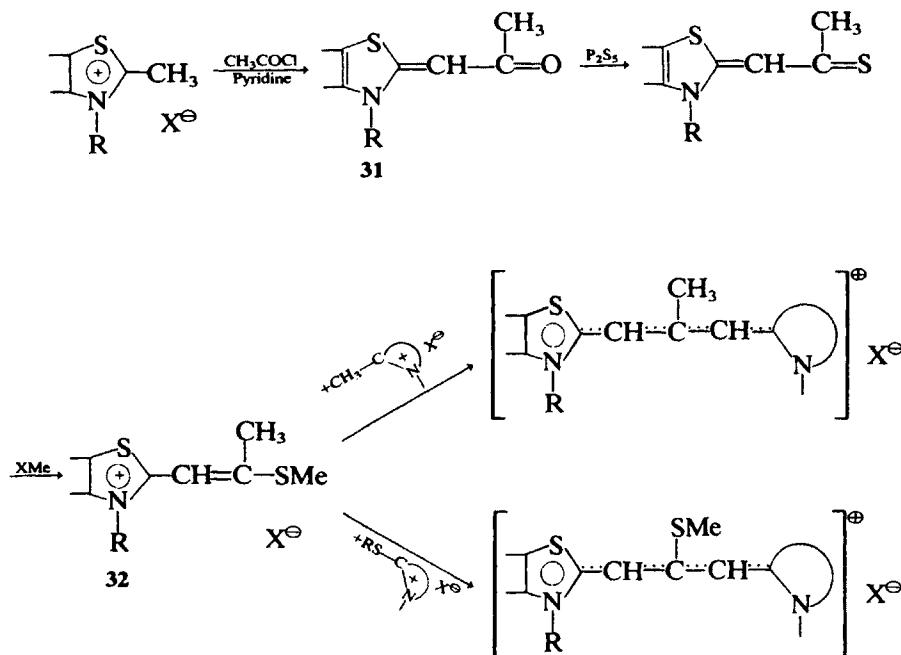
Scheme 41

**OTHER ACCESS ROUTES FOR SYMMETRICAL AND UNSYMMETRICAL THIAZOLO DYES.** Other functionally symmetrical reagents such as diethoxymethylacetate (method C), ethoxymethylenediethylmalonate (method D), diphenyl formamidine, and ethylisoformanilide condense with 2-methylthiazolium to give the trimethine thiazolo dyes.

Anilino vinyl derivatives of thiazolium (**30**, R = H) or acetanilido (**30**, R = COCH<sub>3</sub>), as well as formyl methylene **30b** (methods E-G), give unsymmetrical dyes when condensed with a methyl reactive group of another species (Scheme 42). Mesosubstituted symmetrical or unsymmetrical thiazolocyanines are obtainable via  $\beta$ -alkylmercaptovinyl thiazolium derivatives (**32**) (methods H and I) (Scheme 43).  $\alpha$  or  $\beta$  carbon atoms of the trimethine chain can be substituted by acetyl when a dye is treated with acetic anhydride (method L). The hydrolysis of neocyanines lead to trimethine cyanine by fractional elimination of a componant chain (method K).



Scheme 42



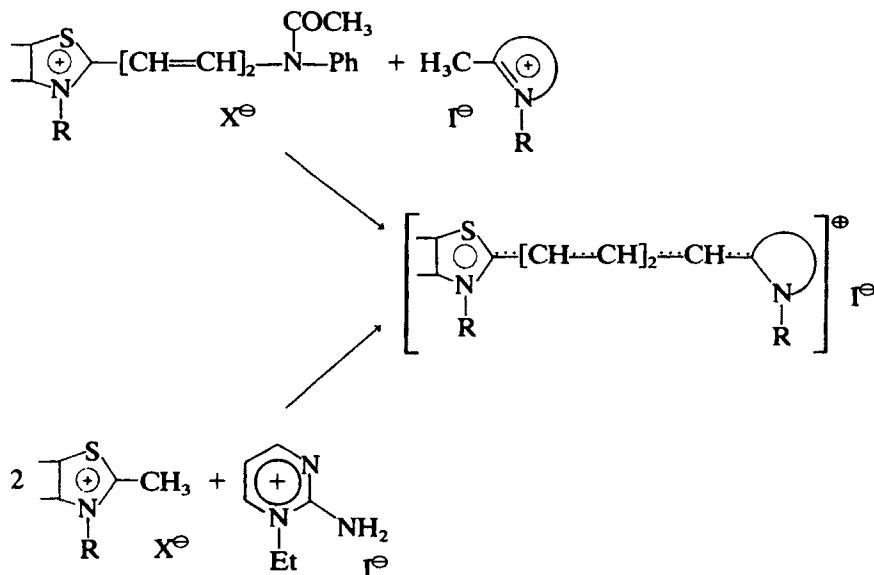
### c. PENTAMETHINE THIAZOLOCYANINES (Table 213)

The three methine carbons of the chain can be provided by 1,3,3-triethoxypropene (method C) or  $\beta$ -anilinoacrolein anil, vinylog of diphenyl formamidine issued from the condensation of aniline on tetraalkyloxyp propane (method A).

Two steps take place in the synthesis, as in the case of trimethine dyes. The first intermediate is a 2-anilinobutadienyl thiazolium, which then reacts with a second mole of 2-methylthiazolium salt or another molecule of a different ring according to the desired product: either a symmetrical or unsymmetrical dye (method B).

The ring fission of pyrimidinium gives good results when this salt is condensed with 2-methylbenzothiazolium; the yield obtained is very poor with 2-methylthiazolium (method D) (545).

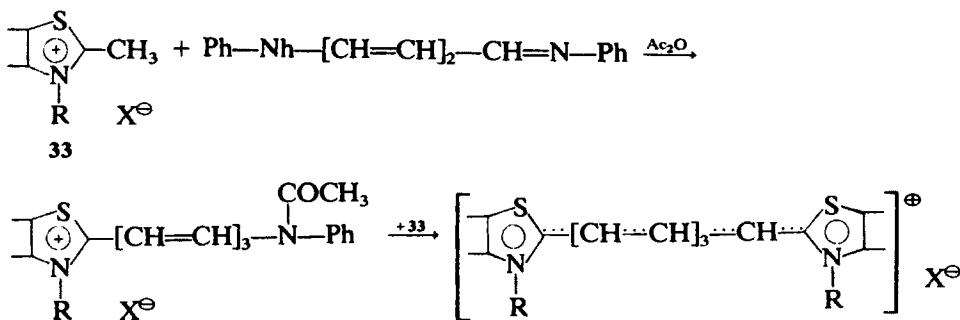
Low yield in pentamethine thiazolocyanine results also from the fission of the pyridine ring in the presence of 2-chloro-3,3-dimethylindolenine (544). Explanations of this unusual reaction have been given (Scheme 44) (67).



Scheme 44

#### d. HEPTAMETHINE THIAZOLOCYANINES (Table 214)

The five carbon atoms of the methine chain are brought by glutaconic aldehyde (method A) or by the dihydrochloride of its dianilide (method B). Both are issued from the fission of the pyridinium ring when a strong electron-attracting substituent group is fixed on the nitrogen atom; the reaction can be performed in one or two steps whether a symmetrical or unsymmetrical dye is concerned, the intermediate being anilino-1,3,5-hexatrienylthiazolium or its acetyl derivative (method C) (Scheme 45).



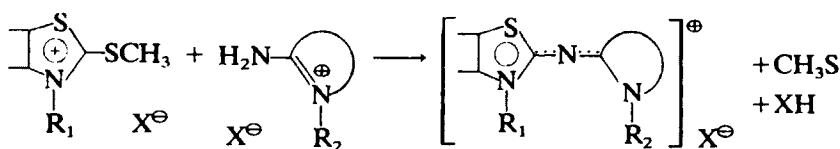
Scheme 45

e. CHAIN-BRIDGED THIAZOLOCYANINES (Tables 2151,  
2152A,B,C,D)

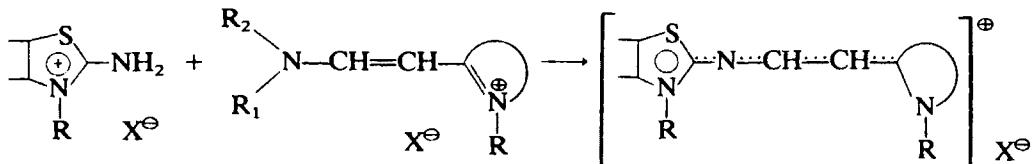
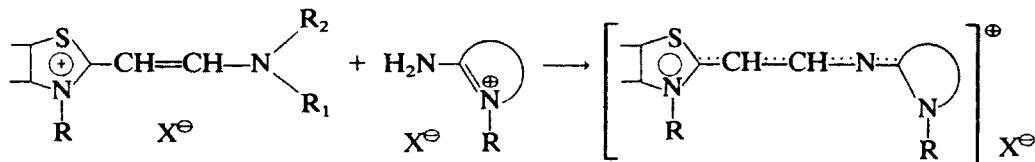
The methine chain is obtained by reacting ethyl *o*-formate (method A) or ethylisoformanilide (method B) with a bis quaternary salt of bis-(2-thiazolyl)butane. Concerning dyes with fused thiazolo rings: pyrrolo[2,1b]thiazole, thiazolo[2,3a]indole, thiazolo[2,3c]1,4-benzoxazaine, the  $\alpha$  carbon directly linked to the carbon 2 of the thiazole ring is also responsible for the classical syntheses giving trimethine or pentamethine dyes.

f. AZACYANINES

MONOAZATHIAZOLOCYANINES (Tables 2171A,B). The same reactive intermediates and the same conditions as in the case of thiazolomethine dyes are used in the synthesis of this class of dyes, except that the  $\text{CH}_3$  group is replaced by a  $-\text{NH}$  group (Schemes 46 and 47). No  $\beta$ -azatrimethine thiazolocyanine has been described, in spite of an access method applied with success to other rings (65).



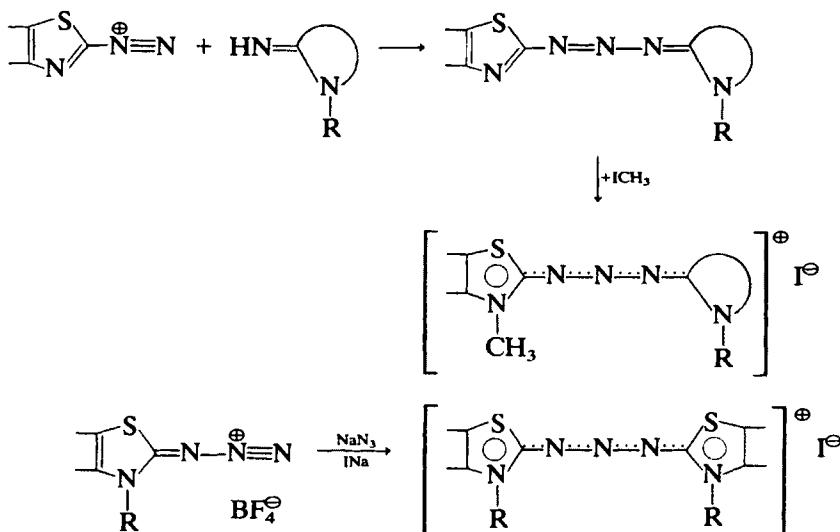
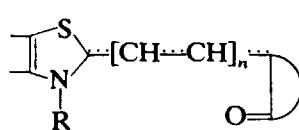
Scheme 46



Scheme 47

$\alpha,\gamma$ -DIAZATRIMETHINE THIAZOLOCYANINES (Table 2172).

TRIAZATHIAZOLOCYANINES (Table 2173). Triazathiazolocyanines are obtained by the methods in Scheme 48.

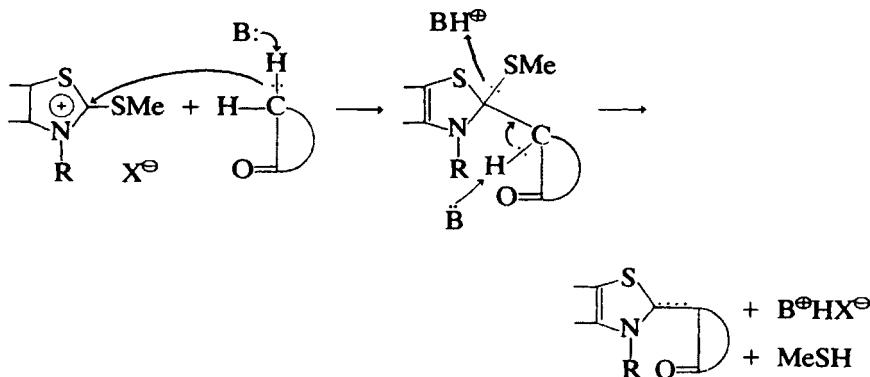
B. *Neutrocyanines* (or Merocyanines) (Scheme 49)

$$n = 0, 2, 4, 6$$

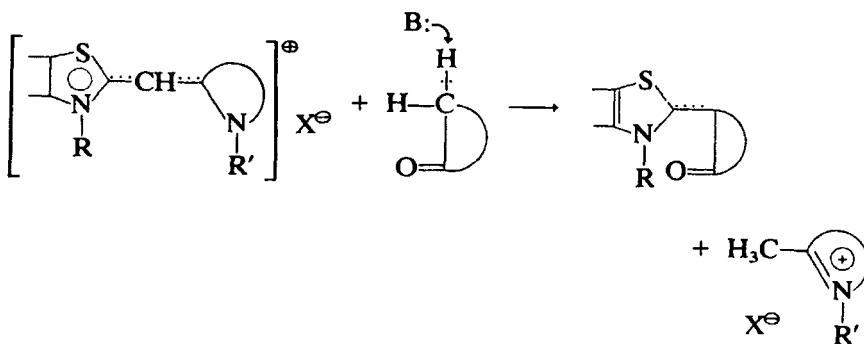
Scheme 49

## a. ZEROMETHINE CYANINES (Table 221)

Zeromethine cyanines result from the reaction of the electrophilic reagent thiazolium salt having a leaving group  $-\text{SR}$ ,  $-\text{SO}_3^-$ , or  $-\text{SO}_3\text{Me}$  on carbon-2 with any ketomethylene compounds that are nucleophilic reagents (Scheme 50). The same mechanism is operating when a ketomethylene compound react on an unsymmetrical monomethine thiazolocyanine with elimination of the hetero ring, which is the more basic (Scheme 51) (26, 67).



Scheme 50

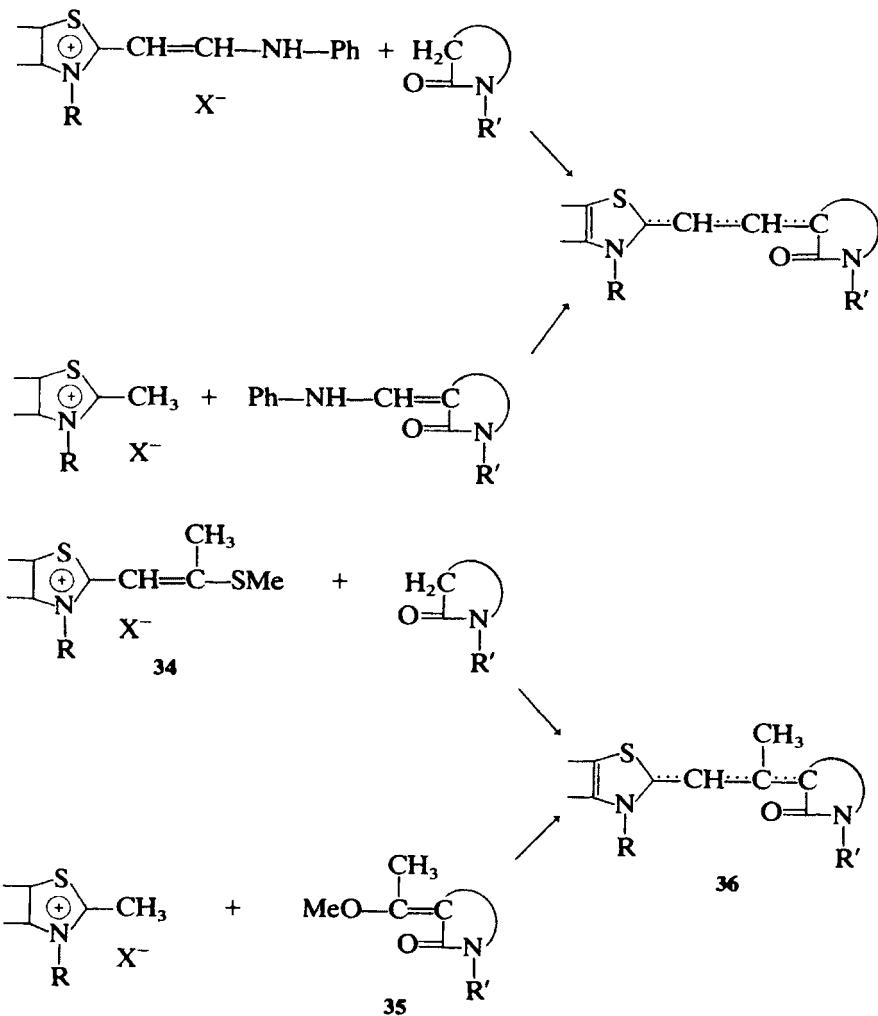


Scheme 51

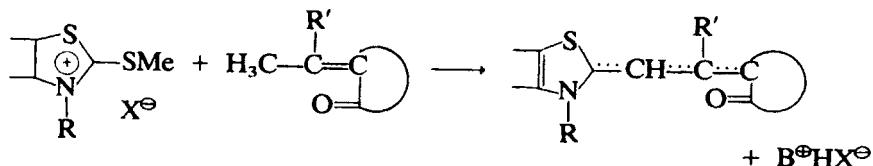
### b. DIMETHINE NEUTROCYANINES (Tables 222A,B,C,D,E)

Two different access routes are used, whether the leaving group is carried on thiazolium derivatives such as anilinovinyl (method A), acetanilidovinyl (method B), formyl methylene, or thioformylmethylene or on the ketomethylene compound (method C). The use of acid anhydride together with pyridine has been patented (method E).

The choice of the intermediate depends on the reactivity of the ring. For a pyrazolone (**34**) rather than **35** is used; it is the reverse for rhodanine (Schemes 52 and 53).

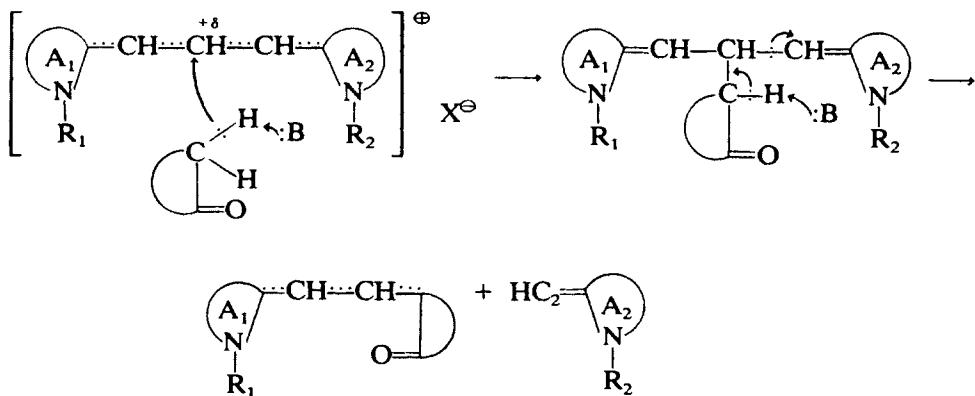


Scheme 52



Scheme 53

The electronic structure of a trimethine asymmetrical cyanine, controls the attack of a ketomethylene (Scheme 54). There is a condensation of the nucleophilic carbon on the electrophilic central carbon atom of the methine chain, leading to a neutrodimethine cyanine and simultaneously elimination of the more basic nucleus.

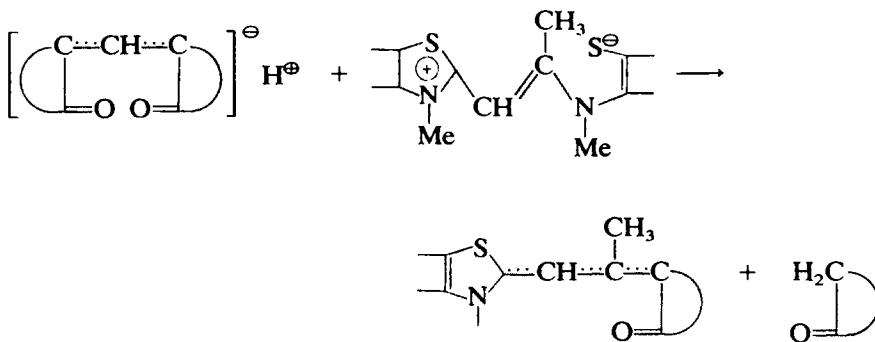


Scheme 54

For example, when *N*-ethylrhodanine is condensed with (quinoline-4)(4-methylthiazole-2)trimethine cyanine or (pyridine-2)(4-methylthiazole-2)trimethine cyanine, the yields obtained for neutrothiazolodimethine dye are 25 and 75%, respectively (53).

The easier elimination of pyridine compared to quinoline-4 may be related to the *pK<sub>a</sub>* value of 4-methylthiazole, which is between those of lepidine and 2-picoline (25, 55). This reaction explains also why a neutrodimethine cyanine is obtained with such good yields when reacting together a quaternary salt, ketomethylene, and *o*-ester in a basic medium. As the reaction proceeds, the trimethine cyanine is attacked by the ketomethylene. The resulting 2-methyl quaternary salt is transformed into trimethine cyanine, consuming the totality of the ketomethylene (1, p. 512; 661). The mesosubstituted neutrodimethine cyanine is practically pure.

The condensation of a thiazolium with an oxonol dye in a basic medium is another example of the combination of electrophilic and nucleophilic reagents (Scheme 55). With a nonopening ring, the obtained neutrodimethine cyanine is not mesosubstituted (68).

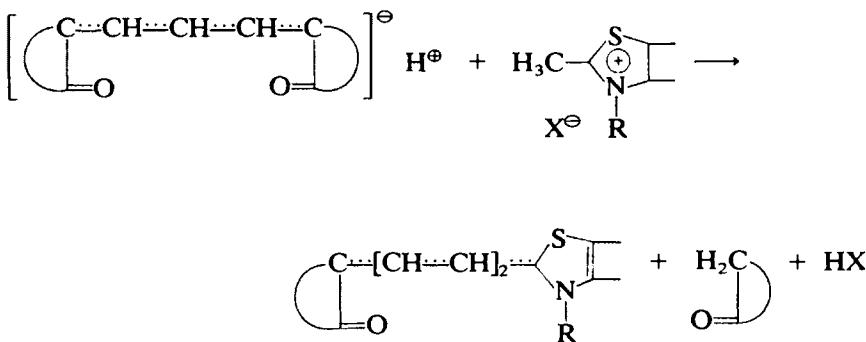


Scheme 55

### c. TETRAMETHINE NEUTROCYANINES (Table 223)

Two access routes are also used with the vinylog derivatives of previous compounds, for example, either 2-(4-anilinobutadienyl)thiazolium with a ketomethylene, or 4-(4-anilinobutadienyldiene)ketomethylene with 2-methylthiazolium.

The cleavage of thiazolopentamethine cyanine and also neocyanine results in a tetramethine neutrocyanine together with neutrodimethine cyanine (26). 2-Methylthiazolium reacts with a trimethine oxonol dye and gives a neutrotetramethine dye (Scheme 56) (26).



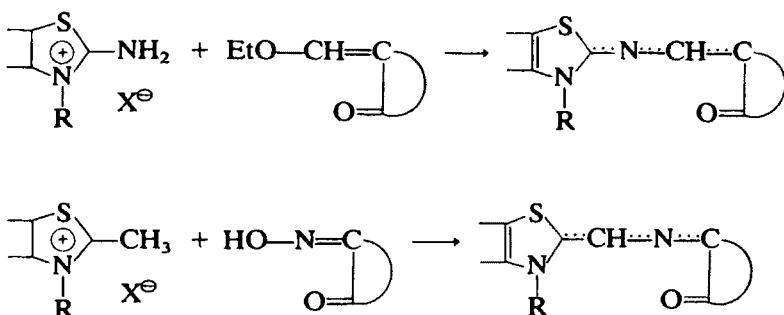
Scheme 56

### d. HEXAMETHINE NEUTROCYANINES (Table 224)

Hexamethine neutrocyanines are obtained by heating 2-(6-acetanilido-1,3,5-hexatrienyl)-4-aryltiazolium with ketomethylene and sodium acetate in acetic anhydride (69).

## e. AZANEUTROCYANINES (Table 225)

According to the position of nitrogen, the leaving substituent may be brought either by ketomethylene (5-ethoxymethylene) (method A) reacting on an aminothiazolium or by an amino derivative of this ketomethylene reacting on a 2-methylthiazolium (method B) (Scheme 57).



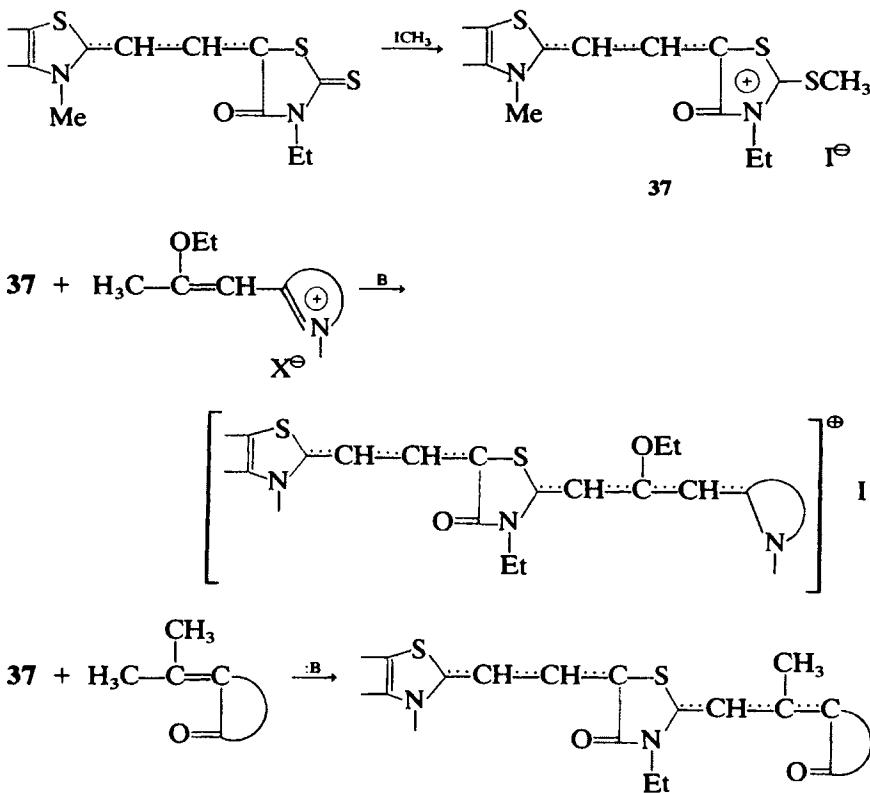
Scheme 57

## 3. Trinuclear Dyes

Among the dyes described in patents or literature listed in Tables 311, 312A,B,C,D, 313, 315A,B, 322A,B,C, the two following classes appear the more important, both from a theoretical and practical point of view.

## A. Rhodacyanines (Tables 3141A,B,C, 3142,A,B)

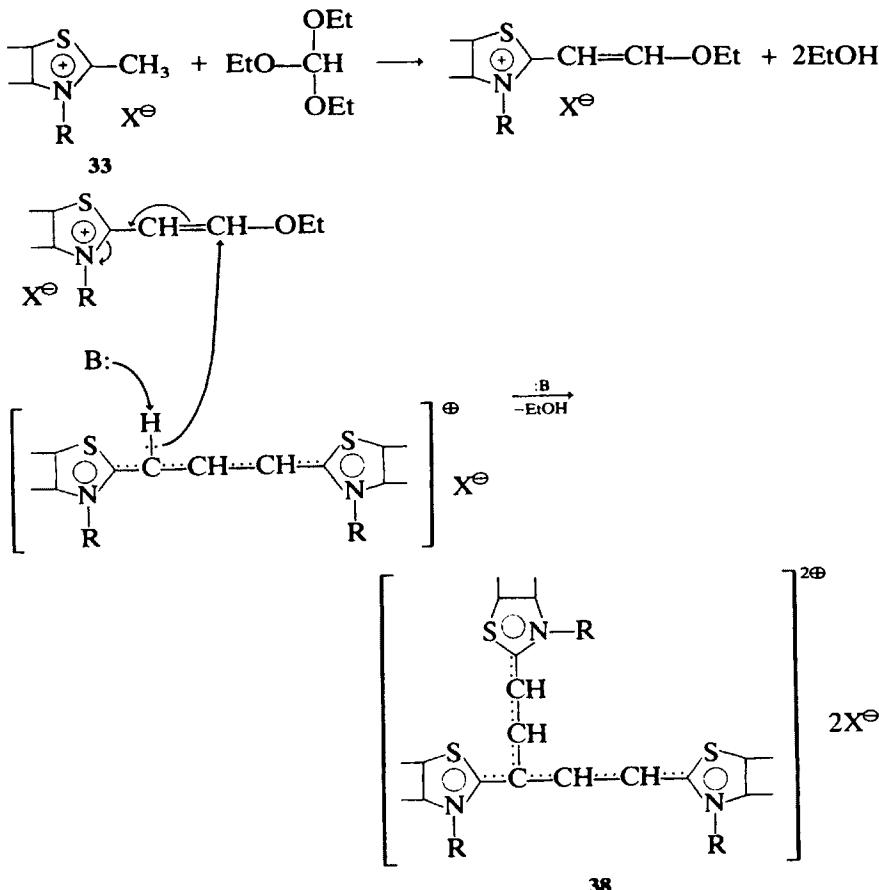
These dyes possess two independent chromophoric chains of even methine (neutro) and uneven methine (cyanine) fixed on a central ketomethylene nucleus. The methylene reactive group is first used for the neutrocyanine synthesis in position 5, the quaternization of which can ensure a subsequent polymethylene synthesis in position 2 of a cationic dye by ordinary means (Scheme 58). As indicated, this quaternized neutrocyanine (37) may as well give another neutrocyanine.



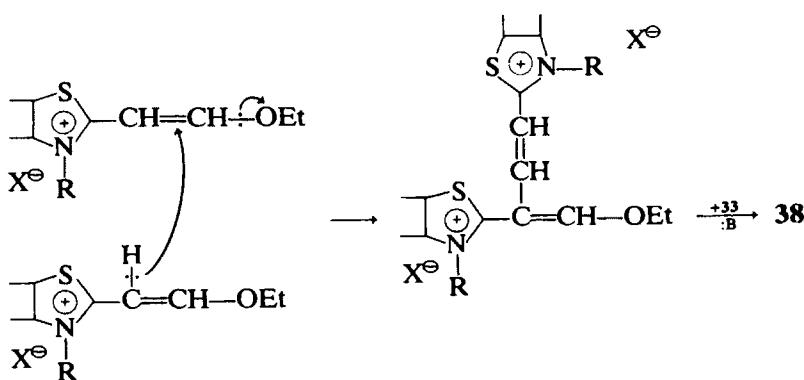
Scheme 58

### B. Neocyanines (Tables 321A,B)

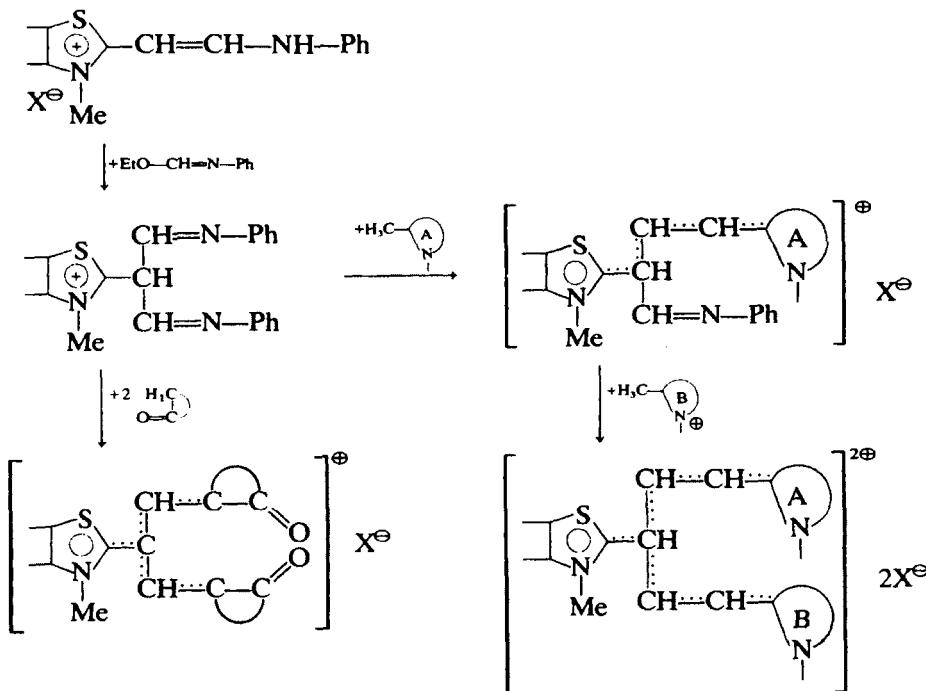
When a reactive methyl group of a heterocycloammonium reacts with ethyl *o*-formate in a solvent, the result of the condensation depends both on the basicity of the quaternary salt and on the nature of the solvent. For example, 2-methylbenzothiazolium in cresol gives chiefly neocyanine, whereas the same reaction performed in pyridine gives trimethine cyanine with a nearly quantitative yield. In the case of 2,4-dimethylthiazolium and cresol, ethyl-*o*-formate is not reactive; if pyridine is used, the reaction gives a low yield of trimethine thiazolocyanine plus neocyanine. Reducing the basicity of the ring by negative substituent as carbethoxy groups in positions 4 and 5 increases the yield of trimethine thiazolocyanine, simultaneously with the disappearance of neocyanine. But the interpretation of this reaction as far as the relative ratio of the two dyes is concerned should take into account the fact that neocyanines derived from benzothiazole decompose readily in a pyridine alcohol mixture,



Scheme 59



Scheme 60



Scheme 61

giving trimethine cyanines. So if in some experiments no neocyanine was found does not mean that it is not produced in the course of the reaction, but only that it is too unstable in the medium. The formation of neocyanines can be interpreted by the two following probable mechanisms, both starting from the very reactive  $\beta$ -alkoxyalcene derivative (64) and which are supported by the electronic state on the methine chain:

1. This derivative condenses either on itself (64) or on the anhydrobase, giving the trimethine dye. Indeed, the nucleophilic  $\alpha$ -carbon of the dye—the proton is labile and can be replaced (70, 71)—is liable to add onto the electrophilic  $\beta$ -carbon of the alcene derivative. The neocyanine results from elimination of a molecule of ethanol.
2. Two moles of  $\beta$ -alkoxyalcene can condense on each other by means of their  $\alpha$ - and  $\beta$ -carbon atoms. The resulting intermediate reacts on the anhydrobase by elimination of a molecule of ethanol resulting in a neocyanine formation (Schemes 59 and 60). Both monoanilino and bis-anilino derivatives resulting from the condensation of dimethylformamide have been isolated. They are capable of furnishing various condensations on either ketomethylene or another reactive nucleus (Scheme 61).

#### 4. Tetrานuclear Dyes

Examples of different kinds of dyes obtained by usual synthesis starting from bis-thiazolium salts are listed in Tables 41A,B,C, 42, 43A,B,C, 44.

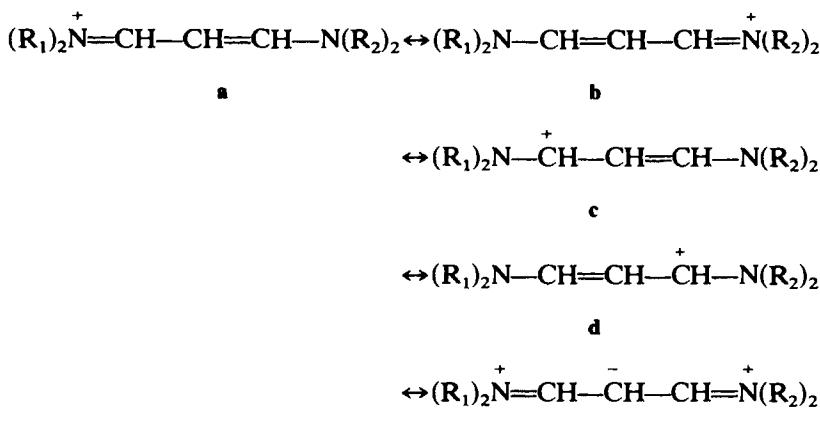
### IV. STRUCTURE AND COLOR

Since the very beginning of chemistry, many efforts have been devoted to find out basic relationships between the characteristics of absorption spectra and the molecular structure of dyes.

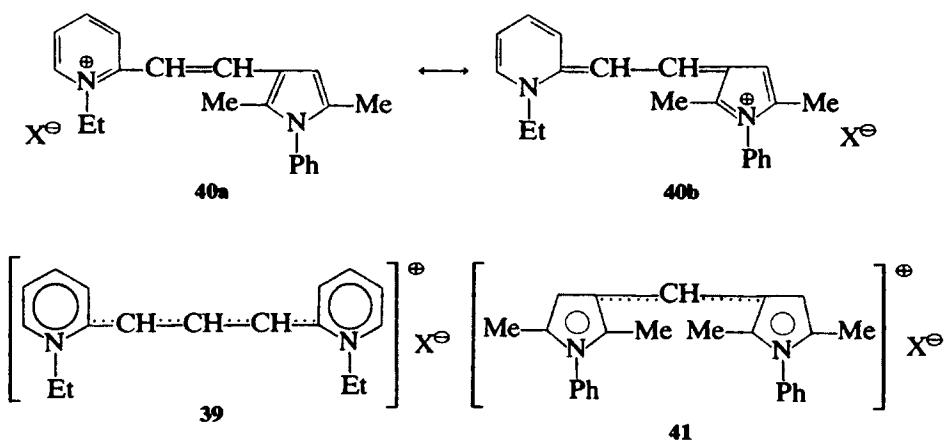
Among the authors whose contribution has been important in the past, either by their experimental work or by their theoretical approach, the names of Koenig, Hamer, Fischer, Brooker, Kiprianov, Sklar, Schwarzenbach, Eistert, Dimroth, and Platt are well familiar to scientists having an interest in the field of methine dyes, as well as more recently those of Kuhn, Wizinger, Scheibe, Hünig, Van Dormael, Dähne, and Fabian.

#### 1. Resonance Theory and Concept of "Basicity"

Most of the qualitative relationships between color and structure of methine dyes based on the resonance theory were established independently during the 1940's by Brooker and coworkers (16, 72-74) and by Kiprianov (75-78), and specific application to thiazolo dyes appeared later with the studies of Knott (79) and Rout (80-84). In this approach, the absorptions of dyes belonging to amidinium ionic system are conveyed by a group of contributing structures resulting from the different ways of localization of the  $2n$   $\pi$  electrons on the  $2n-1$  atoms of the chromophoric cationic chain, rather than by a single formula:



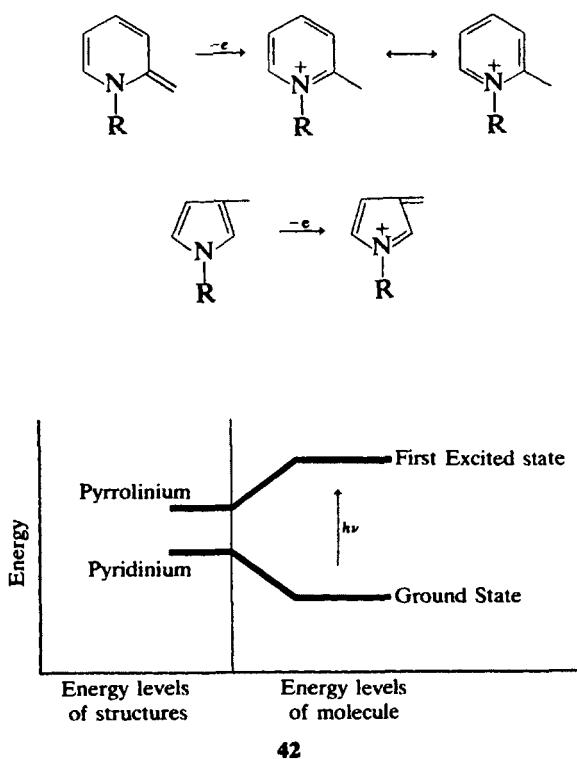
All the structures (a to e) contribute to the dye molecule, both in the ground and excited states. From their interaction, it results that in the ground state, there will be relatively greater contribution from the low-energy extreme structures; higher-energy intermediate structures play a greater part in the excited state. The effect of the absorption of light would be related to the change of the relative contribution of the structures involved in the resonance hybrid in favor of those of higher energy resulting in some displacement of positive charge away from the nitrogen atom to the carbons of the conjugated chain (Scheme 62) (5, 72).



Scheme 62

Dye **40a** is considered by Brooker as a “structural hybrid” of symmetrical dyes **39** and **41** for the reason that five carbon atoms separate the two nitrogen atoms of the terminal groups. If the pyridine and pyrrole rings were to possess the same energy, the  $\lambda_{\max}$  of unsymmetrical dye should be the arithmetic mean of the  $\lambda_{\max}$  of both symmetrical dyes: 562 and 448 nm. The observed  $\lambda_{\max}$ , 426 nm, lies at shorter wavelength, and the difference called “deviation” is attributed to the difference in the energies of the extreme structures, related to the electron-donating abilities or “basicities” of the terminal groups (Scheme 63) (5, 72). According to resonance theory, the pyridinium structure involving two possible charged structures must be more important or stabilized than pyrrolium, implying just one positive design. A pyridine nucleus accommodating more easily a positive charge is more “basic” than pyrrole. The **40b** arrangement is not stabilized by resonance to the same extent as the

pyridinium one, and its energy will be higher as indicated by Brooker (5). Associating all the heterocyclic nuclei able to form methine dyes with either a pyrrole or *p*-dimethylamino phenyl group, Brooker obtained a scale of relative increasing "basicities" in which indole is the least basic and benzimidazole the most basic nucleus, thiazole rings (4-H, 4-Me, 4-Ph substituted) taking place at the end of this table between quinoline and pyridine rings (72, 16). A similar classification was made for the acidic electron-accepting terminal groups, that is, ketomethylene compounds in merocyanine series (72).



42

Scheme 63

"Vinylous" shift of about 100 nm in symmetrical dyes, convergent and nonconvergent series, and solvent effects, have also been successfully interpreted by Brooker's and Kiprianov's conceptions.

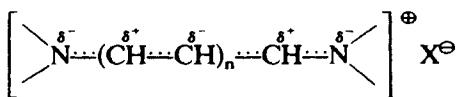
## 2. Limit of the “Basicity” Concept

Deviation includes, in fact, the summation of steric and electronic effects, and basicity is somewhat a useful predictor for properties of complex dyes (solvent sensitivity, isomeric forms of trinuclear dyes) and gives also semiquantitative data for color structure relation:  $\lambda_{\max}$ , (atomic) charge density, redox properties.

But however useful this concept may have been, the formalism of resonance on which it is based is not well adapted to the quantitative electronic description of polymethine dyes. The choice of the limiting structures and the estimations of their relative weight and energy appear arbitrary in many cases, and moreover, according to Moffit (85), the ready acceptance with which qualitative symbolism has been used to operate the splitting of the extreme energy levels in the resonance hybrid is liable to propagate an undesirable misunderstanding (e.g., 42). Equally, no proof has been given concerning this assertion that the absorption of light should be accompanied by some movement of the positive charge away from the nitrogen onto the carbon of the conjugated chain (5). Strict interpretation of the resonance model for an excited-state dye is only partially paralleled by molecular orbital results. Although positive  $\pi$  charge does accumulate on the chain methine carbons as a result of excitation, excess electrons migrating toward the ends of the chromophore do not appreciably change the charge on the nitrogen atoms, but rather changes the charges on other atoms, for example, carbons of the terminal groups. A more serious objection results from the observation that when rings other than pyrrole in dimethine cyanines or dimethylaminophenyl in styryl dyes are taken as reference to establish or verify the scale of “basicity,” the deviations so obtained are dispersed and lead to another order of “basicity” that cannot be interpreted. Striking examples have been given by the use of pyranothiazole and thiapyranothiazole nuclei as reference (86, 87). On the other hand, Brooker’s concept of basicity implying a strictly one-electron process could not be confirmed by the experimental determination of polarographic reduction potentials of the quaternary salts from which dyes are issued (25). On the contrary, some correlation exists between “deviations” and properties deriving from a two-electron process, for example, the linear relationship between the  $pK_a$  of quaternary salts (25) or the  $pK_a$  of symmetrical trimethine cyanines (658) with deviation attached to the nuclei. The deviation has also been related to the Hammett’s  $\sigma$  coefficient (88, 89).

### 3. Modern Conception

The theory of resonance and Valence Bond method appear now inadequate to give an entirely satisfactory structural and energetic description of a methine dye molecule and to predict either its peculiarities or physicochemical characteristics. Following the pioneering theoretical work of Forster (90), Herzfeld and Sklar (91), and of Mulliken (92), Kuhn (6-8) used the free-electron model to calculate the molecular orbital structure of polymethine cation dyes. The conclusion is that all such dyes possess the same conjugated system in which the net positive charge is delocalized, being distributed alternatively into positive and negative partial charges on the carbons of the chain (Scheme 64).



Scheme 64

The nitrogen ending atoms being more electronegative possess a  $\pi$  electronic density greater than that of the carbon atoms directly linked to them.

Finally, Huckel's method allowed the calculation of transition energies (93, 95, 96), and as the access to computers of large capacity becomes more common, the Pariser-Parr-Pople type of treatment within the  $\pi$  framework (11, 97-99) or CNDO in all-valence electron methods (100, 101) allows the predictions of not only  $\lambda_{\max}$ , but also the influence of geometrical isomerism on full spectra (666), the energy for each transition, its oscillator strength, polarization vectors, and other physicochemical properties such as dipole moment, basic strength, oxidation, and reduction polarographic potentials, as well as  $^{13}\text{C}$  (10) and proton nuclear magnetic resonance (94).

As a consequence of the alternative distribution of an even number ( $2n$ )  $\pi$  electrons on an odd number ( $2n - 1$ ) carbon atoms, centers of the methine chain susceptible to nucleophilic attack are effectively the even carbons atoms starting from nitrogen, as it has been proven experimentally (103), particularly with a ketomethylene giving a neutrocyanine compound (53, 67).

The charge distribution may be symmetrical when the system possesses similar nitrogen atoms and identical or unsymmetrical nuclei approaching more or less a polyene-like bond state when the nuclei are of different

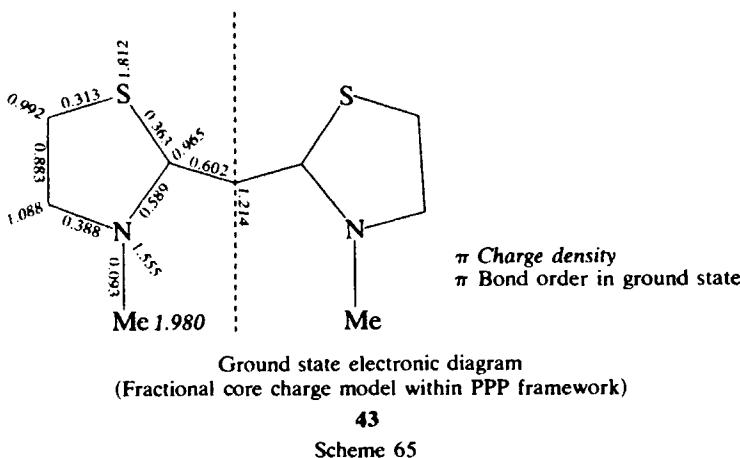
nature. Accompanying the alternative charge distribution, it has been established that the NMR chemical shift  $\tau$  of the methine protons presents corresponding strong alternation (102), and equalization of bond length has been confirmed by X-ray structural analysis (104).

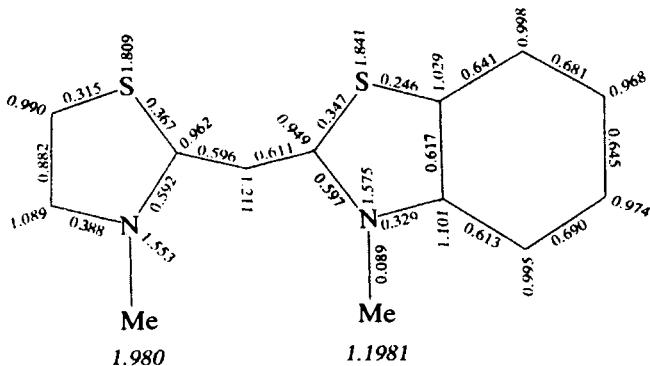
Likewise, quantum mechanical calculation succeeds in giving a theoretical explanation of some facts that the resonance theory could not explain, for example, why bis(pyridine-2)monomethine cyanine and bis(pyridine-4)monomethine cyanine possess the same lowest energy transition contrary to the 2,2'- and 2,4'-quinoline monomethine dyes, together with a molecular coefficient extinction lower than that of the 4,4'-quinoline dye (11). Calculation shows also that there is no theoretical reason for observing a relationship between  $\lambda_{\max}$  and  $pK_a$  in a large series of dyes with different nuclei as it has been postulated, even if limited observations and calculations in short homogeneous series could lead to this conclusion (105).

The ground-state electronic diagrams of some thiazolo dyes have been calculated with the use of theoretical model of fractional core charge model applied to PPP method (659).

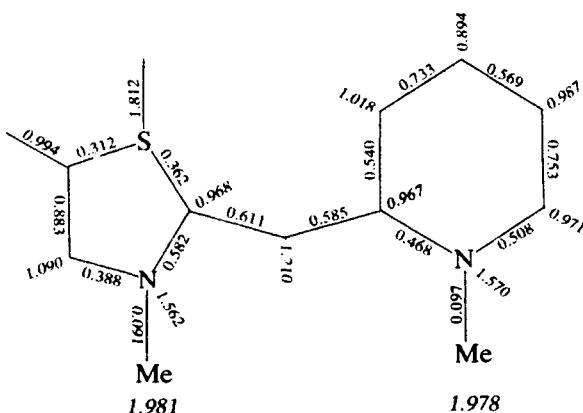
The correspondence between calculated and experimental values for the  $\lambda_{\max}$  of the monomethine dyes, 404 nm for 410 nm, 404 nm for 412 nm, are satisfactory but this does not imply that **43** and **44** possess the same first ionization potential and the same electronic affinity simultaneously.

Considering Highest Occupied Molecular Orbital and Lowest Unoccupied Molecular Orbital (providing that solvation energies are equal) **44** might be a better reducer than **43** (Schemes 65–68).

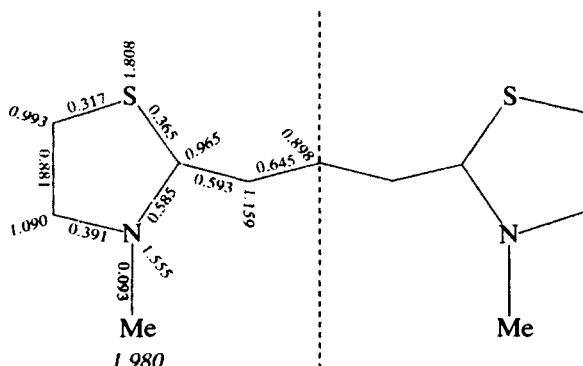




Scheme 66



Scheme 67



Scheme 68

Calculation can also explain why in some thiazole dyes vinylene shift of the first two homologs is larger than the shift between higher members of the series, and also why wavelengths of absorption of nonsymmetrical dyes as calculated by the mean value rule differ from experimental data (667). This deviation is caused by an interannular no-bond SS-interaction in the monomethine ion.

The remarkable solvatochromism found in neutrocyanines was extensively studied. It has been the subject of a long and severe controversy as to its origin (106).

It seems now established by NMR spectroscopic investigations that a change can take place in electronic structures and atomic configuration of the dyes depending on the polarity of the solvent. Parameters describing the transition from one single bond to more double bond character vary according to the nature of the solvent (107).

The relations between structure and electrochemical potential are an important aspect in the study of dyes, since effective sensitizers require both the correct absorption wavelength and suitable electrochemical potentials.

Although the reduction process is not always a reversible one, oxidation and reduction potential values can be sometimes related to the Hückel energies of the highest and lowest filled molecular orbital of the dye (108).

Investigations on thiazolodyes in the infrared region 1600 to 1400  $\text{cm}^{-1}$  have shown that each vinylog gives rise to a characteristic pattern of resonant conjugated stretching modes, exhibiting a low frequency shift as the conjugated length increases (109).

#### 4. Absorption and Substituents

##### A. *Influence of Substituents in the Nuclei*

Whatever their nature may be, phenyl or alkyl, the substituents of the thiazole ring in position 4 or 5 give a bathochromic shift (110, 111) of the absorption of a symmetrical trimethine thiazolocyanine compared to an unsubstituted dye. For a given substituent, this shift is greater for position 5 than for 4 (112).

The "basicity" of a 4-phenyl-substituted thiazole is less than the corresponding methyl-substituted thiazole (16) and the  $pK_a$  values of quaternary salts are in the same order (25).

Several hypotheses have been advanced in order to explain these results. Any substituent in position 4 produces a twist of the molecule resulting from a steric hindrance between this substituent and the group fixed on the nitrogen atom (113, 114).

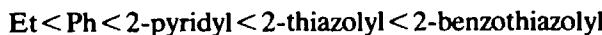
The delocalization could be higher through the sulfur atom in the case of a substituent in position 5 (112).

Unquaternized dyes, substituted by styryl groups in positions 4 and 5, have been used to prevent steric hindrance (115).

Electron-donating or -withdrawing properties of a substituent on the 4 and 5 positions have also been used in order to modulate the "basicity" in the hope to observe either hypsochromic or bathochromic shift (110).

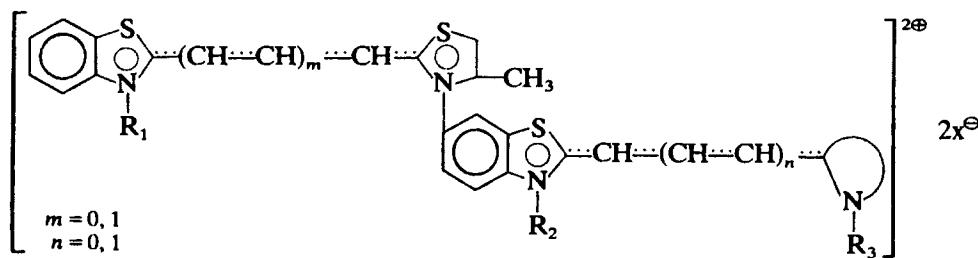
In every case a bathochromic shift that is greater for asymmetrical than for symmetrical dyes has only been observed.

Until now, no final conclusion seems to have been reached as to the origin and amplitude of the phenomenon. The nature of the alkyl group fixed on the nitrogen atoms does not influence the position of  $\lambda_{\text{max}}$ , but substituents of different kinds produce, for trimethine thiazolocyanine as well as styryl derivatives (116), a bathochromic shift in the order:



whereas the reverse effect is observed for neutrorhodathiazolocyanine, the same substituents giving an increasing hypsochromic shift (117).

In tetranuclear dyes such as in Scheme 69 with two independent chromophoric chains, providing the chains are of different length, the absorptions maxima lie at the same  $\lambda$  as each of the component cyanine; but chains of equal lengths produce a shift of the two  $\lambda$ , one toward longer



Scheme 69

wavelength, the other toward shorter wavelength relative to the parent dye (118).

### B. Substituents on the Chain

An alkyl group in the chain of a monomethine thiazolocyanine prevents the molecule from being planar. It gives a bathochromic  $\lambda_{\max}$  shift of 40 nm and a decrease of the oscillator strength (26), as is the case for other methine dyes.

The  $\lambda_{\max}$  of mesomethyl-substituted trimethine thiazolo dyes is shifted by 30 nm toward shorter wavelength. The shift is only of 20 to 25 nm for an ethyl group, whereas phenyl has no effect.

According to the nature of nuclei in the case of asymmetrical dyes, it has been shown that the influence of substituents in the meso position is neither identical nor in the same direction.

The meso carbon atom should present a carbenium ( $C^{6+}$ ) structure with a low  $\pi$  electron density in the ground state, in the excited state this carbon possesses the carbeniate structure ( $C^{6-}$ ) with a high  $\pi$  electron density (119). An electron-donating group in such a position should stabilize the ground state and raise the excited state to the highest level; hypsochromic shift results as a whole.

Any electron-attracting group,  $-NO_2$ ,  $-CN$ ,  $-Ac$ , or  $-CO_2Et$ , on the  $\alpha$ -carbon of the methine chain of a thiazole dye gives a bathochromic shift (120). A reverse effect is observed for an alkoxy group.

Color and redox properties of a dye are related to the two molecular orbitals of  $\epsilon_{LV}$  and  $\epsilon_{HF}^*$ . They are both sensitive to the nature and position of the substituent. A perturbation of both orbitals occurs generally, but in some cases the effect is much more important for one of them.

The  $\lambda_{\max}$  of bridged dyes such as thiazoloindole symmetrical and unsymmetrical trimethine dyes are shifted toward shorter wavelength compared to the corresponding *N*-phenylthiazolocyanine. This hypsochromic shift corresponds to an electron-donating substituent in the  $\alpha$ -position of the chain when steric hindrance is absent (121).

A rigidized molecule obtained when the two  $\alpha$ -carbons of the trimethine chain are linked by a dimethylene bridge cannot be planar. According to the resonance concept, its "stability" increases as a bathochromic effect of 41 nm is observed (122). The  $\lambda_{\max}$  of the bistyryl dye obtained by the substitution of the  $\beta$ -proton in the chain of a styryl dye by a dialkylamino group is nearly the same as for the parent styryl dye (123).

\*  $\epsilon_{LV}$  = energy of the lowest vacant orbital;  $\epsilon_{HF}$  = energy of the highest filled orbital.

### C. Nitrogen Atoms Replacing Methine Groups of the Chain

A great number of monoaza or polyaza, either symmetrical or unsymmetrical, mono trimethine thiazolocyanines have been synthesized in order to verify or to obtain semiempirical rules, more or less based on the resonance theory, concerning the relation between the color of a thiazolo dye and the number and place of nitrogen atoms in the chromophoric chain. For example, Forster's rule applies to ionic dyes and stipulates that the  $\lambda_{\max}$  will increase with the decreasing tendency of chromophoric atoms lying between the two auxochromes to take up the characteristic charges (90).

Knott's rule concerns the importance of the place of the nitrogen atom replacing a methine carbon in the conjugated chain; when the atom is separated from the active auxochromic atoms by an odd number of conjugated atoms, the shift is bathochromic. It is hypsochromic when there is an even number. The importance of the shift could establish a measure of  $\pm M$  effect of various heterocyclic nuclei (79, 124). Many papers have been published, and examples have been given to verify these rules (79-84).

## V. USES OF DYES

### 1. Photography

The use of sensitizing dyes in photography has been the subject of many studies and constitutes still now, one of the most studied areas in specialized periodic publications (125, 126) or in textbooks (88, 127). It can be ascertained that one hundred years after Vogel's discovery of spectral sensitization, the basic mechanisms of action of dyes on their silver halide support still remain not fully understood. However, the theoretical reasons explaining why among many other dye families practically only cyanine methine dyes appear to be spectral sensitizers (128) are better known.

Sensitizers as well as desensitizers form a reversal oxidoreduction system with silver halides, according to both pH and  $pAg$  of the photographic emulsion. But besides the specific influence of the emulsion, the efficiency of a sensitizing dye depends on many other factors such as its adsorption, its spectral absorption, the energetic transfer yield, the dye aggregate to the silver halide, and finally on its desensitizing property in

the spectral region where it is not a sensitizer by itself. Effective sensitizers require both the correct absorption wavelength and suitable electrochemical potential.

Since there do not exist generally accepted test methods to measure the efficiency of a dye, the tables do not mention any characteristic of the efficiency and spectral domain of dyes, as they are sometimes found in patents or the literature. Sensitizing power decreases as the chain length increases, and desensitizing and fogging properties have been related to the increase of the ground-level energy (129) together with electron affinity and basicity (130, 131). Experimentally it has been proved that thiazolo carbocyanines become desensitizers when a quinoline ring is fused to the thiazole one. Electron-accepting substituents such as a  $\text{NO}_2$  group on the nucleus, or nitrogen in polymethine chain can confer strong desensitizing properties to the dyes depending on their position and number (132).

