

ADVANCES IN HETEROCYCLIC CHEMISTRY, VOL. 79

Tellurium–Nitrogen-Containing Heterocycles

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I. Introduction

The chemistry of Te,N-containing heterocycles dates back to 1971 when the first representatives of this class of compounds, 2,5-diaza-1,6-dioxa-6*a*-tellurapentalenes, were prepared (71BSF4591). Currently, more than 20 diverse structural types of the heterocycles containing tellurium and nitrogen atoms in a ring have become known, most having been studied during the last decade. Both the methods for the preparation and the reactions of Te,N-containing heterocycles generally differ from those characteristic of their sulfur and selenium analogues. Thus, owing to the enhanced thermal stability of σ -telluranes [Te(IV) derivatives] and the high electrophilicity of TeHal₃ groups, aryltellurium trichlorides can serve as expedient precursors of various tellurium heterocycles. These compounds readily undergo oxidation–addition reactions, in particular with halogens, to give easily isolated derivatives of tetracoordinate tellurium that are substantially more stable than the corresponding sulfur or selenium compounds. Owing to the higher nucleophilicity of Te(II) centers, tellurium heterocycles readily form telluronium salts and give stable complexes even with relatively soft Lewis acids. In tellurium heterocycles, Te(II) centers are capable of forming intramolecular and intermolecular coordination bonds with oxygen and nitrogen centers which are stronger than the bonds formed by other chalcogen centers. This property of organotellurium compounds is responsible for the peculiar physical characteristics of benzoisotellurazole, 1,2,5-telluradiazole, and some other Te,N-containing heterocycles.

The early literature on Te,N-containing heterocycles has been reviewed up to 1991 [86MI1; 86MI2; 93AHC(58)47]. This review updates our previous work [93AHC(58)47] and covers the literature to the end of 1999. This review is organized into sections according to ring size and the number and origin of heteroatoms. Neither heterocycles containing tellurium and nitrogen in different rings nor inorganic Te,N-containing heterocycles are considered. Extensive reviews on these compounds may be found in [94CCR301; 98AHC(71)115].

II. Heterocycles with One Te Atom and One N Atom

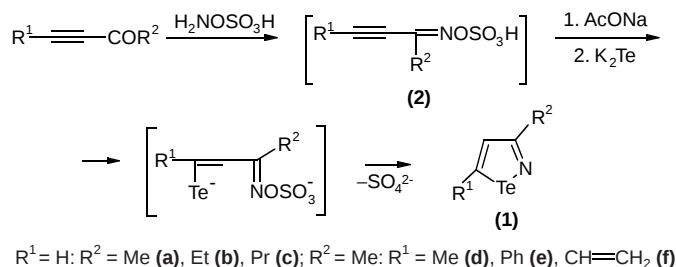
A. FIVE-MEMBERED HETEROCYCLED

Thus far, the known compounds of this type include isotellurazoles, their benzo fused derivatives, and benztellurazoles.

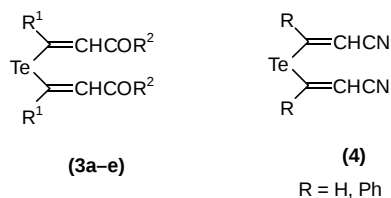
1. *Isotellurazoles*

Isotellurazoles **1a–e** were first prepared by coupling ethynyl ketones with hydroxylamine-*O*-sulfonic acid and K₂Te in aqueous solution of sodium acetate

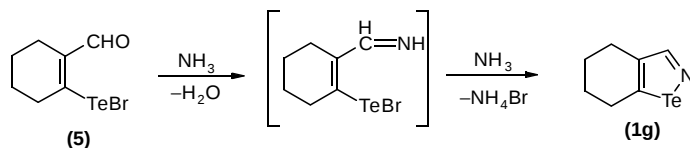
(83S824). More recently, this method was employed for the synthesis of 3-methyl-5-vinyl isotellurazole **1f** (87H1587). It was assumed that the reaction occurs through the intermediate oxime-*O*-sulfonic acids **2**.



3,5-Disubstituted isotellurazoles **1** (4–11%) and bis(β -acylvinyl)tellurides **3** (3–10%) were isolated in very low yields from the reaction mixture as the products of nucleophilic addition of telluride anion to the triple bond of the initial ethynyl ketones (83S824). This method cannot be applied to the synthesis of 3*H*-isotellurazoles. When α -acetylenic aldehydes were used instead of ethynyl ketones, bis(β -cyanovinyl)tellurides **4** obtained in 14–20% yields were the only products (83S824).

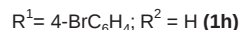
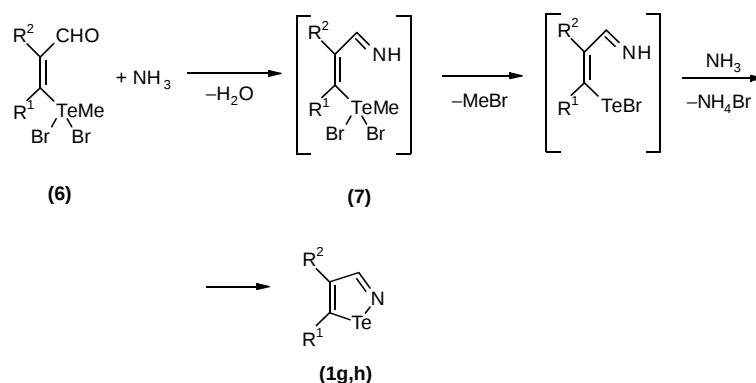


Appreciably higher is the synthetic potential of the methods based on the use of such precursors as β -bromotellurenylvinylaldehydes **5** and β -methyldibromotellurovinylaldehydes **6** [97DOK(357)504]. By bubbling ammonia through a benzene solution of tellurenyl bromide **5**, 4,5-tetramethyleneisotellurazole **1g** was prepared in 70% yield.



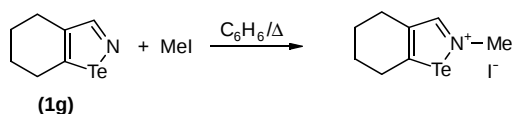
Type **5** tellurenyl bromides are not easily accessible. They were prepared by refluxing an acetic acid solution of tellurium dibromides **6** in the presence of catalytic amounts of HBr [96ZOK1434; 97JOM(536-537)233]. The reaction is not easily reproducible, affording the tellurenyl bromides in rather low yields.

More expedient from the preparative point of view is the method based on compounds **6**, which can be obtained in high yields by the addition of bromine to β -methyltellurovinylaldehydes [96ZOK1434; 97JOM(536-537)233]. When treated in benzene solution with ammonia, β -methyl dibromotellurovinylaldehydes **6** afford isotellurazoles **1g,h** in about 70% yields [97DOK(357)504]. The key step of the reaction is the elimination of a molecule of methyl bromide from the intermediate imine **7**.

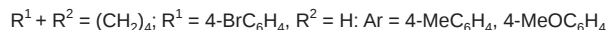
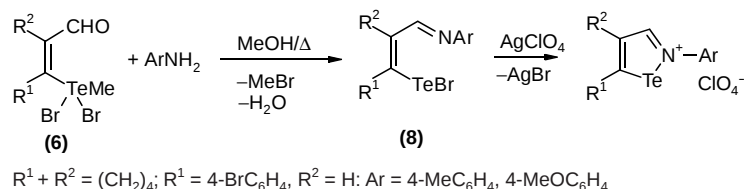


This method is similar to that employed in the synthesis of isoselenazoles (73JHC267). However, owing to the thermal stability of the tellurium dibromides **6**, one can perform the reaction at room temperature, whereas with selenium dibromides it must be carried out at -70°C .

Reactions of isotellurazoles have been little studied so far. By heating a benzene solution of **1g** and methyl iodide, *N*-methyl-4,5-tetramethyleneisotellurazolium iodide was obtained in 70% yield [97DOK(357)504].

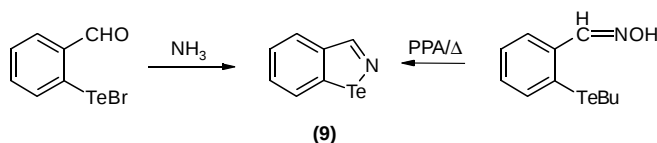


N-Arylisotellurazolium perchlorates were prepared in 78–86% yields by the reaction of β -bromotellurenylvinylaldimines **8** with silver perchlorate in DMSO or acetone [97DOK(357)504].

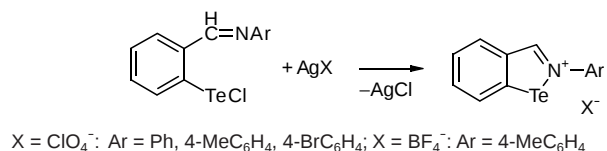


2. Benzoisotellurazoles

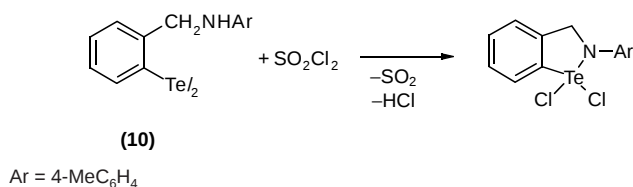
To date, only the parent benzoisotellurazole **9** has been obtained. For its synthesis, methods similar to those used for the preparation of benzoisothiazole and benzoisoselenazole were employed. The reaction of 2-bromotellurenylbenzaldehyde with ammonia affords **9** in 74% yield, whereas cyclization of the oxime of *o*-butyltellurobenzaldehyde catalyzed by PPA gives **9** in 40% yield (78JHC865). In contrast to the reaction of 2-bromoselenenylacetophenone with ammonia, which leads to 3-methylbenzoisoselenazole (73JHC267), 2-bromotellurenylacetophenone reacts with ammonia to give telluroindoxyl (78JHC865).



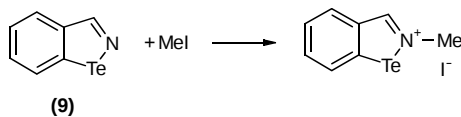
The only other known compounds containing the benzoisotellurazole fragment are the *N*-arylbenzoisotellurazolium salts, prepared in 65–84% yields by a treatment of the easily accessible 2-chlorotellurenylbenzalanilines with AgClO_4 [88KGS1426; 90JOM(391)177; 91JOM(402)331] or AgBF_4 [93JCS(D)619].



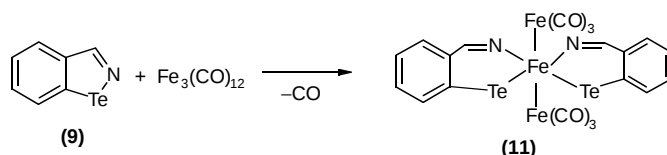
By coupling the ditelluride **10** with sulfuryl chloride, 1,1-dichloro-2-(4-methylphenyl)-3*H*-benzoisotellurazole was obtained [93JCS(D)619].



N-Methylbenzoisotellurazolium iodide is readily formed by the action of methyl iodide on **9** (78JHC865).



On refluxing a toluene solution of benzoisotellurazole and $\text{Fe}_3(\text{CO})_{12}$, cleavage of the Te—N bond occurs, resulting in formation of the metal chelate complex **11** whose structure was determined by X-ray (97MI1).



In contrast with benzoisothiazole and benzoisoselenazole, benzoisotellurazole **9** is poorly soluble in most organic solvents. Its melting point (173°C) is anomalously high compared to those of benzoisothiazole (39°C) and benzoisoselenazole (57°C) (78JHC865). These peculiarities are caused by strong intermolecular coordination $\text{Te} \cdots \text{N}$ bonds between molecules of **9** in the crystalline state. The $\text{Te} \cdots \text{N}$ distances ($2.40 \text{ \AA}$) (78JHC745) are $1.3 \text{ \AA}$ shorter than the sum of the length of van der Waals radii of tellurium and nitrogen ($3.70 \text{ \AA}$) (60MI1) and are very close to the length of the covalent Te—N bond ($2.11 \text{ \AA}$) in benzoisotellurazole. The covalency ratio of the intermolecular $\text{Te} \cdots \text{N}$ bonds estimated according to Weinhold (88CR889) is 0.8. This value is comparable with those calculated for the strongest intramolecular coordination $\text{Te} \cdots \text{N}$ bonds (98CJC766; 99RKZ10).

The UV absorption spectrum of benzoisotellurazole is similar to those of its sulfur and selenium analogues, all absorption bands of the former being bathochromically shifted relative to the latter compounds. The ^1H NMR signal of the H-3 proton of **9** appears at lower field than the respective signals of benzoisothiazole and benzoisoselenazole (Table I).

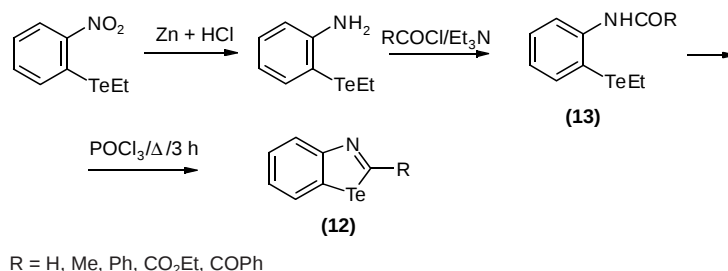
3. Benzotellurazoles

a. *Synthesis.* A three-step method based on 2-ethyltelluronitrobenzene (82TL 3905) as the starting material was used for the synthesis of benzotellurazole and

TABLE I
UV SPECTRA (ETHANOL) AND ^1H NMR CHEMICAL
SHIFTS (DMSO) OF THE H-3 PROTON
OF BENZOISOCALCOGENAZOLES

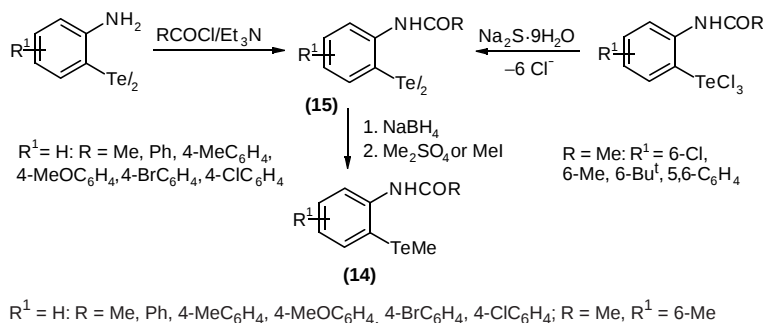
M	λ_{max} , nm (log ϵ)	δ , ppm
S	204(4.19), 222(4.36), 303(3.57)	8.37
Se	203(4.15), 228(4.30), 318(3.67)	9.15
Te	207(4.27), 242(4.15), 344(3.65)	10.16

its derivatives **12** (83TL5873). The cyclization of the intermediately formed *N*-acylanilides **13** proceeds in 2–15% yields during a 3-hour refluxing of their POCl₃ solutions.

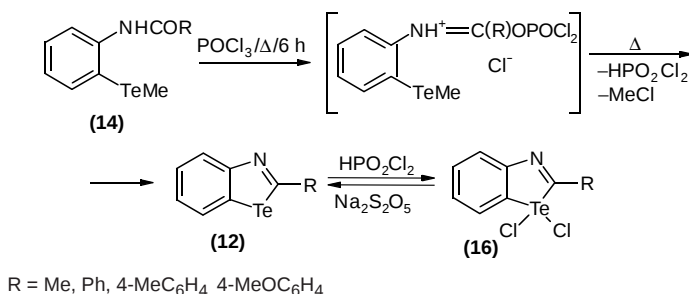


Junk and Irgolic (88MI1) failed to reproduce preparation of 2-ethyltelluronitrobenzene following the procedure described in (82TL3905). Because of this, *N*-acyl derivatives of 2-methyltelluroaniline **14** were used as other precursors for **12**. Compounds **14** were obtained by methylation of sodium arenetellurolates (88KGS276; 88MI1; 89KGS120) prepared *in situ* by reduction of diaryl ditellurides **15** with NaBH₄.

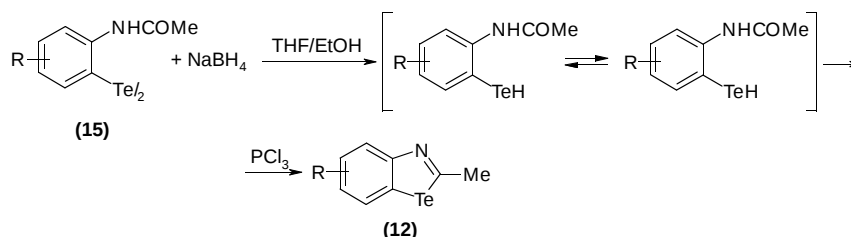
Compounds **15** can be prepared either by acylation of bis(*o*-aminophenyl) ditelluride (88KGS276; 89KGS120) or by reduction of *N*-acyl-2-trichlorotellurobenzenes with sodium sulfide (88MI1).



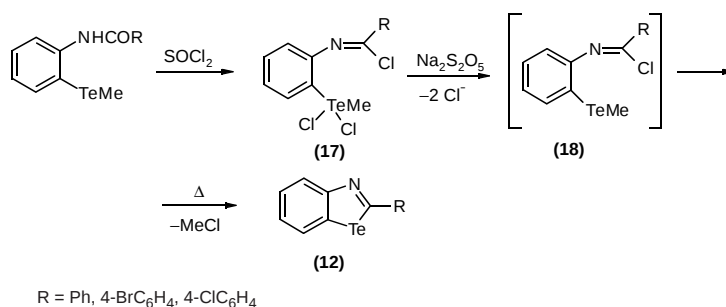
A 6-hour refluxing of purified anilides **14** in POCl₃ led not to benzotellurazoles **12**, but to their 1,1-dichloro derivatives **16** obtained in 35–60% yields (89KGS120).



For the synthesis of various 2-substituted benzotellurazoles **12**, PCl_3 instead of POCl_3 was used as the cyclizing reagent (88MI1). The yields of **12** are in the range of 30–50%.



When sulfuryl chloride was used in this reaction instead of POCl_3 or PCl_3 , imidoyl chlorides **17** were isolated as the products (89KGS120). By reduction of **17** with $\text{Na}_2\text{S}_2\text{O}_5$ aryl methyl tellurides **18** are formed which readily eliminate a molecule of methyl chloride to give 2-arylbenzotellurazoles **12** ($\text{R} = \text{Ar}$) in 40–65% yields.

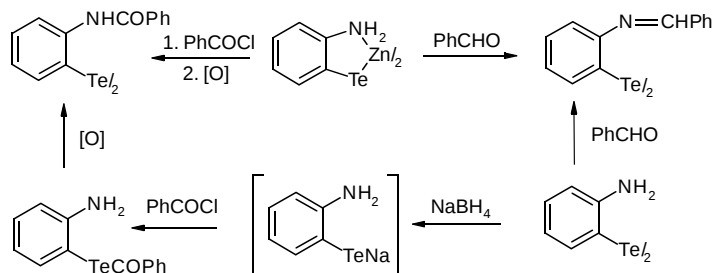


Elimination of a molecule of methyl chloride takes place also in several other cyclizations of organyl methyl tellurides containing active chlorine atoms, e.g., in the synthesis of tellurocoumarin (84JHC1281) and telluroisocoumarin (80JOC3535).

The structure of benzotellurazoles was proved by elemental analysis, spectral data, and X-ray study of 2-phenylbenzotellurazole.

The molecular and crystal structures of 2-phenylbenzotellurazole were determined by X-ray (89KGS1690). The dihedral angle between the planes of the five-membered heterocycle and 2-phenyl ring is 31.2° . In contrast to benzoisotellurazole, no shortened intermolecular $\text{Te} \cdots \text{N}$ contacts were found in the crystal structure of 2-phenylbenzotellurazole. Consequently, no anomalies in solubility and melting point were revealed for this compound as compared with its sulfur and selenium congeners.

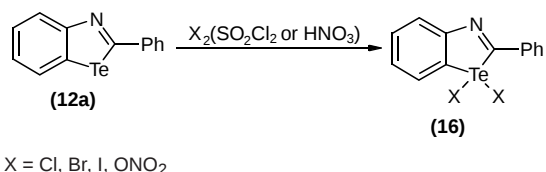
Attempts to use other precursors and cyclizing reagents for the synthesis of benzotellurazoles failed. Thus, phosphonitrile dichloride, which readily reacts with 2-methylthioacetanilides to give benzothiazoles, is inert with respect to the tellurium analogues of thioacetanilides (77S892). In the same way, 2-chlorotellurenylazobenzene does not react with methylene active compounds (acetone, acetophenone, malonic acid), whereas 2-chlorosulphenylazobenzene readily undergoes cyclization leading to benzothiazoles (56JCS648). No benzotellurazoles were obtained in the reactions of sodium or zinc *o*-aminotellurophenolate (86ZOB2168) with benzoyl chloride and benzaldehyde, although the metal salts of *o*-aminothiophenol and *o*-aminoselenophenol afford benzothiazoles and benzoselenazoles, respectively, upon coupling with those reagents.



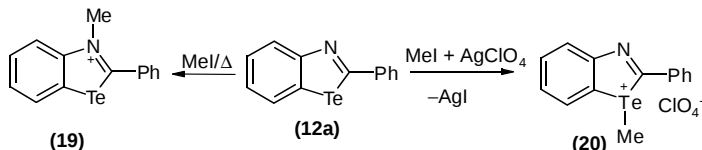
These facts, together with many other examples of the peculiar chemical behavior of organotellurium compounds (95UK527), emphasize their specific reactivity and explain why a number of methods developed for preparation of organosulfur and organoselenium compounds are inapplicable for their tellurium analogues.

b. *Reactions.* Whereas sulfur and selenium atoms in benzothiazoles and benzoselenazoles, respectively, are weak nucleophilicity centers, electrophilic reactions occur at the tellurium atom benzotellurazoles rather readily. They were studied mainly on the example of reactions of 2-phenylbenzotellurazole.

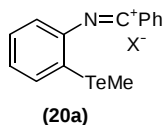
i. REACTIONS AT THE TELLURIUM ATOM. 2-Phenylbenzotellurazole **12a** readily undergoes various oxidation–addition reactions at the Te(II) center to give the Te(IV) derivatives **16** (89KGS989).



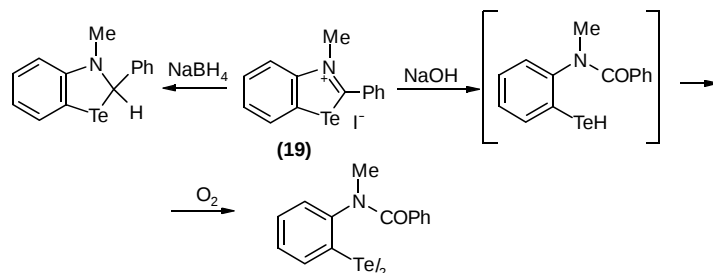
In contrast with benzothiazoles for which alkylation occurs only at the nitrogen atom, 2-phenylbenzotellurazole forms upon methylation both N- and Te-methyl derivatives, depending on the reaction conditions. *N*-Methyl-2-phenylbenzotellurazole **19** was obtained in 90% yield on heating a methyl iodide solution of **12a** (88KGS136; 89KGS120). When silver perchlorate was added to the solution Te-methyl-2-phenylbenzotellurazole **20** was formed in almost quantitative yield. The isomers **19** and **20** can easily be differentiated according 1H NMR chemical shifts of their methyl groups: 3.81 ppm in **19** and 2.28 ppm in **20** (88KGS136; 89KGS120).



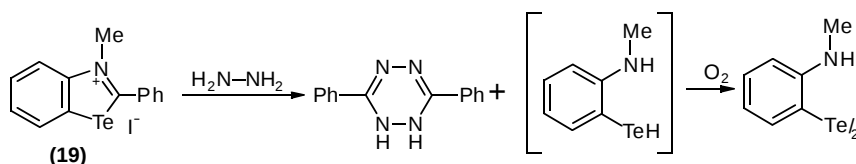
According to semiempirical MNDO/PM3 calculations, the Te-methylated isomer **20** is energy-disfavored relative to **19**. Moreover, the C–Te⁺ bond in **20** is elongated (3.53 Å) to such an extent that its structure should better be described as the carbenium ion **20a** [94JCS(P2)2341].



Reactions of **19** are similar to those of other *N*-methylbenzochalcogenium salts. Under the action of $NaBH_4$ **19** is reduced to 3-methyl-2,3-dihydro-2-phenylbenzotellurazole. The alkaline hydrolysis of **19** gives rise to di(*o*-*N*-methyl-*N*-benzoylaminophenyl) ditelluride (92MI1)

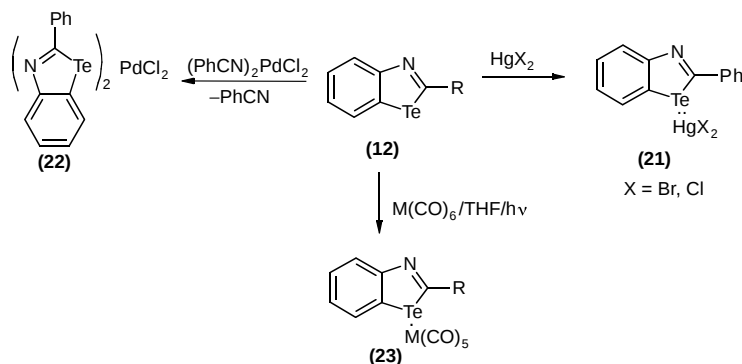


Like *N*-methyl-2-phenylbenzothiazolium iodide (76KGS635), the salt **19** reacts with hydrazine hydrate to give 3,6-diphenyl-1,2-dihydro-1,2,4,5-tetrazine and di(*o*-*N*-methylaminophenyl) ditelluride (92MI1).



Te-Methyl-2-phenylbenzotelluroniumazazole perchlorate **20** behaves as a strong methylating agent in reactions with O- and N-nucleophiles (92MI1).

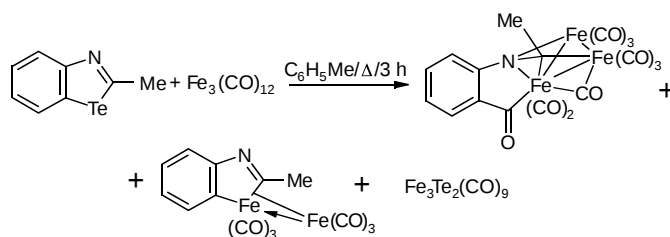
With soft Lewis acids, such as Hg(II) and Pd(II) salts, 2-phenylbenzotellurazole readily affords 1:1 and 2:1 complexes (compounds **21** and **22**, correspondingly) (89KGS989). Their structure has not yet been X-ray determined, and coordination at the Te(II) atoms was proposed by analogy with the properties of complexes of diorganyl tellurides (95MI1) and telluroxanthene (80KGS1342). In a similar way, tungsten pentacarbonyl also coordinates the tellurium center by forming with **12a** a stable complex **23**. As shown by an X-ray diffraction study (99MI1), the length of the W—Te bond in this complex is 2.809 Å, bond lengths in the coordinated and free ligand being insignificantly different (99MI1).



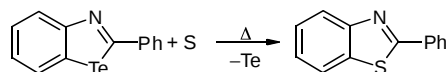
R = Ph: M = W (**a**); R = Me: M = Cr (**b**)

The complex **23** is the first X-ray structurally characterized metal coordination compound with a benzochalcogenazole ligand in which chalcogen behaves as the ligating atom.

Refluxing a toluene solution of 2-methylbenzotellurazole **12b** with $\text{Fe}_3(\text{CO})_{12}$ leads to detelluration of the heterocycle and formation of the iron clusters (97MI1).



Owing to the fact that a C—S bond energy is greater than that of a C—Te bond, the tellurium atoms in phenoxatellurines and phenotellurazines are readily replaced by sulfur under heating with elemental sulfur [93AHC(58)47]. In a similar way, 2-phenylbenzotellurazole converts to 2-phenylbenzothiazole with 42% yield (89KGS989).



ii. REACTIONS AT THE NITROGEN ATOM. Alkylation at the nitrogen atom of **12a** was considered in Section II,A,3,b,i. Protonation of benzotellurazoles occurs at the nitrogen atom. Gas phase basicities (ΔGB) of a series of benzotellurazoles and their oxygen, sulfur, and selenium analogues were determined by means of Fourier transform ion cyclotron resonance spectroscopy [94JCS(P2)2341]. The ΔGB values and the values of $\text{p}K_{\text{BH}^+}$ for the solutions in acetonitrile (91KGS836) are listed in Table II.

As seen from the data given in Table II, the basicities of the heterocycles **24** with the same substituent in position 2 increase in the order $\text{O} < \text{S} < \text{Se} < \text{Te}$ which corresponds to decrease in the negative inductive effects of these atoms. Another factor to be accounted for is that the largest (in this series of compounds) value of the CNC angle is that for benzotellurazoles (89KGS1690). Accordingly, the sp^n orbital of the lone electron pair in benzotellurazoles has the greatest s character, which finding correlates with their higher basicities.

iii. ALDOL CONDENSATION REACTIONS. Coupling 2-methylbenzotellurazole with aromatic aldehydes affords 2-styrylbenzotellurazoles **25** in >60% yields (89KGS120).

TABLE II
GAS PHASE (DGB) BASICITIES (333 K) [94JCS(P2)2341], BASICITY CONSTANTS (pK_{BH^+})
MEASURED IN ACETONITRILE (25°C) (91KGS836), AND MNDO PM3 CALCULATION
[94JCS(P2)2341] PROTON AFFINITIES (PA) OF COMPOUNDS

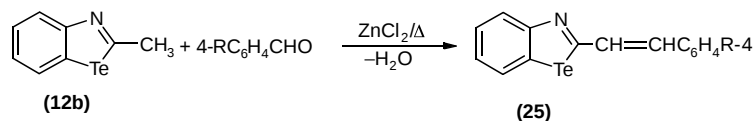
X	R	ΔGB (kcal mol ⁻¹) ^a	pK_{BH^+} ^b	PA (kcal mol ⁻¹) ^c
O	H	-9.8 ± 0.2	6.70	210.2
S	H	-14.9 ± 0.2	7.87	211.3
Se	H	—	8.03	208.1
Te	H	—	—	214.7
O	CH ₃	-13.8 ± 0.1	7.30	214.1
S	CH ₃	-18.8 ± 0.2	8.63	214.9
Se	CH ₃	-18.9 ± 0.2	8.87	212.5
Te	CH ₃	-20.3 ± 0.1	10.10	219.6
O	Ph	—	5.95	116.1 ^d
S	Ph	—	7.24	114.6 ^d
Se	Ph	—	7.46	113.0 ^d
Te	Ph	—	8.67	118.4 ^d
Te	4-ClC ₆ H ₄	—	8.36	—
Te	4-BrC ₆ H ₄	—	8.35	—
Te	4-MeC ₆ H ₄	—	9.20	—
Te	4-MeOC ₆ H ₄	—	9.60	—

^aThe values obtained by averaging of four reference bases.

^b pK_{BH^+} values in water can be estimated by using the equation pK_{BH^+} (water) = -7.75 + 1.14 pK_{BH^+} (acetonitrile).

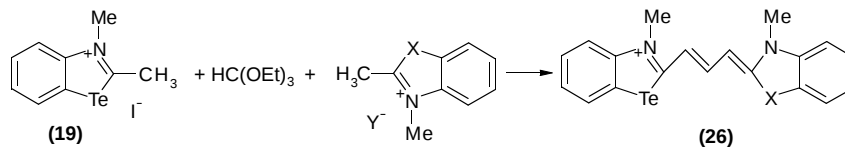
^cProton affinities of the bases calculated using the value $\Delta H_f^0(H^+) = 367.2$ kcal mol⁻¹.

^dMethyl cation affinities.



R = H, OMe, OEt, NO₂

The carbocyanine dyes **26** containing benzotellurazole fragments were obtained by treatment of the salt **19** with triethyl orthoformate (89KGS989).



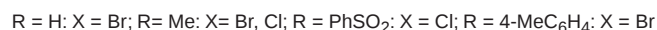
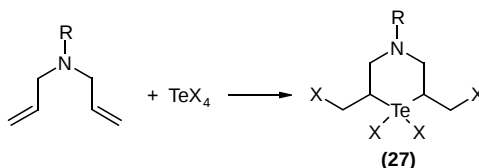
X = O (**a**), Se (**b**), Te (**c**); Y = I, 4-MeC₆H₄SO₃

B. SIX-MEMBERED HETEROCYCLED

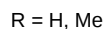
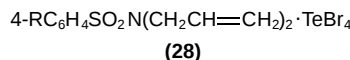
Three types of six-member Te,N-containing heterocycles are known thus far.

1. 1-Aza-4-Telluracyclohexane Derivatives

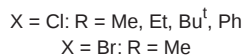
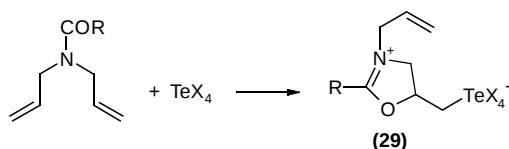
1,1-Dihalogeno-2,5-dihalogenomethyl-1-aza-4-telluracyclohexanes **27** were obtained by the reaction of electrophilic addition of tellurium tetrahalides to diallylamines (78KGS1212) or their *N*-sulfonyl derivatives (89KGS564). The reaction proceeds regiospecifically as an anti-Markovnikov addition, yields of the products being in the wide range of 27–79%.



In some cases the adducts of $TeBr_4$ with diallylarylsulfonylamides **28** were isolated along with tellurazines **27** ($R = 4-MeC_6H_4SO_2: X = Br$) (89KGS564).



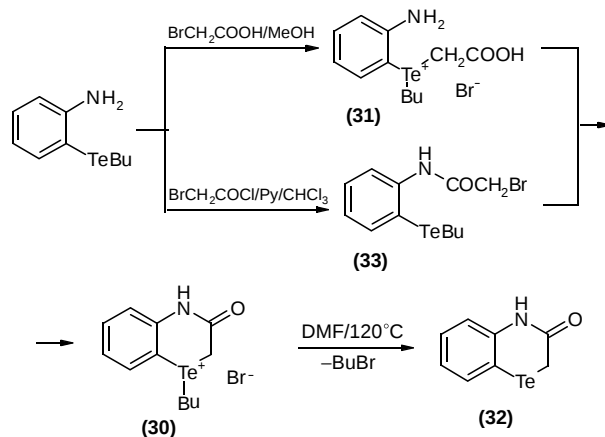
In contrast with diallylamines and their sulfonyl derivatives, *N*-acyldiallylamines react with tellurium tetrahalides to give zwitterionic oxazolines **29** containing five-coordinated tellurium (85T1607). Molecular and crystal structures of one of this type of compound (**29**, $R = Me$, $X = Cl$) were studied by X-rays (85T1607).

2. 2*H*-1,4-Benzotellurazin-3(4*H*)-One

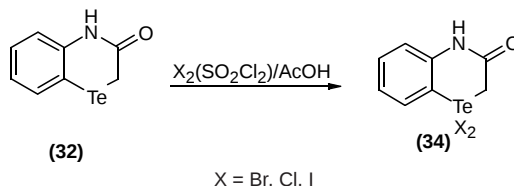
Two methods have been designed for the synthesis of this compound, which is the only Te,N-containing bicyclic six-member heterocycle known so far (93MI1).

A treatment of 2-butyltelluroaniline with an equimolar amount of bromoacetic acid results in spontaneous cyclization of the formed telluronium salt **31** to give 1-butylbenzotellurazinonium bromide **30**. That the alkylation occurs at the tellurium and not at the nitrogen atom of 2-butyltelluroaniline has been proved by the isolation of the methyl ester of **31** in 60% yield when the amine was coupled with methyl bromoacetate under the same reaction conditions. Elimination of butyl bromide from **30** readily occurs on heating of its DMF solution leading to 2H-1,4-benzotellurazin-3(4H)-one **32** in 90% yield.

When bromoacetyl chloride is used instead of bromoacetic acid, the anilide **33** is formed at the first stage. Its subsequent cyclization also leads to **32**. This approach to benzotellurazinone is similar to that developed for the synthesis of 2H-1,4-benzothiazin-3(4H)-ones (66CJC1247). Significantly, attempts to isolate the intermediate sulfonium salts analogous to **30** were unsuccessful.



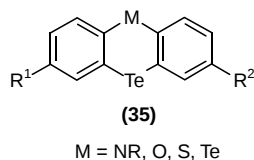
The heterocyclic telluride **32** is susceptible to oxidation–addition reactions and readily adds halogen atoms under a treatment with halogens or suluryl chloride (93MI1). Compounds **34** were obtained in almost quantitative yields.



3. Phenotellurazines

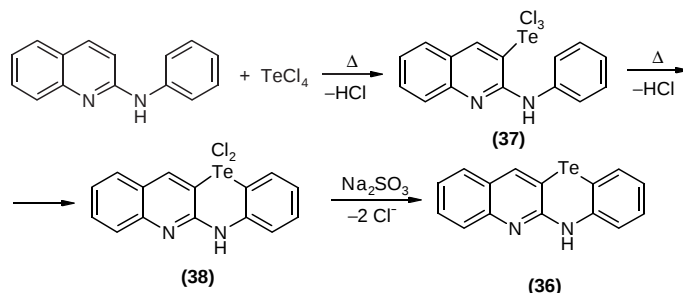
The tricyclic compounds **35** are among the most studied six-member Te-containing heterocycles with two heteroatoms in the ring. The first representative

of this group of compounds, phenoxatellurine (**35**, $M = O$, $R^1 = R^2 = H$) was prepared more than 70 years ago (26JCS223), but *N*-ethyl-2,8-dimethylphenotellurazine (**35**, $M = NEt$, $R^1 = R^2 = Me$) was only obtained 56 years later [82DOK(266)1164; 82KGS707]. Significant progress in the chemistry of phenotellurazines has been seen recently.



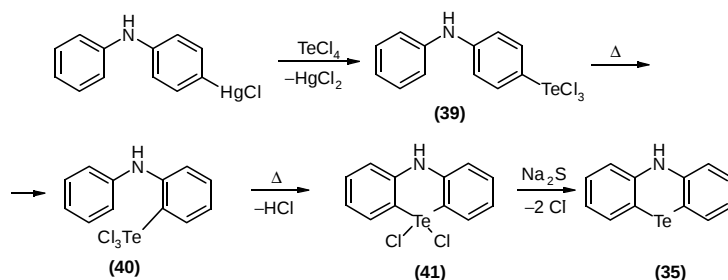
a. *Synthesis.* For the synthesis of phenotellurazines (**35**, $M = NR$), two approaches have been used: (1) cyclization of *o*-trichlorotellurodiarylamines and (2) coupling 2,2-dilithiodiarylamines with TeI_2 or $TeCl_4$.

i. ELECTROPHILIC CYCLIZATION OF *o*-TRICHLOROTELLURODIARYLAMINES. Owing to enhanced stability of aryltellurium trichlorides as compared to their sulfur and selenium analogues (95UK527), and because of the high electrophilicity of a $TeCl_3$ group, intramolecular cyclization of *o*-trichlorotellurodiarylamines is a convenient method for the preparation of phenotellurazines. The approach is well exemplified by the synthesis of 1*H*-dibenzo[*b,g*]-4-tellura-1,8-naphthyridine **36**. The 2-phenylamino-3-quinolytellurium trichloride **37** formed at the first stage of the reaction between 2-phenylaminoquinoline and $TeCl_4$ undergoes thermal ring closure to give the Te,Te -dichloride **38**. Subsequent reduction of **38** affords **36** (77MI1).

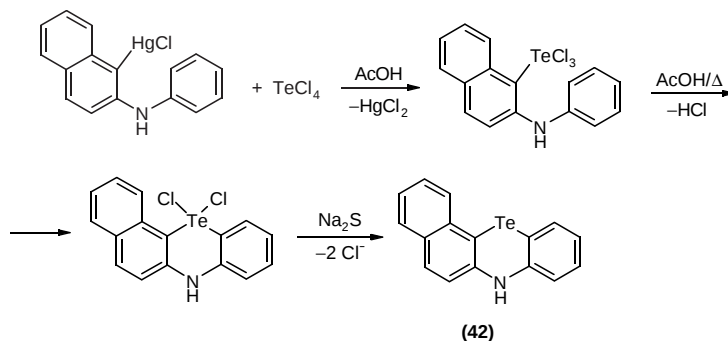


At the same time attempts to apply this approach to the synthesis of phenotellurazine and its *N*-methyl derivative using diphenylamine and *N*-methyldiphenylamine as starting materials failed [83JOM(251)223; 85KGS757]. The parent phenotellurazine **35** ($M = NH$, $R^1 = R^2 = H$) has been prepared in low yield (7%) by refluxing an acetic acid solution of 4-trichlorotellurodiphenylamine **39** (89H1007).

This compound apparently rearranges to its *o*-isomer **40** whose subsequent cyclization gives rise to 10,10-dichlorophenotellurazine **41**.

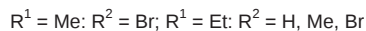
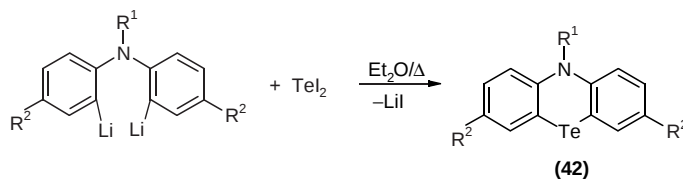


The same method was employed for the synthesis of benzo[*a*]phenotellurazine **42** and proven to be more efficient. The heterocycle **42** was obtained in 55% yield (89H1007). A possible explanation for the higher yield of **42** is that the transmetallation reaction in this particular case dominates the side formation of nonreactive complex of the amine with tellurium tetrachloride. There is no need for an additional step of the isomerization of the formed aryltellurim trichloride.

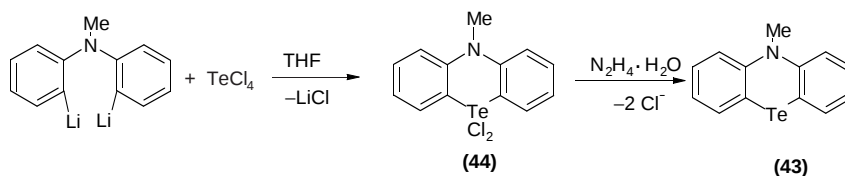


It is worth noting that cyclization reactions of *o*-trichlorotellurodiphenylamine **40** proceed under milder conditions (refluxing their acetic acid solutions) as compared to those required for the ring closure of *o*-trichlorotellurodiphenyloxide and the corresponding sulfide (heating at 200°C and 240–250°C, respectively) (26JCS223; 60T15).

ii. FROM 2,2-DILITHIODIARYLAMINES. A series of phenotellurazines **43** were prepared in 45–55% yields by coupling 2,2-dilithiodiarylamines with TeI_2 [82DOK (266)1164; 82KGS707; 85KGS757].

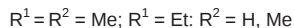
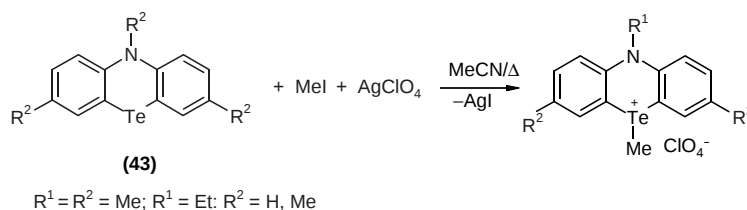


The use in this reaction of TeCl_4 instead of TeI_2 was less efficient. When treated with TeCl_4 , *N*-methyl-2,2-dilithiodiphenylamine afforded the dichloride **44** which was then reduced by hydrazine hydrate to give **43** in an overall yield of only 18% [82JOM(251)223].

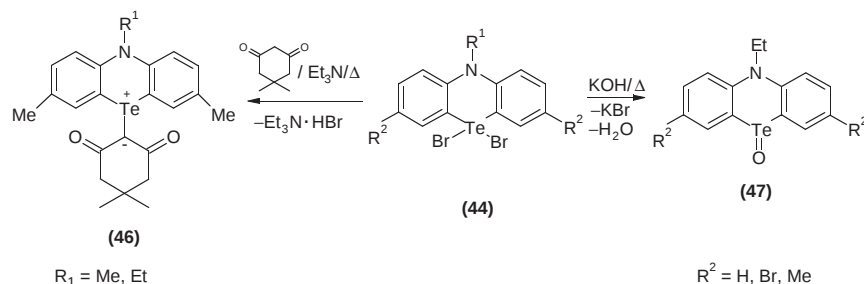


b. *Reactions.* Although three reaction centers of phenotellurazine (benzene rings, tellurium, and nitrogen) are conceivable, until now only reactions related to the former two centers were described in significant detail.

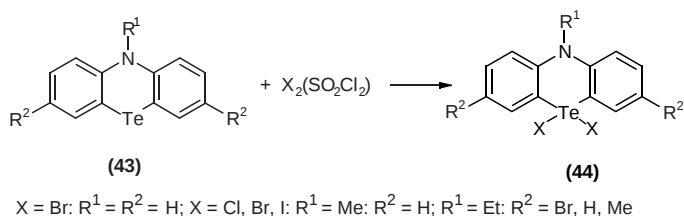
i. REACTIONS AT THE TELLURIUM ATOM. In common with acyclic diorganyl tellurides, phenotellurazines form telluronium salts under the action of alkylating agents. However, in contrast with dialkyl and alkylaryl tellurides (95MI1), addition of methyl iodide to the Te(II) center of phenotellurazines occurs only in the presence of an equivalent amount of silver perchlorate [82DOK(266)1164; 85KGS757].



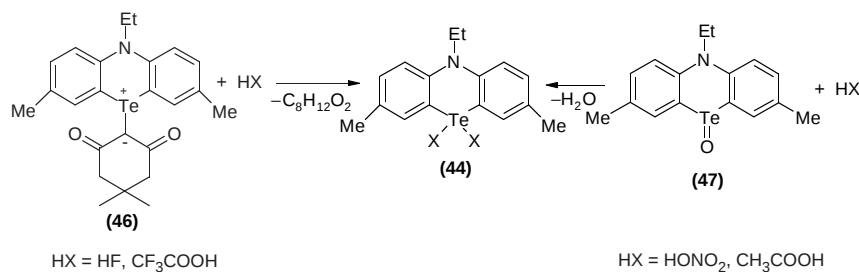
σ -Telluranes **44** were employed for the synthesis of other derivatives of phenotellurazines with a tricoordinated tellurium atom. The tellurium ylides **46** were obtained in high yields by coupling Te,Te -dibromophenotellurazines with dime-done in the presence of equivalent amounts of Et_3N . Alkaline hydrolysis of the dibromides leads to telluroxides **47** in 80–85% yield [82DOK(266)1164; 85KGS757].



In common with various other acyclic (95MI1) and cyclic dicoordinated tellurium compounds [86MI1; 86MI2; 93AHC(58)47; 95AHC(63)1], phenotellurazines are susceptible to oxidation–addition reactions giving rise to σ -telluranes **44**. Halogens add to phenotellurazines under very mild conditions (at room temperature or lower) to form **44** in high yields [82DOK(266)1164; 85KGS757; 89H1007]. The use of CuCl_2 or CuBr_2 as halogenating agents requires more severe reaction conditions (refluxing a solution of the components in aqueous acetone) (85KGS757).

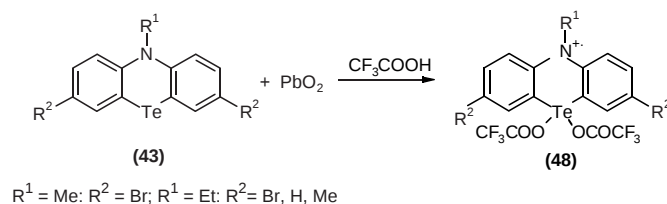


A series of σ -telluranes **44** with $\text{X} = \text{F, ONO}_2, \text{OCOMe}$, and OCOCF_3 were prepared by a treatment of the ylides **46** [82DOK(266)1164; 85KGS757] or the telluroxides **47** (85KGS757) with protic acids.

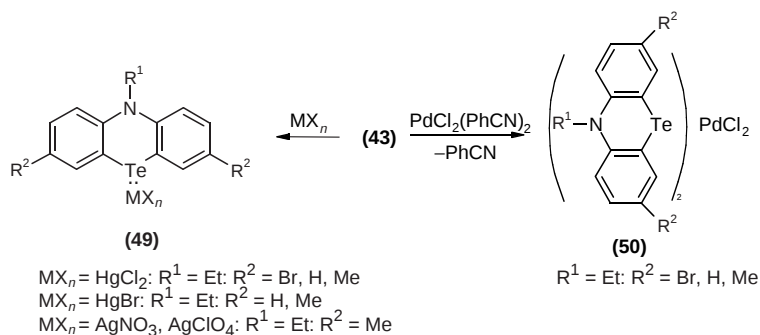


Phenotellurazine bis(trifluoroacetates) can be obtained by the oxidation of *N*-methylphenotellurazines with lead dioxide in trifluoroacetic acid solution. The

reaction involves intermediate formation of the radical cations of **48**, whose well resolved ESR spectra show significant delocalization of the unpaired electron (85KGS757).



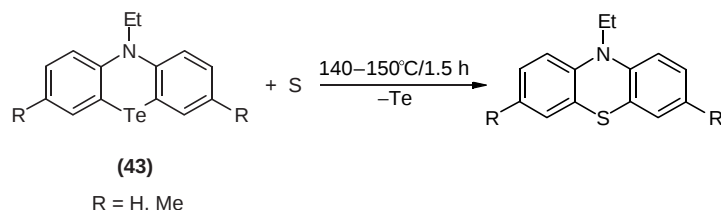
With soft Lewis acids, phenotellurazines **43** form molecular complexes in which the tellurium center serves as the ligating atom [82DOK(266)1164; 85KGS757]. The 1:1 complexes **49** are formed with salts of Hg(II) and Ag(I). PdCl_2 reacts with **43** to give 1:2 complexes **50**. Whereas the Hg(II) and Pd(II) complexes are stable on exposure to air, the Ag(I) complexes rapidly decompose when isolated from their solutions.



N-Ethyl-2,8-dimethylphenotellurazine **50** reacts with Rh(I) carbonyl complex $[\text{Rh}(\text{CO})_2\text{Cl}]_2$ [82DOK(266)1164; 84MI1] and $\text{Rh}[\text{Oxq}(\text{C}_8\text{H}_{14})\text{CO}]$ (where Oxq is 8-hydroxyquinoline and C_8H_{14} is cyclooctene) (84MI1) to give $\text{Rh}[\text{L}_2\text{COCl}]$ **51** and $\text{Rh}[\text{Oxq}(\text{L})\text{CO}]$ **52**, respectively. The vibration frequencies of the CO ligands in these and similar Rh(I) complexes were used for estimation of the strength of the Rh–chalcogen donor–acceptor bonds. The ν_{CO} frequency in **51** (1968 cm^{-1}) is greater than that in the similar Rh(I) complex with diethyl telluride (1955 cm^{-1}), which justifies weaker donor ability of the tellurium centers of phenotellurazines as compared with dialkyl tellurides. On the other hand, the ν_{CO} vibration frequencies in the phenothiazine analogue of the complex **51** (1993 cm^{-1} , 1996 cm^{-1}) are greater than in **51**, which fact was interpreted as a result of the relative weakness of the Rh–S donor–acceptor bond. When treated with phosphines (Ph_3P , Bu_3P), complexes **51** and **52** undergo ligand exchange reactions eliminating

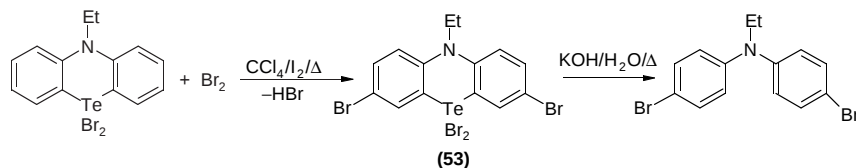
phenotellurazine ligands (84MI1). One may assume, therefore, that the Rh—P donor–acceptor bonds are stronger than the Rh—Te bonds in these complexes.

As in phenoxatellurines, phenothiatellurine [95AHC(63)1] and benzotellurazoles (89KGS757), 10-alkylphenotellurazines **43** react with elemental sulfur at elevated temperature by replacing the ring tellurium atom by sulfur (85KGS757). The yield of this reaction is about 50%.

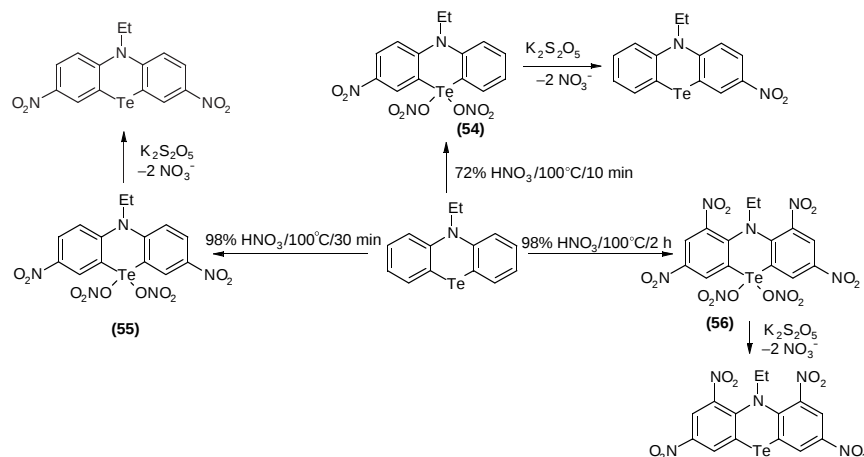


ii. ELECTROPHILIC SUBSTITUTION REACTIONS. Bromination and nitration of *N*-alkylphenotellurazines is accompanied by faster reactions which form Te,Te-dibromides and Te,Te-dinitrates, respectively (85KGS757). The TeX_2 ($\text{X} = \text{Br}, \text{ONO}_2$) groups are strongly electron-withdrawing, whereas the NR groups possess strong electron-releasing ability. The correlated electronic effects direct electrophilic substitution reactions at positions 2(8) and 4(6) of the benzene rings.