Generally an efficient sensitizing dye requires a planar molecular or nearly planar, and so, any steric factor preventing the chromophoric conjugated system from being in a plane leads to an important loss of the sensitizing power. Even if the assistance of the computer allows now the design of new molecules of dyes with appropriate characteristics, practical testing is still necessary for their discrimination as sensitizers. 4-Phenyl-thiazolo dyes are known to be desensitizers (111), though 4,5-diphenyl-substituted trinuclear dyes have been patented as red sensitizers for color photography (133). In general, methine thiazolo dyes are considered to be lacking efficiency; nevertheless some have been patented as supersensitizers. Used with some dyes of other classes, they produce an increase of the intensity and an extent of the sensitization by a suradditive effect. Other physical properties are taken into account for selecting specific sensitizers:

- Easiness of their incorporation by means of their solubility in various media,
- Nonwandering in order to prevent the dyes from migrating from a layer to the next one in multilayer coatings.
- Easy removal during the development process in order to avoid residual and unwanted color stain.

Styryl derivatives have been patented as sensitizers (76) and other methine dyes for sensitization in direct photothermographic material (134).

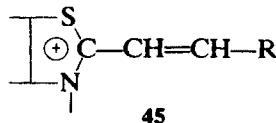
## 2. Textile Dyes

Thiazoloazatrimethine dyes, as generally basic dyes, possess a good affinity for acrylic fibers. It seems that the light fastness of trimethine thiazolo dyes can be increased by the progressive replacement of methine groups by nitrogen atoms, and an excellent fastness to light is reported to be displayed by three nitrogen atoms in the chromophoric chain (66).

## 3. Pharmaceutical Uses

A great number of various substituted aminophenyl derivatives of thiazolium, and their organic or metallic complexes, have been patented as weed-killers or regulating growth factors of plants (135–138).

The bactericidal and enzymatic action of dyes, particularly of vinyl derivatives of 3,4,5-substituted thiazolium, for example, **45** (Scheme 70) (139), have been systematically studied to know more about the basic mechanisms involved (140).



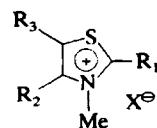
Scheme 70

Many methine cationic dyes, styrylic (141), pyrrolic, or amino-substituted (142) derivatives of thiazolium, possess interesting antihelminthic properties (143). This last class has been used as accelerators of the catabolism and activators of cellular exchanges (144).

Mention must be made of the use of *o*- or *p*-hydroxystyryl thiazolo dyes as indicators for protolytic titrations in nonaqueous media (145), and of the hypotensive action of some neotrinuclear thiazolo cyanines in experiments on animals (146).

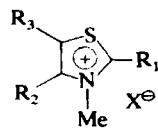
## VI. LIST OF THIAZOLIUM SALTS

TABLE IX-1. 3-METHYLTHIAZOLIUM SALTS



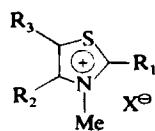
R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	Anion	m.p. (°C)	Ref.*
H	H	H	I <sup>-</sup>	159	147
H	H	H	OTs <sup>-</sup>	—	20 r, 28 k, 1481, 149 k, 150 h
Me	H	H	Cl <sup>-</sup>	—	20 r
Me	H	H	I <sup>-</sup>	—	149 k, 151
Me	H	H	OTs <sup>-</sup>	185	28 k, 150, 152-154
Et	H	H	I <sup>-</sup>	—	149 k, 154 k
n-Pr	H	H	I <sup>-</sup>	—	154
i-Bu	H	H	I <sup>-</sup>	—	154
Neopentyl	H	H	I <sup>-</sup>	—	154
i-Pr	H	H	I <sup>-</sup>	—	149 k, 154
t-Bu	H	H	I <sup>-</sup>	—	149 k, 154
CMe(OH)CONH <sub>2</sub>	H	H	I <sup>-</sup>	152	155
CPh(OCH <sub>2</sub> CH <sub>2</sub> OH) <sub>2</sub>	H	H	I <sup>-</sup>	190-2	156
CH=CHNHPPh	H	H	OTs <sup>-</sup>	—	157
C[NH(p-tolyl)]: N(p-tolyl)]	H	H	I <sup>-</sup>	196-7	158
	H	H	I <sup>-</sup>	210	159 u
Ph	H	H	Cl <sup>-</sup>	—	160
p-(Me) <sub>2</sub> N <sub>2</sub> H <sub>4</sub>	H	H	I <sup>-</sup>	220	137
p-(Me) <sub>2</sub> N <sub>2</sub> H <sub>4</sub>	H	H	Cl <sup>-</sup>	—	161 h
p-(Me) <sub>2</sub> N <sub>2</sub> H <sub>4</sub>	H	H	OTs <sup>-</sup>	—	162 l
SMe	H	H	I <sup>-</sup>	—	159, 163, 164 k
H	Me	H	Br <sup>-</sup>	155-7	18 ir, 19, 21kr, 23 k
H	Me	H	Cl <sup>-</sup>	—	28 k, 150, 165, 166
H	Me	H	I <sup>-</sup>	119-20	156, 167-169
H	Me	H	OTs <sup>-</sup>	148	20 r, 21
H	CH <sub>2</sub> -4'-thiazolyl	H	I <sup>-</sup>	162-3	170
H	CH <sub>2</sub> -4'-thiazolyl	H	2I <sup>-</sup>	240	170
H	t-Bu	H	OTs <sup>-</sup>	140	20 u
H	Ph	H	Cl <sup>-</sup>	—	160 i
H	H	Me	Cl <sup>-</sup>	—	150, 160, 172
H	H	Me	I <sup>-</sup>	67, 1-8, 3	28 k, 171
H	H	Me	IO <sub>4</sub> <sup>-</sup>	75, 1-6	171
H	H	Me	OTs <sup>-</sup>	69	20

TABLE IX-1 (Continued)



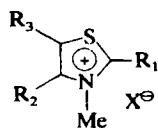
R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	Anion	m.p. (°C)	Ref.
H	H	COOEt	I <sup>-</sup>	153	173
H	H	Ph	Cl <sup>-</sup>	—	160
H	H	Ph	I <sup>-</sup>	—	172
Me	Me	H	Cl <sup>-</sup>	235	147
Me	Me	H	I <sup>-</sup>	—	20 r.u, 28 k, 150 k, 151, 169, 174–177, 178a,
Me	Me	H	OTs <sup>-</sup>	159	179a
Me	Ph	H	I <sup>-</sup>	202	150, 174, 180–182, 183 a, 184 a, 185 a, 186, 187 k
Me	p-ClC <sub>6</sub> H <sub>4</sub>	H	I <sup>-</sup>	—	187
Me	p-BrC <sub>6</sub> H <sub>4</sub>	H	I <sup>-</sup>	—	181, 183, 185, 188
Me	p-NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	H	I <sup>-</sup>	186	184, 185, 187 k
Me	m-NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	H	I <sup>-</sup>	—	187 k
Me	p-MeOC <sub>6</sub> H <sub>4</sub>	H	I <sup>-</sup>	197–8	183 a, 184, 185
Me	p-EtOC <sub>6</sub> H <sub>4</sub>	H	I <sup>-</sup>	204	183 a, 185
Me	2-Naphthyl	H	I <sup>-</sup>	220	189
Me	5-Acenaphthetyl	H	I <sup>-</sup>	186	189
Me	NH <sub>2</sub>	H	I <sup>-</sup>	235	190
CH <sub>2</sub> OH	Me	H	I <sup>-</sup>	—	191 r.u
CH(Me)OH	Me	H	Br <sup>-</sup>	166–7.5	165
CH(Me)OH	Me	H	I <sup>-</sup>	149–50	165, 192 u
CH(Me)OH	Me	H	NO <sub>3</sub> <sup>-</sup>	133–4	192 u
CH(Me)OP(O) (OMe)O <sup>-</sup>	Me	H	Inner salt	—	166
CH(Me)OP(O)OEt <sub>2</sub>	Me	H	Br <sup>-</sup>	—	166
CHPh(OH)	Me	H	I <sup>-</sup>	165–7	193
(CH(Ph)OP(O) (OMe)O <sup>-</sup>	Me	H	Inner salt	—	166
CH(Ph)OP(O)OEt <sub>2</sub>	Me	H	Br <sup>-</sup>	—	166
CMe(OH) <sub>2</sub>	Me	H	I <sup>-</sup>	—	194, 195 k
CH=CHNPh	Me	H	I <sup>-</sup>	248	176
CH=CHN(COMe)Ph	Ph	H	I <sup>-</sup>	162–3	181, 182, 183 a, 184
CH=CHN(COMe)Ph	p-BrC <sub>6</sub> H <sub>4</sub>	H	I <sup>-</sup>	174–5	183, 188 u
CH=CHN(COMe)Ph	p-EtOC <sub>6</sub> H <sub>4</sub>	H	I <sup>-</sup>	188	183 a
Acetyl	Me	H	I <sup>-</sup>	137–8	168 u, 192, 194, 195 k
Benzoyl	Me	H	I <sup>-</sup>	159–60	156, 196
Ph	Me	H	I <sup>-</sup>	—	197
p-Me <sub>2</sub> NC <sub>6</sub> H <sub>4</sub>	Me	H	Cl <sup>-</sup>	—	161, 162
p-Me <sub>2</sub> NC <sub>6</sub> H <sub>4</sub>	Me	H	I <sup>-</sup>	165	137
p-Me <sub>2</sub> NC <sub>6</sub> H <sub>4</sub>	Me	H	OTs <sup>-</sup>	—	138, 198
p-Me <sub>2</sub> NC <sub>6</sub> H <sub>4</sub>	n-Pr	H	I <sup>-</sup>	198	162
NH <sub>2</sub>	Ph	H	I <sup>-</sup>	—	181, 182
NH <sub>2</sub>	p-MeOC <sub>6</sub> H <sub>4</sub>	H	I <sup>-</sup>	—	181

TABLE IX-1 (Continued)



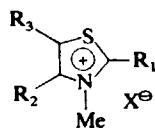
R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	Anion	m.p. (°C)	Ref.
NH <sub>2</sub>	p-EtOC <sub>6</sub> H <sub>4</sub>	H	I <sup>-</sup>	—	181
NH(CH <sub>2</sub> ) <sub>6</sub> NH-2-(3,4-Me di-Me-thiazolyl)	Me	H	2I <sup>-</sup>	202	199
NH(CH <sub>2</sub> ) <sub>6</sub> NH-2(3,4-Me di-Me-thiazolyl)	Me	H	2I <sup>-</sup>	236	199
NH(CH <sub>2</sub> ) <sub>2</sub> NH-2-(3,4-Me di-Me-thiazolyl)	Me	H	2I <sup>-</sup>	265	199
SCH <sub>3</sub>	Me	H	I <sup>-</sup>	—	163, 164
SCH <sub>3</sub>	Ph	H	I <sup>-</sup>	—	200
Me	H	Me	OTs <sup>-</sup>	121	20, 28 k, 153
Me	H	t-Bu	OTs <sup>-</sup>	141	20
Me	H	Ph	I <sup>-</sup>	—	153
Me	H	Br	I <sup>-</sup>	243	157 u
Me	H	OEt	I <sup>-</sup>	109-10	201
Me	H	NH <sub>2</sub>	I <sup>-</sup>	—	157
CH <sub>2</sub> Ph	H	NHCOCH <sub>3</sub>	I <sup>-</sup>	265	202
t-Bu	H	Me	OTs <sup>-</sup>	101	20
Styryl	H	Ph	OTs <sup>-</sup>	90-9	2031
Styryl	H	OEt	I <sup>-</sup>	170	201
CH=CHNHPh	H	Me	I <sup>-</sup>	—	204
CH=CHNHPh	H	Br	OTs <sup>-</sup>	—	157
Ph	H	Ph	OTs <sup>-</sup>	230-3	205
Ph	H	2-Naphthyl	OTs <sup>-</sup>	210-2	2031
p-Biphenylyl	H	Ph	OTs <sup>-</sup>	238-40	2031
N,N,N-trimethyl-p-aniliniumyl	H	Ph	OTs <sup>-</sup>	238-40	2031
p-FC <sub>6</sub> H <sub>4</sub>	H	Ph	OTs <sup>-</sup>	157	2031
p-ClC <sub>6</sub> H <sub>4</sub>	H	Ph	OTs <sup>-</sup>	192-4	2031
m-IC <sub>6</sub> H <sub>4</sub>	H	Ph	OTs <sup>-</sup>	172-4	2031
o-IC <sub>6</sub> H <sub>4</sub>	H	Ph	OTs <sup>-</sup>	210-3	2031
1-Naphthyl	H	Ph	OTs <sup>-</sup>	151	2031
2-Thienyl	H	Ph	OTs <sup>-</sup>	173-6	2031
NHCOCH <sub>3</sub>	H	CH <sub>2</sub> NMe <sub>2</sub>	I <sup>-</sup>	209	206
NHCOCH <sub>3</sub>	H	CH <sub>2</sub> NEt <sub>2</sub>	I <sup>-</sup>	173	206
NHCOCH <sub>3</sub>	H	CH <sub>2</sub> -piperidinyl	I <sup>-</sup>	200	206
NHCOCH <sub>3</sub>	H	CH <sub>2</sub> -Morpholinyl	I <sup>-</sup>	198	206
SMe	H	Me	I <sup>-</sup>	—	164
SMe	H	NH <sub>2</sub>	I <sup>-</sup>	150	207
SMe	H	NHCOCH <sub>3</sub>	I <sup>-</sup>	212	157, 207
H	R <sub>1</sub>	R <sub>1</sub>	MeSO <sub>4</sub> <sup>-</sup>	—	208
H	Me	Me	OTs <sup>-</sup>	135	20, 28 k, 150
H	Me	Et	I <sup>-</sup>	130.5-1.5	209
H	Me	(CH <sub>2</sub> ) <sub>2</sub> Cl	I <sup>-</sup>	165	210, 2111
H	Me	CH <sub>2</sub> CH <sub>2</sub> OH	I <sup>-</sup>	—	35, 158, 167, 2121, 213 p, 214, 215 m, 216

TABLE IX-1 (Continued)



R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	Anion	m.p. (°C)	Ref.
—	Me	CH <sub>2</sub> CH <sub>2</sub> OH	Ylide	—	35, 212
H	Me	CH <sub>2</sub> OH	Cl <sup>-</sup>	—	217 f
H	Me	CHOHMe	I <sup>-</sup>	164.5	218
H	Me	Acetyl	I <sup>-</sup>	168	218
H	Me	COOEt	I <sup>-</sup>	—	167
H	Alkyl	CH <sub>2</sub> CH <sub>2</sub> OH	—	—	219
Me	Me	Me	I <sup>-</sup>	117-18	220
			OTs <sup>-</sup>	149	20 r.u, 28 k, 150, 221
Me	Me	COOH	I <sup>-</sup>	—	222
Me	Me	CO <sub>2</sub> <sup>-</sup>	Betaine	190-1	222
Me	iBu	Me	I <sup>-</sup>	121-3	220
Me	CH <sub>2</sub> Ph	Me	I <sup>-</sup>	—	220
Me	Spiro[4.5]decano[6,7-d]		I <sup>-</sup>	205-7	223
Me	Ph	Me	I <sup>-</sup>	181	220
				111-13	224 a
Me	Ph	Ph	I <sup>-</sup>	208	186, 225
Me	Ph	Ph	OTs <sup>-</sup>	—	187 k, 226
Me	Ph	p-ClC <sub>6</sub> H <sub>4</sub>	I <sup>-</sup>	130	186
Me	Ph	p-NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	I <sup>-</sup>	95	186
Me	Ph	OPh	OTs <sup>-</sup>	—	227
Me	p-Biphenyl	p-Biphenyl	I <sup>-</sup>	226	228
Me	p-ClC <sub>6</sub> H <sub>4</sub>	Ph	I <sup>-</sup>	134	186
Me	p-BrC <sub>6</sub> H <sub>4</sub>	O(p-BrC <sub>6</sub> H <sub>4</sub> )	OTs	—	229 a
Me	p-MeOC <sub>6</sub> H <sub>4</sub>	OPh	OTs <sup>-</sup>	—	229 a
Me	2,4-(MeO) <sub>2</sub> C <sub>6</sub> H <sub>3</sub>	p-ClC <sub>6</sub> H <sub>4</sub>	I <sup>-</sup>	93	186
Me	2-Naphthyl	2-Naphthyl	I <sup>-</sup>	227	228
Me	2-Furyl	COOEt	Me-SO <sub>4</sub>	—	230 a
CH <sub>2</sub> OH	Me	CH <sub>2</sub> CH <sub>2</sub> OH	I <sup>-</sup>	—	191
C(NH-p-MeC <sub>6</sub> H <sub>4</sub> )=N-p-MeC <sub>6</sub> H <sub>4</sub>	Me	CH <sub>2</sub> CH <sub>2</sub> OH	I <sup>-</sup>	194-6	35, 158
C(SH)=NPh	Me	CH <sub>2</sub> CH <sub>2</sub> OH	Cl <sup>-</sup>	206-9	35
C(S <sup>-</sup> )=NPh	Me	CH <sub>2</sub> CH <sub>2</sub> OH	Zwitterion	135-6	35
C(S <sup>-</sup> )=NPh	Me	(CH <sub>2</sub> ) <sub>2</sub> OCSNHPH	Zwitterion	175-6	35
C(S <sup>-</sup> )=N-p-ClC <sub>6</sub> H <sub>4</sub>	Me	CH <sub>2</sub> CH <sub>2</sub> OH	Inner salt	168-9	35
C(S <sup>-</sup> )=N-p-NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	Me	CH <sub>2</sub> CH <sub>2</sub> OH	Inner salt	180-1	35
Ph	Ph	SMc	I <sup>-</sup>	—	231
Ph	Ph	SO <sub>3</sub> <sup>-</sup>	Betaine	303-4	232
p-Me <sub>2</sub> NC <sub>6</sub> H <sub>4</sub>	R'	R"	—	—	161 l, 198
p-R <sub>2</sub> NC <sub>6</sub> H <sub>4</sub>	R	R	X <sup>-</sup>	—	137, 198
p-R <sub>2</sub> NC <sub>6</sub> H <sub>4</sub>	R	R	I <sup>-</sup>	—	198
p-R <sub>2</sub> NC <sub>6</sub> H <sub>4</sub>	R	R	OTs <sup>-</sup>	—	198
p-Me <sub>2</sub> NC <sub>6</sub> H <sub>4</sub>	Me	Me	Cl <sup>-</sup>	—	162
p-Me <sub>2</sub> NC <sub>6</sub> H <sub>4</sub>	Me	Me	I <sup>-</sup>	229	137
p-Me <sub>2</sub> NC <sub>6</sub> H <sub>4</sub>	Me	Me	OTs <sup>-</sup>	—	198
p-Me <sub>2</sub> NC <sub>6</sub> H <sub>4</sub>	Me	Et	I <sup>-</sup>	154-6	162
p-Me <sub>2</sub> NC <sub>6</sub> H <sub>4</sub>	Me	n-Bu	I <sup>-</sup>	134	162

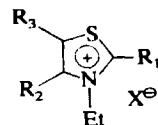
TABLE IX-1 (Continued)



R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	Anion	m.p. (°C)	Ref.
p-Me <sub>2</sub> N <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	Me	<i>n</i> -Octyl	I <sup>-</sup>	102	162
p-Me <sub>2</sub> N <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	Me	<i>i</i> -Pr	I <sup>-</sup>	135	162
p-Me <sub>2</sub> N <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	Et	Me	I <sup>-</sup>	203	162
p-Me <sub>2</sub> N <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	Et	<i>n</i> -Pr	I <sup>-</sup>	149	162
p-Me <sub>2</sub> N <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	<i>n</i> -Pr	Et	I <sup>-</sup>	125	162
p-Me <sub>2</sub> N <sub>2</sub> C <sub>6</sub> H <sub>4</sub>		-(CH <sub>2</sub> ) <sub>4</sub> -	I <sup>-</sup>	233-4	138
p-Me <sub>2</sub> N <sub>2</sub> C <sub>6</sub> H <sub>4</sub>		-(CH <sub>2</sub> ) <sub>4</sub> -	Cl <sup>-</sup>	—	162
p-Me <sub>2</sub> N <sub>2</sub> C <sub>6</sub> H <sub>4</sub>		-(CH <sub>2</sub> ) <sub>4</sub> -	OTs <sup>-</sup>	—	162
p-Me <sub>2</sub> N <sub>2</sub> C <sub>6</sub> H <sub>4</sub>		-(CH <sub>2</sub> ) <sub>5</sub> -	I <sup>-</sup>	190	162
p-Me <sub>2</sub> N <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	Ph	Me	I <sup>-</sup>	159-60	162
NH <sub>2</sub>	Me	Me	MeSO <sub>4</sub> <sup>-</sup>	145	233
NH <sub>2</sub>	Me	Br	X <sup>-</sup>	—	234 p
NH <sub>2</sub>	Me	I	X <sup>-</sup>	—	234 p
NHAc	Me	CH <sub>2</sub> NMe <sub>2</sub>	I <sup>-</sup>	202-4	206
NHAc	Me	CH <sub>2</sub> NEt <sub>2</sub>	I <sup>-</sup>	167	206
NHAc	Me	CH <sub>2</sub> -piperidinyl	I <sup>-</sup>	197	206
NHAc	Me	CH <sub>2</sub> -morpholinyl	I <sup>-</sup>	217	206
N(Me) <sub>2</sub>	Me	Ph	Br <sup>-</sup>	—	235
SMe	Me	Me	I <sup>-</sup>	—	164
SMe	Ph	NH <sub>2</sub>	CH <sub>3</sub> SO <sub>4</sub> <sup>-</sup>	123	202

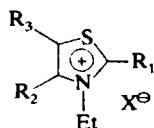
\* Reference code, see p. 2 of Part One. References in italics refer to papers describing experimental conditions for synthesis.

TABLE IX-2. 3-ETHYLTHIAZOLIUM SALTS



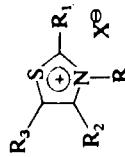
$\text{R}_1$	$\text{R}_2$	$\text{R}_3$	Anion	m.p. (°C)	Ref.
H	H	H	$\text{I}^-$	—	236 r
Me	H	H	$\text{I}^-$	—	237, 238
$\text{CH}=\text{CHN}(\text{COMe})\text{Ph}$	H	H	$\text{I}^-$	—	238
$\text{NH}_2$	H	H	$\text{I}^-$	—	239
H	Me	H	$\text{Br}^-$	170–1	19, 240 I
H	Me	H	$\text{I}^-$	—	241, 242
Me	Me	H	$\text{I}^-$	—	237, 243–248
Me	$\text{COOEt}$	H	$\text{OTs}^-$	—	157
Me	Ph	H	$\text{I}^-$	175.5–6.5	237, 238
Me	<i>p</i> -Xenyl	H	$\text{I}^-$	222	189
Me	<i>p</i> - $\text{HOC}_6\text{H}_4$	H	$\text{I}^-$	—	249
Me	<i>p</i> - $\text{CF}_3\text{SO}_2\text{C}_6\text{H}_4$	H	$\text{I}^-$	—	250
Me	1-Azulenyl	H	$\text{OTs}^-$	—	189
Me	1-Naphthyl	H	$\text{I}^-$	217–18	189
Me	2-Naphthyl	H	$\text{I}^-$	187	189
Me	2-Pyrryl	H	$\text{OTs}^-$	—	251
Me	2-Furyl	H	$\text{OTs}^-$	—	230
Me	2-Benzofuryl	H	$\text{I}^-$	—	230 a
Me	2-Benzofuryl	H	$\text{OTs}^-$	—	230 a
Me	2-Benzothiazolyl	H	$\text{I}^-$	202	252
Me	$\text{NH}_2$	H	$\text{I}^-$	205	190
<i>p</i> -Dimethyl aminostyryl	<i>p</i> -Sulfophenyl	H	Betaine	283–5.5	253
$\text{CH}=\text{CHNH}-$ (2,5-xylyl)	Me	H	$\text{I}^-$	232	243
$\text{CH}=\text{CHNH}-$ ( <i>p</i> -BrC <sub>6</sub> H <sub>4</sub> )	Me	H	$\text{I}^-$	234	243
$\text{CH}=\text{CHNH}-$ ( <i>p</i> -MeOC <sub>6</sub> H <sub>4</sub> )	Me	H	$\text{I}^-$	165	243
$\text{CH}=\text{CHNH}-$ ( <i>o</i> -MeOC <sub>6</sub> H <sub>4</sub> )	Me	H	$\text{I}^-$	251	243
$\text{CH}=\text{CHNH}-$ ( <i>p</i> -EtOC <sub>6</sub> H <sub>4</sub> )	Me	H	$\text{I}^-$	181	243
$\text{CH}=\text{CHNH}(5\text{-Cl}-$ 2-pyridyl)	Me	H	$\text{I}^-$	211	243
$\text{CH}=\text{CHNH}-$ 2-(5-Br-pyridyl)	Me	H	$\text{I}^-$	256	254 I
$\text{CH}=\text{CHNH}-$ 2-(5-I-pyridyl)	Me	H	$\text{I}^-$	274	243
I	Me	H	$\text{I}^-$	171	255
Me	H	COOMe	$\text{OTs}^-$	—	157
Me	H	3-Me-4-MeOC <sub>6</sub> H <sub>3</sub>	$\text{I}^-$	198–202	256, 257
Me	H	3-Ph,4-MeOC <sub>6</sub> H <sub>3</sub>	$\text{I}^-$	—	256
Me	H	3,4(MeO) <sub>2</sub> C <sub>6</sub> H <sub>3</sub>	$\text{I}^-$	193–7	256, 257
Me	H	3,4(EtO) <sub>2</sub> C <sub>6</sub> H <sub>3</sub>	$\text{I}^-$	—	256

TABLE IX-2 (Continued)



R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	Anion	m.p. (°C)	Ref.
Me	H	2,3-Me <sub>2</sub> 4-MeOC <sub>6</sub> H <sub>2</sub>	I <sup>-</sup>	—	256
Me	H	2,5-Me <sub>2</sub> 4-MeOC <sub>6</sub> H <sub>2</sub>	I <sup>-</sup>	—	256
Me	H	3,5-Me <sub>2</sub> 4-MeOC <sub>6</sub> H <sub>2</sub>	I <sup>-</sup>	—	256
Me	H	2-Naphthyl	I <sup>-</sup>	210	228
Me	H	5-Acenaphthyl	I <sup>-</sup>	—	228
Me	H	2-Benzofuranyl	I <sup>-</sup>	—	256
Me	H	2-Benzothiazolyl	I <sup>-</sup>	265	252
Me	H	NHCOCH <sub>3</sub>	OTs <sup>-</sup>	—	157 u
Et	H	COOEt	OTs <sup>-</sup>	—	157
SC <sub>2</sub> H <sub>5</sub>	H	NHCOCH <sub>3</sub>	I <sup>-</sup>	—	258
H	Me	Me	L <sup>-</sup>	—	259
H	Me	CH <sub>2</sub> CH <sub>2</sub> OEt	I <sup>-</sup>	—	259
Me	Me	Acetyl	I <sup>-</sup>	—	157
Me	CH <sub>2</sub> Ph	Me	I <sup>-</sup>	135	220
Me	Ph	Me	OTs <sup>-</sup>	—	260
Me	Ph	Ph	I <sup>-</sup>	184	{ 225, 261, 262, 263, 264, 265}
Me	Ph	Ph	EtSO <sub>4</sub> <sup>-</sup>	—	
Me	p-MeOC <sub>6</sub> H <sub>4</sub>	Ph	OTs <sup>-</sup>	—	229
Me	-spiro(4,5)decane[6,7-d]-		I <sup>-</sup>	188-92	223
CH=C(CH <sub>3</sub> )Cl	Ph	Ph	Cl <sup>-</sup>	119-20	266
SMe	Me	Me	Br <sup>-</sup>	—	267
SMe		-CH <sub>2</sub> -CH <sub>2</sub> -	Cl <sup>-</sup>	—	267

TABLE IX-3. 3-ALKYL- AND 3-ARYLTHIAZOLIUM SALTS



R	R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	Anion	m.p. (°C)	Ref.
n-Pr	H	Me	H	Br <sup>-</sup>	139-41	19
n-Pr	Me	Me	H	Br <sup>-</sup>	—	268
n-Bu	H	Me	H	Cl <sup>-</sup>	—	242
n-Bu	Me	Me	H	I <sup>-</sup>	—	176, 269
n-Bu	CH=CHNHPh	Me	H	I <sup>-</sup>	180	176
n-Bu	CH=CHNH-2-(5-I-pyridyl)	Me	H	I <sup>-</sup>	178	269
n-Bu	Me	Me	H	I <sup>-</sup>	—	248
n-Heptyl	CH=CHNH-2-(5-Br-pyridyl)	Me	H	I <sup>-</sup>	—	254
n-Heptyl	H	Me	CH <sub>2</sub> CH <sub>2</sub> OH	Cl <sup>-</sup>	—	270
		Me	CH <sub>2</sub> CH <sub>2</sub> OH	Cl <sup>-</sup>	—	270
		Me	(CH <sub>2</sub> ) <sub>2</sub> OCOMe	Br <sup>-</sup>	—	270
		Mc	H	Br <sup>-</sup>	224	262, 271
(CH <sub>2</sub> ) <sub>3</sub> COOH			H	I <sup>-</sup>	—	176, 268
(CH <sub>2</sub> ) <sub>3</sub> CO <sub>2</sub> Et	H		CH <sub>2</sub> CH <sub>2</sub> OH	Cl <sup>-</sup> , HCl	156-8	272
(CH <sub>2</sub> ) <sub>3</sub> COOEt	H		CH <sub>2</sub> CH <sub>2</sub> OH	Cl <sup>-</sup> , HCl	150-5	272, 273
(CH <sub>2</sub> ) <sub>3</sub> SO <sub>2</sub> NH <sub>2</sub>	Mc		H	Cl <sup>-</sup>	—	274
i-Amyl	Mc		CH <sub>2</sub> CH <sub>2</sub> OH	Cl <sup>-</sup>	—	274
(CH <sub>2</sub> ) <sub>2</sub> CONH <sub>2</sub>	H		H	Cl <sup>-</sup>	149-50	274
(CH <sub>2</sub> ) <sub>2</sub> CONHEt	H		H	Cl <sup>-</sup>	149-50	274
(CH <sub>2</sub> ) <sub>2</sub> CONHCHM <sub>e</sub> <sub>2</sub>	NH <sub>2</sub>		H	Betaine	164-5	274
(CH <sub>2</sub> ) <sub>2</sub> CONEt <sub>2</sub>	NH <sub>2</sub>		H	1H <sub>2</sub> O	—	274
(CH <sub>2</sub> ) <sub>2</sub> COOH	NH <sub>2</sub>		H			274
(CH <sub>2</sub> ) <sub>2</sub> COOH	NH <sub>2</sub>		H			274

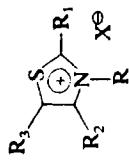
(CH <sub>2</sub> ) <sub>2</sub> COOH	NH <sub>2</sub>	Me	H	Betaine	165	275
(CH <sub>2</sub> ) <sub>2</sub> COOMe	NH <sub>2</sub>	H	H	0.5 H <sub>2</sub> O	—	274
(CH <sub>2</sub> ) <sub>2</sub> Ph	H	Me	H	Cl <sup>-</sup>	—	276
(CH <sub>2</sub> ) <sub>2</sub> NH <sub>2</sub>	H	Me	H	Br <sup>-</sup>	196	274
(CH <sub>2</sub> ) <sub>2</sub> NH <sub>2</sub>	H	Me	H	Cl <sup>-</sup>	—	277 <sup>1</sup>
(CH <sub>2</sub> ) <sub>2</sub> N(Et) <sub>2</sub>	H	Me	CH <sub>2</sub> CH <sub>2</sub> OH	Cl <sup>-</sup> , HCl	—	273 <sup>1</sup>
2-Phthalimidethyl	H	Me	CH <sub>2</sub> CH <sub>2</sub> OH	Cl <sup>-</sup>	—	217 <sup>f</sup>
(CH <sub>2</sub> ) <sub>2</sub> NO <sub>2</sub>	H	Me	H	Br <sup>-</sup>	238	277, 278
(CH <sub>2</sub> ) <sub>2</sub> COOMe	H	Me	H	Cl <sup>-</sup>	—	277 <sup>1</sup>
p-Chlorocymoxymethyl	Me	Me	H	Br <sup>-</sup>	—	217 <sup>f</sup>
Hexahydrobenzyl	H	Me	H	Br <sup>-</sup>	214	279
CH <sub>2</sub> CH(CH <sub>2</sub> O)Br	H	Me	CH <sub>2</sub> CH <sub>2</sub> OH	Br <sup>-</sup>	—	242
CH <sub>2</sub> CH(OH)Ph	H	Me	H	Br <sup>-</sup>	60	280
CH <sub>2</sub> CH(OCO-	H	Mc	H	Br <sup>-</sup>	182.5-3	19, 281
cyclohexyl)Ph			H	Br <sup>-</sup>	—	281
CH <sub>2</sub> CH(OCO-						
cyclohexyl)Ph						
89	C[CH(O)PPh](PPh)(OH)	Me	H	Br <sup>-</sup>	—	281
Allyl	H	Me	H	Br <sup>-</sup>	132-40	19, 27, 152
Allyl	Me	Me	H	Br <sup>-</sup>	—	152, 176
Allyl	CH=CHNHPPh	Me	H	Br <sup>-</sup>	—	176
Allyl	CH=CHNH-	Me	H	Br <sup>-</sup>	190	176
	(3,5-diMeC <sub>6</sub> H <sub>3</sub> )					
Alkyl	H	H	—	—	39	
	CH <sub>3</sub>	CH <sub>3</sub>	CH <sub>2</sub> CH <sub>2</sub> OH	Br <sup>-</sup>	—	
	CH <sub>2</sub> COCHBr <sub>2</sub>	Me	CH <sub>2</sub> CH <sub>2</sub> OH	Br <sup>-</sup>	—	
	3-Bromo-3-phthalimido-					
	acetonyl					
CH <sub>2</sub> COCOOH	H	Me	CH <sub>2</sub> CH <sub>2</sub> OH	Br <sup>-</sup>	—	280
CH <sub>2</sub> COPh	H	H	H	Br <sup>-</sup>	227-9	19, 32
CH <sub>2</sub> COPh	Alkyl	H	X <sup>-</sup>	—	—	39, 282
CH <sub>2</sub> COPh	Me	H	Br <sup>-</sup>	—	41	
CH <sub>2</sub> COPh	H	Me	OH <sup>-</sup>	—	91-2	271
CH <sub>2</sub> COPh	H	Mc	H	Br <sup>-</sup>	—	32

TABLE IX-3 (Continued)

R	R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	Anion	m.p. (°C)	Ref.
CH <sub>2</sub> COPh	Me	Me	H	Br <sup>-</sup>	159-60	283
CH <sub>2</sub> COPh	Et	Me	H	Br <sup>-</sup>	—	283
CH <sub>2</sub> COPh	NH <sub>2</sub>	Me	H	Br <sup>-</sup>	—	284
CH <sub>2</sub> COPh	Mc	Me	Mc	Br <sup>-</sup>	203-4	283
CH <sub>2</sub> CO( <i>p</i> -BrC <sub>6</sub> H <sub>4</sub> )	Mc	H	H	Br <sup>-</sup>	—	41
CH <sub>2</sub> CO( <i>p</i> -NO <sub>2C<sub>6</sub>H<sub>4</sub>)</sub>	H	H	H	Br <sup>-</sup>	226	32
CH <sub>2</sub> CO( <i>p</i> -NO <sub>2C<sub>6</sub>H<sub>4</sub>)</sub>	H	H	H	Betaïne	158	32
CH <sub>2</sub> CO( <i>p</i> -NO <sub>2C<sub>6</sub>H<sub>4</sub>)</sub>	H	H	H	Br <sup>-</sup>	260-1	285]
CH <sub>2</sub> CO( <i>p</i> -NO <sub>2C<sub>6</sub>H<sub>4</sub>)</sub>	H	Mc	H	Betaïne	136-7	27
CH <sub>2</sub> CO( <i>p</i> -NO <sub>2C<sub>6</sub>H<sub>4</sub>)</sub>	H	Mc	CH <sub>2</sub> CH <sub>2</sub> OH	Br <sup>-</sup>	222	285]
CH <sub>2</sub> CO( <i>p</i> -NO <sub>2C<sub>6</sub>H<sub>4</sub>)</sub>	H	Mc	COOEt	Br <sup>-</sup>	205	285]
CH <sub>2</sub> CO( <i>p</i> -NO <sub>2C<sub>6</sub>H<sub>4</sub>)</sub>	NH <sub>2</sub>	Mc	H	Br <sup>-</sup>	174	286
CH <sub>2</sub> -2-furyl	NH <sub>2</sub>	Mc	H	Cl <sup>-</sup>	223	286
CH <sub>2</sub> -2-(5-Br-furyl)	NH <sub>2</sub>	H	H	Br <sup>-</sup>	208-10	286
CH <sub>2</sub> -2-(5-NO <sub>2</sub> -furyl)	NH <sub>2</sub>	Mc	H	Br <sup>-</sup>	253-5	286
CH <sub>2</sub> -2-(5-NO <sub>2</sub> -furyl)	NH <sub>2</sub>	Mc	H	Br <sup>-</sup>	228	262, 271
CH <sub>2</sub> CONHSO <sub>2</sub> CH <sub>3</sub>	Mc	H	H	Betaïne	247	287, 288
CH <sub>2</sub> COOH	NH <sub>2</sub>	Me	CH <sub>2</sub> CH <sub>2</sub> OH	Cl <sup>-</sup>	—	289, 290, 291
CH <sub>2</sub> COOH	H	Me	CH <sub>2</sub> CH <sub>2</sub> OH	OH <sup>-</sup>	178-9	289, 290, 291
CH <sub>2</sub> COOH	NH <sub>2</sub>	H	H	Cl <sup>-</sup>	196	287, 288
CH <sub>2</sub> COOEt	Mc	Me	H	Cl <sup>-</sup>	200	287, 288
CH <sub>2</sub> COOEt	NH <sub>2</sub>	Mc	H	Cl <sup>-</sup>	180-1	287, 288
CH <sub>2</sub> COOEt	H	Me	CH <sub>2</sub> CH <sub>2</sub> OH	Cl <sup>-</sup>	—	270, 289, 290

<chem>CH2COOCH2SO3H</chem>	Me	H	Br <sup>-</sup>	—	292
<chem>CH2Ph</chem>	H	H	Br <sup>-</sup>	—	293
<chem>CH2Ph</chem>	Me	H	—	—	294
<chem>CH2Ph</chem>	CM <sub>2</sub> (OH)CONH <sub>2</sub>	H	Br <sup>-</sup>	151	155
<chem>CH2Ph</chem>	Br <sup>-</sup>	H	Br <sup>-</sup>	—	295 n
<chem>CH2Ph</chem>	—	—	Cl <sup>-</sup>	148	{ 18 i, r, 19, 152, 165, 197, 217, 240 l, 242,
<chem>CH2Ph</chem>	—	—	Br <sup>-</sup>	—	{ 276, 296-300, 301 x
<chem>CH2Ph</chem>	H	H	Br <sup>-</sup>	19	
<chem>CH2Ph</chem>	H	H	Br <sup>-</sup>	—	197, 276
<chem>CH2Ph</chem>	Me	Me	Cl <sup>-</sup>	202	152, 294, 300
<chem>CH2Ph</chem>	Me	Me	Cl <sup>-</sup>	170	197, 276
<chem>CH2Ph</chem>	CH <sub>2</sub> Ph	CH <sub>2</sub> Ph	Br <sup>-</sup>	—	197, 276
<chem>CH2Ph</chem>	CH <sub>2</sub> Ph	CH <sub>2</sub> Ph	Br <sup>-</sup>	—	165
<chem>CH2Ph</chem>	CHOHM <sup>c</sup>	CHOHM <sup>c</sup>	Br <sup>-</sup>	159-160	165
<chem>CH2Ph</chem>	C(NH-p-tolyl) =N-p-tolyl	Me	Br <sup>-</sup>	225-6	158
<b>91</b>	Octylamino	H	Me	Cl <sup>-</sup>	186-9
<chem>CH2Ph</chem>	CH <sub>2</sub> Ph	H	Me	Cl <sup>-</sup>	302
<chem>CH2Ph</chem>	CH <sub>2</sub> Ph	H	Me	Cl <sup>-</sup>	300
<chem>CH2Ph</chem>	CH <sub>2</sub> Ph	H	CH <sub>2</sub> CH <sub>2</sub> OH	141-3	{ 212 e, 215 m 217 f, 303, 304,
<chem>CH2Ph</chem>	CH <sub>2</sub> Ph	H	CH <sub>2</sub> CH <sub>2</sub> OH	—	{ 305, 306
<chem>CH2Ph</chem>	CH <sub>2</sub> Ph	H	CH <sub>2</sub> CH <sub>2</sub> OCOPh	Br <sup>-</sup>	166, 307
<chem>CH2Ph</chem>	CH <sub>2</sub> Ph	H	COOEt	I <sup>-</sup>	186
<chem>CH2Ph</chem>	CH <sub>2</sub> Ph	CH(Me)OP(O)(OEt <sub>2</sub> )	CH <sub>2</sub> CH <sub>2</sub> OB <sup>r</sup>	Br <sup>-</sup>	—
<chem>CH2Ph</chem>	CH <sub>2</sub> Ph	CH(Ph)OP(O)(OEt <sub>2</sub> )	CH <sub>2</sub> CH <sub>2</sub> OB <sup>r</sup>	Br <sup>-</sup>	166, 307 k
<chem>CH2Ph</chem>	CH <sub>2</sub> Ph	C(NH-p-tolyl) =N-p-tolyl	CH <sub>2</sub> CH <sub>2</sub> OH	Br <sup>-</sup>	307 k
<chem>CH2Ph</chem>	CH <sub>2</sub> Ph	C(NP <i>i</i> )S <sup>-</sup>	Me	Cl <sup>-</sup>	193-4
<chem>CH2Ph</chem>	CH <sub>2</sub> Ph	C(N-p-ClC <sub>6</sub> H <sub>4</sub> S <sup>-</sup> )	CH <sub>2</sub> CH <sub>2</sub> OH	Zwitterion	35
<chem>CH2Ph</chem>	CH <sub>2</sub> Ph	Me	CH <sub>2</sub> CH <sub>2</sub> OH	Zwitterion	35
<chem>CH2-p-PhC6H4</chem>	Me	Me	CH <sub>2</sub> CH <sub>2</sub> OH	Cl <sup>-</sup>	184-6
<chem>CH2-p-ClC6H4</chem>	Me	Me	CH <sub>2</sub> CH <sub>2</sub> OH	Cl <sup>-</sup>	128-31
<chem>CH2-o-CC6H4</chem>	Me	Me	H	Cl <sup>-</sup>	190
<chem>CH2-p-NH2C6H4</chem>	Me	H	H	Cl <sup>-</sup>	188

TABLE IX-3 (Continued)



R	R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	Anion	m.p. (°C)	Ref.
CH <sub>2</sub> - <i>p</i> -NH <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	H	Me	H	Cl <sup>-</sup>	183-6	294, 310
CH <sub>2</sub> - <i>p</i> -NH <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	Me	Me	H	Cl <sup>-</sup>	186-7	294, 310
CH <sub>2</sub> - <i>p</i> -NH <sub>2</sub> -C <sub>6</sub> H <sub>4</sub>	H	Me	CH <sub>2</sub> CH <sub>2</sub> OEt	Cl <sup>-</sup>	172-3	310
CH <sub>2</sub> - <i>m</i> -NH <sub>2</sub> -C <sub>6</sub> H <sub>4</sub>	Me	H	H	Cl <sup>-</sup>	228-8.5	294, 310
CH <sub>2</sub> - <i>m</i> -NH <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	H	Me	H	Cl <sup>-</sup>	226-7	294, 310
CH <sub>2</sub> - <i>m</i> -NH <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	Me	Me	H	Cl <sup>-</sup>	228-9.5	294, 310
CH <sub>2</sub> - <i>m</i> -NH <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	H	Me	CH <sub>2</sub> CH <sub>2</sub> OEt	Cl <sup>-</sup>	—	310
CH <sub>2</sub> - <i>m</i> -NH <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	H	H	Cl <sup>-</sup>	Cl <sup>-</sup> , HCl	164-5	294, 310
CH <sub>2</sub> - <i>o</i> -NH <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	Me	H	Cl <sup>-</sup>	Cl <sup>-</sup> , HCl	212	294, 298, 311, 312
CH <sub>2</sub> - <i>o</i> -NH <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	H	Me	H	Cl <sup>-</sup>	—	277 e, 278, 313
CH <sub>2</sub> - <i>o</i> -NH <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	H	Me	H	I <sup>-</sup>	237	310
CH <sub>2</sub> - <i>o</i> -NH <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	H	Me	H	Cl <sup>-</sup>	161-2	294, 310
CH <sub>2</sub> - <i>o</i> -NH <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	Me	Me	Me	Cl <sup>-</sup> , HCl	—	310-312
CH <sub>2</sub> - <i>o</i> -NH <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	H	Me	CH <sub>2</sub> CH <sub>2</sub> OH	Cl <sup>-</sup>	—	310, 314
CH <sub>2</sub> - <i>o</i> -NH <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	H	Me	H	Cl <sup>-</sup>	196-9	276, 285 I, 310
CH <sub>2</sub> - <i>p</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	Me	H	H	Br <sup>-</sup>	—	310
CH <sub>2</sub> - <i>p</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	Br	Me	H	Cl <sup>-</sup>	71-20	310
CH <sub>2</sub> - <i>p</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	Me	Me	CH <sub>2</sub> CH <sub>2</sub> OH	Cl <sup>-</sup>	166-7	276, 285 e
CH <sub>2</sub> - <i>p</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	H	Me	CH <sub>2</sub> CH <sub>2</sub> OH	Br <sup>-</sup>	150	276
CH <sub>2</sub> - <i>p</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	H	Me	CH <sub>2</sub> CH <sub>2</sub> OH	CH <sub>2</sub> CH <sub>2</sub> OH	I <sup>-</sup>	120-1
CH <sub>2</sub> - <i>p</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	H	Me	CH <sub>2</sub> CH <sub>2</sub> OAc	Br <sup>-</sup>	178	276
CH <sub>2</sub> - <i>p</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	H	Me	COOEt	Cl <sup>-</sup>	—	285 e
CH <sub>2</sub> - <i>p</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	H	Me	COOEt	I <sup>-</sup>	160-1	285 e

<chem>CH2-p-NO2C6H4</chem>	<chem>C(NH-p-tolyl)=N-p-tolyl</chem>	<chem>Me</chem>	<chem>CH2CH2OH</chem>	<chem>Br^-</chem>	191-192	158
<chem>CH2-m-NO2C6H4</chem>	<chem>Me</chem>	<chem>H</chem>	<chem>H</chem>	<chem>Cl^-</chem>	149-51	310
<chem>CH2-m-NO2C6H4</chem>	<chem>H</chem>	<chem>Me</chem>	<chem>H</chem>	<chem>Cl^-</chem>	192-2.5	310
<chem>CH2-m-NO2C6H4</chem>	<chem>Me</chem>	<chem>Me</chem>	<chem>H</chem>	<chem>Cl^-</chem>	212-3	310
<chem>CH2-m-NO2C6H4</chem>	<chem>H</chem>	<chem>Me</chem>	<chem>CH2CH2OH</chem>	<chem>Cl^-</chem>	152-3.5	2121, 310
<chem>CH2-o-NO2C6H4</chem>	<chem>Me</chem>	<chem>H</chem>	<chem>H</chem>	<chem>Cl^-</chem>	176-7	310
<chem>CH2-o-NO2C6H4</chem>	<chem>H</chem>	<chem>Me</chem>	<chem>H</chem>	<chem>Cl^-</chem>	198	{278, 297, 298, 310-313, 315}
<chem>CH2-o-NO2C6H4</chem>	<chem>H</chem>	<chem>Me</chem>	<chem>H</chem>	<chem>Br^-</chem>	204	
<chem>CH2-o-NO2C6H4</chem>	<chem>H</chem>	<chem>Me</chem>	<chem>H</chem>	<chem>Cl^-</chem>	199-201	310
<chem>CH2-o-NO2C6H4</chem>	<chem>Me</chem>	<chem>Me</chem>	<chem>H</chem>	<chem>Cl^-</chem>	203-4	311, 312
<chem>CH2-o-NO2C6H4</chem>	<chem>H</chem>	<chem>Me</chem>	<chem>CH2CH2OH</chem>	<chem>Cl^-</chem>	207-8	2121
<chem>CH2-o-NO2C6H4</chem>	<chem>H</chem>	<chem>Me</chem>	<chem>CH2CH2OH</chem>	<chem>Cl^-</chem>	203-5	297, 310, 312, 313-315
<chem>CH2-o-NO2C6H4</chem>	<chem>H</chem>	<chem>Me</chem>	<chem>H</chem>	<chem>Cl^-</chem>	—	3161
<chem>CH(2-benzimidazolyl)</chem>	<chem>H</chem>	<chem>Ph</chem>	<chem>Ph</chem>	<chem>OTs^-</chem>	—	317 a
<chem>CH(2-(2-benzothiazolyl)</chem>	<chem>H</chem>	<chem>Me</chem>	<chem>CH2CH2OH</chem>	<chem>—</chem>	132-3	212
<chem>CH2CN</chem>	<chem>H</chem>	<chem>Me</chem>	<chem>CH2CH2OH</chem>	<chem>Br^-</chem>	—	318
<chem>CH2-N-creatinyl</chem>	<chem>H</chem>	<chem>4-Me</chem>	<chem>H</chem>	<chem>I^-</chem>	136-7	19
<chem>i-Pr</chem>	<chem>H</chem>	<chem>SMe</chem>	<chem>H</chem>	<chem>NHCOCH3</chem>	83	207
<chem>i-Pr</chem>	<chem>SMe</chem>	<chem>SMe</chem>	<chem>H</chem>	<chem>NHCOCH3</chem>	182-3	207
<chem>CH(CH3)(C6H5)</chem>	<chem>Me</chem>	<chem>Me</chem>	<chem>H</chem>	<chem>ClO4^-</chem>	162	319
<chem>CH(CH2COOH)COOH</chem>	<chem>H</chem>	<chem>Me</chem>	<chem>CH2CH2OH</chem>	<chem>Betaine</chem>	161-2	270, 289-291
<chem>CH(CH2COOH)COOH</chem>	<chem>H</chem>	<chem>Me</chem>	<chem>CH2CH2OH</chem>	<chem>Cl^-</chem>	—	270, 289-291
<chem>CH(CH2COOH)COOH</chem>	<chem>H</chem>	<chem>Me</chem>	<chem>CH2CH2OH</chem>	<chem>Br^-</chem>	—	270
<chem>CH(CH2COOH)COOH</chem>	<chem>H</chem>	<chem>Me</chem>	<chem>CH2CH2OH</chem>	<chem>OH^-</chem>	167	270
<chem>CH(CH2CO2Et)CO2Et</chem>	<chem>H</chem>	<chem>Me</chem>	<chem>CH2CH2OH</chem>	<chem>Cl^-</chem>	—	270, 290, 291
<chem>CH(CH=CH2)Ph</chem>	<chem>NH2</chem>	<chem>Ph</chem>	<chem>Ph</chem>	<chem>Cl^-</chem>	—	320
<chem>CH(CO-p-NO2C6H4)</chem>	<chem>H</chem>	<chem>H</chem>	<chem>H</chem>	<chem>Betaine</chem>	172	32
<chem>N(CHO)Ph</chem>	<chem>Me</chem>	<chem>Me</chem>	<chem>H</chem>	<chem>Betaine</chem>	194-5	32
<chem>CH(CO-p-NO2C6H4)</chem>	<chem>H</chem>	<chem>Ph</chem>	<chem>Ph</chem>	<chem>Cl^-</chem>	—	320
<chem>N(CHO)Ph</chem>	<chem>NH2</chem>	<chem>NH2</chem>	<chem>NH2</chem>	<chem>NH2</chem>	—	—
<chem>CH=CHCH2Ph</chem>	<chem>NH2</chem>	<chem>NH2</chem>	<chem>NH2</chem>	<chem>NH2</chem>	—	—

TABLE IX-3 (Continued)

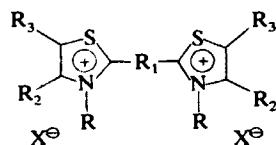
R	R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	Anion	m.p. (°C)	Ref.
Ph	SMe	H	H	I <sup>-</sup>	—	163
Ph	H	Me	H	I <sup>-</sup>	241	147, 152, 241
Ph	Me	Me	H	I <sup>-</sup>	—	147, 152, 238, 241, 244
Ph	Me	Me	H	ClO <sub>4</sub> <sup>-</sup>	180	321
Ph	Me	Ph	H	Br <sup>-</sup>	149	120
Ph	Me	p-Xenyl	H	I <sup>-</sup>	240	189
Ph	CH <sub>2</sub> COMe	Ph	H	Br <sup>-</sup>	—	120
Ph	CH <sub>2</sub> CN	Ph	H	Br <sup>-</sup>	237	120, 322
Ph	Ph	Me	H	ClO <sub>4</sub> <sup>-</sup>	205	323
Ph	Ph	Me	H	I <sup>-</sup>	222	323
Ph	Ph	p-HOC <sub>6</sub> H <sub>4</sub>	H	Br <sup>-</sup>	—	249
Ph	SMe	Me	H	I <sup>-</sup>	—	163
Ph	SEt	NHCOMe	H	HCl	170	324, 1
Ph	SCH <sub>2</sub> Ph	NHCOMe	H	HCl	120	324, 1
Ph	Me	H	Ph	ClO <sub>4</sub> <sup>-</sup>	—	325
Ph	Me	H	p-MeOC <sub>6</sub> H <sub>4</sub>	ClO <sub>4</sub> <sup>-</sup>	—	325
Ph	Me	Me	Me	Br <sup>-</sup>	133	120
Ph	CH <sub>2</sub> COMe	Me	Me	Br <sup>-</sup>	232	120
Ph	Ph	COMe	OH	OH <sup>-</sup>	170-2	326
Ph	Ph	OEt	Ph	—	—	327
Ph	Ph	OCOCH <sub>3</sub>	Ph	—	—	327
p-MeC <sub>6</sub> H <sub>4</sub>	SCH <sub>3</sub>	H	H	I <sup>-</sup>	—	—

<i>p</i> -MeC <sub>6</sub> H <sub>4</sub>	CH <sub>2</sub> CN	Ph	Br <sup>-</sup>	231	322
<i>p</i> -MeC <sub>6</sub> H <sub>4</sub>	SCH <sub>3</sub>	Me	I <sup>-</sup>	—	163
<i>o</i> -MeC <sub>6</sub> H <sub>4</sub>	H	Me	I <sup>-</sup>	230	298
<i>o</i> -MeC <sub>6</sub> H <sub>4</sub>	Me	Me	I <sup>-</sup>	217-18	147
<i>o</i> -MeC <sub>6</sub> H <sub>4</sub>	Me	Me	ClO <sub>4</sub> <sup>-</sup>	172	147
<i>o</i> -MeC <sub>6</sub> H <sub>4</sub>	CH <sub>2</sub> CN	Ph	Br <sup>-</sup>	235	322
<i>p</i> -EtOCOC <sub>6</sub> H <sub>4</sub>	NHNH- <i>p</i> -MeC <sub>6</sub> H <sub>4</sub>	Ph	Br <sup>-</sup>	177	328
<i>p</i> -EtOCOC <sub>6</sub> H <sub>4</sub>	NHNH- <i>p</i> -NH <sub>2</sub> SO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	Ph	Br <sup>-</sup>	180-1	328
<i>p</i> -Biphenyl	Me	Me	I <sup>-</sup>	—	238
<i>p</i> -ClC <sub>6</sub> H <sub>4</sub>	CH <sub>2</sub> CN	Ph	Br <sup>-</sup>	198	322
<i>p</i> -ClC <sub>6</sub> H <sub>4</sub>	NHNH- <i>p</i> -NH <sub>2</sub> SO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	Ph	Br <sup>-</sup>	214	328
<i>p</i> -BrC <sub>6</sub> H <sub>4</sub>	NHNH- <i>p</i> -NH <sub>2</sub> SO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	Ph	Br <sup>-</sup>	221	328
<i>o</i> -MeCONH-C <sub>6</sub> H <sub>4</sub>	H	Me	Cl <sup>-</sup>	222	298
<i>p</i> -Me <sup>2</sup> NC <sub>6</sub> H <sub>4</sub>	Me	Me	Cl <sup>-</sup>	—	244
<i>p</i> -Me <sup>2</sup> NC <sub>6</sub> H <sub>4</sub>	Ph	NH <sub>2</sub>	Br <sup>-</sup>	176	329
<i>p</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	NHNH- <i>p</i> -NH <sub>2</sub> SO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	Ph	Br <sup>-</sup>	193	328
<i>p</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	Me	H	Cl <sup>-</sup>	126	330
2,4,6-(NO <sub>2</sub> ) <sub>3</sub> C <sub>6</sub> H <sub>2</sub>	SM <sub>2</sub>	H	I <sup>-</sup>	—	163
<i>p</i> -MeOC <sub>6</sub> H <sub>4</sub>	Me	Me	ClO <sub>4</sub> <sup>-</sup>	—	244
<i>p</i> -MeOC <sub>6</sub> H <sub>4</sub>	CH <sub>2</sub> CN	Ph	Br <sup>-</sup>	212	322
<i>p</i> -MeOC <sub>6</sub> H <sub>4</sub>	NHNH- <i>p</i> -NH <sub>2</sub> SO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	Ph	Br <sup>-</sup>	191	328
<i>p</i> -MeOC <sub>6</sub> H <sub>4</sub>	SM <sub>2</sub>	Me	I <sup>-</sup>	—	163
<i>p</i> -MeOC <sub>6</sub> H <sub>4</sub>	CH <sub>2</sub> CN	Ph	Br <sup>-</sup>	220	322
<i>o</i> -MeOC <sub>6</sub> H <sub>4</sub>	NHNH- <i>p</i> -MeC <sub>6</sub> H <sub>4</sub>	Ph	Br <sup>-</sup>	139	328
<i>p</i> -EtOC <sub>6</sub> H <sub>4</sub>	NHNH- <i>p</i> -NH <sub>2</sub> SO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	Ph	Br <sup>-</sup>	196-8	328
<i>p</i> -EtOC <sub>6</sub> H <sub>4</sub>	NHNH- <i>p</i> -NaSO <sub>3</sub> C <sub>6</sub> H <sub>4</sub>	Ph	Br <sup>-</sup>	170-2	328
1-Naphthyl	Me	H	ClO <sub>4</sub> <sup>-</sup>	—	244
2-Naphthyl	Me	Ph	ClO <sub>4</sub> <sup>-</sup>	—	325
2-Pyridyl	SM <sub>2</sub>	R <sub>3</sub>	ClO <sub>4</sub> <sup>-</sup>	—	331
5-(6-NH <sub>2</sub> -4-Me-pyrimidinyl)	H	Me	Cl <sup>-</sup> , HCl	254-5	332
5-(6-NH <sub>2</sub> -4-Me-pyrimidinyl)	H	Me	CH <sub>2</sub> CH <sub>2</sub> OH	250	332

TABLE IX-3 (Continued)

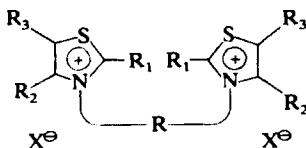
R	R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	Anion	m.p. (°C)	Ref.
5-(6-NH <sub>2</sub> -4-Et-pyrimidinyl)	H	Me	H	Cl <sup>-</sup> , HCl	252-3	332
5-(6-NH <sub>2</sub> -4-Et-pyrimidinyl)	H	Me	CH <sub>2</sub> CH <sub>2</sub> OH	Cl <sup>-</sup> , HCl	220	332
5-(2,6-diNH <sub>2</sub> -4-Me-pyrimidinyl)	H	Me	H	Cl <sup>-</sup> , HCl	315	332
5-(2,6-diOH-4-Me-pyrimidinyl)	H	Me	H	Cl <sup>-</sup>	306	332
2-Benzimidazolyl	H	H	H	Cl <sup>-</sup>	—	316 e
2-Benzimidazolyl	H	H	SO <sub>4</sub> <sup>2-</sup>	—	—	316 e
2-Benzimidazolyl	H	H	CO <sub>3</sub> <sup>2-</sup>	—	—	316 e
2-Benzimidazolyl	H	H	C <sub>1</sub> C <sub>2</sub> H <sub>3</sub> CO <sub>2</sub> <sup>-</sup>	—	—	316 e
2-(1,5,6-triMe-7-CF <sub>3</sub> -benzimidazolyl)	Me	H	Cl <sup>-</sup>	—	—	316 l
2-Thiazolyl	SMe	R <sup>2</sup>	R <sup>3</sup>	ClO <sub>4</sub> <sup>-</sup>	—	331
3-Acetylmino	Me	Me	H	ylide	—	333

TABLE IX-4A. 2,2'-BIS-TIAZOLIUM SALTS



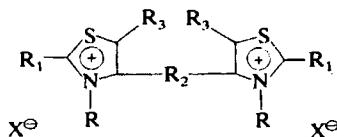
R	R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	Anion	m.p. (°C)	Ref.
Me	(CH <sub>2</sub> ) <sub>4</sub>	Me	H	OTs <sup>-</sup>	216	334
Me	(CH <sub>2</sub> ) <sub>4</sub>	Me	H	ClO <sub>4</sub> <sup>-</sup>	268	334
Me	(CH <sub>2</sub> ) <sub>4</sub>	Ph	H	I <sup>-</sup>	272	334
Et	(CH <sub>2</sub> ) <sub>4</sub>	Ph	H	I <sup>-</sup>	243	334
Et	(CH <sub>2</sub> ) <sub>4</sub>	Et	H	ClO <sub>4</sub> <sup>-</sup>	278	334
Pr	(CH <sub>2</sub> ) <sub>4</sub>	Ph	H	I <sup>-</sup>	201	334
Bu	(CH <sub>2</sub> ) <sub>4</sub>	Ph	H	I <sup>-</sup>	212	334
CH <sub>2</sub> COPh	(CH <sub>2</sub> ) <sub>4</sub>	H	H	Br <sup>-</sup>	142-3	335

TABLE IX-4B. 3,3'-BIS-TIAZOLIUM SALTS



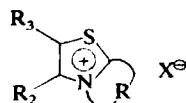
R	R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	Anion	m.p. (°C)	Ref.
3,3'-(CH <sub>2</sub> ) <sub>2</sub>	Me	Me	H	Cl <sup>-</sup>	300	336 a
3,3'-(CH <sub>2</sub> ) <sub>2</sub>	Me	Ph	H	Cl <sup>-</sup>	—	336 a
3,3'-(CH <sub>2</sub> ) <sub>2</sub>	p-Me <sub>2</sub> N-styryl	Ph	H	I <sup>-</sup>	281	336
3,3'-(CH <sub>2</sub> ) <sub>3</sub>	Me	Me	H	Cl <sup>-</sup>	300	336
3,3'-(CH <sub>2</sub> ) <sub>3</sub>	Me	Ph	H	Br <sup>-</sup>	—	336
3,3'-(CH <sub>2</sub> ) <sub>3</sub>	Me	Ph	H	Cl <sup>-</sup>	—	336
3,3'-(CH <sub>2</sub> ) <sub>5</sub>	H	Me	H	Br <sup>-</sup>	244	337
3,3'-(CH <sub>2</sub> ) <sub>5</sub>	Me	Me	H	Br <sup>-</sup>	195	337
3,3'-(CH <sub>2</sub> ) <sub>5</sub>	H	Me	Me	Br <sup>-</sup>	—	337
3,3'-(CH <sub>2</sub> ) <sub>5</sub>	Me	Me	Me	Br <sup>-</sup>	167	337
(CH <sub>2</sub> ) <sub>2</sub> O(CH <sub>2</sub> ) <sub>2</sub>	H	H	I <sup>-</sup>	188	338	
(CH <sub>2</sub> ) <sub>2</sub> O(CH <sub>2</sub> ) <sub>2</sub>	H	H	OH <sup>-</sup>	—	338	
(CH <sub>2</sub> ) <sub>2</sub> O(CH <sub>2</sub> ) <sub>2</sub>	Me	H	I <sup>-</sup>	175	338	
(CH <sub>2</sub> ) <sub>2</sub> O(CH <sub>2</sub> ) <sub>2</sub>	Me	H	OH <sup>-</sup>	—	338	
(CH <sub>2</sub> ) <sub>2</sub> O(CH <sub>2</sub> ) <sub>2</sub>	H	Me	H	I <sup>-</sup>	65	338
(CH <sub>2</sub> ) <sub>2</sub> O(CH <sub>2</sub> ) <sub>2</sub>	H	Me	H	OH <sup>-</sup>	—	338
(CH <sub>2</sub> ) <sub>2</sub> O(CH <sub>2</sub> ) <sub>2</sub>	H	H	Me	I <sup>-</sup>	180	338
(CH <sub>2</sub> ) <sub>2</sub> O(CH <sub>2</sub> ) <sub>2</sub>	H	H	Me	OH <sup>-</sup>	—	338
C <sub>6</sub> H <sub>4</sub> (m)	Me	Me	H	ClO <sub>4</sub> <sup>-</sup>	—	339

TABLE IX-4C. 4,4'-BIS-THIAZOLIUM SALTS



R	R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	Anion	m.p. (°C)	Ref.
Me	H	CH <sub>2</sub>	H	I <sup>-</sup>	162-3	170
Me	H	CH <sub>2</sub>	H	2I <sup>-</sup>	240	170

TABLE IX-5. 2,3-POLYMETHYLENE THIAZOLIUM SALTS



R	R <sub>2</sub>	R <sub>3</sub>	Anion	m.p. (°C)	Ref.
(CH <sub>2</sub> ) <sub>3</sub>	Me	H	ClO <sub>4</sub> <sup>-</sup>	130-2	340, 341
(CH <sub>2</sub> ) <sub>3</sub>		-(CH <sub>2</sub> ) <sub>4</sub> -	Cl <sup>-</sup>	—	341
(CH <sub>2</sub> ) <sub>3</sub>	Ph	H	ClO <sub>4</sub> <sup>-</sup>	187	341
(CH <sub>2</sub> ) <sub>3</sub>	Ph	Ph	IO <sub>4</sub> <sup>-</sup>	—	340, 342
(CH <sub>2</sub> ) <sub>3</sub>	Ph	Ph	ClO <sub>4</sub> <sup>-</sup>	—	340, 342
(CH <sub>2</sub> ) <sub>4</sub>	Me	H	ClO <sub>4</sub> <sup>-</sup>	196	340-342
(CH <sub>2</sub> ) <sub>4</sub>	Ph	H	ClO <sub>4</sub> <sup>-</sup>	190-1	342
(CH <sub>2</sub> ) <sub>4</sub>	Ph	H	I <sup>-</sup>	212	342
(CH <sub>2</sub> ) <sub>4</sub>	Me	Me	ClO <sub>4</sub> <sup>-</sup>	170-1	342
(CH <sub>2</sub> ) <sub>4</sub>	Ph	Ph	I <sup>-</sup>	210	340-342
(CH <sub>2</sub> ) <sub>5</sub>	Me	H	ClO <sub>4</sub> <sup>-</sup>	154	341
(CH <sub>2</sub> ) <sub>5</sub>	Ph	H	I <sup>-</sup>	176-7	341
(CH <sub>2</sub> ) <sub>5</sub>	Me	Me	ClO <sub>4</sub> <sup>-</sup>	104	341
(CH <sub>2</sub> ) <sub>5</sub>	Ph	Ph	I <sup>-</sup>	217-8	340-342

## VII. CLASSIFICATION AND LIST OF THIAZOLOCYANINE DYES

### 1. MONONUCLEAR CYANINES

#### 11. Cationic dyes

##### 111. *Hemicyanines*

- 1111. Aminovinyl compounds
- 1112. Anilinovinyl compounds
- 1113. Aminophenyl compounds

##### 112. *Dimethine compounds*

- 1121. Styryl dyes
- 1122. Dimethine cyanines
- 1123. Azastyryl compounds

#### 12. Non-ionic mononuclear compounds

##### 121. *Ketomethylene compounds*

##### 122. *Anilinovinyl base*

##### 123. *Semicyanines*

### 2. DINUCLEAR CYANINES

#### 21. Cationic cyanines

##### 211. *Monomethine cyanines*

- 2111. Symmetrical
- 2112. Unsymmetrical
- 2113. Substituents on the chain
- 2114. Cyanines bases

##### 212. *Trimethine cyanines*

- 2121. Symmetrical unsubstituted
- 2122. Unsymmetrical unsubstituted
- 2123. Symmetrical substituted on the chain
- 2124. Unsymmetrical substituted on the chain

##### 213. *Pentamethine cyanines*

##### 214. *Heptamethine cyanines*

##### 215. *Chain-bridged cyanines*

- 2151. Chain-bridged
- 2152. Between chain and nitrogen atom

##### 216. *Biscationic dyes*

##### 217. *Azacyanines*

- 2171. Monoazacyanines
- 2172. Diazacyanines
- 2173. Triazacyanines

**22. Neutrocyanines**

- 221. *Zeromethine*
- 222. *Dimethine*
- 223. *Tetramethine*
- 224. *Hexamethine*
- 225. *Azaneutrocyanines*

**3. TRINUCLEAR CYANINES****31. Linear cyanine**

- 311. *Cyanine-cyanine*
- 312. *Cyanine-neutrocyanine*
- 313. *Cyanine-oxonol*
- 314. *Rhodacyanine*
  - 3141. Cationic dyes
  - 3142. Non-ionic dyes
- 315. *Azacyanines*

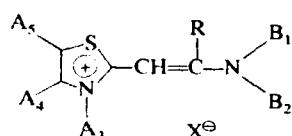
**32. Branched cyanines**

- 321. *Trimethine cyanines substituted in  $\alpha$ -position: neocyanines*
- 322. *Trimethine cyanines substituted in  $\beta$ -position*

**4. TETRANUCLEAR CYANINES**

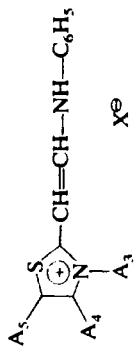
- 41. **Two cyanines**
- 42. **Two neutrocyanines linked by the thiazole ring**
- 43. **Two neutrocyanines linked by the ketomethylene nucleus**
- 44. **One cyanine and one neutrocyanine**

TABLE III. AMINOVINYL COMPOUNDS



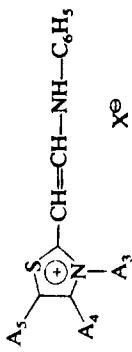
A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	B <sub>1</sub>	B <sub>2</sub>	R	X	m.p. (°C)	Ref.
Me	Ph	Ph	Me	-C(Ph)=C(Ph)SMe	Me	I	183	45
Et	Ph	H	H	COPh	H	OTs	218	114
Et	-C <sub>6</sub> H <sub>4</sub> ( $\sigma$ )-CH <sub>2</sub> -CH <sub>2</sub> -	H	H	COPh	H	OTs	176	114
(CH <sub>2</sub> ) <sub>2</sub> -OH	Me	H	H	H	H	Br	—	343

TABLE 1112A. ANILINOVINYLN COMPOUNDS



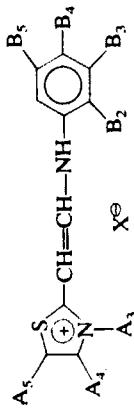
$A_3$	$A_4$	$A_5$	X	$X^\ominus$	Method	m.p. (°C)	$\lambda$ (nm)	Ref.
Me	H	H	OTs	C	—	—	112	112
Me	H	Br	OTs	—	—	—	—	112
Me	Me	H	I	A	248	—	135, 344, 345	346
Me	Me	H	2,4-di-OH-benzoate	—	201	—	—	347
Me	Me	H	$\alpha$ -Naphthenate	—	117	—	347	347, 348
Me	Me	H	4-NH <sub>2</sub> -3-OH-benzoate	—	200	—	—	349
Me	Me	H	$\alpha$ -naphthyl acetate	—	117	—	—	349
Me	Me	Me	I	A	218	—	139	222
Me	Me	COO <sup>-</sup>	—	A	206	412	—	111, 350, 351
Me	Ph	OPh	OTs	C	191	—	—	111, 350, 351
Me	Ph	<i>p</i> -Ph-C <sub>6</sub> H <sub>4</sub> O	I	—	266	—	—	111, 350, 351
Me	Ph	<i>p</i> -Me-C <sub>6</sub> H <sub>4</sub> S	I	—	243	—	—	111, 350, 351
Me	Ph	8-Anilinomethylindeno-1,2-	OTs	C	195	—	—	352
Me	<i>p</i> -Xylyl	Ph	I	C	247	—	—	111
Me	<i>p</i> -Xylyl	<i>p</i> -Me-C <sub>6</sub> H <sub>4</sub>	I	—	230	—	—	111
Me	<i>p</i> -MeOC <sub>6</sub> H <sub>4</sub>	Ph	OTs	C	246	—	—	111, 350, 351
Me	<i>p</i> -MeOC <sub>6</sub> H <sub>4</sub>	OPh	OTs	C	202	—	—	111
Me	<i>p</i> -MeOC <sub>6</sub> H <sub>4</sub>	<i>p</i> - <i>t</i> -Octyl-C <sub>6</sub> H <sub>4</sub> O	OTs	C	236	—	—	353, 354
Et	H	Ph	OTs	C	142	—	—	354
Et	H	$\alpha$ -Naphthyl	OTs	—	204	—	—	354
Et	H	$\alpha$ -Naphthyl	OTs	—	139	—	—	354
Et	H	2-Benzofuryl	OTs	—	150	—	—	354
Et	H	2-Thienyl	OTs	—	185	—	—	354
Et	Me	<i>i</i> -Nicotinate	—	C	157	—	—	355
Et	Me	4-Thiazoyl	I	C	244	—	—	356

TABLE IIIIA (Continued)



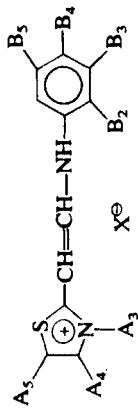
A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	X	Method	m.p. (°C)	λ (nm)	Ref.
Et	Ph	H	OTs	C	176	—	354, 357
Et	Ph	-C <sub>6</sub> H <sub>4</sub> (o)-CH <sub>2</sub> -CH <sub>2</sub> -	OTs	—	218	—	114
Et	Ph	Ph	OTs	C	233	—	111
Et	Ph	OPh	I	C	196	—	111, 350, 351
Et	Ph	p-t-Octyl-C <sub>6</sub> H <sub>4</sub> O	I	—	176	—	111, 350, 351
Et	Ph	p-MeC <sub>6</sub> H <sub>4</sub> S	I	—	225	—	111, 350, 351
Et	Ph	-CH <sub>2</sub> -(o)-C <sub>6</sub> H <sub>4</sub> -	OTs	C	236	—	352
Et	p-Xyl	Ph	I	C	226	—	111
Et	p-Xyl	p-MeC <sub>6</sub> H <sub>4</sub>	I	—	168	—	111
Et	p-BrC <sub>6</sub> H <sub>4</sub>	p-BzC <sub>6</sub> H <sub>4</sub>	I	C	193	—	111, 350, 351
Et	p-ClC <sub>6</sub> H <sub>4</sub>	p-ClC <sub>6</sub> H <sub>4</sub>	I	—	247	—	111, 350, 351
Et	p-MeOC <sub>6</sub> H <sub>4</sub>	p-MeOC <sub>6</sub> H <sub>4</sub>	OTs	C	250	—	111
Et	p-MeOC <sub>6</sub> H <sub>4</sub>	Ph	I	—	233	—	111
Et	p-MeOC <sub>6</sub> H <sub>4</sub>	OPh	I	C	196	—	111, 350, 351
Et	p-MeOC <sub>6</sub> H <sub>4</sub>	Ph	OTs	C	161	—	111
Et	3,4-diMeOC <sub>6</sub> H <sub>3</sub>	p-Phenetylxy	I	C	185	—	111, 350, 351
Et	p-Phenetylxy	p-EOC <sub>6</sub> H <sub>4</sub> O	I	C	185	—	111
Et	Veraaryl	H	OTs	—	197	—	354
Et	β-Naphthyl	H	OTs	C	234	—	111
Et	β-Naphthyl	p-MeC <sub>6</sub> H <sub>4</sub> S	I	C	—	—	358
Et	2-Pyrrolyl	H	I	—	176	—	354, 359
Et	2-Furyl	H	OTs	I	—	—	359
Et	2-Furyl	H	I	A	180	—	135
n-Bu	Me	H	I	A	221	—	139
i-Bu	Me	Me	I	i-Nicotinate	138	—	360
n-Heptyl	Me	H	I	—	—	—	—

TABLE 1112B



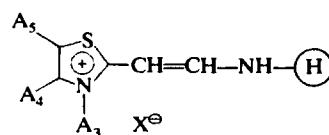
A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	B <sub>2</sub>	B <sub>3</sub>	B <sub>4</sub>	B <sub>5</sub>	X	Method	m.p. (°C)	λ (nm)	Ref.
Me	H	COOEt	H	H	OMe	H	I	B	—	—	361
Me	H	COOEt	OMe	H	H	H	I	B	—	—	361
Me	Me	H	H	OH	COOH	H	I	p-Aminosalicylate	280	—	348
Me	Me	H	OMe	H	H	H	I	A	95	—	362
Me	Me	Me	H	H	Me	H	I	A	263	—	139
Me	Me	Me	H	H	Et	H	I	A	228	—	139
Me	Me	Me	H	H	N-diEt	H	I	A	212	—	139
Me	i-Bu	Me	H	H	Me	H	I	A	23.5	—	139
Me	i-Bu	Me	H	H	COOEt	H	I	A	238	—	139
Me	i-Bu	Me	H	H	OME	H	I	A	21.6	—	139
Me	i-Bu	Me	H	H	OEt	H	I	A	220	—	139
Me	i-Bu	Me	H	H	Me	H	I	A	23.3	—	139
Me	i-Bu	Me	Me	H	H	H	I	A	23.6	—	139
Me	Benzyl	Me	H	H	Me	H	I	A	25.9	—	363
Me	Benzyl	Me	H	H	COOEt	H	I	A	15.3	—	363
Me	Benzyl	Me	H	H	Cl	H	I	A	23.5	—	363
Me	Benzyl	Me	H	H	OMe	H	I	A	25.7	—	363
Me	Ph	Me	H	H	Me	H	I	A	24.5	—	363
Me	Ph	Me	H	H	COOEt	H	I	A	24.3	—	363
Me	Ph	Me	H	H	Cl	H	I	A	22.4	—	363
Me	Ph	Me	H	H	N-diEt	H	I	A	23.5	—	363
Me	Ph	Me	H	H	OH	H	I	A	24.7	—	363
Me	Ph	Me	H	H	OMe	H	I	A	24.7	—	363

TABLE 1112B (Continued)



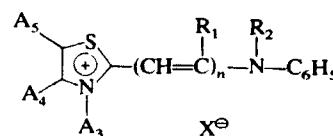
$A_3$	$A_4$	$A_5$	$B_2$	$B_3$	$B_4$	$B_5$	$X$	Method	m.p. (°C)	$\lambda$ (nm)	Ref.
Me	Ph	Me	H	H	OEt	H	I	A	239	—	363
Me	Ph	Me	Me	H	Me	H	I	A	204	—	363
Et	Me	H	H	H	Me	H	<i>i</i> -Nicotinate	A	183	—	360
Et	Me	H	H	H	Br	H	I	A	120	—	362
Et	Me	H	H	H	Cl	H	<i>i</i> -Nicotinate	—	185	—	144 e, 360
Et	Me	H	H	H	N-diEt	H	I	D	—	451	364
Et	Me	H	H	H	OMe	H	I	A	105	—	362
Et	Me	H	H	H	OMe	H	<i>i</i> -Nicotinate	—	163	—	360
Et	Me	H	H	H	OEt	H	I	A	122	—	362
Et	Me	H	Me	H	Me	H	I	A	145	—	362
<i>n</i> -Pr	Me	H	Me	H	Me	H	<i>i</i> -Nicotinate	—	190	—	135
Allyl	Me	H	Me	H	H	H	<i>i</i> -Nicotinate	—	110	—	360

TABLE 1112C.



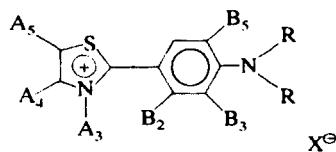
H	A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	X	Method	m.p. (°C)	Ref.
α-naphthyl	Me	i-Bu	Me	I	A	214	139
α-naphthyl	Me	Benzyl	Me	I	A	243	363
β-naphthyl	Me	Me	Me	I	A	225	139
β-naphthyl	Me	i-Bu	Me	I	A	229	139
β-naphthyl	Me	Ph	Me	I	A	247	363
2-(5-Br-pyridyl)	Me	Me	Me	I	A	249	139
2-(5-Br-pyridyl)	Me	i-Bu	Me	I	A	225	139
2-(5-Br-pyridyl)	Me	Benzyl	Me	I	A	229	363
2-(5-Br-pyridyl)	Et	Me	H	I	A	256	362
2-(5-Br-pyridyl)	Et	Me	H	<i>i</i> -Nicotinate	—	154	360
2-(5-Cl-pyridyl)	Et	Me	H	I	A	140	362
2-(5-Cl-pyridyl)	Et	Me	H	<i>i</i> -Nicotinate	—	142	360
2-(5-I-pyridyl)	Me	i-Bu	Me	I	A	242	139
2-(5-I-pyridyl)	Me	Benzyl	Me	I	A	253	363
2-(5-I-pyridyl)	Me	Ph	Me	I	A	220	363
2-(5-I-pyridyl)	Et	Me	H	I	A	274	362
2-(5-I-pyridyl)	n-Bu	Me	H	I	A	178	365
2-(3,5-diBr-pyridyl)	Me	Benzyl	Me	I	A	230	363
2-(3,5-diBr-pyridyl)	Me	Ph	Me	I	A	232	363

TABLE 1112D.



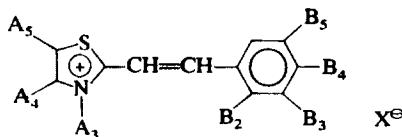
n	R <sub>1</sub>	R <sub>2</sub>	A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	X	Method	m.p. (°C)	Ref.
1	H	COMe	Me	Me	H	I	E	—	348 e
1	H	COMe	Et	H	Ph	OTs	E	130	366
1	H	COMe	Et	Ph	H	OTs	E	138	367
1	H	COMe	Et	2-Furyl	H	I	E	160	359
1	Me	H	Et	p-MeOC <sub>6</sub> H <sub>4</sub>	Ph	I	—	233	111
2	H	H	Me	H	H	I	—	—	368
2	H	H	Et	H	H	I	—	—	369. 669
2	H	COOEt	Me	H	H	I	—	—	368

TABLE 1113. AMINOPHENYL COMPOUNDS



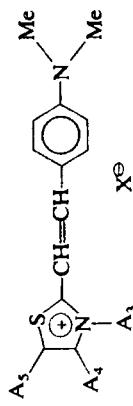
A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	R	B <sub>2</sub>	B <sub>1</sub>	B <sub>3</sub>	X	m.p. (°C)	Ref.
Me	H	H	Me	H	H	H	I	220	142 e
Me	H	H	Me	H	H	H	CO <sub>3</sub>	—	198 e
Me	Me	H	Me	H	H	H	I	165	142 e,
								212	370
Me	Me	H	Me	H	H	H	OTs, SO <sub>4</sub>	—	198
Me	Me	H	Me	H	Me	Me	Br	—	198
Me	Me	H	Et	H	H	H	Br	—	198
Me	Me	Me	Me	H	H	H	Cl, Br, I, OTs	—	136 e, 137 e,
									138 e, 198 e
Me	Me	Me	Me	Br	H	OMe	Oleate	—	198
Me	Me	Me	Et	H	H	H	Butyrate	—	198
Me	Me	Me	n-Pr	H	Me	H	I	—	198
Me	Me	Et	Me	H	H	H	I	154	142 e
Me	Me	Pr	Me	H	H	H	I	134	142 e
Me	Me	i-Pr	Me	H	H	H	I	135	142 e
Me	Me	n-Bu	Me	H	H	H	I	119	370
Me	Me	n-Bu	Me	H	H	H	Formate	—	198
Me	Me	n-Octyl	Me	H	H	H	I	102	142 e
Me	Et	Me	Me	H	H	H	I	203	142 e, 370
Me	Et	Pr	Me	H	H	H	I	149	142 e
Me	Pr	H	Me	H	H	H	I	198	142 e
Me	n-Pr	Et	Me	H	H	H	I	125	142 e
Me	n-Amyl	H	Me	H	H	H	I	134	370
Me	-(CH <sub>2</sub> ) <sub>4</sub> -		Me	H	H	H	I	223	142 e
Me	-(CH <sub>2</sub> ) <sub>4</sub> -		Me	H	H	H	OTs	—	198 e
Me	-(CH <sub>2</sub> ) <sub>5</sub> -		Me	H	H	H	I	190	142 e, 198
Me	-(CH <sub>2</sub> ) <sub>6</sub> -		Me	H	H	H	I	—	198 e
Me	Ph	H	Me	H	H	H	I	—	371
Me	Ph	Me	Me	H	H	H	I	159	142 e
Et	H	Et	i-Bu	Cl	H	Cl	Valerate	—	198 e
Et	-(CH <sub>2</sub> ) <sub>4</sub> -		Me	H	H	H	SO <sub>4</sub>	—	198 e
n-Pr	Et	Et	Me	H	H	H	Cl, Salicylate	—	198 e
i-Bu	Me	Me	Me	H	H	H	I	—	198 e
i-Bu	Me	Me	Et	H	H	H	Cl	—	198 e

TABLE 1121A. STYRYL DYES



A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	B <sub>2</sub>	B <sub>3</sub>	B <sub>4</sub>	B <sub>5</sub>	X	m.p. (°C)	λ (nm)	Ref.
Me	H	H	H	NMe <sub>2</sub>	H	H	I	218	—	372
Me	H	COOEt	OMe	H	H	H	I	—	—	361
Me	H	Ph	H	H	H	H	I	90	—	373 e
Me	Me	H	H	H	OH	H	OH	—	—	374
Me	Me	H	H	H	OH	H	I	—	—	343 e
Me	Me	H	H	H	OMe	H	I	—	—	361
Me	Me	H	H	NH <sub>2</sub>	OMe	H	4-NH <sub>2</sub> -5-OH- benzoate	283	—	347 e
Me	Me	H	OMe	H	H	H	I	—	—	361
Me	Me	Me	H	H	NO <sub>2</sub>	H	I	222	—	139
Me	Me	Me	H	H	OMe	H	I	236	—	139
Me	Me	Me	H	OH	H	H	I	227	—	139
Me	Me	Me	H	OMe	OH	H	I	229	—	139
Me	i-Bu	Me	H	H	NO <sub>2</sub>	H	I	246	—	139
Me	i-Bu	Me	H	NO <sub>2</sub>	H	H	I	198	—	139
Me	i-Bu	Me	H	OMe	OH	H	I	215	—	139
Me	i-Bu	Me	H	—O-CH <sub>2</sub> -O-	H	H	I	213	—	139
Me	Benzyl	Me	H	H	NO <sub>2</sub>	H	I	211	—	363
Me	Benzyl	Me	H	H	OH	H	I	213	—	363
Me	Benzyl	Me	H	OMe	OH	H	I	237	—	363
Me	Benzyl	Me	H	—O-CH <sub>2</sub> -O-	H	H	I	209	—	363
Me	Ph	Me	H	H	NO <sub>2</sub>	H	I	227	—	363
Me	Ph	Me	H	H	OH	H	I	254	—	363
Me	Ph	Me	H	NO <sub>2</sub>	H	H	I	222	—	363
Me	Ph	Me	H	OMe	OH	H	I	214	—	363
Me	Ph	Ph	H	Cl	H	Cl	I	—	—	375
Me	Ph	Ph	OH	H	OH	H	I	—	—	375
Me	Ph	Ph	H	Cl	OH	Cl	Cl	—	—	376
Et	H	H	H	I	OH	I	EtSO <sub>4</sub>	—	—	376
Et	H	H	OH	I	H	I	EtSO <sub>4</sub>	—	—	376
Et	H	Styryl	H	H	H	H	I	234	525	357
Et	H	Ph	H	H	H	H	I	228	—	113
Et	Me	H	H	H	OH	H	I	257	400	76
Et	Me	H	OH	H	H	H	I	255	—	377
Et	Me	H	OH	H	OH	H	I	215	394	76
Et	Me	H	OH	H	OH	H	I	201	—	113
Et	Ph	H	H	H	H	H	I	222	406	76, 145
Et	Ph	H	OH	H	H	H	I	—	—	145
Et	Ph	H	H	H	OH	H	I	—	—	145
Et	Ph	Ph	H	Br	OH	Br	I	—	—	375, 378
Et	Ph	Ph	H	Cl	OH	Cl	Cl	—	—	376
Et	Ph	Ph	H	I	OH	I	EtSO <sub>4</sub>	—	—	376
Et	Ph	Ph	OH	Br	H	Br	EtSO <sub>4</sub>	—	—	376

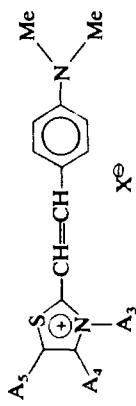
TABLE 1121B.



$\text{A}_3$	$\text{A}_4$	$\text{A}_5$	$\text{X}$	m.p. (°C)	$\lambda$ (nm)	Ref.
Me	H	$\text{CH}_2\text{OH}$	OTs	212	489	379
Me	H	Br	I	238	510	112
Me	Mc	H	Cl	—	—	380
Me	Me	H	Erythrosine	—	—	381
Me	Me	Me	I	—	—	139
Me	Me	COO <sup>-</sup>	—	194	490	222
Me	i-Bu	Me	I	216	—	139
Me	Benzyl	Me	I	218	—	363
Me	$\text{CH}_2\text{OH}$	H	OTs	297	485	379
Me	Ph	H	I	205	490	80, 143, 382, 383
Me	Ph	Me	I	216	—	363
Me	Ph	Ph	I	44	498	83
Me	Ph	$p\text{-ClC}_6\text{H}_4$	I	55	520	83
Me	Ph	$p\text{-NO}_2\text{C}_6\text{H}_4$	I	148	570	83
Me	$p\text{-MeC}_6\text{H}_4$	H	I	187	490	382
Me	$p\text{-MeC}_6\text{H}_4$	H	I	239	—	384
Me	$p\text{-PhC}_6\text{H}_4$	H	I	167	490	382
Me	$p\text{-PhC}_6\text{H}_4$	H	I	253	—	141 e
Me	$m\text{-BrC}_6\text{H}_4$	H	I	225	495	382
Me	$p\text{-BrC}_6\text{H}_4$	H	I	191	490	80, 82, 382
Me	$p\text{-BrC}_6\text{H}_4$	H	I	223	—	384
Me	$p\text{-ClC}_6\text{H}_4$	H	I	232	490	80, 82, 382

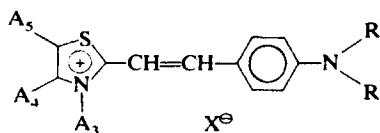
Me	H	I	217	—	384
Me	Ph	I	81	506	83
Me	H	I	229	—	143, 384
Me	H	I	224	490	382
Me	H	I	195	480	143, 382
Me	H	I	225	490	382
Me	<i>p</i> -NO <sub>2C<sub>6</sub>H<sub>4</sub></sub>	I	60	505	83
Me	2,3-diMeOC <sub>6</sub> H <sub>3</sub>	I	—	—	385
Me	$\alpha$ -Furyl	H	—	—	385
Me	$\alpha$ -Thienyl	H	—	—	385
Me	NH <sub>2</sub>	H	—	—	386
Et	H	H	—	—	387
Et	Et	H	142	483	16
Et	H	CH <sub>2</sub> OEt	167	492	379
Et	H	Styryl	234	525	357
Et	H	Ph	262	520	386
Et	H	<i>p</i> -MeOC <sub>6</sub> H <sub>4</sub>	—	—	387
Et	H	<i>p</i> -( <i>p</i> -MeOC <sub>6</sub> H <sub>4</sub> )C <sub>6</sub> H <sub>4</sub>	—	—	388
Et	H	<i>p</i> -PhOC <sub>6</sub> H <sub>4</sub>	—	—	388
Et	H	2-Benzothiazolyl	264	508	388
Et	H	2-Benzothiazolyl	—	—	389
Et	H	H	253	516	389
Et	Et	Br	—	—	377
Et	H	Br	242	470	76
Et	H	Br	241	—	72, 667
Et	H	OTs	—	480	—
Et	Me	Me	—	—	390
Et	Me	Me	243	510	356
Et	Me	Me	235	510	356
Et	Et	Me	—	—	391
Et	Et	Me	230	—	391
Et	Et	4-Thiazolyl	114	—	—
Et	Et	4-(2-Me-thiazolyl)	115	—	391
Et	Et	Tetradecyl	—	—	391
Et	Et	Hexadecyl	—	—	391
Et	Et	<i>p</i> -MeOCOBenzyl	94	—	391
Et	Et	CH <sub>3</sub> COMe	193	496	379
Et	Et	Styryl	228	493	357
Et	Et	Ph	255	495	357

TABLE 1121B (Continued)



$A_3$	$A_4$	$A_5$	$X$	m.p. (°C)	$\lambda$ (nm)	Ref.
Et	Ph	H	I	195	490	16
Et	<i>p</i> -PhC <sub>6</sub> H <sub>5</sub>	H	I	233	—	141 e
Et	<i>p</i> -( <i>p</i> -MeOC <sub>6</sub> H <sub>4</sub> )C <sub>6</sub> H <sub>4</sub>	H	Cl	—	490	388
Et	<i>p</i> -PhOC <sub>6</sub> H <sub>4</sub>	H	I	238	490	141 e, 388
Et	<i>p</i> -SO <sub>3</sub> C <sub>6</sub> H <sub>4</sub>	H	—	—	—	392
Et	B-Naphthyl	<i>p</i> -MeC <sub>6</sub> H <sub>4</sub> S	ClO <sub>4</sub>	—	—	351
Et	2-Benzothiazolyl	H	ClO <sub>4</sub>	234	511	389
Et	OEt	H	ClO <sub>4</sub>	168	491	379
Et	<i>p</i> -HOOCBenzyl	Me	—	—	—	393
Ph	Me	H	I	—	—	394
Ph	Me	H	ClO <sub>4</sub>	240	500	16
Ph	Me	H	ClO <sub>4</sub>	237	504	16
	<i>p</i> -PhC <sub>6</sub> H <sub>4</sub>	Me	ClO <sub>4</sub>	—	—	116
	2-Pyridyl	Me	ClO <sub>4</sub>	—	—	116
	2-Thiazolyl	Me	ClO <sub>4</sub>	—	—	116
	2-Benzothiazolyl	H	ClO <sub>4</sub>	—	—	395
	2-Benzothiazolyl	Me	ClO <sub>4</sub>	—	—	116

TABLE 1121C.



$\text{A}_3$	$\text{A}_4$	$\text{A}_5$	R	X	m.p. (°C)	$\lambda$ (nm)	Ref.
Me	H	Me	Et	I	227	—	396
Me	Me	H	Et	I	157	—	397
Me	Me	Me	Et	I	217	—	396
Me	Ph	H	Et	I	211	490	83, 397
Me	Ph	H	$(\text{CH}_2)_4$	Br	236	—	141 e
Me	Ph	H	$(\text{CH}_2)_4$	I	234	—	141 e
Me	Ph	Me	Et	I	201	—	398
Me	Ph	Ph	Et	I	218	—	398
Me	Ph	<i>p</i> -ClC <sub>6</sub> H <sub>4</sub>	Et	I	149	525	83
Me	Ph	<i>p</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	Et	I	196	570	83
Me	<i>p</i> -MeC <sub>6</sub> H <sub>4</sub>	H	Et	I	184	—	398
Me	<i>p</i> -PhC <sub>6</sub> H <sub>4</sub>	H	Et	I	208	—	141 e
Me	<i>p</i> -PhC <sub>6</sub> H <sub>4</sub>	H	$(\text{CH}_2)_4$	I	262	—	141 e
Me	<i>p</i> -( <i>p</i> -MeOC <sub>6</sub> H <sub>4</sub> )C <sub>6</sub> H <sub>4</sub>	H	$(\text{CH}_2)_4$	I	279	—	141 e
Me	<i>p</i> -BrC <sub>6</sub> H <sub>4</sub>	H	Et	I	198	—	398
Me	<i>p</i> -BrC <sub>6</sub> H <sub>4</sub>	H	$(\text{CH}_2)_4$	I	211	—	141 e
Me	<i>p</i> -ClC <sub>6</sub> H <sub>4</sub>	H	Et	I	160	—	398
Me	<i>p</i> -ClC <sub>6</sub> H <sub>4</sub>	H	$(\text{CH}_2)_4$	I	219	—	141 e
Me	<i>p</i> -ClC <sub>6</sub> H <sub>4</sub>	Ph	Et	I	159	519	83
Me	<i>p</i> -FC <sub>6</sub> H <sub>4</sub>	H	$(\text{CH}_2)_4$	I	238	—	141 e
Me	<i>p</i> -IC <sub>6</sub> H <sub>4</sub>	H	Et	I	211	—	398
Me	2,4-diMeO-C <sub>6</sub> H <sub>3</sub>	<i>p</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>5</sub>	Et	I	120	515	83
Et	Ph	H	$(\text{CH}_2)_4$	I	228	—	141 e
Et	<i>p</i> -Ph-C <sub>6</sub> H <sub>4</sub>	H	$(\text{CH}_2)_4$	I	264	—	141 e
Et	<i>p</i> -BrC <sub>6</sub> H <sub>4</sub>	H	$(\text{CH}_2)_4$	I	223	—	141 e
Et	<i>p</i> -ClC <sub>6</sub> H <sub>4</sub>	H	$(\text{CH}_2)_4$	I	217	—	141 e
C <sub>2</sub> H <sub>4</sub> OH	Me	H	C <sub>2</sub> H <sub>4</sub> OH	Cl	231	482	668
C <sub>2</sub> H <sub>4</sub> OH	Me	H	C <sub>2</sub> H <sub>4</sub> OH	Br	227	482	668
C <sub>2</sub> H <sub>4</sub> OH	Ph	H	C <sub>2</sub> H <sub>4</sub> OH	Cl	217	492	668
C <sub>2</sub> H <sub>4</sub> OH	Ph	H	C <sub>2</sub> H <sub>4</sub> OH	Br	187	492	668

TABLE 1121D.

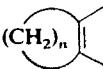
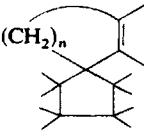
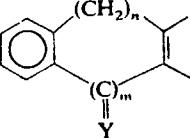
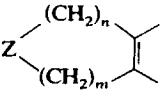
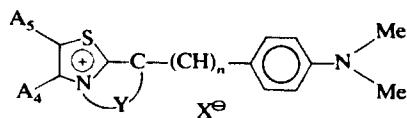
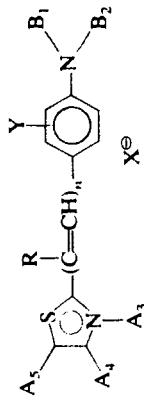
H	p	A <sub>3</sub>	R	m.p. (°C)	λ (nm)	Ref.
	<i>n</i>					
	3	1	Et	Me	267	470 399
	4	1	Et	Me	246	480 16, 72
	4	1	Et	Me	—	490 87
	4	1	Et	Et	—	490 400
	4	1	Me	Me	228	490 401
(4- <i>i</i> -Pr, 7-Me)	5	1	Et	Me	238	476 402
	<i>n</i>					
	2	1	Et	Me	145	490 403
	3	1	Me	Me	232	470 404
	<i>n</i>	<i>m</i>	Y			
	2	0	—	1	Me Me	215 496 405
	0	1	H, H	1	Et Me	249 523 352
	0	1	=CHC <sub>6</sub> H <sub>4</sub> N	1	Et Me	— 534 352
			Et <sub>2</sub> (p)			
	<i>n</i>	<i>m</i>	Z			
	1	2	O	1	Et Me	233 495 87, 406
	3	0	O	1	Et Me	315 494 407
	1	2	S	1	Et Me	247 490 87, 408
	0	3	S	1	Et Me	256 506 87, 409
	0	3	S	2	Et Me	236 548 87, 409

TABLE 1121E.



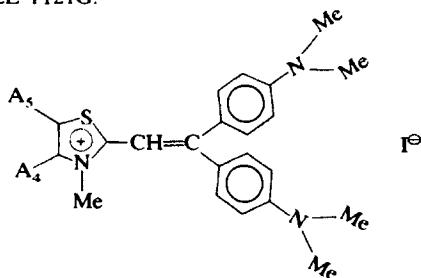
Y	n	A <sub>4</sub>	A <sub>5</sub>	X	m.p. (°C)	λ (nm)	Ref.
—(CH <sub>2</sub> ) <sub>3</sub> —	1	Ph	H	I	281	498	410
—(CH <sub>2</sub> ) <sub>3</sub> —	1	H	Ph	I	—	—	410
—(CH <sub>2</sub> ) <sub>2</sub> O—	1	Me	H	ClO <sub>4</sub>	218	492	411
—(CH <sub>2</sub> ) <sub>2</sub> S—	1	Me	H	ClO <sub>4</sub>	232	502	411
—CH <sub>2</sub> —C <sub>6</sub> H <sub>4</sub> (o)CH <sub>2</sub> —	1	Me	H	ClO <sub>4</sub>	216	468	412
—CH <sub>2</sub> C <sub>6</sub> H <sub>4</sub> (o)—	1	Me	H	ClO <sub>4</sub>	156	446	412
—C <sub>6</sub> H <sub>4</sub> (o)(CH <sub>2</sub> ) <sub>2</sub> —	1	Me	H	ClO <sub>4</sub>	—	444	413, 414
—C <sub>6</sub> H <sub>4</sub> (o)—	1	Me	H	ClO <sub>4</sub>	221	585	415
—C <sub>6</sub> H <sub>4</sub> (o)—	3	Me	H	ClO <sub>4</sub>	233	670	415
—C <sub>6</sub> H <sub>4</sub> (o)O—	1	Me	H	ClO <sub>4</sub>	266	550	121, 413, 414
—C <sub>6</sub> H <sub>4</sub> (o)Se—	1	Me	H	ClO <sub>4</sub>	—	516	413, 414
—C <sub>10</sub> H <sub>6</sub> (1,2)O—	1	H	H	ClO <sub>4</sub>	240	550	121, 413, 414

TABLE 1121F.



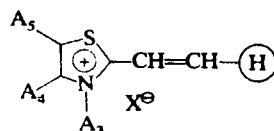
<i>n</i>	R	A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	B <sub>1</sub>	B <sub>2</sub>	Y	X	m.p. (°C)	λ (nm)	Ref.
114	H	Me	H	H	(CH <sub>2</sub> ) <sub>3</sub> OCOOEt	(CH <sub>2</sub> ) <sub>3</sub> SO <sub>3</sub> <sup>-</sup>	H	—	—	674	
	H	Me	Me	H	COMe	COMe	H	I	285	—	416
	H	Me	Me	H	COMe	COMe	H	I	—	—	139
	H	Me	i-Bu	H	COMe	COMe	H	I	255	—	139
	H	Me	Ph	H	COMe	CH <sub>3</sub> CHOH-	H	I	255	—	363
	H	Et	p-KO <sub>2</sub> S-C <sub>6</sub> H <sub>4</sub>	H	Me	CH <sub>3</sub> SO <sub>3</sub> Na	OTs	—	—	674	
	H	(CH <sub>2</sub> ) <sub>3</sub> SO <sub>3</sub> <sup>-</sup>	H	H	Me	(CH <sub>2</sub> ) <sub>2</sub> CHMe-SO <sub>3</sub> <sup>-</sup>	3-OMc-6-NHCOPh	—	—	674	
	H	(CH <sub>2</sub> ) <sub>3</sub> SO <sub>3</sub> <sup>-</sup>	Ph	H	Me	(3-SO <sub>3</sub> K-4-OMe)-C <sub>6</sub> H <sub>3</sub>	H	—	—	674	
	H	(CH <sub>2</sub> ) <sub>3</sub> SO <sub>3</sub> <sup>-</sup>	p-KO <sub>2</sub> S-C <sub>6</sub> H <sub>4</sub>	H	(CH <sub>2</sub> ) <sub>3</sub> SO <sub>3</sub> Na	CH <sub>2</sub> CHMeSO <sub>3</sub> H	2-NH-COPh	—	—	674	
	H	CHMe(CH <sub>2</sub> ) <sub>2</sub> -SO <sub>3</sub> H	Me	(CH <sub>2</sub> ) <sub>3</sub> SO <sub>3</sub> Na	(CH <sub>2</sub> ) <sub>3</sub> SO <sub>3</sub> Na	H	—	—	—	674	
1	COMe	Ph	Mc	Mc	Mc	Mc	H	Br	247	430	120
1	COMe	Ph	Ph	H	Mc	Mc	H	Br	219	435	120
1	COOEt	Ph	Ph	H	Mc	Mc	H	Br	193	422	120
1	CN	Ph	Ph	H	Me	Me	H	Br	192	516	120
4	H	Me	Me	H	Me	Me	I	243	533	416	
4	H	Et	Ph	H	Et	H	ClO <sub>4</sub>	—	702	417	

TABLE 1121G.



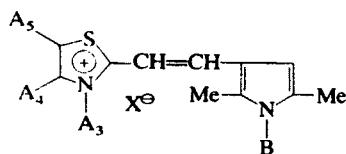
$A_4$	$A_5$	m.p. (°C)	$\lambda$ (nm)	Ref.
Me	H	261	475	123
Me	COOEt	214	526	123
Ph	H	170	492	123

Table 1122A. DIMETHINECYANINES



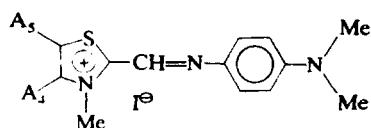
H	$A_3$	$A_4$	$A_5$	X	m.p.	$\lambda$ (nm)	Ref.
$\alpha$ -naphthyl	Me	Me	Me	I	199	—	139
$\alpha$ -(4-NEt <sub>2</sub> -naphthyl)	Me	Me	H	I	202	—	383
1-Tropylium	Me	H	H	ClO <sub>4</sub>	257	481	418
1-Tropylium	Me	Me	H	I	266	481	418
1-(3-Me-tropylium)	Me	H	H	ClO <sub>4</sub>	264	502	418
1-(3-Me-tropylium)	Me	Me	H	ClO <sub>4</sub>	291	500	418
1-(4,6,8-triMe-tropylium)	Me	H	H	ClO <sub>4</sub>	270	499	418
1-(4,6,8-triMe-tropylium)	Me	Me	H	I	268	499	418
1-(4,6,8-triMe-tropylium)	Me	Me	H	ClO <sub>4</sub>	296	499	418
1-(3,8-diMe-5-i-Pr-tropylium)	Me	H	H	ClO <sub>4</sub>	254	525	418
2-cyclopentadienylidene triphenylphosphorane	Et	H	H	ClO <sub>4</sub>	—	460	419
$\alpha$ -Furyl	Me	Me	Me	I	202	—	139
$\alpha$ -Furyl	Me	Benzyl	Me	I	206	—	363
4-Quinolyl	Me	Me	H	I	226	—	57
5-(2-NMe <sub>2</sub> -4-Me-thiazole)	Et	H	H	I	—	—	420
5-(2-MePh-amino-4-Me-thiazole)	Et	H	H	I	—	—	420
5-(2-MePh-amino-4-Ph-thiazole)	Et	H	H	I	—	—	420
5-(2-SMe-4-Me-thiazole)	Et	H	H	I	—	—	420

TABLE 1122B.



<b>A<sub>3</sub></b>	<b>A<sub>4</sub></b>	<b>A<sub>5</sub></b>	<b>B</b>	<b>X</b>	<b>m.p.(°C)</b>	<b>λ(nm)</b>	<b>Ref.</b>
Me	Ph	H	Me	I	228	—	421 e, 422
Me	Ph	H	Et	I	208	—	421 e, 422
Me	Ph	H	n-Bu	I	182	—	421 e, 422
Me	Ph	H	n-Amyl	I	108	—	421 e, 422
Me	Ph	H	Ph	I	223	—	421 e, 422
Me	Ph	H	p-ClC <sub>6</sub> H <sub>4</sub>	I	—	—	422
Me	-C <sub>6</sub> H <sub>4</sub> (o)-(CH <sub>2</sub> ) <sub>2</sub> -	Ph		I	226	456	405
Me	p-PhC <sub>6</sub> H <sub>4</sub>	H	Me	I	222	—	421 e
Me	p-PhC <sub>6</sub> H <sub>4</sub>	H	Et	I	192	—	421 e
Me	p-PhC <sub>6</sub> H <sub>4</sub>	H	Ph	I	216	—	421 e, 422
Me	p-PhC <sub>6</sub> H <sub>4</sub>	H	p-ClC <sub>6</sub> H <sub>4</sub>	I	227	—	421 e
Me	p-BrC <sub>6</sub> H <sub>4</sub>	H	Me	Br	216	—	421 e
Me	p-BrC <sub>6</sub> H <sub>4</sub>	H	n-Bu	I	181	—	421 e
Me	p-BrC <sub>6</sub> H <sub>4</sub>	H	n-Amyl	I	176	—	421 e
Me	p-BrC <sub>6</sub> H <sub>4</sub>	H	Ph	I	222	—	421 e, 422
Me	p-ClC <sub>6</sub> H <sub>4</sub>	H	Me	I	229	—	421 e
Me	p-ClC <sub>6</sub> H <sub>4</sub>	H	n-Bu	I	169	—	421 e
Me	p-ClC <sub>6</sub> H <sub>4</sub>	H	n-Amyl	I	181	—	421 e
Me	p-MeOC <sub>6</sub> H <sub>4</sub>	H	n-Bu	I	148	—	421 e
Me	p-MeOC <sub>6</sub> H <sub>4</sub>	H	Ph	I	—	—	422
Me	β-Naphthyl	H	Me	I	221	—	421 e
Me	β-Naphthyl	H	Ph	I	—	—	422
Et	Me	H	1-Ph-2,5-diMe-pyrrolyl (3)	I	—	434	667
Et	-(CH <sub>2</sub> ) <sub>3</sub> -	Et		I	273	440	87, 399
Et	-(CH <sub>2</sub> ) <sub>2</sub> -O-CH <sub>2</sub> -	Ph		I	>300	440	87, 406
Et	-(CH <sub>2</sub> ) <sub>3</sub> -S-	Ph		I	229	444	87, 409

TABLE 1123. AZASTYRYL COMPOUNDS



A <sub>4</sub>	A <sub>5</sub>	m.p. (°C)	λ (nm)	Ref.
Ph	H	209 235	530 530	80, 82, 83 382
Ph	Ph	113	550	83
Ph	p-ClC <sub>6</sub> H <sub>4</sub>	85	545	83
Ph	p-NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	151	552	83
p-MeC <sub>6</sub> H <sub>4</sub>	H	238	535	382
p-PhC <sub>6</sub> H <sub>4</sub>	H	195	540	382
p-BrC <sub>6</sub> H <sub>4</sub>	H	234	530	383
		193	540	80
m-BrC <sub>6</sub> H <sub>4</sub>	H	215	530	382
p-ClC <sub>6</sub> H <sub>4</sub>	H	225	540	382
		190	530	80, 82
p-ClC <sub>6</sub> H <sub>4</sub>	Ph	>310	545	83
p-MeOC <sub>6</sub> H <sub>4</sub>	H	215	530	382
p-EtOC <sub>6</sub> H <sub>4</sub>	H	200	530	382
m-NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	H	224	530	382
2,4-diMeOC <sub>6</sub> H <sub>3</sub>	p-NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	101	526	83

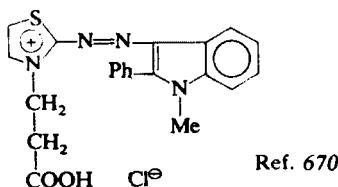


TABLE 121. KETOMETHYLENE COMPOUNDS

	m.p. (°C)	Ref.
	173	76
	Me 178	76
	Ph 150	76
	—	423
	146	424

TABLE 122. ANILINOVINYL BASE

		m.p. (°C)	λ(nm)	Ref.
		111	347	13

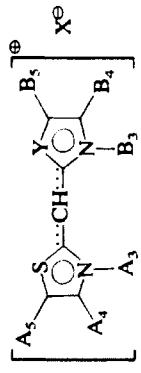
TABLE 123. SEMICYANINES

n	R	A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	m.p. (°C)	λ(nm)	Ref.
1	CS-NMePh	Me	H	H	119	—	425, 426
1	CS-pyrrolidinyl	Me	H	H	153	—	425, 426
1	P-Ph <sub>2</sub> (ClO <sub>4</sub> )	Et	H	H	180	388	419
3	CN	Et	Ph	Ph	208	658	427

TABLE 2111. SYMMETRICAL MONOMETHINE CYANINES

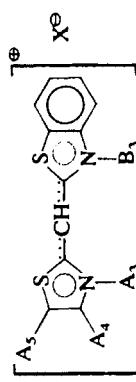
A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	X	Method	m.p. (°C)	λ(nm)	Ref.
Me	Me	H	I	B,C	> 320	—	1(p. 72), 428
Me	Me	H	I <sub>2</sub>	B	233	—	428
Me	Ph	H	I	—	—	420	429
Me	Ph	Ph	I	—	—	440	429
Et	H	H	I	A	240	438	430, 431, 432
Et	Me	H	I	A	298–308	409	109, 432, 433, 666
Et	Ph	H	I	A	260	—	432
Et	Ph	Ph	I	A	265	439	109, 433, 667
<i>n</i> -Heptyl	Me	H	<i>i</i> -Nicotinate	—	132	—	360
Ph	Ph	H	Br	D	—	—	434
p-MeOC <sub>6</sub> H <sub>4</sub>	Ph	H	Br	D	—	—	434
p-EtOC <sub>6</sub> H <sub>4</sub>	Ph	H	Br	D	—	—	434
o-MeOC <sub>6</sub> H <sub>4</sub>	Ph	H	Br	D	—	—	434

TABLE 2112A. UNSYMMETRICAL MONOMETHINE CYANINES



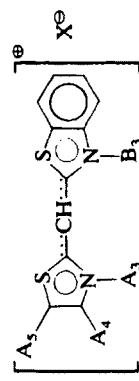
Y	A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	B <sub>3</sub>	B <sub>4</sub>	B <sub>5</sub>	X	Method	m.p. (°C)	λ (nm)	Rcf.
O	Et	Ph	Ph	Et	Ph	Ph	I	F	—	420	1(p. 69), 435
O	Bu	Ph	Ph	Et	Ph	Ph	ClO <sub>4</sub>	F	—	—	1(p. 69), 435
S	Me	Me	H	(CH <sub>2</sub> ) <sub>3</sub> -SO <sub>3</sub> <sup>-</sup>	Me	H	—	—	—	—	343 e
S	Me	Ph	H	Ph	Me	H	I	G	220	440	436
S	Me	Ph	H	p-MeC <sub>6</sub> H <sub>4</sub>	Me	H	I	G	215	440	436
S	Me	Ph	H	p-BrC <sub>6</sub> H <sub>4</sub>	Me	H	I	G	220	440	436
S	Me	Ph	H	o-MeC <sub>6</sub> H <sub>4</sub>	Me	H	I	G	243	440	436
S	Et	Me	H	Ph	Ph	H	I	D	222	—	437, 438
S	Et	Ph	Ph	Et	=O	=CHNHPH	EiSO <sub>4</sub>	—	232	—	440, 441, 439
S	Et	Ph	Ph	Allyl	=O	=CHNHPH	EiSO <sub>4</sub>	—	—	661	439, 440, 441
S	Et	Ph	Ph	Bu	Ph	Ph	ClO <sub>4</sub>	F	—	438	1(p. 69), 435
S	Ph	Ph	H	p-NH <sub>2</sub> SO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	Ph	H	Br	F	>300	—	442
S	p-MeC <sub>6</sub> H <sub>4</sub>	Ph	H	p-BrC <sub>6</sub> H <sub>4</sub>	Ph	H	Br	F	—	—	442
S	p-MeC <sub>6</sub> H <sub>4</sub>	Ph	H	p-ClC <sub>6</sub> H <sub>4</sub>	Ph	H	Br	F	—	—	442
S	p-MeC <sub>6</sub> H <sub>4</sub>	Ph	H	p-NH <sub>2</sub> SO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	Ph	H	Br	F	—	—	442

TABLE 2112B



A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	B <sub>3</sub>	X	Method	m.p. (°C)	λ (nm)	Ref.
Me	H	CH <sub>2</sub> OH	Et	I	—	256	414	379
Me	H	Br	Et	I	F	>320	423	112
Me	H	NH <sub>2</sub>	Et	I	F	254	457	112
Me	Me	H	Me	Br	F	—	—	1(p. 69), 60
Me	Me	H	Et	I	FG	276	—	416, 443, 444
Me	Me	COO <sup>-</sup>	Et	—	F	210	420	222
Me	Ph	H	Me	I	—	—	418	1(p. 72), 43, 80, 82
Et	H	H	Et	I	—	279	412	388
Et	H	Styryl	Et	I	F	257	634	357
Et	H	COMe	Et	OTs	F	235	434	112
Et	H	COOMe	Et	OTs	F	233	430	112
Et	H	COOEt	Et	I	F	202	428	112
Et	H	Ph	Et	I	F	277	437	354, 357, 388
Et	H	p-PhC <sub>6</sub> H <sub>4</sub>	Et	I	—	244	444	388
Et	H	p-(p-MeC <sub>6</sub> H <sub>4</sub> )C <sub>6</sub> H <sub>4</sub>	Et	I	—	268	447	388
Et	H	p-MeOC <sub>6</sub> H <sub>4</sub>	Et	I	—	257	440	388
Et	H	α-Naphthyl	Et	I	—	274	426	354, 445
Et	H	β-Naphthyl	Et	I	—	—	445	354
Et	H	α-Furyl	Et	I	—	267	445	354
Et	H	α-Benzo(b)furyl	Et	I	—	—	267	454
Et	H	β-Benzo(b)thienyl	Et	I	—	—	264	434
Et	H	2-Benzothiazolyl	Et	I	F	277	457	389

TABLE 2112B (Continued)

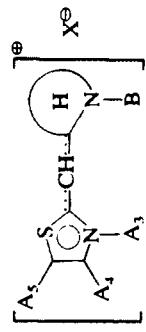


A <sub>3</sub>	A <sub>4</sub>	A <sub>3</sub>	B <sub>3</sub>	X	Method	m.p. (°C)	λ (nm)	Ref.
Et	H	NHCOMe	Et	G	—	430	446	
Et	H	NHCOMe	(CH <sub>2</sub> ) <sub>2</sub>	G	—	430	446, 447	
Et	H	OEt	COO <sup>a</sup>	—	222	425	388	
Et	Me	4-Thiazolyl	Et	F	276	436	356	
Et	Me	4-(2-Me-Thiazolyl)	Et	F	285	437	356	
Et	Styryl	H	Et	F	251	424	357	
Et	Ph	H	Et	F	238	419	114, 354, 357	
Et	p-MeOC <sub>6</sub> H <sub>4</sub>	Ph	Me	I	304	425	1(p. 69), 111	
Et	α-Naphthyl	H	Et	ClO <sub>4</sub>	—	228	419	354
Et	β-Naphthyl	H	Et	I	—	—	419	354
Et	2-Pyrrolyl	H	Et	F	262	417	358	
Et	α-Furyl	H	Et	I	—	248	419	354
Et	α-Benzo(b)furyl	H	Et	I	—	251	418	354
Et	α'-Thienyl	H	Et	I	—	251	417	354
Et	β-Benzo(b)thienyl	H	Et	I	—	243	417	354
Et	2-Benzothiazolyl	H	Et	ClO <sub>4</sub>	F	256	420	389
(CH <sub>2</sub> ) <sub>2</sub> SH	Ph	H	Ph	Br	—	—	63	
Ph	H	Ph	Et	ClO <sub>4</sub>	F	—	—	325
Ph	Me	H	Me	I	G	264	—	60
Ph	Ph	H	Me	I	D	—	—	437
Ph	H	H	Et	Br	D	—	—	1(p. 304), 437

Ph	H	I	D	271	—	437, 438
Ph	H	Et <sup>a</sup>	Br	298	—	438
Ph	H	Et <sup>a</sup>	I	D	—	437
Ph	CH <sub>2</sub> COO	Et <sup>a</sup>	Br	—	—	438
Ph	COOMe	Et <sup>a</sup>	Br	—	—	437
Ph	o-MeOC <sub>6</sub> H <sub>4</sub> -N=N-	Et <sup>a</sup>	Br	—	—	448
Ph	2-(6-EtOpenzo-thiazolyl-N=N-	Et <sup>a</sup>	Br	—	—	448
Me	Me	Et	I	G	—	117
H	H	Et	I	—	—	395
2-Benzothia-zoly		Me	Cl	F	210	—
6-(2Me-benzo-thiazolyl)	Me	H	Me	ClO <sub>4</sub>	F	240
6-(2Me-benzo-thiazolyl)	Me	H	—	—	—	118

<sup>a</sup> S-Cl-benzothiazole.