In the presence of catalytic amounts of iodine, bromination of *N*-ethyl-phenotellurazine with bromine results in *N*-ethyl-2,8,10,10-tetrabromophenotellurazine **53**. Structure **53** was proved by its synthesis by bromination of *N*-ethyl-2,8-dibromophenotellurazine and also by alkaline hydrolysis of **53** into 4,4-dibromo-*N*-ethyldiphenylamine.



Depending on the origin of the nitrating agent and reaction conditions (time, temperature, concentration of HNO_3), nitration of *N*-ethylphenotellurazine leads to Te,Te-dinitrates of mono-**54**, di-**55**, and tetranitro-*N*-ethylphenotellurazines **56**. Reduction of the latter with aqueous $\text{K}_2\text{S}_2\text{O}_5$ gives rise to the corresponding nitro-*N*-ethylphenotellurazines (89KGS989).



c. *Molecular and Crystal Structure of N-Ethyl-2,8-Dimethylphenotellurazine.* X-Ray diffraction (85ZSK120) demonstrates that *N*-ethyl-2,8-dimethylphenotellurazine exists in a butterfly conformation with the dihedral angle of bending along the $Te \cdots N$ axis of 137.7° . This value indicates substantially greater planarization of this heterocycle as compared with telluranthrene ($Te \cdots Te$ angle is 124°) (81CSC1359) and telluroxanthene ($Te \cdots C$ angle is 129.6°) (81ZSK106); however, it is more bent than phenoxatellurine ($Te \cdots O$ angle is 145°) (73JHC527). The dihedral angle in phenotellurazine is approximately equal to that in its sulfur analogue ($S \cdots N$ angle is 135°) [75AX(B)1179] but less than in 2,8-dichlorophenoselenazine ($Se \cdots N$ angle is 146.5°) [74AX(B)1332]. The valence CTeC angle in *N*-ethyl-2,8-dimethylphenotellurazine (89.8°) is smaller than the angles CSeC in 2,8-dichlorophenoselenazine (95.4°) and CSC in *N*-ethyl-2,8-dimethylphenothiazine (97.4°).

Dipole moments and molar Kerr constants of phenotellurazines were measured (85ZOB846). The results suggest a significant shift of electron density from the benzene rings toward the heterocycle.

^{125}Te NMR resonance in phenotellurazine is observed at 409 ppm (89H1007). This value should be compared with ^{125}Te NMR chemical shifts of the tellurium nuclei in phenoxatellurine (424 ppm), telluroxanthene (512 ppm) (89H1007), and telluranthrene (888 ppm) [81JOM(212)141].

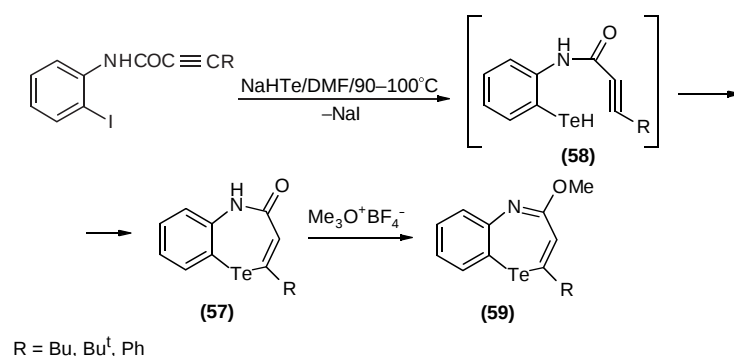
C. SEVEN-MEMBERED HETEROCYCLED

So far, only benzo- and dibenzo-fused telluroazepines are known.

1. 1,5-Benzotelluroazepine Derivatives

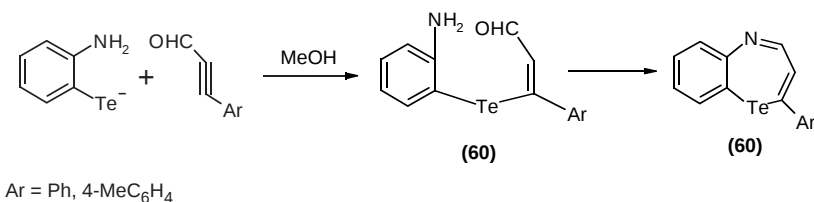
The first representatives of this group of compounds, 1,5-benzotelluroazepinones **57**, have been prepared in 17% yield by the reaction between 2-iodopropylanilides and NaHTe (98H631). The reaction proceeds, most probably, as nucleophilic substitution of the iodine, resulting in telluroles **58** and the subsequent nucleophilic addition of a hydrotelluride group to the triple bond. An alternative mechanism involving initial addition of NaTeH to the triple bond followed by the nucleophilic substitution of the iodine atom was ruled out because the anilides $\text{PhNHCOC}\equiv\text{CR}$ do not react with NaTeH under the conditions at which the heterocycles **57** were obtained. Neither of the adducts $\text{PhNHCOCH}=\text{C}(\text{R})\text{TeH}$ or $[\text{PhNHCOCH}=\text{C}(\text{R})\text{Te}]_2$ was isolated.

Upon treatment of telluroazepinones **57** with trimethyloxonium boron tetrafluoride, 4-methoxy-1,5-telluroazepines **59** were prepared in 40–45% yields (98H631). These compounds are thermally unstable.



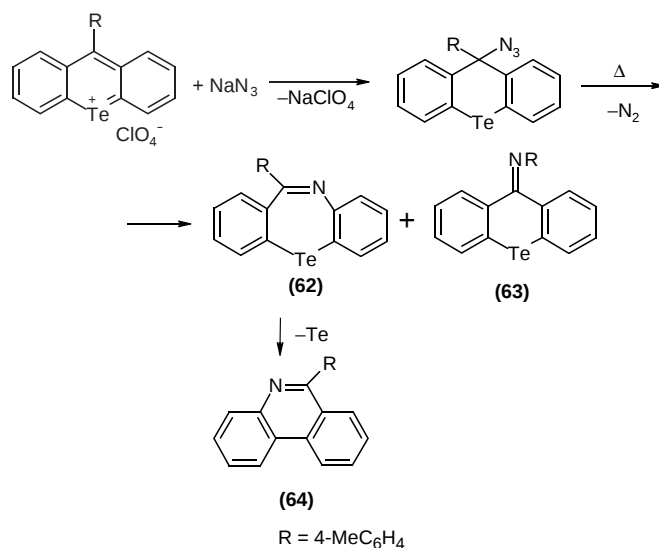
The initial 2-iodopropylanilides are rather inaccessible compounds. Their preparation requires a three-stage synthesis with diphenylphosphinic acid azide as the starting material.

Recently, a one-pot method for preparation of 2-aryl-1,5-benzotelluroazepines **60** has been developed based on the reaction of sodium 2-aminophenyltelluroate [from di(*o*-aminophenyl) ditelluride] with arylpropargyl aldehydes (99MI1). Considering the high affinity of supernucleophilic aryltelluroate anions to a triple bond, one may assume that at the first stage of this reaction arylvinyl tellurides **61** are formed. Cyclization of **61** spontaneously or on silica gel in a chromatographic column forms the heterocycles **60**.



2. Dibenzotelluroazepines

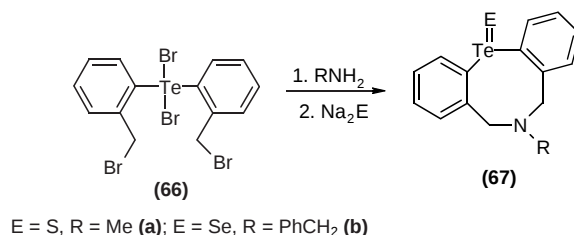
The first compound containing a telluroazepine ring, 11-(4-methylphenyl) dibenzo[*b,f*][1,4]telluroazepine **62** was obtained in 21% yield upon heating *p*-xylene solution of 9-azido-9-(4-methylphenyl)telluroxanthene at 130–140°C (87KGS279). Other products of this pyrolytic process are the imine **63** (32% yield) and phenanthridine **64** (21% yield). Formation of the latter implies extrusion of the tellurium atom from dibenzotelluroazepine **62**.



D. EIGHT-MEMBERED HETEROCYCLED

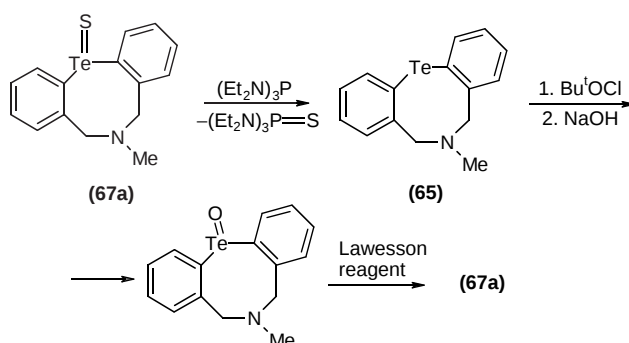
5*H*,7*H*-Dibenzo[*b,g*][1,5]telluroazocines **65** are the only representatives of Te, N-containing eight-membered heterocycles known thus far. Treatment of bis(2-bromomethylphenyl)tellurium dibromide **66** with methylamine followed by reduction of the reaction mixture with sodium sulfide gives, instead of the expected

N-methyl-5*H*,7*H*-dibenzo[*b,g*][1,5]telluroazocine **65**, its thiooxide **67a** (X = S, R = Me) (95JA6388). The same type of reaction with benzylamine and sodium selenide gives *N*-benzyl-5*H*,7*H*-dibenzo[*b,g*][1,5]telluroazocine selenoxide **67b** (X = Se, R = CH₂Ph). Reaction of the tellurium dibromide **66** with sodium sulfide carried out in the absence of the primary amines affords the eight-member Te,S-containing heterocycle 5*H*,7*H*-dibenzo[*b,g*][1,5]telluroazocine (92CL151).



The structure of the compound **67b** was determined by X-ray (95JA6388). The length of the Te=Se bond (2.445 Å) is 0.1 Å shorter than in bis(2-ethylcarboxy-phenyltellurenyl) selenide (81CSC1353), which points to a greater double bond character of this bond in the former compound. Selenoxide **67b** has a boat conformation. The length of the transannular Te...N bond (2.620 Å) is appreciably shorter than the corresponding van der Waals contact (3.70 Å). The intramolecular coordination Te←N bond thus formed contributes to the stabilization of the boat conformation of the compounds **67**.

On treatment with an equivalent amount of (Et₂N)₃P, thiotelluroxide **67a** is reduced to give telluroazocine **65** in almost quantitative yield, which can be converted into telluroxide **68** by the usual procedures for a diorganyl telluride to telluroxide transformation. It is worth noting that when treated with Lawesson reagent, the telluroxide **68** forms thiotelluroxide **67a**, whereas diaryl telluroxides are reduced by this reagent to diaryl tellurides.



III. Heterocycles with One Te Atom and Two N Atoms

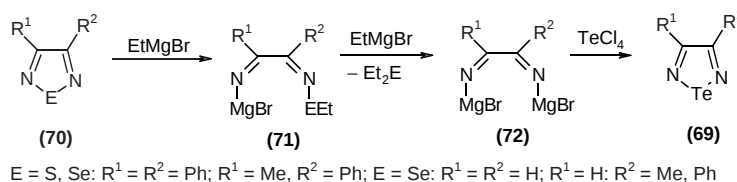
A. FIVE-MEMBERED HETEROCYCLED

Derivatives of three of the four possible structural types of these aromatic heterocycles have been synthesized.

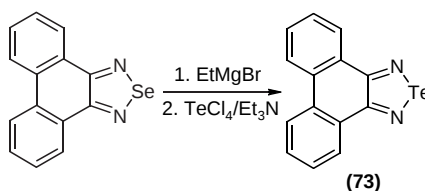
1. 1,2,5-Telluradiazoles

a. *Synthesis and Reactions.* The parent compound **69** and its derivatives were prepared by reaction of 1,2,5-thiadiazoles **70** (E = S) or 1,2,5-selenadiazoles **70** (E = Se) with ethylmagnesium bromide followed by treatment of the reaction mixtures with TeCl_4 and Et_3N (82S681).

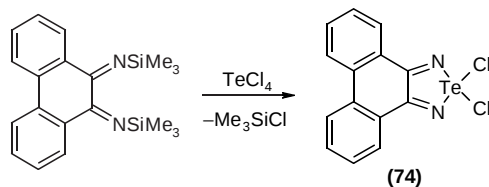
The yields of telluradiazoles **69** vary in the wide range of 6–79%, the highest being achieved with 1,2,5-selenadiazoles as the starting material. Only disubstituted 1,2,5-thiadiazoles afford correspondingly telluradiazoles in this reaction. The parent compound and various monosubstituted derivatives produce mixtures of not fully identified substances. The reaction mechanism involves initial nucleophilic attack of a Grignard reagent at the sulfur (selenium) center that results in cleavage of the heterocycle and formation of the intermediate **71**. Reaction of **71** with another molecule of EtMgBr leads to **72**, which gives telluradiazole when treated with TeCl_4 (82S681)



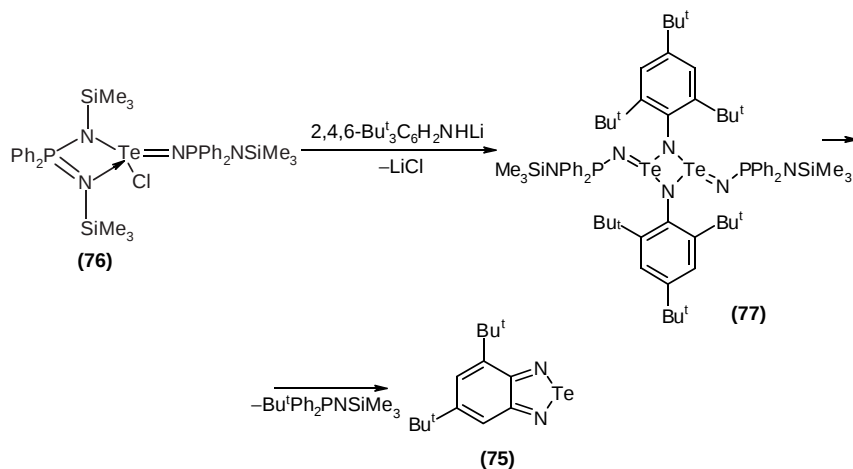
A similar approach provided a synthesis of phenanthro[9,10-*c*]1,2,5-telluradiazole **73** in 35% yield [87ZN(B)84].



The Te,Te-dichloro derivative **74** was prepared in 85% yield by heating phenanthro-9,10-bis(trimethylsilyl)imine with TeCl_4 (87MI1).



Because of the lower stability of Se(IV) compounds as compared with their Te(IV) analogues (95UK527), the reaction of the disilyldiimine with SeCl_4 ends up in phenanthro[9,10-*c*]-1,2,5-selenadiazole. 3,4-(2,4-Di-*tert*-butylbenzo)-1,2,5-telluradiazole **75** was obtained by a treatment of the tellurium diimide **76** with a lithium salt of tris(*tert*-butyl)aniline. Dimer **77** is an intermediate in this reaction (96IC9).



Reactions of telluradiazoles **69** and their analogues **73** and **75** were not studied in detail. Compounds **69** are stable with respect to air and moisture, but decompose when exposed to sunlight. Acidic hydrolysis of telluradiazoles gives dicarbonyl compounds, ammonium salts, Te , and H_2TeO_3 .

b. *Molecular and Crystal Structure of 1,2,5-Telluradiazole and Its Analogues.* 1,2,5-Telluradiazoles have anomalously high melting points and low solubilities in organic solvents as compared with their sulfur and selenium analogues (Table III).

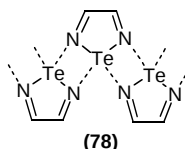
TABLE III
COMPARISON OF PROPERTIES OF
1,2,5-OXA(CHALCOGENO)DIAZOLES (Y = S, Se, Te; R = H)

Y	mp (°C)	UV ^a ($\lambda_{\max}$, ϵ)	NMR ¹ H (δ , ppm)
O	−28	— ^b	8.66
S	−50	253(10100)	8.89
Se	21	285(6600)	9.47
Te	185–188	336(3900)	10.81

^aSolvent, THF.

^bAbsorption in the far UV region.

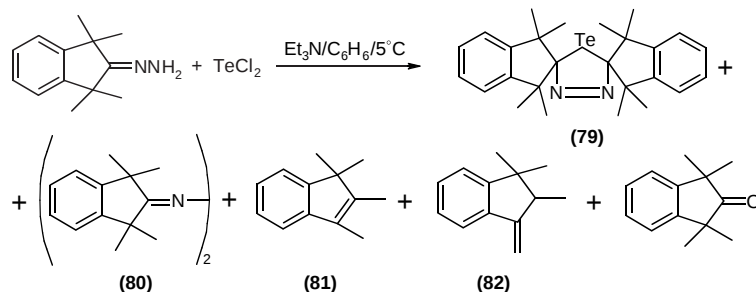
These peculiarities are caused by strong intermolecular coordination Te···N bonds ($l = 2.764 \text{ \AA}$) existing between molecules **69** in the crystal (84CSC653). Crystal packing is determined by formation of two such bonds by a tellurium atom of each molecule, which results in the polymeric structure **78**. The same type of bonds, their lengths being approximately 1 $\text{\AA}$ shorter than the Te···N van der Waals contact, were also found in crystalline **73** ($l = 2.833 \text{ \AA}$) [87ZN(B)84] and its benzo derivative **75** ($l = 2.628 \text{ \AA}$) (96IC9). The lengths of the covalent Te–N bonds in the heterocycles **69**, **73**, and **75** span the narrow range 2.004–2.023 $\text{\AA}$. The valence N–Te–N angles in these compounds are 82.5°, 84.3°, and 85.8°, respectively.



In contrast with **69**, molecules of benztelluradiazole **75** in the solid state are associated in dimers rather than polymeric chains. The bulky *t*-butyl groups of **75** prevent a higher degree of association and provide for good solubility of **75**, which readily crystallizes from its pentane solution.

2. Δ^3 -1,3,4-Telluradiazolines

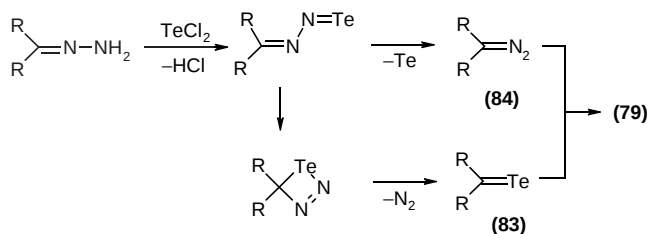
Δ^3 -1,3,4-Telluradiazolines **79** were obtained by coupling crowded hydrazones with TeCl_2 in the presence of triethylamine (93CL1047; 98MI1). Thus, telluradiazoline **79a** was prepared in 26% yield based on the hydrazone of 1,1,3,3-tetramethylindanone. The byproducts of this reaction are azine **80**, alkenes **81** and **82**, and 1,1,3,3-tetramethylindanone.



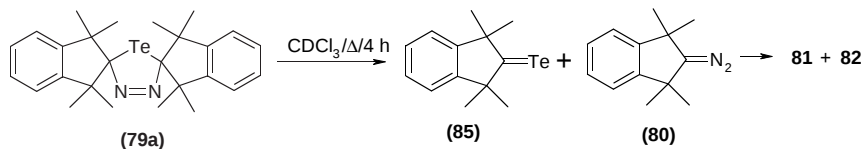
Telluradiazolines **79b** and **79c** were obtained in low yields of 10% and 11%, respectively (93CL1047; 98MI1).



The reaction mechanism involves a stage of 1,3-dipolar cycloaddition of the telluroketone **83** to the diazoketone **84** (93CL1047).

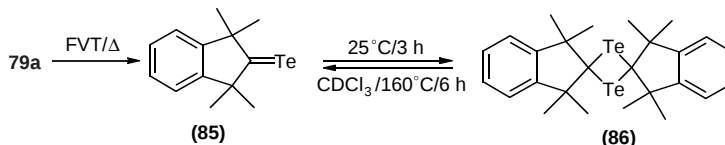


Telluradiazolines are thermally stable crystal compounds, but they are very sensitive to light. When exposed to daylight, telluradiazolines undergo rapid decomposition, even in the solid state. By heating degassed solutions of telluradiazoline **79a** in deuteriochloroform or benzene, telluroketone **85** and alkenes **81** and **82** are formed in almost quantitative yield (93JA7019).



Compound **85** was also prepared by flash-vacuum thermolysis of **79a** (250°C , 10^{-3} mm Hg) (97TL2501). At room temperature the telluroketone **85**, isolated as

green crystals, gradually converts to its dimer, 1,3-ditellurethane **86**. The reaction is reversible. On heating a solution of **86**, this compound dissociates to give the initial telluroketone in quantitative yield.



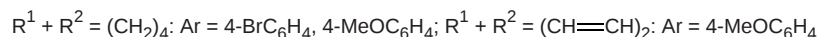
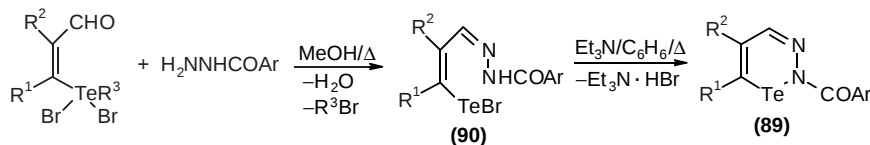
3. 1,2,3-Telluradiazole Derivatives

4,5-Benzo-1,2,3-telluradiazolium perchlorate **87** is the only compound known so far containing a 1,2,3-telluradiazole ring. It was obtained in 65% yield by reaction of the tellurenyl chloride **88** with silver perchlorate (88KGS1426).



B. SIX-MEMBERED HETEROCYCLED

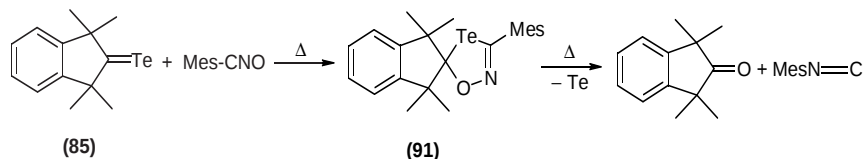
Six-membered heterocycles with one tellurium and two nitrogen atoms in the ring are represented by 2-aryyl derivatives of 1,2,3-telluradiazine and 5,6-benzo-1,2,3-telluradiazine **89**. For the synthesis of these compounds, *N*-aryylhydrazones of 2-bromotellurenylcyclohexenealdehyde and 2-bromotellurenylbenzaldehyde **90** were used as the starting materials (98ZOK959). Dehydrobromination of the hydrazones **90** occurs on treatment with triethylamine and gives the heterocycles **89** in about 80% yields.



IV. Heterocycles Containing Other Elements in the Ring along with Te and N Atoms

A. 1,4,2-OxATELLURAZOLES

The only known representative of this type of compound, **91**, was prepared by 1,3-dipolar addition of mesityl nitrile oxide to telluroketone **85** (93JA7019; 94MI1). The reaction proceeds smoothly on heating equimolar amounts of the reactants at 80°C, giving rise to **91** in 70% yield. The heterocycle is a thermally unstable and light-sensitive compound. Thermolysis of a deuteriochloroform solution of **91** at 60–90°C in a sealed ampule affords 1,1,3,3-tetramethylindanone and mesityl isonitrile (94MI1).

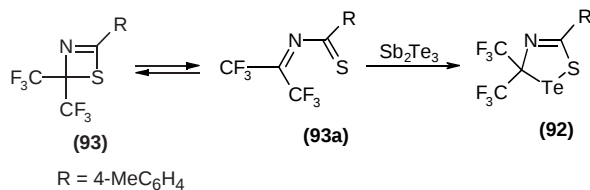


Sulfur and selenium analogues of **91** are also susceptible of this type of rearrangement. Its rate is defined by the strength of the C–chalcogen bonds in **91** and analogues and was found to increase in the order of S < Se < Te (94MI1).

The structure of **91** was studied by X-ray (94MI1). The five-member heterocycle is planar. The valence angle (79.4°) has the lowest value for all known five-member tellurium-containing heterocycles.

B. Δ^4 -1,2,4-ThiotELLURAZOLINES

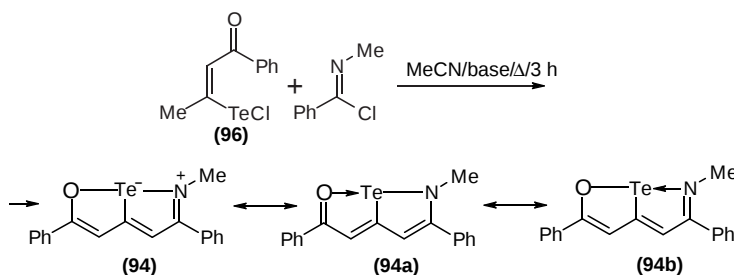
3,3-Di(trifluoromethyl)-5-(4-methylphenyl)- Δ^4 -1,2,4-thiotellurazoline **92** was obtained in 15% yield on heating 2*H*-1,3-thiazete with Sb₂Te₃ (79CC80). In₂Te₃ can be used in this reaction instead of Sb₂Te₃, but the yield of **92** decreases.



The compound **92** is extremely light-sensitive. Under the action of phosphites or compounds with multiple bonds, it readily eliminates a tellurium atom and thus may be considered as a synthetic equivalent of the heterodiene **93a**.

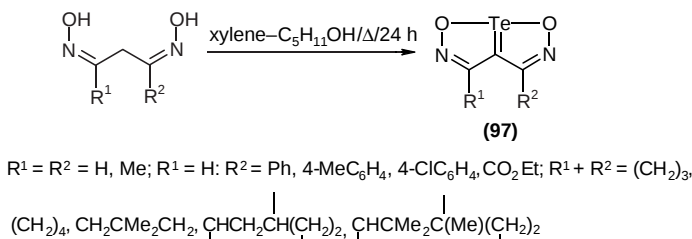
C. 1-AZA-6-OXA-6a-TELLURAPENTALENES

This group of compounds is represented by *N*-methyl-2,5-diphenyl-1-aza-6-oxa-6a-tellurapentalene **94** which was prepared in low yield (11%) by coupling an imidoyl chloride **95** with a tellurenyl chloride **96** in acetonitrile solution containing 2,6-lutidine (87MI2).

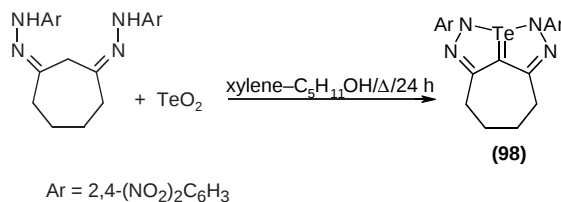


D. 2,5-DIAZA-1,6-DIOXA-6a-TELLURAPENTALENES AND THEIR AZA ANALOGUES

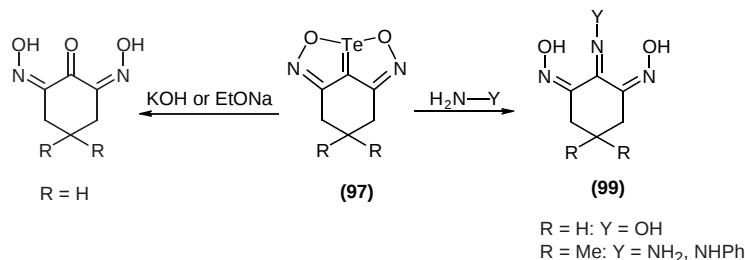
2,5-Diaza-1,6-dioxa-6a-tellurapentalenes **97** were obtained in 4–60% yields by oxidation of 1,3-diketone dioximes with tellurium dioxide (79BSF199).



The aza analogue of **97**, 1,2,5,6-tetraaza-6a-tellurapentalene **98**, was isolated in very low yield (2%) as a product of reaction of TeO_2 with the dihydrazone of 1,3-cycloheptadione (79BSF205).



On treatment with sodium ethanolate or aqueous ethanolic KOH, 2,5-diaza-1,6-dioxa-6a-tellurapentalenes **97** eliminates a tellurium atom to give 1,2,3-triketone 1,3-dioximes (79BSF199). The elimination reaction occurs also when the compounds **97** are treated with hydroxylamine, hydrazine, or phenylhydrazine. The 1,2,3-triketone derivatives **99** are the products of this reaction (79BSF199).

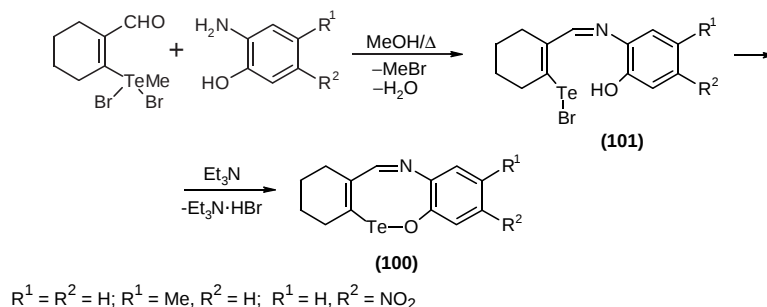


2,5-Diaza-1,6-dioxa-6a-tellurapentalenes **97** do not undergo electrophilic substitution reactions such as halogenation, nitration, or Friedel-Crafts and Vilsmaier reactions (79BSF199).

Mass spectra of 1,6-dioxa-2,5-diaza-6a-tellurapentalenes are given in (75JHC639). The ESCA spectra of the parent heterocycle and of its sulfur and selenium analogues were discussed (80JA1783).

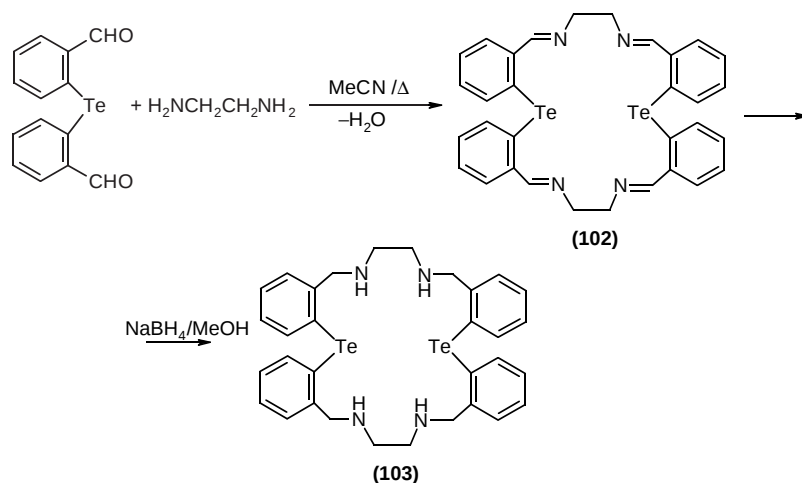
E. BENZO DERIVATIVES OF 1-OXA-2-TELLURA-6-AZACYCLOOCTA-3,5-DIENE

The method developed for preparation of telluradiazines **89** was applied to the synthesis of benzo derivatives of 1-oxa-2-tellura-6-azacycloocta-3,5-diene **100** (99UP2). It involves dehydrobromination of bromotellurenylvinylaldehydes **101**, obtained by condensation of 2-methyldibromotellurocyclohexenealdehyde with *o*-aminophenols. Under a treatment of benzene suspension of **101** with triethylamine followed by short-term refluxing the reaction mixture the heterocycles **100** were obtained in 71–87% yields.

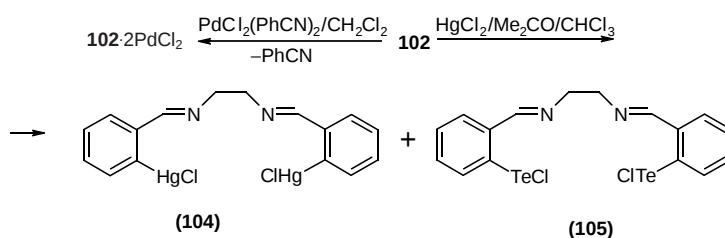


V. Tellurium- and Nitrogen-Containing Macrocycles

The tetraazamacrocycle **102** is the first known representative of tellurium-containing macrocycles. It was prepared in 80% yield by coupling bis-(*o*-formylphenyl) telluride (81KGS121) with 1,2-diaminoethane [96JCS(D)1203]. Reduction of **102** with NaBH₄ gives rise to the tetraazamacrocyclic compound **103** [96JCS(D)1203].

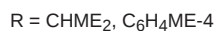
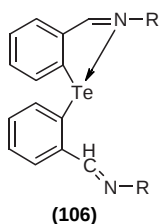


With PdCl₂, the macrocycle **102** forms a stable 1:2 complex. When treated with HgCl₂, **102** undergoes ring opening, resulting in formation of a mixture of **104** and **105** in 41% and 60% yields, respectively [96JCS(D)1203].



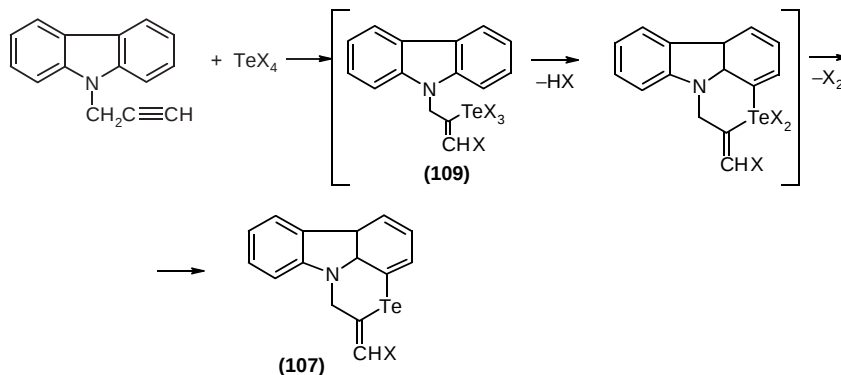
Molecular and crystal structures of the macroheterocycle **102** were studied by X-ray [96JCS(D)1203]. As for bis-imines of di(*o*-formylphenyl) telluride **106**, [89MI1; 91JOM(402)331] only one of two potentially possible intramolecular coordination N→Te bonds exists in a molecule of the macrocycle **102**, which, in

accordance with N-X-L nomenclature (82JA7753), should therefore be assigned to 10-Te-3 telluranes. The length of the N→Te bond in **102** (2.701 Å) is approximately the same as that in **106**.

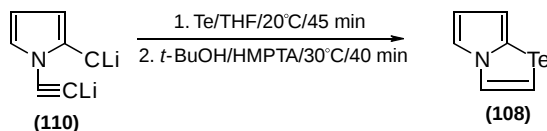


VI. Tellurium-Containing Heterocycles with N-Bridged Atoms

N-Bridged tellurium-containing heterocycles are exemplified by derivatives of 1,4-tellurazine[2,3-*gh*]carbazole **107** and heterocycle **108**. One-pot reaction of *N*-propargylcarbazole with tellurium tetrahalides under conditions of the two-phase tellurohalogenation (Section II,B,1) leads to halogenomethylene derivatives **107** (90KGS126). The reaction proceeds as the spontaneous electrophilic cyclization of initially formed tellurium trihalides **109**.



Heterocycle **108** was obtained in 45% yield by the reaction of dilithiated *N*-ethynylpyrrole **110** with elemental tellurium followed by a treatment of the reaction mixture with hexamethylphosphorus triamide [95JOM(493)271].



ACKNOWLEDGMENTS

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ADVANCES IN HETEROCYCLIC CHEMISTRY, VOL. 79

Photochemical Isomerization of Pentaatomic Heterocycles

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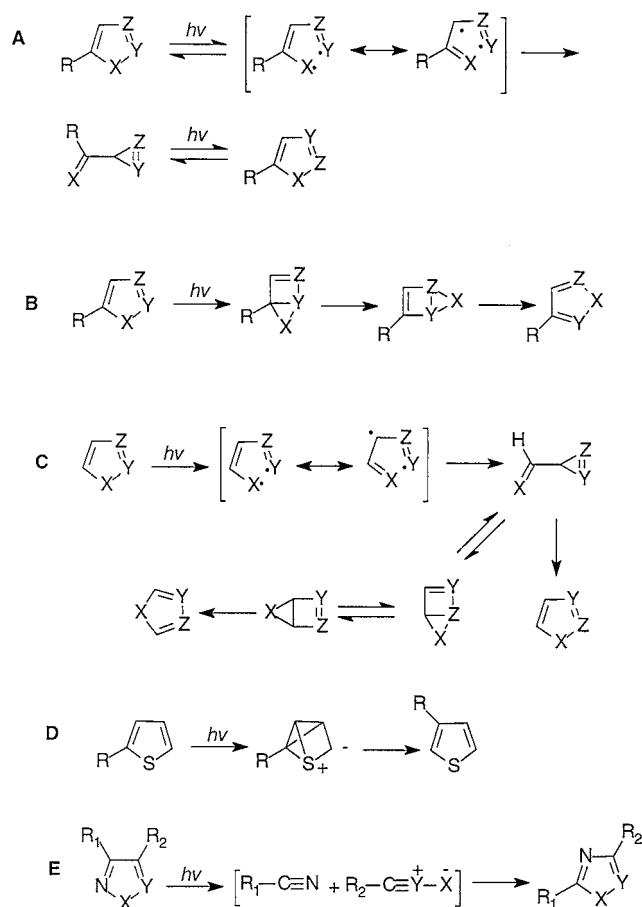
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I. Introduction

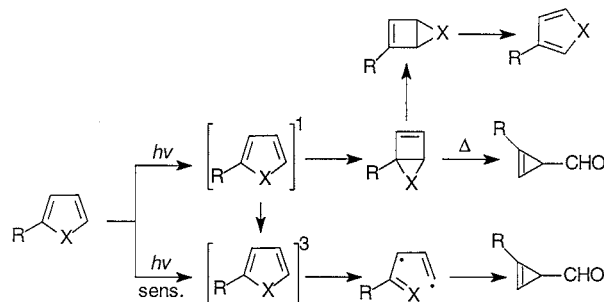
The photochemical isomerization of pentaatomic heterocyclic compounds has been the object of several reviews, starting with that of Lablache-Combiér in 1971 (71BSF679). After this work, reviews of the whole field (76MI123; 77CRV473;

80MI501) or part of it (85MI745; 95MI803; 95MI1063) appeared. Why a new review on this subject? In spite of the amount of work done in this field, the description of the photochemical behavior of pentaatomic heterocyclic compounds is confused. Five mechanisms can be invoked to justify the observed behaviors: (1) the ring contraction–ring expansion route (RCRE) (Scheme 1A); (2) the internal cyclization–isomerization route (ICI) (Scheme 1B); (3) the van Tamelen–Whitesides general mechanism (VTW) (Scheme 1C); (4) the zwitterion–tricycle route (ZT) (Scheme 1D); (5) the fragmentation–readdition route (FR) (Scheme 1E).

Recently, we reported our first attempts to reach a unified description of the photochemical isomerization of pentaatomic heterocyclic compounds [99H(50)1115; 99MI233]. If the first excited state of a molecule is populated, this molecule can



SCHEME 1



SCHEME 2

convert into the corresponding triplet state or into the corresponding Dewar isomer. The efficiency of these processes will depend on energetic factors. If the Dewar isomer is formed, an isomeric product is obtained. If the triplet state is formed, cleavage of the $X-C_\alpha$ bond can occur to give ring-opening products, decomposition products, or ring-contraction products. However, if the radical formed after the $X-C_\alpha$ cleavage shows a higher energy than the triplet state, the triplet state will not be able to give the biradical with high efficiency, and then it will be quenched in radiative and nonradiative processes. In this case, the Dewar isomer could be responsible for the isomerization reaction, but the isomerized product will probably be produced in very low quantum yields (Scheme 2).

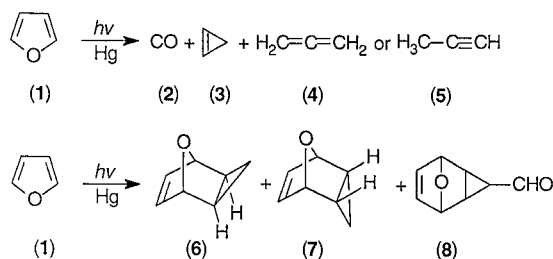
In this chapter we want to describe the conclusion of this work, giving the first unified description of the isomerization of these π -excessive compounds.

II. Furan

A. ISOMERIZATION OF FURAN

The photolysis of furan (**1**) in gas phase (5–150 mm Hg) in the presence of mercury at 254 nm gave carbon monoxide (**2**) and a fraction containing mainly cyclopropene (**3**) and a very small amount of allene (**4**) or methylacetylene (**5**) (Scheme 3) (67JA1758). Subsequently, carrying out the reaction at higher pressure of furan (0.2–1 atm), the same author showed that three new products were obtained (67JA4812; 68PAC65; 69PAC263). The first two compounds (**6** and **7**) were the Diels–Alder adducts obtained by the coupling of furan and cyclopropene, while the third **8** was the Diels–Alder adduct obtained by the coupling of furan and cyclopropene-3-carbaldehyde (Scheme 3). None of the reported products was observed on direct irradiation of furan in solution (cyclopentane as solvent).

These results were justified by assuming that the first photoproduct of the reaction was cyclopropene-3-carbaldehyde. This compound can be obtained via

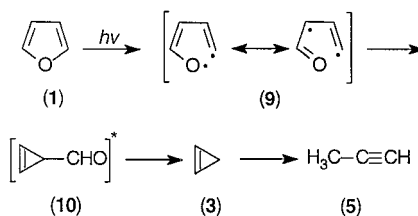


SCHEME 3

homolytic fission of an $\text{O}-\text{C}_\alpha$ bond to give the biradical **9**, which then collapses to **10** (Scheme 4). In this hypothesis, the compound **10** was obtained as excited species, allowing the further decomposition to cyclopropene. The thermal decomposition of cyclopropene to methylacetylene was known (60JA6375). The author did not consider the possibility that **10** could be excited via direct irradiation at 254 nm. Furthermore, when furan (137 mm Hg) was irradiated in the presence of mercury and methanol (37 mm Hg), the amount of cyclopropene and carbon monoxide in the reaction mixture decreased, while methyl 3-butenate was obtained as main product (67JA4812). This product could be generated from vinylketene through addition of methanol. The formation of vinylketene could be a new route to explain the formation of the ring-opening products in the photolysis of furan, but the author did not consider it.

The sensitized reaction on furan led to the formation of a π, π^* triplet state of **1**; then the relaxation of $\text{O}-\text{C}_\alpha$ bonds can induce, by crossing onto a π, σ^* energy sheet, the formation of a π, σ biradical **9** (74JA3486; 76T1729; 99MI1).

The direct irradiation of furan in gas phase gave carbon monoxide, methylacetylene, and allene, while cyclopropene was detected only in traces [68JCP(48)2185]. The direct irradiation leads to the formation of the excited singlet state, in which case cyclopropene is not the main product; then **3** is formed, starting from the excited triplet state. The direct flash photolysis of furan in gas phase gave mass fragments corresponding to C_4H^+ , C_4H_2^+ , C_4H_3^+ , and C_4H_4^+ together with the



SCHEME 4

formation of cyclopropene-3-carbaldehyde [87JP(37)273]. This behavior can be explained by assuming that Dewar furan is a precursor of the cyclopropene derivative. The irradiation at 214 nm of furan at 10 K gave a complex mixture of products in which carbon monoxide, methylacetylene, allene, vinylketene, formylallene, cyclopropene-3-carbaldehyde, and Dewar furan were detected (86JA1691). Furthermore, the irradiation of Dewar furan at 254 nm at 10 K afforded **10**.

Liquid-phase photolysis of furan at room temperature occurred in very low yields (1% conversion), giving a mixture of Diels–Alder adducts deriving from the reaction of cyclopropene-3-carbaldehyde and formylallene with furan (85JOC3034).

The existence of Dewar furan was confirmed by generation of this species using both chemical and photochemical methods. This way, the high reactivity of Dewar furan has been demonstrated to give the corresponding cyclopropenyl derivatives [82JA847; 84JCS(CC)1464; 84JCS(CC)1466; 85JA7176; 91AJC1275]; sometimes, the cyclopropenyl derivatives can be converted into the corresponding furan (78TL1015; 78TL1019). *Ab initio* calculations using STO-3G basis set concluded that Dewar furan has an energy $318.7 \text{ kJ mol}^{-1}$ higher than furan [81JST(85)303].

Semiempirical (PM3) and *ab initio* (6-31G** basis set) calculations are in agreement with the hypothesis described in Section I (99MI233; 000JOC2494). In the case of the sensitized reaction, when the excited triplet state is populated, only the formation of the radical intermediate is allowed. This intermediate can evolve to the corresponding cyclopropenyl derivative or to the decomposition products. In a previously reported mechanism the decomposition products resulted from the excited cyclopropenyl derivative. In our hypothesis the formation of both the decomposition products and the cyclopropenyl derivatives can be considered as competitive reactions.

In the case of the direct irradiation, the singlet excited state is populated, hence the formation of the Dewar furan is energetically possible (Fig. 1). This result is in agreement both with the evidence for the formation of the Dewar furan in the direct irradiation and with the formation of isomeric furans.

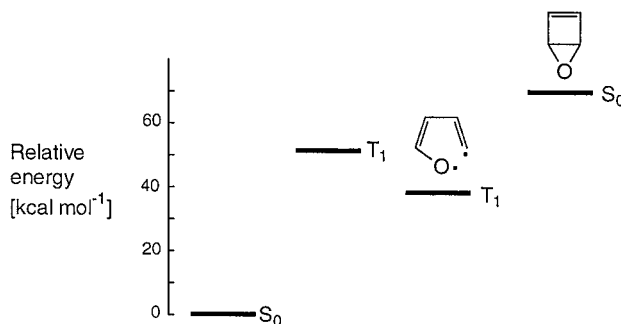
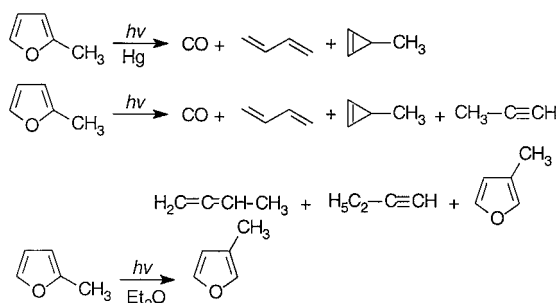


FIG. 1. Relative energy of the excited states of furan and of some reactive intermediates.



SCHEME 5

B. ISOMERIZATION OF METHYLFURANS

2-Methylfuran, irradiated in the presence of mercury vapor, gave carbon monoxide and a fraction containing 1,3-butadiene and 3-methylcyclopropene (45:55) (67JA1758). Subsequently, it was found that in both sensitized and direct photolysis of 2-methylfuran a more complex mixture of products was obtained, where 3-methylfuran was present (Scheme 5) (68JA2720; 70JPC574). 3-Methylfuran was the only product when 2-methylfuran was irradiated in diethyl ether (68JA2720).

In a theoretical work on this compound (SINDO 1), it was established that excitation takes place to the third excited singlet state. Then, two internal conversion processes cause the system to reach the minimum of the lowest excited singlet state. In this hypothesis, reaction can occur in both excited singlet and triplet state (87JA1044). Some previous authors claimed to predict the direction of the ring contraction. In their opinion, during the reaction the weakest bond is broken, and it is that between O—C₂ and O—C₅ (70JPC574; 87JA1044). Other results (6-31G**) did not confirm this hypothesis. We calculate the distance between O and C₂ in the triplet state of 2-methylfuran to be the same as that between O and C₅ (000JOC2494).

Also in this case the relative energy of all the possible intermediates involved in the photochemical isomerization was calculated (000JOC2494). The results are collected in Fig. 2. Also in this case the sensitized irradiation involves the formation of the biradicals. We have to note, however, that the fission of O—C_α bond in the triplet state of the molecule is not so favored as in furan. The process should be quite inefficient. The corresponding biradicals show the same energy as that in the triplet state. In this case, then, the formation of a biradical should depend on the activation energy.

Mercury-sensitized irradiation of 3-methylfuran gave 2-butyne, 1,2-pentadiene, 1-methylcyclopropene, 1-butyne, and 1,3-butadiene (Scheme 6) (68JA2720; 70JPC574). In the direct irradiation, 1-methylcyclopropene was obtained in lower yields while both 3-methylcyclopropene and 2-methylfuran appeared (Scheme 6) (70JPC574).

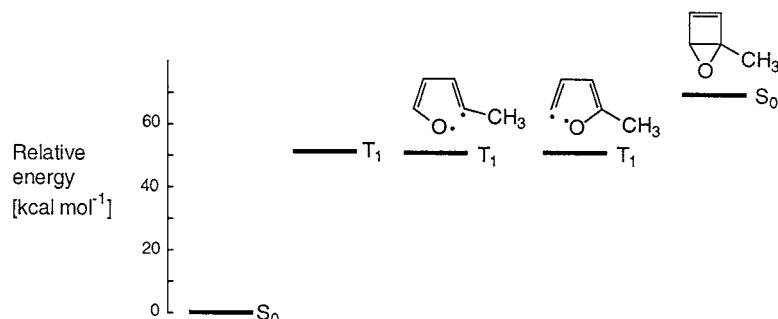


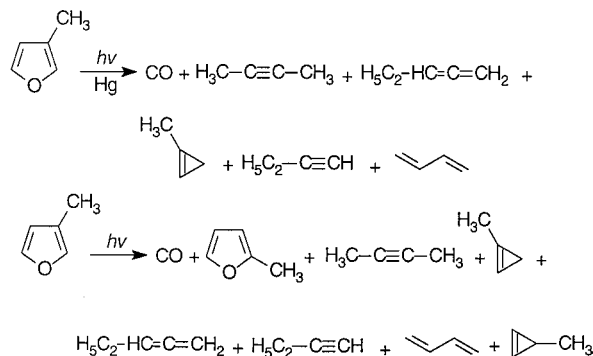
FIG. 2. Relative energy of the excited states of 2-methylfuran and of some reactive intermediates. [Reprinted with permission from M. D'Auria. *Ab initio study on the photochemical isomerization of furan derivatives*. *J. Org. Chem.* **65**, 2494–2498 (2000)].

C. PHOTOISOMERIZATION OF ALKYL FURANS

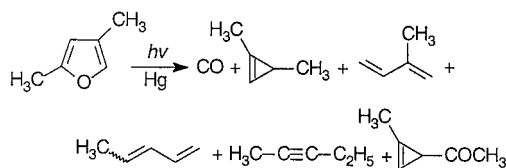
2,4-Dimethylfuran, in a sensitized reaction, gave 1,3-dimethylcyclopropene (the main product), isoprene, *cis*- and *trans*-1,3-pentadiene, 2-pentyne, and 1-methylcyclopropenyl methyl ketone (Scheme 7) (70JPC574); the ring contraction showed a high selectivity.

The irradiation of 2,5-dimethylfuran in the presence of mercury vapor gave a complex mixture of products. Carbon monoxide and propene were removed as gaseous products. Then, *cis*- and *trans*-1,3-pentadiene, isoprene, 1,3-dimethylcyclopropene, 2-pentyne, 2-ethyl-5-methylfuran, hexa-3,4-dien-2-one, 1-methyl-3-acetylcyclopropene, and 4-methylcyclopent-2-enone were obtained (Scheme 8) (68JA2720; 70JA1824). The most abundant product was the cyclopentenone **19**, the second was the 1,3-pentadiene **12**, while the third product was the cyclopropenyl derivative **18**.

By contrast, direct irradiation of 2,5-dimethylfuran gave 2,4-dimethylfuran as the only isolated product (68JA2720).



SCHEME 6

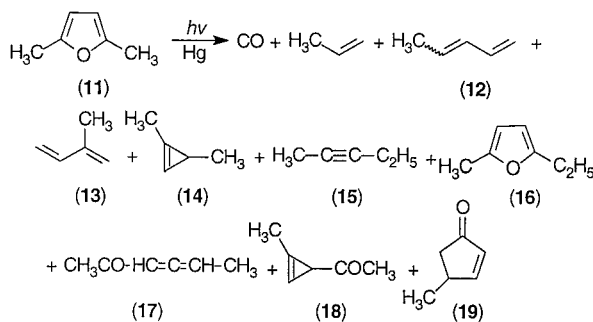


SCHEME 7

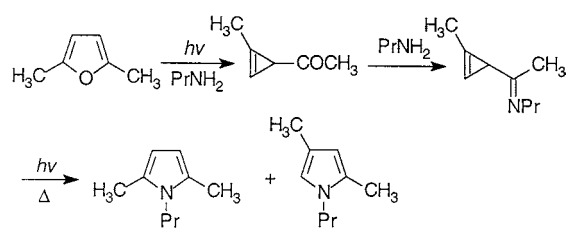
The irradiation of 2,5-dimethylfuran in propylamine led to the formation of the corresponding pyrrole. The reaction mechanism can be explained as depicted in Scheme 9 [71JCS(CC)891; 75T785]. It is noteworthy that the irradiation of **18** in butylamine gave a mixture of the analogous *N*-butylpyrroles, in agreement with the proposed mechanism (73CPB1516; 73T2747); however, the independently synthesized tetrakis(trifluoromethyl)cyclopropene derivative of the proposed imine could not be converted into the corresponding pyrrole (80JOC2968). The latter result is not in agreement with the mechanism depicted in the Scheme 9 and gives evidence for one involving the presence of a Dewar furan.

2,5-Di-*t*-butylfuran **20**, irradiated in pentane, gave the expected cyclopropenyl ketone **21**, 2,4-di-*t*-butylfuran (**22**), and an allene **23** in low yields (4, 9, and 9%, respectively) (Scheme 10) (68JA3894; 71JA6129). The irradiation of the cyclopropenyl ketone **21** gave 2,5-di-*t*-butylfuran (**20**) (8%), 2,4-di-*t*-butylfuran (**22**) (13%), and the allene **23** (19%), showing that it was an intermediate in the reaction mixture (Scheme 10). The irradiation of 2,4-di-*t*-butylfuran (**22**) gave the corresponding cyclopropenyl derivative **24** (Scheme 10). Finally, 2,3,5-tri-*t*-butylfuran (**25**) gave a cyclopentanone derivative **26** (95%) and a cyclopropenyl derivative **27** (5%) (Scheme 10).

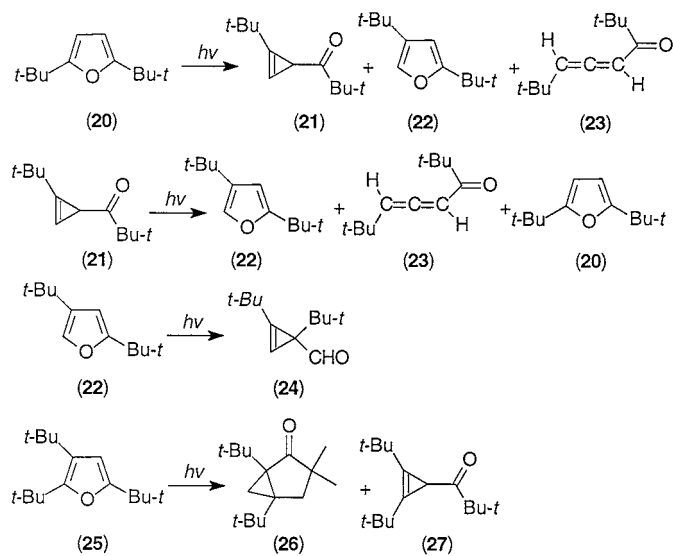
The formation of **26** can be explained on the basis of the mechanism depicted in the Scheme 11, where the irradiation of the cyclopropenyl derivatives **28** induces a radical reaction to give **26**.



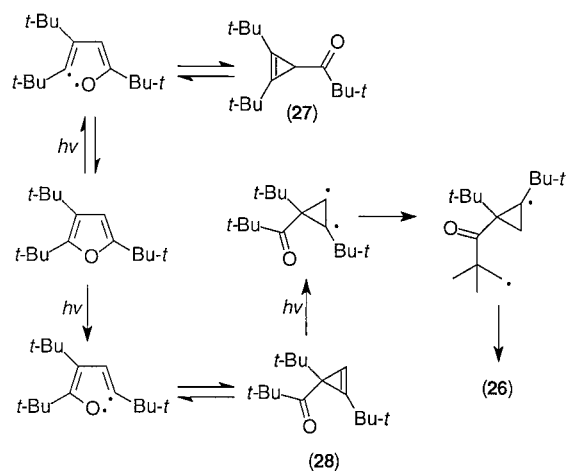
SCHEME 8



SCHEME 9



SCHEME 10



SCHEME 11

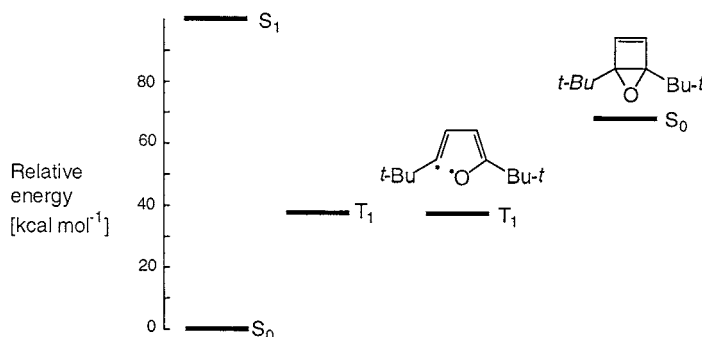


FIG. 3. Relative energy of the excited states of 2,5-di-*t*-butylfuran and of some reactive intermediates.

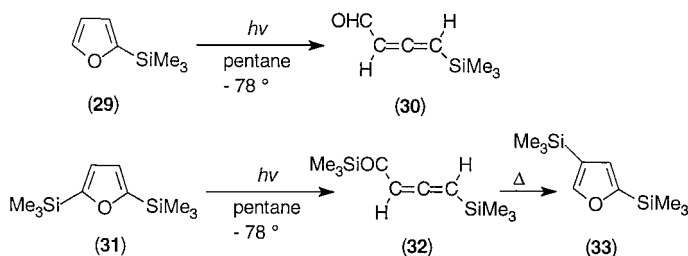
Also in this case theoretical calculations are in agreement with experimental results. In fact, the triplet state of **20** can be converted into the corresponding biradical to give the cyclopropenyl derivative (Fig. 3) (000JOC2494).

D. ISOMERIZATION OF TRIMETHYLSILYL-SUBSTITUTED FURANS

The irradiation of 2-trimethylsilylfuran (**29**) gave the corresponding ring-opening product **30** in 68% yield (Scheme 12) (83JA6316). Other trimethylsilyl derivatives showed the same behavior (Scheme 12). The allene **32**, obtained starting from the furan **31**, can be thermally converted into 2,4-ditrimethylsilylfuran (**33**).

The authors, assuming the intervention of a cyclopropenyl intermediate, explained the formation of this type of product.

The calculated relative energies of all the possible intermediates involved in the photochemical isomerization are collected in Fig. 4 (000JOC2494). In this case the irradiation can involve the excited singlet state, and then the formation of Dewar isomer is possible. As in 2-methylfuran, the fission of a O—C_α bond in the triplet state of the molecule is not so favored as in furan. The corresponding biradicals



SCHEME 12

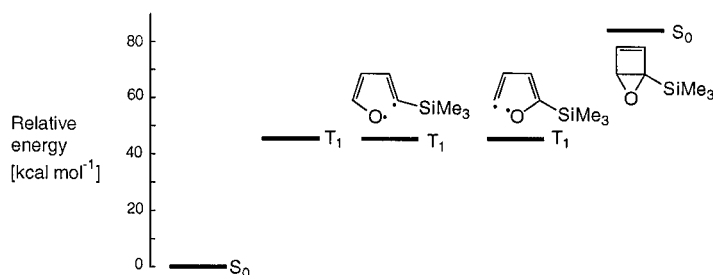
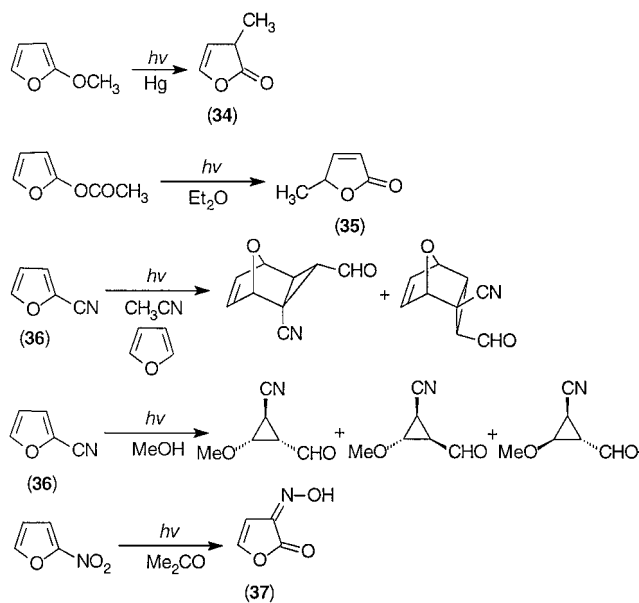


FIG. 4. Relative energy of the excited states of 2-trimethylsilylfuran and of some reactive intermediates. [Reprinted with permission from M. D'Auria. *Ab initio study on the photochemical isomerization of furan derivatives*. *J. Org. Chem.* **65**, 2494–2498 (2000)].

show the same energy as that in the triplet state. In this case, then, the formation of a biradical should depend on the activation energy.

E. ISOMERIZATION OF FURANS BEARING ELECTRON-DONATING OR ELECTRON-WITHDRAWING GROUPS

The sensitized irradiation of 2-methoxyfuran gave a product (**34**) deriving from a transposition reaction of the methyl group (Scheme 13) (69TL2767). The 5-methyl derivative **35** was obtained starting from 2-acetoxymethoxyfuran (Scheme 13).



SCHEME 13

Hiraoka and Srinivasan showed that direct irradiation of furan-2-carbaldehyde gave carbon monoxide, methylacetylene, and allene [68JCP(48)2185]. Subsequently, it was reported that furan, cyclopropene, and acetylene can also be obtained in the direct irradiation of the same molecule at 254 nm (76CJC3095). The sensitized irradiation (Hg) of furan-2-carbaldehyde gave carbon monoxide, methylacetylene, cyclopropene, and furan in one report [68JCP(48)2185] and carbon monoxide, methylacetylene, allene, acetylene, cyclopropene, and furan in another (76CJC3095). Gandini and coworkers found, in contrast with the previous work, that direct and sensitized reactions gave the same mixture of products where furan was present (76CJC3095). The only difference they found was that, in the sensitized reaction, a higher quantity of methylacetylene and a lower quantity of furan were obtained than in direct irradiation. The reaction starts from a vibrationally excited π, π^* triplet that decomposes to give CO and furan.

The irradiation of 2-cyanofuran (**36**) in the presence of furan gave the Diels–Alder adducts deriving from furan and the ring-contraction product of **36** (Scheme 13) [94JPP(A)(78)15]. The reaction of furan-2-carbonitrile in methanol allowed the authors to isolate a possible cyclopropenyl intermediate. In this case, the reaction gives a mixture of isomeric products containing methoxy substituents. These products clearly arise from a cyclopropenyl intermediate through a Michael addition of methanol (Scheme 13) [71JCS(CC)1610; 73T2955].

The relative energies of all the possible intermediates involved in the photochemical isomerization are collected in Fig. 5 (000JOC2494). In this case the irradiation can involve the excited singlet state, and thus the formation of the Dewar isomer is possible. As in furan, the fission of an $O-C_\alpha$ bond in the triplet state of

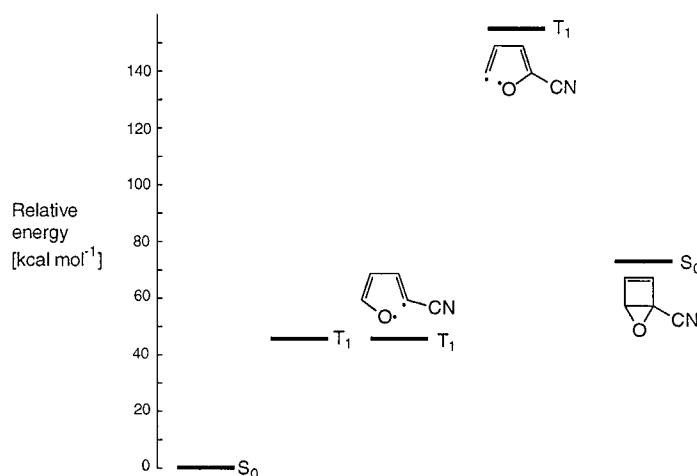
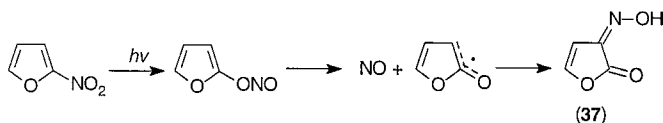


FIG. 5. Relative energy of the excited states of 2-cyanofuran and of some reactive intermediates.



SCHEME 14

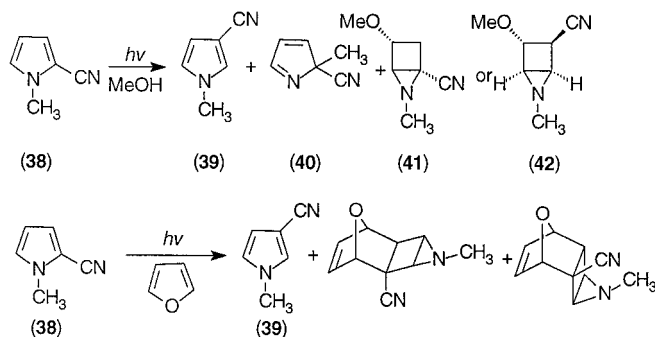
the molecule is favored if the O—C₂ bond is broken. By contrast, the fission of the O—C₅ bond leads to the formation of a high-energy species.

Finally, while the irradiation of 2-nitrofuran in 2-propanol did not give any interesting product (69BAP599), its irradiation in acetone gave the transposition product **37** (Scheme 13) [72JCS(P1)2527]. The observed reaction can be explained on the basis of the mechanism depicted in Scheme 14.

III. Pyrrole

The UV spectrum of the pyrrole in vapor phase showed absorptions at 211.0, 217.0, and 237.5 nm. The absorption at 237.5 nm was identified as relating to a π, π^* transition, and no decomposition of the starting material was observed [69JCP(51)2276]. On irradiation of pyrrole vapor at 214 nm at room temperature, propyne, allene, ethylene, acetylene, and propene were observed as decomposition products in the reaction mixture together with HCN (71JA3432). These authors identified only decomposition products; they did not find isomerization products, as in the vapor phase photochemistry of furan. Furthermore, vapor-phase photolysis of 2,5- and 2,4-dimethylpyrroles at 214 and 229 nm showed only hydrogen, methane, and ethane, while no product derived from ring opening was observed (72CJC1678). To confirm these experimental data, Dewar pyrrole, generated by photofragmentation of a suitable substrate, was shown to be very unstable in comparison to similar compounds obtained from furans or thiophenes [81JST(85)303; 96JCS(CC)1519]. Theoretical calculations on pyrrole are in agreement with the experimental results and showed that decomposition gives acetylene more likely than photoisomerization. Furthermore, they showed that strong electron-donor or -acceptor substituents could modify this behavior (86KGS173).

In 1970, Hiraoka reported that 2-cyanopyrrole, irradiated in methanol with a low-pressure mercury arc for 20 h, gave a mixture of 3-cyanopyrrole and pyrrole-2-carbaldehyde [70JCS(CC)1306]. 1-Methyl-2-cyanopyrrole (**38**) also gave this reaction (Scheme 15) [71JCS(CC)1610]. In this case, the author isolated the product of the isomerization **39**, the product of the shift in C-2 of the *N*-methyl group **40**, and a third product that was assumed to be derived from the addition of methanol to the Dewar pyrrole **41**. The reaction depends on the temperature used; in fact, no reaction occurred when the reaction was performed at -68°C . This result is in agreement with the presence of a thermal-activated step [78JCS(CC)131]. More



SCHEME 15

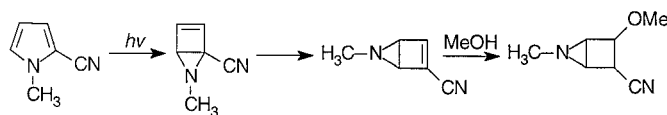
recently, the nature of this third product was re-evaluated and the structure **42** of Scheme 15 was proposed [78JCS(CC)131]. This structure was also confirmed by performing the reaction in the presence of furan. In this case, 4+2 photoadducts were isolated (Scheme 15).

All these data seem to be in agreement with a mechanism depicted in Scheme 16, where the thermal-activated step is the 1,2-sigmatropic shift between the Dewar pyrroles.

Similar results were obtained using methyl-substituted 2-cyanopyrroles [75JCS(CC)786]. All the results can be explained assuming the mechanism depicted in the Scheme 16. Only the by-product obtained in the irradiation of 5-methyl-2-cyanopyrrole cannot be explained. To explain this product, a subsequent 1,2-sigmatropic shift was postulated.

Recently, the 1,3-sigmatropic shift in 2-cyanopyrrole was studied by using the SINDO1 semiempirical method (90JOC2288). This study showed that the reaction occurred via a π, π^* transition and that some biradical intermediates are probably involved in the reaction.

When pyrrole is irradiated, only decomposition products were obtained. Theoretical data can fit this statement (Fig. 6). In fact, the direct irradiation populates the excited singlet state, which can be converted into the Dewar pyrrole or into the corresponding triplet state. Clearly, the intersystem crossing to the triplet state allows the system to reach the lowest energy state. The excited triplet state can give the biradical intermediate, and this intermediate can give either the decomposition



SCHEME 16

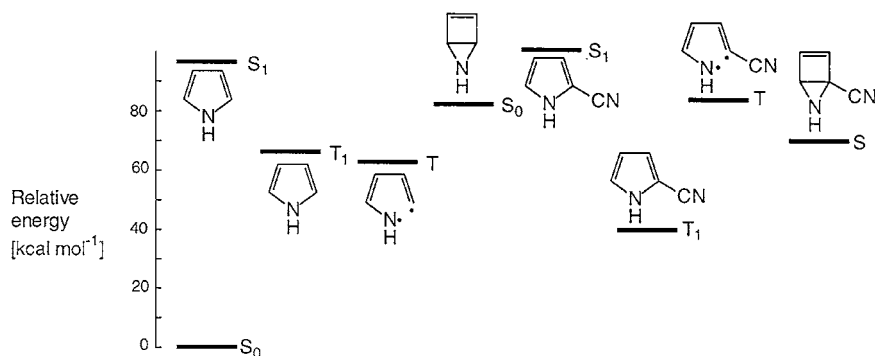


FIG. 6. Relative energy of the excited states of pyrrole and 2-cyanopyrrole and of some reactive intermediates. [Reprinted with permission from M. D'Auria. *Ab initio study on the photochemical isomerization of furan derivatives*. *J. Org. Chem.* **65**, 2494–2498 (2000)].

products only or the cyclopropenyl derivative that thermally evolves to give the decomposition products as reported above [99H(50)1115].

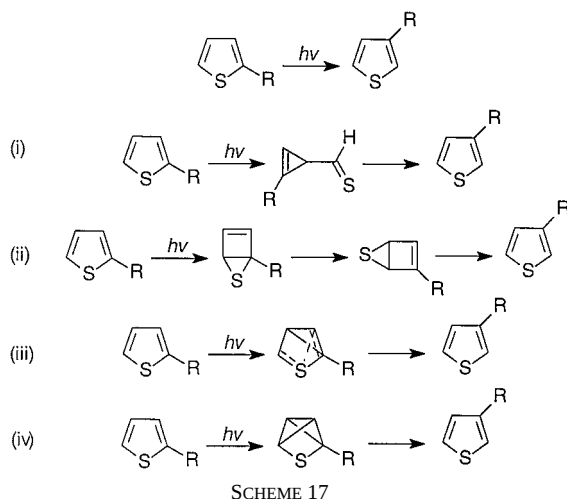
In contrast, when the irradiation is performed on 2-cyanopyrrole, the isomeric products are observed. In fact, in this case, the corresponding Dewar pyrrole shows a lower energy than in the previous case, allowing the formation of the isomeric products (Fig. 6). When 2-methylpyrrole is used as substrate, the formation of the triplet state is favored, but this triplet state cannot evolve through the formation of the biradical intermediate.

IV. Thiophene

The irradiation of the thiophene in gas phase yields ethylene, allene, methylacetylene, carbon disulfide, and vinylacetylene. No Dewar thiophene or cyclopropene derivatives were isolated (69CJC2965). The irradiation in liquid phase gave the Dewar thiophene which can be trapped as a Diels–Alder adduct with furan (85JA723). The Dewar thiophene and cyclopropene-3-thiocarbaldehyde can be obtained by irradiation in argon matrices at 10 K (86JA1691).

Wynberg has discovered the most interesting reaction in the photochemical reactivity of thienyl derivatives. The irradiation of 2-substituted thiophenes gave the corresponding 3-substituted derivatives (Scheme 17).

Several studies have been performed on the mechanism of this photoisomerization showing that the reaction takes place from the singlet excited state of the molecule (68TL5895), that the interchange between C₂ and C₃ occurs without the concomitant interchange between C₄ and C₅, and that the bonds between ring carbons and the substituents are not broken (67JA3501). Four mechanisms have been proposed (Scheme 17). Wynberg preferred (iii) (71ACR65; 72TL1429); more



recently, however, several studies showed that mechanism (ii) is the most probable [83TCA(63)55; 91JOC129].

Also in this case calculation results fit the experimental data (Fig. 7) [99H(50)1115]. In fact, the singlet excited state can evolve, giving the Dewar thiophene (and then isomeric thiophenes) or the corresponding excited triplet state. This triplet state cannot be converted into the biradical intermediate because this intermediate shows a higher energy than the triplet state, thus preventing the formation of the cyclopropenyl derivatives.

A. ISOMERIZATION OF ALKYLTHIOPHENES

Alkylthiophenes reacted to give the corresponding transposition products, but they showed low reactivity (70JOC2737). Better results were obtained using

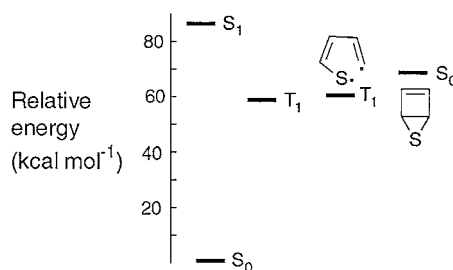
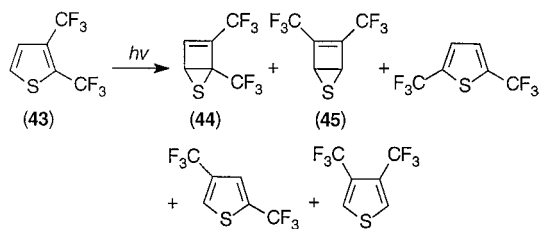


FIG. 7. Relative energy of the excited states of thiophene and of some reactive intermediates.



SCHEME 18

perfluoroalkyl derivatives. The tetrakis(trifluoromethyl) Dewar thiophene, isolated in 1970 by vapor-phase irradiation of the thiophene (70MI1), was the first Dewar isomer isolated in this series. The presence of trifluoromethyl groups induces fast formation and slow decomposition of strained systems. The slow decomposition may be partly due to the “siphoning” of vibrational energy into perfluoroalkyl groups (80T1161). The Dewar structure was accepted with some equivocation (71JA3432) until confirmed by a ¹⁹F NMR study (72CJC2821) and an X-ray structure of the tetramethylfuran adduct (74TL2941; 75CPB2772; 75TL1639). The irradiation of 2,3-di(trifluoromethyl)thiophene (43) gave a mixture of products where the authors found both isomeric thiophenes and an 8:1 mixture of Dewar isomers 44 and 45 (Scheme 18) [83H(20)174; 84TL1917]. The irradiation of the 2,5-di(trifluoromethyl)thiophene gave the corresponding 2,4 isomer, while 3,4-di(trifluoromethyl)thiophene gave the 2,4 isomer only in traces. 2,3,4-Tri(trifluoromethyl)thiophene gave the 2,3,5 isomer and a mixture of the corresponding Dewar isomers [83H(20)174].

As reported for the furan derivatives, thiophenes, when irradiated in the presence of an amine, gave the corresponding pyrroles [69JCS(CC)524; 71JCS(CC)891; 71T1059; 75T785]. The authors proposed the formation of a cyclopropenyl intermediate. Subsequently, however, a Dewar thiophene derivative, treated with aniline, gave the corresponding pyrrole, showing that it is probably the true intermediate in this reaction (80JOC2968).

B. ISOMERIZATION OF ARYLTHIOPHENES

Arylthiophenes were used as substrates in the photoisomerization described in Scheme 17 [65JA3998; 66JA5047; 66JCS(CC)203; 67JA3487; 67JA3495; 67JA3498]. The dithienyls gave this reaction efficiently, while 2-(2-pyridyl)thiophene and 2-(2-furyl)thiophene did not give this reaction in a reasonable yield (69JOC3175; 71JOC1011). Carbonyl and olefinic substituents inhibit the rearrangement [71ACR65; 74CJC1681; 75CJC1; 86G747; 86JCS(P1)1755; 87JCS(P1)1777; 87JOC5243; 89G381; 89JPP(A)(47)191; 94G195; 94JCS(P1)1245].

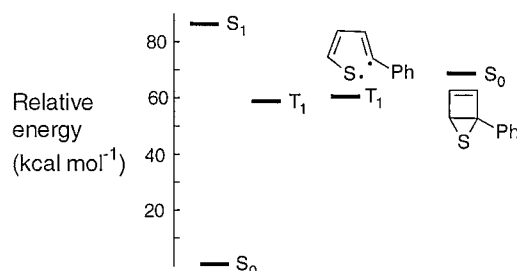


FIG. 8. Relative energy of the excited states of 2-phenylthiophene and of some reactive intermediates. [Reprinted with permission from M. D'Auria. *Ab initio study on the photochemical isomerization of furan derivatives*. *J. Org. Chem.* **65**, 2494–2498 (2000)].

The results of theoretical calculations on 2-phenylthiophene fit the experimental results (Fig. 8) [99H(50)1115]. In fact, in this case, the formation of the triplet state of 2-phenylthiophene cannot allow the formation of the biradical intermediate allowing the formation of the Dewar thiophene.

C. ISOMERIZATION OF CYANTHIOPHENES

The irradiation of 2- and 3-cyanothiophene gave interesting results in agreement with the scheme described above (Scheme 19). The photoisomerization reaction involved only the π, π^* excited singlet state and Dewar thiophenes were isolated when the reactions were carried out at -68°C and shown to be intermediates in the isomerization reactions [79JCS(CC)881; 79JCS(CC)966].

When 2-cyanothiophene is used in calculations, the results fit the experimental results (Fig. 9). In fact, the formation of the triplet state of 2-cyanothiophene cannot allow the formation of the biradical intermediate allowing the formation of the Dewar thiophene [99H(50)1115].

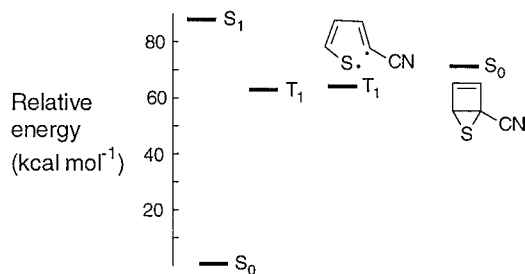
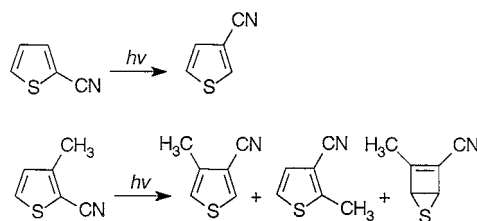


FIG. 9. Relative energy of the excited states of 2-cyanothiophene and of some reactive intermediates.



SCHEME 19

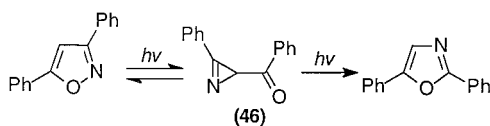
V. Isoxazole

The photorearrangements of isoxazoles are well known reactions. Thus 3,5-diphenylisoxazole, when irradiated at 254 nm, gave 2,5-diphenyloxazole via the corresponding azirine **46** (Scheme 20).

The azirine can be isolated; on irradiation at 254 nm, it gave the oxazole while irradiation at 334 nm gave the isoxazole [66JA1844; 66JCS(CC)689; 67JA6911; 72JA1199; 75JA4682; 81BCJ1293]. It is noteworthy that in this case the formation of Dewar isoxazoles cannot explain the obtained products.

The irradiation of 3,5-diphenylisoxazole in the presence of propylamine gave a mixture of 2,5-diphenyloxazole (20%) and *N*-propyl-2,5-diphenylimidazole (1%). The same distribution of the products was obtained starting from the azirine **46** (75T785).

In the photochemical isomerization of isoxazoles, we have evidence for the presence of the azirine as the intermediate of this reaction. The azirine is stable and it is the actual first photoproduct of the reaction, as in the reaction of *t*-butylfuran derivatives. The fact that it is able to interconvert both photochemically and thermally into the oxazole could be an accident. In the case of 3,5-diphenylisoxazole, the cleavage of the O—N bond should be nearly concerted with N—C4 bond formation (81BCJ1293); nevertheless, the formation of the biradical intermediate cannot be excluded. The results of calculations are in agreement with the formation of the azirine [99H(50)1115]. The excited singlet state can convert into a Dewar isomer or into the triplet state. The conversion into the triplet state is favored, allowing the formation of the biradical intermediate. The same results [99H(50)1115] were obtained using as substrate 3-phenyl-5-methylisoxazole (68ACR353) and 3,5-dimethylisoxazole (76MI1).



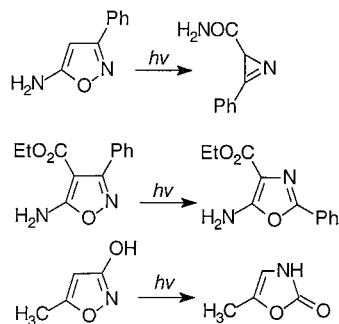
SCHEME 20

In the hypothesis described above the reaction should occur through the triplet state of the molecules involved, as reported in the furan. In contrast, some experimental data (72JA1199) and theoretical calculations (81BCJ1293) are in agreement with a singlet state mechanism. The use of sensitizers does not solve the question, because they were used in order to invoke both singlet (72JA1199) and triplet sensitization (67JA6911). However, the fact that 3,5-diphenyl-4-benzoylisoxazole, a compound with a high intersystem crossing quantum yield, does not react could support a singlet state process. However, calculations on this molecule showed that the triplet state can be formed but it cannot evolve to the high-energy triplet biradical intermediate [99H(50)1115]. In conclusion, there is no evidence for a singlet state process in this isomerization.

The capability of the reaction to occur in the first excited singlet state has been tested on 3,5-dimethylisoxazole (000UP1). The first excited singlet state shows a relative energy of 88 kcal mol^{-1} , and it can convert into a singlet biradical through O—N cleavage (at 39 kcal mol^{-1}), into the Dewar isomer (at 60 kcal mol^{-1}), or into the triplet excited state at 36 kcal mol^{-1} . The triplet state can be converted into the triplet biradical through O—N cleavage at 15 kcal mol^{-1} . Thus, the singlet state mechanism cannot be excluded.

A. ISOMERIZATION OF ISOXAZOLES BEARING ELECTRON-DONATING GROUPS

Some data were obtained from the photochemical isomerization of aminoisoxazoles. 5-Aminoisoxazoles gave the corresponding azirine (Scheme 21) [70JCS(C)1825]; when a 4-carboethoxy-substituted derivative was used, no azirine was isolated and the oxazole was the only product obtained (Scheme 21) (72CB748). The azirine intermediate was not observed upon irradiating 3-amino derivatives [91H(32)1765].



SCHEME 21

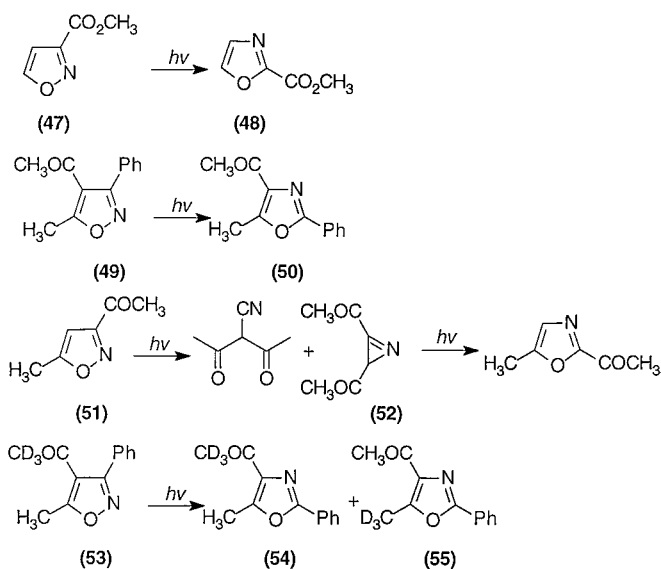
Calculations are in agreement with the formation of excited triplet state, and this intermediate can evolve to the formation of the azirine via the biradical intermediate [99H(50)1115].

The photoisomerization of 3-hydroxyisoxazoles gave the corresponding 2-oxazolones without the isolation of the azirine (Scheme 21) (67HCA137). Also in this case calculations are in agreement with the experimental results [99H(50)1115].

B. ISOMERIZATION OF ISOXAZOLES BEARING ELECTRON-WITHDRAWING GROUPS

The irradiation of 3-carbomethoxyisoxazole (**47**) gave the corresponding oxazole (**48**) in very low yields (5–8%) without the isolation of the corresponding azirine (Scheme 22) [71JCS(C)1196]. Also in this case calculations show that the energy of the triplet state allows the formation of the biradical intermediate and then of the azirine. However, the low yields of the conversion can be explained considering that the transformation of the biradical intermediate into the azirine is an endothermic reaction (Fig. 10) [99H(50)1115].

The irradiation of 3-phenyl-4-acetyl-5-methylisoxazole (**49**) gave the isomeric oxazole (**50**) (Scheme 22) (75JA6484; 76HCA2074). The reaction can involve the formation of the biradical intermediate starting from the triplet state, in agreement



SCHEME 22

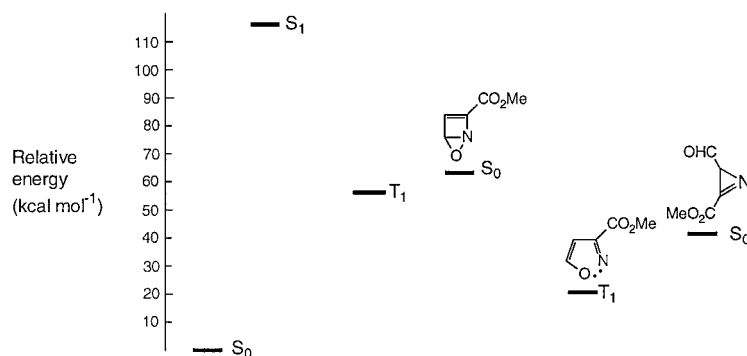
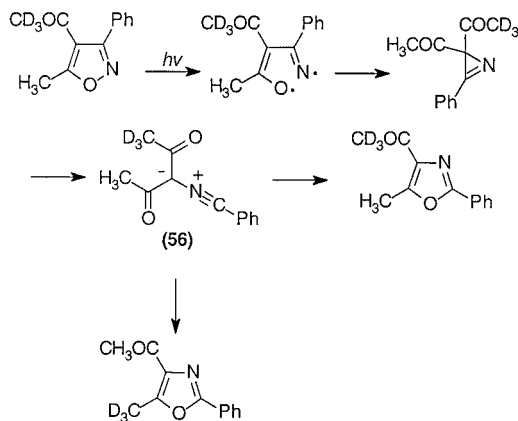


FIG. 10. Relative energy of the excited states of methyl isoxazole-3-carboxylate and of some reactive intermediates.

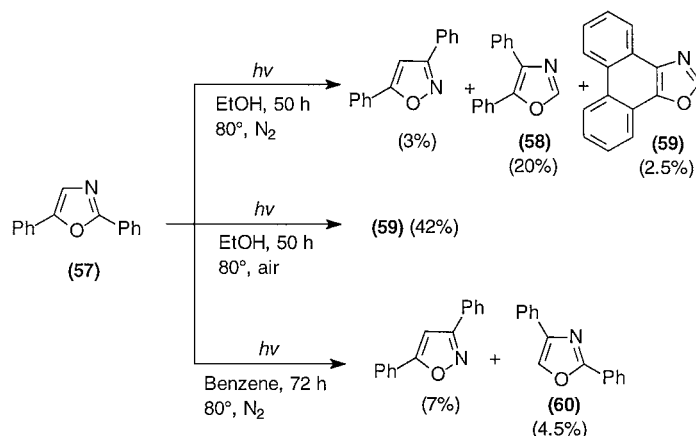
with the presence of the azirine derivative in the reaction mixture [99H(50)1115]. Using a very similar substrate, 3-acetyl-5-methylisoxazole (**51**), the formation of the azirine **52** was detected (Scheme 22) (87TL5797). When a deuterated substrate **53** was used a 1:1 mixture of oxazoles **54** and **55** were obtained (Scheme 22) (76HCA2074). This result was explained assuming the formation of the intermediate **56** after the cleavage of the azirine (Scheme 23).

VI. Oxazole

The photoisomerization of 2,5-diphenyloxazole (**57**) was first reported in 1969. 2,5-Diphenyloxazole, irradiated in ethanol at 80°C, gave three products, where the



SCHEME 23



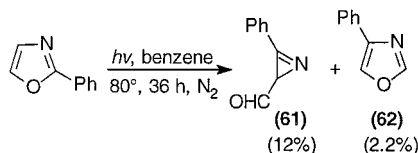
SCHEME 24

more important was 4,5-diphenyloxazole (**58**) (Scheme 24) [69TL2379; 77JCS (P1)239]. 4,5-Diphenyloxazole undergoes condensation to product **59**; in the presence of air, **59** was the only product obtained in good yield. In benzene, the overall yields are lower; however, the most noticeable change is the nature of the products. In fact, the main product was 3,5-diphenyloxazole in the presence of small amounts of 2,4-diphenyloxazole (**60**) (Scheme 24). The irradiation of **57** in the presence of propylamine gave **58** as the main product (75T785).

All the products are compatible with both ring contraction–ring expansion and ICI mechanisms.

2-Phenyloxazole, irradiated in benzene at 80°C, gave two products: **61**, derived from a ring contraction, and 4-phenyloxazole (**62**), obtained in very low yields (Scheme 25) [73JCS(CC)539; 77JCS(P1)239]. The authors thought that 4-phenyloxazole was ascribable to a Dewar intermediate.

4,5-Diphenyloxazole gave only the product of the condensation between the aromatic rings without transposition, while 2,4-diphenyloxazole did not react appreciably [77JCS(P1)239]. However, some important information about the mechanism was obtained when 2-phenyloxazole and 2-phenyl-5-methyloxazole were irradiated with a monochromatic light at 294 nm at 24°C. In both cases, the only



SCHEME 25

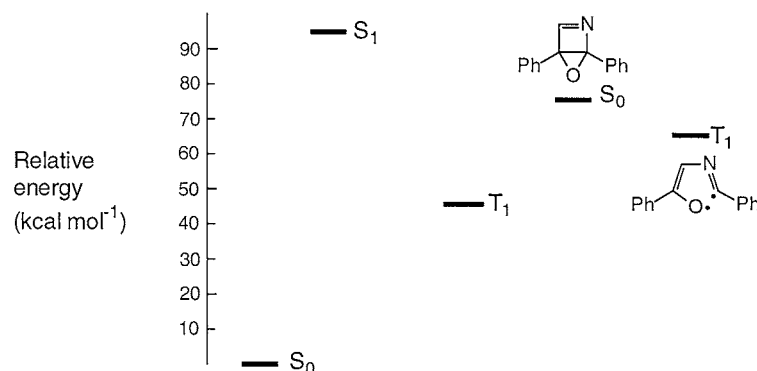


FIG. 11. Relative energy of the excited states of 2,5-diphenyloxazole and of some reactive intermediates.

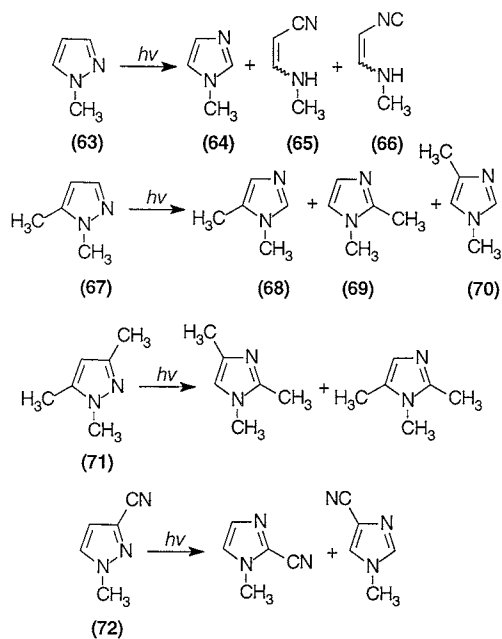
product observed was the corresponding azirine which did not convert into the corresponding isoxazole.

Concerning the mechanism of this reaction, calculations suggest that, in the presence of aromatic substituents on the oxazole ring, the ICI mechanism might be favored (83JA1753).

In agreement with the previously reported theoretical study, the results of semi-empirical calculations showed that the formation of the Dewar isomer is favored [99H(50)1115]. Probably, the observed formation of the azirine derives from a thermal isomerization of the first photoproduct, in line with that described in the case of furan and thiophene derivatives (Fig. 11).

VII. Pyrazole

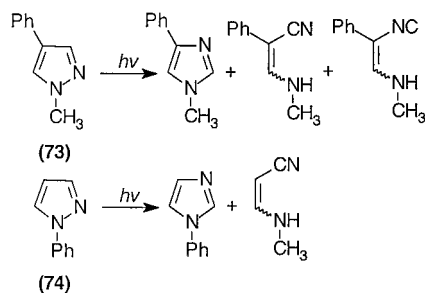
The irradiation of 1-methylpyrazole (**63**) gave 1-methylimidazole (**64**) (Scheme 26) (67HCA2244). Subsequently, a ring-opening product (**65**) was observed in the reaction mixture (91JOC6313), and, finally, **66** was detected in the residue after the reaction (97JOC8325). The irradiation of 1,5-dimethylpyrazole (**67**) gave a mixture of three products (**68–70**). While **69** and **70** can be obtained through both RCRE and ICI mechanisms, **68** can be obtained only via a ring contraction–ring expansion mechanism (Scheme 26) (91JOC6313; 92JOC1937). The formation of **70** is temperature-dependent: at 77 K it was not obtained (92JOC1937). Dewar pyrazole was invoked in the photoisomerization of 1,3,5-trimethylpyrazole (**71**), in the synthesis of **69** and **70** starting from **67**, and in the isomerization of cyanopyrazole (**72**) (Scheme 26) [67TL5315; 69T3287; 81JCS(CC)604; 91JOC6321]. A zwitterionic intermediate was also proposed (70PAC495).



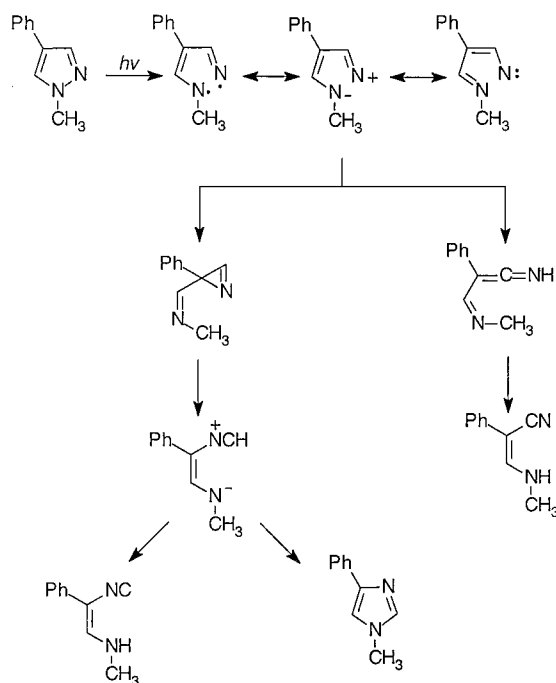
SCHEME 26

Ring-opening products were observed in the photoisomerization of 1-methyl-4-phenylpyrazole (73) (95JOC8138; 97JOC8325) and in the reaction of 1-phenylpyrazole (74) (Scheme 27) (93JA7645).

The mechanism depicted in Scheme 28 accounts for the formation of the ring-opening products (97JOC8325). It is a variation of the RCRE mechanism. By contrast, on irradiation 1-methyl-5-phenylpyrazole (75) gave **76**, **77**, and **78** (Scheme 29) (97JOC8325). Compounds **76** and **77** can be obtained via an ICI



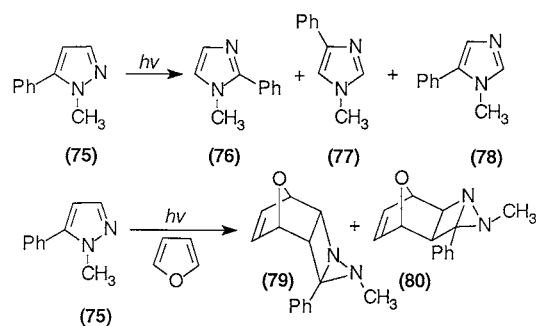
SCHEME 27



SCHEME 28

mechanism; in fact, the irradiation of **75** in the presence of furan gave the corresponding Diels–Alder adducts (**79** and **80**) formed from Dewar pyrazole derivative and furan (Scheme 29) (97JOC8325).

There are several data in agreement with a mechanism involving an excited singlet state (93JA7645; 97JOC8325). Theoretical calculations support this



SCHEME 29

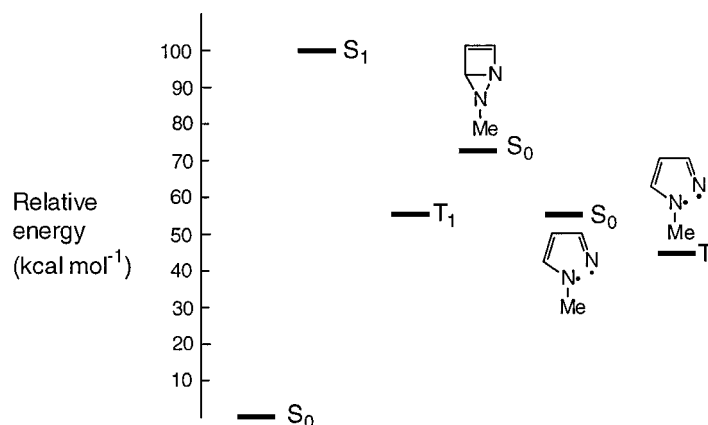


FIG. 12. Relative energy of the excited states of 1-methylpyrazole and of some reactive intermediates.

hypothesis. In fact, the homolytic cleavage of the N—N bond in the excited singlet state gave a biradical that can evolve as described in Scheme 28 (Fig. 12) (70PAC495; 000UP1). The reaction of 1-methyl-3-cyanopyrazole cannot be explained using the same hypothesis. In this case, the formation of the Dewar pyrazole derivative accounts for the experimental results (Fig. 13) (000UP1). The formation of the excited triplet state is energetically favored, but it cannot evolve to give the corresponding biradical.

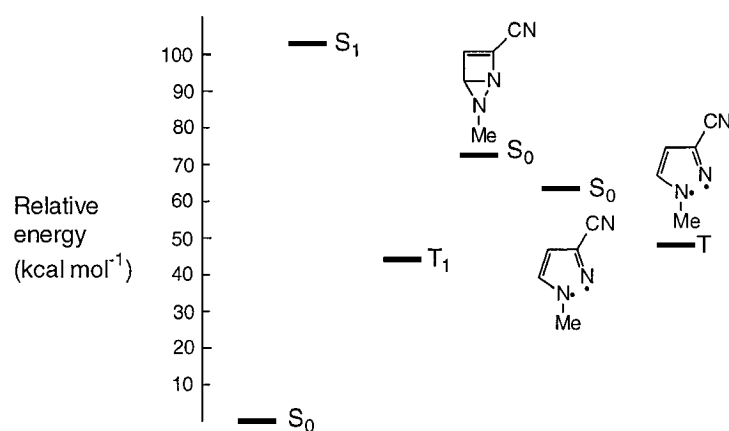
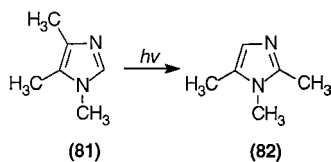


FIG. 13. Relative energy of the excited states of 1-methyl-3-cyanopyrazole and of some reactive intermediates.



SCHEME 30

VIII. Imidazole

The irradiation of imidazole derivatives such as **81** gave isomeric compounds **82** (Scheme 30) (67TL5315; 69T3287). Dewar isomers are invoked to justify the observed photochemical behavior.

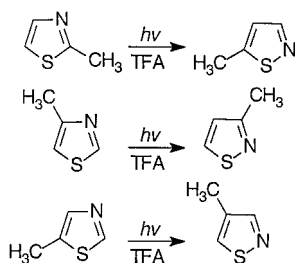
Computational results are reported for the isomerization of 1,4,5-trimethylimidazole (99MI233). They show that the isomerization occurs through the Dewar isomer arising from the excited singlet state. The formation of the triplet state is energetically favored; however, the biradical intermediate cannot be produced because it has higher energy than the excited triplet state.

IX. Thiazole

The irradiation of thiazole did not give any interesting products [69JCS(CC) 1018]. However, 2-, 4-, and 5-methylthiazole gave the corresponding isothiazoles in low yields when irradiated in trifluoroacetic acid (Scheme 31) (93JOC3407).

A. ISOMERIZATION OF ARYL-SUBSTITUTED THIAZOLES

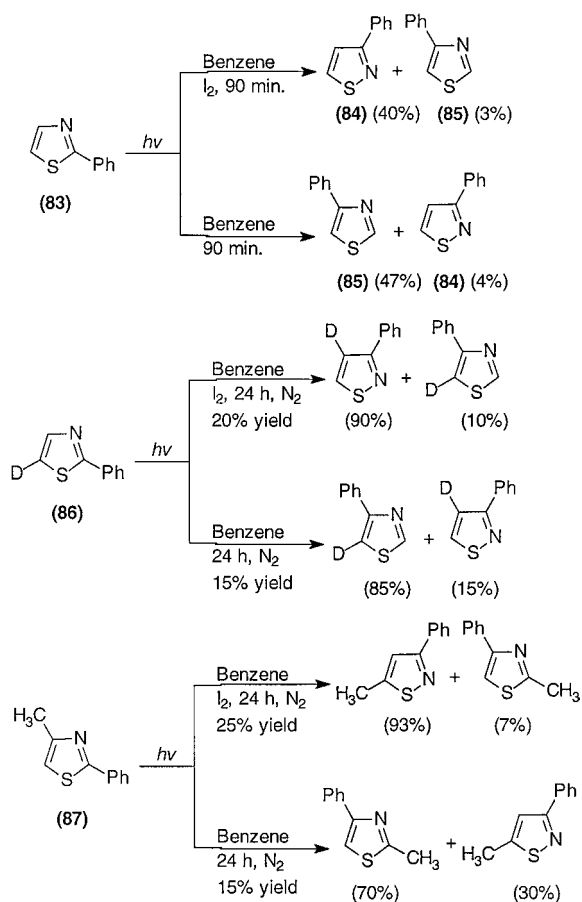
Many data have been reported on the reactivity of 2-phenylthiazole (**83**). In a pioneering work, the irradiation of this compound led to 4-phenylthiazole (**84**)



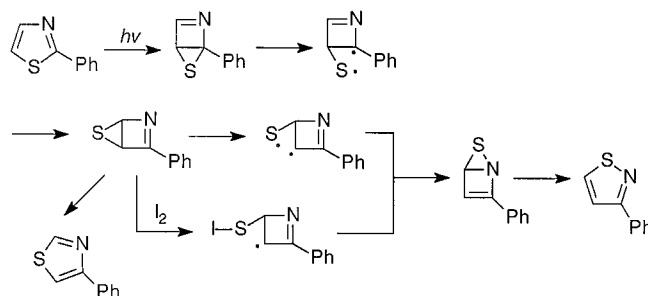
SCHEME 31

and 3-phenylisothiazole (**85**); however, while the experimental conditions were provided (benzene, 12–24 h), the yields of the obtained products were not reported (71BSF1103). Subsequently, the same authors reported that the irradiation of a benzene solution of **83** (obtained through irradiation of 2-iodothiazole in benzene) in the presence of iodine leads to **85**, the major product, in addition to small amounts of **84** (Scheme 32). When the reaction was carried out without iodine, the main product was 4-phenylthiazole (**84**) while 3-phenylisothiazole (**85**) was present in a trace amount [72JCS(P)1145; 94JA2292].

2-Phenyl-5-deuterothiazole (**86**) and 2-phenyl-4-methylthiazole (**87**) showed the same behavior (Scheme 32) (72BSF2673; 94JA2292). By contrast,



SCHEME 32



SCHEME 33

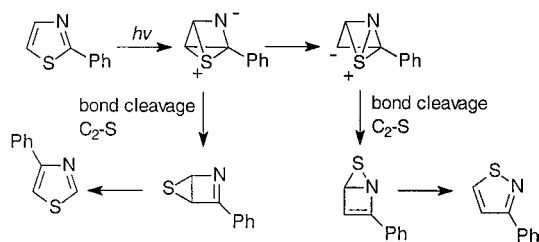
4-phenylthiazole practically did not react, and 5-phenylthiazole gave 4-phenylisothiazole as the main product in very low yield (73BSF1743; 94JA2292).

Other methyl derivatives were additionally studied. 2-Phenyl-5-methylthiazole furnishes mainly 3-phenyl-4-methylisothiazole; 2-methyl-5-phenylthiazole gives 3-methyl-5-phenylisothiazole; 4-methyl-5-phenylthiazole leads to the formation of 3-methyl-4-phenylisothiazole; 2-methyl-4-phenylthiazole gives 3-phenyl-5-methylisothiazole; and, finally, 4-phenyl-5-methylthiazole is converted into 3-phenyl-4-methylisothiazole (74T879).

The authors generally considered the above results consistent with an ICI mechanism. In this contest, iodine favors the intersystem crossing or the opening of the Dewar intermediate, as reported in the Scheme 33 (72BSF2673).

However, such an explanation was not convincing for other authors. Maeda and Kojima found that the irradiation of 2-phenylthiazole in ethanol at 80°C led to the same products described before but in a different ratio. Under the same reaction conditions, 5-phenylthiazole gave 4-phenylisothiazole, while 4-phenylthiazole was converted into 3-phenylisothiazole. The most important observation those authors made was that deuterium incorporation occurred when the reaction was carried out in benzene at 80°C in the presence of deuterium oxide. In fact, 2-phenylthiazole furnished deuterated 3-phenyl-4-deuteroisothiazole and 4-phenylthiazole without any deuterium incorporation. Likewise, 4-phenylthiazole was converted into 3-phenyl-4-deuteroisothiazole; however, 5-phenylthiazole did not undergo any deuterium incorporation [73T3523; 78JCS(P1)685]. On the basis of their results, the authors suggested a new mechanism hypothesis on the ZI route involving the participation of a polar intermediate (Scheme 34) [73T3523; 78JCS(P1)685]. In fact, the formation of a polar intermediate could account for the deuterium incorporation. However, deuterium incorporation could also be explained by using the ICI mechanism (94JA2292).