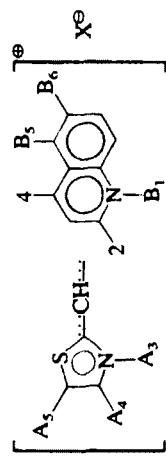
TABLE 2112C



H	A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	B	X	Method	m.p. (°C)	λ(nm)	Ref.
3,3-diMe-indolonyl	Me	Me	H	Me	I	F-G	244	414	449
3,3-diMe-indolonyl	Me	Ph	H	Me	I	F-G	244	417	449
3,3-diMe-indolonyl	Ph	Ph	H	Me	I	D	204	—	437, 438
3,3-diMe-indolonyl	Ph	OH	p-MeC <sub>6</sub> H <sub>4</sub> -N=N-	Et	Br	—	—	—	448
3,3-diMe-indolonyl	Et	H	H	Et	I	—	442	—	450
2-Pyridinyl	Et	Me	H	Me	I	F	—	—	1(p. 66), 451, 452
2-Pyridinyl	Et	Me	H	Et	I	F	447	—	453, 454
2-Pyridinyl	Et	Ph	H	Et	I	F	447	—	1(p. 53), 450, 452
1-Isquinolyl	Et	Me	H	Me	I	F	447	—	450
3-Benzofquinolyl	Et	H	H	Et	I	F	—	—	455
3-Benzofquinolyl	Et	Me	H	Et	I	F	268	483	1(p. 48), 450, 456, 457
3-Benzofquinolyl	Et	Ph	H	Et	I	—	—	—	1(p. 48), 450,
3-Benzofquinolyl	Et	Ph	H	Et	I	—	—	—	456, 457
2-(5-MeS-4,4-diMe-imidazolyl)	Me	Ph	H	Me	ClO <sub>4</sub>	F	246	—	458
2-(3,3-diMe-pyrrolo-(2,3- <i>b</i> )pyridyl	Me	Me	H	Et	I	G	203	—	459
2-(1-Me-benzimidazolyl)	Ph	Me	H	Me	I	G	240	235	436

2-Benzoxazolyl	Et	Me	H										
2-Benzoxazolyl	Ph	Me	H										
2-Benzoxazolyl	<i>p</i> -MeC <sub>6</sub> H <sub>4</sub>	Me	H										
2-Benzoxazolyl	<i>p</i> -BrC <sub>6</sub> H <sub>4</sub>	Me	H										
2-Benzoxazolyl	<i>o</i> -MeOC <sub>6</sub> H <sub>4</sub>	Me	H										
2-(5,6-diMe-benzoxazolyl)	Ph	OH	<i>p</i> -ClC <sub>6</sub> H <sub>4</sub> -N=N-										
2-Thiazolyl	Ph	Ph	H										
2-(4-Oxothiazolinyl)	Me	Me	H										
2-(4-Oxothiazolinyl)	Ph	Ph	Me										
2-(4-Oxo-5- $\alpha$ -MesS-propylidene-thiazolinyl)	Me	Ph	Me										
2- $\beta$ -Naphthothiazolyl	Ph	Ph	H										
2-Benzoselenazolyl	Me	Me	H										
2-Benzoselenazolyl	Ph	Ph	H										
2-Benzoselenazolyl	Ph	Ph	H										
2-(5-Me-benzo-selenazolyl)	Et	H	NHCOMe										
2-(5-MeO-benzo-selenazolyl)	Et	H	NHCOMe										
2-(5,6-diMe-benzoselenazolyl)	Et	H	NHCOMe										
2-(5,6-diMe-benzoselenazolyl)	Ph	OH	<i>p</i> -HO <sub>3</sub> SC <sub>6</sub> H <sub>4</sub> -N=N-										
9-(3-Et-2Me-imidazo(4,5-f)quinolyl)	Me	Me	H										

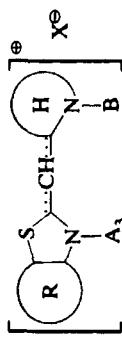
TABLE 2112D



2 or 4	A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	B <sub>1</sub>	B <sub>3</sub>	B <sub>6</sub>	X	Method	m.p. (°C)	λ (nm)	Ref.
2	Me	H	H	Et	H	H	I	F	—	—	456
2	Me	H	COMe	Et	H	H	I	F	282	489	112
2	Me	Me	H	Me	H	Me	OTs	F	—	—	1(p. 63), 60, 295
2	Me	H	Et	Et	H	H	I	F-G	—	472	443, 444, 450, 468
2	Me	Me	COO <sup>-</sup>	Et	H	Me	—	F	206	482	222
2	Me	COOEt	Me	Et	H	OMe	I	—	—	—	303
2	Me	Ph	H	Me	H	H	I	F	—	475	80, 83, 456
2	Me	Ph	Me	Et	H	H	I	F	—	—	456
2	Me	Ph	p-MeC <sub>6</sub> H <sub>3</sub> -S	Me	=O	H	I	—	484	467	1(p. 65), 111, 351
2	Me	Ph	COOEt	Et	H	H	Br	F	227	485	468
2	Me	$\alpha$ -Furyl	H	Et	H	H	I	F	—	—	1(p. 48), 386
2	Me	NH <sub>2</sub>	H	Et	H	H	I	F	270	454	1(p. 48), 111,
2	Et								267	465	387, 457, 469
2	Et	Me	H	Me	H	H	OTs	—	—	—	465
2	Et	Me	H	Et	H	H	I	F	—	472	1(p. 48), 111,
2	Et	Ph	H	Et	H	H	I	F	—	—	456, 457
2	Et	Ph	Me	Et	H	H	I	—	—	—	1(p. 48), 111,
2	Et	Ph	Ph	Me	H	H	I	F	271	490	453, 456, 457
2	Et	Ph	OPh	Me	H	H	I	F	258	480	111
										1(p. 65), 42, 111	

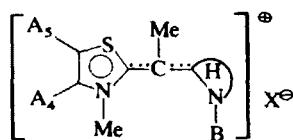
2	Et	<i>p</i> -MeOC <sub>6</sub> H <sub>4</sub>	Ph	Me	H	H	I	F	267	490
2	Et	3,4-diMeOC <sub>6</sub> H <sub>3</sub>	Ph	Me	H	H	I	F	227	485
2	Et	2-Pyrrolyl	H	Et	H	H	I	F	227	479
2	Et	$\alpha$ -Furyl	H	Et	H	OMe	I	F	—	358
2	Et	$\alpha$ -Furyl	H	Et	H	H	I	F	—	468
2	Et	$\alpha$ -Thienyl	H	Et	H	H	I	F	—	468
2	Et	$\alpha$ -Benzox(b)-thienyl	H	Et	H	H	I	F	—	110, 470
2	Et	$\alpha$ -Benzox(b)-thienyl	H	Et	H	Me	I	F	—	468, 471
2	Et	$\alpha$ -Benzox(b)-thienyl	H	Et	H	OMe	I	F	—	468, 471
2	Et	$\alpha$ -Benzox(b)-thienyl	H	Et	H	COOEt	I	F	—	468, 471
2	Et	CH(Me)Ph	Me	Me	H	H	Cl	F	166	480
2	Ph	Ph	Me	Me	H	H	I	F	230	—
2	Ph	Ph	OH	Et	H	H	Br	D	272	62, 394, 473
2	Ph	<i>p</i> -MeC <sub>6</sub> H <sub>4</sub>	Me	Me	H	H	Br	—	—	437, 438
2	Ph	<i>p</i> -BrC <sub>6</sub> H <sub>4</sub>	Me	Me	H	H	I	G	—	448
2	Ph	<i>o</i> -MeC <sub>6</sub> H <sub>4</sub>	Me	Me	H	H	I	G	201	480
2	Ph	<i>o</i> -MeOC <sub>6</sub> H <sub>4</sub>	Me	Me	H	H	I	G	292	480
4	Me	Ph	H	Me	H	H	I	G	249	480
4	Et	H	NHCOMe	Et	H	H	I	G	249	480
4	Et	H	NHCOMe	Et	H	Me	I	G	240	—
4	Et	Ph	H	Et	H	H	I	H	—	1(p. 39), 42
4	Et	Ph	H	Et	H	H	I	G	—	505
4	Et	Ph	NHCOMe	Et	H	Me	I	G	—	523
4	Et	Ph	NHCOMe	Et	H	Me	I	G	—	446
4	Et	Ph	H	Et	H	H	I	F	—	447
4	Et	Ph	H	Et	H	H	I	F	—	474
4	CH(Me)Ph	Me	H	Et	H	H	I	F	200	511
4	Ph	Me	H	Me	H	H	I	G	258	520
4	Ph	Ph	H	Me	H	H	I	D	301	—
4	Ph	<i>p</i> -MeC <sub>6</sub> H <sub>4</sub>	Me	Me	H	H	I	G	195	520
4	Ph	<i>p</i> -BrC <sub>6</sub> H <sub>4</sub>	Me	Me	H	H	I	G	275	520
4	Ph	<i>o</i> -MeC <sub>6</sub> H <sub>4</sub>	Me	Me	H	H	I	G	188	520
4	Ph	<i>o</i> -MeOC <sub>6</sub> H <sub>4</sub>	Me	Me	H	H	I	G	188	520

TABLE 2112E



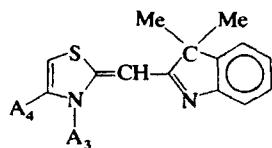
R	H	A <sub>3</sub>	B	X	Method	m.p. (°C)	λ (nm)	Ref.
$(\text{CH}_2)_n$	<i>n</i>							
	3	2-Quinolyl	Et	I	F	284	490	399
	4	4-(6-Me-quinolyl)	Me	$\text{ClO}_4^-$	F	—	515	400
	4	2-(7-Me-quinolyl)	Me	I	F	—	500	400
	4	2-Benzothiazolyl	$(\text{CH}_2)_2\text{SH}$	Et	Br	—	—	63
	4	2-Quinolyl	Me	I	F	240	484	401
(4-i-Pr, 7Me)	5	2-Quinolyl	Et	I	E	263	476	402
	<i>n</i>	<i>m</i>						
	1	2	2-Quinolyl	Me	I	F	280	—
	1	2	2-Quinolyl	Et	I	F	273	—
	3	0	2-Quinolyl	Me	I	F	265	500
	<i>n</i>							
	1	2-Benzothiazolyl	Me	$\text{ClO}_4^-$	F	238	523	352
	2	2-Quinolyl	Me	I	F	246	490	405
	2	2-Benzothiazolyl	Et	I	—	248	435	114
	<i>n</i>							
	2	2-Quinolyl	Et	I	F	268	468	404
	3	2-Quinolyl	Et	I	F	—	476	404

TABLE 2113. MONOMETHINE CYANINES WITH SUBSTITUENT ON THE CHAIN



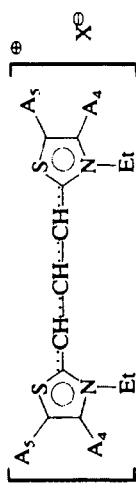
H	A <sub>4</sub>	A <sub>5</sub>	B	X	λ (nm)	Ref.
2-Quinolyl	H	H	Et	OTs	518	26
2-(4-Me-thiazolyl)	H	H	Me	OTs	448	26
2-Benzothiazolyl	H	H	Me	OTs	450	26
2-Benzothiazolyl	-CH <sub>2</sub> ) <sub>4</sub> -		Me	ClO <sub>4</sub>	—	400

TABLE 2114. CYANINE BASES



A <sub>3</sub>	A <sub>4</sub>	m.p. (°C)	λ (nm)	Ref.
Me	H	—	—	1(p. 361), 475
Me	Me	237	405	449
Me	Ph	182	405	449
Et	H	—	—	475

TABLE 2121A. SYMMETRICAL UNSUBSTITUTED TRIMETHINE CYANINES



$\text{A}_4$	$\text{A}_5$	X	Method	m.p. (°C)	$\lambda$ (nm)	Ref.
H	H	I	—	—	541	476
		F	223	542	1(p. 129), 16	
		A	—	543	110	
		—	—	545	113	
		—	—	548	79	
		—	—	553	477	
		A	—	555	387	
		A	—	580	478	
		—	—	—	479	
H	ClO <sub>4</sub>	—	—	—	—	—
H	Me	A	—	553	110, 477	
H	CH <sub>2</sub> OH	A	191	555	379	
H	Styryl	A	199	634	357	
H	COOEt	A	134	583	112	
H	Ph	A	—	592	17, 110	
H	Ph	A	—	592	17, 114, 354, 388,	
					480, 477	
H	p-PhC <sub>6</sub> H <sub>4</sub>	I	A	178	608	481
H	p-(p-NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> )C <sub>6</sub> H <sub>4</sub>	I	—	—	612	480
H	p-(p-MeOC <sub>6</sub> H <sub>4</sub> )C <sub>6</sub> H <sub>4</sub>	Cl	—	—	645	480
H	p-Me <sub>2</sub> NC <sub>6</sub> H <sub>4</sub>	I	A	198	612	388
H	p-Me <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> (ClH)	ClO <sub>4</sub>	—	—	623	17
H					608	17

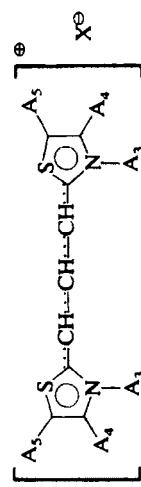
H	<i>p</i> -MeCONHC <sub>6</sub> H <sub>4</sub>	I	—	265	608	17
H	<i>p</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	I	—	230	650	480
H	<i>p</i> -HOC <sub>6</sub> H <sub>4</sub>	ClO <sub>4</sub>	—	266	600	17
H	<i>p</i> -MeOC <sub>6</sub> H <sub>4</sub>	I	A	190	612	17 1(p. 101), 17, 388, 445
H	<i>p</i> -PhOC <sub>6</sub> H <sub>4</sub>	Cl	A	250	606	388
H	<i>α</i> -Naphthyl	I	A	180	578	1(p. 101), 17, 388, 445
H	<i>β</i> -Naphthyl	I	A	135	580	445
H	4-Me- <i>α</i> -Naphthyl	OTs	A	249	610	1(p. 101), 354, 445, 481
H	5-Acenaphthyl	I	A	205	606	481
H	<i>α</i> -Furyl	ClO <sub>4</sub>	—	203	609	354
H	<i>α</i> -Thienyl	I	—	184	610	354
H	<i>α</i> -Benz(o/b)furyl	I	—	244	623	113, 354
H	<i>β</i> -Benz(o/b)thienyl	I	—	200	594	354
H	2-Benzothiazolyl	ClO <sub>4</sub>	A	267	630	389
H	MeCONH	ClO <sub>4</sub>	D	—	576	112
Me	H	I	—	—	548	79
Me	A	A	—	552	<i>I</i> <sup>3</sup>	16, 75 u, 109 i, 110, 387, 433, 466, 482, 483, 484, 485, 667
Me	H	<i>i</i> -nicotinate	—	193	—	360
Me	Me	I	A	—	563	110, 477
Me	CF <sub>3</sub> (CF <sub>2</sub> ) <sub>2</sub>	ClO <sub>4</sub>	A	256	—	487
Me	COMe	I	D	240	598	112
Me	4-Thiazolyl	ClO <sub>4</sub>	D	228	597	356
Me	4-(2-Me-thiazolyl)	ClO <sub>4</sub>	D	202	600	356
Me	NO <sub>2</sub>	I	—	—	640	480
i-Bu	Me	I	A	162	—	391

TABLE 2121A (Continued)

$A_4$	$A_5$	X	Method	m.p. (°C)	$\lambda$ (nm)	Ref.
$\left[ A_5 \begin{array}{c} S \\   \\ \text{---} \text{CH} \text{---} \text{CH} \text{---} \text{CH} \text{---} \text{N} \text{---} \text{Et} \\   \\ \text{---} \text{S} \text{---} \text{C} \text{---} \text{C} \text{---} \text{N} \text{---} \text{Et} \\   \\ \text{---} \text{A}_5 \end{array} \right]^\oplus X^\ominus$						
Tetradecyl	Me	I	—	119	—	391
Hexadecyl	Me	I	—	129	—	391
( <i>p</i> -MeOCOC <sub>6</sub> H <sub>4</sub> )CH <sub>2</sub>	Me	I	—	214	—	391
Phthalimidomethylene	H	I	—	241	545 I(p. 101), 488	
Styryl	H	I	A	239	572	357
CH <sub>2</sub> OH	H	ClO <sub>4</sub>	A	183	553	379
COOEt	H	I	D	173	542	112
Ph	H	I	A	240	559	16, 110, 114, 354, 477, 484, 486
Ph	Ph	I	A, F	247	588	1(p. 104), 110, 111, 477, 483, 489, 667
<i>p</i> -Xylyl	Ph	ClO <sub>4</sub>	F	225	592	1(p. 104), 111
<i>p</i> -PhC <sub>6</sub> H <sub>4</sub>	H	I	A	222	560	1(p. 104), 481, 490
<i>p</i> -PhC <sub>6</sub> H <sub>4</sub>	<i>p</i> -PhC <sub>6</sub> H <sub>4</sub>	I	A	155	602	481
<i>p</i> -( <i>p</i> -MeOC <sub>6</sub> H <sub>4</sub> )C <sub>6</sub> H <sub>4</sub>	H	Cl	A	262	565	388
<i>p</i> -MeOC <sub>6</sub> H <sub>4</sub>	H	I	A	240	560	388
<i>p</i> -MeOC <sub>6</sub> H <sub>4</sub>	Ph	I	F	181	590	1(p. 104), 111
3,4-diMeOC <sub>6</sub> H <sub>3</sub>	Ph	I	Cl	179	590	1(p. 104), 111
<i>p</i> -PhOC <sub>6</sub> H <sub>4</sub>	H	A	198	560	388	
<i>p</i> -CF <sub>3</sub> SO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	H	I	A	239	—	491
<i>o</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	H	I	—	—	—	492
<i>m</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	H	I	—	—	—	492

$\alpha$ -Naphthyl	H	I	A	190	560	1(p. 101), 354,
<i>p</i> -MeO- $\alpha$ -Naphthyl	H	Br	A	144	568	481, 490
$\beta$ -Naphthyl	H	I	A	215	560	1(p. 101), 354,
$\beta$ -Naphthyl	<i>p</i> -MeC <sub>6</sub> H <sub>4</sub> -S	I	F	194	585	1(p. 104), 111,
5-Acenaphthyl	H	ClO <sub>4</sub>	A	201	560	351
2-Pyrrolyl	H	I	A	199	555	481, 490
$\alpha$ -Furyl	H	I	A	234	563	358
$\alpha$ -Thienyl	H	I	—	—	559	354, 468, 493
$\alpha$ -Benzo( <i>b</i> )furyl	H	EtSO <sub>4</sub>	—	220	562	354
2-Benzothiazolyl	H	I	A	215	565	—
2-Benzothiazolyl	H	ClO <sub>4</sub>	—	264	—	389
NH <sub>2</sub>	H	I	A	235	526	1(p. 101), 386

TABLE 2121B



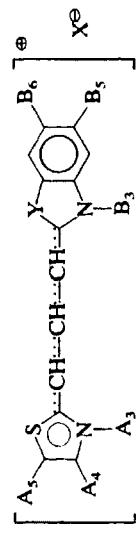
A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	X	Method	m.p. (°C)	λ (nm)	Ref.
Me	H	H	I	E, F	227	550	495
Me	H	Styryl	I	A	199	446	357
Me	H	Ph	I	—	592	17	
Me	H	p-Me <sub>2</sub> NCO <sub>2</sub> H <sub>4</sub>	ClO <sub>4</sub>	—	213	623	17
Me	H	p-Me <sub>2</sub> NCO <sub>2</sub> H <sub>4</sub> , CH	I	—	—	603	17
Me	H	p-NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	I	—	—	650	17
Me	H	p-HO-C <sub>6</sub> H <sub>4</sub>	—	—	—	620	17
Me	H	p-MeOC <sub>6</sub> H <sub>4</sub>	I	—	—	612	17
Me	H	Br	ClO <sub>4</sub>	F	183	566	112
Me	H	NH <sub>2</sub>	OTs	F	—	530–580	112
Me	H	Acetate	A	173	560	496	
Me	H	p-NH <sub>2</sub> salicylate	—	—	—	347, 348	
Me	H	ClO <sub>4</sub>	A	234	—	496	
Me	H	1,3-diCN-1,3-diCOOEt-propene	—	170	558	497	
Me	Me	H	I	A, C	247	550–556	1(p. 101, 110, 129), 17, 387, 482, 484, 485, 486, 496, 498, 499
Me	Me	H	I	E, F	268	—	495
Me	Me	H	3-Indoleacetate	—	218	—	347, 349
Me	Me	H	Resorcylate	—	243	—	346, 347, 349
Me	Me	H	1,2,3,4-Tetra-CN-butadiene	—	251	558	500
Me	Me	COOH	—	F	219	554–574	222
Me	Me	COOEt	I	—	—	—	501

Me	NO <sub>2</sub>	—	17
Me	H	A, F	638 560
Ph	H	—	1(p. 100), 43, 484, 486, 502
Ph	Ph	—	383, 429
Ph	p-MeC <sub>6</sub> H <sub>4</sub> S-	—	503
Ph	p-Ph-C <sub>6</sub> H <sub>4</sub>	—	585
Ph	p-NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	F	585
Me	p-BrC <sub>6</sub> H <sub>4</sub>	A	502
Me	H	224	502
Me	p-NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	A	502
Me	H	229	502
Me	p-MeOC <sub>6</sub> H <sub>4</sub>	—	502
Me	H	191	502
Me	p-MeOC <sub>6</sub> H <sub>4</sub>	—	502
Me	OPh	—	502
Me	H	183	502
Me	p-EtOC <sub>6</sub> H <sub>4</sub>	ClO <sub>4</sub>	580
Me	H	240	502
Me	p-Naphthyl	A	172
Me	β-Naphthyl	SO <sub>4</sub> Me	—
Me	H	—	586
Me	2-Pyrrolyl	A	—
Me	H	—	358
Me	α-Furyl	I	—
Me	H	—	385
Me	α-Furyl	COOEt	—
Me	H	A	468, 493
Me	α-Thienyl	I	—
Me	NH <sub>2</sub>	H	—
Me	2-Pyrrolyl	I	385
Pr	H	—	—
Allyl	Me	Br	—
Ph	Me	E, F	—
Ph	H	A	559
Ph	H	A	394, 473
Ph	H	A	561
Ph	H	A	16
Ph	H	—	561
Ph	p-PhC <sub>6</sub> H <sub>4</sub>	ClO <sub>4</sub>	566
Ph	Me	I	566
Ph	H	—	504
Ph	p-ClC <sub>6</sub> H <sub>4</sub>	Cl	570
Ph	Me	Cl	120
Ph	p-CH <sub>3</sub> OC <sub>6</sub> H <sub>4</sub>	ClO <sub>4</sub>	564
Ph	Me	Cl	120
Ph	p-Me <sub>2</sub> NC <sub>6</sub> H <sub>4</sub>	ClO <sub>4</sub>	565
Ph	α-Naphthyl	ClO <sub>4</sub>	504
Ph	α-Pyridyl	ClO <sub>4</sub>	504
Ph	2-Thiazolyl	ClO <sub>4</sub>	504
Ph	2-Benzothiazolyl	ClO <sub>4</sub>	504

TABLE 2121C

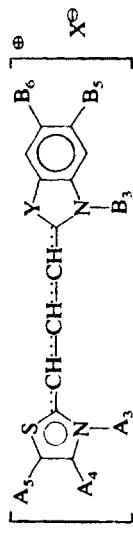
H	$A_3$	X	Method	m.p.		Ref.			
				(°C)	λ(nm)				
<i>n</i>									
	3	Et	I	A	258	580	399		
	4	Et	I	A	—	568	478		
	4	Me	$\text{ClO}_4$	A	216	570	16, 87		
	(4-i-Pr7-Me)	Et	I	C	238	572	401		
	5	Et	$\text{SO}_4\text{Et}$	A	208	566	402		
	5	Et	I	—	—	564	478		
<i>z      n      m</i>									
	0	1	2	Me	I	A	>300	—	406
	0	1	2	Et	I	C	—	570	87
	0	3	0	Et	I	—	170	580	407
	NMe <sub>2</sub>	3	1	Me	3I	—	230	575	505
	S	0	3	Et	$\text{ClO}_4$	F	215	575	87, 409
	S	1	2	Me	I	A	192	566	408
	S	1	2	Et	I	—	—	570	87
<i>n      m</i>									
	0	1	Et	I	F	—	637	352	
	0	2	Et	I	—	247	635	114	
	1	0	Me	$\text{ClO}_4$	A	>300	560	32, 352	
	2	0	Me	I	A	244	586	405	
	2	0	Et	I	A	244	586	405	
	2	0	Et	I	—	213	594	114	
<i>n</i>									
	2	Et	I	C	178	580	403		
	3	Me	I	A	193	570	404		

TABLE 2122A. UMSYMMETRICAL UNSUBSTITUTED TRIMETHINE CYANINES



Y	A <sub>1</sub>	A <sub>4</sub>	A <sub>5</sub>	B <sub>3</sub>	B <sub>5</sub>	B <sub>6</sub>	X	m.p. (°C)	λ(nm)	Ref.
MeCMe	Me	H	p-[5-(2,3-diMe-thiazolium)C <sub>6</sub> H <sub>4</sub> ]	Me	H	H	2ClO <sub>4</sub> -	205	562	506
MeCMe	Me	H	p-[4-(2,3-diMe-thiazolium)C <sub>6</sub> H <sub>4</sub> ]	Me	H	H	2ClO <sub>4</sub> -	220	538	506
MeCMe	Me	NH <sub>2</sub>	H	Me	H	H	I	244	528	1(p. 136), 386
MeCMe	Et	Me	H	Me	H	H	I	-	516	79
MeCMe	Et	Ph	H	(CH <sub>2</sub> ) <sub>2</sub> COO -	H	H	-	-	-	507
MeCMe	Ph	H	H	Me	H	H	ClO <sub>4</sub>	-	-	338
MeCMe	Ph	Me	H	Me	H	H	ClO <sub>4</sub>	-	534	121, 339
MeCMe	p-[5-(2,4-diMe-thiazolium)C <sub>6</sub> H <sub>4</sub> ]	Me	H	Me	H	H	2ClO <sub>4</sub> -	181	540	506
MeCMe	2-Thiazoly	Me	Me	Me	H	H	ClO <sub>4</sub>	-	-	116
MeCMe	2-Benzothiazoly	Mc	H	Me	H	CF <sub>3</sub>	ClO <sub>4</sub>	-	-	116
NEt	Mc	H	H	C <sub>3</sub> H <sub>6</sub> SO <sub>3</sub>	CF <sub>3</sub>	CF <sub>3</sub>	CN	-	-	471
NEt	Mc	H	H	C <sub>3</sub> H <sub>6</sub> SO <sub>3</sub>	CF <sub>3</sub>	Br	ClO <sub>4</sub>	-	-	508
NEt	Me	H	H	C <sub>3</sub> H <sub>6</sub> SO <sub>3</sub>	CF <sub>3</sub>	Cl	Br	-	-	471
NEt	Et	H	H	C <sub>3</sub> H <sub>6</sub> SO <sub>3</sub>	CF <sub>3</sub>	H	Cl	-	-	509
NEt	Et	H	H	C <sub>3</sub> H <sub>6</sub> SO <sub>3</sub>	CF <sub>3</sub>	Br	E	-	-	471
NEt	Et	H	H	C <sub>3</sub> H <sub>6</sub> SO <sub>3</sub>	CF <sub>3</sub>	H	E	-	-	509
NEt	Et	H	H	C <sub>3</sub> H <sub>6</sub> SO <sub>3</sub>	CF <sub>3</sub>	Br	E	-	-	508
NEt	Et	H	H	C <sub>3</sub> H <sub>6</sub> SO <sub>3</sub>	CF <sub>3</sub>	Cl	E	-	-	474
O	Me	Mc	H	C <sub>3</sub> H <sub>6</sub> SO <sub>3</sub>	CF <sub>3</sub>	Cl	E	-	-	508, 509
O	Me	Ph	H	EtOCH <sub>2</sub> COOH	H	OTs	-	-	-	510
O	Me	Ph	Ph	Me	H	H	I	-	-	520
O	Me	NH <sub>2</sub>	H	Me	H	H	I	-	-	525
O	Me	H	H	Me	H	H	F	240	429	1(p. 129), 386

TABLE 2122A (Continued)



<i>Y</i>	<i>A</i> <sub>3</sub>	<i>A</i> <sub>4</sub>	<i>A</i> <sub>5</sub>	<i>B</i> <sub>3</sub>	<i>B</i> <sub>4</sub>	<i>B</i> <sub>5</sub>	<i>B</i> <sub>6</sub>	<i>X</i>	Method	m.p. (°C)	λ (nm)	Ref.
O	Et	H	H	Et	Ph	H	I	—	—	—	—	511
O	Et	Me	H	Et	OH	H	I	F	253	—	—	512
O	Et	Me	H	Et	H	H	I	—	—	508	79	
O	Et	Me	H	Et	OCOMe	H	I	F	217	—	—	512
O	Et	NH <sub>2</sub>	H	Et	H	H	I	F	246	504	386	
O	Et	Me	H	Benzyl	H	H	Br	—	—	—	438	
O	Lauryl	Me	H	Me	H	Cl	I	E	262	—	—	368
O	Benzyl	Me	H	p-HOCO-	H	H	Br	—	—	—	—	368
O	<i>p</i> -HOCO-benzyl	Me	H	benzyl	Et	H	—	—	—	—	—	
O	(CH <sub>2</sub> ) <sub>5</sub> SO <sub>2</sub> NH <sub>2</sub>	Me	H	Et	H	H	—	F	—	517	513	
O	CH <sub>2</sub> CONHSO <sub>2</sub> Me	Me	H	Benzyl	H	H	I	F	—	514	513	
O	CH <sub>2</sub> COOCH <sub>2</sub> SO <sub>3</sub>	Me	H	Et	H	H	—	F	—	518	514	
S	Me	H	CH <sub>3</sub> OH	Et	OMe	ClO <sub>4</sub>	—	—	176	567	379	
S	Me	Me	H	Et	H	H	I	F	255	560	10, 129, 70, 135	
S	Me	Me	H	Et	OMe	Me	I	—	—	560	—	
S	Me	Me	CH <sub>3</sub> OH	Et	H	ClO <sub>4</sub>	—	—	167	560	379	
S	Me	Ph	H	Me	H	I	—	—	540	—	—	80, 85, 429
S	Me	Ph	Ph	Me	H	H	I	—	—	560	—	
S	Me	p-BrPh	H	Me	H	H	I	F	215	554	515	
S	Et	Me	H	Et	H	H	I	—	—	550	79	
S	Et	Me	H	Ph	H	H	I	—	—	246	538	504
S	Et	Ph	<i>p</i> -MeSC <sub>6</sub> H <sub>4</sub>	Et	H	H	I	F	195	560	10, 131, 111	
S	Et	α-Naphthyl	H	Et	H	H	ClO <sub>4</sub>	—	—	562	504	

S	Et	$\beta$ -Naphthyl	<i>p</i> -MeSC <sub>6</sub> H <sub>4</sub>	Et	H	H	CIO <sub>4</sub>	F	205	565	1(p. 131), 111
S	Et	Tropyl	H	Et	H	H	CIO <sub>4</sub>	F	221	555	516
S	Et	Tropyl	H	Et	Me	H	CIO <sub>4</sub>	F	218	560	516
S	Et	Tropyl	H	Et	OMe	H	CIO <sub>4</sub>	F	225	564	516
S	Et	$\alpha$ -Furyl	H	Et	H	H	I	F	—	—	468
S	Et	$\alpha$ -Benzofuryl	H	Et	H	H	I	F	—	—	468, 471
S	Et	Me	H	Et	H	H	I	F	188	—	1(p. 129), 135
S	n-Bu	Me	H	Et	H	H	I	F	207	—	1(p. 129), 135
S	i-amyl	Me	H	Et	H	H	I	—	566	121	
S	Ph	Me	H	Et	H	H	I	—	—	—	
S	Ph	Me	H	Et	H	H	CIO <sub>4</sub>	—	230	558	504
S	2-Benzothiazole	H	H	Et	H	H	I	—	—	—	395
S	6-(2-Me-Benzothiazole)	Me	H	Et	H	H	I	F	190	—	118
Se	Et	Tropyl	H	Et	H	H	CIO <sub>4</sub>	F	208	558	55
Se	Et	Tropyl	H	Et	OMe	H	CIO <sub>4</sub>	F	206	567	516
Se	Et	$\alpha$ -Benzofuryl	H	Et	H	H	I	F	—	—	516
Se	Et	Me	H	Et	H	H	I	F	—	554	79

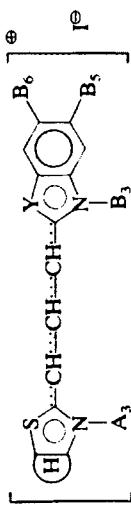
TABLE 2122B



H	A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	B	X	Method	m.p. (°C)	λ(nm)	Ref.
2-Pyrrolidinyl	Et	Me	Ph	Et	ClO <sub>4</sub>	G	—	—	517
2-Pyrrolidinyl	Et	Ph	Ph	Et	ClO <sub>4</sub>	G	186	—	517
2-(3-Me-pyrrolidinyl)	Me	2-thienyl	H	Me	ClO <sub>4</sub>	E	158	—	518
2-(1-Et-pyridyl)	Et	Me	H	Et	I	—	557	55	
3-(1-Isoindolonyl)	Me	Ph	Ph	Ph	ClO <sub>4</sub>	—	450	519	
2-Quinolyl	Me	Me	H	Et	I	F	280	580	1(p. 128), 70, 485, 520
2-Quinolyl	Me	Ph	H	Me	I	—	600	80, 82	
2-Quinolyl	Me	p-BrC <sub>6</sub> H <sub>4</sub>	H	Me	I	F	218	590	515
2-Quinolyl	Et	Me	H	Et	I	—	583	79	
2-Quinolyl	Et	Me	H	Et	ClO <sub>4</sub>	K	263	—	521
2-Quinolyl	Et	α-Benzo( <i>i</i> Pr)furyl	H	Et	I	F	—	468, 469	
2-Quinolyl	Allyl	Me	H	Et	I	F	220	—	1(p. 128), 135
2-Quinolyl	Ph	Me	H	Et	I	—	—	589	121
4-Quinolyl	Me	Me	H	Et	I	—	635	70	
4-Quinolyl	Me	Me	COO <sup>-</sup>	Et	—	F	196	610-	222
4-Quinolyl	Me	Ph	H	Me	I	—	—	648	
4-Quinolyl	Et	Me	H	Et	I	—	—	640	429
4-(2-Me-quinolyl)	Me	Ph	Me	Me	I	—	—	645	79
4-(6-MeS-quinolyl)	Me	Me	Me	Me	I	—	—	699	429
4-(6-MeS-quinolyl)	Me	Me	COOEt	Me	I	—	—	690	522, 523
4-(6-MeS-quinolyl)	Me	Ph	Me	Ph	I	—	—	695	522, 523
4-(6-MeS-quinolyl)	Me	Ph	Me	Ph	I	—	—	705	522, 523
4-(6-MeS-quinolyl)	Me	Ph	Ph	Ph	Ph	Ph	—	700	522, 523
4-(6-MeS-quinolyl)	Me	Ph	Ph	Ph	Ph	Ph	—	705	522, 523
4-(6-MeS-quinolyl)	Me	Ph	Ph	(CH <sub>2</sub> ) <sub>3</sub> SO <sub>3</sub> <sup>-</sup>	I	F	—	700	522, 523

4-(6-MeS-quinolyl)								
9-(10-Et-acridyl)	Et	Ph	Me	Et	Me	Et	—	522, 523
2-(4,4-di-Me-oxazolinyl)	Me	H	H	Et	H	Me	—	524
2-(4,4-di-Me-oxazolinyl)	Et	H	Ph	Me	Ph	ClO <sub>4</sub>	—	419
2-(4,4-di-Me-oxazolinyl)	Et	H	Ph	Me	Ph	ClO <sub>4</sub>	—	525
2-Oxazolyl	Et	H	H	Me	Me	ClO <sub>4</sub>	—	525
2-(4,4,6-triMe-4,5-dihydro-1,3-oxazinyl)	Et	H	H	Me	Me	ClO <sub>4</sub>	—	423
2-(6-Ph-1,3-oxazinyl)	Me	Ph	Ph	Me	Me	ClO <sub>4</sub>	—	496
2-(6-Ph-1,3-oxazinyl)	Et	H	H	Me	ClO <sub>4</sub>	F	—	424
3-(1,2-Benzoisoxazolyl)	Me	Mc	H	Et	Et	ClO <sub>4</sub>	—	424
3-(1,2-benzoisoxazolyl)	Benzyl	H	H	(CH <sub>2</sub> ) <sub>3</sub> SO <sub>3</sub>	—	E	255	527
2-Tetrahydrobenzoxazolyl	Et	Mc	H	Et	Et	—	302	515
2-(5,6-Tetramethylenebenzoxazolyl)	Benzyl	H	H	Et	Et	—	244	518
2-(6,7-Tetramethylenebenzoxazolyl)	Benzyl	Me	H	Me	I	—	206	525
2-Thiazolinyl	Et	Me	Me	Et	I	—	197	522
2-(4-Me-thiazolyl)	Me	Me	COO <sup>-</sup>	Et	—	—	493	79
2-(4-Me-thiazolyl)	Allyl	Me	H	Br	—	—	183	564
2-(4-Ph-thiazolyl)	Me	Ph	Ph	Me	I	—	219	—
$\alpha$ -Naphthothiazole	Me	Ph	H	Me	I	—	—	135
$\alpha$ -Naphthothiazole	Me	Ph	Ph	Me	I	—	—	224
$\alpha$ -Naphthothiazole	Et	Benzo(b)furyl	H	Et	—	—	—	224
$\alpha$ -Naphthothiazole	Me	H	CH <sub>2</sub> OH	Et	ClO <sub>4</sub>	—	—	468
$\beta$ -Naphthothiazole	Me	Me	H	Et	I	—	241	571
$\beta$ -Naphthothiazole	Me	CH <sub>2</sub> OH	H	Et	ClO <sub>4</sub>	—	—	379
$\beta$ -Naphthothiazole	Me	Ph	H	Me	I	—	570	70
$\beta$ -Naphthothiazole	Et	Me	Ph	Me	I	—	—	568
$\beta$ -Naphthothiazole	Et	Tropyl	H	Et	ClO <sub>4</sub>	—	236	568
$\beta$ -Naphthothiazole	Et	$\alpha$ -Furyl	H	Et	I	—	—	429
$\beta$ -Naphthothiazole	Et	$\alpha$ -Benzofuryl	H	Et	I	—	—	429
$\beta$ -Naphthothiazole	Et	$\alpha$ -Furyl	H	Et	I	—	—	111
4-[5,6- <i>b</i> ](3'-Et-2'-Me-thiazo)quinolyl]	Et	Ph	Et	ClO <sub>4</sub>	F	235	574	516
					I	—	—	—
					F	—	—	468
					I	—	—	471
					F	—	—	468
					I	—	—	655

TABLE 2122C



H	Y	A <sub>3</sub>	B <sub>3</sub>	B <sub>5</sub>	B <sub>6</sub>	Method	m.p. (°C)	λ(nm)	Ref.
<i>n</i>									
3	MeCMe	Me	Me	H	H	G	236	524	399
3	O	Me	Me	H	H	F	272	516	399
3	O	Me	Me	Me	Me	F	283	530	399
3	O	Me	Me	Ph	H	F	259	520	399
3	O	Me	Me	Cl	H	F	280	516	399
3	O	Et	Et	H	H	F	249	516	399
4	O	Et	Et	H	H	—	—	512	87
4	S	Et	Me	H	H	F	—	555	399
4	O	Et	Et	H	H	F	235	517	401
(4-i-Pr, 7-Me)									
5	O	Et	Et	H	H	F	236	516	402
<i>n</i>									
2	O	Me	Et	H	H	F	190	526	403
3	O	Et	Et	H	H	F	266	516	404
<i>z</i>	<i>n</i>	<i>m</i>							
0	1	2	MeCMe	Et	H	H	G	165	526
0	1	2	O	Me	Et	H	F	274	—
									406
									406

		$\text{Z}-\text{CH}_2-\text{CH}_2-$		$\text{Z}-\text{CH}_2-\text{CH}=\text{CH}-$		$\text{Z}-\text{CH}_2-\text{C}(\text{H})=\text{CH}-$		$\text{Z}-\text{CH}_2-\text{C}(\text{H})=\text{CH}_2-$		$\text{Z}-\text{CH}_2-\text{C}(\text{H})_2=\text{CH}_2-$	
<i>n</i>	<i>m</i>	Et	Et	Et	Et	Et	Et	Et	Et	Et	Et
0	1	0	Et	H	H	F		275	516		87, 406
0	1	2	O	Et	Me	F		263	526		406
0	1	2	O	Et	Ph	F		274	524		406
0	1	2	O	Et	Cl	H		292	516		406
0	1	2	O	Benzyl	H	H		277	518		406
0	1	2	O	Benzyl	H	H		232	528		407
0	3	0	O	Et	H	H					
0	3	0	O	Et	Ci	H					
S	0	3	O	Et	H	H		246	530		87, 409
S	1	2	O	Me	H	H		240	516		408
S	1	2	O	Et	H	H		—	520		87
S	1	2	O	Et	H	H		196	518		408

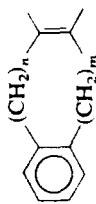
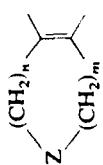
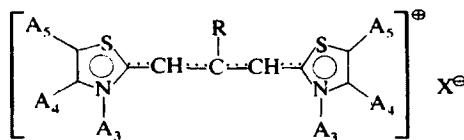
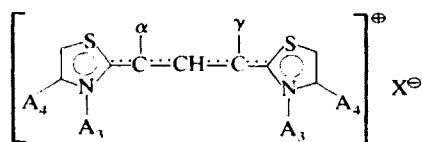


TABLE 2123A. SYMMETRICAL SUBSTITUTED TRIMETHINE CYANINES



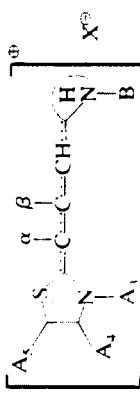
R	A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	X	Method	m.p. (°C)	λ (nm)	Ref.
Me	Me	Ph	H	I	B	—	—	1(p. 167), 484, 486, 530
Me	Et	H	H	I	B	174	553	445
Me	Et	H	Me	I	B	—	526	110
Me	Et	H	Ph	I	B	—	566	110
Me	Et	H	p-MeOC <sub>6</sub> H <sub>4</sub>	I	—	245	600	388
Me	Et	H	3,4-diMeOC <sub>6</sub> H <sub>3</sub>	I	—	—	578	531
Me	Et	H	β-Naphthyl	I	B	218	585	481
Me	Et	H	α-Benzofuryl	ClO <sub>4</sub>	—	226	—	354
Me	Et	Me	H	I	B	—	526	110
Me	Et	Me	Me	I	B	—	536	110
Me	Et	Ph	H	I	B	—	530	110
Me	Et	Ph	Ph	OTs	B	240	—	532
Me	Et	Ph	Ph	I	B	253	560	388, 481, 489
Me	Et	Ph	Ph	ClO <sub>4</sub>	B	—	562	110
Me	Et	p-MeOC <sub>6</sub> H <sub>4</sub>	Ph	I	—	249	561	111
Me	Et	5-Acenaphthyl	H	ClO <sub>4</sub>	B	200	533	490
Me	Et	α-Furyl	H	I	—	188	—	354, 468
Me	Et	α-Thienyl	H	I	B	—	—	470, 494
Me	Et	α-Benzofuryl	H	ClO <sub>4</sub>	—	218	—	354
Me	Ph	Me	H	I	B	—	548	394, 473
Et	Me	Ph	H	I	B	—	—	1(p. 167), 484, 486, 530
Et	Et	H	Me	I	B	—	530	110
Et	Et	H	Ph	I	B	—	570	110
Et	Et	H	p-MeOC <sub>6</sub> H <sub>4</sub>	ClO <sub>4</sub>	—	221	600	388
Et	Et	H	3-ME, 4-MeOC <sub>6</sub> H <sub>3</sub>	I	B	—	583	533
Et	Et	H	3,4-diMeOC <sub>6</sub> H <sub>3</sub>	I	B	—	578	531, 533
						—	583	531
Et	Et	H	α-Naphthyl	I	B	156	558	445
Et	Et	H	β-Naphthyl	I	B	231	585	481
Et	Et	H	5-Acenaphthyl	I	B	185	565	481
Et	Et	Me	H	I	B	—	530	1(p. 167), 110, 484 486
Et	Et	Me	4-Thiazolyl	I	B	240	570	356
Et	Et	Ph	H	I	B	—	534	110
Et	Et	Ph	Ph	I	B	—	564	110
					B	261	567	489
Et	Et	p-PhC <sub>6</sub> H <sub>4</sub>	p-PhC <sub>6</sub> H <sub>4</sub>	OTs	B	233	580	481
Et	Et	α-Furyl	H	I	B	—	—	354
Et	Et	α-Furyl	H	ClO <sub>4</sub>	—	182	—	354
Et	Et	α-Thienyl	H	I	B	—	—	470

TABLE 2123B



$\alpha$	$\gamma$	$A_3$	$A_4$	X	Method	m.p. (°C)	$\lambda$ (nm)	Ref.
CN	CN	Ph	Ph	Br	—	275	503	120
CN	CN	<i>p</i> -MeC <sub>6</sub> H <sub>4</sub>	Ph	Br	—	300	503	120
COMe	H	Me	Me	I	L	210	470	71
COMe	COMe	Me	Me	I	L	227	470	71
COPh	H	Me	Me	I	L	199	480	71
COPh	COPh	Me	Me	I	L	280	445-465	71

TABLE 2124A. UNSYMMETRICAL SUBSTITUTED TRIMETHINE CYANINES

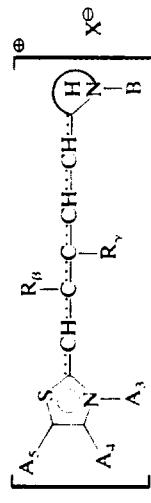


H	$\alpha$	$\beta$	A <sub>1</sub>	A <sub>2</sub>	A <sub>3</sub>	B	X	Method	m.p. (°C)	$\lambda$ (nm)	Ref.
3,3-di Me Indolonyl	CN	H	Ph	Ph	H	Me	Br	—	261	529	120
3,3-di Me Indolonyl	COMe	H	Ph	Me	Me	Me	Br	—	256	442	120
3,3-di Me Indolonyl	COMe	H	Ph	Ph	H	Me	Br	—	277	446	120
3,3-di Me Indolonyl	COOEt	H	Ph	Ph	H	Me	Br	—	210	490	120
2-quinolyl	PhNHCH=	H	Et	Me	H	Et	2ClO <sub>4</sub>	—	264	—	521, 534
2-quinolyl	COMe	H	Me	Me	H	Et	1	L	245	500	70, 71
4-quinolyl	PhNHCH=	H	Et	Me	H	Et	2ClO <sub>4</sub>	—	—	—	521, 534
4-quinolyl	COMe	H	Me	Me	H	Et	1	L	190	550	70, 71
2-thiazolinyl	PhNHCH=	H	Et	Me	H	Et	1	—	—	—	354
2-benzothiazolyl	H	SEt	Me	Me	H	Me	1	—	—	—	535
2-benzothiazolyl	COMe	H	Me	Me	Me	Et	1	L	251	465-470	70-71
2-benzothiazolyl	COMe	H	Ph	Me	H	Et	1	—	205	458	120
2-benzothiazolyl	COMe	H	Ph	Ph	H	Et	1	—	249	460	120
2-benzothiazolyl	COOEt	H	Ph	Ph	H	Et	1	—	Oil	520	120
2-(5-Me benzothiazolyl)	H	Me	Et	I-Azulenyl	H	Et	ClO <sub>4</sub>	H	210	544	516
2-(5-Me benzothiazolyl)	H	Et	Et	I-Azulenyl	H	Et	ClO <sub>4</sub>	H	209	552	516
2-(6-Me Benzothiazolyl)	COMe	H	Me	Me	H	Et	1	L	253	470-490	70
2-(5,6diMe benzothiazolyl)	H	Me	Et	COOEt	Me	Et	ClO <sub>4</sub>	—	—	555	536
2-(5,6diMe benzothiazolyl)	H	Et	Et	COOEt	Me	Et	ClO <sub>4</sub>	—	—	560	536
2-[6(5',6'diMeOC <sub>6</sub> H <sub>3</sub> )benzothiazolyl]	H	Et	Et	H	5,6-diMeOC <sub>6</sub> H <sub>3</sub>	Et	1	—	—	—	531
$\beta$ -naphthothiazolyl	H	Ph	Et	Ph	Ph	Et	ClO <sub>4</sub>	—	—	590	536
$\beta$ -naphthothiazolyl	COMe	H	Me	Me	H	Et	1	L	257	490-500	70-71

TABLE 2124B

H	n	Y	$\alpha$	$\beta$	A <sub>3</sub>	B <sub>3</sub>	Method	m.p. (°C)	$\lambda$ (nm)	Ref.	$\left[ \begin{array}{c} \text{S} \\   \\ \text{H} \\   \\ \text{N} \\   \\ \text{C} \\   \\ \text{C} \\   \\ \text{CH} \\   \\ \text{X} \\   \\ \text{N} \\   \\ \text{C}_6\text{H}_5 \\   \\ \text{I}^\ominus \end{array} \right]^\oplus$	
											A <sub>1</sub>	B <sub>1</sub>
$(\text{CH}_2)_n$	4	MeCMe	Me	Me	G	—	—	515	400			
	3	S	H	Me	Et	H	275	540	399			
	4	S	H	Me	Me	Et	238	540	401			
		(4-i-Pr, 7-Me)										
$(\text{CH}_2)_n$	1	S	H	Me	Et	H	265	540	537			
	2	S	H	Me	Et	H	250	550	405			
$(\text{CH}_2)_n$												

TABLE 213. PENTAMETHINE CYANINES



H	A <sub>1</sub>	A <sub>4</sub>	A <sub>5</sub>	R <sub>6</sub>	R <sub>β</sub>	R <sub>γ</sub>	B	X	Method	m.p.(°C)	λ(nm)	Ref.
2-quinolyl	Me	Me	COOEt	H	H	Et	I	B	—	—	1(p. 211), 538, 539, 540, 541	
2-quinolyl	Me	Ph	H	H	Me	Me	I	—	—	700	80, 82	
2-quinolyl	Me	p-BrC <sub>6</sub> H <sub>4</sub>	H	H	Me	Me	I	B	233	695	80, 515	
4-quinolyl	Me	Me	Me	H	Me	Me	I	B	—	—	1(p. 211), 538, 539	
2-thiazolyl	Me	H	H	H	Me	I	A	215	—	—	1(p. 213), 542, 543	
2-(4-Me thiazolyl)	Me	Me	H	H	Me	I	A	227	—	495	—	
2-(4-Me thiazolyl)	Et	Me	H	H	Et	I	E	241	—	—	544	
2-(4-Me thiazolyl)	Et	Me	H	H	Br	Et	I	C, D	194	637	222, 666, 667	
2-(4-Me thiazolyl)	Ph	Me	H	H	Ph	I	—	—	—	537	109	
2-(4-Me 5-COO thiazolyl)	Me	Me	COO <sup>-</sup>	H	Me	—	B	199	664	657	504	
2-(5-Ph thiazolyl)	Me	Ph	H	H	Me	I	—	—	—	222	—	
2-(4,5-diPh thiazolyl)	Me	Ph	Ph	H	Me	I	D	250	—	—	545, 546	
2-(4,5-diPh thiazolyl)	Et	Ph	Ph	H	Et	I	D	196	663	663	109, 433, 667	
2-[{4,5-(CH <sub>2</sub> ) <sub>3</sub> -N-CH <sub>2</sub> } <sup>+</sup> COOEt]	Et	{(CH <sub>2</sub> ) <sub>3</sub> -N-CH <sub>2</sub> } <sup>+</sup>	COOEt	H	Br	Et	I	—	—	—	547	

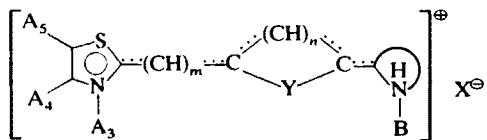
2-(4- $\alpha$ -furyl thiazoly)	Et	$\alpha$ -Furyl	H	H	Ci	Et	I	A	—	—	468
3-Benzisoxazolyl	Me	Me	H	H	H	Et	I	B	197	600	527
2-benzoazolyl	Et	—(CH <sub>2</sub> ) <sub>3</sub> —	H	H	H	Et	I	B	216	610	399
2-Benzothiazolyl	Me	Ph	H	H	H	Me	I	—	—	640	80, 82
2-Benzothiazolyl	Me	<i>p</i> -BrC <sub>6</sub> H <sub>4</sub>	H	H	H	Me	I	—	640	116, 120	
2-Benzothiazolyl	Me	<i>p</i> -EtOC <sub>6</sub> H <sub>4</sub>	H	H	H	Me	I	B	224	652	515
2-Benzothiazolyl	Et	H	Ph	H	H	Et	ClO <sub>4</sub>	—	—	640	116, 120
2(5,6-dimethylbenzothiazolyl)	Me	Ph	H	H	Me	Et	I	B	225	—	548
2-(5,6-diMeO-benzothiazolyl)	Me	Me	H	H	H	Et	I	B	—	665	1(p, 212), 538
2-(5,6-diMeO-benzothiazolyl)	Et	Me	H	H	H	Et	I	B	—	—	539
2-5,6-diMeO-benzothiazolyl)	Me	Me	H	H	H	Et	I	B	—	—	1(p, 212), 538
2-(5,6-diEtO-benzothiazolyl)	Et	Me	H	H	H	Et	I	B	—	—	539
2-(5,6-diEtO-benzothiazolyl)	Et	Me	H	H	H	Et	I	B	—	—	665
2-Benzoselenazolyl	Et	Me	H	H	H	Et	I	B	—	—	538
2-(5-MeO-Benzoselenazolyl)	Et	Ph	H	H	H	Et	I	B	—	660	1(p, 212), 538,
2-(5-EtO-Benzoselenazolyl)	Me	Me	H	H	H	Et	I	B	—	—	539, 540, 541
2-(5-EtO-Benzoselenazolyl)	Et	Me	H	H	Me	Et	I	B	—	650	540, 541
$\alpha$ -Naphthothiazolyl	Me	Me	COOEt	H	H	Et	I	B	—	655	540, 541
$\alpha$ -Naphthothiazolyl	Et	Me	COOEt	H	H	Et	I	B	—	—	540, 541
$\beta$ -Naphthothiazolyl	Et	—(CH <sub>2</sub> ) <sub>2</sub> O-CH <sub>2</sub> —	H	H	Et	I	B	—	675	538	
								B	205	675	406

TABLE 214. HEPTAMETHINE CYANINES



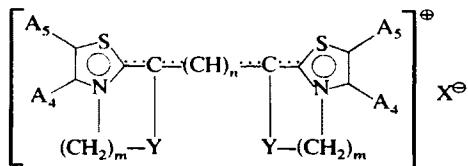
H	A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	B	X	Method	m.p. (°C)	λ (nm)	Ref.
2-Quinolyl	Me	Ph	H	Me	I	—	790	80, 82	
2-Quinolyl	Me	<i>p</i> -BrC <sub>6</sub> H <sub>4</sub>	H	Me	I	C	785	80, 82, 515	
2-Thiazolyl	Me	H	H	Me	I	A, B	174	—	1(p. 258), 493, 498, 542, 543, 549, 550
2-(4-Me-thiazolyl)	Me	Me	H	Me	I	B	155	—	365
2-(4-Ph-thiazolyl)	Et	Ph	Ph	Et	I	—	760	—	1(p. 258), 493, 498, 542, 543, 549, 550
2-(4-α-Furylthiazolyl)	Et	α-Furyl	H	Et	OTs	B	—	—	26
2-(4-α-Benzo(b)furylthiazolyl)	Et	α-Benzo(b)-furyl	H	Et	OTs	B	—	—	468, 471
2-Benzothiazolyl	Me	Ph	H	Me	I	—	740	80, 82	
2-Benzothiazolyl	Me	<i>p</i> -BrC <sub>6</sub> H <sub>4</sub>	H	Me	I	C	202	758	515
2-Benzothiazolyl	Me	<i>p</i> -EtOC <sub>6</sub> H <sub>4</sub>	H	Me	I	—	—	80, 82	
2-(5,6-diMe-benzothiazolyl)	Et	Me	H	Et	I	B, C	—	—	1(p. 253), 551-553
2-(5-Et <sub>2</sub> N-benzothiazolyl)	Et	Me	H	Et	ClO <sub>4</sub>	B	—	—	1(p. 253), 551-553
2-Benzoselenazolyl	Me	Ph	H	Et	I	—	—	—	1(p. 253), 551-553
2-(5-MeO-benzoselenazolyl)	Me	Ph	H	Et	—	C	—	—	553

TABLE 2151. CHAIN-BRIDGED CYANINES



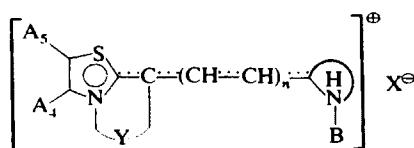
H	Y	<i>m</i>	<i>n</i>	A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	B	X	Method	m.p. (°C)	λ (nm)	Ref.
2-(4-Me-thiazolyl)	(CH <sub>2</sub> ) <sub>2</sub>	0	1	Et	Me	H	Et	OTs	A	—	595	I(p. 272), 122
2-(4-styryl-5-Ph-thiazolyl)	(CH <sub>2</sub> ) <sub>2</sub>	0	1	Me	Styryl	Ph	Me	I	A, B, C,	124	612	503
2-(4-Ph-thiazolyl)	(CH <sub>2</sub> ) <sub>2</sub>	0	1	Et	Ph	H	Et	OTs	A	—	596	I(p. 272), 122
2-(4-Ph-thiazolyl)	(CH <sub>2</sub> ) <sub>2</sub>	0	1	<i>n</i> -Pr	Ph	H	<i>n</i> -Pr	OTs	A	—	600	I(p. 272), 122
2-(4-Ph-thiazolyl)	(CH <sub>2</sub> ) <sub>2</sub>	0	1	<i>n</i> -Bu	Ph	H	<i>n</i> -Bu	OTs	B	—	600	I(p. 272), 122
2-(4,5-diPh-thiazolyl)	(CH <sub>2</sub> ) <sub>2</sub>	0	1	Me	Ph	Ph	Me	I	A, B, C,	206	611	503
2-(4,5-diPh-thiazolyl)	(CH <sub>2</sub> ) <sub>2</sub>	0	1	Et	Ph	Ph	Et	I	A, B, C,	199	623	503
2-Benzothiazolyl	CH <sub>2</sub>	0	1	Me	Ph	Ph	Me	ClO <sub>4</sub>	—	201	556	554
2-Benzothiazolyl	(CH <sub>2</sub> ) <sub>2</sub>	0	1	Me	Ph	Ph	Me	ClO <sub>4</sub>	—	230	596	554
2-Benzothiazolyl	O	1	2	Et	H	Ph	Et	I	—	240	—	548

TABLE 2152A. N CHAIN-BRIDGED CYANINES



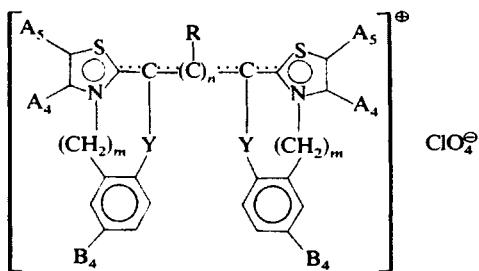
<i>n</i>	Y	<i>m</i>	A <sub>4</sub>	A <sub>5</sub>	X	m.p. (°C)	λ (nm)	Ref.
1	—	2	Me	H	ClO <sub>4</sub>	265	566	555
1	—	2	Ph	H	ClO <sub>4</sub>	215	573	555
1	—	2	Ph	Ph	I	262	608	555
1	—	3	Ph	Ph	ClO <sub>4</sub>	262	615	555
1	—	4	Ph	Ph	ClO <sub>4</sub>	232	609	555
1	O	2	Me	H	Br	244	630	411
1	O	2	Ph	H	ClO <sub>4</sub>	189	638	411
1	S	2	Me	H	ClO <sub>4</sub>	249	622	411
1	S	2	Ph	H	Br	249	630	411
3	—	3	Ph	Ph	ClO <sub>4</sub>	233	707	555
3	O	2	Ph	H	ClO <sub>4</sub>	192	730	411
3	S	2	Ph	H	ClO <sub>4</sub>	249	722	411