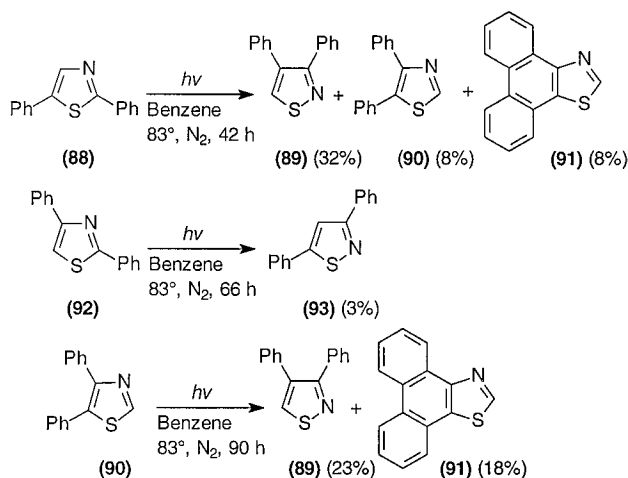
The same authors discussed the reactivity of diphenylthiazoles. 2,5-Diphenylthiazole (**88**), irradiated in benzene at 80°C, gave 3,4-diphenylisothiazole (**89**) as



SCHEME 34

the major product, together with minor amounts of 4,5-diphenylthiazole (**90**) and its cyclized derivative **91** (Scheme 35). 2,4-Diphenylthiazole (**92**) gave only a very low yield of 3,5-diphenylisothiazole (**93**), while 4,5-diphenylthiazole (**90**) was converted into the corresponding cyclized product **91** and 3,4-diphenylisothiazole (**89**) (Scheme 35) [70JCS(CC)386; 78JCS(P1)685]. All the data are in agreement with an ICI mechanism.

Theoretical calculations explain the photochemical behavior of phenylthiazoles (Fig. 14) (99MI233). The RCRE mechanism cannot be invoked because the radical intermediates have higher energies than the corresponding triplet states. Furthermore, the formation of the Dewar isomer is favored in comparison with the formation of the zwitterionic intermediate. Nevertheless, the reaction conditions used by Kojima and Maeda could allow for an endothermic reaction giving this type of intermediate. The same results were obtained using 2,5-diphenylthiazole.



SCHEME 35

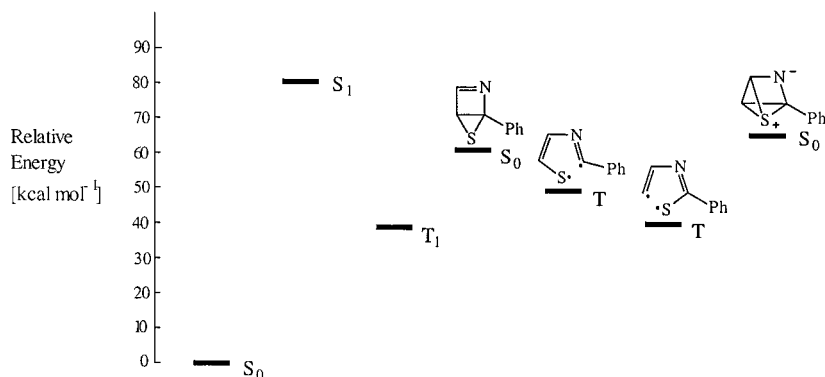


FIG. 14. Relative energy of the excited states of 2-phenylthiazole and of some reactive intermediates.

B. ISOMERIZATION OF BITHIAZOLES

The irradiation a 1:1 Cu–peplomycin complex, an antibiotic of the bleomycin family (Fig. 15), leads to a product of an isomerization of a thiazole ring. In this case, the compound converted from a 2,4'-bithiazole to a 4,4'-bithiazole derivative. When a 5:1 Cu – peplomycin complex was used, the product was a 3-isothiazolyl-4-thiazole derivative (86JA7089; 87JA938).

This behavior has been verified with bithiazole derivative **94** (Scheme 36). Neither acetophenone nor benzophenone sensitized the reaction, in agreement with an ICI mechanism via the excited singlet state (86TL6385; 87JA938).

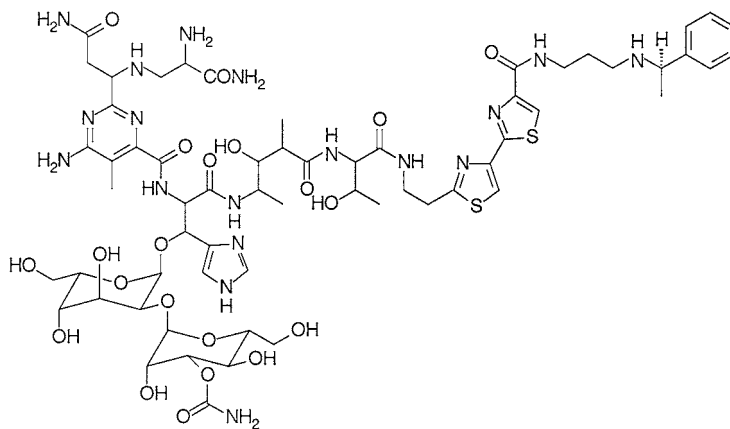
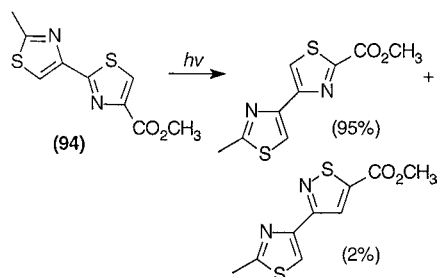


FIG. 15. Peplomycin.



SCHEME 36

Semiempirical calculations on **94** showed that it can isomerize only via the ICI mechanism; in fact, the triplet state cannot evolve to give the corresponding biradical derivatives (Fig. 16) (99MI233).

C. ISOMERIZATION OF TRITHIAZOLES

Trithiazole **95** gave the same type of reaction described above. In this case the isomerization occurred at the second ring (Scheme 37) (88TL3963).

The authors justified their result considering that the bond index $[B_{r,s} = (\text{coefficient } r \text{ LUMO})^2(\text{coefficient } s \text{ LUMO})^2]$ on the LUMO accounted for the formation of the Dewar isomer on the central ring of the trithiazole. By contrast, calculations show that the Dewar isomer should be formed on the third ring of the trithiazole (Fig. 17) (99MI233).

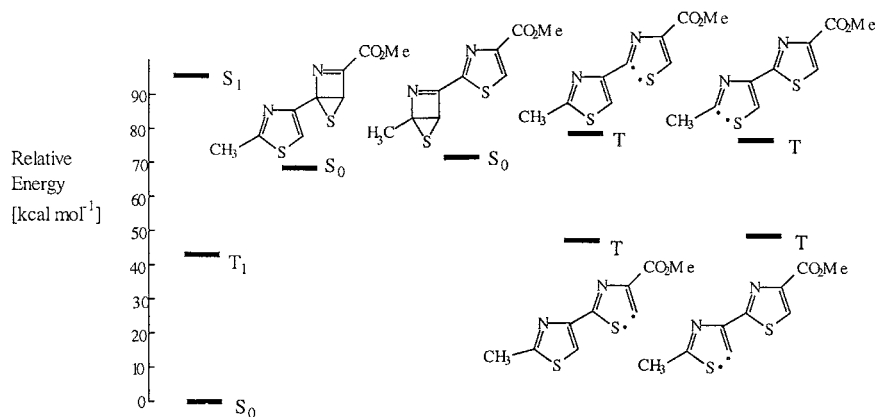
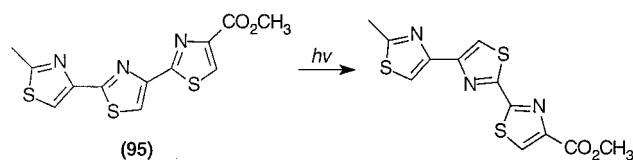
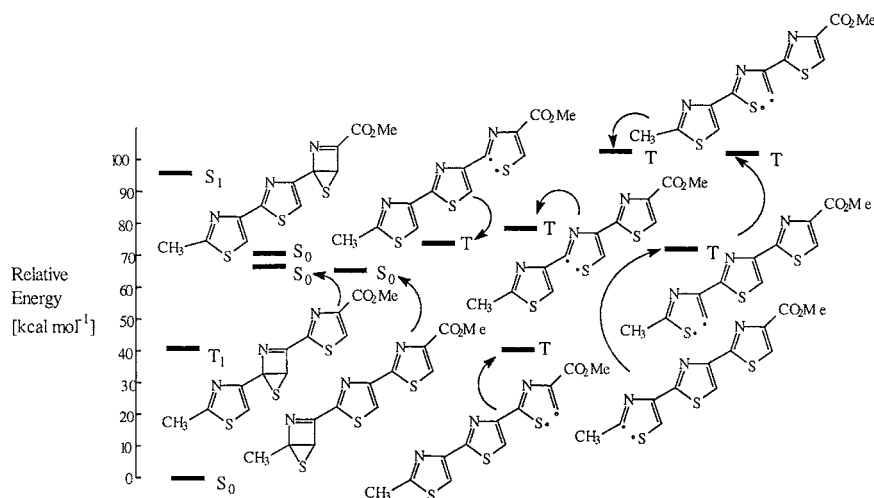


FIG. 16. Relative energy of the excited states of bithiazole derivative **94** and of some reactive intermediates.



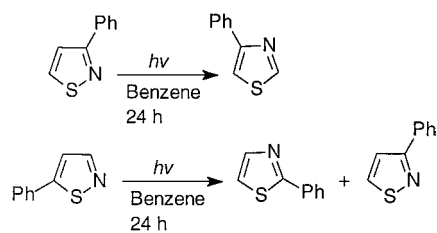
SCHEME 37

FIG. 17. Relative energy of the excited states of trithiazole derivative **95** and of some reactive intermediates.

X. Isothiazole

The irradiation of isothiazole with a low-pressure mercury arc leads to the formation of thiazole [69JCA(CC)1018]. The photochemical behavior of alkylisothiazoles has been studied. 3-Methylisothiazole gave 2-methylthiazole in a low yield. 4-Methylisothiazole was converted into 4-methylthiazole, and 5-methylisothiazole gave a mixture of 5-methylthiazole (55%) and 4-methylisothiazole (Scheme 38) (72T3141; 93JOC3407). Either a ZI (72T3141) or an ICI mechanism was invoked to justify these reactions (93JOC3407).

Semiempirical calculations on 4-methylisothiazole showed that the reaction can occur through an ICI mechanism with the formation of the Dewar isothiazole derivative (Fig. 18) (000UP1). In fact, the triplet state of the isothiazole cannot evolve to the biradical. The ZI mechanism can be excluded: Only the intermediate **96** showed an acceptable energy; however, it is a resonance structure of Dewar isothiazole.



SCHEME 39

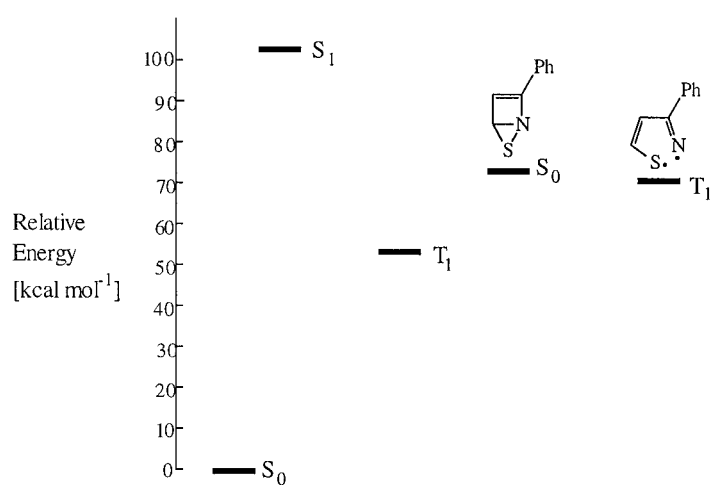
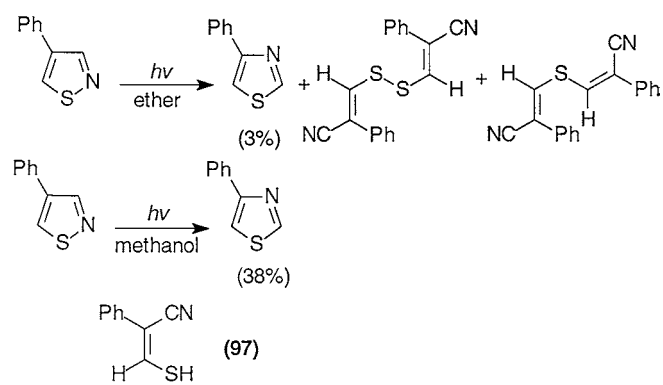


FIG. 19. Relative energy of the excited states of 3-phenylisothiazole and of some reactive intermediates.



SCHEME 40

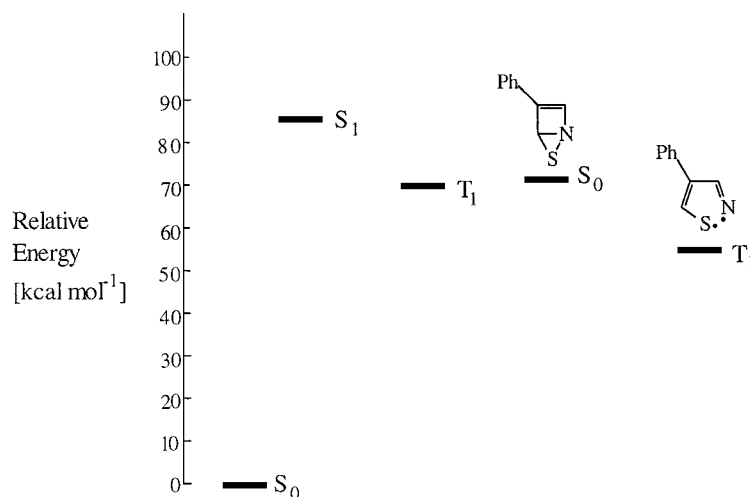


FIG. 20. Relative energy of the excited states of 4-phenylisothiazole and of some reactive intermediates.

led to 4-phenylthiazole in 38% yield. Finally, in the presence of triethylamine, the yields of 4-phenylthiazole increased. All these data can be explained by assuming the formation of the ring-opening intermediate **97** displayed in Scheme 40.

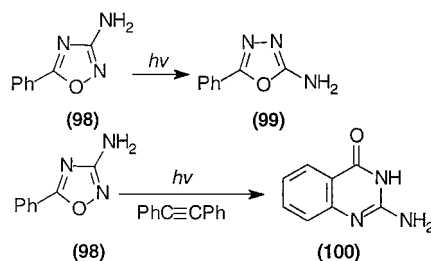
Calculations account for the experimental results (Fig. 20) (99MI233). The first excited singlet state (which accounts for the absorption at 269 nm; calculated value 267 nm) was converted into the corresponding triplet state (69 kcal mol⁻¹; experimental 68 kcal mol⁻¹). The triplet state gives the cleavage of the S—N bond with the formation of the biradical intermediate. This intermediate leads to the product.

XI. Oxadiazoles

A. ISOMERIZATION OF 1,2,4-OXADIAZOLES

The irradiation of 3-amino-5-phenyl-1,2,4-oxadiazole (**98**) gave the corresponding 1,3,4-oxadiazole (**99**) (Scheme 41) [88JCS(P1)1313; 88JHC931; 95H(41)2095]. The formation of **99** has been explained by assuming a RCRE mechanism.

1,3,4-Oxadiazole was obtained through the first excited singlet state. When the reaction was carried out in the presence of a triplet sensitizer, **99** was not detected but the quinazolinone **100** was obtained (Scheme 41) [91JCS(P2)187]. Compound **99** cannot be obtained via the Dewar isomer. The author supposed the formation



SCHEME 41

of a zwitterionic intermediate through the heterolytic cleavage of the O—N bond [91JCS(P2)187]. Semiempirical calculations showed that in the excited singlet state the heterolytic cleavage to give the zwitterionic intermediate **101** is favored in comparison with the formation of both the Dewar isomer and the triplet state (Fig. 21) (000UP1). In the Fig. 21 the energies of both the first excited singlet state and the triplet state are experimental values [91JCS(P2)187]. By contrast, when the reaction occurs through the triplet state, only the biradical can be obtained.

Recently, the reaction of 3-methoxy-5-aryl-1,2,4-oxadiazoles in the presence of diphenylacetylene to give the corresponding quinazolinones has been reinvestigated and an electron transfer mechanism was proposed (99JOC7028).

On irradiation in ether at 254 nm 2,5-diphenyl-1,2,4-oxadiazole (**102**) did not give any interesting result. On the contrary, the irradiation in an immersion apparatus gave, with low conversion (33%), a mixture of three products (**103–105**) (Scheme 42) [68TL2417; 95H(41)2095].

Compound **104** could not be obtained from **103**, and a hypothesis about its formation considered the (homolytic or heterolytic) cleavage of the O—N bond (Scheme 43) (68TL2417). The sensitized reaction did not give a different result; the author supposed that the reaction involved the excited triplet state of the molecule. When the reaction was carried out in methanol, **104** was obtained in ~8% yield

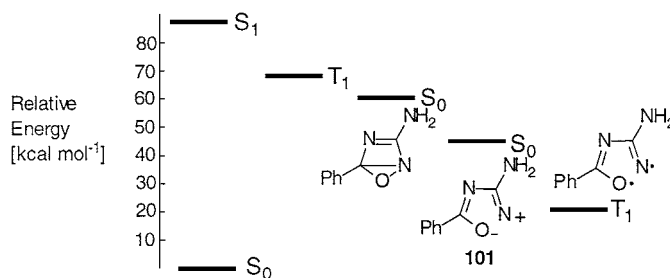
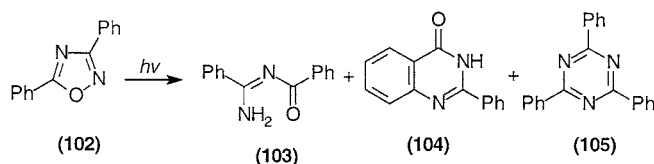


FIG. 21. Relative energy of the excited states of 3-amino-5-phenyl-1,2,4-oxadiazole and of some reactive intermediates.



SCHEME 42

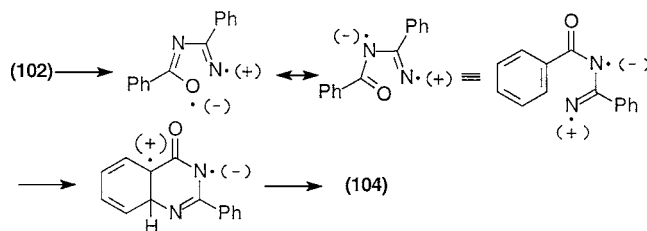
while the methoxy derivative of **103** was isolated as the main product (40%) (68TL2421).

Semiempirical calculations on this compound showed that the excited triplet state can be obtained. This intermediate can evolve to the biradical (Fig. 22) (000UP1).

When the reaction was carried out on the phenoxy derivative **106**, only **107** was obtained (Scheme 44) (88JHC1551). The formation of this product was rationalized assuming a heterolytic cleavage of the O—N bond followed by isomerization (Scheme 44). If the reaction occurs in the excited triplet state of the molecule, the biradical is the most probable intermediate.

The isomerization described in Scheme 44 could not be observed when the 5-methyl derivative **108** was used. The quinazolinone **109** was obtained in high yield, probably via **110** through the cleavage of the O—N bond in the triplet state (Scheme 45) [89H(29)737; 89H(29)1301; 91JCS(P2)187]. The same behavior (but in low yield) was observed with **111** (Scheme 45) (90JHC861).

The irradiation of 5-amino-1,2,4-oxadiazole derivatives gave ring-opening products **112** (Scheme 46) (88JHC931). When external nucleophiles were added to the reaction mixture, products deriving from the attack of these nucleophiles to the ring-opening intermediate deriving from the O—N bond cleavage were observed. In the presence of amines, the triazoline **113** was obtained (Scheme 46) (96JOC8397), while, in the presence of thiourea, **114** was the main product (Scheme 46) (97T12629). However, in the presence of sodium hydrogen sulfide, **115** was obtained without sulfur incorporation (97T12629).



SCHEME 43

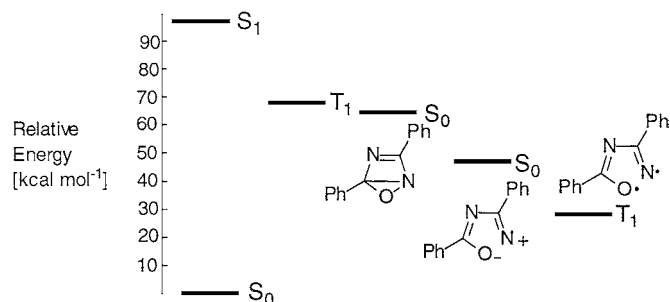
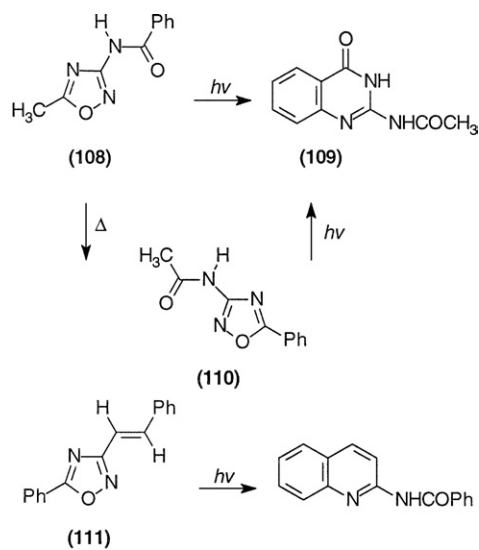
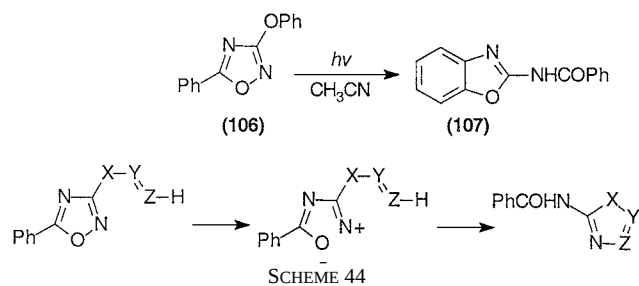
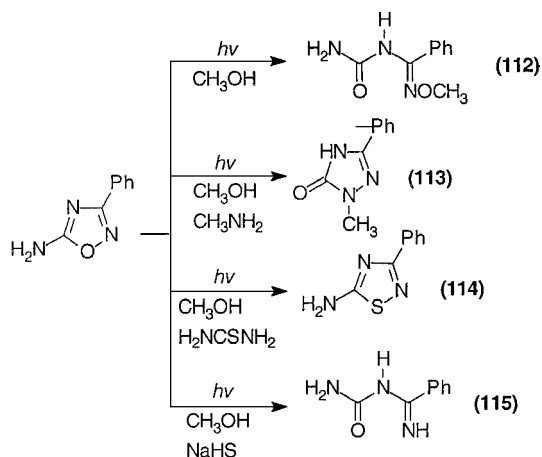


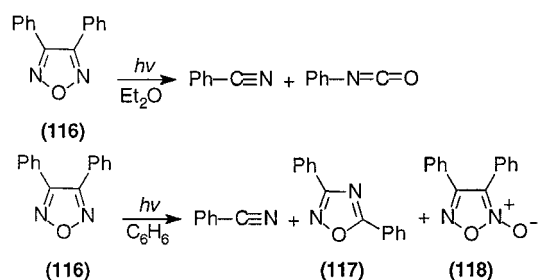
FIG. 22. Relative energy of the excited states of 3,5-phenyl-1,2,4-oxadiazole and of some reactive intermediates.





B. ISOMERIZATION OF 1,2,5-OXADIAZOLES

The photolysis of 3,4-diphenyl-1,2,5-oxadiazole (**116**) in ether with a high-pressure mercury arc through Corex gave benzonitrile and phenyl isocyanate (Scheme 47) [68JCS(CC)977]. When xanthone was used as a triplet sensitizer, the observed quenching of the reaction was in agreement with a reaction occurring in the excited singlet state. When the reaction was carried out in benzene through Pyrex, benzonitrile was the main product, but low yields of 3,5-diphenyl-1,2,4-oxadiazole (**117**) and diphenylfuroxan (**118**) were obtained (Scheme 47) (69BCJ581). The authors considered that **117** was obtained by the coupling of benzonitrile and benzonitrile oxide while **118** was obtained by the recombination of two molecules of benzonitrile oxide.



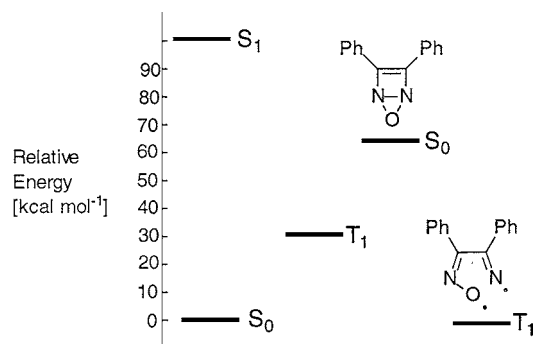
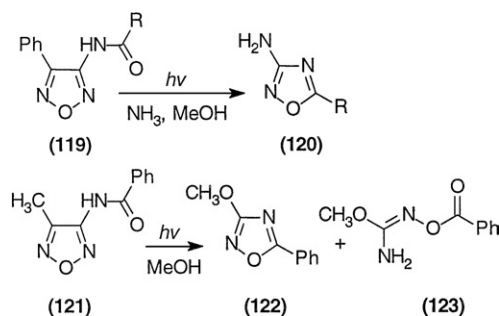


FIG. 23. Relative energy of the excited states of 3,4-phenyl-1,2,5-oxadiazole and of some reactive intermediates.

The results described above represent the first example of the FR mechanism (Scheme 1). Semiempirical calculations on this molecule showed that the intersystem crossing to the excited triplet state is favored: The reaction cannot be sensitized by xanthone because the triplet state of 3,4-diphenyl-1,2,5-oxadiazole is lower than that of xanthone. The cleavage of the triplet state to the biradical is favored, considering the relative energy of this intermediate (Fig. 23) (000UP1).

The irradiation of 3-acylamino derivatives **119** in aqueous ammonia gave the corresponding 1,2,4-oxadiazoles (**120**) in 45–50% yield (Scheme 48) [92H(34)2313; 95S917]. The observed reactivity can be explained by assuming a fragmentation reaction of the substrate to give a nitrile oxide intermediate that can react with ammonia and then with the acyl group. However, when **119** was irradiated in methanol, only benzonitrile, phenylcarbamate, and acetylcyanamide were detected in the reaction mixture (95JOC4096). The irradiation of **121** in methanol gave the 1,2,4-oxadiazole derivatives **122** and **123** in 29 and 26% yield, respectively (Scheme 48) (95JOC4096).



SCHEME 48

XII. Conclusions

In this chapter I have attempted to collect all the data available on the photo-isomerization reactions of pentaatomic aromatic heterocycles. At the end of this work, I can note that all the results fit the hypothesis I reported in the introduction of this chapter.

The direct irradiation of a pentaatomic heterocycle leads to the formation of a singlet excited state. This excited state can interconvert into the corresponding triplet state or into the corresponding Dewar isomer. The Dewar isomer is the origin of the formation of isomeric heterocyclic derivatives. Only in the case of isoxazoles and 1,2,4-oxadiazoles can homolytic or heterolytic cleavage of the N—O bond in this excited singlet state be supposed. The excited triplet state, obtained via intersystem crossing from the corresponding excited singlet state or via a sensitized reaction, can evolve to give a biradical intermediate via the homolytic cleavage of the X—C_α bond. This biradical intermediate can be converted into decomposition products or into ring-contraction products. The ring-contraction products can be irradiated under the reaction conditions used to give isomeric heterocyclic derivatives.

This scheme is very simple if compared with the jungle of hypotheses formulated to justify the photochemical isomerization of pentaatomic heterocycles. I do not know if it is true or if it represents a constriction of the nature in a rigid cage. I hope my work will be useful to direct future research efforts in this field. It is not important if future data will confirm or destroy my hypothesis: The most important thing is to elicit a discussion.

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ADVANCES IN HETEROCYCLIC CHEMISTRY, VOL. 79

Heterocyclic Acyl and Formyl Anion Equivalents

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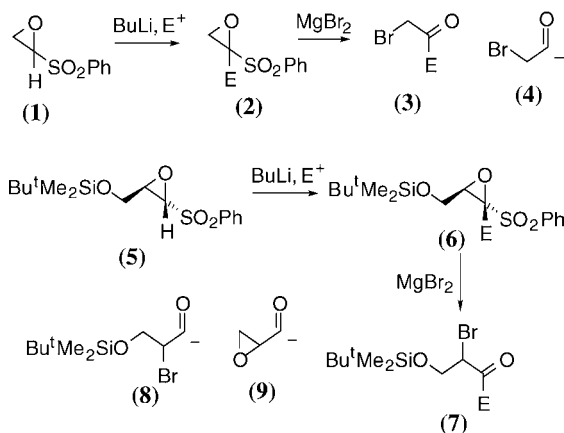
I. Introduction

Although many carbonyl derivatives act as acyl cation equivalents, $R(C=O)^+$ in synthetic chemistry, the inherent polarity of the carbonyl group makes it much more difficult to find compounds that will act as equivalents of acyl anions, $R(C=O)^-$. Since the 1960s, major progress has been made in this area, and there are now a wide variety of compound types that can react in this way. As in so many areas of organic chemistry, heterocyclic compounds take pride of place and form the basis of many of the most useful methods. In recent years there has been particular interest in developing chiral acyl anion equivalents that will show high

diastereoselectivity in their addition to prochiral electrophiles. In this chapter we have attempted to describe the most important acyl and formyl anion equivalents in which the anionic carbon atom is either part of a heterocyclic ring or is attached to one or more adjacent heterocyclic groups. The examples are organized in order of (1) increasing ring size, (2) increasing number of heteroatoms, and (3) decreasing priority of heteroatoms.

II. Oxiranes

The phenylsulfonyloxirane **1** is readily deprotonated at low temperature, and the resulting anion reacts with a wide variety of electrophiles to give **2**. Nucleophilic ring opening of this by magnesium bromide is accompanied by loss of PhSO_2^- to give the functionalized ketone products **3**, thus making **1** a synthetic equivalent for the acyl anion **4** [88JCS(CC)645; 91JCS(P1)897]. The oxirane **5** with additional functionality behaves similarly, giving first **6** and then **7** [89JCS(P1)835; 92JCS(P1)2863]; and since removal of the silyl group in **7** results in oxirane formation, **5** may be considered as an equivalent of **9** as well as **8**. The range of electrophiles that can be used is wide and includes alkyl halides, aldehydes and ketones, acid chlorides, and lactones.

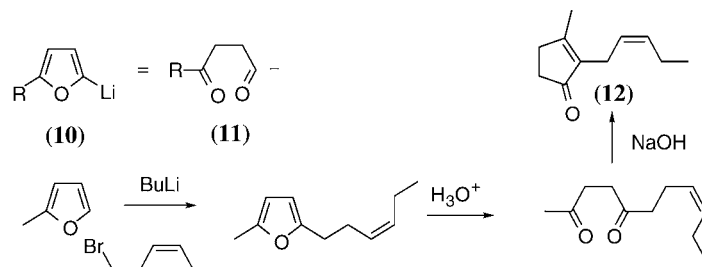


III. Five-Membered Rings

A. FURANS AND DIHYDROFURANS

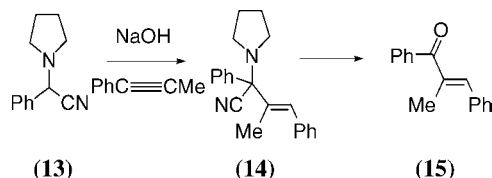
The ease with which furans may be formed from and hydrolyzed back to 1,4-dicarbonyl compounds, together with ready 5-lithiation of 2-substituted furans,

makes **10** a convenient equivalent of the γ -oxoacyl anion **11**. An early example of this is provided by Büchi and Wüest's synthesis of *Z*-jasmone **12** (66JOC977). Lithiation of 2,3-dihydrofuran at the 5 position is also readily achieved, making it a potential γ -functionalized butyryl anion equivalent although this has only been reported for a limited range of electrophiles (77TL4187; 81T3997).



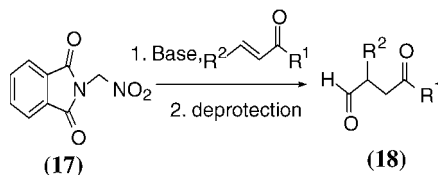
B. PYRROLIDINES AND PHTHALIMIDES

The α -cyanobenzylpyrrolidine **13** may be used as a benzoyl anion equivalent by treatment with NaOH in DMSO and $\text{PhC}\equiv\text{CMe}$ to afford the adduct **14**, although conditions to deprotect this to give the enone **15** were not specified (93LA375).



The hydrazone **16** formed between formaldehyde and (*S*)-1-amino-2-methoxymethylpyrrolidine (SAMP) is one of the most effective chiral formyl anion equivalents. It undergoes nucleophilic addition under mild neutral conditions to a wide variety of electrophiles, including nitroalkenes (94TL471; 96S48), sugar aldehydes (96TL5787), trifluoromethyl ketones [98AG(E)3428], enones (96JA7002; 97JOC5144), and α,β -unsaturated lactones [99SL629; 99JCS(CC)701]. The hydrazone function is readily removed by ozonolysis and the e.e. of the resulting products is often greater than 90%. Some of the applications of this useful reagent are summarized in Scheme 1 (98JPR281).

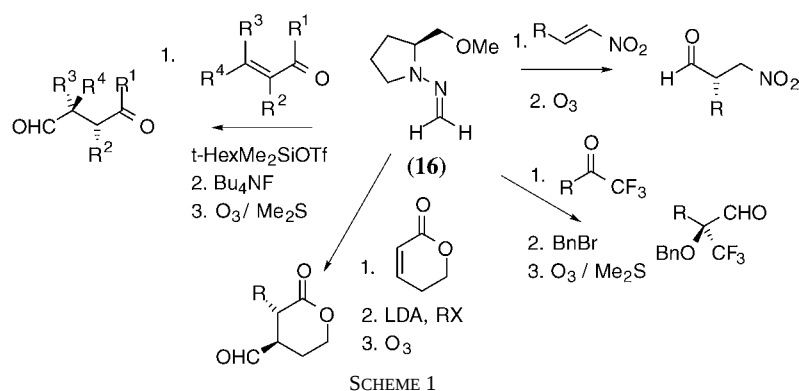
The anion of *N*-nitromethylphthalimide **17** also acts as a formyl anion equivalent with Michael acceptors to afford 1,4-dicarbonyl compounds **18** in good to excellent yields, although difficulty was experienced in isolating the products under the conditions required for removal of the phthalimide group (77CJC2919).



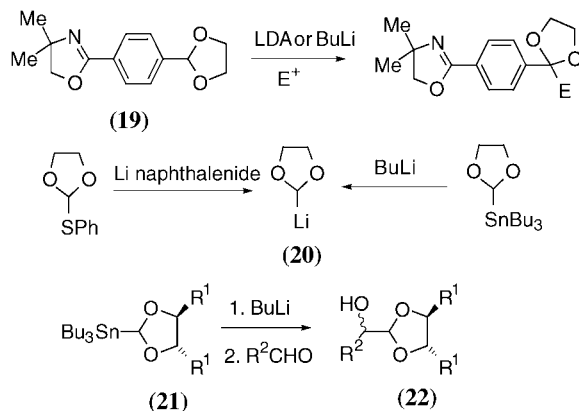
C. 1,3-DIOXOLANES AND DIOXOLANONES

The 2-aryl-1,3-dioxolanes are readily deprotonated at the 2 position, but in most cases the resulting anions are unstable and undergo fragmentation with elimination of an alkene to give the corresponding benzoates. For the oxazoline-substituted compound **19**, however, this does not occur and it acts as an effective acyl anion equivalent with a variety of electrophile types (79TL4155; 79TL4159). Later studies showed that the parent 2-lithio-1,3-dioxolane **20** may be successfully generated either by reductive lithiation of the phenylthio compound or by transmetalation of the stannane. It was shown to be stable at -78°C and added in the expected way to *p*-anisaldehyde (89JA1381). A series of chiral dioxolanes **21** were evaluated as potential chiral formyl anion equivalents by treatment with BuLi at -78°C followed by benzaldehyde or isobutyraldehyde. The expected products **22** were formed, but with disappointing stereoselectivity ranging from 50 to just 10% d.e. (94TL2063).

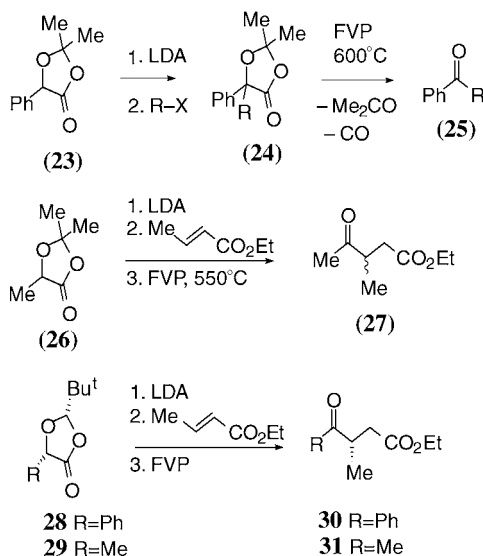
The 5-substituted 1,3-dioxolan-4-one **23** is readily deprotonated at the 5 position and can be alkylated with a variety of alkyl halides. The resulting products **24** decompose upon flash vacuum pyrolysis (FVP) at 600°C with loss of acetone



SCHEME 1

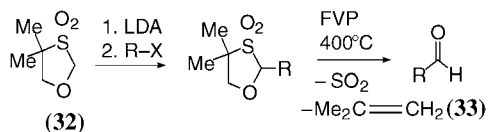


and CO to give the ketones **25**, thus making **23** a useful benzoyl anion equivalent (98SL102). The methyl analogue **26** likewise undergoes deprotonation and conjugate addition to ethyl crotonate to afford, upon FVP at 550°C, the acetyl anion addition product **27**. By using the dioxolanones derived from chiral mandelic and lactic acids, this method was adapted to provide chiral acyl anion equivalents. Treatment of **28** with LDA followed by ethyl crotonate and then FVP at 500°C gave **30** in 91% yield and 33% e.e., while similar reaction of **29** and FVP at 550°C gave **31** in 94% yield and 86% e.e. (98SL102).



D. 1,3-OXATHIOLANE 3,3-DIOXIDES

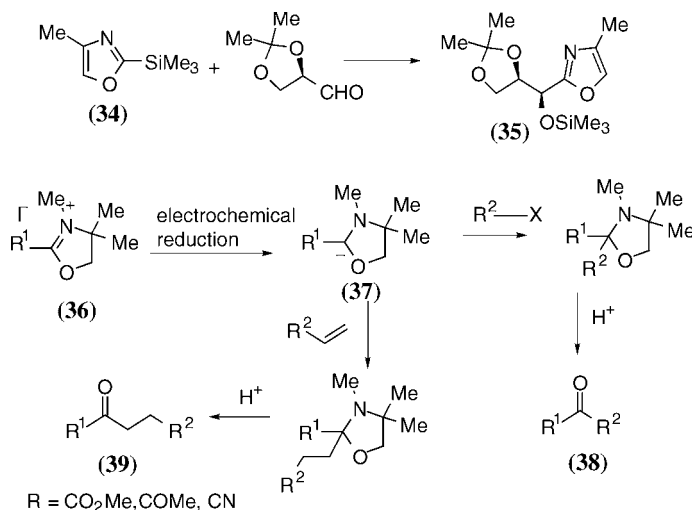
A somewhat similar method to that mentioned above involves alkylation of 4,4-dimethyl-1,3-oxathiolane *S,S*-dioxide **32** at the 2 position followed by FVP at 400°C, which results in fragmentation with loss of SO₂ and isobutene to give the aldehydes **33**. Other electrophiles that may be used include aldehydes, ketones, and Me₃SiCl, making this a convenient formyl anion equivalent (79TL3375).



E. OXAZOLES, OXAZOLINIUM SALTS, OXAZOLINONES, AND OXAZOLIDINES

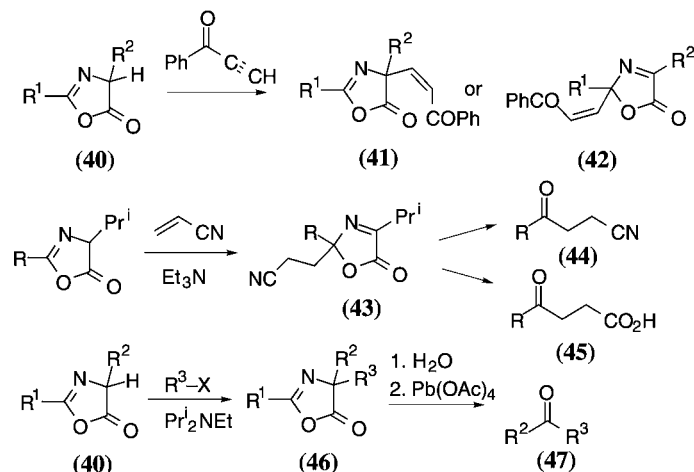
The silyloxazole **34** undergoes addition to chiral aldehydes under mild conditions; and although the stereoselectivity is poorer than for 2-trimethylsilylthiazole (see Section III,G), the opposite diastereomer of the product **35** is obtained (85TL5477).

Electrochemical reduction of oxazolinium salts **36** gives the anions **37**, which add efficiently to alkyl halides or, in the presence of Me₃SiCl, to methyl acrylate, methyl vinyl ketone, and acrylonitrile. Simple acid hydrolysis then gives the ketone products **38** and **39**, and this method is quite general since the starting salts are readily prepared from carboxylic acids, R¹CO₂H (87TL4411).



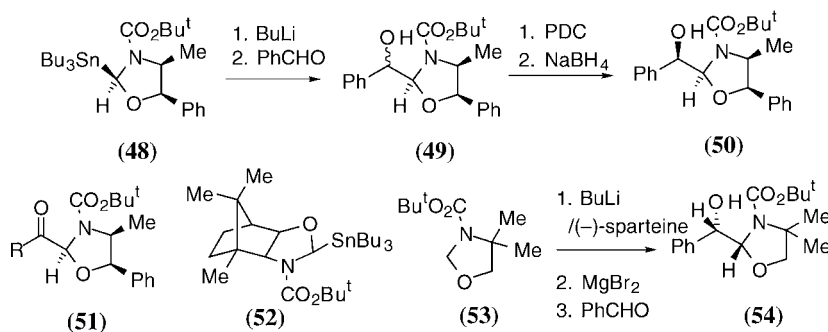
The oxazolin-5-ones **40** are readily formed by dehydration of *N*-acyl α -amino acids, and they may undergo reaction at either the 2 or 4 position depending on the nature of R^1 and R^2 , the electrophile, and the conditions. In either case the oxazolinone may be converted into a carbonyl compound, thus making these useful acyl anion equivalents. The earliest study showed that addition to benzoylacetylene generally gave the 4-adduct **41** except for $R^1 = CF_3$, where the 2 adduct **42** was formed [71AG(E)653]. On the other hand, acrylonitrile, dimethyl fumarate, dimethyl maleate, and the corresponding dinitriles added exclusively at C2 regardless of the nature of R^1 while with DMAD, acrolein, and methyl vinyl ketone an almost equal mixture of 2- and 4-adducts was produced. Simple hydrolysis of the acrylonitrile adducts **43** gave either **44** or **45** depending on the conditions used [71AG(E)655], and this method was later used to obtain derivatives of δ -aminolevulinic acid (80CB787). Reaction of **40** with benzylic and allylic halides occurs at C4, but the products **46** may also be converted into ketones **47** by hydrolysis followed by treatment with lead tetraacetate [78AG(E)450].

More recent work has shown that the 2-unsubstituted compound **40** ($R^1 = H$, $R^2 = Pr^i$) is an effective formyl anion equivalent which reacts at C2 and undergoes both 1,4-addition to α,β -unsaturated carbonyl compounds and 1,2-addition to aldehydes (93TL3907; 96T4719).



A variety of 1,3-oxazolidines have been used as chiral formyl anion equivalents for addition to aldehydes. Thus, for example, reaction of *N*-protected norephedrine with $Bu_3Sn-CH(OEt)_2$ gives **48**, and transmetalation with $BuLi$ followed by addition of benzaldehyde affords the expected adduct **49**. The selectivity at the newly formed alcohol center is poor, but the situation can be salvaged by oxidation and re-reduction, which affords the product **50** with >95% d.e. It is then a simple matter to hydrolyze off the oxazolidine, although the resulting hydroxyaldehydes

are rather sensitive and are usually reduced immediately to the chiral 1,2-diols (94TL2063). Alternatively, the chiral 2-acyloxazolidines **51** may be prepared in other ways and reacted with Grignard reagents to afford, upon hydrolysis, chiral α -hydroxyaldehydes in high e.e., which were again reduced *in situ* (94TL3309). The camphor-derived 2-stannyloxazolidine **52** likewise undergoes transmetalation and reaction with aldehydes to give products analogous to **49** as almost equal mixtures of two diastereomers (95TL2863). Again, oxidation of the secondary alcohol and re-reduction or reaction with Grignard reagents affords the chiral α -hydroxyaldehydes, which would be expected from addition of formyl anion to carbonyl compounds.



The foregoing examples do not represent useful chiral formyl anion equivalents in a direct sense since the stereoselectivity of the initial addition to aldehydes is poor, although as has been explained, the situation is salvaged by oxidation and re-reduction. On the other hand, by lithiation at the 2 position of the achiral oxazolidine **53** in the presence of (–)-sparteine followed by addition of benzaldehyde, useful levels of d.e. and e.e. are achieved directly (98TA3125). For example, by adding MgBr₂ before the benzaldehyde, the major product obtained is **54** in 80% d.e. and 86% e.e.

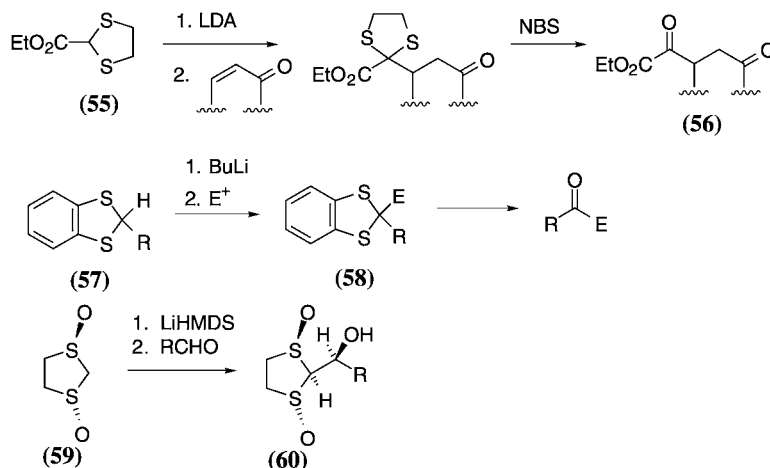
F. 1,3-DITHIOLANES, 1,3-BENZODITHIOLES, AND THEIR S-OXIDES

Although deprotonation of simple 1,3-dithiolanes at the 2 position is usually accompanied by cycloreversion to the alkene and dithiocarboxylate, this does not occur for the 2-ethoxycarbonyl compound **55**. The anion of this is readily generated with LDA and undergoes conjugate addition to α,β -unsaturated ketones, esters, and lactones to give, after deprotection, the α,δ -diketoester products **56** (73TL2599). In this transformation **55** therefore acts as an equivalent of EtO₂C–C(O)[–].

The 2-substituted 1,3-benzodithioles **57** are readily lithiated and react as acyl anions with various electrophiles, including alkyl halides, aldehydes, ketones,

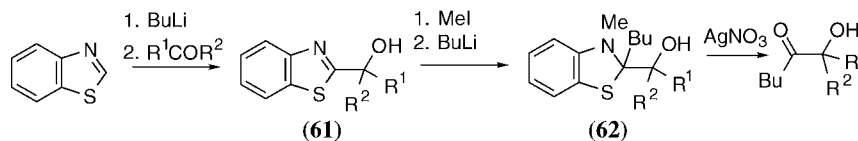
and epoxides. Cleavage of the products **58** to give the corresponding ketones occurs in good yield although toxic mercury compounds are usually used for this (78TL2345).

The (racemic) *trans* disulfoxide of 1,3-dithiolane **59** is readily deprotonated at C2 by lithium hexamethyldisilazide, and the resulting anion reacts with aldehydes at -78°C with moderate to excellent diastereoselectivity to give mainly the products **60**, although subsequent cleavage of these to give the α -hydroxyaldehydes was not described (97JOC1139).



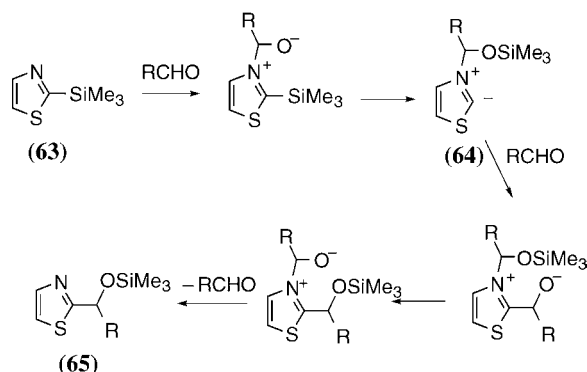
G. THIAZOLES AND BENZOTHAZOLES

Benzothiazole is readily lithiated at the 2 position and the resulting anion adds to ketones to give products **61** (78TL5), although removal of the benzothiazole to give α -hydroxyketones was not described. In a later study the range of electrophiles was extended to include aldehydes, esters, lactones, benzonitrile, DMF, Me_3SiCl , and haloketones (88BCJ3637). Although direct removal of the benzothiazole was again not reported, α -hydroxyketone products were obtained by reaction of **61** with methyl iodide followed by butyllithium to give **62** and then treatment with silver nitrate.

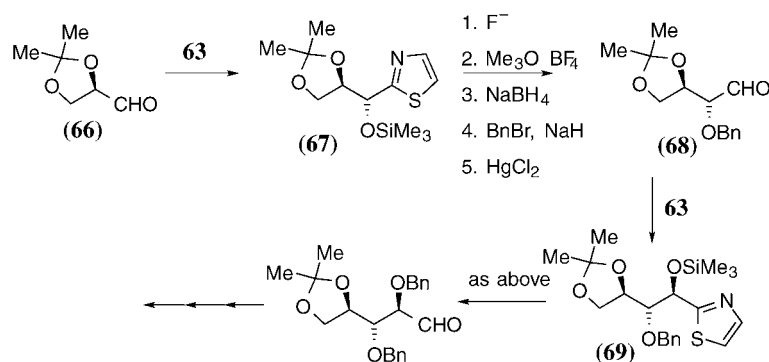


The deprotonation of 4,5-dimethylthiazole and addition of the resulting anion to aldehydes was demonstrated as early as 1948 (48HCA652); and 2-lithiothiazoles were later shown to react with aldehydes, ketones, methyl iodide, and epoxides

(62BSF2072). However, it is only in the last 20 years with the extensive work of Dondoni that 2-lithiothiazole and particularly 2-trimethylsilylthiazole **63** have emerged as among the most versatile formyl anion equivalents (98S1681). The latter compound reacts readily at room temperature with a variety of electrophiles including acid chlorides, ketenes, and aldehydes (83TL2901; 88JOC1748). The reaction with aldehydes, which is perhaps the most useful, gives the products **65**; and both the isolation of intermediates (93JOC3196) and theoretical studies (96JOC1922) point to a multistep mechanism involving two molecules of aldehyde and the ylide **64** as shown.

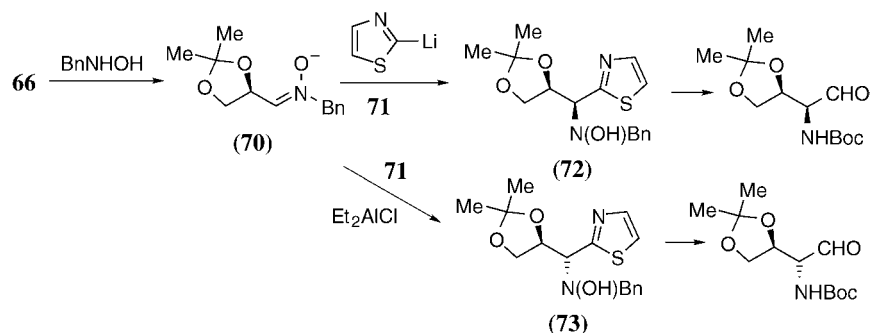


Where the aldehyde is chiral, good diastereoselectivity is generally obtained at the new center, and since the thiazole in **65** is readily removed to generate a new aldehyde function, the procedure lends itself ideally to the stepwise homologation of carbohydrates. The method is illustrated by reaction of D-glyceraldehyde acetonide **66** with **63** to give **67** in 93% yield and >90% d.e. Treatment of this with fluoride, N-methylation and borohydride reduction, protection of the alcohol, and hydrolysis using mercuric chloride then gives the homologous aldehyde **68**, which reacts with **63** to afford **69** (85TL5477; 89JOC693). The sequence may be



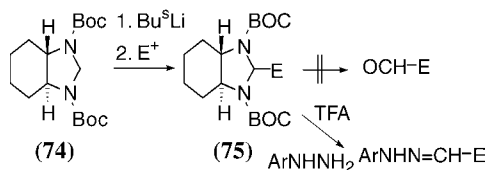
continued to give sugars with up to eight carbon atoms and has been applied to a number of interesting carbohydrate targets [86AG(E)835; 87T3533; 89JOC693; 92S201; 97JOC6261]. By starting from aminoaldehydes a variety of chiral amino sugars and unnatural amino acid derivatives may be prepared [88JCS(CC)10; 90JOC1439; 93S1162; 95S181].

The related addition of 2-lithiothiazole **71** to carbohydrate-derived nitrones provides access to α -aminoaldehydes, as illustrated by reaction of **70** to give either **72** or, in the presence of a Lewis acid, the opposite diastereomer **73**; this route has been used in the synthesis of a variety of amino sugars [92TL4221; 93SL78; 93TL5475; 94JCS(CC)1731; 95CEJ505]. A variety of other synthetic applications of both **63** (95S654) and **71** (89T5141; 94JOC6404; 95JOC4749) as formyl anion equivalents have also been described.



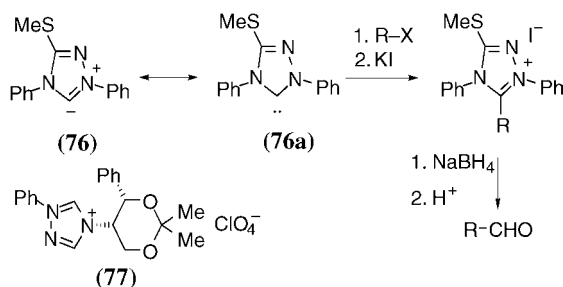
H. IMIDAZOLIDINES

The chiral bicyclic imidazolidine **74** is deprotonated at the 2 position by $s\text{-BuLi}$; and the resulting anion adds to alkyl halides, acid chlorides, chloroformates, phenyl isocyanate, and aldehydes. The use of this compound as a chiral formyl anion equivalent seems to be limited, however, since the diastereoselectivity in the addition to aldehydes is poor and hydrolysis of the products **75** to give aldehydes also produces cyclohexane-1,2-diamine, necessitating isolation of the aldehyde as its 2,4-dinitrophenylhydrazone (96SL1109; 98T14255).

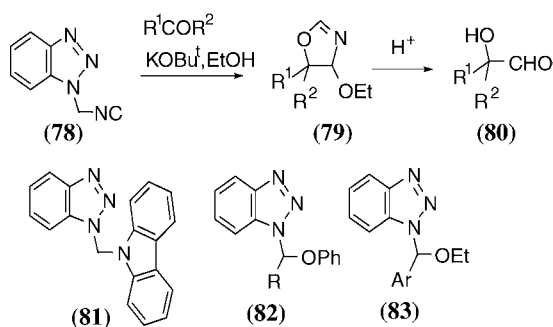


I. TRIAZOLES AND BENZOTRIAZOLES

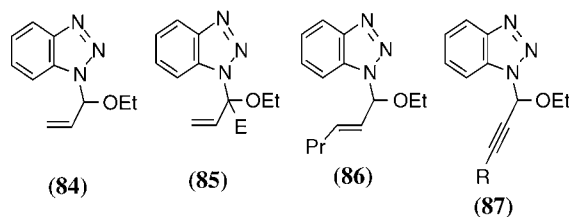
The triazole **76**, which is more accurately portrayed as the nucleophilic carbene structure **76a**, acts as a formyl anion equivalent by reaction with alkyl halides and subsequent reductive cleavage to give aldehydes as shown (75TL1889). The benzoin reaction may be considered as resulting in the net addition of a benzoyl anion to a benzaldehyde, and the chiral triazolium salt **77** has been reported to be an efficient asymmetric catalyst for this, giving the products (*R*)-ArCH(OH)COAr, in up to 86% e.e. (96HCA1217). In the closely related intramolecular Stetter reaction e.e.s of up to 74% were obtained (96HCA1899).



Katritzky and coworkers have developed a wide range of effective acyl anion equivalents based on benzotriazole. Benzotriazolymethyl isocyanide **78** adds to ketones to give the oxazoline products **79**, and these are readily hydrolyzed under acidic conditions to give α -hydroxyaldehydes **80** (89TL6657). Carbazolymethyl-benzotriazole **81** may be deprotonated at the CH₂ group by butyllithium, and the resulting anion is an effective formyl anion equivalent for addition to alkyl halides, aldehydes, ketones, PhNCO, and PhNCS (91JOC2143). Deprotection to give the corresponding aldehydes is readily achieved by acid hydrolysis, and this was usually done in the presence of 2,4-dinitrophenylhydrazine to give the hydrazones in 60–80% yield.



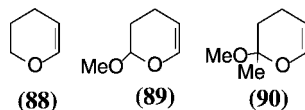
A variety of valuable acyl anion equivalents are provided by α -alkoxyalkylbenzo triazoles. The readily available α -phenoxy compounds **82**, for example, act as the equivalent of RCO^- by treatment with butyllithium and electrophiles such as alkyl halides, aldehydes, ketones, imines, enones, and silyl halides, followed by acid hydrolysis to give the ketones (96JOC7551). In a similar way the α -ethoxy compounds **83** act as equivalents of ArCO^- for addition to alkyl halides, aldehydes, ketones, and imines (95JOC7619). The approach may be extended to unsaturated systems as illustrated by the α -ethoxyallyl compound **84**, which acts as an equivalent of $\text{CH}_2=\text{CH}-\text{CO}^-$ for addition to alkyl halides, aldehydes, α,β -unsaturated esters, and α,β -unsaturated ketones (95JOC7589), and ketones (95JOC7597). If the initial adducts **85** are treated with a Grignard reagent rather than simply being hydrolyzed, conjugate addition occurs to give $\text{RCH}_2\text{CH}_2\text{CO}-\text{E}$ after hydrolysis, thus adding a further element of versatility to the method (95JOC7605). The related reagent **86** acts as a hex-2-enoyl anion equivalent for addition to alkyl halides, aldehydes, ketones, imines, and α,β -unsaturated ketones (97JOC706) while the alkynoyl anion equivalents **87** add effectively to aldehydes, ketones, imines, esters, carbamates, isocyanates, and silyl halides (95JOC7612). By using esters as the electrophiles with either **86** or **87**, unsaturated 1,2-diketones can be produced (97JOC4125).



IV. Six-Membered Rings

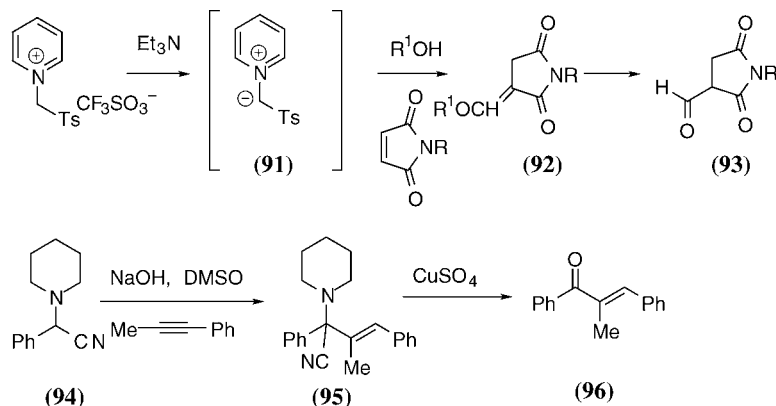
A. DIHYDROPYRANS

Dihydropyrans **88–90** are deprotonated at the vinylic position adjacent to oxygen by $t\text{-BuLi}$ and the resulting anions add readily to alkyl halides, aldehydes, and ketones. Subsequent acid hydrolysis provides the products expected from reaction of an ω -functionalized pentanoyl anion: **88** acts as $\text{HOCH}_2(\text{CH}_2)_3\text{CO}^-$, **89** as $\text{OCH}(\text{CH}_2)_3\text{CO}^-$, and **90** as $\text{MeCO}(\text{CH}_2)_3\text{CO}^-$ (77TL4187; 81T3997).



B. PYRIDINES AND PIPERIDINES

Pyridinium *p*-toluenesulfonylmethylide **91** has been used as a formyl anion equivalent for conjugate addition to *N*-substituted maleimides to give the enol ethers **92**, which were readily deprotected to give the aldehydes **93** (80TL705).

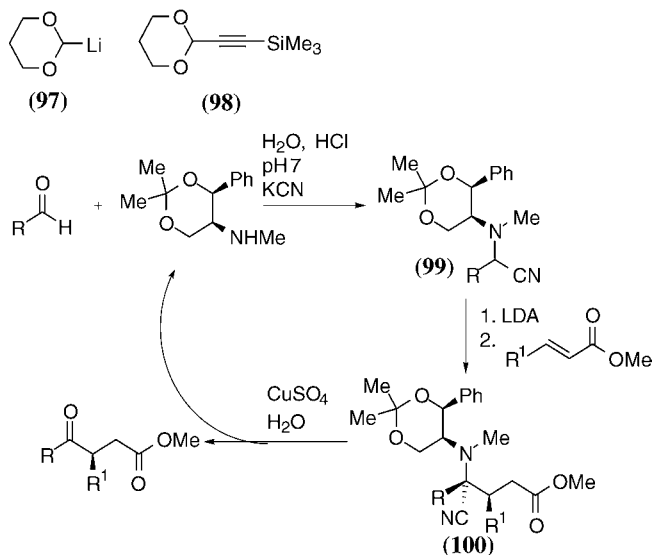


The cyanobenzylpiperidine **94** acts as an effective benzoyl anion equivalent for addition to alkynes. As shown, the initial adduct **95** is readily cleaved with copper sulfate to give the ketone product **96** (93LA375).

C. 1,3-DIOXANES

2-Lithio-1,3-dioxane **97** is readily prepared either by treatment of the 2-phenylthio compound with lithium naphthalenide or by transmetallation of the 2-tributylstannyl compound with BuLi and acts as an effective formyl anion equivalent for addition to alkyl halides, aldehydes, ketones, and epoxides (89JA1381). With cyclohexenone, **97** undergoes exclusive 1,2-addition, but this may be diverted entirely toward 1,4-addition by transmetallation with $\text{CuI}/\text{Bu}_3\text{P}$. The trimethylsilyldioxane **98** provides an effective equivalent of the $\text{Me}_3\text{Si}-\text{C}\equiv\text{C}-\text{CO}^-$ anion for addition to alkyl halides and epoxides as well as trimethylsilyl-, -germyl, and -stannyl chlorides (83T3073). Treatment with butyllithium followed by the electrophile and finally acid hydrolysis gives the alkynyl ketone products in good yield, although in some cases the conditions have to be carefully controlled to avoid the formation of allene-derived products.

Among the most successful classes of asymmetric acyl anion equivalents are the dioxane-containing α -amino nitriles **99** introduced by Enders and coworkers. These are deprotonated by LDA , and the resulting anions act as efficient equivalents of RCO^- for addition to α,β -unsaturated esters [90AG(E)179],

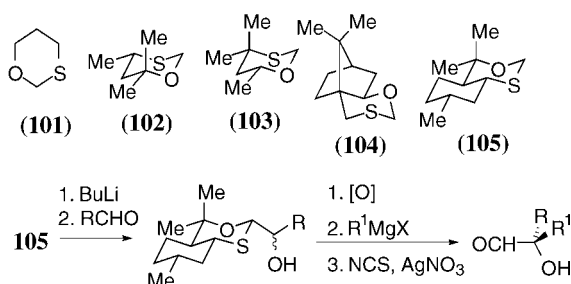


cyclohexenone (92SL837; 95S659), and butenolide (96LA1361). The stereoselectivity obtained is usually excellent; but even if it is not, the adducts **100** can be recrystallized to effect diastereomeric enrichment before the auxiliary is removed by simple treatment with copper sulfate or silver nitrate to give the final products in over 95% e.e. This regenerates the amine required to prepare **99**, which can be reused repeatedly. A further attractive feature of the method is that for cyclohexenone and α,β -unsaturated lactones the enolates resulting from conjugate addition may be trapped by adding an electrophile to afford final products with two new stereogenic centers (95S659; 96LA1361; 96T10083). The structure and reactivity of the lithiated species has recently been examined in detail, lead to a good understanding of the mechanism of these efficient asymmetric reactions (98EJOC63).

D. 1,3-OXATHIANES

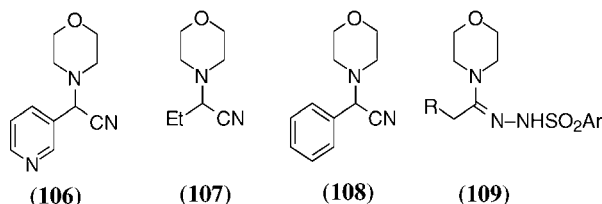
1,3-Oxathiane **101** is readily deprotonated using *s*-BuLi, and the resulting anion reacts with alkyl halides, ketones, and benzonitrile (85JOC657). The majority of work in this area, however, is due to Eliel and coworkers and has involved chiral 1,3-oxathianes as asymmetric acyl anion equivalents. In the earliest work it was demonstrated that the oxathianes **102** and **103**, obtained in enantiomerically pure form by a sequence involving resolution, could be deprotonated with butyllithium and added to benzaldehyde. The products were formed with poor selectivity at the new stereocenter, however, and oxidation followed by addition

of a Grignard reagent was required to improve this before deprotection to give the α -hydroxyaldehyde products in excellent e.e. (78JA1614; 84JA2937). Later, the more readily accessible oxathianes **104** (79JOC3598) and **105** (81TL2855; 84JA2943; 87OS215), derived respectively from camphorsulfonic acid and pulegone, were introduced; but these again gave poor selectivity upon addition to aldehydes, and the situation was salvaged only by oxidation and addition of a Grignard reagent or re-reduction (84T1333) to eventually give α -hydroxyaldehyde products. Although these oxathianes may only be considered to act as formyl anion equivalents in an indirect way, there has been considerable interest in their use. Newer developments include improved syntheses of 2-acyloxathianes (90JOC4951; 93JOC2920) and use of lanthanide salts to improve selectivity (90JA8189; 95SL501), and the method has been used in syntheses of several important natural products (81TL2859; 85TL3907; 90JOC5625; 91JOC2086).



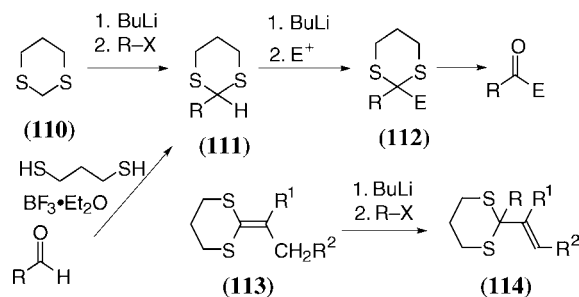
E. TETRAHYDRO-1,4-OXAZINES

There are several examples of the use of α -cyanoalkyltetrahydro-1,4-oxazines (morpholines) as acyl anion equivalents. The pyridine compound **106**, for example, is deprotonated by KOH and the resulting anion undergoes conjugate addition to acrylonitrile (72JOC4465) and acrylophenone (76JOC3438) to give, after simple acid hydrolysis, functionalized 3-acylpyridines of value for synthesis of nicotine metabolites. The simpler alkyl compound **107** may be deprotonated and alkylated with alkyl halides to provide a convenient synthesis of ethyl ketones (79S127). The anion of the phenyl compound **108** acts as a benzoyl anion equivalent for reaction with acrylonitrile, ethyl acrylate, acid chlorides, and activated haloarenes to give phenyl ketones upon acid hydrolysis (79JOC4597), this method has also been used in the synthesis of the alkaloid anibine (89JOC3985). Finally, the amidrazones **109** act as equivalents of RCH_2CO^- for addition to alkyl halides and ketones by treatment with *t*-BuLi followed by the electrophile and then water [81JCS(CC)1121]. The reaction is likely to involve an intermediate α -lithioenamine.



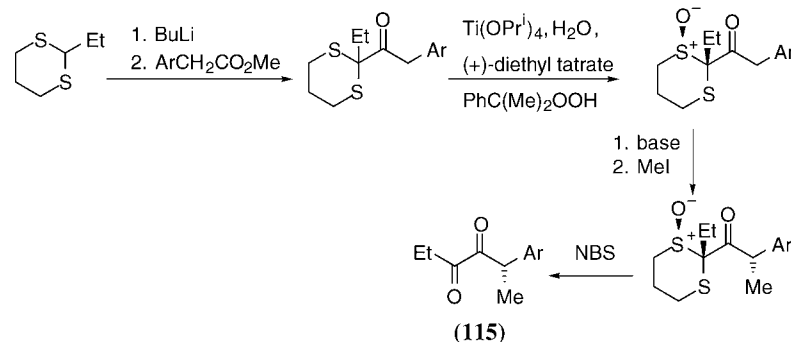
F. 1,3-DITHIANES AND THEIR S-OXIDES

The 1,3-dithianes are perhaps the classic heterocyclic acyl anion equivalents. Since their introduction by Corey and Seebach [65AG(E)1075; 65AG(E)1077], they have found widespread use as standard tools of the synthetic chemist as described in detail in a number of reviews (69S17; 75JOC231; 77S357; 89T7643). The parent 1,3-dithiane **110** is readily deprotonated at the 2 position with butyllithium and the resulting anion can be alkylated to give **111**; alternatively, treatment of an aldehyde with propane-1,3-dithiol in the presence of BF_3 diethyl etherate gives **111** directly. This can be deprotonated and reacted with almost all major classes of electrophiles to give the products **112**. The original method of deprotection to give the ketone products involved treatment with a mercury salt, but numerous alternative methods avoiding this have now been developed, including *N*-halosuccinimides (71JOC3553), sulfuryl chloride and wet silica gel (76S678), and bis(trifluoroacetoxy)iodobenzene (89TL287). Some electrophiles of particular significance that have been used are D_2O , which allowed the first highly efficient one-pot synthesis of deuterioaldehydes (66JOC4303); trialkylsilyl and -germyl halides, which gave convenient access to silyl and germlyl ketones (67JA431; 67JA434); and enantiomerically pure alkyl halides giving chiral aldehydes and ketones [68AG(E)619; 68AG(E)620]. A dependable large-scale procedure for the use of dithianes has been described (71OS76). One practical difficulty that has not been overcome is the foul odor of the propanedithiol used in the preparation of dithianes.



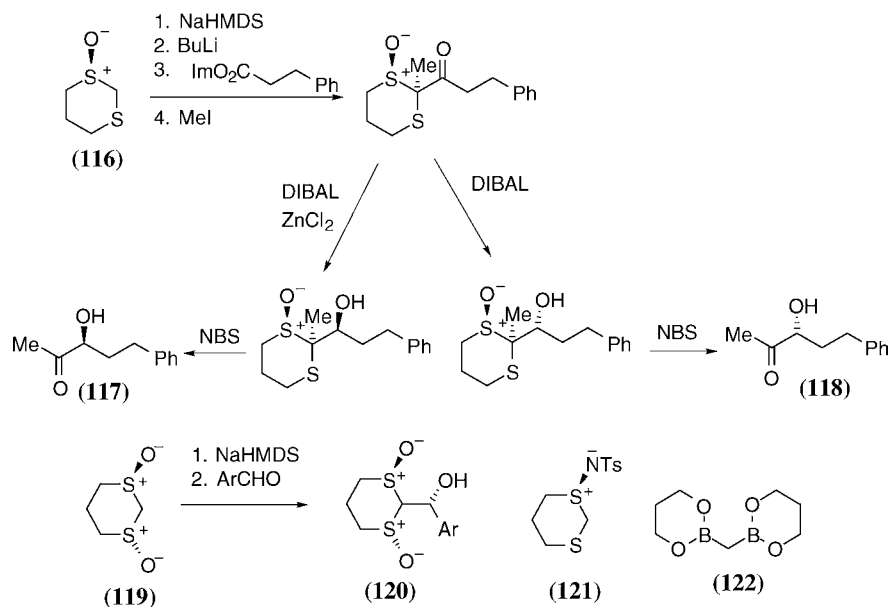
The alkylidenedithianes **113** are readily formed by treatment of methyl esters $R^2CH_2CH(R^1)CO_2Me$ with $Me_2AlS(CH_2)_3SAlMe_2$ and undergo deprotonation and alkylation at the dithiane carbon to give **114**, thus making them the equivalent of $R^2CH=C(R^1)CO^-$ (75TL925). The conformational properties of 2-lithio-1,3-dithianes have been extensively studied by labeling (74JA1807), NMR (77JA8262), and theoretical methods (97JA7545), and the great preference they show for the equatorial lithium conformation is well understood.

There has been no success in adapting chiral 1,3-dithianes to achieve asymmetric synthesis, and this led Eliel to examine the 1,3-oxathianes, as described in Section IV.D. More recently, however, there has been a good deal of work involving both mono- and disulfoxides of 1,3-dithiane in stereoselective synthesis. The work of Page has focused on 1,3-dithiane monoxides as chiral auxiliary groups for stereoselective alkylation [95JCS(P1)2673] or reduction (96TL8929) of a 2-acyl substituent which may then be liberated to give the chiral 1,2-dione or α -hydroxyketone products. Two examples of this approach are provided by the syntheses of **115** and of **117** and **118**, although it should be noted that the acyl anion character of the dithiane is only involved in assembling the starting material for the key asymmetric step in each case. A dependable large-scale procedure for the preparation of the starting dithiane oxide **116** has recently appeared (99OS37).



Aggarwal's group has developed the C_2 -symmetric 1,3-dithiane 1,3-dioxide **119** as an effective formyl anion equivalent for addition to aldehydes; but in order to achieve high diastereoselectivity, it is necessary to use an aromatic aldehyde and to carry out the reaction under conditions of thermodynamic control to give the products **120** in up to 94% d.e. [90TL135; 91TL7743; 94JCS(CC)1653; 95JOC2174; 97T16213].

The related 1,3-dithiane *S*-imides **121** have also been examined but were found not to be synthetically useful [96JCS(P1)313].

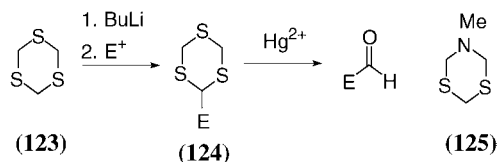


G. DIOXABOROLINES

The methylenebis(boronic acid) **122** may be deprotonated and alkylated at the central position and may thus behave as an acyl anion equivalent. Monoalkylation of **122** followed by hydrolysis gives aldehydes in good yield, and a second alkylation led to a ketone in one case (77JA3196).

H. TRITHIANES AND DIHYDRODITHIAZINES

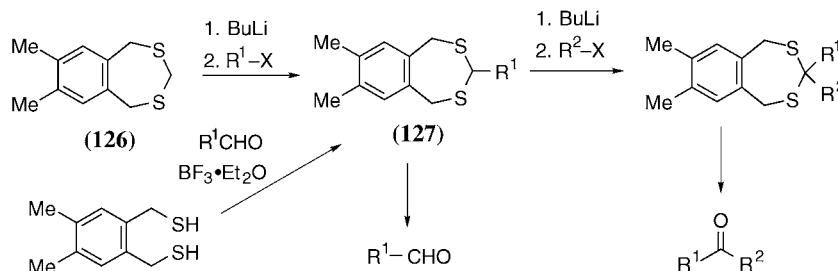
Like 1,3-dithiolane, 1,3,5-trithiolane **123** is readily deprotonated by butyllithium, and the resulting anion reacts with electrophiles such as alkyl halides, ketones, epoxides, nitriles, chloroformates, and trialkylsilyl halides. Hydrolysis of the adducts **124** to give the corresponding aldehydes is readily achieved with mercury salts (74CB367). A dependable large-scale procedure for the use of trithianes has been described (71OS39). It should be noted that a second alkylation of **124** cannot be used to prepare ketones because deprotonation occurs at one of the unsubstituted positions, but further alkylation of a symmetric 1,3,5-trialkyltrithiolane to give a ketone upon hydrolysis has been demonstrated in one case. Hydrolysis of trithianes is also possible using iodine in DMSO (73TL3735).



The *N*-methyldihydrodithiazine **125** has also been used as an effective formyl anion equivalent for reaction with alkyl halides, aldehydes, and ketones (77JOC393). In this case there is exclusive alkylation between the two sulfur atoms, and hydrolysis to give the aldehyde products is considerably easier than for dithianes. However, attempts to achieve a second alkylation at C2 were unsuccessful, thus ruling out the use of this system as an acyl anion equivalent for synthesis of ketones. Despite this limitation, the compound has found some use in synthesis (82TL4995).

V. Aromatic Fused Dithiepinines and Their S-Oxides

The benzodithiepinines **126** and **127** have been used as formyl and acyl anion equivalents, respectively, although the range of electrophiles was restricted to alkyl halides (75S720) and epifluorohydrin (72TL1837). The carbonyl products were formed by hydrolysis with either mercury or copper salts.



There has been recent interest in naphtho-fused dithiepinines as chiral acyl anion equivalents, particularly since the starting dithiol **128** can be obtained in enantiomerically pure form (89TL2575). This is transformed using standard methods into the dithiepinine **129**, but showed only moderate diastereoselectivity in its addition to carbonyl compounds. On the other hand, as we have seen previously for other systems, formation of the 2-acyl compound **130** and reduction or addition of a Grignard reagent gave the products **131** with much better stereoselectivity (91JOC4467).

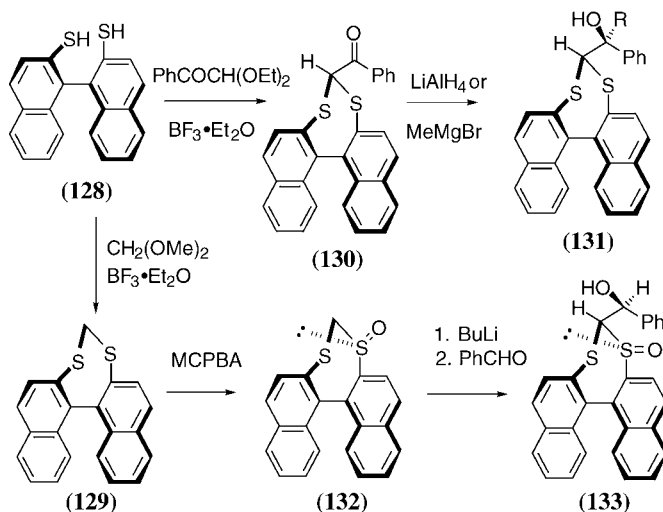
In contrast to this, the monosulfoxide **132**, readily prepared from **129** by oxidation, acts as a highly diastereoselective formyl anion equivalent and adds to

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benzaldehyde to give the product **133** as a single diastereomer in 92% yield [89JCS(CC)411; 91JOC4467].



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 65AG(E)1077
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 66JOC4303
 67JA431
 67JA434
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 68AG(E)620
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ADVANCES IN HETEROCYCLIC CHEMISTRY, VOL. 79

Organometallic Compounds of Pyrrole, Indole, Carbazole, Phospholes, Siloles, and Boroles

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Abbreviations: Alk, alkyl; AN, acetonitrile; Ar, aryl; Bu, butyl; cod, 1,5-cyclooctadiene; Cp, cyclopentadienyl; Cp*, pentamethylcyclopentadienyl; Cy, cyclohexyl; dppm, diphenylphosphinomethane; dpme, Ph₂PC₂H₄PMe₂; Et, ethyl; fod, 6,6,7,7,8,8,8-heptafluoro-2,2-dimethyl-3,5-octanedionate; HOMO, highest occupied molecular orbital; LUMO, lowest unoccupied molecular orbital; Me, methyl; MO, molecular orbital; nbd, norbornadiene; Nuc, nucleophile; OTf, triflate; Ph, phenyl; Pr, propyl; py, pyridine; THF, tetrahydrofuran; TMEDA, *N,N,N',N'*-tetramethylethylenediamine.

I. Introduction

This chapter is a continuation of the one on organometallic chemistry of furans and thiophenes [01AHC(78)1]. It starts with the trends of coordination of

pyrrole and benzannulated derivatives. Material presented for azaferrocene serves as a classical subsection on the modification of reactivity of this heterocycle in the complexed state. Phospholes offer a wide versatility of coordination modes and reactivity patterns, especially in the case of phosphacymantrene, phosphoferrocene, and diphosphoferrocene. Organometallic complexes of silole, germole, and borole are still regarded as a rarity, although achievements in this field are already noticeable.

II. Organometallic Compounds of Pyrrole

A. INTRODUCTORY REMARKS

Pyrrole is a classical example of a π -excessive heterocycle in which a nitrogen atom can supply two electrons to the heteroring, giving six electrons per five carbon atoms. It has a negative net π -electron charge of the heteroring and the average π charge per carbon atom. Thus, it fulfills a predominant π -donor function followed by formation of radicals and radical ions of π type (high energy of the HOMO). These features follow from the quantum chemical studies of pyrrole and its simple derivatives (69TCA278; 70T4505; 71TCA52; 78CPL224; 83JMS115). Abstraction of the hydrogen atom leads to the pyrrolate anion bearing a lone σ -electron pair on the nitrogen atom called pyrrolic nitrogen. The nature of an electron to be photodetached from the pyrrolate system is dual. It can be a σ electron belonging to the nonbonding lone pair or a π electron. According to calculations, π electrons are more weakly bound, but experiment favors further electron reorganization to give the σ radical (75JA1160). The general trend is thus the $\eta^5(\pi)$ -donor function of pyrrole. Although pyrrole and cyclopentadienyl anions are isoelectronic, the presence of the sp^2 -hybridized nitrogen atom in pyrrole leads to a wider variety of its possible coordination modes [78JHC1057; 87CCR279; 87JOM(330)231; 92MI1]. The predicted η^5 complexes are, as a rule, unstable and difficult for structural studies. This may be due to the fact that the ionization potential of the nitrogen lone pair is less than that of π electrons. This opens a possibility for $\sigma(\text{N})$ -coordinated or polynuclear complexes, or η^2 -bridged structures.

Comparison of the reactivity pattern for the uncoordinated and coordinated pyrroles may be of interest. Therefore, we shall very briefly characterize the trends in protonation and electrophilic substitution in the free pyrrole and later compare them with those in the corresponding organometallic species. The longstanding discussion concerns the direction of protonation of pyrrole [68MI1; 70AHC383; 74MI1; 75T915; 77MI1; 78T275; 79JCS(P2)1627; 81LAC789; 81NJC505; 82JA7084; 82JA7091; 90JA1678; 96MI1]. Experimental data show that position 2

is the most attractive protonation site in solution. Theoretical data, however, inevitably indicate that position 3 acquires a higher electronic charge [68IJQC165; 73IJQC(S)207; 73IJQC(S)249; 79NJC473; 81JMS163]. Later, some experiments (84JA37) indicated the possibility of C3 protonation, or simultaneous protonation at both C2 and C3 (96JMSP1173). Calculations also did not necessarily favor the C3 position. The semiempirical approach predicted C2 as the most probable protonation site [93JMS(T)199]. *Ab initio* calculations in a rigorous 6-311G(d) basis set with electron correlation point to a possibility of simultaneous protonation at C2 and C3 but definitely not at nitrogen (96JMSP1173).

Electrophilic additions to free pyrrole are really restricted to position 2 (84MI1; 90MI1). Direct addition to position C3, which is of biological importance, is extremely difficult. The problem of derivatization of pyrrole has been solved by the η^2 coordination of the $\text{Os}(\text{NH}_3)_5^{2+}$ fragment, allowing remarkable transformations of the pyrrole ring. This series of work is considered under organoosmium derivatives, where it serves as an illustration that dearomatization of the pyrrole ring induced by the η^2 coordination modifies the reactivity of this heterocycle substantially. Reactivity of the η^5 -coordinated pyrrole has been reviewed as well (87CCR279; 90BSCB707; 90H383; 93AHC123).

Nontransition metal cyclopentadienyl derivatives of pyrrole are quite scarce (99CRV969). The derivatives $\text{M}(2,5\text{-C}_4\text{H}_2\text{Bu}_2\text{N})_2$ ($\text{M} = \text{Sn}, \text{Pb}$) were studied [92AG(E)778; 92JCS(CC)760].

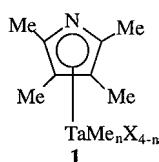
B. COMPLEXES OF TITANIUM, ZIRCONIUM, AND TANTALUM

Interaction of Cp_2MCl_2 ($\text{M} = \text{Ti}, \text{Zr}$) with sodium pyrrolate leads to the formation of $\text{Cp}_2\text{M}(\eta^1\text{-NC}_4\text{H}_4)_2$ ($\text{M} = \text{Ti}, \text{Zr}$), where the π -electron density is localized in the pyrrolate ligands [80IC2368; 87JOM(321)143]. The cyclopentadienyl ligands in the zirconium complex can be easily eliminated to form $[\text{Na}(\text{THF})_6]_2[\text{Zr}(\eta^1\text{-NC}_4\text{H}_4)_6]$. These data indicate less π -donor nature and more σ -donor property of the pyrrolate ion in comparison with the cyclopentadienyl ion. In the cyclopentadienylbis(2,5-dimethylpyrrolyl)zirconium complex obtained by direct interaction of $\text{NaNc}_4\text{H}_2\text{Me}_2$ with Cp_2ZrCl_2 , the methyl groups weaken the Zr-N bond, but the η^1 coordination is retained (86CJC1304).

There are, however, illustrations of the η^5 coordination. Thus, 2,3,4,5-tetramethylpyrrole and titanium tetrachloride give $[\text{Ti}(\eta^5\text{-NC}_4\text{Me}_4)\text{Cl}_3]$ [92JOM(440)289]. The lithium salt of this heterocycle and TiCl_4 gives $[\text{Ti}(\eta^5\text{-NC}_4\text{Me}_4)\text{Cl}_3]$ and $[\text{Ti}(\eta^5\text{-NC}_4\text{Me}_4)_2\text{Cl}_2]$ (97JCS(D)1055). The first of these products substitutes chlorine by benzenethiolate groups, giving both $[\text{Ti}(\eta^5\text{-NC}_4\text{Me}_4)(\text{SPh})\text{Cl}_2]$ and $[\text{Ti}(\eta^5\text{-NC}_4\text{Me}_4)(\text{SPh})_3]$. Lithium pyrrolate leads to the η^5 -pyrrolate complex $[\text{CpTi}(\eta^5\text{-NC}_4\text{Me}_4)\text{Cl}_2]$ even with CpTiCl_2 . The η^5 coordination was also

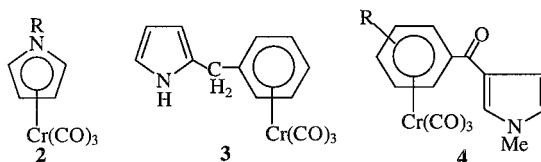
postulated in this complex earlier but without essential proof [82JOM(238)177; 84ACH361].

Lithium tetramethylpyrrolate forms the η^5 complexes **1** ($n = 3$, $X = \text{Cl}$) and **1** ($n = 2$, $X = \text{Cl}$) reacting with TaMe_3Cl_2 (96IC3228). Complex **1** ($n = 3$, $X = \text{Cl}$) enters a reaction with LiX ($X = \text{Me}$, pyrrolyl, indolyl) to yield **1** ($n = 3$, $X = \text{Me}$, *N*-pyrrolyl, *N*-indolyl), the latter two products being the mixed coordinated $\eta^5:\eta^1$ species. Both of them undergo thermolysis to give **1** ($n = 2$, $X = \text{N-pyrrolyl}$, *N*-indolyl).



C. GROUP VI METAL COMPLEXES

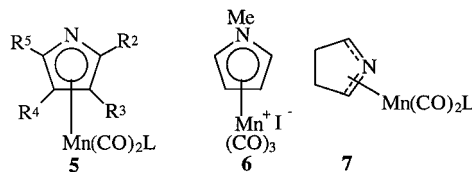
N-Methylpyrrole and $\text{Cr}(\text{CO})_3(\text{AN})_3$ give **2** ($R = \text{Me}$) [71JOM(30)211; 72CB301; 94JOM(470)C4]. Pentamethylpyrrole and tetracarbonyl(norbornadiene)chromium(0) also give the η^5 -coordinated complex, where the ligand reveals stronger donor ability than *N*-methylpyrrole [93JOM(459)125]. Pyrrole and 1-phenylpyrrole form the η^5 -coordinated tricarbonylchromium complexes **2** ($R = \text{H}$, Ph), while 2-benzylpyrrole produces the η^6 -coordinated species **3** [86JOM(317)55; 90JCR(S)64]. Reaction with 3-benzoylpyrrole also gives the π complexes, in which the $\text{Cr}(\text{CO})_3$ framework is bonded to the benzene ring, **4** ($R = \text{H}$, *p*-Me, *o*-Me, *p*-OMe, *p*-Cl) (89CJC433). Radical anions of these complexes are obtained by treating **4** with potassium [87JOM(336)137].



Reaction between $[\text{W}(\text{RC}\equiv\text{C})\text{Cl}(\text{CO})_2(\text{py})_2]$ ($R = \text{Ph}$, Me) with the anionic chelating Schiff base pyrrole-2-carboxaldehyde methylimine yields the cationic complexes $[\text{NEt}_4][\text{W}(\text{RCCO})(\text{NN})_2(\text{CO})]$ (where NN is the dianion of the pyrrole ligand). These complexes react with methyltriflate, forming the neutral acetylenic complexes $[\text{W}(\text{NN})_2(\text{CO})(\text{RC}\equiv\text{COMe})]$ (87OM1503). One of the pyrrolic Schiff bases is coordinated via the pyrrole and imino nitrogen atoms, and another one only via the imino nitrogen atom.

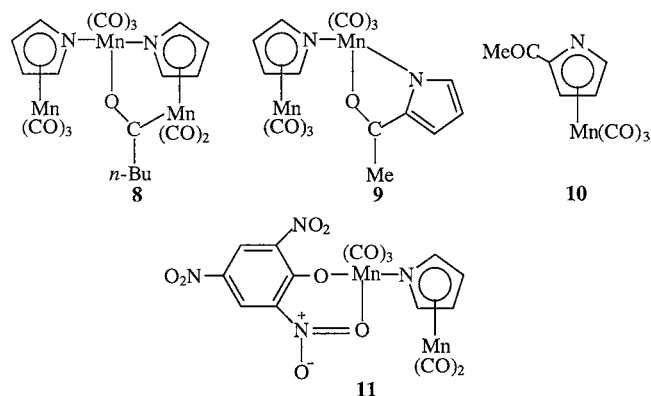
D. GROUP VII METAL COMPLEXES

Tricarbonylmanganese derivatives of pyrrole, **5** ($L = \text{CO}$; $R^2 = R^3 = R^4 = R^5 = \text{H}$; $R^2 = \text{Me}$, $R^3 = R^4 = R^5 = \text{H}$; $R^2 = R^5 = \text{Me}$, $R^3 = R^4 = \text{H}$), called azacymantrenes, are obtained as a result of direct interaction of the corresponding pyrrole or potassium pyrrolate with $\text{BrMn}(\text{CO})_5$ [62PCS326; 64JOM(1)471; 69JOM(20)-264]. Complexes of the unsubstituted pyrrole are much more stable than the cyclopentadienyl analogs. The pyrrolate derivative is a better σ acceptor than cyclopentadienyl [74JOM(77)69]. The weakly basic manganese complex **5** ($R^2 = R^3 = R^4 = R^5 = \text{H}$) does not react with methyl iodide under mild conditions. The salt **6** is obtained by reacting $\text{Mn}(\text{CO})_5\text{I}$ with *N*-methylpyrrole in the presence of aluminium chloride [69JOM(20)264]. Substitution reactions at the carbonyl group in azacymantrene and its 2,5- and 3,4-dimethyl derivatives proceed through intermediate **7** (79CB2423). The pyrrolyl compound reacts much faster than the cyclopentadienyl compound owing to the electron-donor effect of the nitrogen atom [85JOM(296)83; 88PAC1193; 90P1503]. Reaction of azacymantrene with triphenylphosphine yields the monosubstituted product of the carbonyl group **5** ($L = \text{PPh}_3$, $R^2 = R^3 = R^4 = R^5 = \text{H}$) [85JOM(297)69].

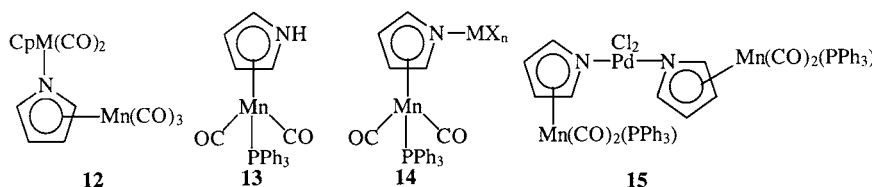


Azacymantrene **5** ($L = \text{CO}$, $R^2 = R^3 = R^4 = R^5 = \text{H}$) was treated with *n*-butyllithium, then the reaction mixture was elaborated with heavy water to locate the position of the lithium atom. Lithium attacks not the pyrrolyl nitrogen but the carbonyl group followed by the formation of the trinuclear complex **8** [73DAN367; 75DAN123; 81JOM(206)169]. Acylation of azacymantrene by acetic anhydride in the presence of phosphoric acid gives **9** rather than **10** [77JOM(128)381]. The complex **9** contains π , σ , donor-acceptor, and chelate bonds with the azacymantrene molecule as a two-electron ligand. To elucidate the nature of the protonation site, an attempt was made to prepare a crystalline product from the interaction of the manganese complex and picric acid. Species **11** was obtained among other products [81JOM(206)177]. Earlier indications of such type of interaction are known [67JOM(7)325].

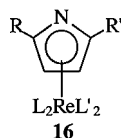
The nitrogen atom in η^5 -pyrrolylmanganesetricarbonyl forms a donor-acceptor bond with transition metals. Complexes in which the pyrrolyl ring behaves as a π ligand for the manganese atom and *n*-donor for the other metal were synthesized: **12** ($M = \text{Mn}$, Re) [78JOM(157)431]. The binuclear heterobimetallic complexes



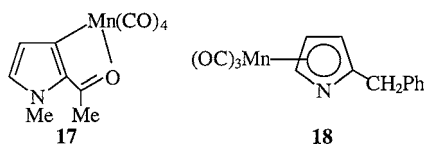
of chromium, molybdenum, and tungsten hexacarbonyls with η^5 -pyrrolylmanganetricarbonyl are known as well [81IZV654]. In acidic medium, the complex $(OC)_5W \cdot C_4H_4Mn(CO)_3$ is protonated at the nitrogen atom, which is indicative of the high donor property of azacymantrene as ligand. Attack of electrophile on **5** ($R^2 = R^3 = R^4 = R^5 = H$, $L = PPh_3$) may be directed toward a series of centers of basicity: the pyrrolic nitrogen atom, manganese atom, and so on. With trifluoroacetic acid, the product of proton addition over the pyrrolic nitrogen, **13**, is obtained. Reaction of **5** ($R^2 = R^3 = R^4 = R^5 = H$, $L = PPh_3$) with aprotic acids, HgX_2 ($X = Cl, OCOMe$), $M(OCOCF_3)_2$ ($M = Hg, Cd$), ZnX_2 , AlX_3 , SnX_4 ($X = Cl, Br$), $GaCl_3$, and $SbCl_3$ leads to the coordination of the latter, again via the pyrrolic nitrogen, **14**. The same situation is realized in the case of $PdCl_2$, which gives **15**.



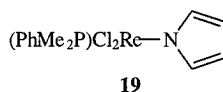
Rhenium heptahydride, $(Ph_3P)_2ReH_7$, reacts with pyrrole in the presence of 3,3-dimethyl-1-butene followed by formation of **16** ($L'_2 = H_2$, $R = R' = H$) [85JA3374; 87JOM(326)C17; 87JOM(337)71; 89JOM(363)C31; 90H383]. In this complex not only does the substitution of the hydride anions by other ligands take place, but also C2 substitution of the pyrrole ring is possible, so that a variety of η^5 -pyrrolyl half-sandwich complexes may be obtained. Treatment of **16** ($L'_2 = H_2$, $R = R' = H$) with iodine in excess potassium carbonate gives **16** ($L'_2 = HI$, $R = R' = H$), with phenyllithium forms **16** ($L'_2 = H_2$, $R = Ph$, $R' = H$) with subsequent formation of **16** ($L'_2 = H_2$, $R = R' = Ph$).



For the substituted pyrroles, π -complex formation is limited due to the stabilization of the σ complexes. Complexes of manganese containing pyrrole bearing an acetyl group such as $\text{Mn}(\text{CO})_3\text{L}$ were synthesized, where $\text{L} = 3\text{-acetyl-2-methylpyrrole}$ (87JA7396; 87OM196), and the *o*-manganated 2-acetyl-1-methylpyrrole, **17**, were isolated [88JOM(349)197]. Unlike the chromium complex **3**, which coordinates via the phenyl ring of 2-benzylpyrrole, interaction of $\text{Mn}(\text{CO})_5\text{Br}$ with the potassium salt of the pyrrole derivative leads to 2-benzyl- η^5 -pyrrolylmanganesetricarbonyl, **18** [70JCS(CC)1414; 86JOM(317)55].



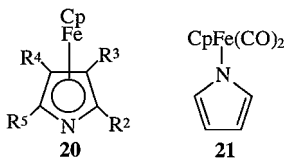
The $\eta^1(\text{N})$ coordinated rhenium complex $[\text{CpRe}(\text{PPh}_3)(\text{NO})(\text{NC}_4\text{H}_4)]$ is known (93OM4728; 94OM60). It was protonated by the strong acids to position 3 of the heteroring. However, the product undergoes isomerization followed by the re-switch of the coordination mode to the $\eta^1(\text{C})$ and migration of the proton partly to C3 and partly to the C5 sites. Lithium pyrrolate and $(\text{PMe}_2\text{Ph})_3\text{ReCl}_3$ give **19** (99OM2230). This compound is interesting in the sense that, formally, this is the 1-substituted pyrrole where the N-functional group is bulky and directs electrophilic substitution and Michael addition to position 3 of the heteroring. However, the protonation basically goes to position 2.



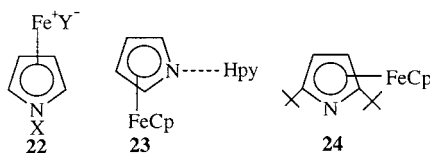
E. COMPLEXES OF IRON

Dicarbonylcyclopentadienyliron iodide reacts with potassium pyrrolate to form azaferrocene **20** ($\text{R}^2 = \text{R}^3 = \text{R}^4 = \text{R}^5 = \text{H}$; $\text{R}^2 = \text{Me}$, $\text{R}^3 = \text{R}^4 = \text{R}^5 = \text{H}$; $\text{R}^2 = \text{R}^5 = \text{Me}$, $\text{R}^3 = \text{R}^4 = \text{H}$) [64JOM(1)471; 68JOM(14)405; 70JINC441; 84JA1646; 86JCS(F2)1543; 90JOM(388)175]. During the formation of **20**, it is possible to anticipate the existence of an intermediate σ -pyrrolate complex **21**, which then

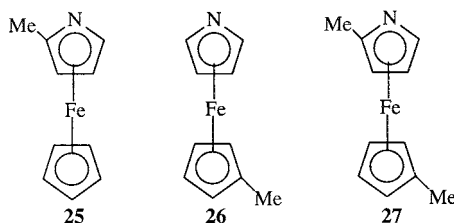
eliminates CO to give the π complex [67JOM(7)321]. The intermediate can be isolated if the reaction is conducted under mild conditions.



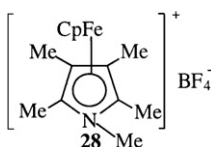
Azaferrocene is very soluble in picric acid, giving picrate **22** ($X = \text{H}$, $Y = \text{C}_6\text{H}_2(\text{NO}_2)_3\text{O}^-$), which reacts with methyl iodide to form an unstable salt **22** ($X = \text{Me}$, $Y = \text{I}$) [64JOM(1)471]. The nitrogen atom changes its hybridization from sp^2 in free pyrrole to sp^3 in azaferrocene upon complex formation. Protonation and quaternization occur at the heteroatom. The N-coordinated pentacarbonyl-tungsten adduct of azaferrocene is known [93JOM(456)107]. Parent azaferrocene is labile and can be stabilized, either by engaging the nitrogen lone pairs as a result of the hydrogen bonding with pyridinium, **23**, or introduction of the sterically bulky substituents to positions 2 and 5 of the heteroring, **24** [89CB1891; 91CB89; 93JOM(456)97]. The nitrogen atom has a basic character and with methyl iodide, borane, acetyl chloride, and $\text{Fe}_2(\text{CO})_9$, forming the N-Me , N-BH_3 , N-COMe , and N-Fe(CO)_4 species.



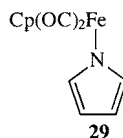
Azaferrocene is methylated by *n*-butyllithium with subsequent treatment with methyl iodide resulting in formation of **25–27** [83JOM(251)C41]. The acetylated complex η^5 -(3-acetyl-2,4-dimethylpyrrolyl)cyclopentadienyliron was also described [74JOM(77)69]. The structure of 2-methylazaferrocene was studied extensively (69AG150; 96JOC7230; 97JA1492; 97JOC444).



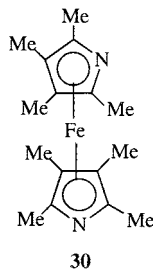
Azaferrocene reacts with aromatic hydrocarbons in the presence of aluminium chloride, giving rise to the cationic complexes of the type $(\eta^6\text{-arene})(\eta^5\text{-cyclopentadienyl})\text{iron}(1+)$ isolated as BF_4^- salts [87JOM(333)71]. The complex **28** is obtained by reaction of the sulfane compound $[\text{Cp}(\text{SMe}_2)_3\text{Fe}]\text{BF}_4$ with pentamethylpyrrole [88AG(E)579; 88AG(E)1468; 90ICA(170)155]. The metallic site in this center reveals expressed Lewis acidity (89CB1891).



Carbonylation of azaferrocene includes a $\pi \rightarrow \sigma$ rearrangement of the pyrrolyl ligand and is reversible (82IC868). The similar process under the influence of the other π -acidic ligands $[\text{PF}_3, \text{R}_2\text{NPF}_2 (\text{R} = \text{Me}, \text{Et}), \text{MeN}(\text{PF}_2)_2, t\text{-BuNC}, n\text{-PrNC}, \text{Me}_2\text{N}(\text{CH}_2)_2\text{NC}, \text{and PhNC}]$ occurs. Derivatives based on **29** are isolated.

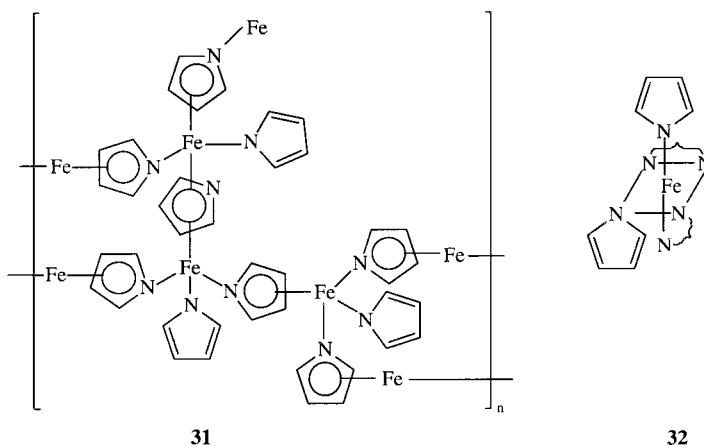


The complex $[(\eta^1\text{-C}_4\text{Me}_4\text{N})_2\text{Fe} \cdot 2\text{THF}]$ is obtained by reaction of FeCl_3 with $\text{C}_4\text{Me}_4\text{NLi}$. It decomposes in solution or by traces of water to give diazaferrocene **30** (89CB2275). Reactions with Lewis acids are also targeted at the pyrrolic nitrogen, giving rise to mono- and disubstituted derivatives (91CB997). It is interesting that silver tetrafluoroborate also attacks nitrogen sites and gives a bridged dimer. The adduct $[\text{Fe}(\eta^5\text{-NC}_4\text{Me}_4)_2] \cdot 2\text{NHC}_4\text{Me}_4$ was also described [88AG(E)342; 88AG(E)1368].



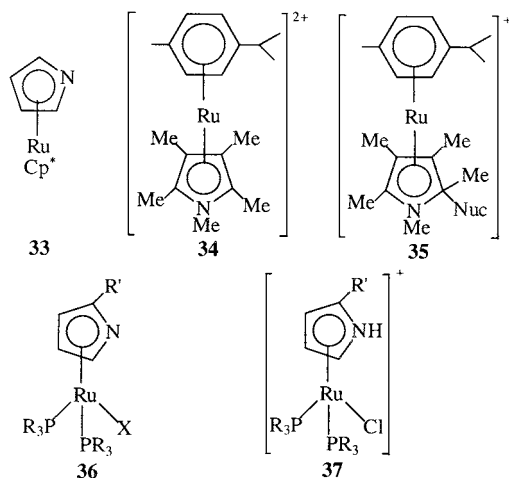
Using two synthetic routes—i.e., interaction of iron with pyrrole in the vapor phase and reaction of ferrous chloride with sodium pyrrolate— $\text{Fe}_x(\text{pyrrolate})_y$ was

obtained, for which model **31** was proposed (87JA2193). It includes π -pyrrolyl ligand simultaneously acting as the σ donor. Reaction with excess sodium cyclopentadienyl leads to the corresponding ferrocenes, while reaction with excess 2,2'-bipyridine or 3,4,7,8-tetramethyl-1,10-phenanthroline results in formation of complexes **32**.

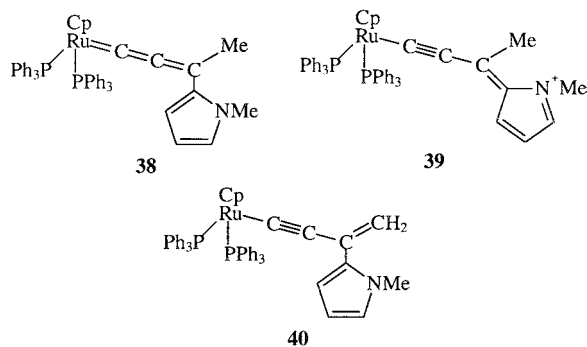


F. COMPLEXES OF RUTHENIUM AND OSMIUM

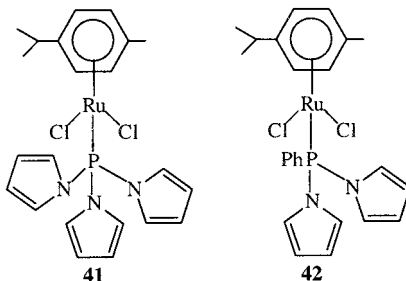
Lithium pyrrolate forms an η^5 complex, **33**, on reaction with $[\text{Cp}^*\text{RuCl}_2]_x$ (98JA7479). This species catalyzes different processes. The tetramethylpyrrolate derivative was prepared according to the same route (92OM4348). The η^5 coordination activates pyrrole and makes it susceptible to nucleophilic attacks. Thus, the ruthenium(II) species **34** experiences nucleophilic addition of H^- and OR^- ($\text{R} = \text{Alk}$) to position 2 of the heteroring, and the coordination mode changes from η^5 in **34** to η^4 in **35** ($\text{Nuc} = \text{H}, \text{OR}$) (94OM60). Lithium pyrrolate and $(\text{Ph}_3\text{P})_3\text{RuCl}_2$ give the η^5 -coordinated species **36** ($\text{X} = \text{Cl}$, $\text{R} = \text{Ph}$, $\text{R}' = \text{H}$) (97OM2325). If $\text{RuH}(\text{Cl})(\text{PPh}_3)_3$ is used, product **36** ($\text{R} = \text{Ph}$, $\text{R}' = \text{X} = \text{H}$) results. The ligand exchange with potassium iodide allows the conversion of **36** ($\text{R} = \text{Ph}$, $\text{X} = \text{Cl}$, $\text{R}' = \text{H}$) to **36** ($\text{R} = \text{Ph}$, $\text{X} = \text{I}$, $\text{R}' = \text{H}$). A similar couple of triethylphosphine complexes **36** ($\text{R} = \text{Et}$, $\text{X} = \text{Cl}, \text{I}$, $\text{R}' = \text{H}$) were also synthesized. The latter were tested for the nucleophilic substitution with alkyl- and aryllithium and found to give the ruthenium(II) hydrides **36** ($\text{R} = \text{Et}$, $\text{X} = \text{H}$, $\text{R}' = \text{Me}, \text{Ph}, \text{Me}_2\text{N}, \text{BEt}_3\text{D}$). The chloride or iodide ligands in this process were displaced by the hydrogen atom of position 2 of the heteroring. Complex **36** ($\text{R} = \text{Ph}$, $\text{R}' = \text{H}$, $\text{X} = \text{Cl}$) is protonated at the pyrrolic nitrogen to give **37**.