TABLE 2152B



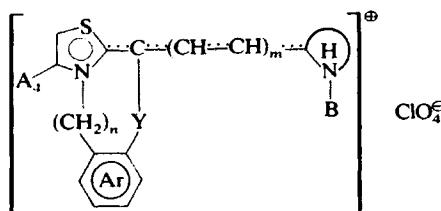
H	n	Y	A <sub>4</sub>	A <sub>5</sub>	B	X	m.p. (°C)	λ(nm)	Ref.
2-(3,3-diMe-indoleny)	1	(CH <sub>2</sub> ) <sub>2</sub>	Ph	Ph	Me	I	207	540	410
2-(3,3-diMe-indoleny)	1	(CH <sub>2</sub> ) <sub>3</sub>	Ph	Ph	Me	ClO <sub>4</sub>	256	548	410
2-(3,3-diMe-indoleny)	1	(CH <sub>2</sub> ) <sub>2</sub> -O-	Ph	H	Me	ClO <sub>4</sub>	235	554	411
2-(3,3-diMe-indoleny)	1	(CH <sub>2</sub> )-S-	Ph	H	Me	ClO <sub>4</sub>	250	564	411
2-Quinolyl	1	(CH <sub>2</sub> ) <sub>2</sub>	Ph	Ph	Et	I	292	606	410
2-Quinolyl	1	(CH <sub>2</sub> ) <sub>3</sub>	Ph	Ph	Et	I	301	610	410
2-Quinolyl	1	(CH <sub>2</sub> ) <sub>2</sub> -O-	Me	H	Et	ClO <sub>4</sub>	245	617	411
2-Quinolyl	1	(CH <sub>2</sub> ) <sub>2</sub> -S-	Me	H	Et	ClO <sub>4</sub>	246	610	411
2-(6-Me-quinolyl)	0	(CH <sub>2</sub> ) <sub>2</sub>	Ph	Ph	Et	I	261	521	555
2-(6-Me-quinolyl)	0	(CH <sub>2</sub> ) <sub>3</sub>	Ph	Ph	Et	I	227	572	555
2-(6-OMe-quinolyl)	0	(CH <sub>2</sub> ) <sub>2</sub>	Me	H	Et	I	229	506	555
2-(6-OMe-quinolyl)	0	(CH <sub>2</sub> ) <sub>2</sub>	Ph	Ph	Et	I	242	539	555
2-(6-OMe-quinolyl)	0	(CH <sub>2</sub> ) <sub>3</sub>	Me	H	Et	I	—	542	555
2-(6-OMe-quinolyl)	0	(CH <sub>2</sub> ) <sub>3</sub>	Ph	H	Et	I	154	549	555
2-(6-OMe-quinolyl)	0	(CH <sub>2</sub> ) <sub>3</sub>	Ph	Ph	Et	I	221	578	555
2-(6-OMe-quinolyl)	0	(CH <sub>2</sub> ) <sub>4</sub>	Ph	Ph	Et	I	—	596	555
2-(4-Me-thiazolyl)	1	CH=C(Ph)-	Me	H	Y	ClO <sub>4</sub>	194	597	40
2-Benzoxazolyl	1	(CH <sub>2</sub> ) <sub>2</sub>	Ph	Ph	Me	I	279	525	410
2-Benzoxazolyl	1	(CH <sub>2</sub> ) <sub>3</sub>	Ph	Ph	Me	I	268	535	410
2-Benzothiazolyl	0	(CH <sub>2</sub> ) <sub>2</sub>	Ph	Ph	Me	I	289	452	555
2-Benzothiazolyl	1	(CH <sub>2</sub> ) <sub>2</sub>	Ph	Ph	Et	I	265	563	410
2-Benzothiazolyl	1	(CH <sub>2</sub> ) <sub>3</sub>	Ph	Ph	Et	I	280	574	410
2-Benzothiazolyl	1	(CH <sub>2</sub> ) <sub>2</sub> -O-	Me	H	Et	ClO <sub>4</sub>	237	568	411
2-Benzothiazolyl	1	(CH <sub>2</sub> ) <sub>2</sub> -S-	Me	H	Et	ClO <sub>4</sub>	228	578	411

TABLE 2152C



<i>n</i>	R	Y	<i>m</i>	B <sub>4</sub>	A <sub>4</sub>	A <sub>5</sub>	m.p. (°C)	λ (nm)	Ref.
1	H	—	—	H	Me	H	304	552	415
1	H	—	2	H	Me	H	—	580	412
1	H	—	2	H	Ph	H	174	610	412
1	H	CH <sub>2</sub>	—	H	Ph	H	229	634	556
1	H	CH <sub>2</sub>	1	H	H	H	140	599	412
1	H	CH <sub>2</sub>	1	H	Me	H	196	606	412
1	H	CH <sub>2</sub>	1	H	Ph	H	146	613	412
1	H	CH <sub>2</sub>	1	H	Ph	Me	174	620	412
1	H	CH <sub>2</sub>	1	H	Ph	Ph	212	643	412
1	H	CH <sub>2</sub>	—	Me	Ph	H	224	637	556
1	H	CH <sub>2</sub>	—	OH	Ph	H	257	637	556
1	H	(CH <sub>2</sub> ) <sub>2</sub>	—	H	Me	H	—	600	556
1	H	O	—	H	Me	H	234	756	121, 413
1	H	S	—	H	Me	H	241	654	121, 413, 414, 557
1	Me	S	—	H	Me	H	132	620	557
1	Et	S	—	H	Me	H	168	620	557
1	H	Se	—	H	Me	H	—	626	413, 414
1	H	-N-CH <sub>2</sub> -COMe	—	H	Me	H	—	586	413, 414
3	H	—	—	H	Me	H	305	668	415
3	H	CH <sub>2</sub>	—	H	Ph	H	230	733	556
3	H	CH <sub>2</sub>	1	H	Me	H	205	680	412
3	H	CH <sub>2</sub>	1	H	Ph	H	190	686	412
3	H	O	—	H	Me	H	—	845	412

TABLE 2152D

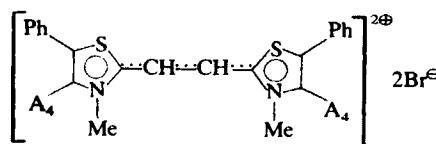


H	Ar	n	Y	m	A <sub>4</sub>	B	m.p. (°C)	λ(nm)	Ref.
2-(3,3-diMe-indolenyl)	1,2-Benzo	0	—	1	Me	Me	252	556	415
2-(3,3-diMe-indolenyl)	1,2-Benzo	0	CH <sub>2</sub>	1	Ph	Me	205	581	556
2-(3,3-diMe-indolenyl)	1,2-Benzo	0	(CH <sub>2</sub> ) <sub>2</sub>	1	Me	Me	—	525	413, 414
2-(3,3-diMe-indolenyl)	1,2-Benzo	0	O	1	Me	Me	265	611	121
2-(3,3-diMe-indolenyl)	1,2-Benzo	0	S	1	Me	Me	197	580	121, 413, 414, 557
2-(3,3-diMe-indolenyl)	1,2-Benzo	0	Se	1	Me	Me	—	576	413, 414
2-(3,3-diMe-indolenyl)	1,2-Benzo	0	N(AC)CH <sub>2</sub>	1	Me	Me	—	542	200, 201
2-(3,3-diMe-indolenyl)	1,2-Benzo	1	CH <sub>2</sub>	1	Me	Me	234	534	412
2-(3,3-diMe-indolenyl)	1,2-Benzo	2	—	1	Me	Me	246	505	412
2-(3,3-diMe-indolenyl)	1,2-Naphtho	0	O	1	H	Me	250	614	121
2-(3,3-diMe-indolenyl)	1,2-Naphtho	0	O	1	Me	Me	—	619	121, 413, 414
2-Quinolyl	1,2-Benzo	0	—	1	Me	Et	298	566	415
2-Quinolyl	1,2-Benzo	0	CH <sub>2</sub>	1	Ph	Et	209	616	556
2-Quinolyl	1,2-Benzo	0	(CH <sub>2</sub> ) <sub>2</sub>	1	Me	Et	—	602	413, 414
2-Quinolyl	1,2-Benzo	0	O	1	Me	Et	—	674	413, 414
2-Quinolyl	1,2-Benzo	0	S	1	Me	Me	205	630	557
2-Quinolyl	1,2-Benzo	0	S	1	Me	Et	—	—	412
2-Quinolyl	1,2-Benzo	0	S	1	Me	Ph	170	645	557
2-Quinolyl	1,2-Benzo	0	S-CH <sub>2</sub> -	1	Me	Me	214	586	557
2-Quinolyl	1,2-Benzo	0	S-CH <sub>2</sub> -	1	Me	Ph	193	630	557
2-Quinolyl	1,2-Benzo	0	Se	1	Me	Et	—	614	413, 414
2-Quinolyl	1,2-Benzo	0	Se	2	Me	Et	211	726	413, 414
2-Quinolyl	1,2-Benzo	0	N(AC)CH <sub>2</sub>	1	Me	Et	—	604	413, 414
2-Quinolyl	1,2-Benzo	0	CH <sub>2</sub>	1	Me	Et	243	600	412
2-Quinolyl	1,2-Benzo	0	O	1	H	Et	246	682	121, 413, 414
2-Quinolyl	2,1-Naphtho	0	O	1	Me	Et	—	674	413, 414
2-Quinolyl	2,3-Naphtho	0	S	1	Me	Et	—	—	412
2-(6-OMe-quinolyl)	1,2-Benzo	0	CH <sub>2</sub>	0	Ph	Et	297	562	556
2-Thiazolyl	1,2-Benzo	0	S-CH <sub>2</sub>	1	Me	—	—	582	557
2-(4-Me-thiazolyl)	1,2-Benzo	0	—	1	Me	Ph	205	542	415
2-Benzoxazolyl	1,2-Benzo	0	—	1	Me	Et	>360	515	415
2-Benzoxazolyl	1,2-Benzo	0	O	1	Me	Et	—	644	413, 414
2-Benzoxazolyl	1,2-Benzo	0	Se	1	Me	Et	—	570	413, 414
2-Benzothiazolyl	1,2-Benzo	0	—	0	Me	Et	245	438	415
2-Benzothiazolyl	1,2-Benzo	0	—	1	Me	Et	287	553	415
2-Benzothiazolyl	1,2-Benzo	0	CH <sub>2</sub>	0	Ph	Et	218	481	556
2-Benzothiazolyl	1,2-Benzo	0	CH <sub>2</sub>	1	Ph	Et	212	596	556
2-Benzothiazolyl	1,2-Benzo	0	(CH <sub>2</sub> ) <sub>2</sub>	1	Me	Et	—	560	413, 414
2-Benzothiazolyl	1,2-Benzo	0	O	0	Me	Et	244	502	121
2-Benzothiazolyl	1,2-Benzo	0	O	1	Me	Et	247	644	121, 413, 414

TABLE 2152D (Continued)

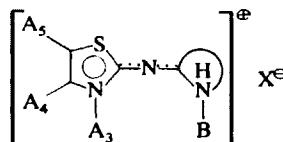
H	Ar	n	Y	m	A <sub>4</sub>	B	m.p. (°C)	λ(nm)	Ref.
2-Benzothiazolyl	1,2-Benzo	0	S	0	Me	Me	200	421	556
2-Benzothiazolyl	1,2-Benzo	0	S	0	Me	Et	—	421	121
2-Benzothiazolyl	1,2-Benzo	0	S	1	Me	Me	85	609	121
2-Benzothiazolyl	1,2-Benzo	0	S	1	Me	Et	—	609	121
2-Benzothiazolyl	1,2-Benzo	0	SCH <sub>2</sub>	1	Me	Me	182	549	557
2-Benzothiazolyl	1,2-Benzo	0	Se	1	Me	Et	—	600	413, 414
2-Benzothiazolyl	1,2-Benzo	0	Se	2	Me	Et	192	668	413, 414
2-Benzothiazolyl	1,2-Benzo	0	N(AC)CH <sub>2</sub>	1	Me	Et	—	565	413, 414
2-Benzothiazolyl	1,2-Benzo	1	CH <sub>2</sub>	1	Me	Et	237	556	412
2-Benzothiazolyl	1,2-Benzo	2	—	1	Me	Et	243	565	412
2-Benzothiazolyl	2,1-Naphtho	0	O	1	H	Et	238	648	121
2-Benzothiazolyl	2,1-Naphtho	0	O	1	Me	Et	—	646	413, 414

TABLE 216. BISCATIONIC DYES



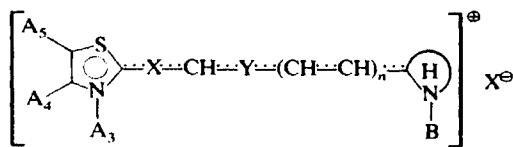
A <sub>4</sub>	m.p. (°C)	Ref.
Me	268	558
Ph	257	558

TABLE 2171A. MONOAZACYANINES



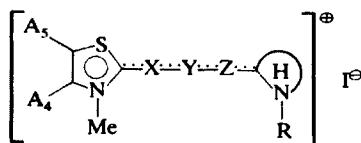
H	A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	B	X	m.p. (°C)	λ(nm)	Ref.
2-Quinolyl	Me	Me	H	Me	I	—	—	374(1)
2-Quinolyl	Me	Ph	H	Me	I	247	405	80, 82
2-Quinolyl	Et	H	H	Et	I	239	395	1(p. 385), 559
2-(4-Me-thiazolyl)	Et	Me	H	Ph	I	190	—	560
2-(4-Ph-thiazolyl)	Me	Ph	H	Me	I	187	390	429
2-(4,5-diPh-thiazolyl)	Me	Ph	Ph	Me	I	232	400	429
2-(4-OH-thiazolyl)	Me	Mc	H	Ph	Br	—	—	1(p. 387), 561
2-(4-OH-thiazolyl)	Me	Ph	Me	Ph	Br	—	—	1(p. 387), 561 562
2-Benzothiazolyl	Me	Ph	H	Me	I	168	390	429
						246	375	80, 82
2-Benzothiazolyl	Me	Ph	Ph	Me	I	209	410	429

TABLE 2171B



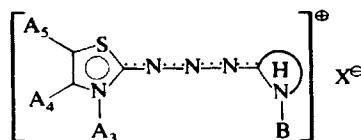
H	X	Y	n	A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	B	m.p. (°C)	λ (nm)	Ref.
2-Pyridyl	N	CH	0	Et	Me	H	Et	161	413	79
2-(3,3-diMe-indolenyl)	N	CH	0	Et	Me	H	Me	211	469	79
2-Quinolyl	CH	N	0	Et	Me	H	Et	230	460	79
2-Quinolyl	N	CH	0	Me	Ph	H	Me	217	520	80, 82
2-Quinolyl	N	CH	0	Et	Me	H	Et	225	500	79
2-Quinolyl	N	CH	1	Me	Ph	H	Me	195	590	80, 82
2-Quinolyl	N	CH	1	Me	p-BrC <sub>6</sub> H <sub>4</sub>	H	Me	—	590	80, 82
2-Quinolyl	N	CH	2	Me	Ph	H	Me	210	670	80, 82
2-Quinolyl	N	CH	2	Me	p-BrC <sub>6</sub> H <sub>4</sub>	H	Me	—	670	80, 82
4-Quinolyl	N	CH	0	Me	Ph	H	Me	205	500	429
4-Quinolyl	N	CH	0	Et	Me	H	Et	219	534	79
4-(2-Me-quinolyl)	N	CH	0	Me	Ph	Ph	Me	215	530	429
2-Thiazolinyl	N	CH	0	Et	Me	H	Et	250	427	79
2-(4-Me-thiazolyl)	N	CH	0	Et	Me	H	Et	220	449	79
2-(4-Ph-thiazolyl)	CH	N	1	Me	Ph	H	Me	230	555	429
2-(4-Ph-thiazolyl)	N	CH	0	Me	Ph	H	Me	198	455	429
2-(4-Ph-thiazolyl)	N	CH	0	Me	Ph	Ph	Me	215	465	429
2-Benzoxazolyl	N	CH	0	Me	Ph	H	Me	225	450	429
2-Benzoxazolyl	N	CH	0	Me	Ph	Ph	Me	210	455	429
2-Benzoxazolyl	N	CH	0	Et	Me	H	Et	203	439	79
2-Benzothiazolyl	CH	N	0	Et	Me	H	Et	257	448	79
2-Benzothiazolyl	N	CH	0	Me	Ph	H	Me	240	470	80, 82 492
2-Benzothiazolyl	N	CH	0	Me	Ph	Ph	Me	228	485	429
2-Benzothiazolyl	N	CH	0	Et	Me	H	Et	252	472	79
2-Benzothiazolyl	N	CH	1	Me	Ph	H	Me	160	565	80, 82
2-Benzothiazolyl	N	CH	1	Me	p-BrC <sub>6</sub> H <sub>4</sub>	H	Me	185	570	80, 82
2-Benzothiazolyl	N	CH	1	Me	p-MeOC <sub>6</sub> H <sub>4</sub>	H	Me	135	—	80
2-Benzothiazolyl	N	CH	1	Me	p-EtOC <sub>6</sub> H <sub>4</sub>	H	Me	175	560	80, 82
2-Benzothiazolyl	N	CH	2	Me	Ph	H	Me	175	655	80, 82
2-Benzothiazolyl	N	CH	2	Me	p-EtOC <sub>6</sub> H <sub>4</sub>	H	Me	212	650	80, 82
2-Benzoselenazolyl	N	CH	0	Et	Me	H	Et	263	473	79
2-α-Naphthothiazolyl	CH	N	0	Me	Ph	H	Me	205	460	429
2-α-Naphthothiazolyl	N	CH	0	Me	Ph	H	Me	248	475	429
2-α-Naphthothiazolyl	N	CH	0	Me	Ph	Ph	Me	198	500	429
2-β-Naphthothiazolyl	CH	N	0	Me	Ph	H	Me	240	468	429
2-β-Naphthothiazolyl	N	CH	0	Me	Ph	H	Me	240	475	429
2-β-Naphthothiazolyl	N	CH	0	Me	Ph	Ph	Me	200	495	429
2-β-Naphthothiazolyl	N	CH	0	Et	Me	H	Et	239	489	79

TABLE 2172. DIAZACYANINES



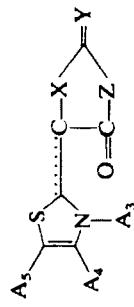
H	R	X	Y	Z	A <sub>4</sub>	A <sub>5</sub>	m.p. (°C)	λ (nm)	Ref.
2-(4-Ph-thiazolyl)	Me	CH	N	N	Ph	H	235	494	429
2-(4-Ph-thiazolyl)	Me	CH	N	N	Ph	Ph	140	495	429
2-(4-Ph-thiazolyl)	Me	N	CH	N	Ph	H	200	405	429
2-(4-Ph-thiazolyl)	Me	N	CH	N	Ph	Ph	200	408	429
2-(4,5-diPh-thiazolyl)	Me	CH	N	N	Ph	H	220	500	429
2-(4,5-diPh-thiazolyl)	Me	CH	N	N	Ph	Ph	115	500	429
2-(4,5-diPh-thiazolyl)	Me	N	CH	N	Ph	Ph	218	375	429
2-(1-Me-benzimidazolyl)	Me	N	N	CH	H	H	—	—	676
2-(1-Et-benzimidazolyl)	Et	N	N	CH	H	H	—	—	676
2-(1-Et-benzimidazolyl)	Et	N	N	CH	Me	H	—	—	676
2-(1-Me-6-Cl-benzimidazolyl)	Me	N	N	CH	H	NO <sub>2</sub>	—	—	676
2-Benzothiazolyl	Me	CH	N	N	Ph	H	218	463	429
2-Benzothiazolyl	Me	CH	N	N	Ph	Ph	210	475	429
2-Benzothiazolyl	Me	N	CH	N	Ph	H	250	380	429
2-Benzothiazolyl	Me	N	CH	N	Ph	Ph	250	385	429
2-Benzothiazolyl	Me	N	N	CH	Ph	H	238	463	429
2-Benzothiazolyl	Me	N	N	CH	Ph	Ph	230	510	429
2-α-Naphthothiazolyl	Me	N	N	CH	Ph	H	227	505	429
2-α-Naphthothiazolyl	Me	N	N	CH	Ph	Ph	170	530	429
2-β-Naphthothiazolyl	Me	CH	N	N	Ph	H	205	482	429
2-β-Naphthothiazolyl	Me	N	N	CH	Ph	H	240	505	429
2-β-Naphthothiazolyl	Me	N	N	CH	Ph	Ph	150	530	429

TABLE 2173. TRIAZACYANINES



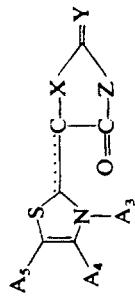
H	A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	B	X	m.p. (°C)	λ (nm)	Ref.
2-Thiazolyl	Et	H	H	Et	BF <sub>4</sub>	200	465	671-673
2-(4-Me-thiazolyl)	Et	Me	H	Et	BF <sub>4</sub>	257	475	563
2-(5-Me-thiazolyl)	Et	H	Me	Et	BF <sub>4</sub>	238	477	671
2-(4,5-diMe-thiazolyl)	Et	Me	Me	Et	BF <sub>4</sub>	133	490	563
2-(5-Cl-thiazolyl)	Et	H	Cl	Et	BF <sub>4</sub>	220	483	671
2-(5-Br-thiazolyl)	Et	H	Br	Et	BF <sub>4</sub>	230	486	671
2-(5-COOEt-thiazolyl)	Et	H	COOEt	Et	BF <sub>4</sub>	225	487	671
2-(5-NO <sub>2</sub> -thiazolyl)	Et	H	NO <sub>2</sub>	Et	BF <sub>4</sub>	204	502	671
2-(5-SO <sub>2</sub> Me-thiazolyl)	Et	H	SO <sub>2</sub> Me	Et	BF <sub>4</sub>	210	477	671
2-Benzothiazolyl	Me	H	H	Me	Cl	—	—	564
2-Benzothiazolyl	Et	Me	H	Et	BF <sub>4</sub>	245	477	565
2-Benzothiazolyl	Et	Me	Me	Et	BF <sub>4</sub>	231	490	565

TABLE 221. O-METHINENEUTROCYANINES



X	Y	Z	A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	m.p. (°C)	λ (nm)	Ref.
CONEt	S	NEt	Me	Me	CH=CHCOOH	—	406	566, 567, 568
CONEt	S	NEt	Me	α-Furyl	H	—	—	359
CONEt	S	NEt	Ph	Ph	Ph	—	—	566
NH	S	NH	Me	α-Furyl	H	—	—	359
NMe	S	NEt	Mc	Mc	CH=CHCOOH	—	457	567, 568
NMe	S	NPh	Me	Me	CH=CHCOOH	—	455	567, 568
NEt	S	NPh	Et	α-Furyl	H	—	—	359
NPh	S	NMc	Me	-(CH <sub>2</sub> ) <sub>2</sub> CO-	—	>300	—	569
NPh	S	NPh	Et	α-Furyl	H	—	—	569
O	O	O	Et	α-Furyl	H	—	—	569
O	O	S	Me	Me	CH=CHCOOH	—	—	444
O	O	S	NEt	Me	CH=C(CN)CN	26.3	444	567, 568
O	O	S	NEt	Me	CH=C(CN)COOH	—	500	567, 568
O	O	S	NEt	Me	CH=C(CHCOO <sup>⊖</sup> ) <sup>⊕</sup>	—	478	567, 568
O	O	S	NEt	Ph	pyridinium	—	449	567, 568
O	S	NEt	Et	H	NHCOME	—	425	446
S	N(Et)Ph	N	Me	α-Furyl	H	—	—	359
S	NPh <sub>2</sub>	N	Et	α-Furyl	H	281	400	359
S	C(CN)CS-	NEt	Mc	Me	CH=CHCOOEt	—	480	567, 568
S	O	N-Piperidyl	Mc	—	-(CH <sub>2</sub> ) <sub>3</sub> CO-	>335	—	569

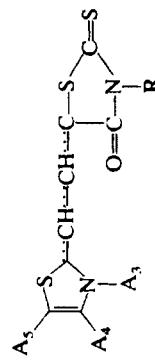
TABLE 221 (Continued)



X	Y	Z	A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	m.p. (°C)	λ (nm)	Ref.
S	S	NH	Mc	α-Furyl	H	—	359	
S	S	NH	Et	Me	H	—	—	423
S	S	NEt	Mc	H	H	—	532	667
S	S	NEt	Mc	H	CH <sub>2</sub> COONa	—	—	570
S	S	NEt	Mc	Mc	H	—	—	443
S	S	NEt	Mc	Mc	CH=CHCONC <sub>4</sub> H <sub>9</sub>	—	462	567, 568
S	S	NEt	Mc	Mc	CH=CHCOOH	—	459	566, 567, 568
S	S	NEt	Mc	Mc	CH=CHCOOEt	—	460	567, 568
S	S	NEt	Mc	Mc	CH=C(CN)CN	—	510	567, 568
S	S	NEt	Mc	Mc	CH=C(CN)COOH, NEt <sub>3</sub>	—	483	567, 568
S	S	NEt	Mc	COCH <sub>3</sub>	—	—	569	
S	S	NEt	Ph	CH=CHCOOH	—	462	567, 568	
S	S	NEt	—(CH <sub>2</sub> ) <sub>3</sub> CO-	—	310	—	569	
S	S	NEt	Mc	α-Furyl	H	218	424	359
S	S	NEt	Et	H	NHCOMe	234	443	112
S	S	NEt	Et	H	—	—	425	446, 447
S	S	NEt	(CH <sub>2</sub> ) <sub>2</sub> SO <sub>3</sub> Na	Me	CH=CHCOOEt	—	—	423, 571
S	S	NEt	Et	-CH <sub>2</sub> - <sup>(o)</sup> C <sub>6</sub> H <sub>4</sub> -	—	233	460	567, 568
S	S	NEt	α-Pyridyl	Mc	Me	—	—	352
S	S	NEt	α-Thiazolyl	Mc	Me	—	—	117
S	S	NEt	α-Benzo-thiazolyl	H	H	—	—	117
S	S	NEt				—	—	395

S	S	N-n-Pr	Et	H	NHCOMe	—	443	446, 447
S	S	NCH <sub>2</sub> CH <sub>2</sub> OH	Me	α-Furyl	H	—	359	359
S	S	N(CH <sub>2</sub> ) <sub>2</sub> COOH	Et	H	NHCOMe	—	442	446, 447
S	S	NCH(Me)COOH	Me	-(CH <sub>2</sub> ) <sub>3</sub> CO-	—	265	—	569
S	S	NCH <sub>2</sub> COOH	Me	Me	CH CHCOOEt	—	461	567, 568
S	S	NCH <sub>2</sub> COOH	Me	-(CH <sub>2</sub> ) <sub>3</sub> CO-	290	331	331	569
S	S	NCH <sub>2</sub> COOH	Et	H	H	—	—	—
S	S	NCH <sub>2</sub> COOH	Et	Me	H	—	572	572
S	S	NCH <sub>2</sub> COOH	Et	Me	H	—	—	—
S	S	NCH <sub>2</sub> COOH	Ph	Mc	H	—	—	573
S	S	NPh	Me	-(CH <sub>2</sub> ) <sub>3</sub> CO-	380	—	—	569
S	S	NPh	Et	H	NHCOMe	—	444	446, 447
S	S	NPh	Et	α-Furyl	H	—	—	359

TABLE 222A. DIMETHINE NEUTROCYANINE



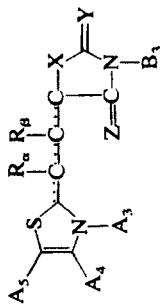
A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	R	Method	m.p. (°C)	λ (nm)	Ref.
Me	H	H	Et	A	220	535	112
Me	H	Br	Et	A	168	537	112
Me	Me	CH=CHCOOH	Et	D	—	562	567, 568
Me	Me	COOH	Et	C	258	547	222
Me	Ph	H	Ph	—	510	80, 82	
Me	p-Xylyl	Ph	Et	B	111	552	111
Me	p-MeOC <sub>6</sub> H <sub>4</sub>	Ph	Et	B	216	552	111
Me	p-MeOC <sub>6</sub> H <sub>4</sub>	OPh	Et	B	186	547	111
Me	p-MeOC <sub>6</sub> H <sub>4</sub>	p-(t-C <sub>8</sub> H <sub>17</sub> )C <sub>6</sub> H <sub>4</sub> O	Et	B	161	546	111, 351
Me	p-MeOC <sub>6</sub> H <sub>4</sub>	H	Et	C	270	510	386
Me	NH <sub>2</sub>	H	H	—	—	—	423
Et	H	H	Et	—	—	—	423, 574, 575
Et	H	H	Et	MeSO <sub>4</sub> <sup>-</sup> salt	165	—	576
Et	H	H	(CH <sub>2</sub> ) <sub>2</sub> SO <sub>3</sub> K	—	—	—	423
Et	H	H	CH <sub>2</sub> COOH	—	—	—	423
Et	H	Styryl	Et	B	205	570	357
Et	H	Ph	Et	B	209	556	354, 357, 575
Et	H	p-NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	Et	—	—	575	
Et	H	p-MeOC <sub>6</sub> H <sub>4</sub>	Et	—	—	575	
Et	H	α-Naphthyl	Et	—	—	548	354, 575
Et	H	β-Naphthyl	Et	—	221	562	354, 575

Et	H	$\alpha$ -Furyl	Et		198	556	354, 575
Et	H	$\alpha$ -Thienyl	Et		192	560	354
Et	H	$\alpha$ -Benzo(b)furyl	Et		220	562	354, 575
Et	Me	H	Me		210	541	577
Et	Et	Me	Et		—	—	—
Et	Et	$(CF_2)_2CF_3$	Et	C	198	539	72
Et	Et	4-Thiazoyl	Et	A	136	487	
Et	Et	4-(2-Me-thiazoyl)	Et	A	210	554	356
Et	Et	H	Et	A	220	555	356
Et	Et	Stryl	Et	B	228	544	357
Et	Et	Ph	Et	B	215	540	114, 354,
Et	Et	Ph	Et	B	—	—	357, 575
Et	Et	Ph	(CH <sub>2</sub> ) <sub>5</sub> COOH	A	242	550	111, 578
Et	Et	Ph	(CH <sub>2</sub> ) <sub>5</sub> COOEt	A	106	553	579, 580
Et	Et	Ph	(CH <sub>2</sub> ) <sub>2</sub> COOEt	A	176	604	581
Et	Et	Ph	CH <sub>2</sub> COOH	—	139	—	579
Et	Et	Ph	CH <sub>2</sub> COOEt	—	—	—	579
Et	Et	Ph	OPh	B	—	500	483
Et	Et	Ph	p-MeC <sub>6</sub> H <sub>4</sub> S	Et	174	544	111, 351
Et	Et	Ph	p-ClC <sub>6</sub> H <sub>4</sub>	Et	165	544	111, 351
Et	Et	p-Xylyl	Et	B	190	554	111
Et	Et	p-QIC <sub>6</sub> H <sub>4</sub>	Et	B	—	—	351
Et	Et	p-MeOC <sub>6</sub> H <sub>4</sub>	Et	B	225	540	111
Et	Et	p-MeOC <sub>6</sub> H <sub>4</sub>	Et	B	197	548	111
Et	Et	p-MeOC <sub>6</sub> H <sub>4</sub>	Et	—	150	544	111
Et	Et	p-CF <sub>3</sub> SO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	Et	C	222	—	491
Et	Et	3,4-diMeOC <sub>6</sub> H <sub>3</sub>	Ph	Et	199	554	111
Et	Et	$\alpha$ -Naphthyl	H	Et	—	—	—
Et	Et	$\beta$ -Naphthyl	H	Et	205	540	354
Et	Et	$\beta$ -Naphthyl	p-MeC <sub>6</sub> H <sub>4</sub> -S	Et	224	540	354
Et	Et	$\alpha$ -Azulene	H	Et	198	544	111, 351
Et	Et	$\alpha$ -Furyl	H	Et	199	542	516
Et	Et	$\alpha$ -Benzo(b)furyl	H	Et	200	536	354, 575
Et	(CH <sub>2</sub> ) <sub>2</sub> SH	Ph	H	Et	243	536	354
					—	—	63

TABLE 222A (Continued)

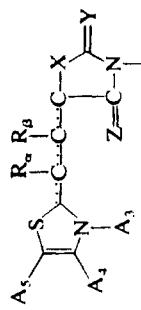
$A_3$	$A_4$	$A_5$	$R$	Method	m.p. (°C)	$\lambda$ (nm)	Ref.
(CH <sub>2</sub> ) <sub>2</sub> SH	Ph	Ph	Et	—	—	—	63
(CH <sub>2</sub> ) <sub>2</sub> SCOMe	Ph	Ph	Et	—	—	—	63
Ph	H	Ph	Et	—	—	—	325
Ph	H	<i>p</i> -MeOC <sub>6</sub> H <sub>4</sub>	Et	—	—	—	325
Ph	H	$\beta$ -Naphthyl	Et	—	—	—	325
Ph	Me	H	Et	—	—	—	530
$\alpha$ -Pyridyl	H	H	Et	D	—	—	117
$\alpha$ -Pyridyl	Me	H	Et	D	—	—	117
$\alpha$ -Pyridyl	Me	Me	Et	—	—	—	117
2-Thiazolyl	H	H	Et	D	—	—	117
2-Thiazolyl	Me	H	Et	D	—	—	117
2-Thiazolyl	Me	Me	Et	—	—	—	117
2-Benzothiazolyl	H	H	Et	—	—	—	395
6-(2-Me-benzo-thiazolyl)	Me	H	Et	C	220	—	118

TABLE 222B

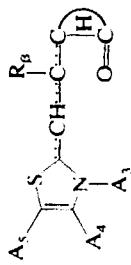


X	Y	Z	A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	R <sub>a</sub>	R <sub>b</sub>	B <sub>3</sub>	Method	m.p. (°C)	λ (nm)	Ref.
NH	S	O	Et	H	H	H	H	H	—	—	—	423
NH	S	O	Et	Me	H	H	H	H	—	—	—	423
NET	S	O	Et	Ph	Ph	H	H	ET	—	—	—	423
NET	S	O	Et	Ph	Ph	H	H	COMe	A	—	—	582
NCOMe	S	O	Et	Ph	Ph	H	H	Et	A	—	—	582
NPh	S	O	Et	Me	H	H	H	—	—	240	—	521
NPh	S	O	Et	Me	H	CH=NPh	H	—	—	—	—	521
NPh	S	O	Et	Me	H	CH=NPh-	H	—	—	165	—	521
								ClHsalt	C	226	547	222
O	S	O	Me	Me	COOH	H	H	Et	—	—	—	423
O	S	O	Et	Me	H	H	H	Et	—	—	—	423
O	S	O	Et	H	H	H	H	Et	—	—	—	423
S	O	O	Et	H	H	H	H	Et	—	—	—	423
S	S	O	Et	Ph	Ph	H	H	Et	—	224	591	583
S	S	O	Me	H	H	H	Me	Et	—	—	—	423
S	S	O	Me	Me	H	H	Me	Me	E	225	—	584
S	S	O	Me	Me	CH=CHCOOH	H	Me	Me	D	—	548	567, 568
S	S	O	Me	Me	CH=CHCOOH	H	Me	Et	D	—	560	567, 568
S	S	O	Me	Me	CH=CHCOOH	H	Me	Et	D	—	560	567, 568
S	S	O	Me	Me	CH=CHCOOEt	H	Me	Et	D	—	565	567, 568
S	S	O	Me	Me	COOH	H	Me	Et	C	214	635	222
S	S	O	Me	Ph	H	H	Me	Me	E	232	—	584

TABLE 222B (Continued)



X	Y	Z	A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	R <sub>α</sub>	R <sub>β</sub>	B <sub>3</sub>	Method	m.p. (°C)	λ (nm)	Ref.
S	S	O	Et	H	H	H	Me	Et	-	-	-	575
S	S	O	Et	H	H	H	Me	CH <sub>2</sub> COOH	-	-	-	585
S	S	O	Et	H	H	H	(CH <sub>2</sub> ) <sub>2</sub> OH	Et	-	-	-	423
S	S	O	Et	H	H	H	(CH <sub>2</sub> ) <sub>2</sub> COOH	Et	-	-	-	423
S	S	S	O	Et	H	H	σ-HOOCC <sub>6</sub> H <sub>4</sub>	Et	-	-	-	423
S	S	S	O	Et	H	H	σ-HOOCC <sub>6</sub> H <sub>4</sub> Allyl	Et	-	-	-	423
S	S	S	O	Et	H	H	OEt	Et	-	-	-	570
S	S	S	O	Et	H	H	CH=NPh	Et	-	-	-	521
S	S	S	O	Et	Me	Me	CH=NPh	Et	-	-	-	521
S	S	S	O	Et	Ph	H	Me	Et	-	-	-	586
S	S	S	O	Et	Ph	H	σ-HOOCC <sub>6</sub> H <sub>4</sub>	Et	-	-	-	423
S	S	S	O	α-Pyridyl	H	H	Me	Et	D	-	-	117
S	S	S	O	α-Pyridyl	Me	H	Me	Et	D	-	-	117
S	S	S	O	α-Pyridyl	Me	H	H	OEt	Et	D	-	117
S	S	S	O	2-Thiazoyl	H	H	H	Me	Et	D	-	117
S	S	S	O	2-Thiazoyl	Me	H	H	Me	Et	D	-	117
S	S	S	O	2-Thiazoyl	Me	Me	H	Me	Et	-	-	117
S	S	S	O	2-Thiazoyl	H	H	H	OEt	Et	-	-	117
S	S	S	O	2-Thiazoyl	Me	H	H	OEt	Et	-	-	117
S	S	S	O	2-Thiazoyl	Me	Me	H	OEt	Et	-	-	117
S	S	S	S	Et	H	H	H	Et	-	-	-	423



H	A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	R	Method	m.p. (°C)	λ (nm)	Ref.
Chroman-2,4-dione	Me	Ph	H	H	—	149	505	587
7-Hydroxychroman-2,4-dione	Me	Ph	H	H	—	208	452	587
5,6-Benzochroman-2,4-dione	Me	Ph	H	H	—	212	485	587
7,8-Benzochroman-2,4-dione	Me	Ph	H	H	—	92	478	587
3-Dihydro-2-keto-4,5-benzo-thianaphthalene)	Et	Ph	Ph	H	C	234	551	588
4-(3-Me-1-Ph-pyrazolone-5)	Me	Me	COOH	Me	C	247	560	222
4-(3-Me-1-Ph-pyrazolone-5)	Me	Ph	H	H	—	—	501	80, 82
4-(3-Me-1-Ph-pyrazolone-5)	Et	Ph	H	H	—	—	—	84
4-(3-Me-1-Ph-pyrazolone-5)	Et	p-BrC <sub>6</sub> H <sub>4</sub>	H	H	—	—	—	84
4-(3-Me-1-Ph-pyrazolone-5)	Et	p-ClC <sub>6</sub> H <sub>4</sub>	H	H	—	—	—	84
4-(3-Me-1-Ph-pyrazolone-5)	Ph	Me	H	H	—	—	480	473
4-(3-Ph-isoxazolone-5)	Me	Ph	H	H	—	—	—	423
4-(3-Ph-isoxazolone-5)	Et	H	H	H	—	—	496	80, 82
5-(4-Ph-thiazolone-4)	Me	H	H	H	—	—	504	589
5-(4-Ph-thiazolthione-4)	Me	H	H	H	—	—	580	589
4-(2-EtS-thiazolone-5)	Me	Ph	p-Xylyl	p-MeC <sub>6</sub> H <sub>4</sub>	B	133	544	111, 351
4-(2-EtS-thiazolone-5)	Me	p-MeOC <sub>6</sub> H <sub>4</sub>	Ph	H	B	108	548	111
4-(2-EtS-thiazolone-5)	Me	p-MeOC <sub>6</sub> H <sub>4</sub>	OPh	H	B	168	543	111, 351
4-(2-EtS-thiazolone-5)	Me	p-MeOC <sub>6</sub> H <sub>4</sub>	p-(t-C <sub>8</sub> H <sub>17</sub> )C <sub>6</sub> H <sub>4</sub> O	H	B	—	—	351

TABLE 222C (Continued)

H	A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	R	Method	m.p. (°C)	λ (nm)	Ref.
4-(2-EtS-thiazolone-5)	Me	β-Naphthyl	p-MeC <sub>6</sub> H <sub>4</sub> S	H	B	180	544	111, 351
4-(2-EtS-thiazolone-5)	Et	Ph	Ph	H	B	208	547	111
4-(2-EtS-thiazolone-5)	Et	Ph	p-MeC <sub>6</sub> H <sub>4</sub>	H	B	—	—	351
4-(2-EtS-thiazolone-5)	Et	p-BrC <sub>6</sub> H <sub>4</sub>	p-BrC <sub>6</sub> H <sub>4</sub> O	H	B	—	—	351
4-(2-EtS-thiazolone-5)	Et	p-MeOC <sub>6</sub> H <sub>4</sub>	H	B	182	527	111	
4-(2-EtS-thiazolone-5)	Et	p-MeOC <sub>6</sub> H <sub>4</sub>	Ph	H	B	181	548	111
4-(2-EtS-thiazolone-5)	Et	p-MeOC <sub>6</sub> H <sub>4</sub>	OPh	H	B	164	542	111, 351
4-(2-EtS-thiazolone-5)	Et	3,4-diMeO-C <sub>6</sub> H <sub>3</sub>	Ph	H	B	229	550	111

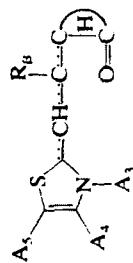


TABLE 222D

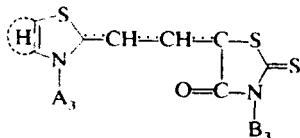
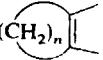
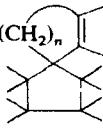
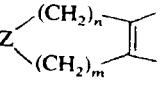
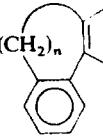
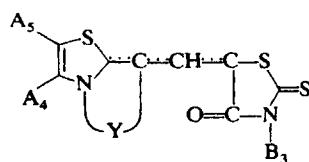
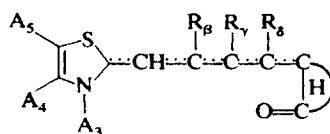
H	A <sub>3</sub>	B <sub>3</sub>	Method	m.p. (°C)	λ(nm)	Ref.	
							
	$\frac{n}{3}$	Et	Et	C	233	556	399
	$\frac{n}{4}$	Et	Et	—	222	546	72
					495		87
	$\frac{n}{4}$ (4-i-Pr, 7-Me)	Me	Et	C	188	550	401
	$\frac{n}{5}$	Et	Et	C	192	550	402
	$\frac{n}{2}$	Me	Et	C	216	556	402
	$\frac{n}{3}$	Et	Et	C	238	550	404
	$\frac{n}{1}$ $\frac{m}{2}$ Z	Et	Et	C	234	491	87, 406
	$\frac{n}{3}$ $\frac{m}{0}$ O	Et	Et	C	216	556	407
	$\frac{n}{0}$ $\frac{m}{3}$ S	Et	Et	C	235	549	409
					598		87
	$\frac{n}{1}$ $\frac{m}{2}$ S	Et	Et	C	235	544	408
					492		87
	$\frac{n}{1}$	Me	Et	C	248	556	537
	$\frac{n}{2}$	Me	Et	C	291	554	405
	$\frac{n}{2}$	Et	Et	—	237	554	114

TABLE 222E



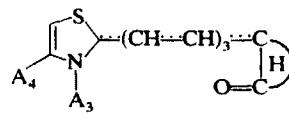
Y	A <sub>4</sub>	A <sub>5</sub>	B <sub>3</sub>	m.p. (°C)	λ(nm)	Ref.
-(CH <sub>2</sub> ) <sub>2</sub> -	Ph	Ph	Me	266	572	410
-(CH <sub>2</sub> ) <sub>3</sub> -	Me	H	Me	271	558	410
-(CH <sub>2</sub> ) <sub>3</sub> -	Me	Me	Me	282	568	410
-(CH <sub>2</sub> ) <sub>3</sub> -	Ph	H	Me	269	560	410
-(CH <sub>2</sub> ) <sub>3</sub> -	Ph	Ph	Me	262	571	410
-(CH <sub>2</sub> ) <sub>4</sub> -	Ph	Ph	Me	235	580	410
-CH <sub>2</sub> -(1,2-cyclohexenyl)-CH <sub>2</sub> -	Me	H	Me	215	568	412
-(CH <sub>2</sub> ) <sub>2</sub> -O-	Me	H	Et	306	578	411
-(CH <sub>2</sub> ) <sub>2</sub> -S-	Me	H	Et	285	572	411
-(o)C <sub>6</sub> H <sub>4</sub> -	Me	H	Et	326	508	415
-(o)C <sub>6</sub> H <sub>4</sub> -(CH <sub>2</sub> ) <sub>2</sub> -	Me	H	Et	—	574	413, 414
-(o)C <sub>6</sub> H <sub>4</sub> -Se-	Me	H	Et	237	545	413, 414

TABLE 223. TETRAMETHINE NEUTROCYANINES



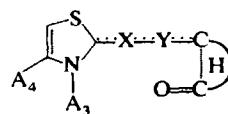
H	R <sub>β</sub>	R <sub>δ</sub>	R <sub>γ</sub>	A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	m.p. (°C)	λ(nm)	Ref.
Chroman-2,4-dione	H	H	H	Me	Ph	H	168	583	587
7-OH-chroman-2,4-dione	H	H	H	Me	Ph	H	190	556	587
5,6-Benzochroman-2,4-dione	H	H	H	Me	Ph	H	>300	550	587
7,8-Benzochroman-2,4-dione	H	H	H	Me	Ph	H	295	554	587
5-(1-Me-3-Ph-pyrazolone)	H	H	H	Et	Ph	H	—	—	84
diEt-thiohydantoine	H	H	H	Me	Me	CH=CHCOOH	—	623	567
4-(2-Ph-oxazolone)	H	OEt	H	Et	Ph	Ph	210	—	590
N-Et-rhodanine	H	H	H	Me	Me	CH=CHCOOH	—	648	567
N-Et-rhodaninc	H	H	H	Me	Ph	H	225	—	69
N-Et-rhodanine	H	H	H	Me	p-BrC <sub>6</sub> H <sub>4</sub>	H	75	—	69
N-Et-rhodanine	H	H	H	Me	p-MeOC <sub>6</sub> H <sub>4</sub>	H	142	—	69
N-Et-rhodanine	H	H	H	Me	p-EtOC <sub>6</sub> H <sub>4</sub>	H	142	—	69
N-Et-rhodanine	Me	H	H	Et	Ph	Ph	213	—	591
N-Et-rhodanine	-(CH <sub>2</sub> ) <sub>3</sub> -	H	Me	Me	CH=CHCOOH	—	—	566	

TABLE 224. HEXAMETHINE NEUTROCYANINES



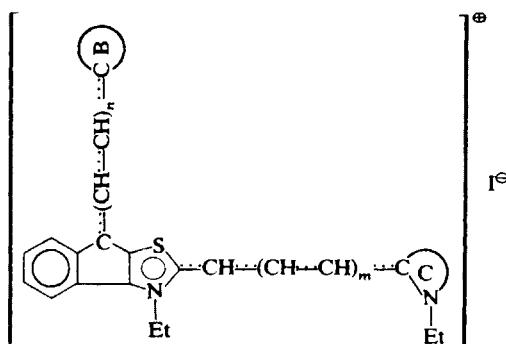
H	A <sub>3</sub>	A <sub>4</sub>	m.p. (°C)	Ref.
5-(1-Me-3-Ph-pyrazolone)	Et	Ph	—	84
5-N-Et-rhodanine	Me	Ph	145	69
5-N-Et-rhodanine	Me	p-BrC <sub>6</sub> H <sub>5</sub>	222	69
5-N-Et-rhodanine	Me	p-MeOC <sub>6</sub> H <sub>4</sub>	146	69
5-N-Et-rhodanine	Me	p-EtOC <sub>6</sub> H <sub>4</sub>	178	69

TABLE 225. AZANEUTROCYANINES



H	X	Y	A <sub>3</sub>	A <sub>4</sub>	Method	m.p. (°C)	λ (nm)	Ref.
4-(3-Me-1-Ph-pyrazolone)	CH	N	Et	Me	B	186	—	592
4-(3-Me-1-Ph-pyrazolone)	N	CH	Me	Ph	A	176	490	80, 82
4-(3-Ph-isoxozolone)	N	CH	Me	Ph	A	182	490	80, 82
5-(N-H-rhodanine)	N	CH	Me	Ph	A	233	470	80
5-(N-Et-rhodanine)	N	CH	Me	Ph	A	192	480	80
5-(N-Ph-rhodanine)	N	CH	Me	Ph	A	300	485	80, 82
5-(N-(o)Me-C <sub>6</sub> H <sub>4</sub> -rhodanine)	N	CH	Me	Ph	A	224	485	80
5-(N-(m)Me-C <sub>6</sub> H <sub>4</sub> -rhodanine)	N	CH	Me	Ph	A	221	480	80
5-(N-(p)Me-C <sub>6</sub> H <sub>4</sub> -rhodanine)	N	CH	Me	Ph	A	230	480	80
5-(N-(m)Cl-C <sub>6</sub> H <sub>4</sub> -rhodanine)	N	CH	Me	Ph	A	300	478	80

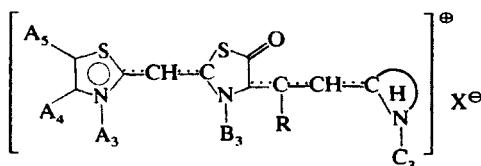
TABLE 311. TRINUCLEAR CYANINES: CYANINE-CYANINE



B	C	m.p.			$\lambda$ (nm)	Ref.
		<i>m</i>	<i>n</i>	(°C)		
4-(1-Me-quinolyl)	2-Benzothiazolyl	0	0	240	448-625	1(p. 622), 537
2-(3-Et-benzoxazolyl)	2-Benzoxazolyl	1	1	288	458-460	1(p. 622), 537
2-(3-Et-benzoxazolyl)	2-Benzothiazolyl	1 <sup>a</sup>	1	236	474-650	1(p. 622), 537
2-(3-Et-thiazolinyl)	2-Thiazolyl	1	1	286	446-625	1(p. 622), 537
2-(3-Et-benzothiazolyl)	2-Benzothiazolyl	0	0	280	430-524	1(p. 622), 86, 537

<sup>a</sup> Mesomethyl.

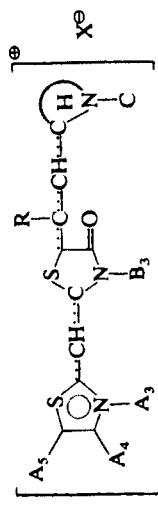
TABLE 312A. CYANINE-NEUTROCYANINE



H	A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	B <sub>3</sub>	C <sub>3</sub>	R	m.p. (°C)	λ(nm)	Ref.
2-(4,5-diPh-oxazolyl)	Et	p-MeOC <sub>6</sub> H <sub>4</sub>	Ph	Me	Et	H	247	598	1(p.656), 593
2-(4,5-diPh-thiazolyl)	Et	Ph	Ph	Me	Et	H	243	643	593
2-(4-Ph-5-p-MeC <sub>6</sub> H <sub>4</sub> S-thiazolyl)	Me	Ph	p-MeC <sub>6</sub> H <sub>4</sub> S	Me	Me	H	205	633	351, 593
2-(4-p-Xylyl-5-Ph-thiazolyl)	Me	p-Xylyl	Ph	Me	Me	H	220	642	593
2-(4-p-Xylyl-5-Ph-thiazolyl)	Et	p-Xylyl	Ph	Me	Et	H	265	651	593
2-(4-β-Naphthyl 5-MeC <sub>6</sub> H <sub>4</sub> S-thiazolyl)	Me	β-Naphthyl	p-MeC <sub>6</sub> H <sub>4</sub> S	Me	Me	H	222	635	593
2-(4-p-BrC <sub>6</sub> H <sub>4</sub> 5-p-BrC <sub>6</sub> H <sub>4</sub> O-thiazolyl)	Me	p-BrC <sub>6</sub> H <sub>4</sub>	p-BrC <sub>6</sub> H <sub>4</sub> O	Me	Et	H	190	629	351, 593
2-(4-p-MeOC <sub>6</sub> H <sub>5</sub> -5-Ph-thiazolyl)	Et	p-MeOC <sub>6</sub> H <sub>4</sub>	Ph	Me	Et	H	263	643	593
2-Benzoxazolyl	Et	p-MeOC <sub>6</sub> H <sub>4</sub>	Ph	Me	Et	H	278	538	593
2-Benzothiazolyl	Me	Ph	OPh	Me	Et	H	—	—	351
2-Benzothiazolyl	Et	Ph	Ph	Me	Et	H	246	620	593
2-Benzothiazolyl	Et	Ph	Ph	Et	Et	Me	264	631	594
2-Benzothiazolyl	Et	Ph	OPh	Me	Et	H	242	619	593
2-Benzothiazolyl	Et	p-MeOC <sub>6</sub> H <sub>4</sub>	Ph	Me	Et	H	269	624	593
2-(5-Cl-benzothiazolyl)	Et	Ph	Ph	Me	Et	H	249	621	593
2-Benzoselenazolyl	Me	Ph	OPh	Me	Et	H	263 <sup>a</sup>	622	351, 593 595
2-Benzoselenazolyl	Et	Ph	Ph	Me	Et	H	268	624	1(p. 656), 593
2-Benzoselenazolyl	Et	p-OMeC <sub>6</sub> H <sub>4</sub>	Ph	Me	Et	H	271	624	1(p. 656), 593
2-β-Naphthothiazolyl	Me	Ph	OPh	Me	Et	H	264 <sup>a</sup>	636	1(p. 656), 593
2-β-Naphthothiazolyl	Et	Ph	Ph	Me	Et	H	263	643	593

<sup>a</sup> X = OTs<sup>⊖</sup>.

TABLE 312B



H	A <sub>3</sub>	A <sub>4</sub>	A <sub>x</sub>	B <sub>3</sub>	C	R	X	m.p. (°C)	λ(nm)	Ref.
4-Pyridinyl	Et	Ph		Et	Et	H	I	210	—	596
2-(1,1-diMe-indolonyl)	Et	Ph		Et	Et	H	I	—	—	597, 598
2-Quinolyl	Et	Ph		Et	Et	H	I	—	—	596
2-(5-Oxoquinolyl)	Et	Ph		Et	Et	H	I	—	—	617
4-Quinolyl	Et	Ph		Et	Et	H	I	—	—	467
4-Quinolyl	Et	COOEt		Et	Et	H	I	—	—	599
4-Quinolyl	Et	Ph		Et	(CH <sub>2</sub> ) <sub>3</sub> SO <sub>3</sub>	H	I	—	—	599
4-Quinolyl	(CH <sub>2</sub> ) <sub>3</sub> SO <sub>3</sub>	Ph		Et	Allyl	H	I	—	—	600
4-Quinolyl	(CH <sub>2</sub> ) <sub>3</sub> SO <sub>3</sub>	Ph		Et	Et	H	I	—	—	600
4-Quinolyl	(CH <sub>2</sub> ) <sub>3</sub> CH(OH)- CH <sub>2</sub> OSO <sub>3</sub>	Ph		Et	Et	H	I	—	—	600
4-Quinolyl	(CH <sub>2</sub> CH <sub>2</sub> O) <sub>2</sub> - (CH <sub>2</sub> ) <sub>3</sub> SO <sub>3</sub>	Ph		Et	Allyl	H	I	—	—	600
4-Quinolyl	CH(Me)CH <sub>2</sub> SO <sub>3</sub>	Ph		Et	Et	H	I	—	—	600
4-Quinolyl	(CH <sub>3</sub> CH <sub>2</sub> O) <sub>2</sub> SO <sub>3</sub>	Ph		Et	Et	H	I	—	—	600
4-Quinolyl	(CH <sub>2</sub> ) <sub>2</sub> SO <sub>3</sub>	Ph		Et	Hexyl	H	I	—	—	600
4-(2-Me-oxo-5-quinolyl)	Me	Ph		Et	Me	H	I	—	—	641
2-(4,5-diPh-oxazolyl)	Et	p-MeOC <sub>6</sub> H <sub>4</sub>	Ph	Et	Et	H	I	210	575	593
2-(4-Me-thiazolyl)	Et	Ph		Et	Et	H	I	—	—	596, 675
2-(4,5,6,7-Tetrahydrobenzo-thiazolyl)	Et	Ph		Et	Et	H	I	—	—	601
2-(4,5-diPh-thiazolyl)	Et	Ph		Et	Me	H	I	228	610	594, 61/2
2-(4,5-diPh-thiazolyl)	Et	Ph		Et	H	H	I	275	615	593
2-(4,5-diPh-thiazolyl)	Et	Ph		Et	Allyl	H	I	—	—	596
2-(4-Ph-5-p-MeC <sub>6</sub> H <sub>4</sub> S- thiazolyl)	Et	p-MeC <sub>6</sub> H <sub>4</sub> S	Et	Et	H	H	I	—	—	1(p. 656), 351

2-(4- <i>p</i> -MeC <sub>n</sub> H <sub>n</sub> 5-Ph-3-Me-thiazolyl)	Me	<i>p</i> -MeC <sub>n</sub> H <sub>n</sub>	Ph	Et	Me	H	1	237	—	603
2-(4- <i>p</i> -Xylyl-5-Ph-thiazolyl)	Me	<i>p</i> -Xylyl	Ph	Et	Me	H	1	240	613	593
2-(4- <i>p</i> -Xylyl-5-Ph-thiazolyl)	Et	<i>p</i> -Xylyl	Ph	Et	Et	H	1	262	614	593
2-(4- <i>p</i> -Naphthyl-5- <i>p</i> -Me-C <sub>n</sub> H <sub>n</sub> S-thiazolyl)	Me	$\beta$ -Naphthyl	<i>p</i> -Me-C <sub>n</sub> H <sub>n</sub> S	Et	Et	H	1	241	613	351, 593
2-(4- <i>p</i> -MeOC <sub>n</sub> H <sub>n</sub> 5-Ph-thiazolyl)	Et	<i>p</i> -MeOC <sub>n</sub> H <sub>n</sub>	Ph	Et	Et	H	1	282	613	593
2-[4- <i>p</i> -MeOC <sub>n</sub> H <sub>n</sub> 5-( <i>p</i> -1,1',3',3'-tetramethylbutyl-C <sub>n</sub> H <sub>n</sub> -thiazolyl)]	Et	<i>p</i> -MeOC <sub>n</sub> H <sub>n</sub>	Ph	Et	Me	H	1	228	608	593
2-(4-5-diMeO-thiazolyl)	Et	4,5-diMeO-C <sub>n</sub> H <sub>n</sub>	H	Et	Et	H	1	227	616	593
2-(4- <i>p</i> -MeOC <sub>n</sub> H <sub>n</sub> 5-PhO-thiazolyl)	Me	<i>p</i> -MeOC <sub>n</sub> H <sub>n</sub>	PhO	Et	Me	H	1	266	605	593, 351
2-(4- <i>p</i> -MeOC <sub>n</sub> H <sub>n</sub> 5-PhO-thiazolyl)	Et	<i>p</i> -MeOC <sub>n</sub> H <sub>n</sub>	PhO	Et	Me	H	1	—	—	351, 593
2-Benzoxazolyl	Et	Ph	Ph	Et	Et	H	1	283	552	593, 603
2-Benzoxazolyl	Et	<i>p</i> -MeOC <sub>n</sub> H <sub>n</sub>	Ph	Et	Et	H	1	—	—	1(p, 294), 511
2-(5-Ph-benzoxazolyl)	Et	H	H	Et	Et	H	1	—	—	—
2-Benzothiazolyl	Et	Me	COOEt	Et	Et	H	1	—	—	599
2-Benzothiazolyl	Et	Ph	Ph	Me	Et	H	1	—	—	596
2-Benzothiazolyl	Et	Ph	Ph	Me	Et	Me	1	265	594	594, 602
2-Benzothiazolyl	Et	Ph	Ph	Et	Me	Et	1	242	598	594, 602
2-Benzothiazolyl	Et	Ph	Ph	Me	Et	<i>n</i> -Pr	1	—	—	602
2-Benzothiazolyl	Et	Ph	Ph	Et	Me	(CH <sub>2</sub> ) <sub>2</sub> Ph	1	240	600	594, 602
2-Benzothiazolyl	Et	Ph	Ph	Et	Me	CH <sub>2</sub> Ph	1	—	—	602
2-Benzothiazolyl	Et	Ph	Ph	Et	Et	H	1	—	—	596
2-Benzothiazolyl	Et	Ph	Ph	Et	Et	H	1	264	584	1(p, 651), 593, 599,
2-Benzothiazolyl	Et	Ph	Ph	Et	Et	Me	1	—	—	604, 605
2-Benzothiazolyl	Et	Ph	Ph	Et	Et	Et	1	—	—	594, 602
2-Benzothiazolyl	Et	Ph	Ph	Et	Et	Ph	1	—	—	599, 606
2-Benzothiazolyl	Et	Ph	Ph	Hetyl	Et	ClO <sub>4</sub>	252	596	594, 602	

TABLE 312B. (Continued)

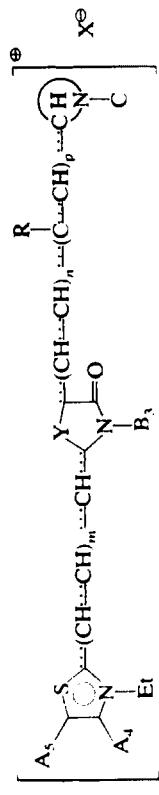
H	A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	B <sub>3</sub>	C	R	X	m.p. (°C)	λ(nm)	Ref.
2-Benzothiazolyl	Et	Ph	Ph	Allyl	Et	CH <sub>3</sub> Ph	I	200	605	594
2-Benzothiazolyl	Et	Ph	Ph	Allyl	Et	H	I	—	590	607
2-Benzothiazolyl	Et	p-MeOC <sub>6</sub> H <sub>4</sub>	Ph	Et	Et	3-p-HOOC-C <sub>6</sub> H <sub>4</sub> -CH <sub>2</sub>	H	285	583	593
2-Benzothiazolyl	Et	p-HOOC-C <sub>6</sub> H <sub>4</sub>	Ph	Et	Me	Me	Br	215	606	594, 602
2-Benzothiazolyl	Et	C <sub>6</sub> H <sub>5</sub> CH <sub>2</sub>	Ph	Allyl	Et	Ph	I	—	611	607
2-Benzothiazolyl	Et	C <sub>6</sub> H <sub>5</sub> CH <sub>2</sub>	Ph	Et	Et	Et	I	—	—	608
2-Benzothiazolyl	Et	(2-Benzo-thiazolyl)-CH <sub>2</sub>	Ph	Et	Et	Et	I	—	—	608
2-Benzothiazolyl	Et	(2-Benzo-thiazolyl)-CH <sub>2</sub>	Ph	Et	Et	Et	I	—	640	609
2-(5-Cl-benzothiazolyl)	Et	Ph	Et	Et	(CH <sub>2</sub> ) <sub>2</sub> COO-	H	I	—	—	596
2-(7-Styryl-benzothiazolyl)	Et	Ph	Ph	Et	Me	Et	Cl	204	604	610
2-(7-Styryl-benzothiazolyl)	Et	Ph	Ph	Et	Et	Et	Cl	207	615	610
2-(7-Styryl-benzothiazolyl)	Et	Ph	Ph	Et	n-Pr	Ph	Cl	196	618	610
2-(5,6-diMe-benzothiazolyl)	Et	Ph	Ph	Et	Et	Et	I	—	—	597, 598
2-(5-EtO <sub>2</sub> 6-Me-benzothiazolyl)	Et	Ph	Ph	Et	Et	Et	I	—	—	596
2-(5-EtO <sub>2</sub> 6-Me-benzothiazolyl)	Et	(CH <sub>2</sub> ) <sub>2</sub> COOH	Ph	Et	Et	p-HOOC-C <sub>6</sub> H <sub>4</sub>	—	—	597, 598	
2-(5-EtO <sub>2</sub> 6-Me-benzothiazolyl)	Et	(CH <sub>2</sub> ) <sub>2</sub> COOH	Ph	Et	Et	p-HOOC-C <sub>6</sub> H <sub>4</sub>	—	—	596	

2-(5-EtO <sub>2</sub> Me-benzo-thiazolyl)	(CH <sub>2</sub> ) <sub>2</sub> OH	Ph	Ph	Et	Et	H	I	—	—	596
2-(5-EtO <sub>2</sub> Me-benzo-thiazolyl)	Et	Ph	Ph	Et	Et	n-Pr	H	I	—	596, 611
2-Benzoselenazolyl	Et	Ph	Ph	Et	Et	H	I	—	—	599, 604
2-(5-MeO-benzo-selenazolyl)	Et	Ph	Ph	Et	Et	Ph	I	—	—	596
2-(5,6-diMe-benzo-selenazolyl)	Et	Ph	Ph	Et	Et	Ph	I	—	—	596
2-(5,6-Tetramethylene benzothiazolyl)	Et	H	2-Benzo-thiazolyl	Et	Et	Ph	I	292	635	389
2-(5,6-Tetramethylene benzothiazolyl)	Et	2-Benzo-thiazolyl	H	Et	Et	Ph	I	246	607	389
2-(6,7-Tetramethylene benzothiazolyl)	Et	H	α-Naphthyl	Et	Et	Ph	SO <sub>2</sub> Me	—	—	612
2-(6,7-Tetramethylene benzothiazolyl)	Et	Me	4-thiazolyl	Et	Et	H	SO <sub>2</sub> Me	>300	615	356
2-(6,7-Tetramethylene benzothiazolyl)	Et		-C <sub>6</sub> H <sub>4</sub> (o)(CH <sub>2</sub> ) <sub>2</sub> -	Et	Et	Ph	SO <sub>2</sub> Me	273	612	114
2-(6,7-Tetramethylene benzothiazolyl)	Et	Ph	Ph	Et	Et	Ph	I	262	—	603, 611
2-α-Naphthothiazole	Et	Ph	Ph	Et	Et	Ph	I	—	—	599, 675
2-β-Naphthothiazole	Et	H	Ph	Et	Et	Ph	SO <sub>2</sub> Me	278	619	354
2-β-Naphthothiazole	Et	H	2-Naphthyl	Et	Et	Ph	SO <sub>2</sub> Me	—	622	354
2-β-Naphthothiazole	Et	H	α-Furyl	Et	Et	Ph	SO <sub>2</sub> Me	287	622	354
2-β-Naphthothiazole	Et	H	α-Benzo-furyl	Et	Et	Ph	SO <sub>2</sub> Me	288	630	354
2-β-Naphthothiazole	Et	H	β-Benzo-furyl	Et	Et	Ph	SO <sub>2</sub> Me	293	616	354
2-β-Naphthothiazole	Et	Ph	Et	Et	Et	H	I	—	—	596
2-β-Naphthothiazole	Et	Ph	Ph	Et	Et	H	I	—	—	596
2-β-Naphthothiazole	Et	(CH <sub>2</sub> ) <sub>2</sub> COO	Ph	Et	Et	Ph	—	—	—	596
2-β-Naphthothiazole	Et	(CH <sub>2</sub> ) <sub>2</sub> SO <sub>3</sub>	Ph	Et	Et	H	OTs	242	655	353
2-(Acenaphtho[1,2-d]-thiazolyl)	Et	Ph	H	Et	Et	H	SO <sub>2</sub> Me	274	645	353

TABLE 312B (Continued)

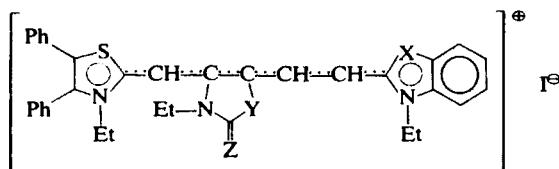
H	A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	B <sub>3</sub>	C	R	X	m.p. (°C)	λ(nm)	Ref.
2-(Acenaphthod[1,2-d]-thiazolyl)	Et	Ph	Ph	Et	Et	H	OTs	284	651	353
5-(7,7-diMe-hexahydro-1,4-diazepine)	Me	Ph	Ph	Et	Et	H	I	175	584	613
7-(5,5-diMe-hexahydro-1,5-diazepine)	Et	Ph	Ph	Et	Et	H	I	210	549	613
7-(5-Et5-Me-hexahydro-1,5-diazepine)	Me	Ph	Ph	Et	Et	Et	I	194	560	613
7-(3,5,5-triMe-hexahydro-1,5-diazepine)	Et	Ph	Ph	Et	Et	H	I	180	551	613
2-(3'-Me-1'-Ph-pyrazolo-[4,5-d]thiazolyl)	Et	Ph	Ph	Et	Et	H	I	243	—	264
2-[Thienot(3,2-e)thiazolyl]	Et	Ph	Ph	Et	Et	H	EtSO <sub>4</sub>	269	607	497, 578
2-[Thieno(3,2-d)thiazolyl]	Et	Ph	Ph	Et	Et	H	I	236	—	578
5-(4-Me-tetrazole)	Et	Ph	Ph	Et	Et	H	I	—	—	597, 598
										675

TABLE 312C



H	<i>m</i>	<i>n</i>	<i>p</i>	<i>Y</i>	<i>A</i> <sub>4</sub>	<i>A</i> <sub>5</sub>	<i>B</i> <sub>3</sub>	C	R	X	m.p. (°C)	λ (nm)	Ref.
4-Quinolyl	1	0	1	S	H	Ph	Et		H	ClO <sub>4</sub>	263	650	1(p. 649), 614
2-(4,5-diPh-thiazolyl)	0	1	1	S	Ph	Ph	Et	Me	OEt	I	—	671	439-441
2-Benzothiazolyl	0	0	1	NEt	Ph	Ph	Et	Et	H	I	—	—	604
2-Benzothiazolyl	0	0	0	S	H	Ph	Et	Et	—	I	187	530	1(p. 649), 614
2-Benzothiazolyl	0	1	1	S	H	Ph	Et	Et	H	I	—	—	1(p. 651), 615
2-Benzothiazolyl	0	1	1	S	H	Ph	Et	Et	OEt	ClO <sub>4</sub>	—	—	439-441
2-Benzothiazolyl	0	1	1	S	Ph	Ph	Et	(CH <sub>2</sub> ) <sub>2</sub> OH	OEt	ClO <sub>4</sub>	—	653	439-441
2-Benzothiazolyl	0	1	1	S	Ph	Ph	Allyl	(CH <sub>2</sub> ) <sub>2</sub> OH	OEt	ClO <sub>4</sub>	—	439-441	
2-Benzothiazolyl	1	0	0	S	H	Ph	Et	Et	OEt	I	228	625	1(p. 649), 614
2-Benzothiazolyl	1	0	1	S	H	Ph	Et	Et	—	I	239	672	1(p. 649), 614
2-(5-Me-benzothiazolyl)	0	1	1	S	Ph	Ph	Allyl	(CH <sub>2</sub> ) <sub>2</sub> OH	H	ClO <sub>4</sub>	—	440	
2-(5-Cl-benzothiazolyl)	0	1	1	S	Ph	Ph	Et	(CH <sub>2</sub> ) <sub>2</sub> <sup>-</sup>	OEt	ClO <sub>4</sub>	—	651	439-441
								OCOMe					
2-(5-MeO-benzothiazolyl)	0	1	1	S	Ph	Ph	Et	Et	OEt	I	—	—	439-441
2-Benzoselenazolyl	0	1	1	S	Ph	Ph	Et	Et	ClO <sub>4</sub>	—	—	—	439-441
2-(5,6-diMc)-benzoselenazolyl	0	1	1	S	Ph	Ph	Et	Et	OEt	I	—	—	439-441
2-β-Naphthothiazolyl	0	1	1	S	Ph	Ph	Et	Et	OEt	I	—	—	439-441
2-(6,7-Tetramethylene benzothiazolyl)	0	1	1	S	Ph	Ph	Et	Et	OEt	I	—	660	439-441

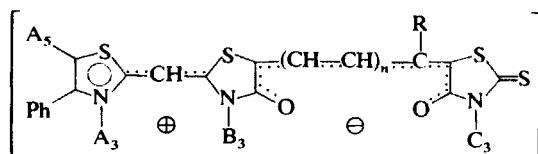
TABLE 312D



X	Y	Z	m.p. (°C)	$\lambda$ (nm)	Ref.
O	S	O	185	660	1(p. 656), 605, 616
S	S	NEt	—	—	1(p. 656), 605
Se	O	O	—	—	1(p. 656), 605

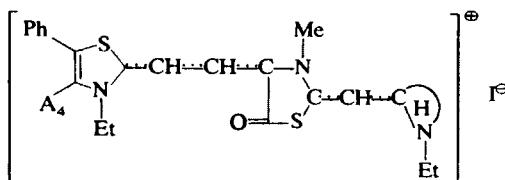
X	Y	Z	m.p. (°C)	$\lambda$ (nm)	Ref.
O	S	O	185	660	1(p. 656), 605, 616
S	S	NEt	—	—	1(p. 656), 605
Se	O	O	—	—	1(p. 656), 605

TABLE 313. CYANINE-OXONOL



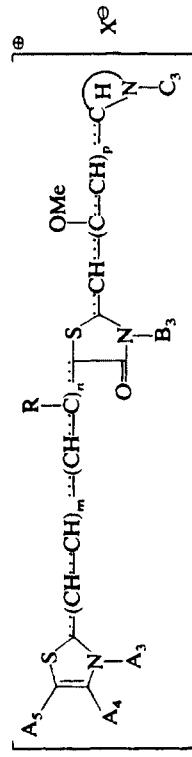
A <sub>3</sub>	A <sub>5</sub>	B <sub>3</sub>	C <sub>3</sub>	n	R	$\lambda$ (nm)	Ref.
Me	Me	Ph	Allyl	1	Et	583	617
Et	Ph	Et	Et	3	H	—	618

TABLE 3141A. CATIONIC RHODACYANINES



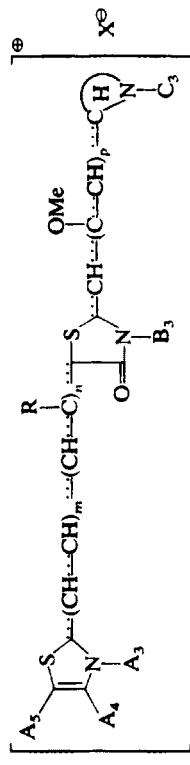
H	A <sub>4</sub>	m.p. (°C)	$\lambda$ (nm)	Ref.
2-(4,5-diPh-Oxazolyl)	p-MeOC <sub>6</sub> H <sub>4</sub>	186	616	1(p. 656), 593
2-Benzothiazolyl	Ph	269	629	1(p. 656), 593
2-Benzothiazolyl	p-MeOC <sub>6</sub> H <sub>4</sub>	272	630	1(p. 656), 593

TABLE 3141B



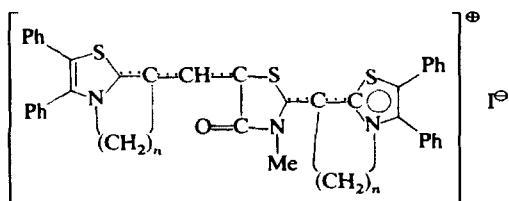
H	<i>m</i>	<i>n</i>	<i>p</i>	A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	B <sub>3</sub>	C <sub>3</sub>	R	X	m.p. (°C)	λ (nm)	Ref.
2-(4,5-diPh-Oxozoyl)	0	0	0	Me	Me	CH=CH-COOEt	Et	Et	—	1	—	507	567
2-(4,5-diPh-Oxozoyl)	0	0	0	Me	Me	CH=CH-COOEt	CH <sub>2</sub> COOH	Et	—	1	—	505	567
2-(4-Me-thiazolidinyl)	0	1	0	Et	Me	H	Et	Et	H	1	—	—	134
2-(4,5-diPh-thiazoyl)	0	0	0	Et	Ph	Ph	Et	Et	Et	1	—	—	606
181	0	1	0	Me	Me	CH=CH-COOEt	Et	Et	Me	1	—	621	567
2-(4,5-diPh-thiazoyl)	0	0	0	Et	Ph	Ph	Et	Et	Me	—	—	—	606
2-(1-Me-benzimidazoyl)	0	0	0	Me	Me	CH=CH-COOH	Et	Et	EtSO <sub>4</sub>	—	—	515	567
2-Benzoxazoyl	0	0	0	Me	Me	CH=CH-COOEt	Et	Et	EtSO <sub>4</sub>	—	—	508	567
2-Benzoxazoyl	1	0	0	Et	Ph	Ph	Et	Et	OEt	EtSO <sub>4</sub>	—	669	439-441
2-Benzothiazoyl	0	0	0	Me	Me	CH=CH-COOH	Et	Et	EtSO <sub>4</sub>	—	—	529	567
2-Benzothiazoyl	0	0	0	Me	Me	CH=CH-COOEt	Et	Et	EtSO <sub>4</sub>	—	—	520	567
2-Benzothiazoyl	0	0	0	Me	Me	CH=CH-COOEt	Et	Et	EtSO <sub>4</sub>	—	—	580	567
2-Benzothiazoyl	0	1	0	Et	Me	H	Et	Et	H	1	—	—	620
2-Benzothiazoyl	0	1	0	Et	Ph	Me	Et	Et	H	1	—	—	619
2-Benzothiazoyl	0	1	0	Et	Ph	Ph	Et	Et	H	1	—	—	621

TABLE 3141B (Continued)



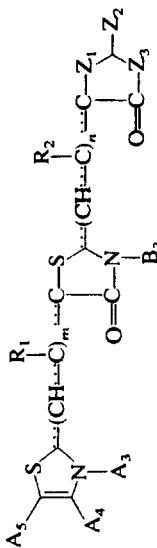
H	m	n	p	A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	B <sub>3</sub>	C <sub>3</sub>	R	X	m.p. (°C)	λ (nm)	Ref.
2-Benzothiazolyl	0	1	0	Et	Azulenyl	H	Et	Et	H	ClO <sub>4</sub>	223	620	516
2-Benzothiazolyl	0	1	1	Me	Me	CH-CH-COOEt	Et	Et	Me	MeSO <sub>4</sub>	—	672	567
2-Benzothiazolyl	0	1	1	Me	Ph	Ph	Et	Et	Me	I	—	—	622
2-Benzothiazolyl	0	1	1	Me	Ph	Ph	Et	Et	CH <sub>2</sub> Pr	MeSO <sub>4</sub>	—	—	622
2-Benzothiazolyl	0	1	1	Me	Ph	Ph	Et	Et	Ph	MeSO <sub>4</sub>	—	—	622
2-(5,6-diMe-benzothiazolyl)	0	1	0	Me	Me	CH-CH-COOEt	Et	Et	Me	I	—	626	567
2-(5MeO-benzothiazolyl)	0	1	0	Et	Azulenyl	H	Et	Et	H	ClO <sub>4</sub>	230	625	516
2-Benzoselenazolyl	0	0	0	Et	Ph	Ph	Et	Et	Br	—	—	606	516
2-Benzoselenazolyl	0	1	0	Et	Azulenyl	H	Et	Et	H	I	220	628	516
2-Benzoselenazolyl	0	1	0	CH <sub>2</sub> -Me	Me	H	Et	(CH <sub>2</sub> ) <sub>2</sub> H	H	I	—	612	623
2-α-Naphthothiazolyl	0	1	0	Et	H	H	Et	Et	H	COOH	—	—	675
2-α-Naphthothiazolyl	0	1	0	Et	Azulenyl	H	Et	Et	H	ClO <sub>4</sub>	234	630	516
2-Acenaphtho(1,2-d)-thiazolyl	0	1	0	Et	H	Ph	Et	Et	H	OTs	259	640	353
2-Benzothieno(2,3-d)-thiazolyl	0	1	0	Et	Ph	Ph	Et	Et	H	Br	229	—	578
2-Benzothieno(3,2-d)-thiazolyl	0	1	0	Et	Ph	Ph	Et	Et	H	I	249	—	578
2-[4-Me-pyrimidino-(1,2-a)benzimidazolyl]	0	1	0	Et	Ph	Ph	(CH <sub>3</sub> ) <sub>2</sub> N <sup>+</sup>	Et	H	2OTs	218	640	624
2-[4-Me-pyrimidino-(1,2-a)benzimidazolyl]	0	1	0	Et	Ph	Ph	(CH <sub>3</sub> ) <sub>2</sub> N <sup>+</sup>	(CH <sub>3</sub> ) <sub>2</sub> Et	H	EtSO <sub>4</sub>	230	649	624

TABLE 3141C



<i>n</i>	m.p. (°C)	$\lambda$ (nm)	Ref.
1	212	655	410
2	226	652	410

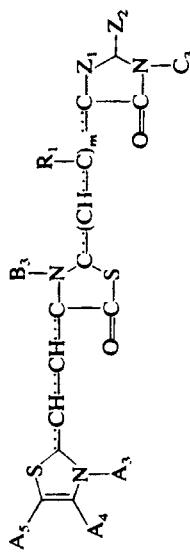
TABLE 3142A. NEUTRO RHODACYANINES



Z <sub>1</sub>	Z <sub>2</sub>	Z <sub>3</sub>	m	n	A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	B <sub>3</sub>	R <sub>1</sub>	R <sub>2</sub>	m.p. (°C)	λ (nm)	Ref.
N	SEt	S	1	0	Me	p-MeOC <sub>n</sub> H <sub>4</sub>	p-(1,1,3,3-tetraMe-Bu)C <sub>n</sub> H <sub>4</sub> O	Et	H	—	201	572	593
N	SEt	S	1	0	Et	Ph	Ph	Et	H	—	246	593	593
N	SEt	S	1	0	Et	p-MeOC <sub>n</sub> H <sub>4</sub>	OPh	Et	H	—	276	574	351, 593
N	SEt	S	1	1	Et	Ph	Ph	CH <sub>3</sub> COOEt	H	OEt	240	630	1(p. 674), 625
N	SEt	S	1	1	Et	Ph	Ph	CH <sub>3</sub> COOEt	H	SEt	—	675	1(p. 674), 625
O	S	NEt	0	0	Me	Me	CH=CH-COOEt	Et	—	—	505	567	—
O	O	S	NEt	1	0	Me	p-MeOC <sub>n</sub> H <sub>4</sub>	p-(1,1,3,3-tetraMe-Bu)C <sub>n</sub> H <sub>4</sub> O	Et	H	—	—	351
O	O	S	NEt	1	0	Et	p-MeOC <sub>n</sub> H <sub>4</sub>	OPh	Et	H	—	—	351
S	S	NEt	0	0	Me	Me	CH=CHCOOEt	Et	—	—	—	—	—
S	S	NEt	0	1	Me	Me	CH=CHCOOEt	CH <sub>3</sub> COOEt	—	—	517	567	—
S	S	NEt	1	0	Me	Me	CH=CHCOOEt	Et	Me	—	—	577	567
S	S	NEt	1	0	Me	p-Xylyl	Ph	Et	Me	—	—	614	567
S	S	NEt	1	0	Me	p-MeOC <sub>n</sub> H <sub>4</sub>	OPh	Et	H	—	—	594	1(p. 656), 593
S	S	NEt	1	0	Me	p-MeOC <sub>n</sub> H <sub>4</sub>	Ph	Et	H	—	240	584	1(p. 656),
S	S	NEt	1	0	Me	p-MeC <sub>n</sub> H <sub>4</sub>	p-(1,1,3,3-tetraMe-Bu)C <sub>n</sub> H <sub>4</sub> O	Et	H	—	252	585	351, 593
S	S	NEt	1	0	Et	Ph	Ph	Et	H	—	298	594	593, 626
S	S	NEt	1	0	Et	Ph	OPh	Et	H	—	274	584	351, 593
S	S	NEt	1	0	Et	p-ClC <sub>n</sub> H <sub>4</sub>	p-ClC <sub>n</sub> H <sub>4</sub> O	Et	H	—	246	584	351, 593
S	S	NEt	1	0	Et	p-Xylyl	Ph	Et	H	—	—	596	593
S	S	NEt	1	0	Et	p-MeOC <sub>n</sub> H <sub>4</sub>	Ph	Et	H	—	286	595	—
S	S	NEt	1	0	Et	p-MeOC <sub>n</sub> H <sub>4</sub>	OPh	Et	H	—	278	586	593
S	S	NEt	1	0	Et	3,4-diMeO-	Ph	Et	H	—	243	594	593
S	S	NEt	1	0	Et	C <sub>n</sub> H <sub>3</sub>	—	Et	H	—	—	—	—
S	S	NEt	1	0	Et	β-Naphthyl	p-MeC <sub>n</sub> H <sub>4</sub> S	Et	H	—	247	585	351, 593

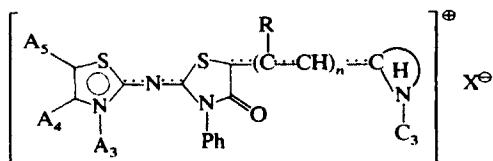
S	S	NEt	—	0	Me	Me	CH=CHCOOEt	Et	Et	—	671
S	S	NBu	—	0	Et	H	Ph	Et	Et	—	626
S	S	NBu	—	0	Et	H	2-naphthyl	Et	Et	—	626
S	S	NBu	—	1	0	Et	$\alpha$ -Furyl	Et	Et	—	626
S	S	NBu	—	1	0	Et	H	Et	Et	—	626
S	S	NBu	—	1	0	Et	Ph	Et	NHPh	—	561
S	S	NBu	—	1	0	Et	Ph	Et	NHPh	—	627
S	S	NBu	—	1	0	Et	Ph	Bu	H	—	626
S	S	NBu	—	1	0	Et	Ph	(CH <sub>2</sub> ) <sub>5</sub> COOEt	H	—	579
S	S	NBu	—	1	0	Et	Ph	Bu	H	—	601
S	S	NC <sub>3</sub> H <sub>11</sub> (n)	—	1	0	Et	Ph	C <sub>4</sub> H <sub>11</sub> (n)	H	—	579
S	S	NC <sub>3</sub> H <sub>11</sub> (n)	—	1	0	Et	Ph	Bu	H	—	605
S	S	NC <sub>7</sub> H <sub>15</sub> (n)	—	1	0	Et	Bh	H	H	—	579
S	S	NC <sub>7</sub> H <sub>15</sub> (n)	—	1	0	Et	Ph	H	C <sub>7</sub> H <sub>15</sub> (n)	—	605
S	S	N(CH <sub>3</sub> ) <sub>2</sub> COOEt	—	1	0	Et	Ph	Et	H	—	579
S	S	N(CH <sub>3</sub> ) <sub>2</sub> COOEt	—	1	0	Et	Mc	Et	H	—	595
S	S	N(CH <sub>3</sub> ) <sub>2</sub> COOEt	—	1	0	Et	Mc	Et	H	—	579
S	S	N(CH <sub>3</sub> ) <sub>2</sub> COOEt	—	1	0	Et	Ph	Et	H	—	587
S	S	N(CH <sub>3</sub> ) <sub>2</sub> COOEt	—	1	0	Et	Ph	Et	H	—	579
S	S	N(CH <sub>3</sub> ) <sub>2</sub> COOEt	—	1	0	Et	Ph	Et	H	—	589
S	S	N(CH <sub>3</sub> ) <sub>2</sub> COOEt	—	1	0	Et	Ph	Et	H	—	579
S	S	N(CH <sub>3</sub> ) <sub>2</sub> COOEt	—	1	0	Et	Ph	Ph	H	—	602
S	S	N(CH <sub>3</sub> ) <sub>2</sub> COOEt	—	1	0	Et	Ph	Bu	H	—	579
S	S	N(CH <sub>3</sub> ) <sub>2</sub> COOEt	—	1	0	Et	Ph	(CH <sub>2</sub> ) <sub>5</sub> COOEt	H	—	604
S	S	N(CH <sub>3</sub> ) <sub>2</sub> COOEt	—	1	0	Et	Ph	(CH <sub>2</sub> ) <sub>5</sub> COOEt	H	—	579
S	S	N(CH <sub>3</sub> ) <sub>2</sub> COOEt	—	1	0	Et	Ph	(CH <sub>2</sub> ) <sub>5</sub> COOBu	H	—	602
S	S	N(CH <sub>3</sub> ) <sub>2</sub> COOPr	—	1	0	Et	Ph	Bu	H	—	579
S	S	N(CH <sub>3</sub> ) <sub>2</sub> COOPr	—	1	0	Et	Ph	n-Pr	H	—	579
S	S	N(CH <sub>2</sub> ) <sub>5</sub> COO <i>i</i> -Pr	—	1	0	Et	Ph	Bu	H	—	602
S	S	N(CH <sub>2</sub> ) <sub>5</sub> COOBu	—	1	0	Et	Ph	Et	H	—	579
S	S	N(CH <sub>2</sub> ) <sub>5</sub> COOBu	—	1	0	Et	Ph	Bu	H	—	603
S	S	N(CH <sub>2</sub> ) <sub>5</sub> COOBu	—	1	0	Et	Ph	N(CH <sub>2</sub> ) <sub>5</sub> COOBu	H	—	579
S	S	N(CH <sub>2</sub> ) <sub>5</sub> COOEt	—	1	0	Et	Ph	Bu	H	—	601
S	S	N(CH <sub>2</sub> ) <sub>5</sub> COOEt	—	1	0	Et	Ph	Bu	H	—	579
S	S	N(CH <sub>2</sub> ) <sub>5</sub> COOEt	—	1	0	Et	Ph	Bu	H	—	602
S	S	N(CH <sub>2</sub> ) <sub>5</sub> COOEt	—	1	0	Et	Ph	Bu	H	—	579
S	S	(i)-Bu	—	0	Et	H	H	Et	H	—	589
S	S	NCH <sub>2</sub> COOEt	—	1	0	Et	H	Pr	H	—	579
S	S	NNHCOMe	—	1	0	Et	Ph	Pr	H	—	628

TABLE 3142B



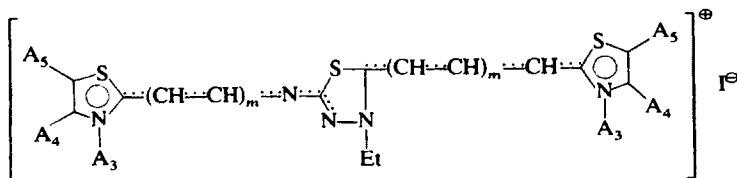
Z <sub>1</sub>	Z <sub>2</sub>	m	A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	B <sub>3</sub>	C <sub>3</sub>	R	m.p. (°C)	λ (nm)	Ref.	
O	S	0	Me	p-MeOC <sub>6</sub> H <sub>4</sub>		p-(1,1,3,3-tetra-MeBu)C <sub>6</sub> H <sub>4</sub> O	Me	Et	—	248	608	1(p. 656), 351, 593
O	S	0	Et	p-MeOC <sub>6</sub> H <sub>4</sub>	Ph	Ph	Me	Et	—	281	613	593
O	S	0	Et	p-MeOC <sub>6</sub> H <sub>4</sub>	OPh	Ph	Me	Et	—	233	607	351, 593
O	S	0	Me	p-Xylyl	Ph	Ph	Me	Et	—	277	624	1(p. 656), 593
S	S	0	Me	p-MeOC <sub>6</sub> H <sub>4</sub>	OPh	OPh	Me	Et	—	286	613	1(p. 656), 351, 593
S	S	0	Me	p-MeOC <sub>6</sub> H <sub>4</sub>		p-(1,1,3,3-tetra-MeBu)C <sub>6</sub> H <sub>4</sub> O-	Me	Et	—	258	613	1(p. 656), 351, 593
S	S	0	Me	β-Naphthyl		p-MeC <sub>6</sub> H <sub>4</sub> S-	Me	Et	—	276	613	593
S	S	0	Et	Ph	Ph	p-MeC <sub>6</sub> H <sub>4</sub> S-	Me	Et	—	298	622	593
S	S	0	Et	Ph	Ph	p-MeC <sub>6</sub> H <sub>4</sub> S-	Me	Et	—	224	615	351, 593
S	S	0	Et	p-BrC <sub>6</sub> H <sub>4</sub>		p-BrC <sub>6</sub> H <sub>4</sub> O-	Me	Et	—	236	618	351, 593
S	S	0	Et	p-ClC <sub>6</sub> H <sub>4</sub>		p-ClC <sub>6</sub> H <sub>4</sub> O-	Me	Et	—	275	600	351, 593
S	S	0	Et	p-MeOC <sub>6</sub> H <sub>4</sub>	Ph	Ph	Me	Et	—	288	623	593
S	S	0	Et	p-MeOC <sub>6</sub> H <sub>4</sub>	OPh	OPh	Me	Et	—	253	617	351, 593
S	S	0	Et	3,4-diMeO	Ph	Ph	Me	Et	—	288	622	593
S	S	0	Et	C <sub>6</sub> H <sub>3</sub>		p-MeC <sub>6</sub> H <sub>4</sub> S-	Me	Et	—	—	—	351
S	S	0	Et	β-Naphthyl	Ph	p-MeC <sub>6</sub> H <sub>4</sub> S-	Et	Allyl	—	—	682	1(p. 674), 625
S	S	1	Et	Ph	Ph	Ph	Et	Allyl	Me	—	730	1(p. 674), 625
S	S	1	Et	Ph	Ph	Ph	Et	Allyl	OEt	—	710	1(p. 674), 625
S	S	1	Et	Ph	Ph	Ph	Et	CH <sub>2</sub> COO	SEt	—	752	1(p. 674), 625
S	NPh <sub>2</sub>	0	Et	p-MeOC <sub>6</sub> H <sub>4</sub>	OPh	Me	Et	—	193	600	351, 593	

TABLE 315A. AZA CYANINES

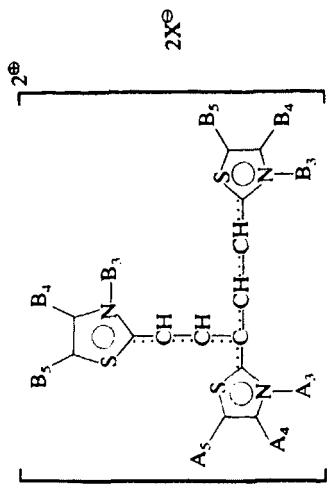


H	<i>n</i>	A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	C <sub>3</sub>	R	X	λ (n.m.)	Ref.
2-(5,6-diMe-benzoxazolyl)	1	Me	Me	H	Et	H	I	524	562
2-Benzothiazolyl	1	Et	Ph	Me	Et	Et	I	—	562
2-(5-EtO-benzoselenazolyl)	0	Et	Ph	Me	Me	—	Br	460	562

TABLE 315B



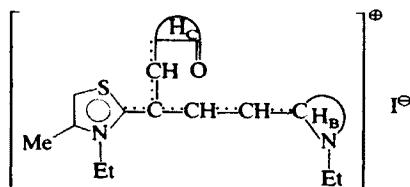
<i>m</i>	A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	Ref.
0	Me	<i>p</i> -MeC <sub>6</sub> H <sub>4</sub>	Ph	629, 630
1	Me	H	H	629, 630
1	Et	Ph	Ph	629, 630

TABLE 321A.  $\alpha$ -BRANCHED CYANINES: NEOCYANINES

$A_3$	$A_4$	$A_5$	$B_3$	$B_4$	$B_5$	X	m.p. (°C)	$\lambda$ (nm)	Ref.
Me	H	H	Me	H	H	I	—	—	374 e
Me	Me	H	Me	Me	H	I	—	—	1(p. 629), 344, 631
Me	Me	H	Me	Me	H	Nicotinate	148	—	360, 632 a
Me	Me	H	Me	Me	H	Aspartate	—	—	633 a
Me	Me	H	Me	Me	H	Glucuronate	—	—	634 a
Me	Me	H	Me	Me	H	Glutamate	—	—	634 a
Et	H	Styryl	Et	$\alpha$ -(4-MeO-naphthyl)	Styryl	$EtOCO(CN)C=CH-C(CN)COOEt$	170	595	500
Et	H	Styryl	Et	$Styryl$	H	$CN-CH=C(CN)CH-$ $(CN)CN$	244	600	500
Me	$-(CH_2)_3^-$	Me	$Et$	$-(CH_2)_3^-$	H	I	264	620	399
Et	H	Styryl	Et	$Styryl$	H	I	247	605	388
Et	H	Styryl	Et	$Styryl$	H	I	—	668	357

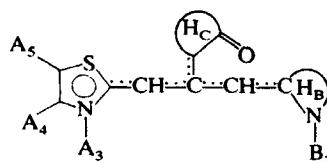
Et	Me	H	Et	Me	H	I		—	587
Et	Me	H	Et	CH <sub>2</sub> -thiazolinyl	CH <sub>2</sub> -thiazo-linyl	ClO <sub>4</sub>		—	534
Et	<i>p</i> -( <i>p</i> -MeOC <sub>6</sub> H <sub>4</sub> )C <sub>6</sub> H <sub>4</sub>	H	Et	<i>p</i> -( <i>p</i> -MeOC <sub>6</sub> H <sub>4</sub> )C <sub>6</sub> H <sub>4</sub>	H	I	190	638	388
Et	<i>p</i> PhOC <sub>6</sub> H <sub>4</sub>	H	Et	<i>p</i> -PhOC <sub>6</sub> H <sub>4</sub>	H	I	180	600	388
Pr	Me	H	Pr	Me	H	I	208	—	635
<i>i</i> -Am	Me	H	<i>i</i> -Am	Me	H	I	200	—	635
<i>n</i> -Heptyl	Me	H	<i>n</i> -Heptyl	Me	H	Cl	—	—	146, 636
<i>n</i> -Heptyl	Me	H	<i>n</i> -Heptyl	Me	H	Nicotinate	80	—	360

TABLE 321B

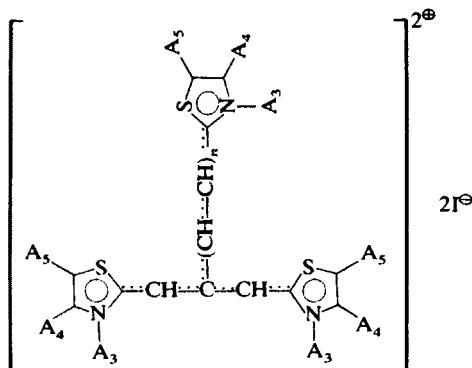


H <sub>B</sub>	H <sub>C</sub>	m.p. (°C)	λ (nm)	Ref.
2-Thiazolinyl	N-Et-rhodanine	214	536	1(p. 629), 13, 534
2-Benzothiazolyl	1-Ph-3-Et-thiohydantoin	225	576	1(p. 629), 13, 534

TABLE 322A. β-BRANCHED HOLOPOLAR CYANINES

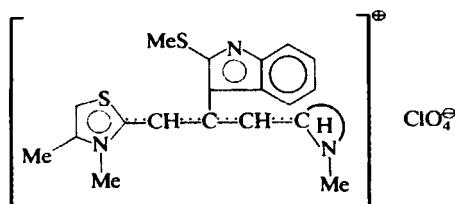


H <sub>B</sub>	H <sub>C</sub>	A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	B <sub>3</sub>	m.p. (°C)	Ref.
2-(4,5-diPh-thiazolyl)	4-(1-Ph-3-Me-2,3'-sulfo-propenyl pyrazolonylidene)	Me	Ph	Ph	Me	320	637-640
2-Benzothiazolyl	4-(1-Ph-3-Me-2,3'-sulfo-propenyl pyrazolonylidene)	Me	Ph	Ph	Me	320	637-640
2-Benzothiazolyl	3-(Chroman-2,4-dione)	Et	H	H	Et	—	641
2-(6-MeO-benzo-	4-(1-Ph-3-Me-2,3'-sulfopropenyl pyrazolonylidene)	Me	Ph	Ph	Me	283	637-640
2-(5,6-diMeO-benzo-	4-(1-Ph-3-Me-2,3'-sulfopropenyl pyrazolonylidene)	Me	Ph	Ph	Me	329	637-640

TABLE 322B.  $\beta$ -BRANCHED CYANINES

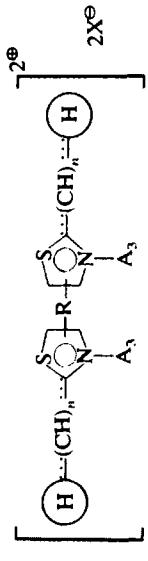
<i>n</i>	A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	m.p. (°C)	Ref.
0	Me	H	H	250	495
0	Me	Me	H	283	495
1	Me	H	H	—	1(p. 677), 485, 530
1	Me	Me	H	—	1(p. 677), 485, 530
1	Me	Ph	H	—	1(p. 677), 485
1	Et	H	H	—	1(p. 677), 485, 530
1	Et	Me	H	—	1(p. 677), 485
1	Et	Et	H	—	530
1	Et		-(CH <sub>2</sub> ) <sub>4</sub> -	—	1(p. 628), 400
1	Et	Ph	H	—	1(p. 677), 485
1	Et	$\alpha$ -Benzofuryl	H	—	468
1	Et	$\alpha$ -Thienyl	H	—	470, 494

TABLE 322C



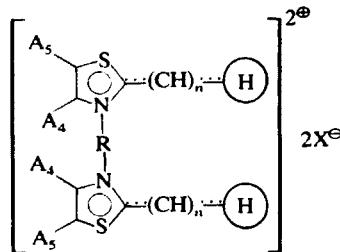
H	m.p. (°C)	$\lambda$ (nm)	Ref.
2-(4-Ph-thiazolyl)	125	585	642
2-Benzothiazolyl	191	620	642

TABLE 41A. TETRANUCLEAR CYANINES: TWO CYANINES



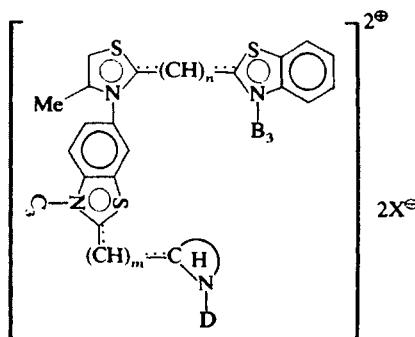
H	n	R	Link	A <sub>3</sub>	X	m.p. (°C)	λ (nm)	Ref.
p-Me <sub>2</sub> N <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	2	p-C <sub>6</sub> H <sub>4</sub>	4-4'	Me	I	—	—	643
p-Me <sub>2</sub> NC <sub>6</sub> H <sub>4</sub>	4	p-C <sub>6</sub> H <sub>4</sub>	4-4'	Me	I	—	—	643
p-Me <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> =	1	p-C <sub>6</sub> H <sub>4</sub>	4-4'	Me	I	—	—	643
1,1,3-triMe-indolonyl	3	p-C <sub>6</sub> H <sub>4</sub>	4-4'	Me	ClO <sub>4</sub>	281	544	506
1,1,3-triMe-indolonyl	3	p-C <sub>6</sub> H <sub>4</sub>	5-5'	Me	ClO <sub>4</sub>	275	530-	506
							602	
2-(6-Me-1-Et-quinolyl)	1	—	4-4'	Et	I	245	497	644, 645
2-(3-Et-benzoxazolyl)	3	—	4-4'	Et	I	273	532	644, 645
2-(3-Et-benzoxazolyl)	5	—	4-4'	(CH <sub>2</sub> ) <sub>2</sub> OPh	I	193	613	644, 645
2-(3-Et-benzothiazolyl)	3	—	4-4'	Et	I	23.5	577	644, 645
2-(3-Et-5,6-methylene dioxybenzothiazolyl)	3(μ-Me)	—	4-4'	Et	I	249	600	644, 645
2-(3-Et-5,6-methylene dioxybenzothiazolyl)	3(μ-Me)	—	4-4'	Et	I	267	573	644, 645
2-(3-Et-5,6-methylene dioxybenzothiazolyl)	3	—	4-4'	Et	I	23.0	571	644, 645
2-(3-Me-β-naphthothiazolyl)	3	—	4-4'	Et	I	23.6	590	644, 645

TABLE 41B



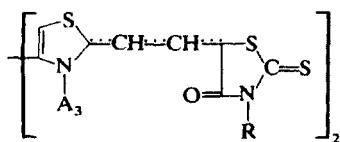
H	n	R	A <sub>4</sub>	A <sub>5</sub>	X	m.p. (°C)	λ (nm)	Ref.
p-Me <sub>2</sub> NC <sub>6</sub> H <sub>4</sub>	2	(CH <sub>2</sub> ) <sub>2</sub>	Ph	H	I	281	—	1(p. 677), 646
2-(1-Et-quinolyl)	1	(CH <sub>2</sub> ) <sub>2</sub>	Ph	H	I	292	—	1(p. 677), 646
2-(1-Bu-quinolyl)	3	(CH <sub>2</sub> ) <sub>2</sub>	Ph	H	ClO <sub>4</sub>	—	—	1(p. 677), 646
2-(3-Et-benzoxazolyl)	3	(CH <sub>2</sub> ) <sub>2</sub>	Me	H	I	208	—	1(p. 677), 646
2-(3-Et-benzoxazolyl)	3	(CH <sub>2</sub> ) <sub>2</sub>	Ph	H	I	288	—	1(p. 677), 646
2-(5-Me-3-Et-benzoxazolyl)	3	(CH <sub>2</sub> ) <sub>2</sub>	Ph	H	I	261	—	1(p. 677), 646
2-(5,6-diMe3-Et-benzoxazolyl)	3	(CH <sub>2</sub> ) <sub>2</sub>	Me	H	I	—	—	1(p. 678), 460
2-(5-Ph-3-Et-benzoxazolyl)	3	(CH <sub>2</sub> ) <sub>2</sub>	Ph	H	I	314	—	1(p. 677), 646
2-(3-Et-benzothiazolyl)	3	(CH <sub>2</sub> ) <sub>2</sub>	Ph	H	I	298	—	1(p. 677), 646
2-(3-Et <sup>5</sup> -Cl-benzothiazolyl)	3	(CH <sub>2</sub> ) <sub>2</sub>	Ph	H	I	292	—	1(p. 677), 646
2-(3-Et-β-naphthothiazolyl)	3	(CH <sub>2</sub> ) <sub>2</sub>	Ph	H	I	254	—	1(p. 677), 646
2-(3-Et-benzoxazolyl)	3	(CH <sub>2</sub> ) <sub>3</sub>	Ph	H	I	162	—	1(p. 677), 646
2-(1,1,3-triMe-indoleny)	3	p-C <sub>6</sub> H <sub>4</sub>	H	H	ClO <sub>4</sub>	—	—	339
2-(1,1,3-triMe-indoleny)	3	p-C <sub>6</sub> H <sub>4</sub>	H	Ph	ClO <sub>4</sub>	—	—	339, 647
2-(1,1,3-triMe-indoleny)	3	m-C <sub>6</sub> H <sub>4</sub>	H	Ph	ClO <sub>4</sub>	—	—	339, 647
2-(1,1,3-triMe-indoleny)	3	p-C <sub>6</sub> H <sub>4</sub>	Me	H	ClO <sub>4</sub>	230	516— 548	339, 506
2-(1,1,3-triMe-indoleny)	3	p-C <sub>6</sub> H <sub>4</sub>	Ph	H	ClO <sub>4</sub>	—	—	647
2-(1,1,3-triMe-indoleny)	3	m-C <sub>6</sub> H <sub>4</sub>	Ph	H	ClO <sub>4</sub>	—	—	647
2-(3-Et-2-Me-benzothiazolyl)	3	p-C <sub>6</sub> H <sub>4</sub>	Me	H	ClO <sub>4</sub>	—	—	339

TABLE 41C



H	m	n	B <sub>3</sub>	C <sub>3</sub>	D	X	m.p. (°C)	Ref.
2-Quinolyl	3	1	Me	Me	Me	I	300	118
2-Benzothiazolyl	1	1	Me	Me	Me	ClO <sub>4</sub>	300	118
2-Benzothiazolyl	3	1	Me	Me	Et	ClO <sub>4</sub>	310	118
2-Benzothiazolyl	3	3	Et	Et	Et	ClO <sub>4</sub>	240	118

TABLE 42 TWO NEUTROCYANINES LINKED BY THE THIAZOLE RING



A <sub>3</sub>	R	Ref.
Me	Et	1 (p. 626), 648
Me	Allyl	1(p. 626), 648
Me	Ph	1(p. 626), 648
Et	Allyl	1(p. 626), 649, 650
Et	Ph	1(p. 626), 649, 650

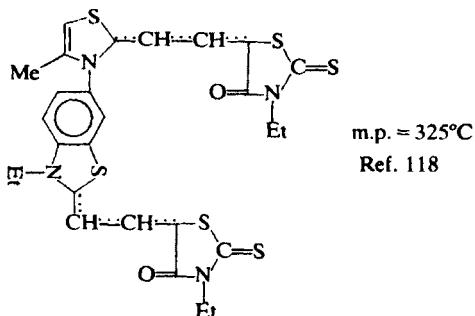
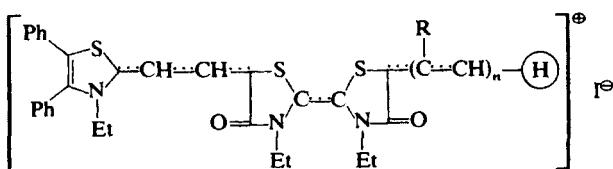
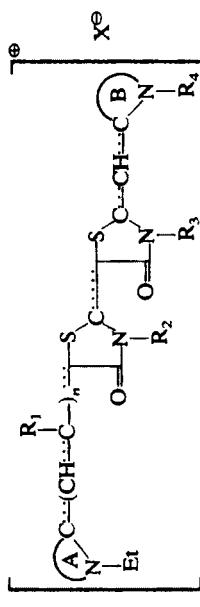


TABLE 43A TWO NEUTROCYANINES LINKED BY THE KETOMETHYLENE



H	n	R	Ref.
4-(1-Et-quinolylidene)	1	H	651-654
2-(3-Et-benzothiazolylidene)	2	H	651-654
2-(3-Et-5,6-diMe-benzothiazolylidene)	1	Ph	651-654
2-(3-Et-5-EtO-6-Me-benzothiazolylidene)	1	Ph	651-654
2-(3-Et-5,6-diMe-benzoselenazolylidene)	1	Ph	651-654
5-(5-MeO-benzoselenazolylidene)	1	Ph	651-654

TABLE 43B

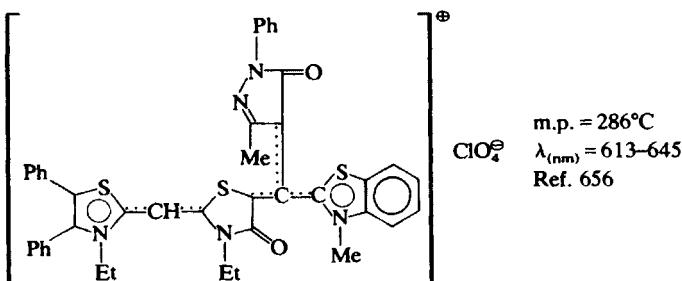
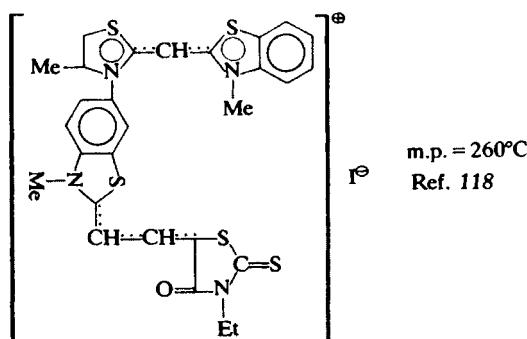


A	B	n	R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	R <sub>4</sub>	X	m.p. (°C)	$\lambda$ (nm)	Ref.
4-Quinolyl	4,5-diPh-thiazolyl	1	H	Et	Et	Et	I	—	708	665
4-(3,4-diMeOC <sub>6</sub> H <sub>3</sub> )- 5-Ph-thiazolyl	4-(3,4-diMeOC <sub>6</sub> H <sub>3</sub> )- 5-Ph-thiazolyl	1	H	Et	Et	Et	OTs	192	650	1(p. 656), 593
4-(p-MeO-C <sub>6</sub> H <sub>4</sub> )- 5-Ph-thiazolyl	2-β-Naphthothiazolyl	1	H	Et	Et	Et	OTs	266	620	1(p. 656), 593
4-(p-MeO-C <sub>6</sub> H <sub>4</sub> )- 5-MeO-benzothiazolyl	4,5-diPh-thiazolyl	1	Ph	CH <sub>2</sub> COOMe	Et	Allyl	I	—	657	665
5,6-diMe-benzothiazolyl	4,5-diPh-thiazolyl	1	Ph	Et	Et	Et	I	—	649	665
5,6-diMe-benzothiazolyl	4,5-diPh-thiazolyl	1	Ph	CH <sub>2</sub> Ph	CH <sub>2</sub> Ph	Et	I	—	650	665
5-EtO-6-Me- benzothiazolyl	4,5-diPh-thiazolyl	1	Ph	Et	Et	Et	I	—	659	665
5,6-diMe-benzo- selenazolyl	4,5-diPh-thiazolyl	1	Ph	Et	Et	Et	I	—	651	665
5,6-diMe-benzo- selenazolyl	4,5-diPh-thiazolyl	1	Ph	CH <sub>2</sub> Ph	CH <sub>2</sub> CH <sub>2</sub> OH	C <sub>3</sub> H <sub>6</sub> -Br	—	652	665	
5-MeO-benzoselenazolyl	4,5-diPh-thiazolyl	1	Ph	Et	Et	COOH	—	—	—	—
Benzothiazolyl	4,5-diPh-thiazolyl	2	H	Et	Et	Et	I	—	650	665
						Et	I	—	690	665
										Ref. 655

TABLE 43C

$\left[ \text{A}_5 \text{---} \text{S} \text{---} \text{CH} \cdots \text{N} \text{---} \text{CH} \cdots \text{S} \text{---} \text{C}(\text{R})_n \cdots \text{S} \text{---} \text{CH} \cdots \text{N} \text{---} \text{CH} \cdots \text{S} \text{---} \text{C}(\text{Et})_2 \right]^{2\Theta} \cdot 2\text{I}^\ominus$						
$\text{A}_4$	$\text{A}_5$	$n$	$\text{R}$	m.p. (°C)	$\lambda$ (nm)	Ref.
Me	H	1	Pr	279	518-437	1(p. 676), 461, 462
Me	H	5	H	237	420-734	1(p. 676), 461, 462
Ph	Me	1	Et	225	627	1(p. 676), 461, 462

TABLE 44. ONE CYANINE AND ONE NEUTROCYANINE



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X

# Selenazole and Derivatives

ROBERT J. GUGLIELMETTI

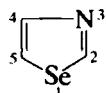
*Université de Bretagne Occidentale, Brest, France*

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## I. INTRODUCTION

1,3-Selenazole is a 5-membered heterocycle of the azole series, containing two heteroatoms. Other members of this group include thiazole and oxazole, whose chemistry is more developed because of their easier access and lower toxicity. The numbering of the heterocyclic system is as follows:



The earliest work on selenazole dates back to 1889, but intensive studies of the compound and its derivatives only really started in 1940. In 1889, the question of the existence under normal conditions of nonsubstituted selenazole was posed as a result of the first studies of the azoles by the group led by Hantzsch.

Hofmann (1), of the Zurich School, was the first to have tried unsuccessfully to prepare the unsubstituted parent compound, selenazole; much later, in 1955, Metzger and Bailly (2) were equally unsuccessful in trying to prepare selenazole from 2-aminoselenazole by reduction of the diazo compound.

This chapter is an attempt to present the important results of studies of the synthesis, reactivity, and physicochemical properties of this series of compounds. The subject was surveyed by Bulka (3) in 1963 and by Klayman and Gunther (4) in 1973. Unlike the oxazoles and thiazoles, there are few convenient preparative routes to the selenazoles. Furthermore, the selenium intermediates are difficult to synthesize and are often extremely toxic: selenoamides tend to decompose rapidly depositing metallic selenium. This inconvenience can be alleviated by choice of suitable reaction conditions. Finally, the use of selenium compounds in preparative reactions is often complicated by the fragility of the cycle and the deposition of metallic selenium.

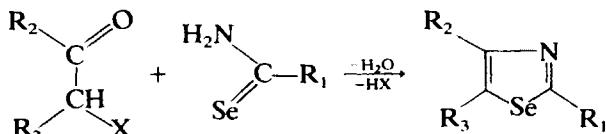
Selenazoles and their derivatives, that is, selenazolines and selenazolidines, are mainly used in cyanine-type dyes and photographic sensitizers as well as in pharmacology and chemotherapy.

## II. GENERAL PREPARATIVE METHODS OF SELENAZOLES

The different functional categories of the selenazoles have been classed in according to the functional priority or to the quantity of prepared compounds.

### 1. Substituted Alkyl and Arylselenazoles

Hoffmann (1), a student of Hantzsch, used the condensation of selenobenzamide with  $\alpha$ -halogenated carbonyl derivatives to prepare a series of 2-phenylselenazoles according to the Scheme 1.



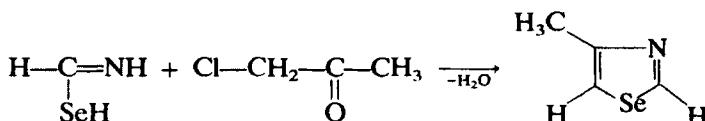
Scheme 1

The alkylselenazoles were prepared much later from selenoacetamide and selenopropionamide ( $\text{R}_1 = \text{Me}$  and  $\text{Et}$ ) (5, 6).

The difficulties posed by the synthesis of 2-alkyl and 2-arylselenazoles substituted in the 4- and 5-positions arise mainly from the preparation of the intermediates, especially the selenoamides.

Thus a second method was envisaged, the reaction of a nitrile, hydrogen selenide, and an  $\alpha$ -halogenated ketone in the presence of a condensation catalyst, which can be  $\text{POCl}_3$  or  $\text{POCl}_3$  with a Lewis acid such as  $\text{PCl}_3$  or anhydrous  $\text{ZnCl}_2$ . The use of fresh  $\text{AlCl}_3$  leads to the formation of tarry side-products.

The maximum yield of 2-alkylselenazole is 25%. In this way, 4-methylselenazole (7) was obtained starting from hydrogen cyanide, hydrogen selenide and chloroacetone. It is the only known selenazole not substituted in the 2-position. The yield relative to chloroacetone is very low (2.5%) (Scheme 2).



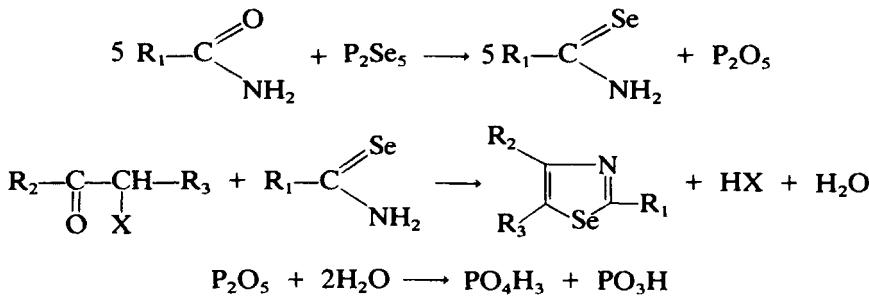
Scheme 2

In thiazole series the application of Tcherniac's method (8), which consists of cyclizing the intermediary iminoketothioether, obtained by reaction of a thiocyanoketone upon a labile hydrogen compound, is suitable and occurs with an average yield of 50%. In selenazole series this cyclization does not happen from selenocyanoketones and by using different acidic media (9).

The O-S exchange method in presence of  $\alpha$ -halogenated carbonyl compound is a very good one for thiazole compounds. The thioamide is prepared in situ by the action of amide upon phosphorus pentasulphide with solvent. The  $\alpha$ -halogenated aldehyde reacts directly. But the O-Se exchange cannot be performed with  $\alpha$ -halogenated carbonyl compounds because of the apparition of phosphoric acid. (Scheme 3). The C-Se bond is very sensitive to acid pH.

Moreover, selenamides are more instable than thioamides; so  $\text{P}_2\text{Se}_5$  was replaced by  $\text{Al}_2\text{Se}_3$ . In that case the cycle is less broken, but the yield of reaction is not as good.

Lena (9) has used this method to prepare the 2-phenyl-4-methylselenazole and the 2,4-dimethylselenazole. An optimization of the experimental conditions in the O-Se exchange must be necessary.

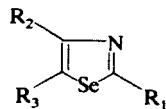


The physical properties of the principal alkyl- and arylselenazoles synthesized are given in Table X-1 along with the synthetic methods used and bibliographic references.

Alkylselenazoles are oily alkaline liquids possessing a smell similar to that of the corresponding thiazole or pyridine derivatives. The crystalline picrates or 3-methylselenazolium iodides have been used for the purpose of characterization. Alkyl derivatives are partially soluble in water; aryl derivatives are insoluble.

Alkyl and aryl selenazoles are weakly basic, and their quaternary salts are easily hydrolyzed in aqueous solution.

TABLE X-1. ALKYL AND ARYL SELENAZOLES

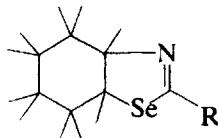


R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	b.p./mm (°C)	m.p. (°C)	m.p. (picrate) (°C)	Ref.
Me	H	H	149/760 32-4/20	—	170	5, 7, 11, 12
H	Me	H	152-3/760	—	197	6
Me	Me	H	163-4/760 56-8/17	—	155	6, 7, 11-13
Me	Me	Me	176/760	—	164	11
Me	Ph	H	—	63-4	—	7, 11-13
Et	Me	H	74-6/20	—	—	5, 11, 12
Ph	Me	H	282-3/737	—	—	1
Ph	Ph	H	—	99	—	1
Ph	Me	COOH	—	206-7	—	1
Ph	Me	COOEt	—	123-4	—	1

It can be noted that the thiazoles are generally more volatile than the corresponding pyridines, while the boiling points of the selenazoles are much higher than others:

	b.p. (°C)		b.p. (°C)		b.p. (°C)
Pyridine	114.8	Thiazole	116.8	Not isolated	—
2-Picoline	129	2-Methyl-thiazole	128	2-Methyl-selenazole	149
3-Picoline	143	4-Methyl-thiazole	130	4-Methyl-selenazole	152.3
2,4-Lutidine	157	2,4-Dimethyl-thiazole	144	2,4-Dimethyl-selenazole	162.4

2-Alkyl or arylhexahydrobenzoselenazoles (Scheme 4) are synthesized by reaction of  $\alpha$ -chlorocyclohexylamine with selenocarboxamides (10).