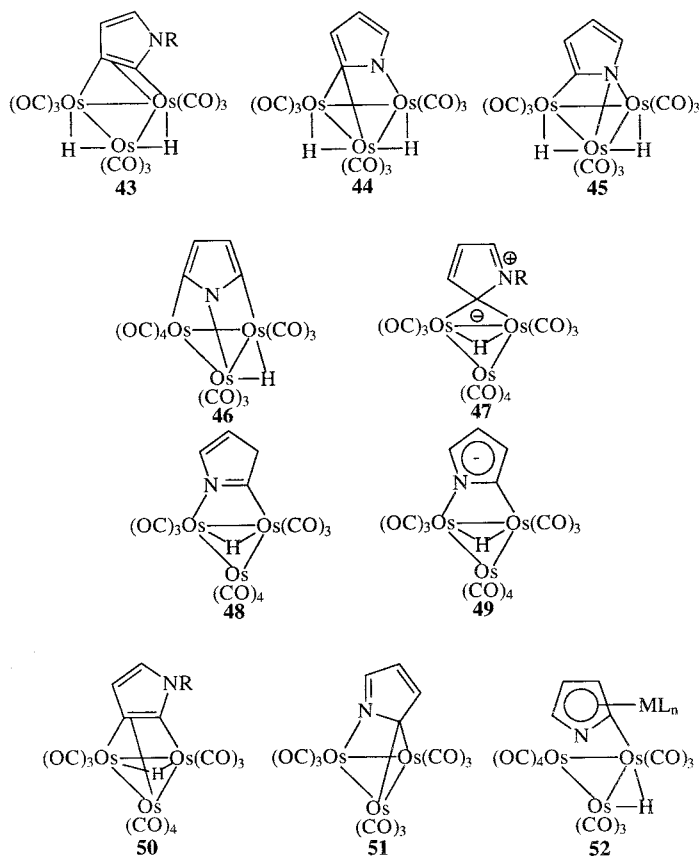
N-Methylpyrrole oxidatively adds to $[\text{Ru}(\text{C}=\text{C}=\text{C}=\text{CH}_2)(\text{PPh}_3)_2(\text{Cp})][\text{PF}_6]$ via its C2 center, the product being the allenylidene species **38** [98JCS(D)467]. Alkyne mesomeric form **39** was postulated to make a significant contribution, which explains well the nature of the deprotonated product **40**, obtained from **38** and *n*-butyllithium.



Pyrrolylphosphines act as P-donor ligands with respect to $[(p\text{-cymene})\text{RuCl}_2]_2$, as exemplified by the products **41** and **42** (95JA7696; 98OM104). The electron-withdrawing character of the pyrrolyl groups was also tested in tri-*N*-pyrrolylsilane (L) with respect to $\text{M}(\text{CO})_2(\text{PPh}_3)_3$ (M = Ru, Os) and $\text{OsCl}(\text{NO})(\text{PPh}_3)_3$ (97OM2730). The properties of the products indicate that this group reveals π -acceptor character and forms species $(\text{OC})_2\text{M}(\text{PPh}_3)_2(\text{X})(\text{L})$ (M = Ru, Os, X = H; M = Os, X = Cl) and $(\text{OC})\text{M}(\text{PPh}_3)_2(\text{Cl})(\text{L})$ (M = Ru, Os) with an unusually strong metal-phosphorus bond.

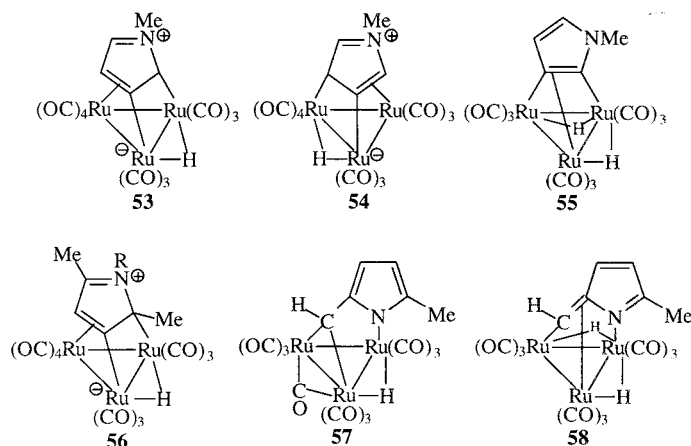


Clusters $\text{Ru}_3(\text{CO})_{12}$ and $\text{Os}_3(\text{CO})_{12}$ as well as their substitution products, e.g. $[\text{Os}_3(\text{CO})_{10}(\text{AN})_2]$, activate pyrrole and its derivatives in many different ways. Thus, dihydrides **43** ($\text{R} = \text{H}, \text{Me}$) follow from triosmium dodecacarbonyl and pyrrole or 1-methylpyrrole [82JCS(D)2563; 84P1175; 86JOM(311)371]. Complex **43** ($\text{R} = \text{H}$) isomerizes as a result of proton transfer to the more stable species **44** and

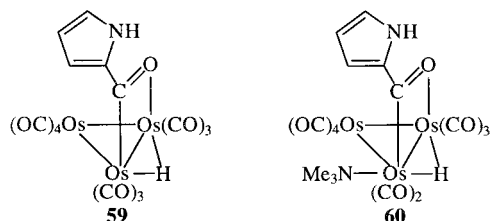


45. Compound **45** undergoes rapid and complete (N)H,D-exchange when treated by $D_2O/CDCl_3$ in the presence of catalytic amounts of triethylamine. In excess D_2O , $[Os_3H_2(CO)_9(C_4H_2ND)]$ gradually transforms to $[Os_3HD(CO)_9(C_4H_3N)]$ and probably to **46**. The ligand in these complexes is nonaromatic [86JOM(311)371; 89JOM(368)119]. Later, the idea of zwitterionic complexes was considered. Thus, it was shown that $[Os_3(CO)_{12}]$ and $[Os_3(CO)_{10}(AN)_2]$ cause the C—H activation of pyrrole; and initially a reactive and unstable zwitterionic species **47** is formed, which easily isomerizes to **48** when $R = H$ [86JOM(311)371; 89OM1408; 90OM6]. Deprotonation of **47** and **48** gives **49**. In isomer **48**, the ligand is presented as C_4H_3N , and is thus a four-electron donor able to form triple bridges **50** ($R = H$) and **51**. These species contain the so-called pyrrolyne framework [91JOM(412)177]. An interesting function is an eight-electron-donor capacity in the triple bridges. It is realized in the mixed metal clusters **52** [$ML_n = FeCp$, $Mn(CO)_3$] formed from $[Os_3(CO)_{10}(AN)_2]$ and azaferrocene or azacymantrene, which enter oxidative addition by virtue of easier C—H activation [88JCS(CC)478; 88JOM(356)C47; 91JCS(D)1111].

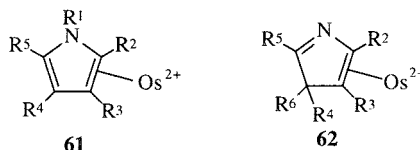
1-Methylpyrrole reacts with $Ru_3(CO)_{12}$ to give **53** and **54**, both zwitterionic complexes with a five-electron-donor heterocycle (97OM1735). Thermolysis of **53** and **54** gives the product of β elimination, **55**, the process being reversible. 1,2,5-Trimethyl- and 2,5-dimethylpyrrole with $Ru_3(CO)_{12}$ give a simple zwitterionic initial complex **56** ($R = H, Me$) in THF. 2,5-Dimethylpyrrole in toluene bypasses the step of zwitterionic complex formation and gives **57** and **58** straightforwardly. Complex **58** is remarkable at least in two respects: an imine coordination of the heterocycle, and the exocyclic C=C bond becoming a μ, η^2 -vinyl donor site. The pyrrole ligand behaves as a μ_3 -bridging five-electron donor.



2-Formylpyrrole oxidatively adds to $[\text{Os}_3(\text{CO})_{10}(\text{AN})_2]$ by its formyl group giving rise to product **59** followed by cleavage of the C—H bond of this moiety [86JOM(311)371; 90OM6]. The complex **59** easily decomposes to **47** and **48**. Species **59** further reacts with triethylamine to yield **60** (97P3775).

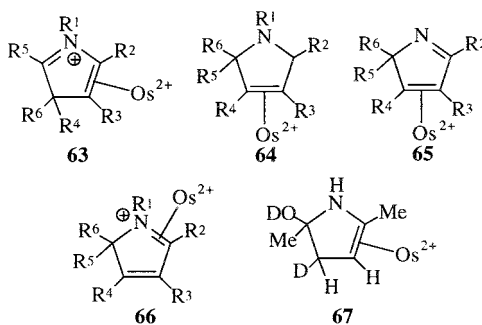


Activation of the pyrrole heteronucleus with respect to electrophilic substitution is enhanced by coordination of the $[\text{Os}(\text{NH}_3)_5]^{2+}$ unit. Species $\text{Os}(\text{NH}_3)_5(\text{OTf})_3$ is reduced in the presence of pyrroles to yield a series **61** ($\text{R}^1 = \text{R}^2 = \text{R}^3 = \text{R}^4 = \text{R}^5 = \text{H}$; $\text{R}^1 = \text{Me}$, $\text{R}^2 = \text{R}^3 = \text{R}^4 = \text{R}^5 = \text{H}$; $\text{R}^2 = \text{Me}$, $\text{R}^1 = \text{R}^2 = \text{R}^3 = \text{R}^4 = \text{R}^5 = \text{H}$; $\text{R}^4 = \text{Me}$, $\text{R}^1 = \text{R}^2 = \text{R}^3 = \text{R}^5 = \text{H}$; $\text{R}^2 = \text{R}^5 = \text{Me}$, $\text{R}^1 = \text{R}^3 = \text{R}^4 = \text{H}$; $\text{R}^1 = \text{R}^2 = \text{R}^5 = \text{Me}$, $\text{R}^3 = \text{R}^4 = \text{H}$; $\text{R}^5 = \text{Et}$, $\text{R}^1 = \text{R}^2 = \text{R}^3 = \text{R}^4 = \text{H}$) (89JA5969; 91JA6682; 92JA5684; 94JA7931). The latter product, **61** ($\text{R}^5 = \text{Et}$, $\text{R}^1 = \text{R}^2 = \text{R}^3 = \text{R}^4 = \text{H}$), is not dynamic, while all the other $\eta^2\text{-C2}=\text{C3}$ complexes experience the shift to the $\eta^2\text{-C4}=\text{C5}$ coordination. Another rearrangement is observed in protic solvents, when tautomerization of **61** ($\text{R}^2 = \text{R}^5 = \text{Me}$, $\text{R}^1 = \text{R}^3 = \text{R}^4 = \text{H}$) to the 3H form **62** ($\text{R}^2 = \text{R}^5 = \text{Me}$, $\text{R}^3 = \text{R}^4 = \text{R}^6 = \text{H}$) is known (95JOC2125). Part of the heteronucleus remote from the transition metal ion resembles the enamine, where the C4 position is more subject to further attacks compared to that of C5.

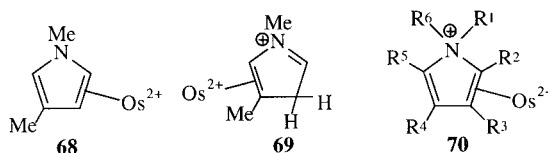


Protonation of complexes **61** by triflic acid occurs preferentially at C4, giving rise to **63** ($\text{R}^6 = \text{H}$), the process being reversible and β deprotonation by mild acids being easy. An alternative protonation was noted for complex **61** ($\text{R}^2 = \text{R}^5 = \text{Me}$, $\text{R}^1 = \text{R}^3 = \text{R}^4 = \text{H}$) (91JA6682; 92JA5684), which with anilinium triflate first gives **63** ($\text{R} = \text{H}$); but then, in the presence of aniline, the rearrangement to **64** ($\text{R}^2 = \text{R}^5 = \text{Me}$, $\text{R}^1 = \text{R}^3 = \text{R}^4 = \text{R}^6 = \text{H}$) occurs where the coordination mode switches to $\eta^2\text{-C3}=\text{C4}$. Deprotonation of **64** gives the 2H-pyrrole complex **65** ($\text{R}^2 = \text{R}^5 = \text{Me}$, $\text{R}^3 = \text{R}^4 = \text{R}^6 = \text{H}$). The latter converts into the tautomer **61** ($\text{R}^2 = \text{R}^5 = \text{Me}$, $\text{R}^1 = \text{R}^3 = \text{R}^4 = \text{H}$). Prolonged protonation of **61** ($\text{R}^4 = \text{Me}$, $\text{R}^1 = \text{R}^2 = \text{R}^3 = \text{R}^5 = \text{H}$)

allows one to obtain, among numerous other tautomers, species **66** with a unique $\text{N}=\text{C}2 \text{ Os}^{2+}$ coordination. Hydrolysis of **61** ($\text{R}^2 = \text{R}^5 = \text{Me}$, $\text{R}^1 = \text{R}^3 = \text{R}^4 = \text{H}$) with heavy water gives **67**.

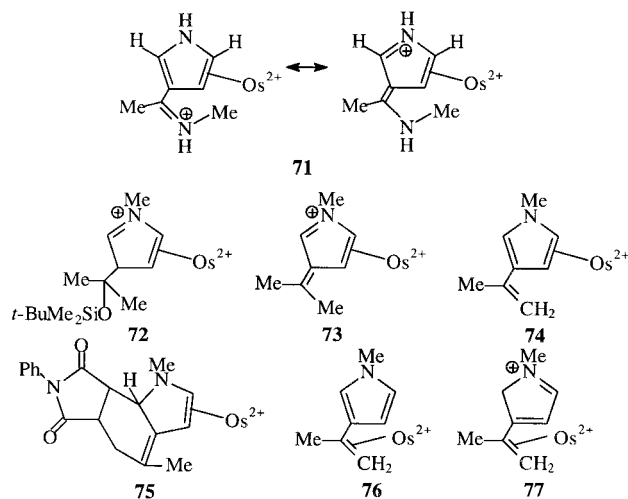


Alkylation of the parent complex **61** ($\text{R}^1 = \text{R}^2 = \text{R}^3 = \text{R}^4 = \text{R}^5 = \text{H}$) is achieved by methyl triflate and gives **63** ($\text{R}^1 = \text{Me}$, $\text{R}^2 = \text{R}^3 = \text{R}^4 = \text{R}^5 = \text{R}^6 = \text{H}$) (92JA5684; 93JOC4788); thus, it occurs at the pyrrolic nitrogen and is followed by migration of the proton to the β position. The methyl group goes to the β position of the starting complex **61** ($\text{R}^1 = \text{Me}$, $\text{R}^2 = \text{R}^3 = \text{R}^4 = \text{R}^5 = \text{H}$), and the reaction leads to **63** ($\text{R}^1 = \text{R}^4 = \text{Me}$, $\text{R}^2 = \text{R}^3 = \text{R}^5 = \text{R}^6 = \text{H}$). Deprotonation of this product with *n*-propylamine gives **68**. It is reprotonated to **69**, the $\eta^2 \text{C}4=\text{C}5$ species, but alkylated at the pyrrolic nitrogen to give **70** ($\text{R}^1 = \text{R}^4 = \text{R}^6 = \text{Me}$, $\text{R}^2 = \text{R}^3 = \text{R}^5 = \text{H}$).

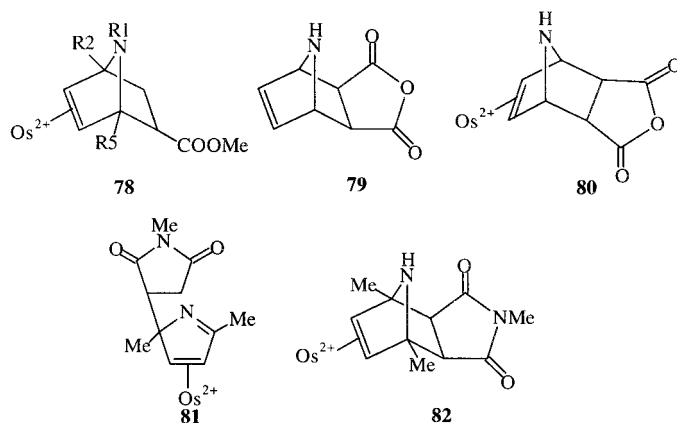


The only example of the β -acetylation was observed in the reaction of **61** ($\text{R}^1 = \text{Me}$, $\text{R}^2 = \text{R}^3 = \text{R}^4 = \text{R}^5 = \text{H}$) with acetic anhydride to give **61** ($\text{R}^1 = \text{Me}$, $\text{R}^4 = \text{MeCO}$, $\text{R}^2 = \text{R}^3 = \text{R}^5 = \text{H}$). However, in the absence of the methyl substituent at N^1 , *N*-acetylation takes place, e.g., for **61** ($\text{R}^1 = \text{R}^2 = \text{R}^3 = \text{R}^4 = \text{R}^5 = \text{H}$; $\text{R}^2 = \text{R}^5 = \text{Me}$, $\text{R}^1 = \text{R}^3 = \text{R}^4 = \text{H}$) (95JOC2125). Electrophilic addition of methylacetonitrilium triflate on **61** ($\text{R}^1 = \text{R}^2 = \text{R}^3 = \text{R}^4 = \text{R}^5 = \text{H}$) goes to the β position of the heteroring and gives **71**. The similar type of process in case of *tert*-butyldimethylsilyl triflate and **61** ($\text{R}^1 = \text{Me}$, $\text{R}^2 = \text{R}^3 = \text{R}^4 = \text{R}^5 = \text{H}$) gives an aldol-type 3-substituted complex **72** (94JA7931). The base, 1,8-diazabicyclo[5.4.0]undec-7-ene, leads first to the elimination of the silicon-containing group to give **73**, and second to deprotonation to yield **74**, which can be reverted to **73** by triflic acid protonation. Complex **74** containing a dienic framework enters Diels–Alder cycloaddition of

the *endo* type with *N*-phenylmaleimide affording **75**. Upon heating, the re-switch of the coordination mode of **74** is observed, and the product **76** is protonated to the C2 position of the heteroring to generate **77**.

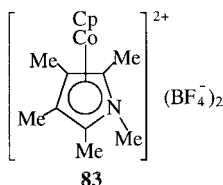


Uncomplexed pyrrole does not enter cycloaddition reactions (86H1835). In contrast, complexes **61** ($R^1 = \text{Me}$, $R^2 = R^3 = R^4 = R^5 = \text{H}$; $R^2 = R^5 = \text{Me}$, $R^1 = R^3 = R^4 = \text{H}$) undergo 1,3-dipolar cycloaddition with methyl acrylate to give **78** ($R^1 = \text{Me}$, $R^2 = R^5 = \text{H}$; $R^1 = \text{H}$, $R^2 = R^5 = \text{Me}$) (95JOC2125). The parent **61** ($R^1 = R^2 = R^3 = R^4 = R^5 = \text{H}$) enters Diels–Alder cycloaddition and yields *exo*-**79** and *endo*-**80** adducts (89JA5969). Formation of **82** is a Diels–Alder reaction of **61** ($R^2 = R^5 = \text{Me}$, $R^1 = R^3 = R^4 = \text{H}$) and 1-pyrrolidin-2,5-dione via the intermediate **81**.



G. COMPLEXES OF COBALT, RHODIUM, AND IRIUM

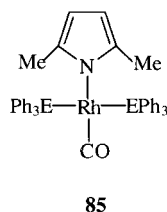
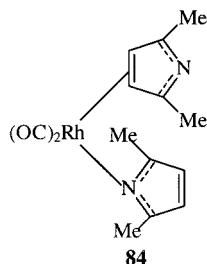
Cobalt complex **83** was obtained by the reaction of $[\text{CpCo}(\text{SMe}_2)_3]_2(\text{BF}_4)_2$ with pentamethylpyrrole [88AG(E)579]. Full cobalt sandwich of 2,5-di-*tert*-butylpyrrole is also known [91JCS(CC)1368]. Meanwhile, attempts to synthesize other pyrrolyl complexes of cobalt, $(\eta^5\text{-2,5-dimethylpyrrole})\text{cobalttricarboxyl}$ and the 3,4-dimethyl analog, have been unsuccessful [87JOM(330)231].



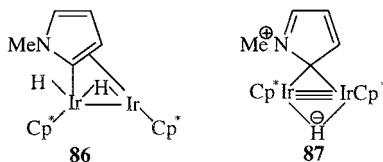
Potassium pyrrolate with $(\text{NMe}_4)(7\text{-R}^1\text{-8-R}^2\text{-7,8-C}_2\text{B}_9\text{H}_{10})$ ($\text{R}^1 = \text{R}^2 = \text{H}$; $\text{R}^1 = \text{Me}, \text{R}^2 = \text{H}$; $\text{R}^1 = \text{Me}, \text{R}^2 = n\text{-Bu}$; $\text{R}^1 = \text{Me}, \text{R}^2 = \text{C}_3\text{H}_6\text{OEt}$) and CoCl_2 yields the sandwich $[(\eta^5\text{-NC}_4\text{H}_4)\text{Co}(7\text{-R}^1\text{-8-R}^2\text{-7,8-C}_2\text{B}_9\text{H}_{10})]$ (96NJC909). Other illustrations of the mixed pyrrolyl/carborane complexes include $[(\eta^5\text{-NC}_4\text{Me}_2\text{R}_2)\text{Co}(\text{Et}_2\text{C}_2\text{B}_4\text{H}_4)]$, $[(\eta^5\text{-NC}_4\text{Me}_2\text{R}_2)\text{Co}(\text{Et}_2\text{C}_2\text{B}_3\text{H}_5)]$ ($\text{R} = \text{Me}, \text{H}$) (89OM2492), $[(\eta^5\text{-NC}_4\text{Me}_2\text{R}_2)\text{Co}(\text{Et}_2\text{C}_2\text{B}_3\text{H}_3)\text{CoCp}]$, $[(\eta^5\text{-NC}_4\text{Me}_2\text{R}_2)\text{Co}(\text{Et}_2\text{C}_3\text{B}_3\text{H}_3)\text{Ru}(\eta^6\text{-1,4-MeC}_6\text{H}_4\text{CHMe}_2)]$ ($\text{R} = \text{Me}, \text{H}$), and $[(\eta^5\text{-NC}_4\text{Me}_4)_2\text{Co}_2(\text{Et}_2\text{C}_2\text{B}_3\text{H}_3)]$ (91OM2631).

A model similar to that of the iron complex **31** was proposed for the cobalt species synthesized as a result of co-condensation of cobalt vapors with pyrrole in vacuum. A frozen matrix formed is subsequently warmed to room temperature (89JA3881). An oligomer or a polymer results, in which σ - and π -donor functions are realized simultaneously. The model proposed differs from that for the iron pyrrolyl complex by inclusion of the Co—Co bonds to attain the 18-electron configuration.

Reaction of lithium 2,5-dimethylpyrrolate ion with $[\text{RhCl}(\text{CO})_2]_2$ leads to formation of **84** (88PAC1193; 90P1503). This is the first example of the mixed mode, when the $\eta^1(\text{N})$ and $\eta^2(\text{C}=\text{C})$ coordination are realized simultaneously. Nucleophilic addition of triphenylphosphine and triphenylarsine gives **85** ($\text{E} = \text{P}, \text{As}$). The iridium analogs of **84** and **85** have also been synthesized.

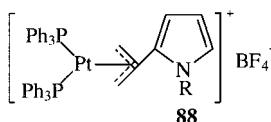


N-Methylpyrrole with $(\text{Cp}^*\text{IrH}_3)_2$ and 3,3-dimethyl-1-butene gives a couple of unique organometallic products, **86** and **87** (99OM134). In **86**, the C—H bond in position 2 is activated and a rare $\eta^1(\text{C}):\eta^2(\text{C}=\text{C})$ coordination mode is realized. Species **87** is a zwitterionic compound containing a triple bond between the iridium atoms.

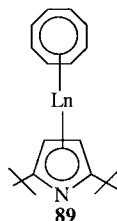


Complex $[\text{RhCl}_2(\eta^1\text{-Ph})(\text{py})_3]$ with *N*-(2-cyanoethyl)pyrrole (L) and EtOH yields $[\text{RhCl}_2(\eta^1\text{-Ph})(\text{L})(\text{py})_2]$ with the $\eta^1(\text{N})$ coordination mode of the heteroaromatic ligand (97P4045).

Pyrrole and 1-methylpyrrole enter the electrophilic substitution with $[\text{Pt}(\text{PPh}_3)_2(\eta^3\text{-C}_3\text{H}_5)](\text{BF}_4)$ to give **88** (R = H, Me) (98JA3243).



2,5-Di-*tert*-butylsodium pyrrolate serves as a source of the η^5 complexes of lanthanides [93CB2657; 95JOM(495)C12]. Thus, with cyclooctadienyl chlorides of samarium, thulium, and lutetium, it affords species **89** [96JOM(507)287]. The N-coordinated samarium(II) calix-pyrrole complex is known [99AG(E)1432].

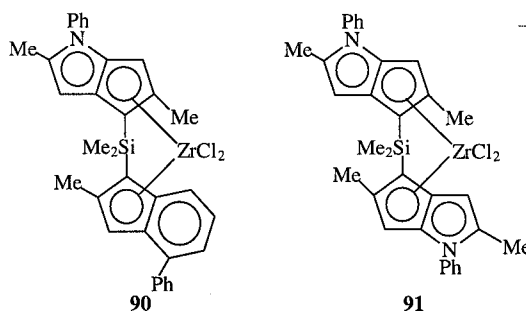


III. Organometallic Complexes of Indole and Carbazole

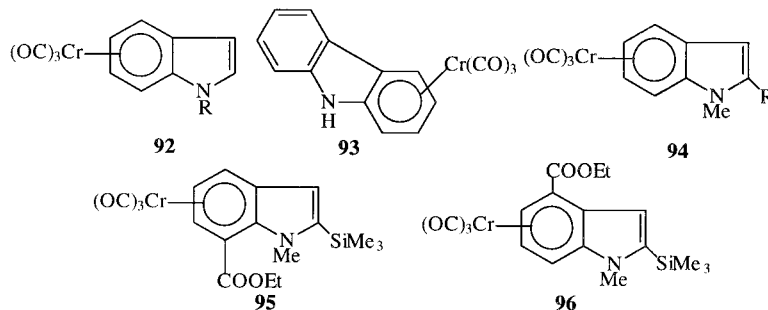
Indole and carbazole are characterized by the enhanced π -electron density within the six-member cycles, which allows one to predict η^6 coordination as the preferential coordination route. In contrast to pyrrole, indole, in accord with experimental (62JA2534; 64JA3796; 71JA5102) and theoretical (82T3693) data, has

the C3 center as the most reactive one with respect to electrophiles and protonation agents (84JA421). Factors of greater thermodynamic stability of the β -protonated form include the electronic influence of the fused six-member ring (83T2851) and effects of polarizability [71JA2914; 77JOC3316; 83AG(E)323].

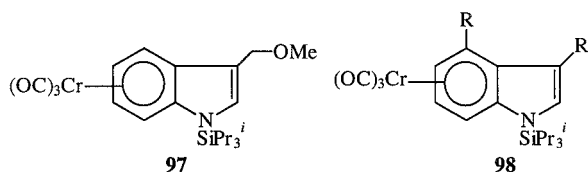
Unusual annulated derivatives of pyrrole, **90** and **91**, were found as efficient catalysts for the polymerization of propene (98JA10786).



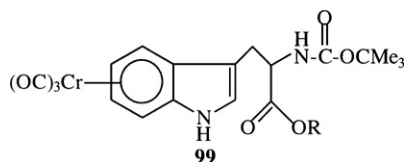
Chromium tricarbonyl complexes with indole and 1-methylindole, **92** ($R = H, Me$), and carbazole, **93**, have been studied [68JOM(14)359; 69JCS(B)1204; 75MI1; 82JCS(CC)467; 82JOM(231)5; 83JOM(255)317]. Two synthetic routes are usually described as direct interaction of chromium hexacarbonyl with the heterocycle, and interaction of the latter with $(NH_3)_3Cr(CO)_3$ or $(AN)_3Cr(CO)_3$. The indole and carbazole complexes are deprotonated by *t*-BuOK followed by formation of the potassium salts, which react with electrophiles [MeI, MeCOCl, *p*-MeC₆H₄SO₂N(NO)Me], addition occurring at the nitrogen atom, e.g., forming **92** ($R = COMe$). For the η^6 complex of 1-methylindole, the sequence of transformations leads to differently substituted η^6 -coordinated products [81JCS(CC)1260]. Thus, action of *n*-butyllithium and then ClCOOEt gives **94** ($R = COOEt$), and further combination of *n*-butyllithium and trimethylchlorosilane gives **94** ($R = SiMe_3$). The next stage of *n*-butyllithium and ClCOOEt allows one to obtain **95** and **96**. Nucleophilic addition of $LiCH_2CN$ to **92** ($R = H$) occurred preferentially at position 4 [79JA217; 82JOM(240)C5].



Chromium hexacarbonyl and N-protected indoles interact and give **92** (R = SiPr₃¹) and **97** [82JCS(CC)467; 84JCS(CC)46; 88T7325]. The combination of lithiation and electrophilic quench leads to the C4-substituted complexes **98** (R = SiMe₃, COOMe, COOEt, CH₂CH=CMe₂, SnMe₃, I, SPh; R' = H, CH₂OMe). This kind of derivatization is difficult for the uncomplexed indole (70MI1; 72MI1).



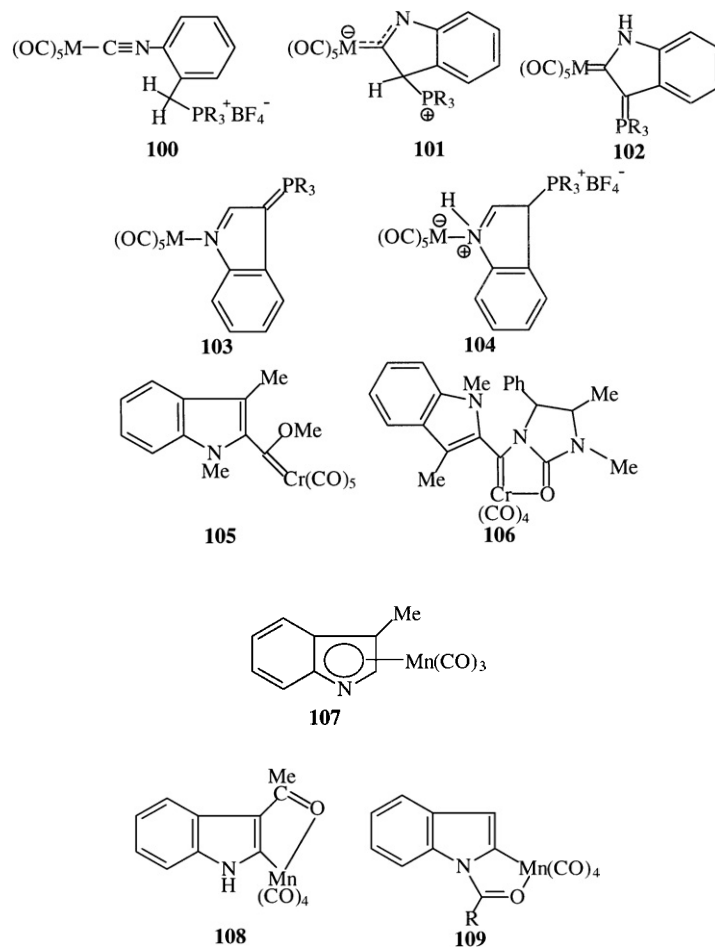
Reaction of chromium hexacarbonyl with the derivative of tryptophan yields **99** (R=Me), which is hydrolyzed to **99** (R=H) and transformed into **99** (R=C₆H₄NO₂) when treated with *p*-nitrophenol and dicyclohexylcarbodiimide [82JOM(240)163].



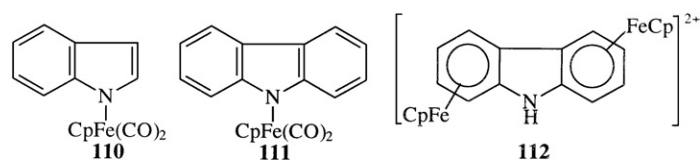
Cyclization of **100** [M = Cr, Mo, W; R₃ = Me₃, Ph₂(CH₂Ph), Ph₃] with Na[N(SiMe₃)₂] proceeds through the stage of **101** to give a mixture of the carbene **102** and N-coordinated complexes **103** and **104** [92JCS(D)2827]. Annulation of pyrrole and indole by the tungsten carbene complexes was studied (99JA3065). Carbene complex **105** (89JOC3249) reacts with the lithium salt of imidazolidinone to give **106** (97OM4945).

The organomanganese chemistry of indole is primarily characterized by the activation of the pyrrolic counterpart. First of all, this is the η^5 coordination, which is a rarity for the benzannulated five-member heterocycles. Thus, the carbocycle in cyclopentadienyltricarbonylmanganese may be substituted by the 3-methylindolyl anion to give **107** [77JCS(D)624]. The pyrrole ring is not planar, while the benzene ring preserves planarity. Tricarbonylmanganese(η^5 -2-methylindolyl) is also known [67JOM(7)325]. Indolylsodium reacts with manganese pentacarbonyl in a more complicated way to give $\text{C}_9\text{H}_8\text{Mn}(\text{CO})_5$.

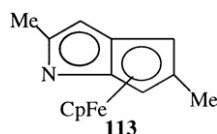
3-Acetylindole with $\text{PhCH}_2\text{Mn}(\text{CO})_5$ gives the *o*-manganated product **108** [88JOM(349)197]. Treatment of *N*-acetyl- and *N*-benzoylindole with $\text{PhCH}_2\text{Mn}(\text{CO})_5$ gives complexes **109** (R = Me, Ph), in which the $\text{Mn}(\text{CO})_4$ group is bonded to the ligand via the Mn—O and Mn—C bonds [88JOM(349)209].



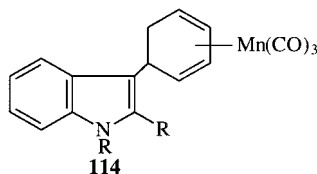
The synthesis of cyclopentadienyliron(η^5 -indolyl) proceeds through formation of **110** (88PAC1193). Transformation into the η^5 -complex occurs at elevated temperatures. Carbazole forms only the *N*-carbazolyl complex **111** under these circumstances. However, carbazole with ferrocene and aluminum chloride/aluminum powder gives the η^6 -coordinated dication **112** [80JOM(186)265].



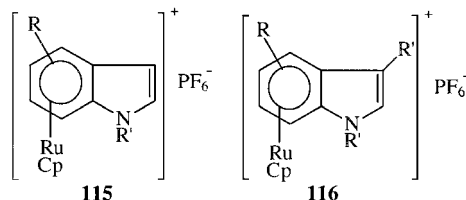
2,3-Dimethylindole undergoes thermolysis with $(C_8H_{10})Fe(Et_2C_2B_4H_4)$, the product being the η^6 -coordinated sandwich $(\eta^6-Me_2C_8H_4NH)Fe^{II}(Et_2C_4B_4H_4)$ (91IC3957). *n*-Butyllithium or potassium hydride serve as deprotonating agents, abstracting the pyrrolic hydrogen, while in THF the process is reverted. The deprotonated species is methylated at nitrogen atom using methyl iodide. The fused species combining cyclopentadienyl and pyrrolyl rings **113** is known [75TL3209; 77JOM(136)C27] where the η^5 coordination embraces the carbocycle.



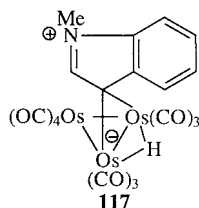
An extraordinary reaction is known for indoles with $[Fe(C_6H_6X)(CO)_3]^+$ ($X = H, OMe$) when the electrophilic substitution of the $C_6H_6M(CO)_3$ fragment goes to position 3 of the heteroring to yield **114** ($R = H, Me$) [73JCS(CC)540; 74JOM(71)C11; 76JCS(D)2192].



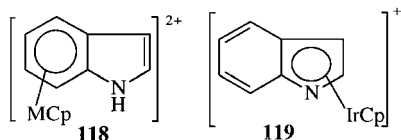
Indole complexes **115** ($R = 5-Cl, 5-Me, 5-Br, 4-Cl, R' = H, Me$) were obtained by heating indole with $[CpRu(AN)_3](PF_6)$ (88OM660). The complexes where $R = 5-Cl, 4-Cl, R' = Me$ undergo nucleophilic substitution of chlorine by $R' = (R''COO)CH_2CH_2$ ($R'' = Me, Et$), MeO , $PhCH_2O$, $HOOCCH_2S$, CH_3NH groups under mild conditions, giving **116**. Other cyclopentadienylruthenium complexes were also obtained by heating the corresponding indole substrate and $[CpRu(AN)_3](PF_6)$ [87JCS(CC)1493; 87JCS(CC)1837]. The coordinated nitroindoles similarly undergo the nucleophilic substitution of the nitro group.



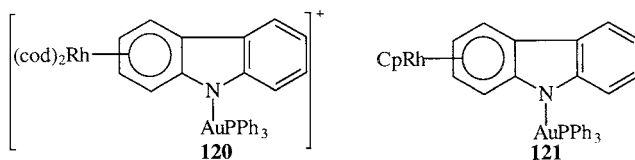
N-Methylindole forms a zwitterionic product **117** with $[\text{Os}_3(\text{CO})_{10}(\text{AN})_2]$ [91JOM(412)177].



Reaction of the cyclopentadienyl rhodium and iridium tris(acetone) complexes with indole leads to the η^6 species **118** ($M = \text{Rh}, \text{Ir}$) [77JCS(D)1654; 79JCS(D)1531]. None of these compounds deprotonates easily in acetone, but the iridium complex loses a proton in reaction with bases (Na_2CO_3 in water, *t*-BuOK in acetone) to form the η^5 -indolyl complex **119**. This reaction is easily reversed in the presence of small amounts of trifluoroacetic acid.

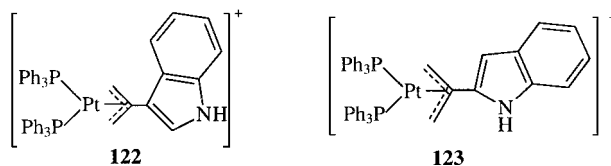


$[(\text{cod})\text{Rh}(\text{acetone})_x]\text{ClO}_4$ reacts with carbazole (L) followed by formation of the η^6 -coordinated derivative $[(\text{cod})\text{RhL}]\text{ClO}_4$ (84P497). Species ClAuPPh_3 with the potassium salt of carbazole forms the *N*-carbazolyl complex, which further reacts as the η^6 donor with acetone-solvated complexes of Rh(I) and Rh(III) affording **120** and **121**.



Indole with $[\text{Pt}(\text{PPh}_3)_2(\eta^3\text{-C}_3\text{H}_5)]\text{BF}_4$ gives the product of insertion of the C_3H_4 moiety to position 3 of the heteroring **122**, while for 3-methylindole, the 2-inserted product **123** is formed (98JA3243).

Unusual π -complex compounds were obtained when reacting with $\text{Yb}(\text{fod})_3$ and $\text{Ag}(\text{fod})$ with 9-vinylcarbazole (83PJC1393). The ^1H NMR spectrum of 9-vinylcarbazole in the presence of $\text{Yb}(\text{fod})_3 + \text{Ag}(\text{fod})$ as the lanthanide shift



reagent was interpreted in such a way that Ag(fod) forms a bridge between Yb(fod)₃ and carbazole. This bridge simultaneously forms the π complex with the double bond of the vinyl group.

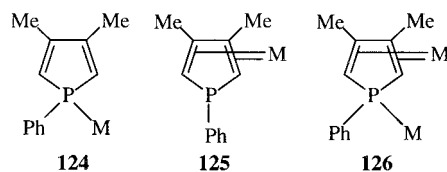
IV. Organometallic Complexes of Phospholes and Analogs

A. INTRODUCTORY REMARKS

Formally, phosphole is reminiscent of pyrrole in its ability to get involved in a 6π -electron delocalized aromatic system. However, the degree of aromaticity of phosphole and analogs is among the lowest in the series of five-member monoheterocycles [88JA4204; 92JA4080; 94MI1; 95AG(E)337; 96BCSF33; 96JA6317; 98IC4413]. Unsubstituted phospholes are pyramidal [70JA5779; 73JCS(D)1888], and to achieve planarity it appears necessary to overcome the inversion barrier at the phosphorus atom [70JHC1; 71JA6507; 74JA6904; 74JCS(P)420; 75JCS(P)974; 76JA407; 76JA2066; 76JA4365; 76JHC1; 79MI1; 81MI1; 90MI2; 92H563; 92MI2; 96MI2]. Aromaticity becomes high for the planarized phosphole (94JA9638; 95JPC586). Polarization is often the result of η^5 -complex formation. The analog of the cyclopentadienyl moiety, phospholyl, has an expressed aromaticity (92JOC3694; 93CCR237; 94CCR1; 96OM1755; 98IC4413; 98MI1). Although phospholyl has a comparable delocalization energy to that of cyclopentadienyl, arsolyl anion is characterized by the least value in this series [75AG(E)232; 78ACR153; 82TCC525; 94OM4732].

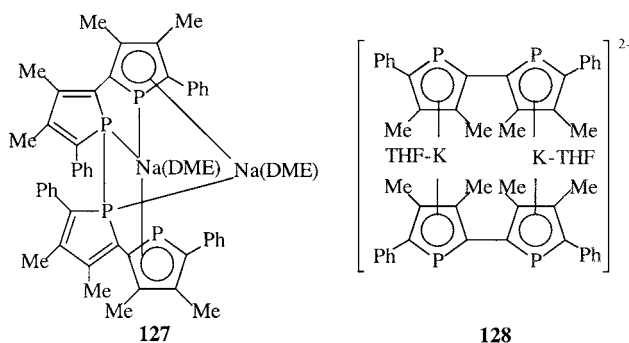
Neutral phospholes, arsoles, and stiboles may formally be considered as two-electron donors, when only the lone-pair electrons of phosphorus, arsenic, and antimony take part in coordination as in **124**. They also behave as four-electron donors, when the diolefinic part of the system coordinates to a metal-carbonyl framework as in **125**. If both functions operate simultaneously, the cyclic system is a formal six-electron donor, e.g. **126** (75CCR239; 80TPC1; 83SB153; 87NJC585; 88CRV429). A combination of the η^5 - and $\eta^1(\text{P})$ functions leads to a chelate character of a phosphole ligand performing the role of a bridge. In compounds containing an ordinary chemical bond between phosphorus, arsenic, or antimony, and a transition metal of the metal-carbonyl framework, phosphole, arsole, and stibole are one-electron donors. However, they behave as three-electron donors when the heteroatom acts as a bridge. Phosphole, arsole, and stibole can also act

as formal five-electron donors similar to cyclopentadiene and pyrrole. Phosphole is sometimes known to be a formal seven-electron donor. The diversity of possible coordination modes is a function of the nature of substituents at the ring (87MI1). The phospholyl group retains the capability to act as an $\eta^1(\text{P})$ two-electron donor ligand and fulfil the four- and eight-electron bridging functions.



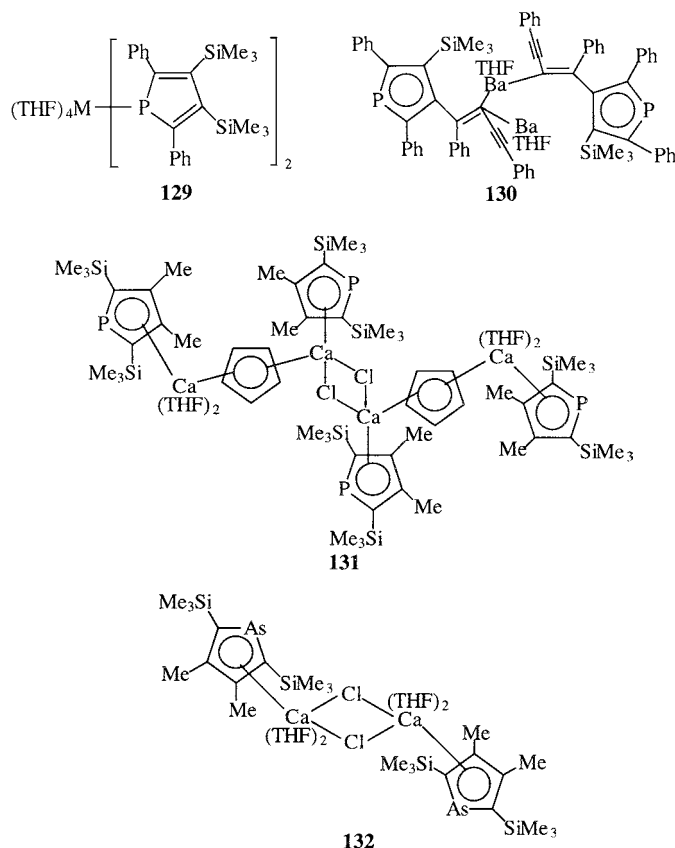
B. COMPLEXES OF NONTRANSITION METALS, TITANIUM, AND ZIRCONIUM

The half-sandwich and sandwich complexes of phospholides and phosphole tetramer are known even for the nontransition metals. The half-sandwich arrangement was studied for lithium tetramethylphospholide [89AG(E)1367] and sodium derivative **127** generated from the phospholide tetramer and sodium in the presence of 1,2-dimethoxyethane [94JA3306; 96AG(E)1125]. Potassium and 1,2-dimethoxyethane in THF in these conditions give a full dianionic sandwich **128**.

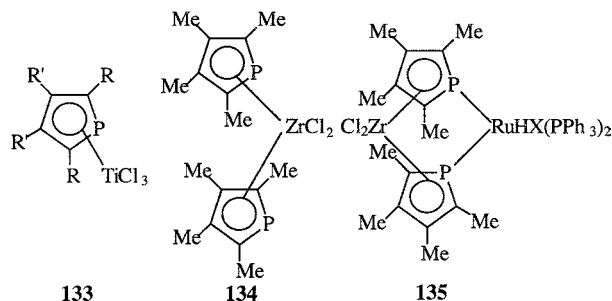


Organomagnesium derivatives have not so far been isolated [80JA994; 80JOM(193)C13]. Bis[bis(trimethylsilyl)phosphonamide] with diphenylbutadiyne and calcium or strontium in THF yields the $\eta^1(\text{P})$ -coordinated species **129**. Reaction of $(\text{THF})_4\text{Ba}[\text{P}(\text{SiMe}_3)_2]_2$ with diphenylbutadiyne gives an η^5 -coordinated species **130** (98JA6722). 1-Chloro- and 1-cyclopentadienyl-3,4-dimethyl-2,5-bis-(trimethylsilyl)-1-phosphacyclopenta-2,4-dienes both react with calcium to give

131 (99OM2491). 1-Chloro-3,4-dimethyl-2,5-bis(trimethylsilyl) arsole is reduced by calcium to give dimer **132**.

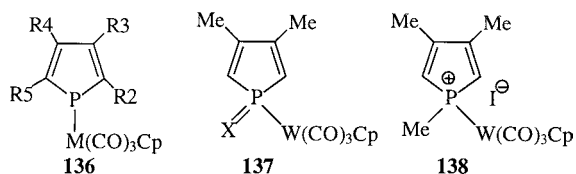


Complexes bis(η^5 -3,4-dimethylphospholyl)-[80JOM(193)C13] and bis (2,3,4,5-tetramethylphospholyl)zirconium dichloride [88OM921; 90JOM(384)271] have been synthesized. Attempts of direct synthesis of (η^5 -phospholyl) MCl_3 ($M = Ti, Zr$) by reaction of lithium 3,4-dimethylphospholide with metal tetrachlorides were successful only in the case of zirconium, but not titanium, when 3,3',4,4'-tetramethyldiphospholyl was isolated [88JCS(CC)770]. Precursors of the titanium complexes are 1-trimethylstannylphospholes. They give rise to species **133**. Complex **134** appears to be an η^1 -P donor (95NJC921), in particular with respect to $RuH_4(PPh_3)_2$ when **135** ($X = H$) is formed. The product exchanges one of the hydrides for a chloride from CCl_4 to yield **135** ($X = Cl$) (96OM4178).



C. GROUP VI METAL COMPLEXES

1-Chlorophenylphosphole and $\text{Na}[\text{M}(\text{CO})_3\text{Cp}]$ give the $\eta^1(\text{P})$ -coordinated species **136** ($\text{M} = \text{Mo}, \text{W}$; $\text{R}^2 = \text{R}^3 = \text{R}^4 = \text{R}^5 = \text{Ph}$) [79JCS(D)814]. A similar reaction course is taken by 1-cyano-3,4-dimethyl- and 1-bromo-2,5-diphenylphosphole and $[\text{Cp}(\text{OC})_3\text{W}]^-\text{Na}^+$, the products being **136** ($\text{M} = \text{W}$, $\text{R}^2 = \text{R}^5 = \text{H}$, $\text{R}^3 = \text{R}^4 = \text{Me}$) and **136** ($\text{M} = \text{W}$, $\text{R}^2 = \text{R}^5 = \text{Ph}$, $\text{R}^3 = \text{R}^4 = \text{H}$), respectively (93OM98). The heteroring in these species is more aromatic than in 1-substituted uncomplexed phosphole. Compound **136** ($\text{M} = \text{W}$, $\text{R}^2 = \text{R}^5 = \text{H}$, $\text{R}^3 = \text{R}^4 = \text{Me}$) is oxidized to **137** ($\text{X} = \text{O}$) by hydrogen peroxide and sulfurized by S_8 to **137** ($\text{X} = \text{S}$), or quarternized by methyl iodide to **138**. Alkylation at the heteroatom takes place in the reaction with 1,2-dichloroethane with formation of the chelate cycle in **139**. Protonation with acetic acid gives **140**, while Diels–Alder cycloaddition of alkynes gives **141** ($\text{R} = \text{COOMe}$, $\text{R}' = \text{Me}$; $\text{R} = \text{Ph}$, $\text{R}' = \text{Et}$). Finally, thermolysis of **136** ($\text{M} = \text{W}$, $\text{R}^2 = \text{R}^5 = \text{H}$, $\text{R}^3 = \text{R}^4 = \text{Me}$) gives the binuclear species **142**. Complex **136** ($\text{M} = \text{W}$, $\text{R}^2 = \text{R}^5 = \text{Ph}$, $\text{R}^3 = \text{R}^4 = \text{H}$) with diphenylacetylene behaves differently and enters ligand substitution to yield **143**. Thermolysis route is also peculiar, leading to **144** containing a hydrido bridge.

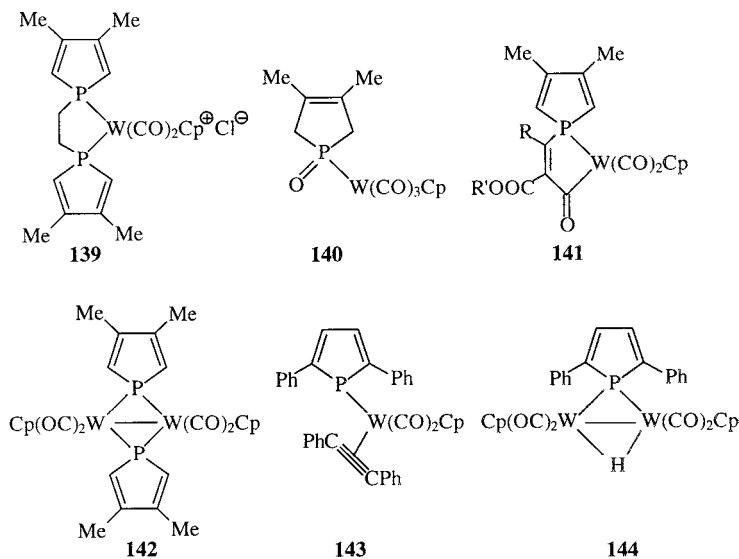


Lithium 3,4-dimethylphospholide with $\text{Cr}(\text{CO})_5(\text{THF})$ gives **145** and then with methyl iodide, **146** ($\text{M} = \text{Cr}$, $\text{R}^1 = \text{R}^3 = \text{R}^4 = \text{Me}$, $\text{R}^2 = \text{R}^5 = \text{H}$) (84JA826; 93BSCF695). Complex **146** ($\text{M} = \text{Cr}$, $\text{R}^1 = \text{R}^3 = \text{R}^4 = \text{Me}$, $\text{R}^2 = \text{R}^5 = \text{H}$) undergoes protonation to give finally the 2*H*-phosphole species **147**. In contrast, the same phospholyllithium salt with $\text{W}(\text{CO})_5(\text{THF})$ yields **148**. It can be reprotonated

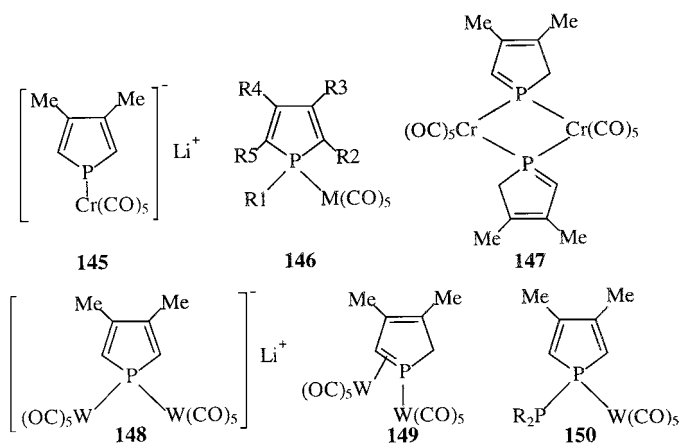
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ALEXANDER P. SADIMENKO

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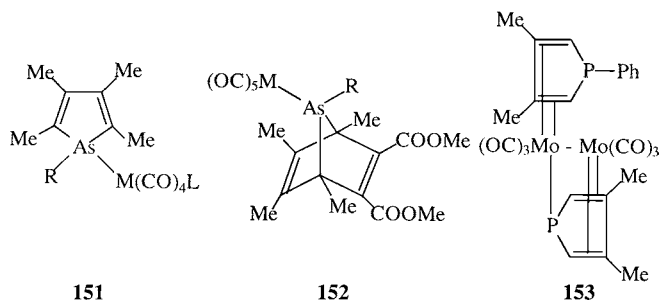


by a sulfonic resin to the $\eta^1:\eta^2$ 2H-phosphole product **149**, the reaction being reversed by *tert*-butyllithium. Complex **148** with a series of chlorophosphanes gave **150** ($R = t\text{-Bu}, i\text{-Pr}, \text{Et}, \text{Ph}$) [97JOM(529)197]. In addition, species similar to **150** but with alkyl (86OM1161), aryl [68T3437; 70JCS(C)386; 78JHC1319], alkoxy (86NJC321), and amino (86TL1323; 89JA9098) substituents are known.