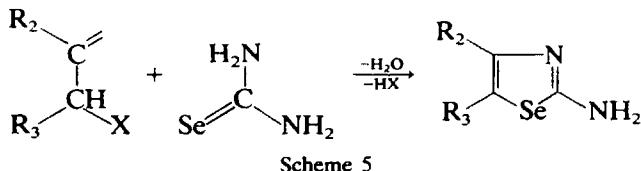


Scheme 4

## 2. 2-Aminoselenazoles Substituted in the 4- and 5-positions

### A. Synthesis from Selenourea

The replacement of selenoamide by selenourea in the Hantzsch's synthesis (1st method) leads to 2-aminoselenazoles (2, 14, 15). This series of compounds has been well developed, mainly because selenourea is much more easily accessible than the selenoamides, but also because a wide variety of  $\alpha$ -halogenated carbonyl compounds are available for the Hantzsch's cyclization reaction (Scheme 5). 2-Aminoselenazole itself was prepared from commercially available chloroacetaldehyde semihydrate

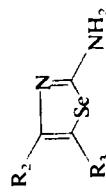


Scheme 5

TABLE X-2. 2-AMINOSELENAZOLES

$R_2$	$R_3$	m.p. (°C)	m.p. (picrate) (°C)	m.p. (acetyl derivative) (°C)	m.p. (HCl) or HBr derivative (°C)	Ref.
H	H	121 (121.5)	236 (209)	210	—	1, 2, 14
Me	H	79-80	—	122	—	1
		78-79	225	—	189 (HCl)	16, 18, 19
		99	219	—	223 (HCl)	14
Et	H	63	213	—	—	14
Me	Me	51-2	220 (dec.)	125-125.5	—	18
tBu	H	108-9	257 (dec.)	155-155.5	HCl 227 (dec.)	18
	$(CH_2)_4$	98.5	212-213	160.5	HBr 196	18
Ph	H	125.5-126	240 (dec.)	141	—	18
		132-3	—	—	—	16, 20
<i>p</i> -Cl-C <sub>6</sub> H <sub>4</sub>	H	132	198-200 (dec.)	196.5-197.5	—	1, 2, 18
		160-160.5	245-8 (dec.)	263-4	—	18
		160-2	—	269-270	—	20
<i>m</i> -NO <sub>2</sub> -C <sub>6</sub> H <sub>4</sub>	H	194-5	—	306-7	—	20
Ph	Me	141-2	—	206-7	—	20
Ph	Ph	189-190	—	204-5	—	20
Me	COOH	181-2 (~195)	— <sup>a</sup>	200 <sup>a</sup>	—	16, 1
Me	COOEt	180-1	— <sup>a</sup>	216-8 <sup>a</sup>	—	20
Ph	COOMe	253	—	—	—	17, 21, 22
Ph	CH <sub>2</sub> COOH	253	—	—	—	21
Ph	COOEt	250 (dec.)	—	—	—	21
Ph	(CH <sub>2</sub> ) <sub>2</sub> COOH	250 (dec.)	—	—	—	21
<i>p</i> -Me-C <sub>6</sub> H <sub>4</sub>	CH <sub>2</sub> COOH	246 (dec.)	—	—	—	21
<i>p</i> -Me-C <sub>6</sub> H <sub>4</sub>	COOMe	246 (dec.)	—	—	—	21
NH <sub>2</sub>	H	—	—	220-250 (HCl)	—	15

<sup>a</sup> These results seem very surprising because of the little difference between the melting point of the compound with COOH and COOEt.



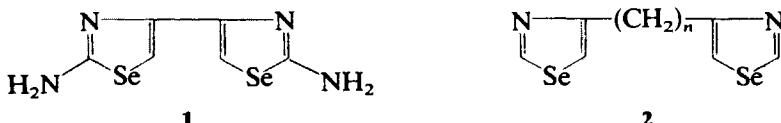
(14, 2) and from  $\alpha,\beta$ -dichlorodiethylether (1). Selenourea is obtainable by action of hydrogen selenide on an aqueous solution of cyanamide at 50 to 60°C (14) in the form of colorless crystals, which after recrystallization melt at 217°C. The yield of selenium reaches 80%. The cyanamide solution is obtained by the action of carbon dioxide upon calcium cyanamide at 0°C followed by neutralization and precipitation of the residual calcium with oxalic acid.

4-Methyl-2-aminoselenazole and 4-ethyl-2-aminoselenazole were prepared similarly from 2-chloropropanal and 2-chlorobutanal, respectively (14). With  $\alpha$ -haloketones, 2-aminoselenazoles, substituted in the 4- and 5-positions with alkyl or aryl groups, are obtained (1, 2, 16).  $\alpha$ -Haloketoacids or their esters with selenourea give 2-amino-5-carboxy selenazoles or their corresponding esters (1, 16, 17).

The most important 2-aminoselenazoles, along with their physical properties are shown in Table X-2.

With  $\alpha$ -halogenated carbonyl derivatives such as  $\alpha,\alpha'$ -dibromobiacyl,  $\text{Br}-\text{CH}_2-\overset{\text{O}}{\text{C}}-\text{C}-\text{CH}_2-\text{Br}$ , and selenourea, 2,2'-diamino-4,4'-biselenazole

(1) can be obtained (Scheme 6) (18). 4,4-Biselenazole derivatives ( $n = 0$ ) (2) (95) (Scheme 6), as well as the corresponding  $\alpha,\omega$  bis (selenazol-4-yl) alkanes ( $n = 3, 4$ ) may be synthesized with dibromo diacetyl or other  $\alpha,\alpha'$ -dibromo)1,2-diketones .

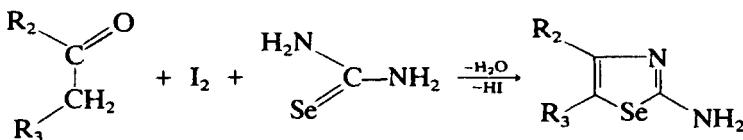


Scheme 6

The dibromohydrate is first produced, and treatment with ammonia gives the free base, which after recrystallization in nitrobenzene gives brown crystals (m.p. 215°C). The picrate decomposes around 254°C.

2,4-Diaminoselenazole was obtained by the action of selenourea on chloracetonitrile (15). Its salts are fairly unstable and decompose to give metallic selenium. All attempts to obtain the free base or 2,4-dihydroxyselenazole from 2,4-diaminoselenazole hydrochloride were unsuccessful and led to decomposition of the heterocycle.

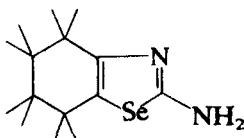
As in the case of the thiazoles, a variation on the Hantzsch's method has been used. This consists of using a nonhalogenated carbonyl derivative directly in the presence of iodine in the reaction with selenourea (Scheme 7) (20). However, in this case the reaction with selenourea is slower than with thiourea, and normally an excess of carbonyl compound is used.



Scheme 7

For 2-amino-4-(*m*-nitrophenyl) selenazole, the yield is particularly high. This has been explained by the oxidizing effect of the nitro group, which liberates iodine from the hydrogen iodide eliminated in the condensation reaction.

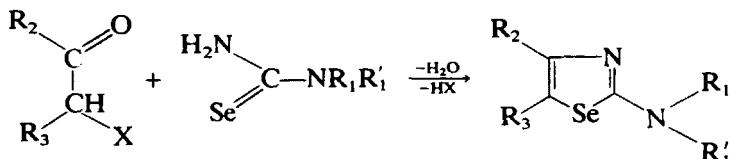
Derivatives analogous to benzoselenazole but possessing an alicyclic ring, such as 2-amino-4,5,6,7-tetrahydrobenzoselenazole (Scheme 8) are obtained from  $\alpha$ -chlorocyclohexanone and selenourea (18, 20).



Scheme 8

### B. Synthesis from *N*-substituted Selenoureas

*N*-substituted selenoureas give the same condensation reaction (Scheme 9, Table X-3).

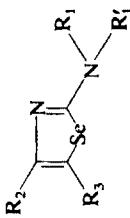


Scheme 9

Mono ( $H_2N-C(=Se)-NR_1$ ) or disubstituted ( $H_2N-C(=Se)-NR_1R_1'$ ) selenoureas can be used. The group  $R_1$ ,  $R_1'$  can be hydrogen, alkyl, aryl, acyl, or benzoyl.

The selenoureas needed for condensation can usually be prepared by addition of hydrogen selenide to a solution of the corresponding cyanamides or carbodiimides. They can be less dangerously and quite

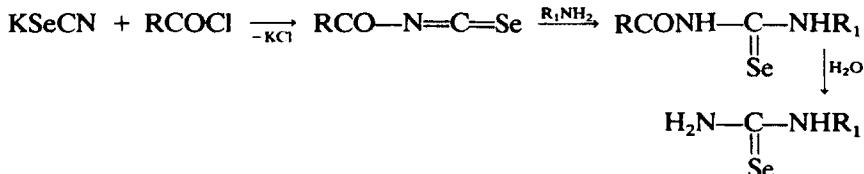
TABLE X-3a. 2-(ALKYLAMINO) AND 2-(ARYLAMINO)-SELENAZOLES



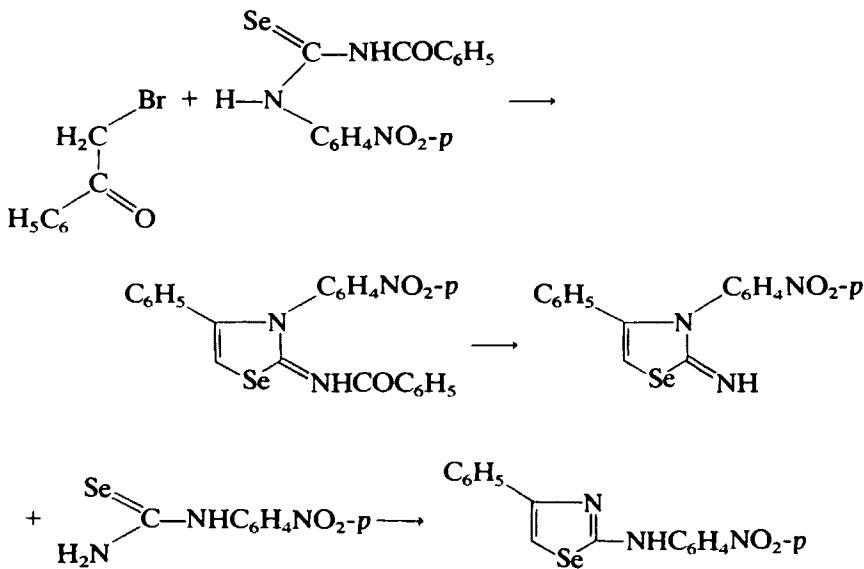
R <sub>1</sub>	R' <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	b.p./mn (°C)	m.p. (°C)	m.p. (picrate) (°C)	m.p. (acetyl derivative) (°C)	m.p. (HCl) or HBr derivative) (°C)	Ref.
Me	H	Me	H	126-7/3	69	213-4 (dec.)	—	—	26
Me	Me	Me	H	—	176-8 (dec.)	—	—	121-4 (HCl)	27
Et	Et	Me	H	125-7/20	—	166-7	—	—	26, 27
Ph	H	H	H	—	145	—	105	—	28
Ph	H	H	Me	—	132	—	120	—	28
Ph	H	H	Me	H	103	204-6 (dec.)	91	154 (HCl)	27, 28
Ph	H	Me	Me	—	81	—	121	129 (HCl)	28
Ph	H	Me	CO <sub>2</sub> Et	—	144	—	172	185 (HCl)	28
Ph	H	Et	H	—	78	—	98	143 (HCl)	28
Ph	H	Ph	H	—	132	—	134	191 (HBr)	28, 29
Ph	H	Ph	Ph	—	162	—	187	196 (HCl)	28, 29
Ph	H	p-MeC <sub>6</sub> H <sub>4</sub>	H	—	128	—	160	198 (dec.) (HCl)	28
Ph	H	p-MeOC <sub>6</sub> H <sub>4</sub>	H	—	149	—	93	243 (dec.) (HCl)	28
Ph	H	p-ClC <sub>6</sub> H <sub>4</sub>	H	—	175	—	148	238 (dec.) (HBr)	28
Ph	H	p-NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	H	—	231	—	132	251 (HBr)	28
p-MeC <sub>6</sub> H <sub>4</sub>	H	H	H	—	127	207	—	—	28
p-MeC <sub>6</sub> H <sub>4</sub>	H	Me	H	—	105	—	102	—	28
p-MeC <sub>6</sub> H <sub>4</sub>	H	Et	H	—	106	—	106	105 (HCl)	28
p-MeC <sub>6</sub> H <sub>4</sub>	H	Ph	H	—	138	—	147	199 (HBr)	28
p-MeC <sub>6</sub> H <sub>4</sub>	H	p-MeC <sub>6</sub> H <sub>4</sub>	H	—	141	—	167	218 (dec.) (HCl)	28
o-MeC <sub>6</sub> H <sub>4</sub>	H	H	H	—	123	—	80	—	28
o-MeC <sub>6</sub> H <sub>4</sub>	H	Me	H	—	127 (dec.)	—	89	176 (dec.) (HCl)	28

<i>o</i> -MeC <sub>6</sub> H <sub>4</sub>	H	Et	H	—	118	206 (dec.)	—	—
<i>o</i> -MeC <sub>6</sub> H <sub>4</sub>	H	Ph	H	—	135	—	130	202 (HBr)
<i>o</i> -MeC <sub>6</sub> H <sub>4</sub>	H	p-MeC <sub>6</sub> H <sub>4</sub>	H	—	143	—	128	188 (dec.) (HCl)
<i>p</i> -MeOC <sub>6</sub> H <sub>4</sub>	H	H	H	—	125	180	—	—
<i>p</i> -MeOC <sub>6</sub> H <sub>4</sub>	H	Me	H	—	118	—	135	203 (dec.) (HCl)
<i>p</i> -MeOC <sub>6</sub> H <sub>4</sub>	H	Et	H	—	144	—	122	109 (HCl)
<i>p</i> -MeOC <sub>6</sub> H <sub>4</sub>	H	Ph	H	—	188	—	176	201 (HCl)
<i>p</i> -MeOC <sub>6</sub> H <sub>4</sub>	H	Et	H	—	180	—	160	214 (HCl)
<i>p</i> -MeOC <sub>6</sub> H <sub>4</sub>	H	p-MeC <sub>6</sub> H <sub>4</sub>	H	—	219	—	158	248 (dec.) (HBr)
<i>p</i> -MeOC <sub>6</sub> H <sub>4</sub>	H	p-ClC <sub>6</sub> H <sub>4</sub>	H	—	80	195 (dec.)	—	—
<i>o</i> -MeOC <sub>6</sub> H <sub>4</sub>	H	Me	H	—	99 (dec.)	—	101	208 (dec.) (HBr)
<i>o</i> -MeOC <sub>6</sub> H <sub>4</sub>	H	Et	H	—	54	—	104	—
<i>o</i> -MeOC <sub>6</sub> H <sub>4</sub>	H	Ph	H	—	74	—	147	208 (HBr)
<i>o</i> -MeOC <sub>6</sub> H <sub>4</sub>	H	p-MeC <sub>6</sub> H <sub>4</sub>	H	—	159	—	151	202 (HCl)
<i>p</i> -ClC <sub>6</sub> H <sub>4</sub>	H	p-ClC <sub>6</sub> H <sub>4</sub>	H	—	175	—	178	236-7 (dec.) (HBr)

smoothly synthesized (101, 25) according to Douglass (24) via the isoselenocyanates by the alkaline hydrolysis of the corresponding acyl-substituted selenoureas (Scheme 10). But in this way one cannot obtain selenourea itself, because the 1-benzoyl-2-selenourea is completely destroyed by the alkaline or acidic hydrolysis. In this case one may first condense the 1-benzoyl-2-selenourea with the  $\alpha$ -haloketone followed by hydrolysis of the resulting derivative of 2-benzamidoselenazole to the 2-amino selenazole (4). This method cannot be successful for 1-acyl-substituted 3-alkyl(aryl)-2-selenoureas, because derivatives of  $\Delta^4$ -selenazolines are formed instead of selenazoles. The ring closure occurs due to the reduced nucleophile character of the acylated amino group with the other nitrogen atom of the selenourea, by giving 2-acylimino-3-alkyl(aryl)- $\Delta^4$ -selenazolines. For instance the reaction of 1-benzoyl-3-*p*-nitrophenyl-2-selenourea with phenyl bromide forms the 2-benzoylimino-3-*p*-nitrophenyl-4-phenyl- $\Delta^4$ -selenazoline (Scheme 11). It



Scheme 10

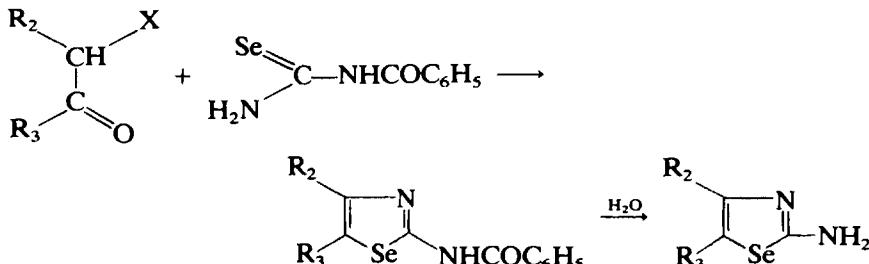


Scheme 11

gives on acidic hydrolysis the 2-imino-3-*p*-nitrophenyl-4-phenyl- $\Delta$ 4-selenazole (102). This is not identical with the 2-*p*-nitranilino-4-phenylselenazole (103) obtained from the 1-*p*-nitrophenyl-2-selenourea and phenacyl bromide.

### a. HYDROLYSIS OF 2-BENZAMIDOSELENAZOLES (23)

The hydrolysis of 2-benzamido selenazoles in aqueous phosphoric or sulfuric acid gives 2-amino selenazoles identical to those prepared from selenourea (Scheme 12).

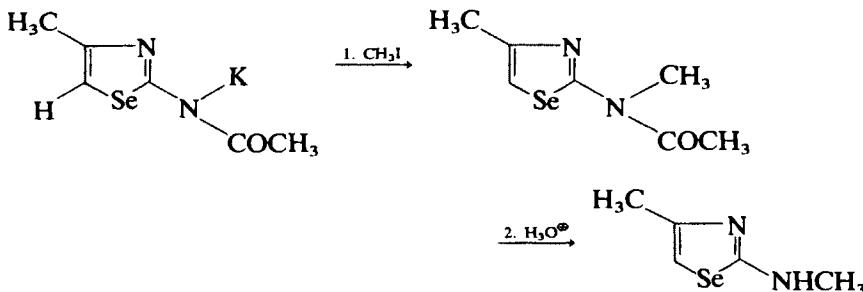


Scheme 12

The advantage of this method is that it avoids the use of hydrogen selenide, necessary for the preparation of selenourea from cyanamide (14). Benzoylselenourea is synthesized by the method of Douglass (24) by the action of potassium selenocyanate on benzoyl chloride in acetone solution.

### b. CONDENSATION OF $\alpha$ -HALOKETONES

The condensation of  $\alpha$ -haloketones with monosubstituted alkyl or aryl selenoureas (25) leads to 2-alkylamino- (26, 27) or 2-arylaminoselenazoles (28) while disubstituted selenoureas give 2-(dialkylamino)selenazoles (26, 27) (Table X-3a).

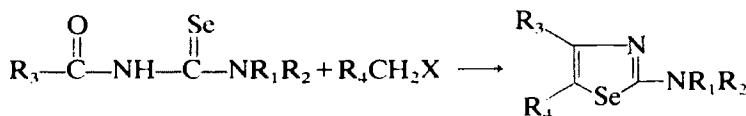


Scheme 13

2-(Methylamino)-4-methylselenazole was obtained by methylation of the potassium salt of 2-acetamido-4-methylselenazole with methyl iodide, followed by hydrolysis (Scheme 13) (26).

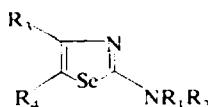
### C. Synthesis from N-acyl Selenoureas (104)

The general method is described in Scheme 14. The compounds so prepared are listed in Table X-3b.



Scheme 14

TABLE X-3b. 2-(ALKYLAMINO) OR 2-(ARYLAMINO)SELENAZOLES (104)



$\text{R}_1$	$\text{R}_2$	$\text{R}_3$	$\text{R}_4$ or Ar	m.p. (°C)	Yield (%)	Ultraviolet $\lambda_{\text{max}}$ (nm)	(log ε)
$\text{C}_6\text{H}_5$	$\text{CH}_3$	$\text{C}_6\text{H}_5$	$\text{C}_6\text{H}_5\text{CO}$	138–40 (ethanol)	82	231 s (4.44)	258 s (4.32) (4.08)
$\text{C}_6\text{H}_5$	$\text{CH}_3$	$\text{C}_6\text{H}_5$	$p\text{-BrC}_6\text{H}_4\text{CO}$	147–148 (ethanol)	86	237 (4.29)	271 (4.24) (4.11)
$\text{C}_6\text{H}_5$	$\text{CH}_3$	$\text{C}_6\text{H}_5$	$p\text{-ClC}_6\text{H}_4\text{CO}$	139–141 (ethanol)	91	237 (4.29)	256 (4.26) (4.08)

### D. General Properties

2-Aminoselenazoles are crystalline compounds, and with the exception of the nonsubstituted derivative (1) they are stable and practically odorless. They are more basic than 2-aryl- or 2-alkylselenazoles. The hydrochlorides are not easily hydrolyzed in aqueous solution. Some of the N-substituted compounds are oily liquids.

2-Amino- and 2-(methylamino)-4-methylselenazole can be considered

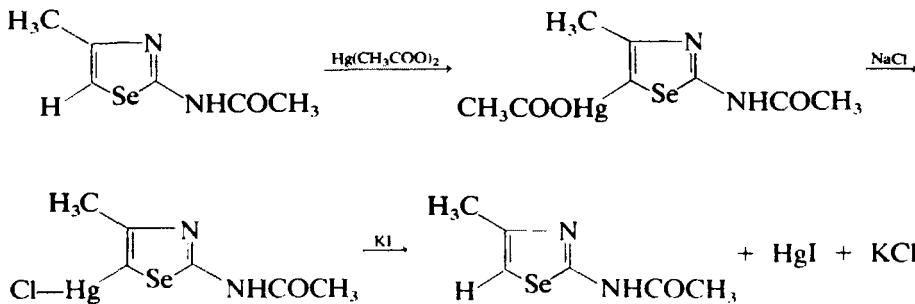
to be in prototropic equilibrium with the 2-iminoselenazolines, as indicated by a comparison of their ultraviolet spectra with that of 2-(diethylamino)-4-methylselenazole (26). In the 2-position there is a substituent with a labile hydrogen (Scheme 15); tautomerism is possible, and this leads to a reduction of the aromatic character of the system. This particularly affects the stability of selenazole and encourages ring opening. These theoretical considerations are in very good agreement with experimental observations (19, 26).



Scheme 15

The 5-position of 2-diethylamino-4-methylselenazole is very reactive. Thus it can undergo coupling with phenyldiazonium chloride or even nitration under mild conditions.

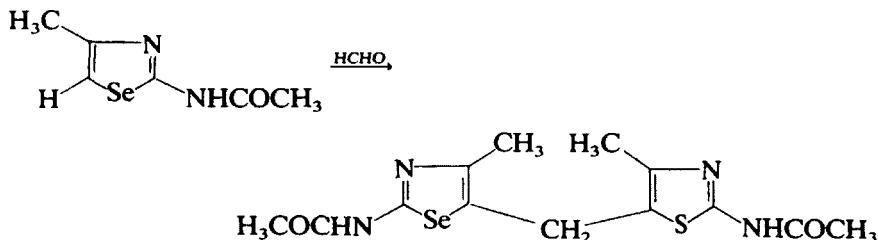
2-Acetamido-4-methylselenazole can react with mercuric acetate to yield 5-mercuriacetate derivatives that can be converted to the chloro derivatives by the action of sodium chloride. Treatment with potassium iodide leads to reduction regenerating the initial compound with loss of mercury (Scheme 16) (4).



Scheme 16

The action of formaldehyde on 2-acetamido-4-methylselenazole can lead to dimerization in the 5-position (Scheme 17) (4).

2-Amino-4-aryl-5-acetic acid selenazoles were used by Knott (21) as intermediates in the preparation of 1',2',4,5-naphthoselenazoles (21, 30, 31).

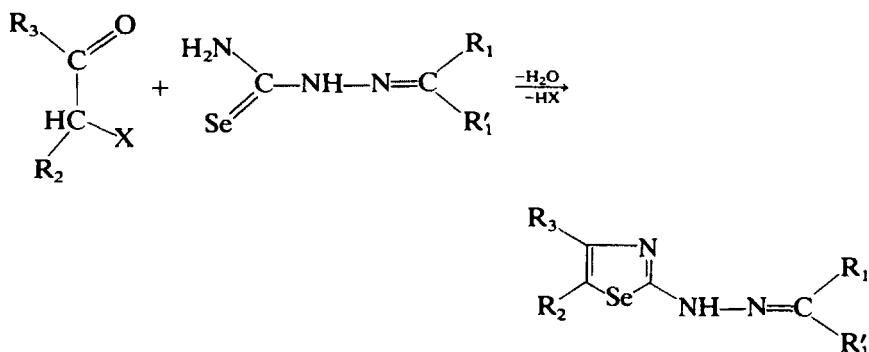


Scheme 17

From the point of view of reactivity, there is little difference between 2-amino-selenazoles and aryl- or alkyl-2-aminoselenazoles, except that the *N*-aryl derivatives are generally less basic and that their salts are more easily hydrolyzed.

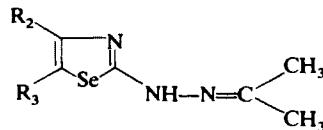
### 3. 2-Hydrazinoselenazoles and 2-Isopropylidenehydrazino or 2-Benzylidenehydrazinoselenazoles

If in the Hantzsch's synthesis, selenoamide is replaced by a selenosemicarbazide ( $\text{H}_2\text{N}-\text{NH}-\overset{\text{C}}{\underset{3}{\text{Se}}}-\text{NH}_2$ ), condensation can take place upon any of the three nitrogen atoms of the assymmetric molecule. To obtain a hydrazine-type structure, the nitrogen in the 1-position is blocked by forming a hydrazone. Thus selenosemicarbazones were obtained with acetone ( $\text{R}_1 = \text{R}_2 = \text{Me}$ ) and with benzaldehyde ( $\text{R}_1 = \text{Ph}$ ,  $\text{R}_2 = \text{H}$ ). Reaction of these selenosemicarbazones with  $\alpha$ -halocarbonyls leads to 2-isopropylidenehydrazino or 2-benzylidenehydrazinoselenazoles (32, 33) (Scheme 18), (Tables X-4 and X-5).



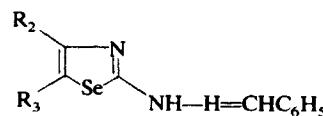
Scheme 18

TABLE X-4. 2-ISOPROPYLIDENE HYDRAZINOSELENAZOLES (32, 33)



R <sub>2</sub>	R <sub>3</sub>	m.p. (°C)	m.p. (HCl or HBr derivative) (°C)
H	Me	156-7 (dec.)	156-7 (dec.) (HBr)
Me	H	101	171-2 (dec.) (HCl)
Me	Me	138 (dec.)	173-5 (dec.) (HCl)
Ph	H	108-9	187 (dec.) (HCl)
p-MeC <sub>6</sub> H <sub>4</sub>	H	129	220 (dec.) (HCl)
p-MeOC <sub>6</sub> H <sub>4</sub>	H	125	207 (HCl)
p-ClC <sub>6</sub> H <sub>4</sub>	H	153	223 (HBr)
p-BrC <sub>6</sub> H <sub>4</sub>	H	152	216 (dec.) (HCl) 227 (dec.) (HBr)
Ph	Ph	172 (dec.)	—
Me	COOEt	154-5 (dec.)	169-170 (dec.) (HCl)
Ph	COOEt	150	194 (HCl)

TABLE X-5. 2-BENZYLIDENE HYDRAZINOSELENAZOLES (32, 33)



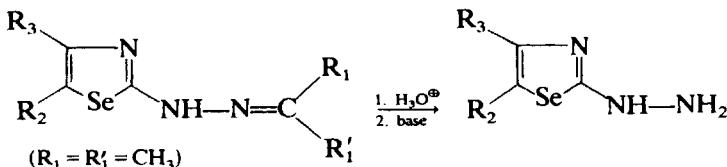
R <sub>2</sub>	R <sub>3</sub>	m.p. (°C)	m.p. (HCl or HBr derivative) (°C)	m.p. (acetyl derivative) (°C)
Me	H	176	168 (HCl)	—
H	Me	209-210 (dec.)	197 (HBr)	125
Me	Me	192 (dec.)	202 (dec.) (HCl)	112
Me	COOEt	180 (dec.)	204 (dec.) (HCl)	114-5
Ph <sup>a</sup>	H	188 (dec.)	214 (dec.) (HCl)	169-70
Ph	Ph	230 (dec.)	—	150-1
Ph	COOEt	213 (dec.)	—	148
p-MeC <sub>6</sub> H <sub>4</sub>	H	181	206 (dec.) (HBr)	189
p-MeOC <sub>6</sub> H <sub>4</sub>	H	196 (dec.)	218 (dec.) (HCl)	156
p-ClC <sub>6</sub> H <sub>4</sub>	H	215	240 (dec.) (HBr)	—
p-BrC <sub>6</sub> H <sub>4</sub>	H	220 (dec.)	216 (dec.) (HBr)	194

<sup>a</sup> Homolog compound from acetophenone has m.p. = 171°C (33).

Except in the case of the 4,5-diphenyl derivatives, these compounds are obtained as their hydrohalide salts with yields of 70 to 90%: the free heterocyclic base is obtained by treatment with weak alkali.

The salts of derivatives possessing phenyl substituents on the selenazole ring are easily hydrolyzed in aqueous solution.

2-Benzylidenehydrazinoselenazoles are stable to acids and do not decompose with time. The isopropylidene homologs are only stable in the form of the hydrochloride, and they can undergo acid hydrolysis, thus providing a convenient pathway to the free hydrazine (32). Hydrolysis is carried out with hot 2 N hydrochloric acid, which, after recooling and filtration, leads to 2-hydrazinoselenazole hydrochloride, yielding the free base upon neutralization (Scheme 19, Table X-6).



Scheme 19

TABLE X-6. 2-HYDRAZINOSELENAZOLES (32)

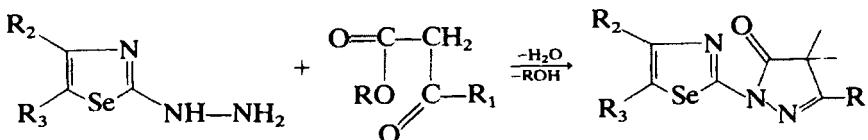
R <sub>2</sub>	R <sub>3</sub>	m.p. (°C)	m.p. (HCl derivative) (°C)
H	Me	100-2	167-8 (dec.)
Me	Me	115	161-2 (dec.)
Me	COOEt	187 (dec.)	198-210 (dec.)
Ph	H	144-5	166-7 (dec.)
Ph	Ph	199 (dec.)	—
Ph	COOEt	189 (dec.)	179
p-MeC <sub>6</sub> H <sub>4</sub>	H	166	168
p-MeOC <sub>6</sub> H <sub>4</sub>	H	156	170 (dec.)
p-BrC <sub>6</sub> H <sub>4</sub>	H	180	187 (dec.)

2-Hydrazinoselenazoles are solids, often in the form of crystals. They possess the characteristic properties common to hydrazines, especially as

reducing agents. Thus they reduce hot Fehling's solution and give, even in the cold, metallic silver from an ammoniacal solution of silver nitrate.

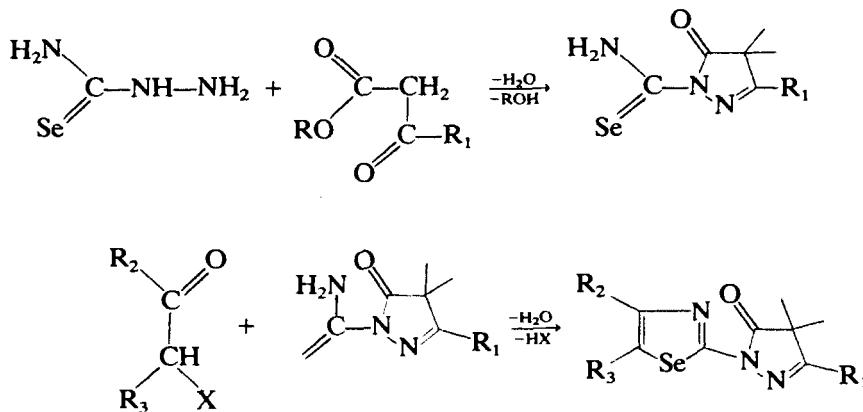
Furthermore, they react with carbonyl derivatives such as acetone and benzaldehyde to give selenohydrazone identical to those prepared from the condensation of selenosemicarbazones with an  $\alpha$ -halocarbonyl compound.

Condensation of 2-hydrazinoselenazoles with  $\beta$ -keto esters ( $R_1-CO-CH_2-COOR$ ) yields 1-(selenazol-2-yl)-3-alkylpyrazol-5-ones (Scheme 20) (34).



Scheme 20

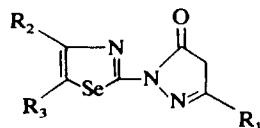
These latter compounds can also be obtained by direct cyclization in a Hantzsch's reaction of the selenosemicarbazone of a  $\beta$ -ketoester, that is, a 1-selenocarbamoyl-3-alkyl-5-pyrazolone (Scheme 21).



Scheme 21

These selenopyrazolones (Table X-7) can undergo further reaction, as described in Section III. 4 (34).

TABLE X-7. 1-SELENAZOL-2-YL-3-ALKYLPYRAZOL-5-ONES (34)

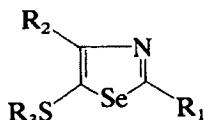


R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	m.p. (°C)	Yield (%)
Me	H	Me	201	58
Me	Me	Me	206-8	62
Me	Me	COOEt	208-9	50
Me	Ph	H	212	76
Me	Ph	Ph	180-1	84
Me	Ph	COOEt	220	41
Me	p-BrC <sub>6</sub> H <sub>4</sub>	H	226	73
Me	p-MeC <sub>6</sub> H <sub>4</sub>	H	210	86
Me	p-MeOC <sub>6</sub> H <sub>4</sub>	H	203	75
C <sub>17</sub> H <sub>35</sub>	Me	COOEt	94-5	44
C <sub>17</sub> H <sub>35</sub>	Ph	H	90-1	42
C <sub>17</sub> H <sub>35</sub>	Ph	Ph	73	56
C <sub>17</sub> H <sub>35</sub>	Ph	COOEt	118-9	8

#### 4. Other Functional Derivatives of Selenazole

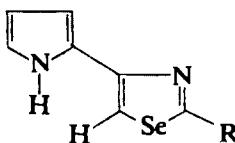
##### A. Thioethers

The preparation of substituted selenazole thioethers (Scheme 22) has already been described (35, 36). These compounds are obtained by the action of a haloketothioether on, for example, selenoacetamide, selenobenzamide, and *N*-ethylselenourea. These selenazoles have not been characterized, but they have been used as intermediates in the preparation of cyanine dyes.



Scheme 22

4-(2-Pyrrolyl)-selenazoles (Scheme 23), obtained by condensation of 2-chloroacetylpyrrole with selenoamides, are also used in dye chemistry (37).



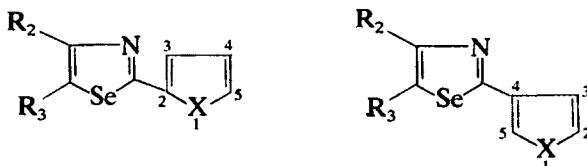
Scheme 23

### B. 2-Mercaptoselenazoles

2-Mercaptoselenazoles with C<sub>6</sub> to C<sub>10</sub> cyclic hydrocarbon substituents in the 4-position have been mentioned as giving negative images from photosoluble emulsions (38, 39). Equally cited are other heterocycles such as thiazole, oxazole, or imidazole.

### C. Synthesis of 2-(2- or 4-furyl, thienyl, or selenienyl)selenazoles

The general formula of the 2-(2- or 4-furyl, thienyl, or selenienyl)selenazoles is shown in Scheme 24 (40, 105, 106). (Selenienyl-2), (furyl-2), (thienyl-2), and (chloromethyl-4)selenazoles may be prepared by Hantzsch's reaction from selenoamides as described in Scheme 25. Hydrolysis give hydroxymethyl derivatives.



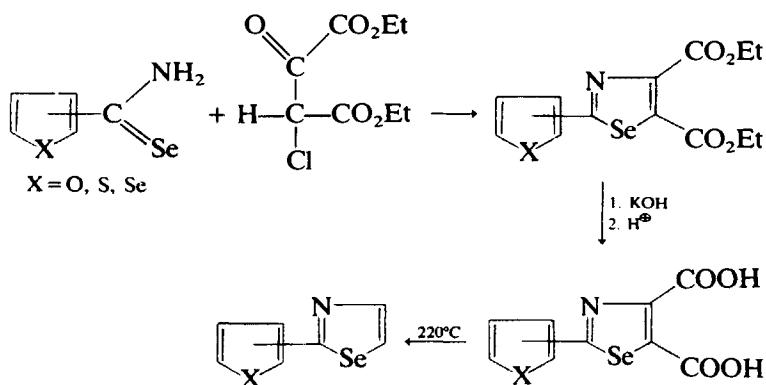
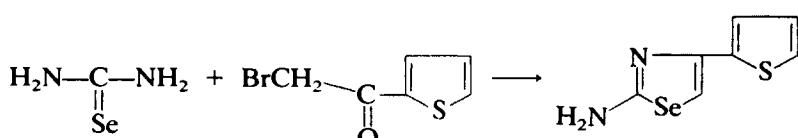
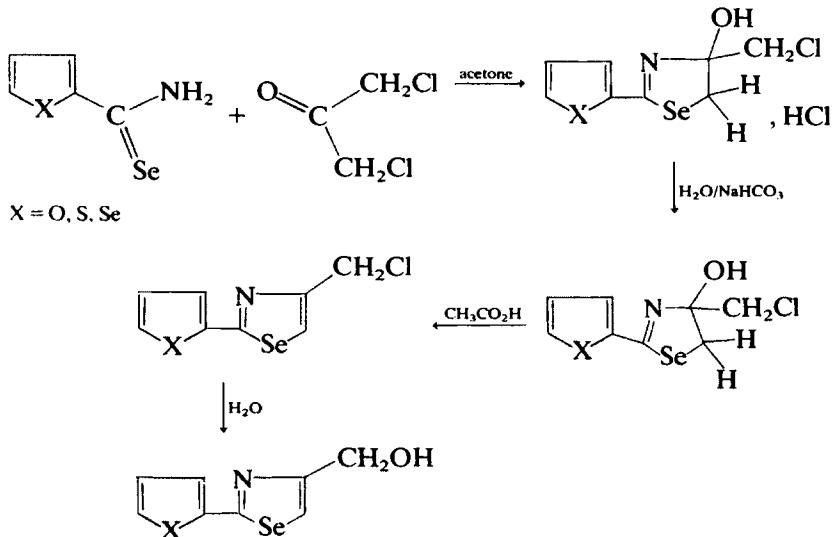
where R<sub>2</sub>, R<sub>3</sub> = H, CO<sub>2</sub>Et and X = O, S, Se

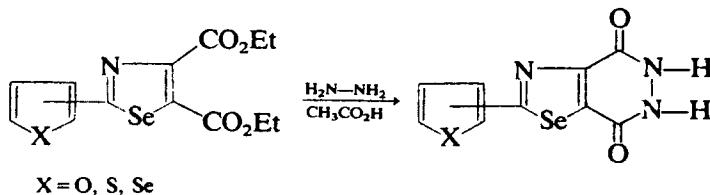
Scheme 24

2-Amino-4-(2-thienyl)selenazole (:n.p. 132°C, yield = 82%) also has been prepared by condensation of selenourea on 2 bromoacetylthiophene (Scheme 26). In a first step the bromhydrate is obtained (105).

(2- or 4-Furyl, thienyl, or selenienyl)selenoamides on ethyl- $\alpha$ -chloro- $\alpha$ -ethoxalyl acetate as shown in Scheme 27. The diesters may be decarboxylated by a saponification, an acid hydrolysis, and heating to 220°C successively (Scheme 27) (105).

The diesters may react with hydrazine in acidic medium to give 4,7-dioxo-4,5,6,7-tetrahydro[4,5,d]azolopyridazine (Scheme 28) (107). The



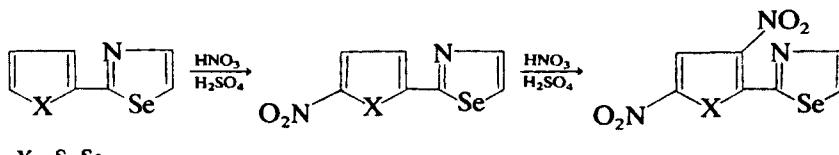


Scheme 28

study of tautomeric equilibrium of azolopyridazines by infrared spectroscopy is in complete agreement with the N-H structure.

The 2-[2-thienyl]selenazole is formylated in the 5-position by action of *n*-butyllithium, dimethyl formamide, and hydrolysis (106).

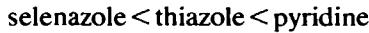
The nitration of 2-[2-thienyl] and [2-selenienyl] selenazoles is achieved according to Scheme 29 (106).



Scheme 29

### III. REACTIVITY OF THE SELENAZOLE RING AND OF DERIVED COMPOUNDS

A comparison of the reactivity of the heterocycles, selenazole, thiazole, and pyridine, was made by Ochiai (41), who used theoretical considerations to show that the degree of aromaticity was:

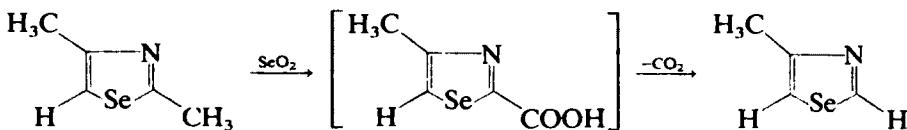


Furthermore, the strongly metallic character of selenium weakens the C-Se bond and thus favors reactions involving opening of the ring. The basicity of the three heterocycles is approximately in the same order, the nitrogen atom of selenazole and thiazole possessing much the same properties as the heteroatom of pyridine. Of the two carbon atoms ortho to nitrogen, that is, the 2-carbon and the 4-carbon, only the one in the 2-position is fairly active as a result of its interaction with selenium or sulfur. The 4- and 5-positions of thiazole and selenazole are more susceptible to electrophilic substitution than the 3- and 5-positions of pyridine. This is particularly true of the 5-position of selenazole. Thus it can be said that the 2- and 5-positions of the selenazoles and thiazoles

correspond to the para position of benzene, while the 2- and 4-positions correspond to the meta position. The 2-position of selenazole seems to be less reactive towards nucleophilic reagents than that of thiazole (19); however, being based on few experimental results, due to difficulties in preparing suitably substituted selenazoles, this hasty conclusion merits confirmation.

Thus Haginiwa (19) has studied the reactivity of the 2-position of selenazoles towards nucleophilic substitution, especially amination. The reaction of 4-methylselenazole with sodium amide in decalin at high temperature gave only rupture of the selenazole ring. It must be noted, however, that such experiments are limited because of the difficulties resulting from the preparation of 4-methylselenazole or other substrates unsubstituted in the 2-position. All attempts by Haginiwa (19) to diazotize 2-amino-4-methylselenazole led to ring destruction. Similar, later, investigations by Metzger and Bailly (2), who studied the deamination of 2-amino-4-phenylselenazole, were also unsuccessful.

The reaction of 2,4-dimethylselenazole with an excess of benzaldehyde gives a monostyryl derivative exclusively (12). Oxidation of 2,4-dimethylselenazole with selenium dioxide yields 4-methylselenazole (Scheme 30) (4).



This implies that only the 2-methyl group is sufficiently active to undergo oxidation to a carboxylic acid intermediate that then undergoes rapid decarboxylation.

The greater reactivity of the 5-position of selenazoles, compared to thiazoles, toward electrophilic substitution has also been demonstrated (19). Substituents in the 2-position possessing a mesomeric donor effect increase the reactivity, but, as Haginiwa (19) observed, also increase the tendency to ring opening.

Thus reactions of the selenazole ring are mainly limited to the 5-position, especially for 2-aminoselenazoles and 2-hydrazinoselenazoles that undergo electrophilic substitution.

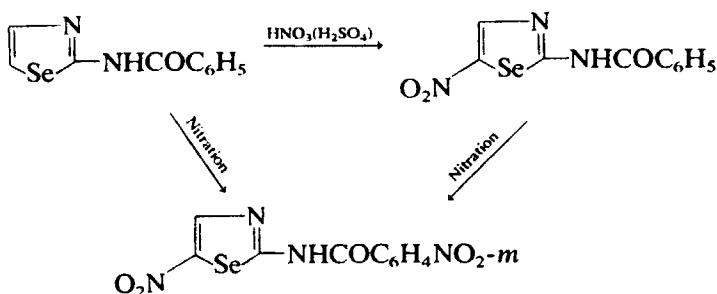
All attempts to prepare selenazole derivatives by the Gatterman (formylation) or Friedel-Crafts (alkylation) methods failed (19, 26), indicating that the electrophilic reactivity of the 5-position is less than that of benzene or toluene.

## 1. Electrophilic Substitution Reactions

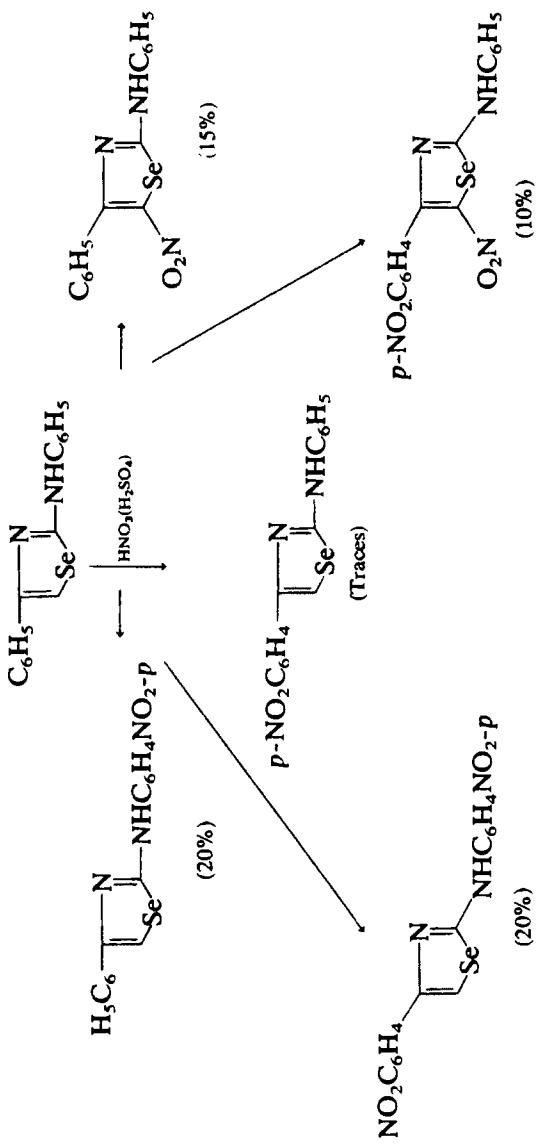
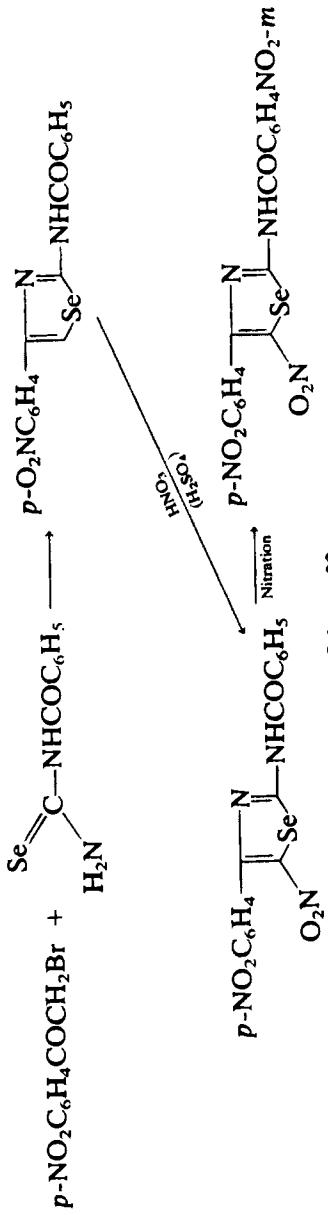
### A. Nitration

Nitration in the 5-position of 4-methyl- and 2,4-dimethylselenazoles with  $\text{HNO}_3\text{-H}_2\text{SO}_4$  is more rapid than for thiazoles [4-methyl-5-nitroselenazole, m.p. 45°C (19); 2,4-dimethyl-5-nitroselenazole, m.p. 115–120°C (decomp.) (19)]. Direct nitration of 2-amino 4-methyl-selenazole leads to ring rupture (19).

The 2-benzamido 4-aryl(alkyl)selenazoles (96) form the corresponding 5-nitro derivatives under mild conditions using the nitrate–sulfuric acid method (Scheme 31). The nitro compounds are well-defined, crystalline compounds. They may be most favorably obtained by dissolving the 2-benzamidoselenazoles in acetone and adding concentrated nitric



acid under cooling, whereupon the nitro derivatives are deposited. One can keep them for rather a long time without decomposition. Adding the nitro derivatives very carefully into the sulfuric acid is recommended, because otherwise secondary reactions occur with a violent release of gas. The use of cold mixed nitric and sulfuric acids also affects phenyl groups that may be present (97), leading to dinitro compounds (Scheme 31), or in the case of the 4-phenyl derivative even a trinitro compound. The structure as the 5-nitro derivative was established partly on the basis of the disappearance of the band between 3100 and 3175  $\text{cm}^{-1}$  in the infrared spectrum on substitution. It might be assigned to the =C–H stretching vibration in the 5-position in analogy to that in the 2-aminothiazoles. The second part of the structure proof is that compounds of the type in Scheme 31 only form benzoic acid on oxidative degradation. The entry of the second nitro group into the meta position of the



benzoyl group, forming compounds of type in Scheme 32, results from the *m*-nitrobenzoic acid obtained by the oxidative degradation. The third nitro group introduced in the dinitro derivative is in the para position of the phenyl group in the 4-position of the selenazole ring. This could be established by an independent synthesis starting with the *p*-nitrophenacyl bromide and the subsequent stepwise nitration of the 2-benzamido-4-*p*-nitrophenylselenazole (Scheme 32). The compound obtained in this way is identical with the trinitro compound formed by direct nitration of the 2-benzamido-4-phenylselenazole.

The nitration of the 2-anilino-4-phenylselenazole (103) is much more complicated. Even careful nitration using the nitrate-sulfuric acid method leads to the formation of a mixture of variously nitrated compounds in an almost violent reaction. By the use of column chromatography as well as thin-layer chromatography a separation could be made, and the compounds could be partly identified by an independent synthesis. Scheme 33 shows a general view of the substances prepared. Ring fission was not observed under mild conditions.

However, prior protective acetylation of the amino group leads to a good yield of the 5-nitro compound [2-acetamido-4-methyl-5-nitroselenazole, m.p. 185°C (19)]. Similarly, 2-diethylamino-4-methylselenazole with nitric acid gives the 5-nitro derivative [yellow needles, m.p. 93°C (26)].

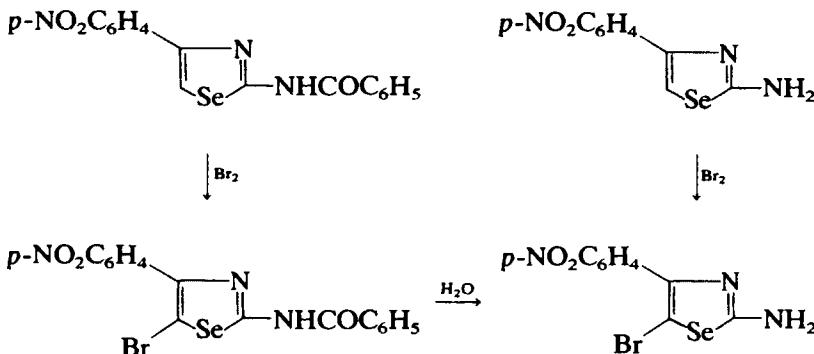
### B. *Sulfonation*

2-Alkyl selenazoles have been sulfonated. Thus 2,4-dimethylselenazole with fuming sulfuric acid (5% SO<sub>3</sub>) at 100°C gives 2,4-dimethyl-5-sulfonylselenazole [m.p. 238°C (decomp.) (26)], which is relatively unstable, decomposing to give metallic selenium.

### C. *Halogenation*

As a further electrophilic substitution the bromination of selenazoles has been investigated. This is not as complicated as nitration. Bromination was carried out in several solvents and with various amounts of bromine. In spite of the great variation in conditions, monobromo derivatives containing the bromine atom in the 5-position are always formed. This could be established, for example, by the bromination of the 2-amino-4-*p*-nitrophenylselenazole (Scheme 34) and its 2-benzamino compound (98). The 2-benzamido bromo compound gives the same bromo

derivative by hydrolysis as it does by the direct bromination in Scheme 34. This simultaneously shows halogens in the 5-position possessing a very low reactivity, because the hydrolysis is done without displacement of bromine. Further displacement reactions were also unsuccessful (Scheme 34).



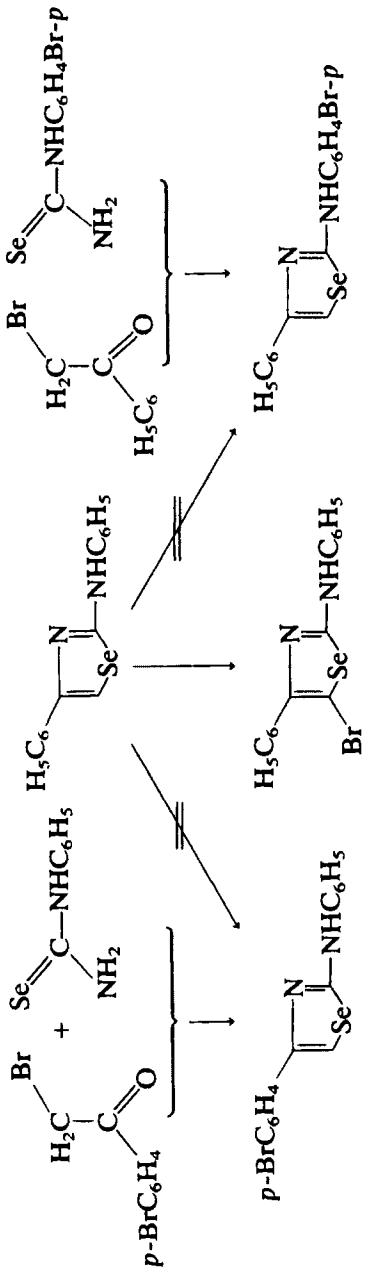
The 2-anilinoselenazoles follow an analogous reaction path. The entry of bromine into the 5-position is in agreement as seen in Scheme 35. The 2-anilino-4-phenylselenazole gives a 5-bromo derivative by bromination. It is not identical with either of the bromo derivatives prepared by direct synthesis (Scheme 35) (99).

Bromination of 2,4-dimethylselenazole with cold bromine gives an unstable monobrominated derivative initially (m.p. 168°C), which is easily converted to a product [m.p. 205°C (decomp.)] considered by Haginiwa to be 5-bromo-2,4-dimethylselenazole hydrobromide (19).

Unlike nitration, 2-amino-4-methylselenazole can be directly brominated, using bromine in carbon tetrachloride solution, to give 2-amino-5-bromo-4-methylselenazole hydrobromide [m.p. 180°C (decomp.)] (19). The free base cannot be isolated. Use of excess of bromine can lead to destruction of the molecule.

2-Acetamido-5-bromo-4-methylselenazole (m.p. 196°C) was obtained by the same method (19).

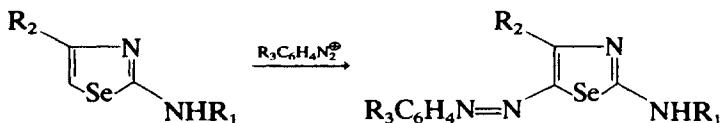
2-Amino-5-bromoselenazole has been used as an intermediate in the synthesis of pharmaceutical compounds (42).



Scheme 35

### D. Diazo Coupling

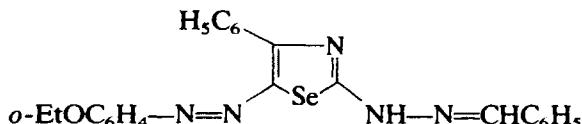
The high reactivity of the 5-position in 1,3-selenazoles toward electrophilic substitution was also observed on azocoupling. By reacting molar quantities of an aqueous solution of a diazonium salt with an ethanolic solution of a 2-arylamino selenazole, for example, the corresponding 2-arylamino-5 azoselenazoles are formed in a smooth reaction (100). They deposit from the deeply colored solution and form intensely red-colored compounds after their recrystallization from a suitable solvent (Scheme 36).



Scheme 36

Coupling of 2-diethylamino-4-methylselenazole with phenyldiazonium chloride gives the corresponding 5-phenylazo compound [orange granules, m.p. 101°C (26)].

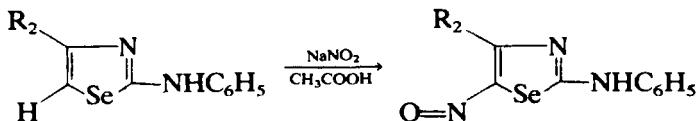
Bulka et al. (43) have demonstrated the electrophilic reactivity of selenazoles possessing an hydrazone in the 2-position and nonsubstituted in the 5-position toward diazonium salt to give 5-phenylazo derivatives preferentially. For example, the main product of the coupling of 2-benzylidene hydrazino-4-phenylselenazole with diazo-*o*-phenetidine is the 5-(*o*-ethoxyphenylazo)-selenazole (Scheme 37) (ruby red prisms, m.p. 206°C, yield 67%). A formazan is obtained as by-product. (See Section III.6) (43).



Scheme 37

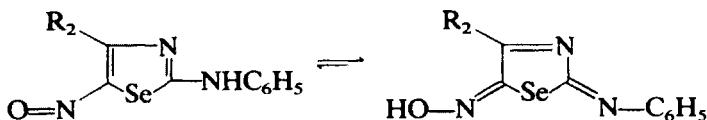
### E. Nitrosation

Treatment of 2-(arylamino)selenazoles with sodium nitrite in glacial acetic acid at room temperature gives the corresponding nitroso compounds instantaneously (Scheme 38) (29). These compounds give rise to

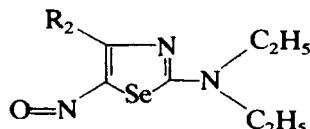


Scheme 38

the tautomeric equilibrium in Scheme 39. They possess properties notably different from analogous aromatic nitroso derivatives not having a mobile hydrogen on the exocyclic nitrogen, as, for example, 2-diethylaminoselenazoles that have the unambiguous 5-nitroso structure (Scheme 40), and properties similar to *p*-nitrosodialkylanilines.

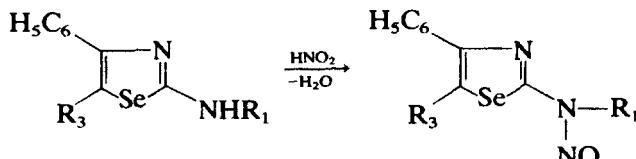


Scheme 39



Scheme 40

*N*-nitrosamino derivatives are only obtained when the 5-position is blocked, for example, with 2-arylmino-4,5-diphenylselenazole or 2-arylmino-5-phenylselenazole (Scheme 41). Different compounds have been prepared (Table X-8) (29).