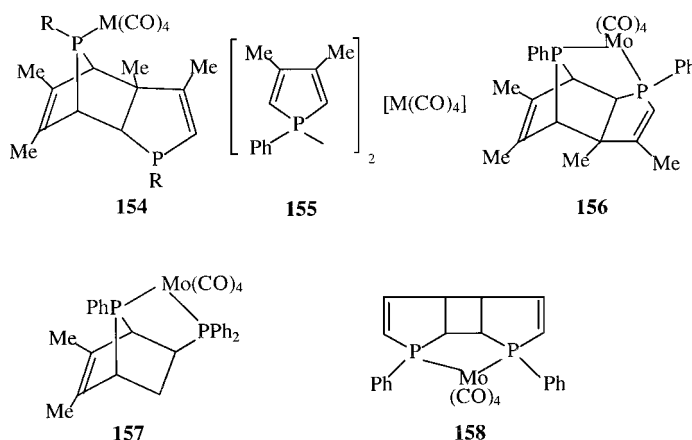
1-Phenyl-3,4-dimethylphosphole is characterized by the transfer of the phenyl group and a [1,5]-hydrogen shift at temperatures higher than 420 K [81JA4595;

82JCS(CC)1272; 82TL511; 83JA6871]. Consequently, it is in equilibrium with 5-phenyl-3,4-dimethyl-2*H*-phosphole [84JOM(263)55]. At temperatures lower than 420 K, the ligand reacts as the 1-phenyl derivative (59CI250; 61JA4406; 64JCS6406; 70JHC1; 71BSCB651). At 413 K, the classical σ complex **146** ($M = \text{Mo}$, $R^1 = \text{Ph}$, $R^2 = R^5 = \text{H}$, $R^3 = R^4 = \text{Me}$) is formed (80JA5809). A series of tetramethylarsoles behaves similarly and yields products **151** ($M = \text{Cr}$, $L = \text{CO}$, $R = \text{Ph}$, Me ; $M = \text{W}$, $L = \text{CO}$, $R = \text{Ph}$, Me , *t*-Bu; $M = \text{W}$, $L = \text{C}_5\text{H}_{10}\text{NH}$, $R = \text{Ph}$, Me ; $M = \text{W}$, $R = \text{Ph}$, *i*-Pr, $L = \text{CO}$) [94JOM(467)67]. For **151**, when $M = \text{Cr}$, W , $R = \text{Me}$, Ph , and $L = \text{CO}$, Diels–Alder addition of acetylenedicarboxylic acid dimethyl ester gives **152** ($M = \text{Cr}$, W , $R = \text{Me}$, Ph). If the reaction leading to **146** ($M = \text{Mo}$, $R^1 = \text{Ph}$, $R^2 = R^5 = \text{H}$, $R^3 = R^4 = \text{Me}$) is conducted for a longer time, **153**, in which both σ and π bonding occur, is formed.



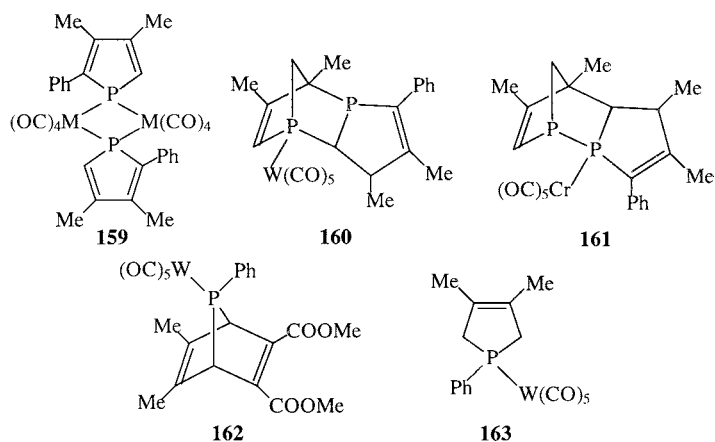
λ^5 -Phospholes tend to dimerize at low temperatures. In contrast, the λ^3 derivatives are not dimerized under normal conditions. [2 + 2] Head-to-tail dimerization of 1,2,5-triphenylphosphole by UV irradiation was observed [81MI1; 82JCS(CC)667; 87AG(E)275; 88JOM(354)83; 90CRV997; 90MI3; 90RHC39; 92JOC6557; 93BSCF843; 93JOC1800; 93JOC6786; 93OM1401; 94JA10966; 94JOC5207; 99JOM(586)166]. However, during a study of the reaction of 1-phenyl-3,4-dimethylphosphole with molybdenum hexacarbonyl, [4 + 2] dimerization leading to the *exo* Diels–Alder dimer has been noted (80JA5809; 81JA4595). The reaction of 3,4-dimethylphospholes with chromium, molybdenum, and tungsten hexacarbonyls under UV irradiation resulted in the complexes **146** ($M = \text{Cr}$, Mo , W , $R^1 = \text{Ph}$, $R^2 = R^5 = \text{H}$, $R^3 = R^4 = \text{Me}$; $M = \text{Mo}$, $R^1 = \text{Me}$, *t*-Bu, $R^2 = R^5 = \text{H}$, $R^3 = R^4 = \text{Me}$) and **154**. The first of them corresponds to the composition $\text{LM}(\text{CO})_5$, while complexes **154** are formulated as $\text{L}_2\text{M}(\text{CO})_4$ ($M = \text{Cr}$, $R = \text{Ph}$; $M = \text{Mo}$, $R = \text{Me}$, *t*-Bu, Ph ; $M = \text{W}$, $R = \text{Ph}$) and represent the derivatives of Diels–Alder dimers with an unusual *exo* configuration. They have a *cis* arrangement around the metal atom. The first step of transition-metal-assisted dimerization includes formation of **155** ($M = \text{Cr}$, W) where two phosphole units are quite close to each other. The second stage, intramolecular dimerization, is promoted by

UV irradiation. Indeed, thermal reaction of 1-phenyl-3,4-dimethylphosphole with $(C_5H_{10}NH)Mo(CO)_4$ leads to **155** ($M = Mo$) and not to **154** ($M = Mo$, $R = Ph$). Complex **155** ($M = Mo$) converts into **154** ($M = Mo$, $R = Ph$) under UV irradiation. This route was confirmed by a photochemical reaction between 3,4-dimethyl-1-phenylphosphole and $Mo(CO)_6$ when both **146** ($M = Mo$, $R^1 = Ph$, $R^2 = R^5 = H$, $R^3 = R^4 = Me$) and **155** ($M = Mo$) resulted (89IC4536). In excess phosphole, the product was **156**. A similar chromium complex is known [82JCS(CC)667]. Complex **146** ($M = Mo$, $R^1 = Ph$, $R^2 = R^5 = H$, $R^3 = R^4 = Me$) enters [4 + 2] Diels–Alder cycloaddition with diphenylvinylphosphine to give **157**. However, from the viewpoint of Woodward–Hoffmann rules and on the basis of the study of UV irradiation of 1,2,5-trimethylphosphole, it is highly probable that [2 + 2] dimers are the initial products of dimerization, and [4 + 2] dimers are the final results of thermally allowed intramolecular rearrangement of [2 + 2] dimers. This hypothesis was confirmed by the data obtained from the reaction of 1-phenylphosphole with molybdenum hexacarbonyl under UV irradiation: the head-to-tail structure of the complex **158**.

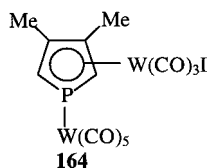


If the reaction temperature is raised to 430 K and the carbon monoxide pressure to 3 atm, coordination of the metal atom in the rearranged product occurs via the phosphorus site, as in **159** ($M = Cr, Mo, W$) [84JOM(263)55]. Along with this product ($M = W$) at 420 K, formation of the dimer of 5-phenyl-3,4-dimethyl-2*H*-phosphole, **160** (the σ complex), is possible as a consequence of [4 + 2] cycloaddition reactions. Chromium hexacarbonyl in turn forms phospholido-bridged $\eta^1(P)$ -coordinated complex **161**. At 420 K in excess 2,3-dimethylbutadiene, a transformation **162** $\rightarrow$ **163** takes place (82JA4484).

A number of papers are devoted to the reaction ability of the $W(CO)_5$ σ complexes of the type **146** [86P1413; 87AG(E)275; 88CRV429; 88OM1796;

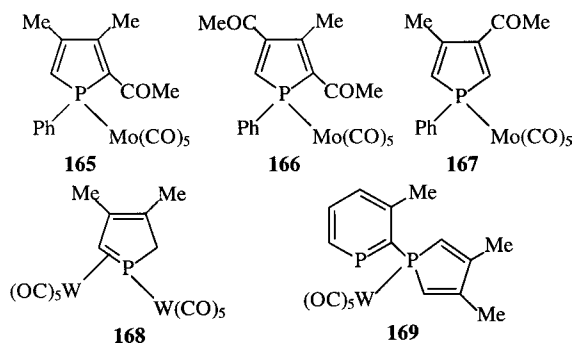


90JOM(400)149; 96BSCF33]. The complex **164** is the first known η^5 -phospholyl species. The tungsten atoms have a coordination number of 9, and the carbon atoms of the phospholyl ring are coplanar. The phosphorus atom deviates from the plane of carbon atoms by 0.015 nm. The basic difference between the η^5 -cyclopentadienyl and η^5 -phospholyl complexes is the existence of a low-lying LUMO localized mainly at the phosphorus atom.

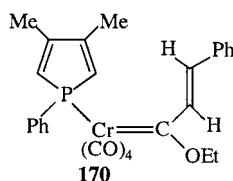


Acylation of the carbon atom in 1-phenyl-3,4-dimethylphosphole becomes possible only after blocking the phosphorus atom by complex formation with Mo(CO)_6 as in **146** ($\text{M} = \text{Mo}$, $\text{R}^1 = \text{Ph}$, $\text{R}^2 = \text{R}^5 = \text{H}$, $\text{R}^3 = \text{R}^4 = \text{Me}$). A series of complexes **165–167** was described. Another consequence of blocking the lone pair of phosphorus by group VI metal hexacarbonyls (particularly tungsten hexacarbonyl) is interaction of the aromatic cyclopentadienyl cycle or the ferrocene molecule with the diene system of phosphole yielding the 2-arylphospholene complexes [87JOM(332)41]. An example of the $\eta^1:\eta^2$ coordination is complex **168**, in which the phosphorus–carbon bond length is intermediate between those of P–C and P=C (88CRV1327). It is interesting that 2-(3,4-dimethyl-1-phospholyl)-3-methylphosphinine coordinates the W(CO)_5 group from $\text{W(CO)}_5(\text{THF})$ via the phosphoric phosphorus, **169** (95BSCF910).

The facile route for introduction of the phosphole ring into the coordination sphere of the chromium vinylcarbene complex is via $[4 + 2]$ intramolecular



cycloaddition of the phosphole dienic system to the C=C carbene double bond (88OM2233). The intermediate complex **170** was proposed, where phosphole behaves as a classical P-ligand.

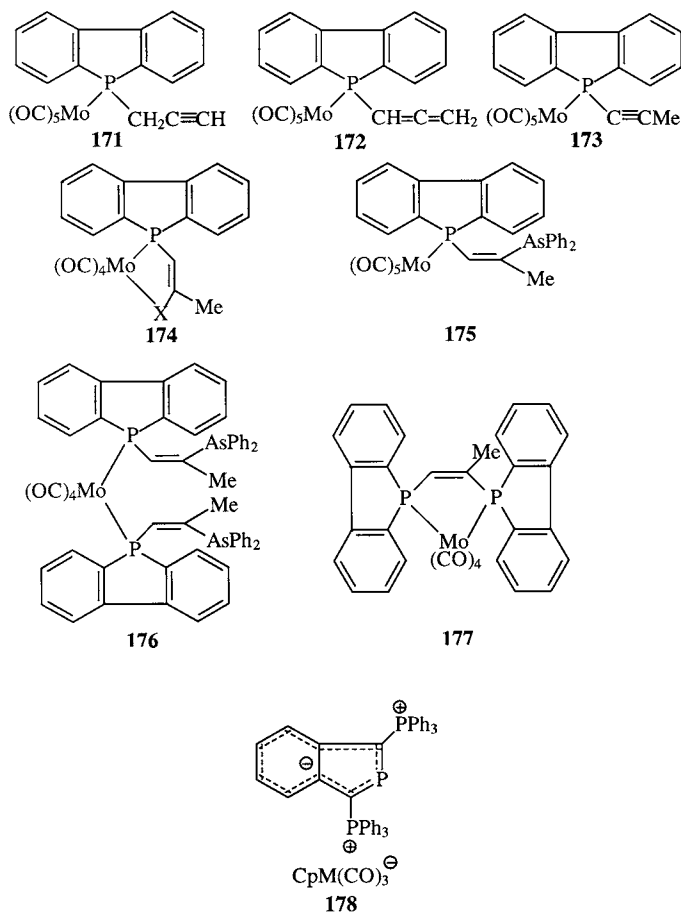


Dibenzophosphole (L) σ complexes of the type $L_nM(CO)_{6-n}$ ($M = Cr, Mo, W$; $n = 1-3$) were reported (72CJC3714; 75CCR239; 87IC4294; 88OM1724; 88OM1735; 90IC425). The complex $L_2Cr(CO)_4$ has a *cis* structure and $L_3Cr(CO)_3$ has a *mer* structure in the solid state.

The derivative **171** is of interest in this series of complexes since it undergoes isomerization of the alkyne substituent in position 1 to yield **172** and **173**. The allyl complex **172** reacts with diphenylarsine to yield predominantly the chelate **174** ($X = AsPh_2$) together with **175** and **176** (98IC1105). Complexes of the types **174** ($X = PPh_2$) and **177** are known (93BSCF673; 97BSCF471).

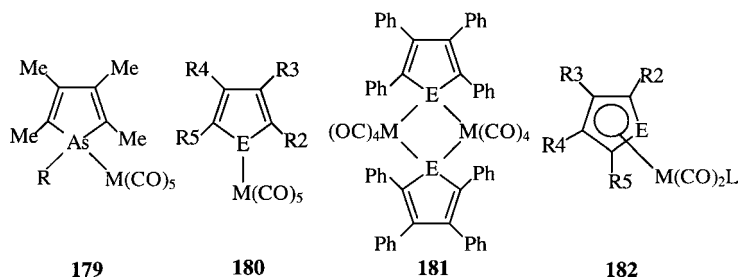
Bis(phosphonio)isophosphindolium cations were expected to act as attractive ambidentate ligands toward the transition-metal-containing moieties. These expectations did not materialize with respect to $CpM(CO)_3$ ($M = Mo, W$), since the anionic metathesis occurred to yield the ionic products **178** (96PSS125).

1-Methyl- and 1-phenyltetramethylarsole react with $[Cp^*Mn(CO)_2(NO)](PF_6)$ to yield the η^1 -coordinated species **179** ($R = Me, Ph$) [94JOM(467)67]. The $\eta^1(E)$ complexes **180** ($M = Mn, Re, E = P, As, Sb$; $R^2 = R^3 = R^4 = R^5 = Ph$) were obtained in two ways: from (i) 1-chlorophosphole, -arsole, or -stibole and $[M(CO)_5]^-$; (ii) from phospholylolithium or -sodium with $Mn(CO)_5Cl$ [78ICA(30)L294; 79JCS(D)814]. Mild thermolysis gives **181** ($M = Mn, Re, E = P, As$). Higher temperatures lead to **182** ($M = Mn, Re, E = P, As, L = CO$; $R^2 = R^3 = R^4 = R^5 = Ph$) [79JCS(D)1552] and **182** ($M = Mn, E = Sb, L = CO$; $R^2 = R^5 = Me, R^3 = R^4 = H$)



[73JCS(CC)258; 80JOM(202)C95]. 1-Phenyl-2,5-dimethylbismole was first lithiated and then reacted with $\text{Mn}(\text{CO})_5\text{Br}$ to give the $\eta^1(\text{Bi})$ -coordinated species **180** ($\text{M} = \text{Mn}$, $\text{E} = \text{Bi}$, $\text{R}^2 = \text{R}^5 = \text{Me}$, $\text{R}^3 = \text{R}^4 = \text{H}$) [93JOM(447)197]. Thermolysis of the product leads to the $\eta^1(\text{Bi}) \rightarrow \eta^5$ rearrangement of the coordination mode with the formation of **182** ($\text{M} = \text{Mn}$, $\text{E} = \text{Bi}$, $\text{L} = \text{CO}$, $\text{R}^2 = \text{R}^5 = \text{Me}$, $\text{R}^3 = \text{R}^4 = \text{H}$). It is quite probable that transformation **180** $\rightarrow$ **182** proceeds through dimer **181**.

Delocalization of the π -electron density in the heterorings takes place, although phosphole and arsole themselves are characterized by lack of aromatic stabilization. Valence force fields of the Cp groups in cymantrene and $\text{C}_4\text{H}_4\text{P}$ in phosphacymantrenes were compared [81JOM(218)416]. The data and the results of MO calculations (79NJC725) showed that the phospholyl rings are more electrophilic and weaker six-electron π donors than the cyclopentadienyl rings. The phospholyl ring is approximately planar with the orthogonal conformation of the exocyclic

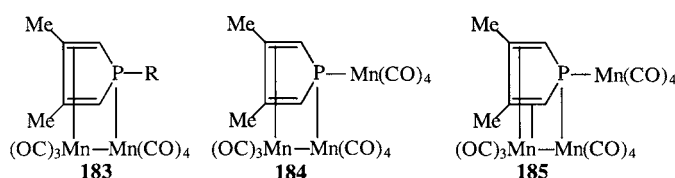


methyl groups for **182** ($M = \text{Mn}$, $E = \text{P}$, $L = \text{CO}$, $R^2 = R^4 = R^5 = \text{H}$, $R^3 = \text{Me}$) [79JOM(181)349; 84JOM(275)53; 86JOM(305)199]. Further data on the extent of the π -electron delocalization were obtained from the information on reactivity. In the free phospholyl anions, electrophilic attacks proceed via the phosphorus atom since the heteroatom has a high negative charge. Phosphacymantrenes have been chosen as references [77JOM(128)297; 78JA5748] to study the influence of π -complex formation on aromaticity of the phosphole nucleus. C-Acylation of **182** ($M = \text{Mn}$, $E = \text{P}$, $L = \text{CO}$, $R^2 = R^5 = \text{H}$, $R^3 = R^4 = \text{Me}$) by $\text{MeCOCl} + \text{AlCl}_3$ proceeds easily to give **182** ($M = \text{Mn}$, $E = \text{P}$, $L = \text{CO}$, $R^2 = \text{COMe}$, $R^3 = R^4 = \text{Me}$, $R^5 = \text{H}$) (76TL4155; 78JA5748), while C-benzoylation ($\text{PhCOCl} + \text{AlCl}_3$) yields **182** ($M = \text{Mn}$, $E = \text{P}$, $L = \text{CO}$, $R^2 = \text{COPh}$, $R^3 = R^4 = \text{Me}$, $R^5 = \text{H}$). Thus, electrophilic substitution via the carbon atoms of phospholyl is, in principle, possible. The reactivity of phospholyl toward electrophiles is, however, considerably less than that of the cyclopentadienyl ring in cymantrene. Phospholyl is a weaker electron donor than cyclopentadienyl toward the $\text{Mn}(\text{CO})_3$ group. The electron density at the cyclic carbon atoms is much less in phosphacymantrenes than in cymantrene. Although the phosphorus atom takes part in the delocalization in the phospholyl ring, it also plays the role of an electron withdrawer. X-Ray analysis of **182** ($M = \text{Mn}$, $E = \text{P}$, $L = \text{CO}$, $R^2 = \text{COPh}$, $R^3 = R^4 = \text{Me}$, $R^5 = \text{H}$) [79JOM(165)129] has shown that the phospholyl ring is not strictly planar.

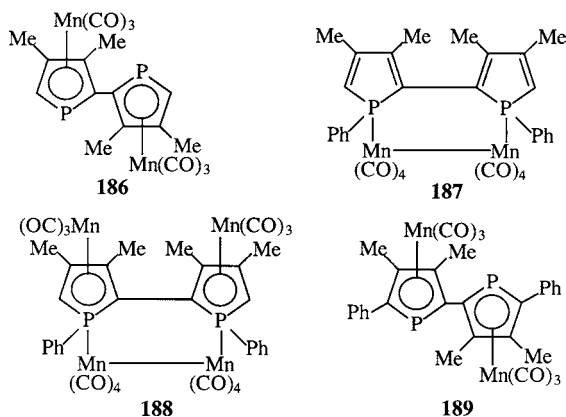
The π complexes of tricarbonylmanganese **182** ($M = \text{Mn}$, $E = \text{P}$, As , $L = \text{CO}$, $R^2 = R^3 = R^4 = R^5 = \text{Ph}$) may be subjected to a series of substitution reactions of carbon monoxide to yield **182** ($M = \text{Mn}$, $E = \text{P}$, As , $R^2 = R^3 = R^4 = R^5 = \text{Ph}$, $L = \text{Ph}_3\text{P}$; $E = \text{As}$, $L = \text{Ph}_2\text{C}=\text{CCPh}_2$) [79JCS(D)1552]. Under UV irradiation, 3,4-dimethylphosphacymantrene **182** ($M = \text{Mn}$, $E = \text{P}$, $L = \text{CO}$, $R^2 = R^5 = \text{H}$, $R^3 = R^4 = \text{Me}$) forms monosubstituted products with various phosphines and trimethylphosphite, **182** [$M = \text{Mn}$, $E = \text{P}$, $R^2 = R^5 = \text{H}$, $R^3 = R^4 = \text{Me}$, $L = \text{PhPMe}_2$, Ph_3P , $(\text{MeO})_3\text{P}$]. A disubstituted derivative was also obtained in the case of trimethylphosphite [78JOM(144)C9].

UV irradiation and heating of $\text{Mn}_2(\text{CO})_{10}$ in the presence of phospholes give rise to the unstable σ complexes $\text{L}_x\text{Mn}_2(\text{CO})_{10-x}$ ($x = 1, 2$) and to the σ, π complexes **183** ($R = \text{Ph}$, $t\text{-Bu}$), in which the ligands are tridentate [75JOM(93)377].

When **183** ($R = \text{Ph}$) is irradiated further in the presence of dimanganese decacarbonyl, the $\text{P}-\text{Ph}$ bond is broken. As a result, **184** was proposed where the $\text{P}-\text{Ph}$ bond is replaced by $\text{P}-\text{Mn}(\text{CO})_4$. Pyrolysis of the product at 420 K yields **182** ($M = \text{Mn}$, $E = \text{P}$, $L = \text{CO}$, $R^2 = R^5 = \text{H}$, $R^3 = R^4 = \text{Me}$). Later [79JOM(176)307], it was shown that the structure **184** was erroneous. When **183** is heated, it gives decomposition products, **185** (minor) and mainly **182** ($M = \text{Mn}$, $E = \text{P}$, $L = \text{CO}$, $R^2 = R^5 = \text{H}$, $R^3 = R^4 = \text{Me}$). The structures of **183** ($R = t\text{-Bu}$) and **185** were confirmed by X-ray analysis (77IC3307).



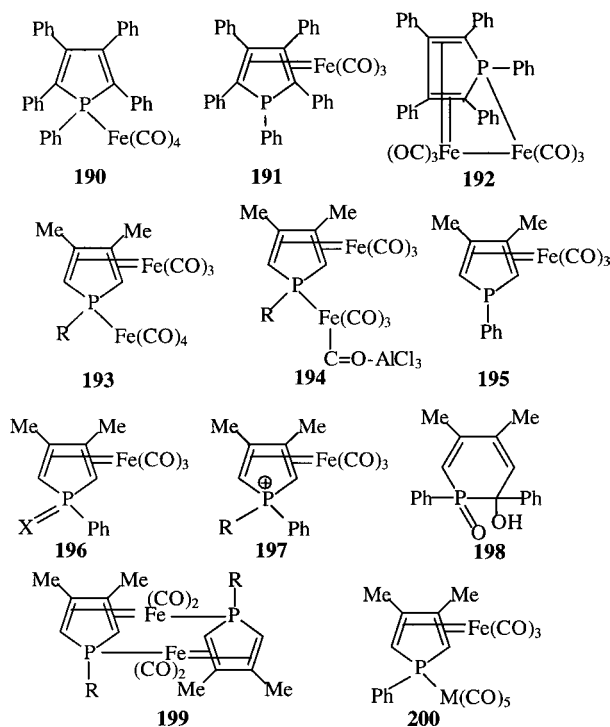
The reaction between 2,2'-biphosphole and dimanganese decacarbonyl was studied under different conditions [86JOM(316)271]. In boiling xylene and under inert atmosphere, the main product is the isomeric bis(η^5 -diphospholyl) complex **186**, and complexes **187** and **188** are also produced. When performing the reaction in a closed vessel and at 420 K, the new π complex **189** is formed.



D. COMPLEXES OF IRON

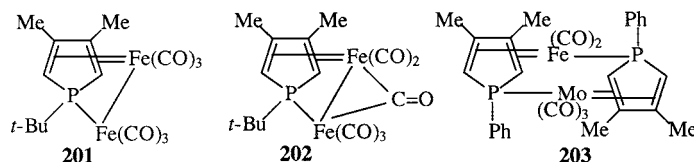
Interaction of pentaphenylphosphole with iron pentacarbonyl gives rise to the $\eta^1(\text{P})$ complex **190** [61JA4406; 77JOM(136)241; 79ICA(35)231]. On the other hand, reaction of $\text{Fe}_3(\text{CO})_{12}$ leads to the formation of the η^4 complex **191** and

$\eta^1:\eta^4$ -complex **192**, where the ligand reveals the property of a nonaromatic conjugated diene. Interaction of 1-phenyl-3,4-dimethylphosphole with diiron nonacarbonyl gives rise to a complex in which $\text{Fe}(\text{CO})_3$ is η^4 -coordinated to the diene while $\text{Fe}(\text{CO})_4$ is η^1 -coordinated to the phosphorus atom, **193** ($\text{R} = \text{Ph}$) [84JOM(266)285]. Reaction of **193** with aluminum trichloride followed by ammonia leads to formation of **194**. The mononuclear complex **195** is formed through an intermediate product **194**. The complex **195** reacts with hydrogen peroxide to give **196** ($\text{X} = \text{O}$), with sulfur to give **196** ($\text{X} = \text{S}$), and with benzyl bromide and methyl iodide to produce **197** ($\text{R} = \text{Me}$, PhCH_2). The complex **195** also possesses properties of the normal phosphole and takes part in ring expansion with benzoyl chloride and triethylamine to give **198** [84JOM(266)285; 86JOM(298)77]. In the presence of phenylnitrilepalladium dichloride, it loses carbon monoxide followed by formation of the sandwich complex **199** ($\text{R} = \text{Ph}$). When interacting with pentacarbonyltungsten tetrahydrofuran, it yields bimetallic complexes, in which the tungsten atom is coordinated via the phosphorus lone pair, **200** ($\text{M} = \text{W}$).

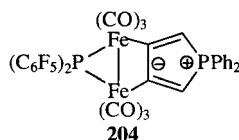


The chemistry of 1-*tert*-butyl-3,4-dimethylphosphole toward iron carbonyls differs in some respects (81IC2848). Thus, with $\text{Fe}_3(\text{CO})_{12}$, complexes containing the

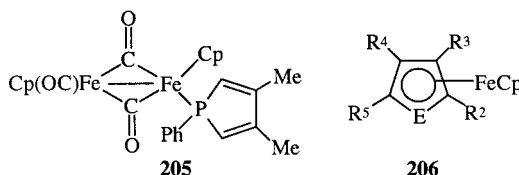
Fe—Fe bond can be assigned structures **201** or **202** based on spectral data. The other product of this reaction is **193** ($R = t\text{-Bu}$), however, it is produced in minor amounts. Complexes **199** ($R = R' = t\text{-Bu}$, $R = \text{Ph}$, $R' = t\text{-Bu}$) were obtained. Reaction of **146** ($M = \text{Mo}$, $R^1 = \text{Ph}$, $R^2 = R^5 = \text{H}$, $R^3 = R^4 = \text{Me}$) with (benzylideneacetone)iron carbonyl gives rise to the bimetallic complex **200** ($M = \text{Mo}$), which reacts further with the free phosphole to form the bimetallic heteronuclear sandwich **203**. The preferable coordination of the molybdenum atom to the dienic system of the second phosphole nucleus is rather unusual. The molybdenum atom is believed to have a greater tendency to coordinate via the trivalent phosphorus atom than via the dienic system.



$\text{Fe}_2(\text{CO})_9$ reacts with $(\text{C}_6\text{F}_5)_2\text{PC}\equiv\text{CPh}$ to yield **204** [75JOM(91)C13; 78JOM(151)1].



1-Phenyl-3,4-dimethylphosphole with dicyclopentadienyliron dicarbonyl gives mainly the classical σ complex **205**, in which the heterocyclic ligand substitutes one terminal CO group [77JOM(139)77]. The first attempt at preparation of phosphaferrrocene included the reaction of potassium tetraphenylphospholyl with $\text{CpFe}(\text{CO})_2\text{FeI}$ (71BSCB651). However, it led to the $\eta^1(\text{P})$ complex $\text{CpFe}(\text{CO})_2\text{L}$ and was not followed by the projected elimination of the CO ligands. 1-Phenylphosphole and 3,4-dimethyl-1-phenylphosphole with $[\text{CpFe}(\text{CO})_2]_2$, however, led to the cleavage of the phosphorus–phenyl bond and formation of **206** ($E = \text{P}$, $R^2 = R^3 = R^4 = R^5 = \text{H}$; $E = \text{P}$, $R^2 = R^5 = \text{H}$, $R^3 = R^4 = \text{Me}$) (77JA3537).

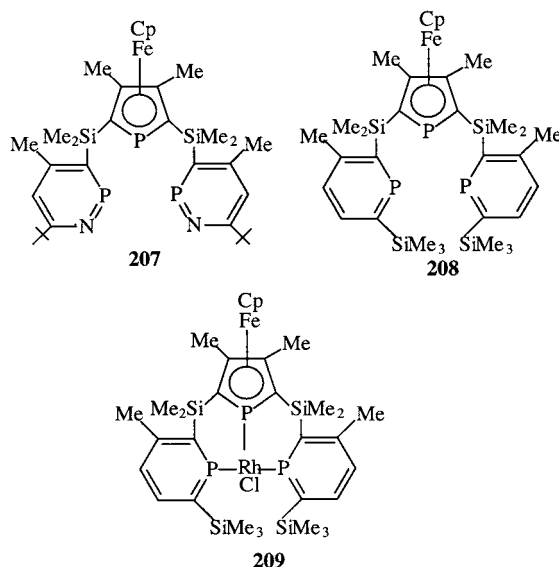


A synthetic route for 2-phenyl-3,4-dimethyl-1-phosphaferrocene **206** ($E = P$, $R^2 = Ph$, $R^5 = H$, $R^3 = R^4 = Me$) from 3,4-dimethyl-1-phenylphosphole and dicyclopentadienyliron dicarbonyl at 430 K and 3 atm was reported. The property of migration of the phenyl group in 1-phenyl-3,4-dimethylphosphole at high temperatures leading to 2*H*-phosphole was utilized (81JA4595). Trapping of the product gives phosphaferrrocene [77JA3537; 84JA425; 84JOM(263)55; 85IC4141; 88OM921]. 2-Phenyl-1-phosphaferrocene **206** ($E = P$, $R^3 = R^4 = R^5 = H$, $R^2 = Ph$) was obtained under identical conditions. Nucleophilic attack of the phospholyl anions on the metastable acetylacetonate of pentamethylcyclopentadienyl iron leads to **206** ($E = P$, $R^2 = R^5 = H$, $R^3 = R^4 = Me$; $R^2 = R^3 = R^5 = H$, $R^4 = Me$; $R^2 = R^3 = R^4 = R^5 = Me$; Cp^* instead of Cp) [86JOM(309)323]. The iron-containing derivatives [$Fe(CO)_2Cp(EC_4Ph_4)$] ($E = P, As$) have greater thermal stability than the corresponding σ complexes of manganese and rhenium. Continuous heating of these complexes causes abstraction of carbon monoxide and formation of phosphaferrrocenes **206** ($E = P, As$; $R^2 = R^3 = R^4 = R^5 = Ph$) [79JCS(D)1552]. Reaction of the stibolyl anion with cyclopentadienyllithium and then $FeCl_2$ gives monostibaferrocene **206** ($E = Sb$, $R^2 = R^5 = SiMe_3$, $R^3 = R^4 = Me$) (92OM1491). Reaction of 1-phenyl-2,5-bis(trimethylsilyl)-3,4-dimethylbismole with lithium gives the corresponding bismolyl. The latter, when reacting with $AlCl_3$ and then $LiCp$ and $FeCl_2$, produces a mixture of ferrocene and bismaferrocene **206** ($E = Bi$, $R^2 = R^5 = SiMe_3$, $R^3 = R^4 = Me$) (92JA372). The Bi—C bond has a multiple character and the C—C bonds are equivalent.

The range of phosphaferrrocenes has been extended to those containing functional silicon-containing exocyclic groups. Thus, disilylphosphaferrrocene can be obtained by isomerization of the bis(methylene)phosphane iron complex [91AG(E)312]. Complex **206** [$E = P$, $R^2 = R^5 = SiMe_2(C\equiv CMe)$, $R^3 = R^4 = Me$] was obtained by interaction of the corresponding silyl anion with [$FeCp(\eta^6-C_9H_{12})PF_6$] (99OM4205). The product enters the reaction with 4,6-di-*tert*-butyl-1,3,2-diazaphosphinine to give **207**, which in turn with excess trimethylsilylacetylene gives the phosphinine–phosphole compound **208**. It reveals the chelating properties with respect to $[Rh(cod)Cl]_2$, yielding **209**.

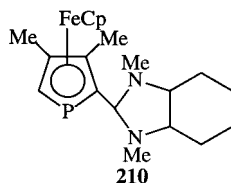
The phospholyl group in phosphaferrrocene is aromatic compared with the free ligand. The phosphole ring is almost planar. The d_{z^2} , d_{xy} , and $d_{x^2-y^2}$ orbitals of iron are more populated than the d_{xz} and d_{yz} orbitals. The phosphorus lone pair is in proximity to the five-member cycle. However, the phosphorus atom is a two-coordinate site and does not take part in direct bond formation with the iron atom. The lone pair is localized on the phosphorus atom but yet loses its nucleophilicity (81IC2966). The phospholyl rings are bonded more strongly than their cyclopentadienyl counterparts [83JOM(256)103; 83OM1008; 84CPL560; 89JPC6043; 93JOM(456)107].

Phosphaferrrocene **206** ($E = P$, $R^2 = R^5 = H$, $R^3 = R^4 = Me$) may be selectively acetylated via the C2 atom, leading to **206** ($E = P$, $R^2 = COMe$, $R^3 = R^4 = Me$,

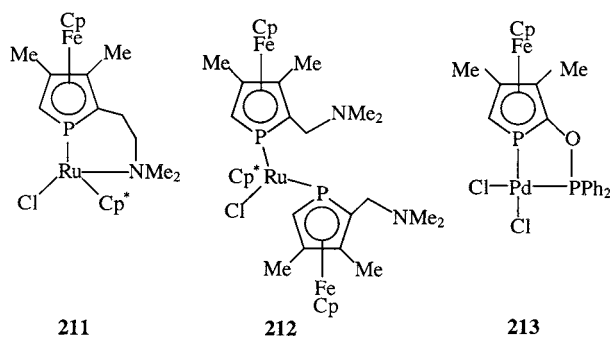


$R^5 = H$) (77JA3537). The unsubstituted phosphoferrocene **206** ($E = P$, $R^2 = R^3 = R^4 = R^5 = H$) is acetylated at positions 2 and 3, giving rise to the products **206** ($E = P$, $R^2 = COMe$, $R^3 = R^4 = R^5 = H$) and **206** ($E = P$, $R^3 = COMe$, $R^2 = R^4 = R^5 = H$) [77JOM(139)77]. Meanwhile, acetylation of the uncomplexed phosphole proceeds via the phosphorus atom. Vilsmeier formylation of **206** ($E = P$, $R^3 = R^4 = Me$, $R^2 = R^5 = H$) goes to position 2 of the heteroring (80JA994) to yield **206** ($E = P$, $R^2 = CHO$, $R^3 = R^4 = Me$, $R^5 = H$). The latter with nitromethane gives **206** ($E = P$, $R^2 = CH=CHNO_2$, $R^3 = R^4 = Me$, $R^5 = H$) (97OM2862). The reaction chain was continued, and reduction with sodium borohydride led to **206** ($E = P$, $R^2 = CH_2CH_2NO_2$, $R^3 = R^4 = Me$, $R^5 = H$) and then to **206** ($E = P$, $R^2 = CH_2CH_2NH_2$, $R^3 = R^4 = Me$, $R^5 = H$). Further nitroaldol condensation using H_2CO , $NaBH_3CN$, and $ZnCl_2$ gives **206** ($E = P$, $R^2 = CH_2CH_2NMe_2$, $R^3 = R^4 = Me$, $R^5 = H$). The formyl complex **206** ($E = P$, $R^2 = CHO$, $R^3 = R^4 = Me$, $R^5 = H$) also enters reductive amination with $HNMe_2$, NEt_3 , $TiCl_4$, and $NaBH_3CN$ and produces **206** ($E = P$, $R^2 = CH_2NMe_2$, $R^3 = R^4 = Me$, $R^5 = H$). In turn, with lithium alumohydride, diphenylchlorophosphine, and triethylamine in ether, the formyl derivative gives **206** ($E = P$, $R^2 = CH_2OPPh_2$, $R^3 = R^4 = Me$, $R^5 = H$). With (*R*),(*R*)-1,2-di(*N*-methylamino)cyclohexane **206** ($E = P$, $R^2 = CHO$, $R^3 = R^4 = Me$, $R^5 = H$) forms **210** [97T(A)2607]. These and some other derivatives find application in homogeneous catalysis [97CB177; 97JOC4534; 97JOM(548)17; 98EJIC1163; 98JOC4168; 98OM773].

Complexes **206** ($E = P$, $R^2 = CH_2CH_2NMe_2$, CH_2NMe_2 , $R^3 = R^4 = Me$, $R^5 = H$) were utilized as ligands with respect to $[Cp^*RuCl]_4$ (97OM2862). Their properties



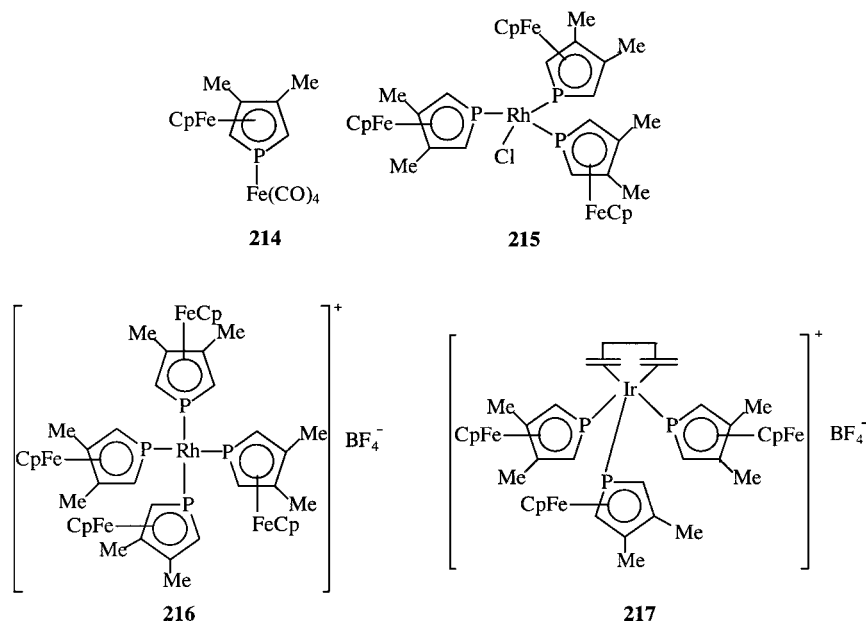
appeared different and species with $R^2 = \text{CH}_2\text{CH}_2\text{NMe}_2$ formed a chelate **211**, while that with $R^2 = \text{CH}_2\text{NMe}_2$ formed a trinuclear complex **212**. Chelating properties of **206** ($E = \text{P}$, $R^2 = \text{CH}_2\text{OPPh}_2$, $R^3 = R^4 = \text{Me}$, $R^5 = \text{H}$) with respect to $(\text{PhCN})_2\text{PdCl}_2$ were illustrated by isolation of **213**.



The nucleophilic properties of phosphorus in phosphoferrocene were demonstrated by reaction with *n*-butyllithium occurring at the phosphorus atom (81IC3252; 82OM312).

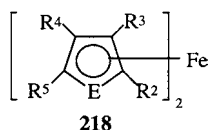
Reaction of phosphoferrocene with $\text{Fe}_2(\text{CO})_9$ leads to the corresponding P complex **214** [78JOM(154)C13]. The lengths of the three C—C bonds in the phosphole ring are equal and coincide with those in the initial phosphoferrocene [80JCS(D)2522]. The phosphole ring remains aromatic. This has two interesting consequences. First, the lone pair of the phosphorus atom does not take part in the delocalization in the phosphole ring of the free phosphoferrocenes. Second, the possibility of the C-electrophilic attacks via the phosphole ring remains even after the P-complex formation. Other illustrations of the P-donor properties of phosphoferrocenes exist [80NJC683; 84IC3455; 86ICA(119)165; 87ICA(130)93; 90JOM(384)271]. 3,4-Dimethylphosphoferrocene with $[\text{Rh}(\text{cod})\text{Cl}]_2$ gives species **215** (99OM807). In excess phosphoferrocene, **216** is formed. Reaction of **206** ($E = \text{P}$, $R^2 = R^5 = \text{H}$, $R^3 = R^4 = \text{Me}$) with $[\text{Rh}(\text{cod})_2]\text{BF}_4$ gives **216**, while with $[\text{Ir}(\text{cod})_2]\text{BF}_4$, only **217** is formed.

The synthesis, chemical properties, and structural analysis of 1,1'-diphosphoferrocene have been described [78JOM(156)C33; 80AX(B)1344; 80JA994;

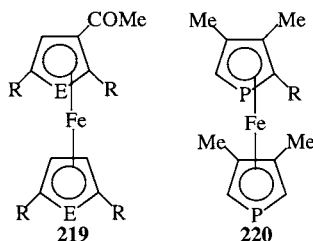


84OM1303; 87JOM(318)157; 87NJC585; 88OM921; 91AG(E)547; 93OM3373; 96MI3; 97JOC4534; 97T(A)2607; 98OM773]. The basic synthetic scheme includes preliminary splitting of the phospholyl-phenyl bond by alkali metals. The product **218** (E = P, R² = R⁵ = Ph, R³ = R⁴ = H) was not used for further studies because of low solubility. The product **218** (E = P, R² = R³ = R⁴ = R⁵ = H) is more soluble and more readily subject to C-electrophilic substitutions. A group of diphosphaferrocenes **218** (E = P, R² = SMe, SiMe₃, SnMe₃, R³ = R⁴ = Me, R⁵ = H) was obtained from the corresponding lithium phospholide and FeCl₂ (96BSCF541). Electrochemical studies of **218** (E = P, R² = R⁵ = H, R³ = R⁴ = Me) point to weak σ-donor and strong π-acceptor functions of the phosphorus atom [85JOM(295)189]. The phospholide group in diphosphaferrocenes is aromatic [97JOM(527)305]. Tetrakis(trimethylsilyl) diphosphaferrocene (97PSS203) and derivative **218** [E = P, R² = R⁵ = SiMe₂(C≡CMe), R³ = R⁴ = Me] (99OM4205) were obtained using traditional synthetic methods. 1,1'-Diarsaferrocene **218** (E = As, R² = Ph, R³ = R⁴ = R⁵ = H) has been obtained from 1-phenylarsole [79ICA(32)L67; 87AG(E)229; 94OM4067]. The arsacyclopentadienyl ring is more effective in accepting electron density from the metal, which stabilizes the HOMO and adds charge to the LUMO with respect to cyclopentadienyl. The arsolyl ring is aromatic. The complex **218** (E = As, R² = R³ = R⁴ = R⁵ = H) is subject to a rapid catalyzed exchange and to the C-acetylation at position 2. Reaction of the stibacyclopentadienyl with ferrous chloride gives a separable mixture

of bis(2,5-dimethylstibacyclopentadienyl)iron and 2,2',5,5'-tetramethylstibolyl [80JOM(202)C95]. A more or less complete series of 1,1'-diheteroferrocenes **218** ($E = P, As, Sb, Bi, R^2 = R^3 = R^4 = R^5 = H$; $E = P, As, Sb, Bi, R^2 = R^5 = Me, R^3 = R^4 = H$; $E = P, As, Sb, R^2 = R^3 = R^4 = R^5 = Me$; $E = P, As, Sb, R^2 = R^5 = H, R^3 = R^4 = Me$; $E = Sb, R^2 = R^5 = SiMe_3, R^3 = R^4 = Me$) was synthesized on the basis of 1-phenylheteroles, lithium, and further $FeCl_2$ [80JOM(202)C95; 81JA207; 91OM2689; 92OM1491; 92OM2743; 95OM2689].

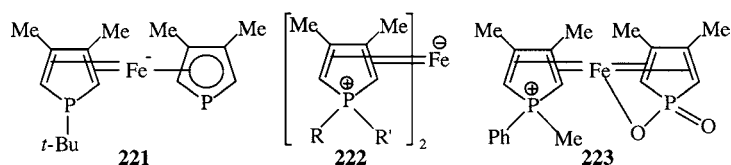


Friedel–Crafts acetylation of **218** ($E = P, R^2 = R^5 = H, R^3 = R^4 = Me$) using stoichiometric amounts of $MeCOCl-AlCl_3$ occurs at room temperature [80JA994; 83JOM(257)209]. This diphosphaferrocene gives the diacetylated product **218** ($E = P, R^2 = COMe, R^3 = R^4 = Me, R^5 = H$) with excess $MeCOCl-AlCl_3$, which is a mixture of two diastereomers (95OM2689). For the diphosphaferrocenes with the occupied positions 2 and 5, the 3 substitution takes place to yield **219** ($E = P, R = Ph; E = As, R = Me$). Vilsmeier hydroformylation of **218** ($E = P, R^2 = R^5 = H, R^3 = R^4 = Me$) gave monoaldehyde **220** ($R = CHO$) but attempts to obtain the dialdehyde appeared unsuccessful (80JA994). Secondary and tertiary alcohols were obtained as a result of reaction of the monoacetyl derivative with lithium alumohydride or borohydride. The secondary alcohol **220** [$R = C(OH)Me_2$] was obtained from the reaction of the monoacetyl derivative with Grignard reagent. Reaction of the complex with $(AlH_3)_n$ gave the ethyl derivative **220** ($R = Et$). Reaction with *n*-butyllithium did not lead to formation of the lithium derivative.



However, reaction of **218** ($E = P, R^2 = R^5 = H, R^3 = R^4 = Me$) with *tert*-butyllithium most probably yields **221**. The phospholyl loses its electrophilicity and the iron atom bears a considerable negative charge. Addition of *tert*-butyllithium (one equivalent) followed by methyl iodide (one equivalent) does not give any isolable product but leads to recovery of the starting **218** only. In excess *tert*-butyllithium and methyl iodide, **222** ($R = t-Bu, R' = Me$) was isolated (81IC3252).

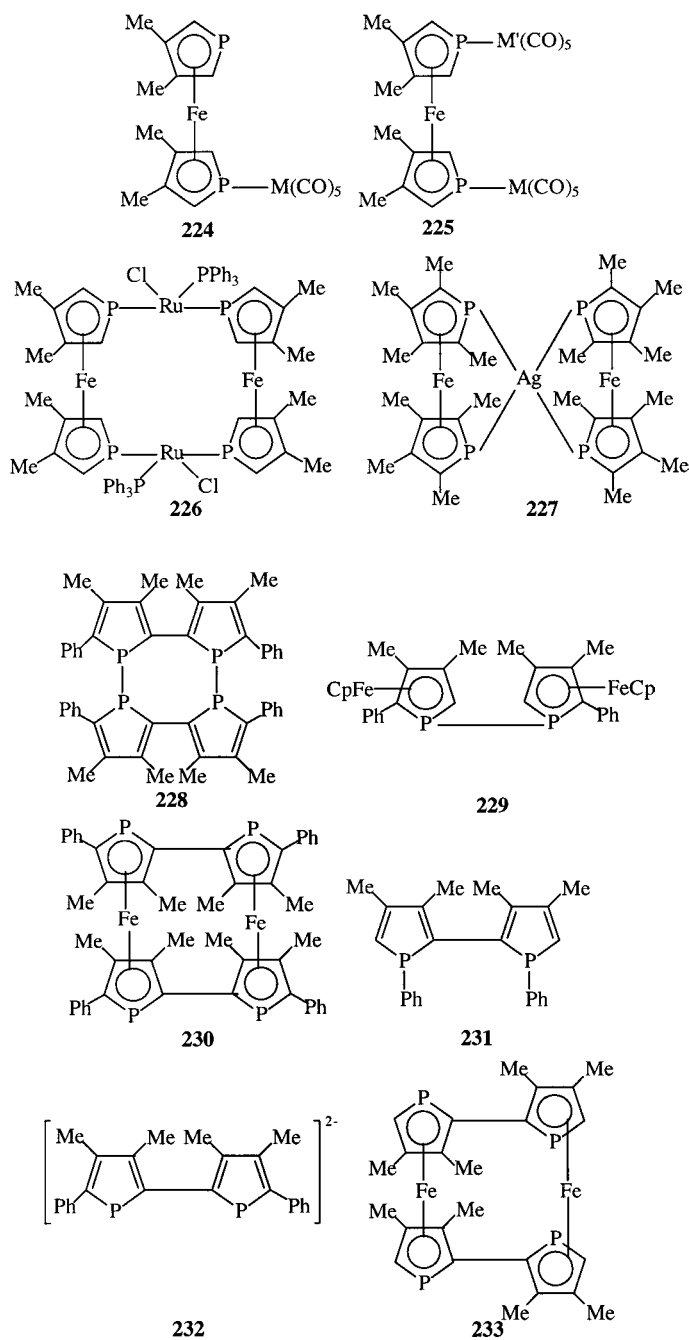
The phosphole groups in **222** ($R = t\text{-Bu}$, $R' = \text{Me}$) are η^4 -ligands through their dienic systems. Formally, **222** ($R = t\text{-Bu}$, $R' = \text{Me}$) contains the 17-electron iron atom, forming a sandwich between two phosphole groups. The product reacts easily with an aqueous solution of hydrochloric acid with the formation of the free phospholium. Similar experiments with methyllithium led to the isolation of **222** ($R = R' = \text{Me}$). If benzyl bromide and *tert*-butyllithium are used, **222** ($R = \text{PhCH}_2$, $R' = t\text{-Bu}$) is formed. Addition of phenyllithium and excess methyl iodide to a THF solution of **218** ($E = \text{P}$, $R^2 = R^5 = \text{H}$, $R^3 = R^4 = \text{Me}$) gives a covalent diamagnetic complex **223**.



The complex **218** ($E = \text{P}$, $R^2 = R^5 = \text{Ph}$, $R^3 = R^4 = \text{H}$) is easily dissolved in concentrated sulfuric acid [89ICA(155)45]. Protonation occurs at the iron atom. Sulfonation is more favorable at the phenyl rather than the five-member cycle. The latter is deactivated with respect to electrophilic attack by the phosphorus atom. The strongest of the known protic acids, trifluoromethanesulfonic acid, having neither oxidizing nor sulfonating properties, causes protonation at the iron atom. Protonation of **218** ($E = \text{P}$, $R^2 = R^5 = \text{H}$, $R^3 = R^4 = \text{Me}$) by means of trifluoroacetic acid proceeds via the iron atom. Acetyl derivatives are protonated at the oxygen atom of the $\text{C}=\text{O}$ group, and in the case of the secondary alcohols, the α -carbenium ions are formed [89ICA(157)45].