Scheme 41

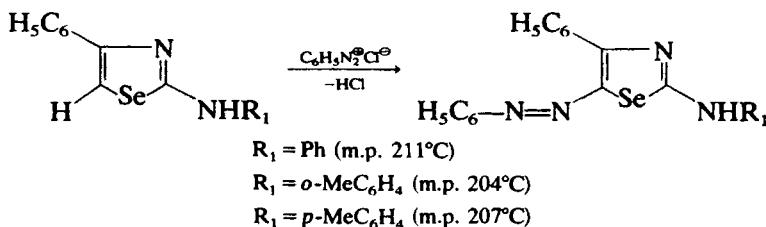
Diazotization of 2-arylmino-4-phenylselenazoles with phenyldiazonium chloride gives 2-arylmino-4-phenyl-5-phenylazoselenazoles (Scheme 42) (29).

TABLE X-8. N-NITROSAMINO  
5-PHENYL-SELENAZOLES (29)

$R_3$	$R_1$	m.p. (°C)
H	Ph	176
H	<i>o</i> -MeC <sub>6</sub> H <sub>4</sub>	183
H	<i>p</i> -MeC <sub>6</sub> H <sub>4</sub>	161
Ph	Ph	193

In conclusion, in terms of electrophilic reactivity a *methyl* group in the 2-position is equally reactive in the two categories of heterocycles (selenazole and thiazole). Of the two positions ortho to nitrogen, only the 2-position is activated. The 5-position is sensitive to electrophilic reagents and resembles more closely the para position of a benzene ring.

With respect to thiazole, the selenazole system displays a lesser nucleophilic reactivity in the 2-position and a greater electrophilic reactivity of the 5-position, but undergoes fission of the cycle more easily.



Scheme 42

## 2. Sulfanilamidoselenazoles

Much of the development of the chemistry of sulfanilamidoselenazole derivatives is a result of the important role played by sulfonamides in chemotherapy and more particularly the good activity of sulfathiazole against bacterial infections. Backer and De Jonge (44) prepared these derivatives by reaction of 2-amino-4-methyl- and 2-amino-4-phenyl-selenazoles with *N*-acetylsulfanilic acid chloride in pyridine. Alkaline

hydrolysis of the resulting *N*-acetyl sulfanilamides gave the corresponding 2-sulfanilamido selenazoles. The principal compounds thus prepared are shown in Table X-9 (16, 45, 46).

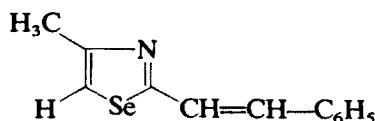
TABLE X-9. 2-SULFANILAMIDOSELENAZOLES

$R_2$	$R_3$	m.p. (°C)	m.p. (acetyl derivative) (°C)	Ref.
H	H	206	—	45
Me	H	222–3, 235	228–9, 225	16, 46
		236–7 (dec.)	—	44
Ph	H	231–2	238–9	16, 46
		209.5–210.5	244–5	44
Me	COOH	231–2	238–9	16, 46

The investigations of Jensen and Schmith (45) indicate that in vitro activity of 2-sulfanilamido and 4-methyl-2-sulfanilamido selenazoles against pneumonial infections is comparable to that of sulfathiazole or sulfadiazine. Frisk (47) found that the activity of the selenium compounds was much lower than that of sulfathiazole.

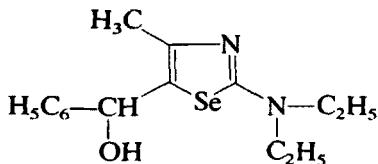
### 3. Condensation with Carbonyl Derivatives

The reactivity of 2-methylselenazole toward carbonyl compounds is the same as its thiazole homolog. Reaction of 2,4-dimethylselenazole with benzaldehyde in the presence of anhydrous zinc chloride as catalyst gives 4-methyl-2-styrylselenazole [m.p. 74–75°C (19)] (Scheme 43).



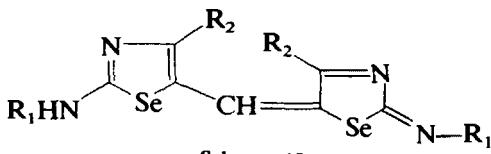
Scheme 43

2-Diethylamino-4-methylselenazole with benzaldehyde in the same conditions as for Scheme 43 yields 2-diethylamino-4-methyl-5-phenylcarbonyl selenazole (*m.p.* 254°C) (Scheme 44) (26).



Scheme 44

Using formaldehyde, compounds of type 5-[*(2-amino-5-selenazolyl)-methylene]*selenazoline are obtained (Scheme 45) (48).



Scheme 45

#### 4. Condensation of Selenated Alkylidene or Benzylidene Hydrazones or Pyrazolones with *p*-nitroso Dialkyanilines

##### A. Isopropylidene and Benzylidene Hydrazones

Isopropylidene and benzylidene hydrazones of selenazole unsubstituted in the 5-position react with *p*-nitrosodimethylanilines or *p*-nitrosodiethyl-anilines when heated in organic solvents in the presence of acetic acid or pyridine (49). Highly colored crystalline 2-hydrazone-5-(*p*-dialkylamino-phenylimino)selenazoles are recovered from the reaction medium (Table X-10).

Seemingly the presence of an aromatic substituent in the 4-position of the selenazole ring is necessary for obtaining well-crystallized products; compounds with an alkyl group in the 4-position are amorphous and quite difficult to obtain as crystals.

The mechanism of the reaction can be interpreted as involving a mesomeric effect of the 2-hydrazone group that leads to a negative charge on the 5-carbon (Scheme 46).

TABLE X-10. ALKYLIDENE OR BENZYLIDENE DERIVATIVES OF 5-(*p*-DIALKYLAMINOPHENYLIINO)SELENAZOL-2-ONE HYDRAZONES (49)<sup>a</sup>

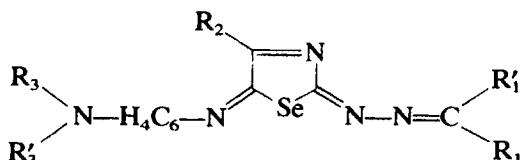
<i>R</i> <sub>2</sub>	<i>R</i> <sub>3</sub>	<i>R</i> <sub>3'</sub>	<i>R</i> <sub>1</sub> = <i>R</i> <sub>1'</sub> = CH <sub>3</sub>	<i>R</i> <sub>1</sub> = H, <i>R</i> <sub>1'</sub> = C <sub>6</sub> H <sub>5</sub>
			m.p. (isopropylidene derivative) (°C)	m.p. (benzylidene derivative) (°C)
Ph	Me	Me	154-5	176
<i>p</i> -MeC <sub>6</sub> H <sub>4</sub>	Me	Me	—	185-6
<i>p</i> -MeOC <sub>6</sub> H <sub>4</sub>	Me	Me	—	165
<i>p</i> -BrC <sub>6</sub> H <sub>4</sub>	Me	Me	—	207
Ph	Et	Et	125	166-7
<i>p</i> -MeC <sub>6</sub> H <sub>4</sub>	Et	Et	—	188
<i>p</i> -MeOC <sub>6</sub> H <sub>4</sub>	Et	Et	—	121
<i>p</i> -BrC <sub>6</sub> H <sub>4</sub>	Et	Et	—	163

<sup>a</sup>  $\lambda_{\text{max}}$  (EtOH) = 530 to 580 nm,  $\lambda_{\text{max}}$  (acetone) = 520 to 570 nm,  $\lambda_{\text{max}}$  (benzene) = 517 to 562 nm.

Electrophilic attack on the 5-position gives formation of an azomethinic bond and elimination of a molecule of water.

The dyes prepared in this way show a positive solvatochromism as the dielectric constant of the solvent increases, indicating that they possess a predominantly nonpolar structure. Substituents on the phenyl group in the 4-position of the selenazole ring have little influence on the absorption spectra.

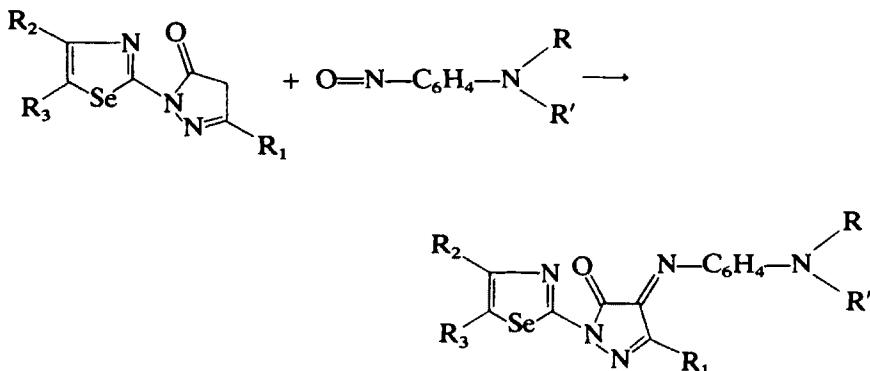
However, benzylidene derivatives show a strong bathochromic shift in comparison with alkylidene derivatives. Thus absorption is a result of the whole conjugated system that is comparable to that of the quinoid dyes. The color of this type of compound is sensitive to acids and bases.



Scheme 46

### B. 2-Pyrazoloneselenazoles

Reaction of 2-pyrazoloneselenazoles with *p*-nitrosodialkylanilines leads to a series of azomethinepyrazolones (Scheme 47) used as purple magenta dyes in color photography (34). The main regions of absorption are:  $\lambda_{\text{max}}$  (EtOH), 535 to 555 nm;  $\lambda_{\text{max}}$  (acetone 531 to 552 nm; and  $\lambda_{\text{max}}$  (benzene), 521 to 537 nm.



Scheme 47

### 5. Oxidation to Quinoid Dyes

Isopropylidene or benzylidene 2-hydrazinoselenazole derivatives can be converted to highly colored 2,2'-dioxo- $\Delta$ -3,3'-biselenazol-5,5'-inylidene-bis-hydrazone (Table X-11) by oxidation with ferric chloride and hydrogen peroxide (33).

Mechanistically, the reaction results from the mesomeric donor effect of the 2-hydrazino group. The coupling between the two molecules takes place in the 5-position (Scheme 48).

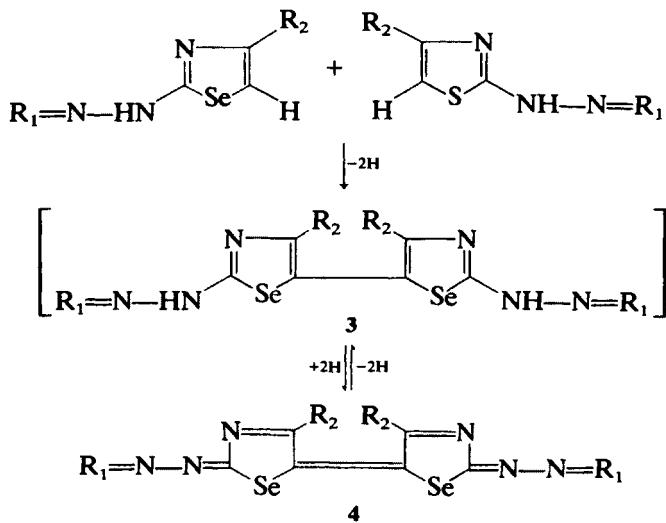
The primary dihydro product (**3**) can be obtained by reduction of the quinoid (**4**). Compound **3**, in which R<sub>1</sub> = C<sub>6</sub>H<sub>5</sub>-CH= and R<sub>2</sub> = C<sub>6</sub>H<sub>5</sub> can also be obtained by direct condensation of benzylidene selenosemicarbazone with 2,3-dibromo-1,4-diphenyl-1,4-butanedione (Scheme 49).

This reaction is proof of the structure of the dye, easily obtained from the dihydro compound (**3**). Yields are low as a result of numerous side reactions. The stereochemical configuration of these quinoid dyes is trans.

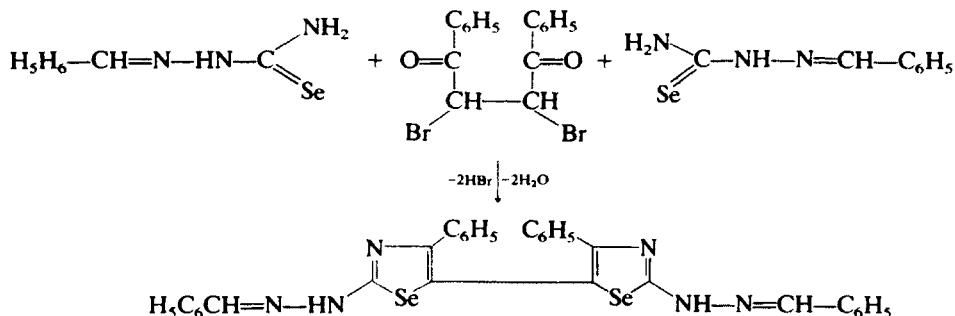
TABLE X-11. BIS-ISOPROPYLIDENE OR BENZYLIDENE DERIVATIVES OF 2,2'-DIOXO- $\Delta$ -3,3'-BISELENAZOL-5,5'-INYLIDENE BIS HYDRAZONES (33)

	$R_1 = C(CH_3)_2$ (Bisisopropylidene derivative)	$R_1 = C(H)_C_6H_5$ (Bisbenzylidene derivative)		
$R_2$	m.p. (°C)	$\lambda_{max}$ (nm) ( $CHCl_3$ )	m.p. (°C)	$\lambda_{max}$ (nm) ( $CHCl_3$ )
4,4'-diPh	246	571	263 <sup>a</sup>	578
4,4'-di-p-MeC <sub>6</sub> H <sub>4</sub>	—	—	292	578
4,4'-di-p-ClC <sub>6</sub> H <sub>4</sub>	251	575	270	590
4,4'-di-p-BrC <sub>6</sub> H <sub>4</sub>	252	580	291	593

<sup>a</sup> Bis- $\alpha$ -methylbenzylidene derivative: m.p. = 274°C,  $\lambda_{max}$  ( $CHCl_3$ ) = 590 nm.



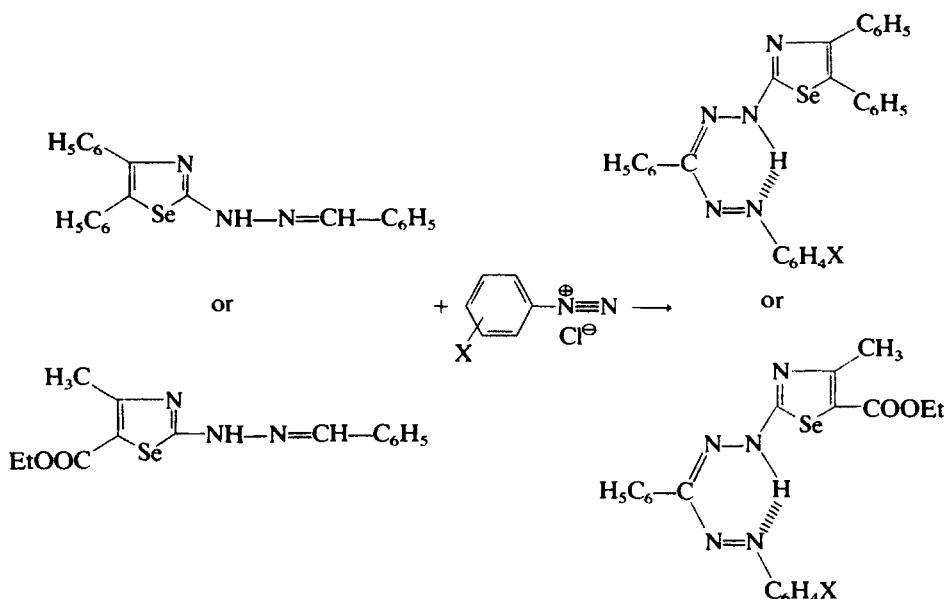
Scheme 48



Scheme 49

### 6. Selenazoleformazans

Formazans possessing a selenazole ring can be prepared from benzylidene hydrazones of selenazole. For example, 2-benzylidene hydrazino-4,5-diphenyl- and 2-benzylidenehydrazino-5-carbethoxy-4-methylselenazole with diazonium salts give, by nitrogen coupling [unambiguously since the 4- and 5-positions are blocked (Scheme 50)], formazans (43) (Table X-12). Nitrogen coupling occurs preferentially in the



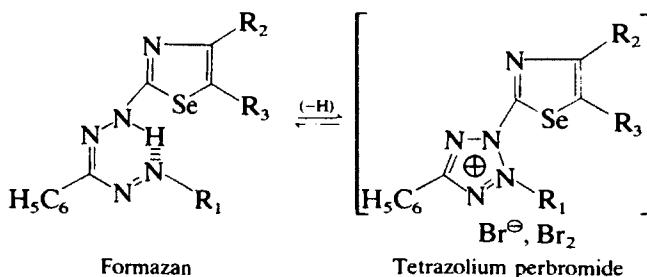
Scheme 50

TABLE X-12. FORMAZANS (43)

$R_1$	$R_2$	$R_3$	Yield (%)	m.p. (°C)
Ph	Ph	Ph	60	176
<i>p</i> -MeC <sub>6</sub> H <sub>4</sub>	Ph	Ph	70	175
<i>o</i> -MeC <sub>6</sub> H <sub>4</sub>	Ph	Ph	71	185
<i>p</i> -MeOC <sub>6</sub> H <sub>4</sub>	Ph	Ph	75	184
<i>o</i> -MeOC <sub>6</sub> H <sub>4</sub>	Ph	Ph	67	200
<i>p</i> -EtOC <sub>6</sub> H <sub>4</sub>	Ph	Ph	61	172
<i>o</i> -EtOC <sub>6</sub> H <sub>4</sub>	Ph	Ph	83	193
$\beta$ -C <sub>10</sub> H <sub>7</sub>	Ph	Ph	79	201
Ph	Me	COOEt	68	184
<i>p</i> -MeC <sub>6</sub> H <sub>4</sub>	Me	COOEt	75	175
<i>o</i> -MeC <sub>6</sub> H <sub>4</sub>	Me	COOEt	53	187
<i>p</i> -MeOC <sub>6</sub> H <sub>4</sub>	Me	COOEt	68	173
<i>o</i> -MeOC <sub>6</sub> H <sub>4</sub>	Me	COOEt	68	192
<i>o</i> -EtOC <sub>6</sub> H <sub>4</sub>	Me	COOEt	57	198
$\beta$ -C <sub>10</sub> H <sub>17</sub>	Me	COOEt	77	183

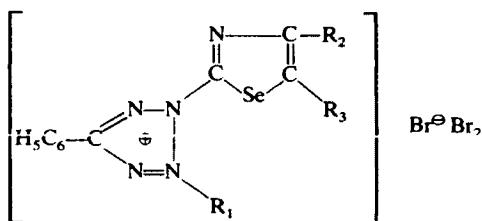
5-position when this latter is free, as this reaction has a higher rate constant.

The diazonium salts precursors can be aniline, *o*- and *p*-toluidine, *o*- and *p*-anisidine, *o*- and *p*-phenetidine, or  $\beta$ -naphthylamine. The resulting formazans are crystalline and intensely colored. They are soluble in organic solvents, giving a red-violet coloration that darkens to blue. Dehydrogenation gives the corresponding tetrazolium salts, which are isolated as perbromides (Scheme 51, Table X-13).



Scheme 51

TABLE X-13. TETRAZOLIUM PERBROMIDES (43)



R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	Yield (%)	m.p. (°C)
Ph	Ph	Ph	80	177
p-MeC <sub>6</sub> H <sub>4</sub>	Ph	Ph	64	158
<i>o</i> -MeC <sub>6</sub> H <sub>4</sub>	Ph	Ph	39	156
p-MeOC <sub>6</sub> H <sub>4</sub>	Ph	Ph	47	156
<i>o</i> -MeOC <sub>6</sub> H <sub>4</sub>	Ph	Ph	51	161
p-EtOC <sub>6</sub> H <sub>4</sub>	Ph	Ph	49	150
<i>o</i> -EtOC <sub>6</sub> H <sub>4</sub>	Ph	Ph	59	173
β-C <sub>10</sub> H <sub>7</sub>	Ph	Ph	20	156
Ph	Me	COOEt	90	129
p-MeC <sub>6</sub> H <sub>4</sub>	Me	COOEt	31	121
<i>o</i> -MeC <sub>6</sub> H <sub>4</sub>	Me	COOEt	57	128
p-MeOC <sub>6</sub> H <sub>4</sub>	Me	COOEt	49	108
<i>o</i> -MeOC <sub>6</sub> H <sub>4</sub>	Me	COOEt	69	121
<i>o</i> -EtOC <sub>6</sub> H <sub>4</sub>	Me	COOEt	55	120
β-C <sub>10</sub> H <sub>7</sub>	Me	COOEt	51	108

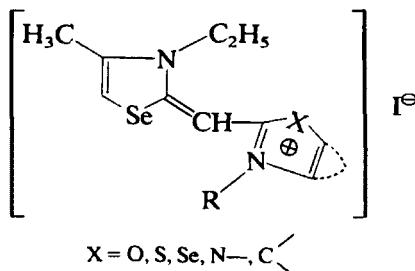
## 7. Quaternary Salts

Quaternary salts are obtained by alkylation of selenazole bases, the heterocyclic nitrogen atom playing the role of nucleophile with regard to the electrophilic carbon of the alkylating agent.

They are used principally as intermediates for cyanine dyes that serve in photography (5, 6, 35, 36, 50–52). 3-Ethyl-2,4-dimethylselenazole iodide (m.p. 157–8°C) was prepared as colorless crystals in 87% yield by Brooker et al. (6) by heating 2,4-dimethylselenazole with an excess of ethyl iodide for 48 hr.

By reaction of a corresponding quaternary salt in the presence of a condensation agent such as amyl nitrite or with a heterocycloammonium salt possessing a labile group, such as Cl, I, or SR (6, 11) in the 2-position, cyanine dyes are obtained (Scheme 52).

The heterocycloammonium salts can be made of 5- or 6-membered rings or even be (5+6) or (6+6) condensed systems. Among the cyanines

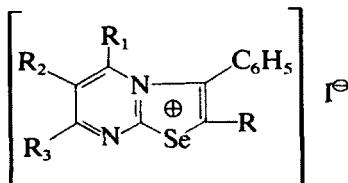


Scheme 52

prepared are: 1',3-diethyl-4-methylselenazolo-2'-cyanine iodide, m.p. 256 to 260°C (dec.); 1',3-diethyl-4-methylselenazolo-2'-pyridocyanine iodide, m.p. 232 to 233°C (dec.); and 1',3-diethyl-4-methyl-5',6'-benzo-selenazolo-2'-cyanine iodide, m.p. 275 to 277°C (dec.). 2-Methyl-4-phenylselenazole (51) can also be used after quaternization to give such dyes.

Carbocyanines with three methine groups can be prepared using two equivalents of selenazolium quaternary salt and one equivalent of ethyl orthoformate in pyridine solution (53).

Selenazolo[3,2-*a*]pyrimidinium salts (Scheme 53) can be obtained by condensation of 2-amino-4-phenylselenazoles with an excess of  $\beta$ -diketone of the type,  $R_1COCHR_2COR_3$ .

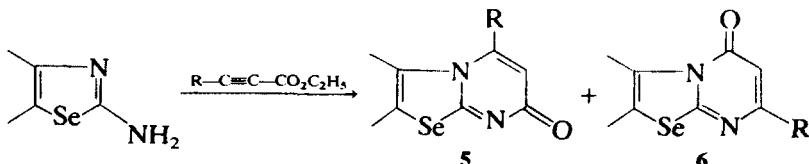


Scheme 53

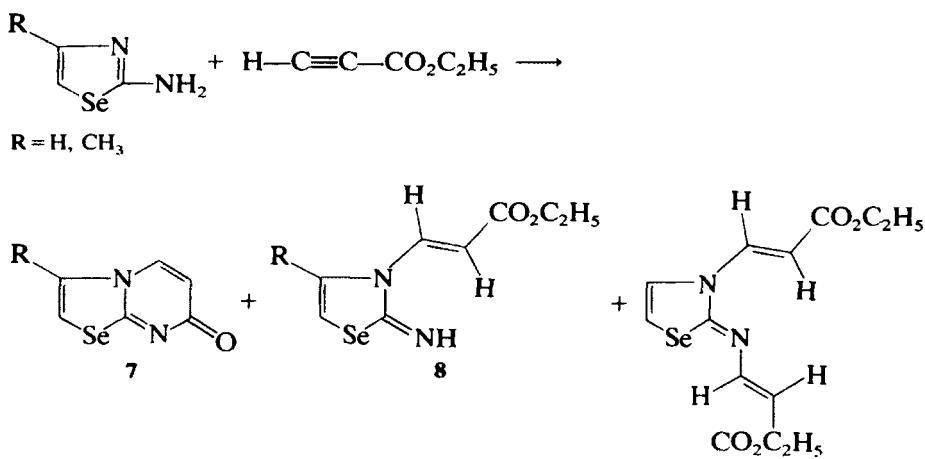
### 8. Reactivity of 2-Amino Selenazoles with Ethyl Propiolate (54) and Dimethyl Acetylene Dicarboxylate

In the reaction of 4-substituted 2-aminoselenazoles with ethyl propiolate and dimethylacetylene dicarboxylate, the major products obtained from such a condensation are substituted 7*H*-selenazolo[3,2-*a*]pyrimidin-7-ones (**5**) and not the alternative isomeric substituted 5*H*-selenazoles[3,2-*a*]pyrimidin-5-ones (**6**). Distinction between the alternative structures was based on infrared, ultraviolet, and NMR data (Scheme

54). In the reaction of 2-aminoselenazole with ethyl propiolate, in addition to compound type **7** (50% yield), compounds **8** and **9** were also isolated. Compound **7** is probably formed through the cis isomer of **8** (Scheme 55).



Scheme 54



Scheme 55

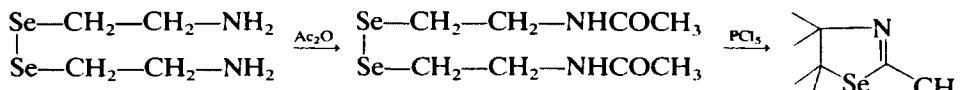
#### IV. OTHER HETEROCYCLES DIRECTLY DERIVED FROM SELENAZOLE

##### 1. Selenazolines

###### A. Alkyl or Aryl- $\Delta_2$ -Selenazolines

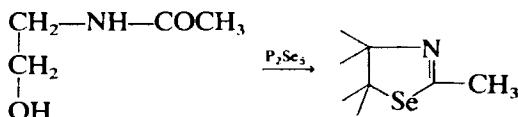
The first compounds with a  $\Delta_2$  selenazoline structure were mentioned in the literature in 1892 by Michels (55). He prepared 2-methyl- $\Delta_2$ -selenazoline in the course of his investigations into sulfur and selenium

derivatives of ethylamine. The preparative method used was the reaction of acetic anhydride on bis(2-aminoethyl)diselenide to give a diacetyl derivative that cyclizes under the action of phosphorus pentachloride (Scheme 56). 2-Methyl- $\Delta_2$ -selenazoline has also been prepared by the action of 2-bromoethylamine hydrobromide on selenoacetamide (56).



Scheme 56

Another method, due to Van Dormael (57) involves heating acetamidoethanol with phosphorus pentaselenide (Scheme 57).

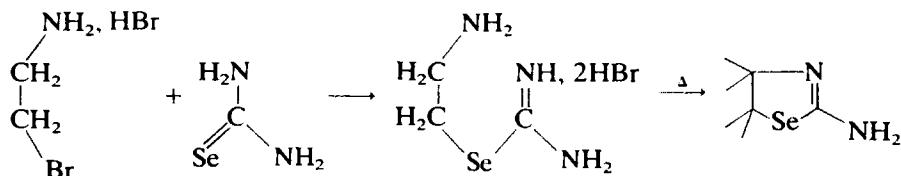


Scheme 57

Other 2-alkyl or 2-aryl- $\Delta_2$ -selenazolines have been prepared in the same way starting from suitably substituted *N*-acylethanolamines (58, 59).

### B. 2-Amino- $\Delta_2$ -Selenazolines

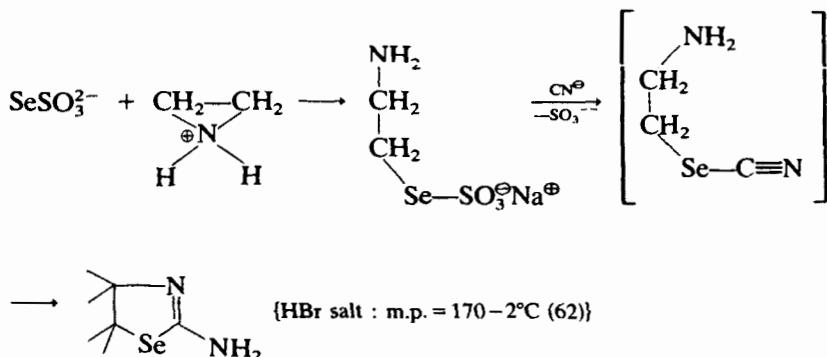
Starting from 2-bromoethylamine hydrobromide and selenourea, Chu and Mautner (60) prepared the salt of 2-(aminoethyl)-selenopseudourea, which cyclizes upon heating in aqueous solution to give 2-amino- $\Delta_2$ -selenazoline (Scheme 58).



Scheme 58

2-Amino- $\Delta_2$ -selenazoline and its 5-methyl derivative have previously been obtained by Baringer (61) starting from 2-bromoethylamine hydrobromide and 1-amino-2-bromopropane hydrobromide, respectively, by reaction with potassium selenocyanate.

Finally, Klayman (62) also prepared 2-amino selenazoline by reaction of a cyanide with the sodium salt of 2-aminoethane selenosulfuric acid (Scheme 59).

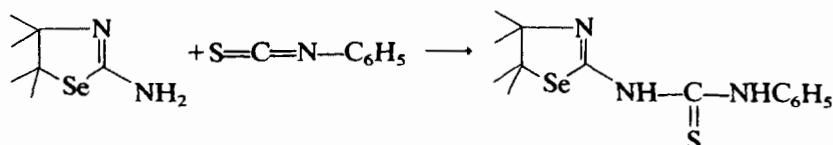


Scheme 59

Treatment of 2-amino selenazoline with aqueous alkaline base (63) or with hydrogen sulfide (64) leads to ring rupture and gives 2-ureidoethane-selenol and 2-thioureidoethane-selenol, which are not isolated but give by oxidation 1,1'-(diselenodiethylene)-bis(2-thiourea) :  $(H_2N-C\overset{||}{=}\text{NH}-CH_2-\text{Se}-)_2$ .

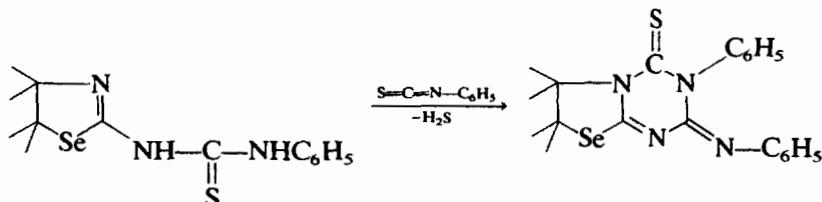
A similar reaction occurs with 2-aminothiazoline (63).

Addition of phenyl isothiocyanate to 2-amino selenazoline leads to 1-phenyl-3-(2-selenazolin-2-yl)-2-thiourea (Scheme 60).



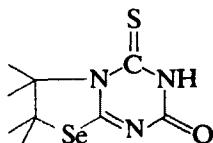
Scheme 60

Heating of this latter compound with an excess of phenyl isothiocyanate in acetonitrile (65) leads to 2-phenylimino-3-phenyl-4-thioxo-selenazole[3,2-*a*]tetrahydro-*s*-triazin (Scheme 61).



Scheme 61

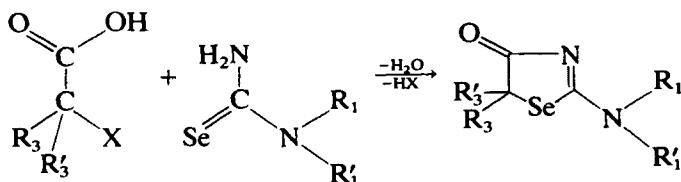
Similarly, the reaction of carbethoxyisothiocyanate ( $\text{EtO}_2\text{CNCS}$ ) with 2-amino selenazole leads to 2,3,6,7-tetrahydro-4*H*-selenazolo[3,2-*a*]-*s*-triazin-2-one-4-thione Scheme 62 (66).



Scheme 62

### C. 4-Oxo-Disubstituted 2-Amino- $\Delta_2$ -Selenazolines

4-Oxoselenazolines with a 2-(disubstituted amino) group are obtained by the action of  $\alpha$ -halocarboxylic acid or rather their esters on *N*-disubstituted selenoureas (Scheme 63, Table X-14) (27, 67, 68).



Scheme 63

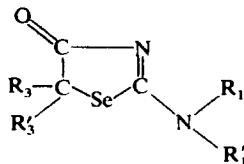
Compounds of this type possess a definite  $\Delta_2$ -selenazoline structure, while homologous compounds with at least one labile hydrogen on the 2-amino group can exist as a tautomeric equilibrium (Scheme 64).

Such is the case of 2-amino or 2-(monoalkyl or arylamino)-4-oxo-selenazolines. Thus because of this  $\text{amino} \rightleftharpoons \text{imino}$  tautomerism, these compounds can be called 2-amino selenazolines or 2-imino selenazolidines and can be classed either as selenazolines or selenazolidines. This has been a source of controversy that has been resolved by recent spectroscopic studies (67, 68).

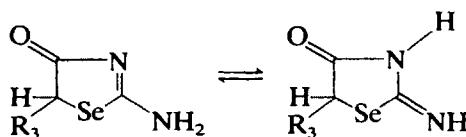
Comrie et al. (68) have thus prepared several compounds of this type by condensation of various selenoureas with  $\alpha$ -halocarboxylic acids and have assigned them the "imino" structure (Table X-15a).

Compounds of this type were first obtained from chloroacetic acid and selenourea (1) by Hofmann in 1889, who also assigned them the "imino"

TABLE X-14. 4-OXO-DISUBSTITUTED 2-AMINOSELENAZOLINES



$R_1$	$R'_1$	$R_3$	$R'_3$	m.p. ( $^{\circ}\text{C}$ )	m.p. (HCl or HBr derivative) ( $^{\circ}\text{C}$ )	Ref.
H	H	Ph	H	266	—	70
H	H	Ph	Me	240	—	70
Me	H	Ph	H	174-5	—	70
Me	Me	H	H	—	229-231 (dec.) (HCl)	27
Me	Me	Me	H	—	211-212 (HCl) 230-2 (dec.) (HBr)	27
Me	Me	Et	H	—	201-5 (dec.) (HBr)	27
Me	Me	Ph	H	144	—	67, 70
Et	Et	H	H	—	204-6 (dec.) (HCl)	27
Et	Et	Me	H	—	203-5 (HCl)	27
Ph	H	Ph	H	176	—	69
Ph	Me	Ph	H	134-5	—	69



Scheme 64

structure. Guidicelli et al. (67) synthesized tautomeric compounds: 2-amino-4-oxo-5-phenylselenazoline and 2-(methylamino)-4-oxo-5-phenylselenazoline, as well as three compounds possessing a fixed structure: 2-(dimethylamino)-4-oxo-5-phenylselenazoline ("amino" structure), 2-imino-3-methyl-4-oxo-5-phenylselenazolidine ("imino" structure), and 2-(methylinimo)-3-methyl-4-oxo-5-phenylselenazolidine ("imino" structure). A study of the ultraviolet spectra in ethanol solution and of the  $pK_a$  (67) led to the conclusion that the "amino" structure is predominant in tautomeric equilibria, contrary to the conclusion reached by Comrie et al. (68).

TABLE X-15a. 2-IMINOSELENAZOLIDINE-4-ONES

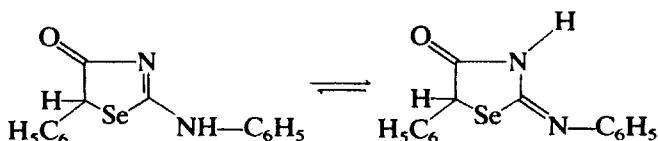
$R_1$	$R_2$	$R_3$	$R'_3$	m.p. ( $^{\circ}\text{C}$ )	m.p. (HBr or HCl derivative) ( $^{\circ}\text{C}$ )	Ref.
H	H	H	H	— <sup>a</sup>	219–224 (HCl) 220–224 (dec.)	68
H	H	Me	H	—	178–180 (dec.) (HBr)	68
H	H	Et	H	178–180 (dec.)	220–4 (dec.) (HBr)	68
H	H	Ph	H	200–4 (dec.) 265–6	—	68
H	Me	Et	H		223 (dec.) (HI)	—
H	Me	Ph	H	115 <sup>b</sup>	—	67, 70
Me	Me	Ph	H	98–9	—	67, 70
Et	Et	H	H	50–1	179–180 (HCl)	68
Et	Et	Et	H	oil	154–6 (HBr)	—
Et	Et	Ph	H	96–7	—	—
Ph	H	Ph	H	176 <sup>c</sup>	—	70
Ph	Me	Ph	H	98–100	—	69
CONHC <sub>6</sub> H <sub>5</sub>	Me	Ph	H	184–5	—	67, 70

<sup>a</sup> Acetyl derivative, m.p. = 236 to 240°C (dec.); 5-benzylidene derivative, m.p. = 270 to 285°C (dec.).

<sup>b</sup> 2,4-Dione after hydrolysis, m.p. = 96°C.

<sup>c</sup> 2,4-Dione after hydrolysis, m.p. = 164 to 165°C (67), m.p. = 160 to 162°C (71).

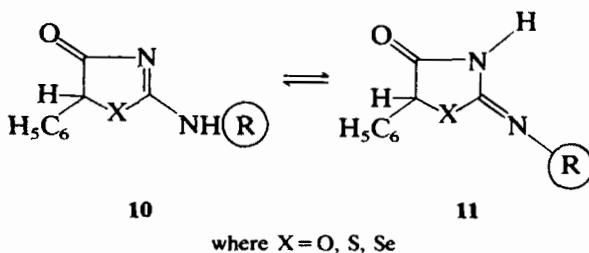
However, in the case of derivatives of the type 2-(phenylamino)-4-oxo-5-phenylselenazoline (Scheme 65), the tautomeric equilibrium is displaced towards the "imino" form (69, 70). This was also shown by the ultraviolet spectra.



Scheme 65

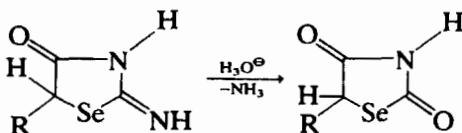
In fact, in this example, the phenyl group takes part in the conjugation of the system and a bathochromic shift is observed.

To sum up, spectroscopic data indicate the "amino" form (**10**) is predominant when R is a hydrogen atom or an alkyl group, while the "imino" form (**11**) is predominant when R is an aryl group (Scheme 66).



Scheme 66

The reactivity of these compounds suggests that they exist mainly in the "imino" form, which explains the ease of hydrolysis to give 2-oxo derivatives (Scheme 67) (71).



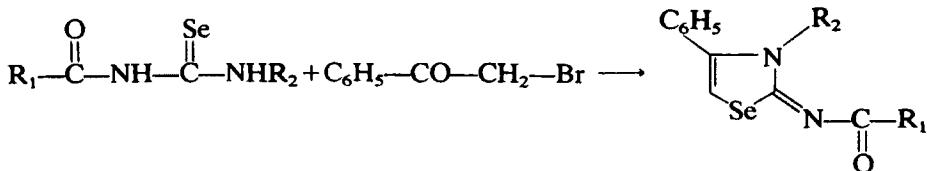
Scheme 67

Hofmann (1) had already demonstrated this type of hydrolysis, which occurs often where the possibility of a tautomerism towards the "imino" form exists. In the case of 2-(dialkylamino) selenazolines, hydrolysis does not take place.

In certain carbocyanines, used as photographic sensitizers in silver halide emulsions, selenazoline rings linked by trimethine bridges are found (56, 72).

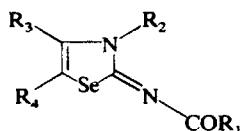
#### D. 2-Acylimino- $\Delta_4$ -Selenazolines (104)

They were prepared as in Scheme 68, and their characteristics are summarized in Table X-15b.



Scheme 68

TABLE X.15b. 2-ACYLIMINO- $\Delta_4$ -SELENAZOLINES    2-ACYLIMINO =  $\Delta_{-4}$   
= SELENAZOLINES



R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	R <sub>4</sub> or Ar	m.p. (°C)	Yield (%)	Ultraviolet $\lambda_{max}$ (nm)/(log ε)			
C <sub>6</sub> H <sub>5</sub>	H	C <sub>6</sub> H <sub>5</sub>	C <sub>6</sub> H <sub>5</sub>	207–209 (ethanol)	96	237 s (4.46)	279 s (3.82)	287 s (3.75)	345 (4.25)
C <sub>6</sub> H <sub>5</sub>	H	C <sub>6</sub> H <sub>5</sub>	p-BrC <sub>6</sub> H <sub>4</sub>	203–204 (nitromethane)	95	235 (4.38)	283 s (3.46)	345 (4.27)	
C <sub>6</sub> H <sub>5</sub>	H	C <sub>6</sub> H <sub>5</sub>	p-NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	295–297 (nitromethane)	81	235 s (4.44)	346 (4.27)		

## 2. Selenazolidines

### A. Alkyl or Arylselenazolidines

A certain number of selenazolidines were first prepared by Draquet and Renson (73) from aziridine using two different pathways (Table X-16).

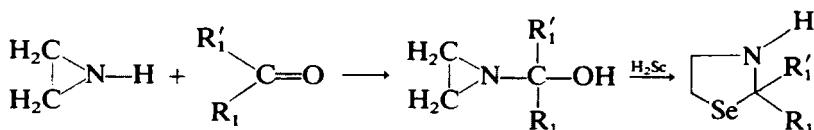
The first involves the reaction of aziridine with an aldehyde or a ketone and the treatment of the resulting carbinol with hydrogen selenide (Scheme 69) (Methode I).

Selenazolidines can also be obtained by first treating aziridine with hydrogen selenide and condensing the intermediate product with a carbonyl compound (Scheme 70) (Methode II).

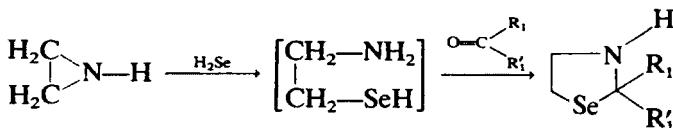
Similarly 2- and 4-disubstituted selenazolidines were prepared from 2-methyl- and 2,2-dimethylaziridines (Table X-17) (74) and 2- and 3-disubstituted selenazolidines were obtained from N-substituted aziridines (Table X-18) (74).

TABLE X-16. 2-ALKYL OR 2-ARYLSELENAZOLIDINES (73)

$R_1$	$R'_1$	b.p./mm ( $^{\circ}$ C)	m.p. ( $^{\circ}$ C)	Method	Yield (%)
H	H	76/20	—	I	33
Me	H	72/20	—	I, II	56, 53
Me	Me	80/20	—	I	50
Me	Et	90/20	—	I	57
Et	H	84/20	—	I	52
<i>n</i> -Pr	H	110/20	—	I	60
Ph	H	—	105	I, II	63, 61
<i>p</i> -MeC <sub>6</sub> H <sub>4</sub>	H	—	98	I	44
<i>p</i> -MeOC <sub>6</sub> H <sub>4</sub>	H	—	96	I	62
<i>o</i> -ClC <sub>6</sub> H <sub>4</sub>	H	—	65	I	48
$-(CH_2)_5-$		95/1	—	I	82



Scheme 69



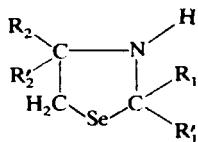
Scheme 70

Treatment of 2,2-dimethylselenazolidines with reactive aromatic aldehydes gives exchange of carbonyl radicals (Scheme 71).

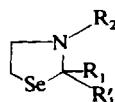
Benzoylation of selenazolidines in aqueous solution leads to opening of the selenazolidine ring and gives dibenzoylselenoalkylamines (Scheme 72) (73).

Selenazolidines are acylated on carbon without ring rupture when the reaction is carried out in benzene in the presence of pyridine (73).

TABLE X-17. 2,4-ALKYL OR ARYLSelenazolidines (74)

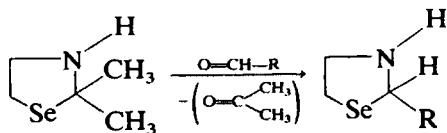


R <sub>1</sub>	R' <sub>1</sub>	R <sub>2</sub>	R' <sub>2</sub>	b.p./mm (°C)	m.p. (°C)	Method	Yield (%)
H	H	Me	H	98/40	—	I	55
H	H	Me	Me	75/25	—	I	40
H	Me	Me	H	79/20	—	I	71
H	Et	Me	H	88/20	—	I	65
H	Et	Me	Me	88/20	—	i	56
Me	H	Me	Me	76/20	—	I	48
Me	Me	Me	H	78/20	—	I	40
Ph	H	Me	H	125/0.5	—	I	49
Ph	H	Me	Me	120/0.3	—	I	25

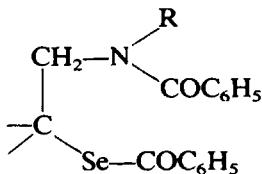
TABLE X-18. 2,3-ALKYL OR ARYL SELENAZOLIDINES<sup>a</sup> (74)

R <sub>1</sub>	R' <sub>1</sub>	R <sub>2</sub>	b.p./mm (°C)	m.p. (°C)	Yield (%)
Me	H	Me	73/20	—	15
Me	H	Ph	102/0.1	—	59
Me	H	Acetyl	99/0.6	—	>90
Me	H	Benzoyl	—	53	>90
Me	Me	Me	76/20	—	21
Et	H	Me	83/20	—	25
Et	H	Ph	108/0.1	—	—
Ph	H	Me	103/0.1	—	17
Ph	H	Acetyl	160/0.4	—	>90
Ph	H	Benzoyl	—	70	>90
Di- <i>o,p</i> -MeOC <sub>6</sub> H <sub>3</sub>	H	H	—	89	>90
di- <i>m,p</i> -MeOC <sub>6</sub> H <sub>3</sub>	H	H	—	78	>90
<i>m</i> -MeOC <sub>6</sub> H <sub>4</sub>	H	H	—	79	>90
<i>o</i> -EtOC <sub>6</sub> H <sub>4</sub>	H	H	—	62	>90
<i>p</i> -ClC <sub>6</sub> H <sub>4</sub>	H	H	—	105	>90
<i>m</i> -ClC <sub>6</sub> H <sub>4</sub>	H	H	—	98	>90
<i>m</i> -BrC <sub>6</sub> H <sub>4</sub>	H	H	—	83	>90
-(CH <sub>2</sub> ) <sub>5</sub> -		Benzoyl	—	125	>90

<sup>a</sup> By method II.

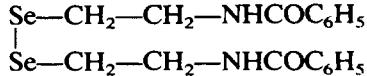


Scheme 71



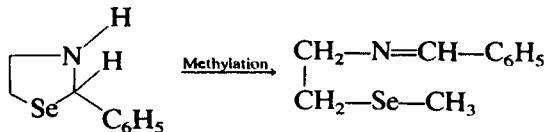
Scheme 72

Hydrolysis and subsequent oxidation by air of benzoylated selenazolidines gives *N,N'*-dibenzoylbis(2-aminoethyl)diselenide (Scheme 73)



Scheme 73

Finally, methylation of 2-phenylselenazolidine leads to benzalamino Se-methylselenocysteamine (Scheme 74).

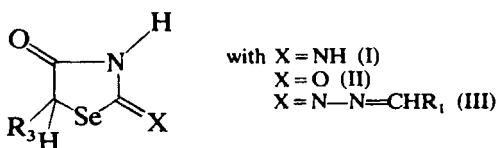


Scheme 74

Methylation takes place on the selenium (73), in contrast to the thiazolidines where it occurs on the heterocyclic nitrogen (75).

**B. 2-Iminoselenazolidin-4-ones (I), Selenazolidin-2,4-diones (II), and 2-Alkylidenehydrazones (III)**

The general structure of these compounds is described in Scheme 75. Type I compounds can show a protomeric equilibrium and thus can be classed eventually with the 2-aminoselenazolines when this latter structure predominates.



Scheme 75

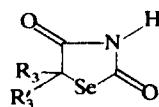
- 2-Iminoselenazolidin-4-ones (I) are prepared by condensation of  $\alpha$ -halo acids with selenourea in ethanol solution. The free base is obtained at the end of the reaction by neutralization of the hydrohalide (Table X-19) (71).

TABLE X-19. 5-ALKYL-2-IMINOSELENAZOLIDIN-4-ONES  
(71)

$R_3$	$R'_3$	m.p. (°C)	Yield (%)
n-Pr	H	184-6	80
i-Pr	H	211-3	61
Bu	H	179-181 (dec.) (HBr)	43
Ph	H	200-4	60

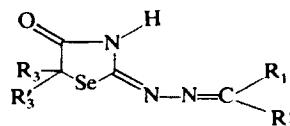
- Selenazolidin-2,4-diones (II) are obtained in the same way as the 2-iminoselenazolidin-4-ones, except that the reactants are under reflux in aqueous solution (Table X-20) (71).
- Selenazolidin-2,4-diones-2-alkylidene (or arylidene) hydrazones (III) are simply prepared by base-catalysed condensation of  $\alpha$ -haloacids on selenosemicarbazones of acetone or benzaldehyde (Table X-21) (71).

TABLE X-20. 5-SUBSTITUTED SELENAZOLIDIN-2,4-DIONES (71)



R <sub>3</sub>	R' <sub>3</sub>	m.p. (°C)	Yield (%)	m.p. (5-derivative) (°C)
Me	H	74-5	82	250-2 (5-benzylidene) 214-6 (5-salicylidene)
Me	Me	82.5-83.5	68	—
Et	H	69-71	75	—
n-Pr	H	55-6	67	—
i-Pr	H	73-4	73	—
n-Bu	H	92-3	89	—
Ph	H	160-2	60	—

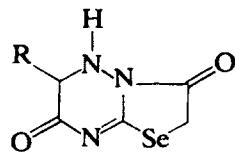
TABLE X-21. SELENAZOLIDIN-2,4-DIONES-2-ALKYLIDENE OR ARYLIDENE HYDRAZONE (71)



R <sub>1</sub>	R' <sub>1</sub>	R <sub>3</sub>	R' <sub>3</sub>	m.p. (°C)	Yield (%)	Ref.
H	Ph	H	H	254-6 (dec.)	—	—
H	Ph	Et	H	200-2	64	—
Me	Me	H	H	101-4	61	—
Me	Me	H	Et	103-6	69	—
Me	Me	H	Ph	208	39	—
p-Me <sub>2</sub> N <sub>C</sub> <sub>6</sub> H <sub>4</sub>	H	H	H	—	—	76
p-MeOC <sub>C</sub> <sub>6</sub> H <sub>4</sub>	H	H	H	—	—	76
<i>o</i> -HOC <sub>C</sub> <sub>6</sub> H <sub>4</sub>	H	H	H	—	—	76

2-Iminoselenazolidin-4-one is benzoylated on the "imino" nitrogen with benzoyl chloride in pyridine solution (77).

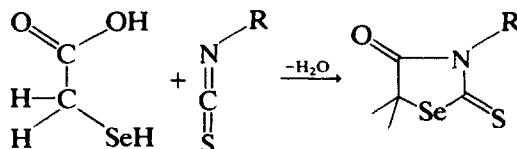
Selenotriazines (Scheme 76) can be obtained starting from seleno-semicarbazide (76).



Scheme 76

### C. 2-Thio-4-oxoselenazolidines

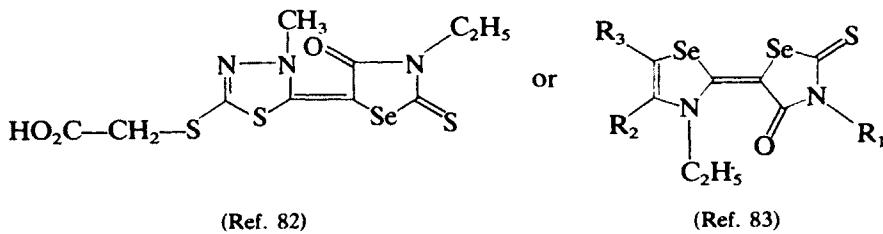
2-Thio-4-oxoselenazolidines substituted in the 3-position, have been described in a number of patents, (79, 80). They are used as intermediates in the manufacture of pharmaceutical products and color sensitizers in photography. They are obtained by action of isothiocyanates on  $\alpha$ -hydroselenoacetic acid in the presence of strong base and in the absence of air (Scheme 77).



Scheme 77

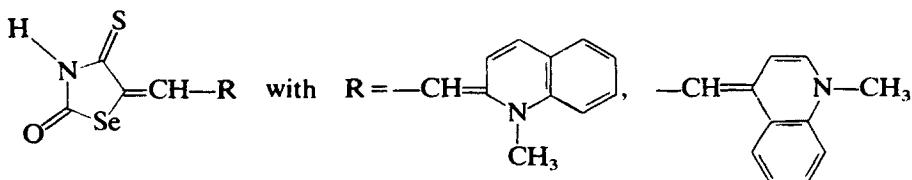
The following compounds have been prepared: R = Et, m.p. = 61–2°C; R = Ph, m.p. = 202°C; R = allyl, m.p. = 45–6°C; R = cyclohexyl, m.p. = 159°C; and R = benzyl, m.p. = 120–1°C.

4-Thio-2-oxoselenazolidine can be obtained in 89% yield by treating the 2,4-dione with  $P_4S_{10}$  in boiling dioxane (81). This structure is found in heterocyclic indigo-type dyes (Scheme 78).



Scheme 78

Merocyanines with selenazolidine nuclei are obtained in 70 to 80% yield from the appropriate derivatives of lepidine or quinaldine (Scheme 79) (84).



Scheme 79

## V. PHYSICOCHEMICAL STUDIES

### 1. Infrared

Comparative studies have been undertaken between different heterocyclic series, especially the azoles, thiazoles, oxazoles, imidazoles, and selenazoles. In a large number of these studies, the heterocycles are condensed with aromatic nuclei.

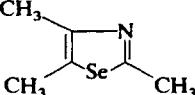
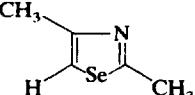
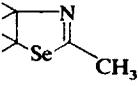
#### A. *Methylselenazoles*

Bassignana et al. (85) have studied 2,4-dimethylselenazole, 2,4,5-trimethylselenazole, and 2-methylselenazoline. The main infrared absorption bands observed are summarized in Table X-22.

Benzoselenazoles have an additional band at 1590 to 1610  $\text{cm}^{-1}$ . The presence of high-intensity infrared bands in selenazolines is further evidence for the assignment of the "selenazole I" vibrations to  $\nu_{\text{C}=\text{N}}$  ( $1650$ – $1680 \text{ cm}^{-1}$ ) and the "selenazole II" vibration to the group  $-\text{N}=\text{C}-\text{Se}-$  ( $1537$ – $1570 \text{ cm}^{-1}$ ).

The authors (85) have also mentioned the deformational vibrations ( $\gamma$ ) of the out-of-plane group  $-\text{CH}=$  of a pentatomic heterocyclic nucleus, showing that there exists a relationship between the absorption frequencies of this group and the electronegativity of the heteroatom (86).

TABLE X-22. INFRARED ABSORPTION

Compounds	Selenazole I	$\nu_{C=C}$	Selenazole II	Heterocyclic nucleus
	1650 M	—	1550 M	950 M
	1650 M	1572 W	1531 S	930 S
	1656 s	—	1558 S	930 W

**B. Substituted Selenazolidinediones  
(and their sulfur and oxygen homologs)**

These compounds have infrared spectra that are greatly complicated by harmonics and combination bands in the region of carbonyl group vibrations.

The two absorption bands in the spectral region, 1690 to 1850  $\text{cm}^{-1}$ , of oxazolidinediones, thiazolidinediones, and selenazolidinediones (87), cannot be assigned to two independent carbonyl groups. They are in fact the result of an electronic coupling analogous to that observed with imides (88). The intensity of the out-of-plane vibrational absorption bands ( $\gamma_1$ ) favors a planar conjugated group and also undoubtedly a nearly planar ring, even in the case of selenazolidine dimers (bulky heavy atom). The low-frequency C=O vibration (1695–1782  $\text{cm}^{-1}$ ) is the result of an out-of-phase vibration of the two C=O groups ( $\nu_1$ ), while the high-frequency band (1757–1849  $\text{cm}^{-1}$ ) corresponds to an in-phase vibration of the C=O groups ( $\nu_2$ ). With the replacement of the oxygen of oxazolidine-2,4-diones by S, Se, or NH, the frequency of the coupled C=O vibrations increases linearly with the increase in electronegativity of the heteroatom. The separation ( $\Delta\nu = 37\text{--}79 \text{ cm}^{-1}$ ) and the relative intensities of these two bands are independent of the substitution and dependent primarily on the

nature of the substituents on the 5-carbon and on the nitrogen between the 2 C=O groups (89).

### C. 2-Thio-4-oxoselenazolidines (and their oxygen, sulfur, and nitrogen homologs)

Cogrossi (90) studied these spectra in the region 4000 to 400 cm<sup>-1</sup>. The different characteristic bands have been assigned as follows:  $\nu_{C=O}$  at 1708 to 1765 cm<sup>-1</sup>; the group N-C=S, thioureide I at 1300 to 1550 cm<sup>-1</sup>, thioureide II at 1200 to 1300 cm<sup>-1</sup>, and thioureide III at 1100 to 1180 cm<sup>-1</sup>.

Methyl bonding, methylene scissor, and  $\gamma_{Car-N}$  modes occur at 1460 to 1340 cm<sup>-1</sup>. Ring bonding and methyl rocking modes are found at 1040 to 1070 and 980 cm<sup>-1</sup>, respectively. Four bands, at 600 to 700, 550 to 600, 520 to 500, and 430 to 510 cm<sup>-1</sup> are attributed to the  $\beta_{ring} + \delta_{C=O}$ ,  $\beta_{ring} + \delta_{C=S}$ ,  $\gamma_{C=O} + \beta_{ring}$  modes, respectively.

## 2. Ultraviolet Spectroscopy

Comparison of the ultraviolet spectra of analogous sulfur and selenium compounds shows that there is very little difference in the absorption curves, except for a slight bathochromic shift in the case of the selenium derivatives.

## 3. NMR and Mass Spectrometry

Noncondensed aromatic or saturated selenated heterocycles are little mentioned in NMR or mass spectrometry literature.

# VI. APPLICATIONS OF SELENIUM HETEROCYCLES

## 1. Dyes

Carbocyanine or merocyanine dyes prepared from selenazole or selenazoline rings have a particular interest in photography.

2-Hydrazono-5-(*p*-dialkylaminophenylimino)selenazoles, pyrazolone azomethin selenazoles, quinoid dyes, and formazans are other important dyes used in photography and the color industry (cf. previous sections).

## 2. Biological Activity and Radioprotection

A large number of selenium derivatives such as the selenium analog of vitamin B<sub>1</sub> have been tested for physiological activity (91).

Sulfonamides prepared from 2-aminoselenazoles by normal methods sometimes possess activity comparable to that of sulfathiazole (45, 47).

The ability of various selenium heterocycles to check the loss of orthophosphate caused by irradiation of ATP has been studied by Brucker and Bulka (92). They found that only 2-amino-4,5-dimethylselenazole shows radioprotective properties, while other 2-aminoselenazoles, selenosemicarbazides, and acetone selenosemicarbazones possess no such activity but are in addition very sensitive to radiation (93).

A series of selenazolidines substituted in the 2-position have been synthesized to be used as antiradiation agents, but no results are yet available (94).

## VII. CONCLUSION

Selenium heterocycles receive far less mention in the literature than do such homologs as oxazole, thiazole, or imidazole. In fact, preparative methods of selenium heterocycles are much more limited than for the other series, mainly because of manipulatory difficulties arising from the toxicity of selenium (hydrogen selenide is even more toxic) that can produce severe damage to the skin, lungs, kidneys, and eyes. Another source of difficulty is the reactivity of the heterocycle itself, which can easily undergo fission, depending on the reaction medium and the nature of the substituents.

Condensed aromatic compounds (benzoselenazoles) are considered much more stable, and they are in fact more commonly used in the area of cyanine-type photographic dyes.

Despite the inconveniences, a certain number of studies have been carried out, particularly concerning dyes containing azomethine groups, such as hydrazones, pyrazolones, formazans, and selenazoles quinoids. Saturated heterocycles, that is, selenazolines and selenazolidines, have also been tackled. Selenium derivatives for pharmacological or physiological applications are little developed by comparison with their thiazole homologs.

Finally, as far as physicochemical or theoretical studies are concerned, except for a few infrared studies, especially comparative ones on azoles (O, S, Se), little work has been done. This is particularly true of recent

spectrometric techniques (NMR and mass spectrometry) and in the field of quatochemical studies.

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