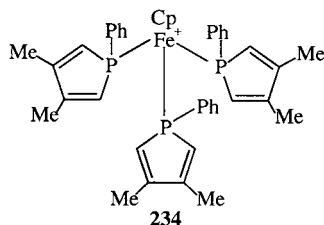
Diphosphaferrocenes are potential bidentate ligands [84IC3455; 86JOM(298)133]. However, reaction with $\text{M}(\text{CO})_5(\text{THF})$ ($\text{M} = \text{Cr}, \text{Mo}, \text{W}$) leads to formation of the monocoordinated complexes **224** ($\text{M} = \text{Cr}, \text{Mo}, \text{W}$). Further reaction of **224** ($\text{M} = \text{Cr}, \text{Mo}, \text{W}$) with the same or other metal carbonyls $\text{M}'(\text{CO})_5(\text{THF})$ leads to homo- and heterobinuclear complexes of trinuclear nature **225** ($\text{M} = \text{M}' = \text{Cr}, \text{Mo}, \text{W}$; $\text{M} = \text{Mo}, \text{M}' = \text{Cr}$; $\text{M} = \text{Mo}, \text{M}' = \text{W}$). With $(\text{Ph}_3\text{P})_3\text{RuCl}_2$, the dimer **226** was produced containing the bridging ligand and terminal chlorine atoms. The phosphoferrocene framework forms stronger bonds between ruthenium and phosphorus atoms than the phosphine ligand. Diphosphaferrocene **218** ($E = \text{P}$, $R^2 = R^3 = R^4 = R^5 = \text{Me}$) in an electrochemical cell with silver anode forms a chelate **227** (93IC1527).

1-Phenyl-3,4-dimethylphosphole isomerizes into 2-phenyl-3,4-dimethyl-5*H*-phosphole at 440 K, giving an unstable two-coordinate compound which transforms into **228** containing four phosphole units. It reacts with $[\text{CpFe}(\text{CO})_2]_2$ at 420 K, leading to **229**, the phosphorus analog of biferrocene (82JA2077). Similarly,



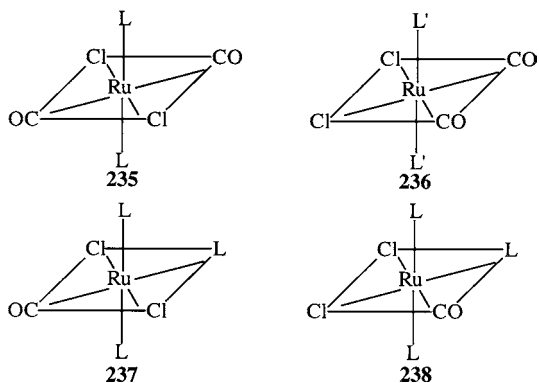
reaction of **228** with MgBr_2 followed by FeCl_2 leads to the bis(fulvalene)diiron analog **230**. Biphospholyl **231** undergoes splitting of exocyclic P—Ph bonds upon the action of lithium in THF [86JOM(316)271]. As a result, a dianion **232** is formed. The latter is the initial reagent for bimetallic η^5 complexes. Thus, reaction with anhydrous ferrous chloride gives the bis(diphosphafulvalene) diiron complex as a mixture of the two isomeric forms, one of which is the head-to-tail isomer **233**.

The complex cation $[\text{Cp}(\eta^1\text{-2,5-dimethylthiophene})\text{Fe}]^+$ with 3,4-dimethylphosphole gives the σ complex **234** on photolysis [84JOM(272)417].

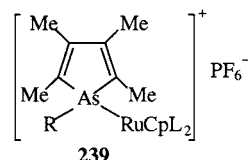


E. COMPLEXES OF RUTHENIUM, OSMIUM, NICKEL, PALLADIUM, PLATINUM, AND LANTHANIDES

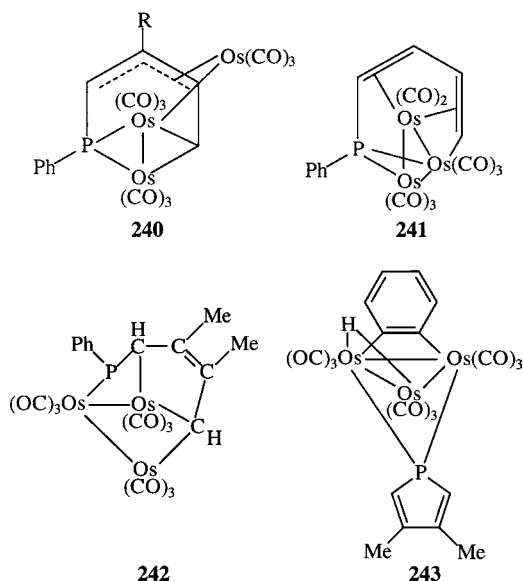
Upon carbonylation of $\text{RuCl}_3 \cdot 3\text{H}_2\text{O}$ in boiling 2-methoxyethanol and subsequent addition of 3,4-dimethylphosphole (L) under CO, **235** is formed (89IC3831). Dibenzophosphole (L') with $[\text{RuCl}_2(\text{CO})_2]_n$ forms **236**. Species **235** is stable below 425 K; at 430 K, ligand redistribution process starts, leading to a mixture of **236**, **237**, and **238** (L instead of L'). Other complexes of similar nature were described (75MI2; 83IC2476; 87OM2216).



1-Substituted tetramethylarsoles react with CpRuL_2Cl [$\text{L}_2 = (\text{PPh}_3)_2$, dppm , dpme , $(\text{PMe}_3)_2$, C_7H_8] to yield the cationic complexes **239** [$\text{R} = \text{Me}$, $\text{L}_2 = (\text{PPh}_3)_2$, dppm , dpme ; $\text{R} = \text{Me}$, Cp^* instead of Cp , $\text{L}_2 = (\text{PMe}_3)_2$, C_7H_8 ; $\text{R} = t\text{-Bu}$, $\text{L}_2 = \text{dppm}$] [94JOM(467)67].

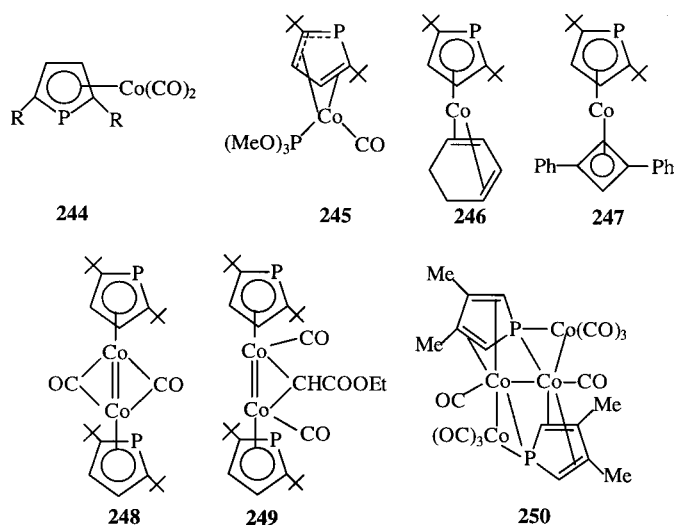


1-Phenylphosphole with $[\text{Os}_3(\text{CO})_{12}]$ and $[\text{Os}_3(\text{CO})_{11}(\text{AN})]$ under reflux conditions gives rise to **240** ($\text{R} = \text{H}$) and **241** as isolable products [91JOM(408)C18]. 1-Phenyl-3,4-dimethylphosphole with $[\text{Os}_3(\text{CO})_{12}]$ or $[\text{Os}_3(\text{CO})_x(\text{AN})_{10-x}]$ ($x = 1, 2$) yields **242** and **243**. The latter, however, experiences subsequent oxidative addition to give **240** ($\text{R} = \text{Me}$). Species **242** and **240** ($\text{R} = \text{Me}$) mutually transform into each other, the direct process induced by light and the reverse occurring in dark [91JCS(D)3381].

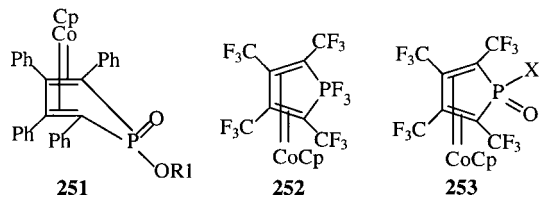


2,2',5,5'-Tetraphenyl- and -tetra-*tert*-butylphospholyls react with $\text{Co}_2(\text{CO})_8$ to give **244** ($\text{R} = \text{Ph}$, *t*-Bu) catalytically active in cyclooligomerization of acetylene [82JOM(231)361; 94JCS(CC)1167; 94JCS(CC)2459; 97OM2049]. Substitution of one of the carbonyl ligands by trimethyl phosphite in **244** ($\text{R} = t\text{-Bu}$) causes the re-switch of the coordination mode to the $\eta^3:\eta^2$, **245**. Substitution of both

carbonyls by cyclohexadiene gives the η^5 -coordinated species **246**, which interacts with phenylacetylene giving rise to **247**, the cyclobutadiene complex. Photolysis of **244** ($R = t\text{-Bu}$) leads to **248**, the binuclear complex with the cobalt–cobalt double bond. The product **248** with diazoalkane reacts via the cobalt–cobalt bond to yield carbene **249**. 3,3',4,4'-Tetramethyl-1,1'-biphospholyl in these circumstances gives the $\eta^1:\eta^4$ coordinated species **250** (83OM1234).

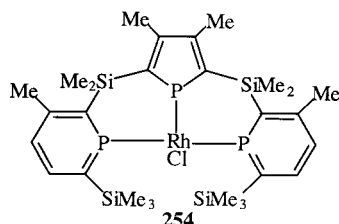


η^5 -Cyclopentadienyl(triphenylphosphine)cobalt reacts with phosphites and forms complexes of 1-alkoxyphosphole oxides **251** ($R = \text{Me, Et, Ph}$) through a step involving $(\eta^5\text{-cyclopentadienyl})(\text{phosphite})\text{cobalt}$ (80JA4363). $(\eta^5\text{-Cp})\text{Co}(\text{PF}_3)_2$ reacts with hexafluorobut-2-yne and **252** is formed, which hydrolyzes into **253** ($X = \text{OH}$) [73JCS(CC)583; 75JCS(D)197]. The five-member ring has the envelope conformation, in which the carbon atoms are coplanar, and the phosphorus atom deviates from this plane in the direction opposite to the cobalt atom. The heterocycle is a four-electron donor relative to the metal center.

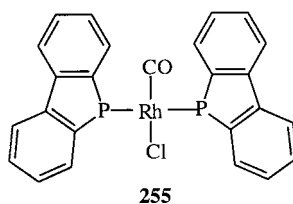


Some other phospholyl complexes of cobalt are known (91OM2631; 95CCR201).

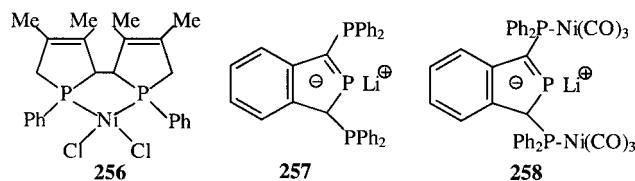
The phosphine–phosphole ligand with $[\text{Rh}(\text{cod})\text{Cl}]_2$ affords complex **254** (99OM4205).



1-Phenyl-3,4-dimethylphosphole (L) or 1-phenyldibenzophosphole (L) with $\text{RhCl}_3 \cdot 3\text{H}_2\text{O}$ and CO give $\text{L}_2\text{Rh}(\text{CO})\text{Cl}$, e.g., **255** (57JCS2284; 78JHC1239; 93IC1048). Complex **255** appeared to be an efficient homogeneous catalyst of hydrogenation processes [74CJC735; 82JCS(CC)721]. This has been attributed to the fact that the phosphorus heteroatom participates in the π -conjugated system (76JHC1; 87MRC774).

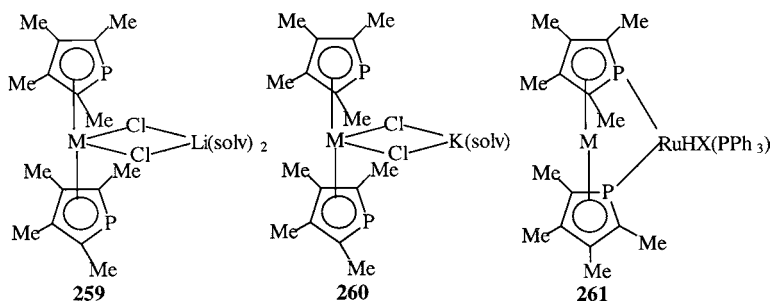


1-Phenyl-3,4-dimethylphosphole experiences an unusual transformation in the presence of nickel(II) chloride and yields **256** (84JA425; 85IC4141). The study of the nickel(II) complexes of 1-methyl- and 1-ethyldibenzophosphole (75CCR239) points to better donor properties of dibenzophosphole ligands compared to triphenylphosphine [71JCS(A)1803; 71JCS(CC)1037; 77PS381; 89IC3453]. Ligand **257** with $\text{Ni}(\text{CO})_4$ forms the dinuclear complex **258** where the coordination is via the exocyclic phosphorus atoms (95BSCF280; 99EJC1169)

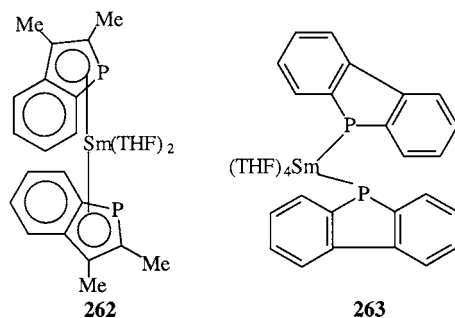


The classical $\eta^1(\text{P})$ complexes were basically described for the phosphole chemistry of palladium(II) and platinum(II) (80IC709; 82IC2145; 84IC449; 85JA6939; 89IC217).

Reaction of lithium 2,3,4,5-tetramethylphospholide with yttrium(III) and lutetium(III) chlorides yields bis(phospholyl)complexes **259** ($M = Y, Lu$) [89JCS(CC)800; 95MI1]. In turn, potassium 2,3,4,5-tetraphenylphospholide with $MCl_3(THF)_3$ ($M = Sm, Nd$) gives **260** ($M = Sm, Nd$) (95IC1306; 99EJIC1041). Some derivatives of these complexes were described. Thus, **260** ($M = Sm, Nd$) with $LiCH(SiMe_3)_2$ gives rise to $[(C_4Me_4P)_2MCH(SiMe_3)_2]$ ($M = Sm, Nd$). The behavior of Sm and Nd species **260** with respect to molecular hydrogen is different. For samarium species, the products are $[(C_4Me_4P)_2Sm(THF)_n]$ and bis(trimethylsilyl)-methane, while for neodymium complex $(C_4H_4P)_2NdH$ results. The $\eta^1(P): \eta^5(PC_4)$ bonding mode in phospholyl-samarium complexes is noted as well [90AG(E)1485; 92JCS(D)3047; 94JOM(466)107]. Complex **259** ($M = Yb$) reacted with $RuH_4(PPh_3)_3$ to give **261** ($M = Yb, X = THF, Y = Cl$). Similar products **261** ($M = U, X = BH_4, Cl, Y = H, Cl$) result from **259** ($M = U$) and **259** [$M = U$, instead of $(solv)_2, (BH_4)_2$] (93P19) and $RuH_4(PPh_3)_3$. However, the uranium species were not isolated (95NJC921; 96OM4178).



Potassium 2,3-dimethylphosphindolide and $SmI_2(THF)_2$ give the η^5 complex **262** [94JOM(464)149]. Bis(dibenzophospholyl) and metallic samarium activated by $HgCl_2$ lead to the η^1 -coordinated species **263**.

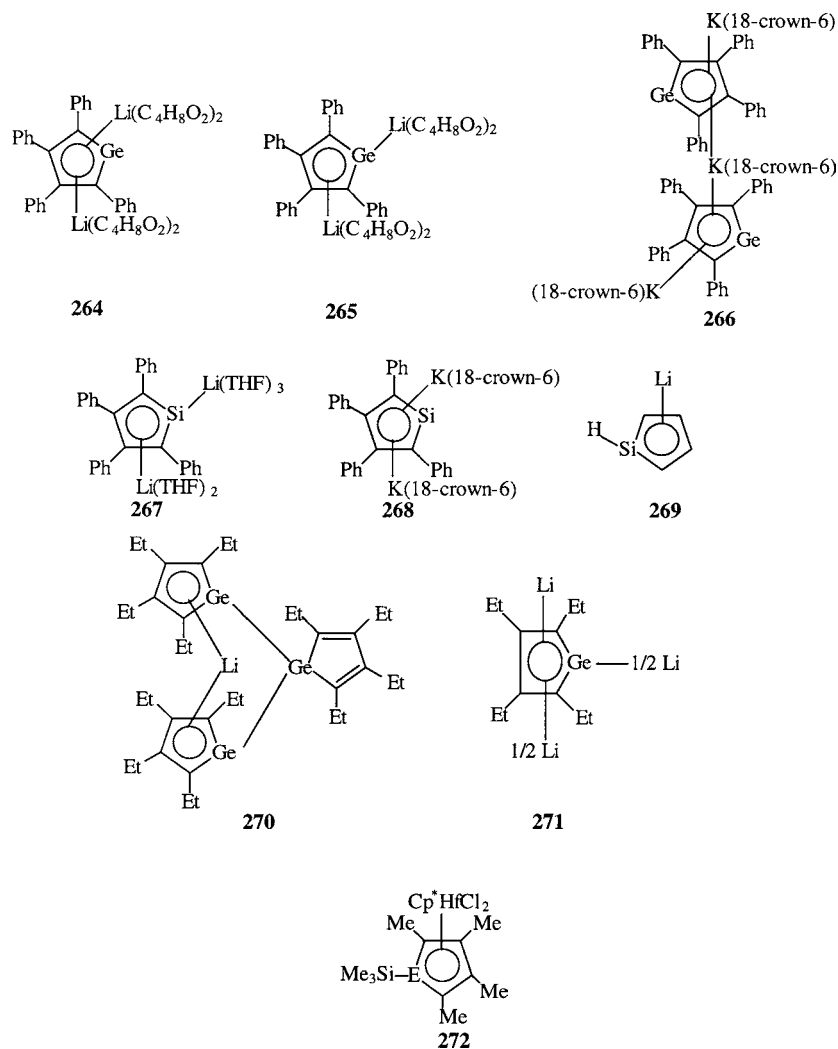


V. Organometallic Complexes of Siloles and Germoles

Because of the similarity between the cyclopentadienyl anion and anions of siloles and germoles, the syntheses of the sila- or germacyclopentadienyl ligands are of interest [89MI1; 90CRV215; 98JCS(D)3693; 98MI2]. The η^5 mode was proposed based on mass-spectral data but a long time passed before it could be further substantiated [73JOM(63)C10; 74HCA167; 76JOM(110)303]. Then followed a lengthy period of prevalence of the η^4 complexes [80JOM(191)209]. The free silole or germole is almost planar, but in the η^4 complexes, the dihedral angles between SiC2C5 and the diene framework appear to be 20–32°. Sila- or germacyclopentadienyl is, however, considered to be aromatically stabilized [84JOM(273)141; 85CRV419; 86MI1; 86PIC1; 87AG(E)1201; 87PAC1011; 90CRV265; 90CRV283; 90JOM(391)27; 93CCR237; 93JA5883; 94JA8428; 94OM3387; 95AG(E)337; 95JA11361; 95JA11608; 95OM1553; 96AG(E)828; 96AG(E)882; 96JA5745; 96JA10457; 96OM1755; 97CL1179; 97OM1543; 97OM2270]. Thus, the tetraphenylgermole dianion was considered as a π -delocalized aromatic compound (93JA5883; 95BSCF495), while the monoanion [(MeC)₄GeSi(SiMe₃)₃][−] is described as a species with localized bonds (90OM3001; 95AG(E)1887). Although for silole the HOMO level is relatively high, the LUMO energy is substantially lower than that of cyclopentadiene (75CP385; 83JA4972; 85OM636; 86JOC5028; 94JA320; 96BCSJ2327). This determines the acceptor properties of the silole ring. LUMO describes the $\sigma^*-\pi^*$ conjugation of the silylene and butadiene moieties [96JCS(CC)1873; 99IC2464].

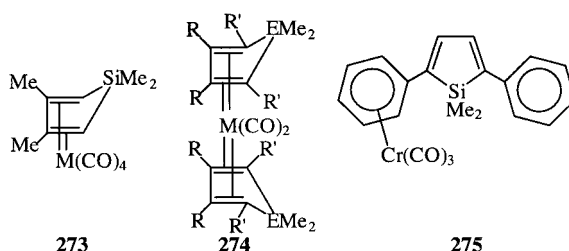
These considerations led to the preparation of the η^5 -coordinated complexes of siloles and germoles (94PAC1431). A number of facts in support of this view were obtained by X-ray determination of the structures of the organometallic derivatives of nontransition metals. Thus, the dianion of tetraphenylgermole forms a sandwich dilithium salt **264** in dioxane at −20°C [96AG(E)1002]. At 25°C, however, the salt with a mixed $\eta^5:\eta^1$ coordination mode **265** results. Columnar structure is observed for **266** (97OM1543). The product **267** (94OM3387; 95BSCF495; 95JA11608; 96OM1755), similar to **265**, was made from the dianion of tetraphenylsilole in THF. The π delocalization within the heterorings in **265** and **267** is noted. Species **268** possesses similar properties [96AG(E)882; 96JA10457]. Moreover, Li complexation increases the aromaticity of the silole monoanion **269** (95OM1553). The same situation can be observed in tris(germole) **270** [96AG(E)186]. 1,1-Dichloro-2,3,4,5-tetraethylgermole and excess lithium yield compound **271** (99OM2919). The mixed $\eta^5:\eta^1$ coordination mode was assigned to the product of interaction of 1,1-dichloro-3-*n*-butylsilaindene and lithium (98JA5814).

The first η^5 -transition metal organometallic complexes **272** were made from Li(Me₄C₄ESiMe₃) (E = Si, Ge) and Cp^{*}HfCl₃ (98JA8245). Species **272** (E = Si) with trimethylphosphine forms the Hf–PMe₃-adduct.



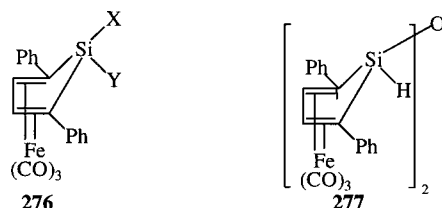
Thermal reactions of 1,1-dimethylsilole and 1,1,3,4-tetramethylsilole and germole with $(\eta^4\text{-cod})\text{M}(\text{CO})_4$ lead to formation of chromium, molybdenum, and tungsten complexes **273** ($\text{M} = \text{Cr}, \text{Mo}, \text{W}$) and **274** ($\text{E} = \text{Si}, \text{R} = \text{H}, \text{Me}, \text{R}' = \text{H}$ $\text{M} = \text{Mo}, \text{W}$; $\text{E} = \text{Ge}, \text{R} = \text{Me}, \text{R}' = \text{H}, \text{M} = \text{Mo}$) (83OM1901; 87OM1398). 1,1-Dimethyl-2,5-diphenyl-1-silacyclopentadiene reacts differently with $\text{Mo}(\text{CO})_6$ and $\text{Cr}(\text{CO})_6$. Prolonged reaction with molybdenum hexacarbonyl gives **274** ($\text{E} = \text{Si}, \text{R} = \text{H}, \text{R}' = \text{Ph}, \text{M} = \text{Mo}$), while reaction with chromium hexacarbonyl gives **275**

where $\text{Cr}(\text{CO})_3$ is coordinated via the arene cycle [75JCS(CC)698; 76JCS(D)2484; 87JOM(335)91]. (η^6 -1-Methyl-1-(trimethylsilyl)dibenzosilacyclopentadiene]tricarboxyl chromium is also known [84JOM(271)C4].)

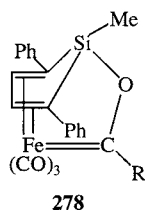


Until recently, the viewpoint predominated that siloles play a role as η^4 donors in organometallic complexes of group VIII metals [70AC(P)199; 73AC(P)123; 73JOM(63)C7; 74JOM(71)C8; 77JOM(128)57; 79JOM(165)399; 80AG(E)484; 81CR(2)331; 81JOM(214)289; 81JOM(215)27; 83JOM(244)C12; 83TL3521; 85JOM(293)295; 86OM910; 87JOM(320)C7]. Thus, $[(\text{C}_4\text{Ph}_4\text{GeMe})\text{Fe}(\text{CO})_3]^+$ represents an η^4 -diene complex with the positive charge localized on the germanium atom [80AG(E)484]. Treatment of 1,1-dimethoxy-2,5-diphenylsilacyclopentadiene with $\text{Fe}_2(\text{CO})_9$ gives **276** ($\text{X} = \text{Y} = \text{OMe}$) [85JOM(293)295]. Reduction of **276** ($\text{X} = \text{Y} = \text{OMe}$) with *i*- Bu_2AlH gives the stable dihydro complex **276** ($\text{X} = \text{Y} = \text{H}$). The availability of the Si-disubstituted η^4 -silole derivatives makes it possible to compare the relative reactivity of the *exo* and *endo* substituents at one silicon atom by directly using the complex **276** ($\text{X} = \text{Y} = \text{H}$). Reaction with PX_5 ($\text{X} = \text{Cl}, \text{Br}$) leads to **276** ($\text{X} = \text{Cl}, \text{Br}, \text{Y} = \text{H}$), while with $\text{Ph}_3\text{C}^+\text{BF}_4^-$, **276** ($\text{X} = \text{F}, \text{Y} = \text{H}$) is obtained; again, reaction with water yields **277**, and with alcohols, ROH ($\text{R} = \text{Me}, p\text{-MeOC}_6\text{H}_4$) gives **276** ($\text{X} = \text{OMe}, p\text{-MeOC}_6\text{H}_4$) is formed. Thus, irrespective of the nature of the reactant, the *exo* isomer is selectively formed [87JOM(320)C7]. Si—H bond cleavage occurs with retention of configuration at the Si atom. Nucleophilic substitution is governed not by the steric hindrance of the metal-containing groups but by ring strain. If one uses **276** ($\text{X} = \text{H}, \text{Y} = \text{Me}$) as the reference complex, then substitution occurs via the Si—H bond [87JOM(320)C1]. Substitution reactions of **276** ($\text{X} = \text{H}, \text{Y} = \text{Me}$) lead to the products **276** ($\text{X} = \text{Cl}, \text{OMe}, \text{Y} = \text{Me}$). **276** ($\text{X} = \text{Cl}, \text{Y} = \text{Me}$) was obtained by reaction with PCl_5 , and **276** ($\text{X} = \text{OMe}, \text{Y} = \text{Me}$) by reaction with MeOH and H_2O . Substitution reactions with **276** ($\text{X} = \text{Cl}, \text{Y} = \text{Me}$) refer to the silicon–chlorine bond. The product of the reaction of **276** ($\text{X} = \text{Cl}, \text{Y} = \text{Me}$) with LiAlH_4 is **276** ($\text{X} = \text{H}, \text{Y} = \text{Me}$), while with EtMgBr it is **276** ($\text{X} = \text{Et}, \text{Y} = \text{Me}$).

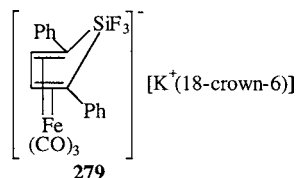
Retention of configuration may be used for the synthesis of the intramolecular carbene complexes [87JOM(336)C1]. Methyl- or phenyllithium or di-



isopropylaminolithium react with **276** ($X = \text{Me}$, $Y = \text{Cl}$) leading to the new cyclic carbenes, again with retention of configuration, **278** ($R = R' = \text{Me}$, $R = \text{Me}$, $R' = \text{Ph}$; $R = \text{Me}$, $R' = i\text{-Pr}_2\text{N}$). The silole moiety is very near to planar.



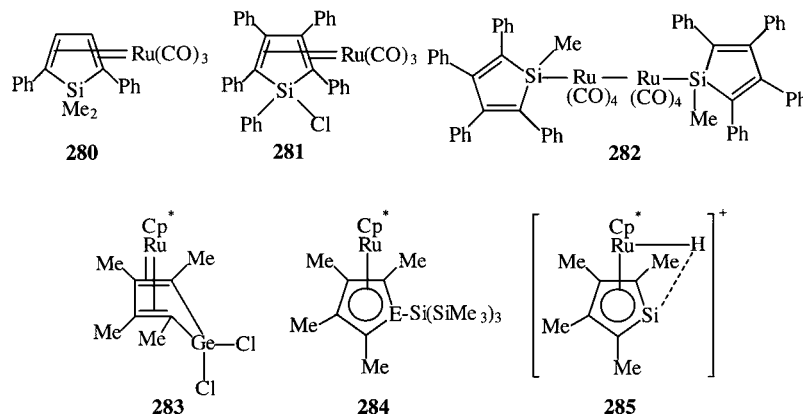
Treatment of (1,1-difluoro-2,5-diphenylsilacyclopentadiene)tricarbonyliron with a mixture of potassium fluoride and 18-crown-6 gives **279** [88JOM(347)C1].



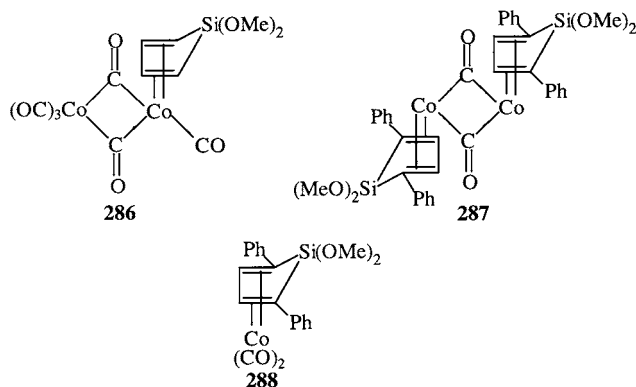
The polymeric species containing silole nuclei also form the $\text{Fe}(\text{CO})_3$ complexes η^4 -coordinated via the heteroring on interaction with $\text{Fe}(\text{CO})_5$ [93JOM(456)35; 99OM1717].

Reaction between $\text{Ru}_3(\text{CO})_{12}$ and 1,1-dimethyl-2,5-diphenylsilacyclopentadiene gave **280** [76JCS(D)2484; 78JOM(151)1]. 1-Chloro-1,2,3,4,5-pentaphenyl-1-silacyclopentadiene with $\text{Ru}_3(\text{CO})_{12}$ gives the same type of a product, **281**. If there is a 1-hydroxido group in the starting ligand, like $\text{C}_4\text{Ph}_4\text{SiMeH}$, a unique silicon-coordinated binuclear product **282** was generated [76JCS(D)2484]. 1,1-Dichloro-2,3,4,5-tetramethylgermole with $\{\text{Cp}^*\text{Ru}(\mu\text{-Cl})\}_4$ also gives the η^4 complex **283** [93AG(E)1744]. The existence of the $\text{Ru} \rightarrow \text{Ge}$ dative interaction between ruthenium and σ^* orbital of the germanium-chlorine bond prompted the authors to search for the first η^5 -coordinated germole in the group VIII series. Such a

complex, **284** (E = Ge), was successfully made from the (germacyclopentadienyl)lithium containing the bulky Si(SiMe₃)₃ group in position 1 and [$\{\text{Cp}^*\text{Ru}(\mu\text{-Cl})_4\}$]. Later, the analogous complexes of silole, **284** (E = Si) and **285**, were prepared [94JA8428].

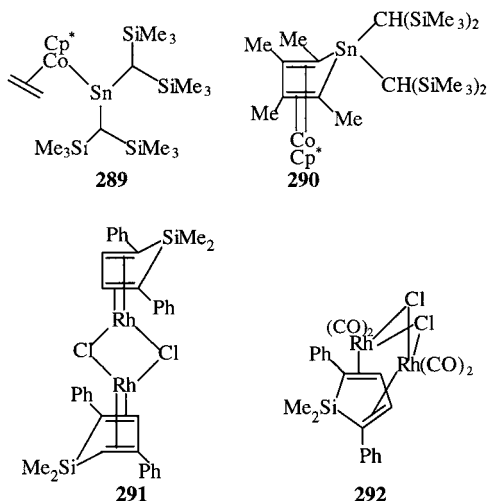


Silole reacts with Co₂(CO)₈ to form the monosubstituted η^4 -silacyclopentadiene complex **286** [85JOM(293)295]. The 2,5-diphenyl analog of the latter with excess silole yields the corresponding disubstituted derivative **287** [87JOM(320)C7]. The similar complex was previously made for the 1,1-dimethyl derivative [76JCS(D)2484]. Treatment of **287** with iodine gives **288**.



The only stannole complex **290** was found by cycloaddition of species **289** to 2-butyne [98ICA(281)53].

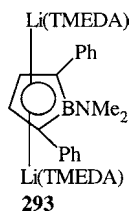
1,1-Dimethyl-2,5-diphenylsilacyclopentadiene with [Rh(CO)₂Cl]₂ gives **291** and mainly **292** [76JCS(D)2484].



VI. Organometallic Complexes of Boroles

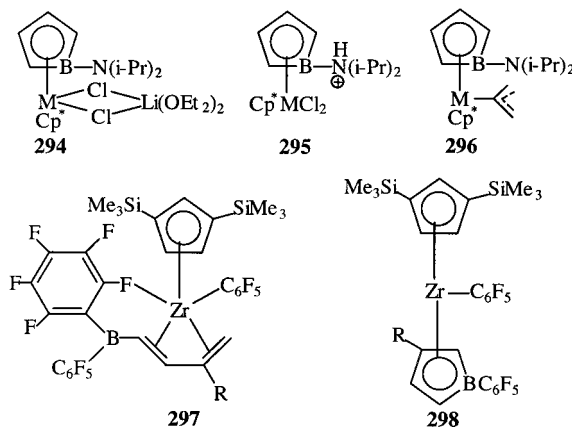
1*H*-Boroles are 4 π -electron systems with a small HOMO–LUMO energy gap (96OM1315). They generally tend to form the η^5 -coordinated organometallic species when the exocyclic substituent at the boron atom is alkyl or allyl. In this case, the effect of the π -back donation from the transition metal toward the electron-deficient boron site enhances the electrophilicity of the metal. However, when this substituent possesses π -donor properties such as an amino group [83ZN(B)1388; 85CB4303; 86CB420; 90AG(E)317; 91CB17; 91CB25], the situation becomes unequivocal (95MI2). Such substituents may weaken the metal–boron interaction and lead to the dienic type η^4 coordination.

Reaction of 1-(dimethylamino)-2,5-dihydro-2,5-diphenyl-1*H*-borole with diisopropylaminolithium and then TMEDA gives **293** [90AG(E)317; 95JOM (502)67].



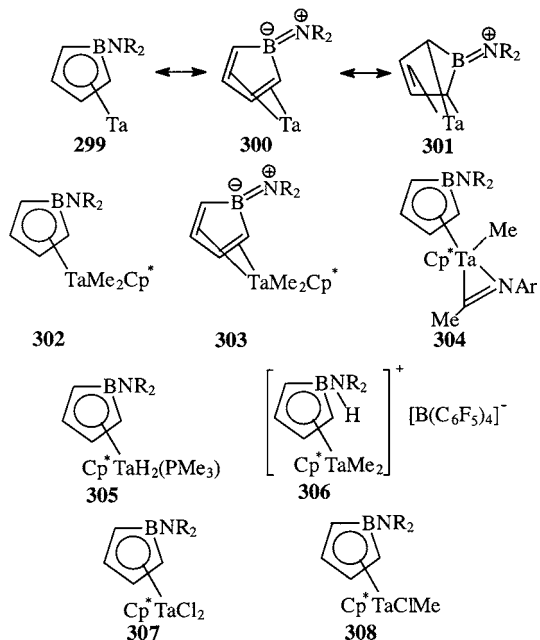
Aminoborolide dianion [C₄H₄BN(*i*-Pr)₂]^{2−} taken as the THF solvate of a lithium salt with Cp**M*Cl₃ (*M* = Zr, Hf) in ether gives **294** (94JA4489). The nitrogen atom

appears to be the protonation site for HCl whose H—Cl bond is cleaved heterolytically to yield **295**. Methyl iodide adds oxidatively and gives rise to $\text{Cp}^*[(\eta^5\text{-C}_4\text{H}_4\text{BN}(\text{Me})(i\text{-Pr})_2)\text{M}-\text{Cl}(\text{I})]$ ($\text{M} = \text{Zr}, \text{Hf}$) (96JA2291; 96OM387). Reactions of $\text{Cp}^*(\eta^5\text{-C}_4\text{H}_4\text{BN}(i\text{-Pr})_2)\text{ZrR}_2\text{Li}$ ($\text{R} = \text{Me}, \text{CH}_2\text{Ph}, \text{C}\equiv\text{CCMe}_3, \text{C}\equiv\text{CC}_6\text{H}_4\text{Me-}p$) were studied [84OM28; 97JOM(528)65]. The same species and its hafnium analog with allyl magnesium bromide give **296** ($\text{M} = \text{Zr}, \text{Hf}$). Complex **296** ($\text{M} = \text{Hf}$) forms adducts with trimethylphosphine, pyridine, and carbon monoxide. Complexes of the group IV metals were also made by cyclization of species **297** ($\text{R} = \text{H}, \text{Me}$) that occurs upon thermolysis to give **298** ($\text{R} = \text{H}, \text{Me}$) (98JA6816). The products **298** ($\text{R} = \text{H}, \text{Me}$) form adducts with diethyl ether.



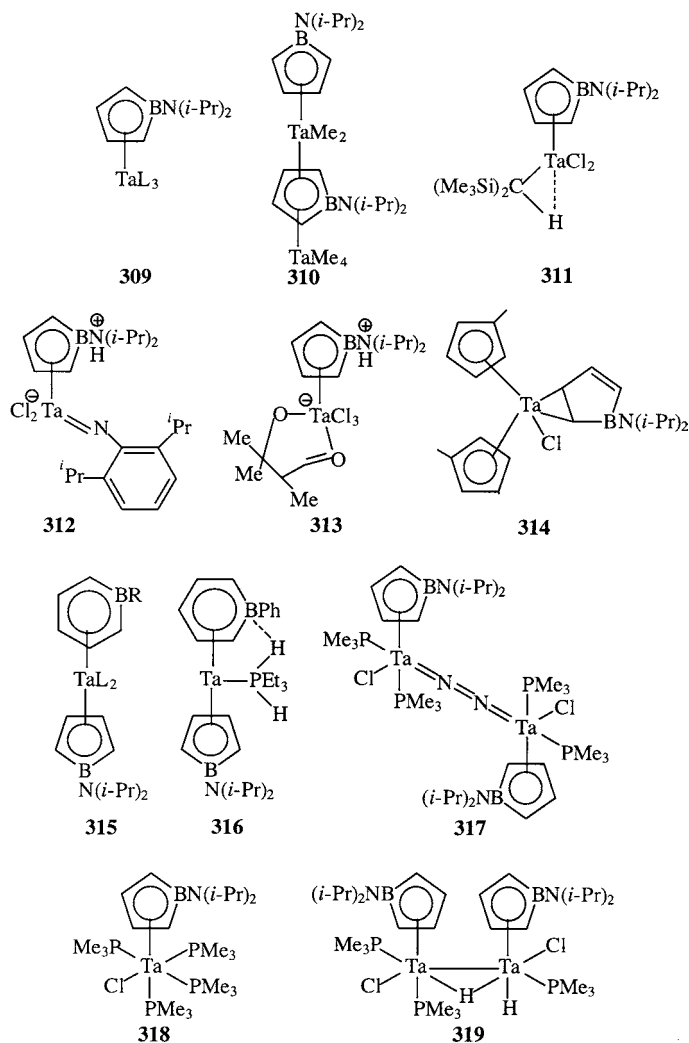
Among the vanadium group complexes, $(\mu\text{-}\eta^5\text{-C}_4\text{H}_4\text{BPh})[\text{V}(\text{CO})_4]_2$ is known [89AG(E)319]. The low-valent tantalum complexes are best described by the resonance structures **300** and **301** but not **299** (96JA10317; 97JA9305; 97OM163; 97OM2492). Thus, $\text{Li}_2[\text{C}_4\text{H}_4\text{BN}(i\text{-Pr})_2]$ and $\text{Cp}^*\text{TaMe}_2\text{Cl}(\text{OSO}_2\text{CF}_3)$ give **302**, the contribution of the resonance structure **303** being significant (95JA2671). 2,6-Dimethylphenyl isocyanide inserts into the Ta—Me bond of **302** to give **304**. Hydrogenolysis of **302** in the presence of trimethylphosphine produces **305**. With the protonation agent $[\text{PhNMe}_2\text{H}][\text{B}(\text{C}_6\text{F}_5)_4]$, **306** was obtained. Photolysis of **302** in methylene chloride or chloroform yields **307**. Reaction with $[\text{HNMe}_3]\text{Cl}$ affords **308**.

$\text{Li}_2[\text{C}_4\text{H}_4\text{BN}(i\text{-Pr})_2] \cdot \text{THF}$, AlCl_3 , and TaCl_5 give the η^5 -coordinated species **309** ($\text{L}_3 = \text{Cl}_3$) [85CB4303; 91CB25; 97JOM(548)1; 98JA7791]. Interaction of the latter with methylmagnesium chloride or methyllithium leads to a homobimetallic complex **310**, while that with $\text{LiCH}(\text{SiMe}_3)_2$, $\text{H}_2\text{N}(2,6\text{-}i\text{-Pr}_2\text{C}_6\text{H}_3)$, and acetone gives mononuclear products **311**, **312**, and **313**, respectively. Of interest is the reaction of **309** ($\text{L}_3 = \text{Cl}_3$) with lithium pentamethylcyclopentadienyl. An

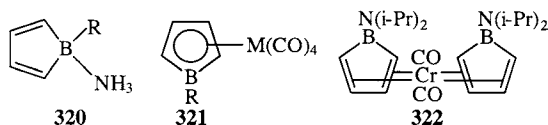


equimolar ratio of reactants gives **309** ($L_3 = \text{Cp}^*\text{Cl}_2$), and further carbonylation of the product leads to **309** [$L_3 = \text{Cp}^*(\text{CO})_2$], where the low-valent tantalum is nevertheless η^5 -coordinated. In excess LiCp^* , however, the η^2 -coordinated borole **314** was found. Further studies of reactivity of **309** ($L_3 = \text{Cl}_3$) were related to the lithium salts $\text{Li}(\text{C}_5\text{H}_5\text{BPh})$ and $\text{Li}(\text{C}_5\text{H}_5\text{BNMe}_2)$, the products being **315** ($R = \text{Ph}$, NMe_2 , $L = \text{Cl}$) (91IC2009; 92IC3558). They are methylated using methyllithium to give **315** ($R = \text{Ph}$, NMe_2 , $L = \text{Me}$). Species **315** ($R = \text{Ph}$, $L = \text{Me}$) on hydrogenation and subsequent treatment with trimethylphosphine gives **315** ($R = \text{Ph}$, $L = \text{PMe}_3$), and with triethylphosphine gives **316** [89JOM(362)243; 94OM619]. Finally, species **309** ($L_3 = \text{Cl}_3$) reacts with $\text{Li}_2[\text{C}_4\text{H}_4\text{BN}(i\text{-Pr})_2] \cdot \text{THF}$ and PMe_3 under nitrogen to give **317**, and under argon to give **318**. Complex **318** with molecular hydrogen yields a hydride-bridged species **319** (98JA7791). Catalytic aspects of the tantalum chemistry of borole were discussed (91JA1455; 92OM3098; 93OM2126; 94JA2177).

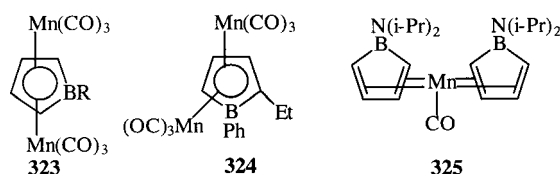
The most satisfactory route to the synthesis of the η^5 -borole complexes is the reaction of dihydroboroles (2-borolenes and 3-borolenes) with metal carbonyls. An alternative method of synthesis includes formation of the borole adducts with ammonia, **320** ($R = \text{Me}$, Ph) [87JOM(336)29]. Thermal reaction of **320** ($R = \text{Me}$, Ph) with $\text{M}(\text{CO})_6$ ($M = \text{Cr}$, Mo , W) gives **321** ($M = \text{Cr}$, $R = \text{Me}$, Ph ; $M = \text{Mo}$, W , $R = \text{Ph}$). There are data in favor of the π -electron delocalization over the borole



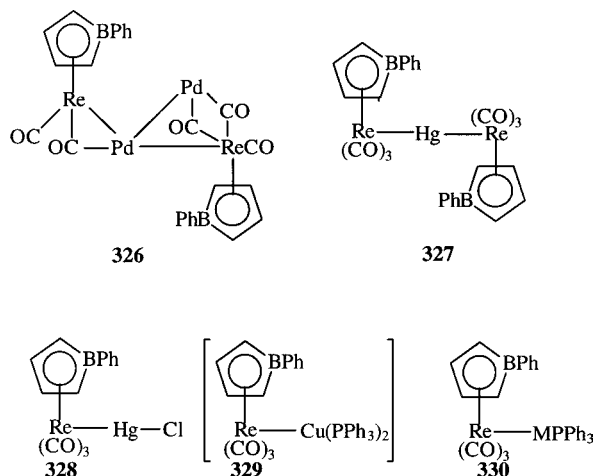
ring, $M \rightarrow L$ back-bonding, which is a peculiarity of boroles in comparison with siloles, and a consequence of participation of the borole LUMO in the bond. The exocyclic π -donor substituents at the boron atom influence electron delocalization in the conjugated heterorings via the p_z orbitals of the boron atom. Thus, the structure of 322 [88JOM(348)305] contains the *cis*- $\text{Cr}(\text{CO})_2$ framework. In contrast to 1-methyl- and 1-phenylboroles, the ligand is distorted. Aminoborole is coordinated as the dienic ligand and direct interaction between the chromium and boron atoms is very weak.



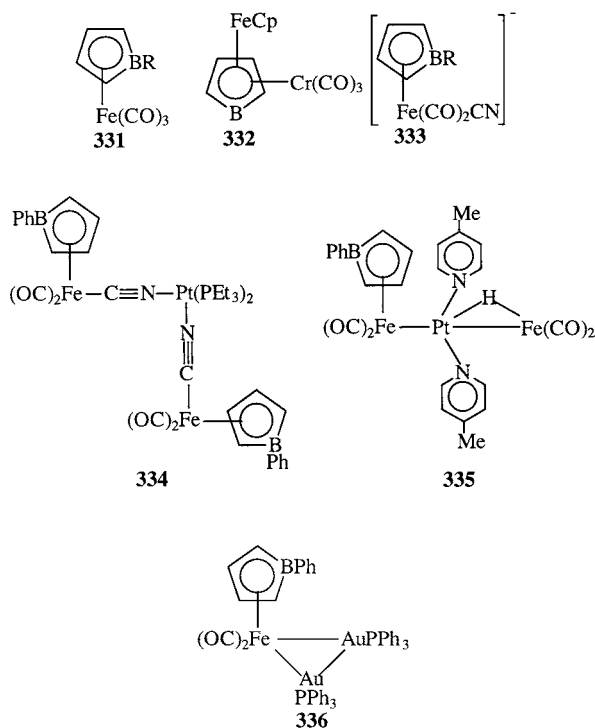
The reaction of 2-borolenes and 3-borolenes, $\text{C}_4\text{H}_6\text{BR}$ ($\text{R} = \text{Ph}, \text{Me}, \text{Cy}, \text{OMe}$) with $\text{Mn}_2(\text{CO})_{10}$ leads to dehydrogenation and formation of the simple C-substituted (η^5 -borole) metal species **323**, which represent the so-called triple-decker complexes [83AG(E)1024; 86JOM(308)153]. The general synthetic route involves the interaction of the derivatives of transition metals with 1-*R*-2,5-dihydroborole followed by their isomerization into 1-*R*-2,3-dihydroborole, dehydrogenation, and formation of the η^5 -metallocomplexes. A similar product **324** is known [76AG(E)433]. When the 1-substituent has π -donor properties, the η^4 -coordinated species, such as **325**, is formed [88JOM(348)305].



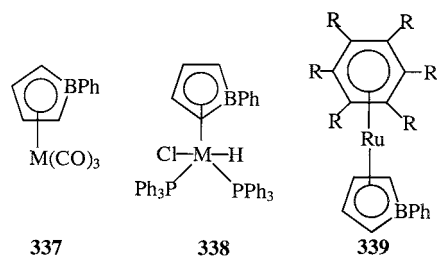
$[\text{NMe}_4][(\eta^5\text{-C}_4\text{H}_4\text{BPh})\text{Re}(\text{CO})_3]$ [95AG(E)1010] gives rise to the Re_2Pd_2 organometallic species **326**. When this rhenium complex enters interaction with HgCl_2 , it yields the ReHgRe system **327** [99JCS(D)2807]. Excess HgCl_2 gives **328**; **329** and **330** ($\text{M} = \text{Ag}, \text{Au}$) were also made and structurally proved.



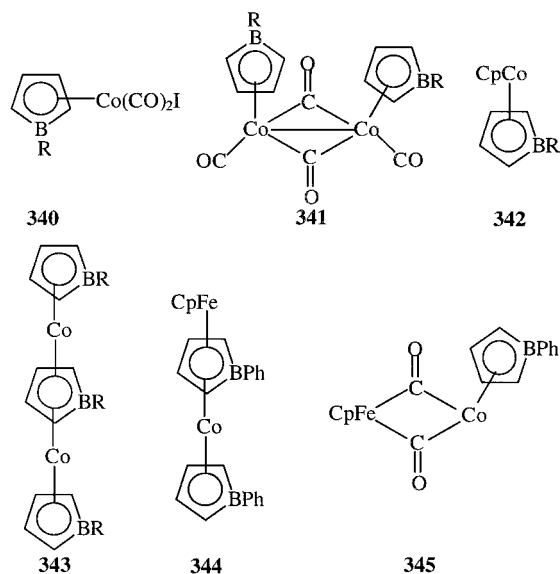
Analogous synthesis using $\text{Fe}(\text{CO})_5$ gives the half-sandwich complexes **331** ($\text{R} = \text{Me}, \text{Ph}$) [87JOM(319)311]. Sandwiches of the type **332** are mentioned [88JOM(343)1]. Pentaphenylborole reacts with $\text{Fe}_2(\text{CO})_9$, yielding $[(\text{OC})_3(\eta^5\text{-pentaphenylborole})\text{iron}]^-$ [77AG(E)42]. Another synthetic route is based on 1-phenyl-4,5-dihydroborepine, which is subject to ring contraction in the process of complex formation. The borole ligands are bonded to a metal as the pentahapto four-electron ligands. Species **331** ($\text{R} = \text{Ph}$) with lithium(trimethylsilyl)amide gives **333** (92CB1801), which with *cis*- $[\text{PtCl}_2(\text{PEt}_3)_2]$ gives the heterotrinnuclear complex **334** with cyano bridges [99JOM(580)66]. $(\text{NBu}_4)[(\eta^5\text{-C}_4\text{H}_4\text{BPh})\text{Fe}(\text{CO})_2\text{H}]$ was a precursor for the other heteronuclear complexes. Thus, with *trans*- $\text{PdBr}_2(4\text{-Mepy})_2$ it leads to **335** [99JCS(D)2807]. FeAu_2 species **336** is also known (98OM2177).



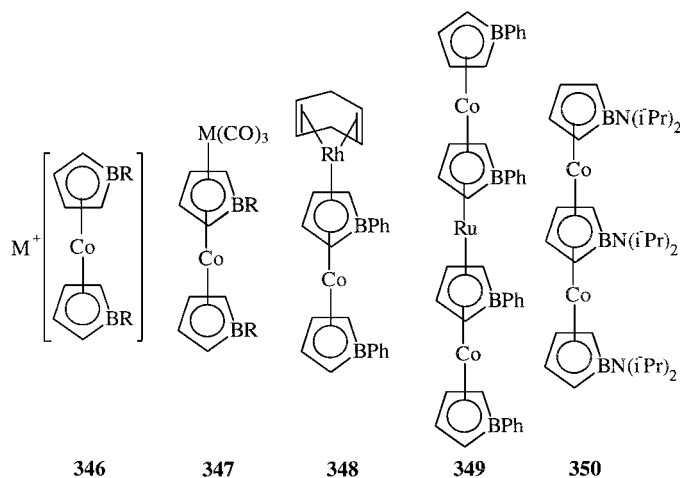
Interaction of borolenes with $\text{Ru}_3(\text{CO})_{12}$, $\text{Os}_3(\text{CO})_{12}$, $\text{RuCl}_2(\text{PPh}_3)_2$, $\text{RuHCl}(\text{PPh}_3)_3$, $\text{OsCl}_2(\text{PPh}_3)_3$, $[\text{Ru}(\eta^6\text{-C}_6\text{H}_6)(\eta^4\text{-C}_6\text{H}_8)]$, and $[\text{Ru}(\eta^6\text{-C}_6\text{Me}_6)\text{Cl}_2]_2$ leads to formation of the η^5 -borole metallic complexes of ruthenium and osmium **337** ($\text{M} = \text{Ru}, \text{Os}$), **338** ($\text{M} = \text{Ru}, \text{Os}$), and **339** ($\text{R} = \text{H}, \text{Me}$) [83AG(E)1024, 87JOM(319)9].



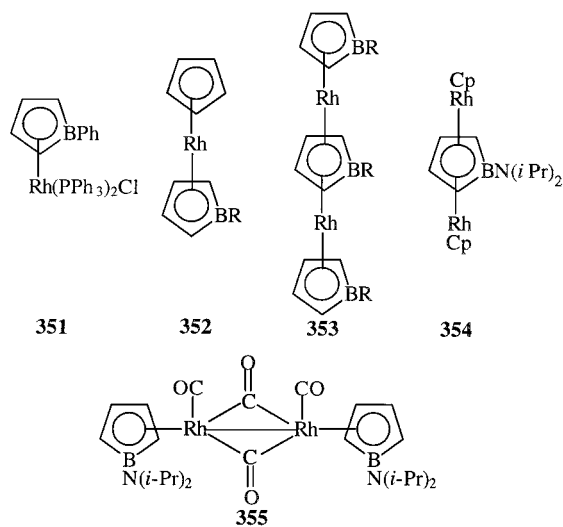
The complex **340** ($R = \text{Me}, \text{Ph}$) was described [87JOM(336)29]. If excess ammonia is introduced to the THF solution of **340**, then **341** ($R = \text{Me}, \text{Ph}$) is formed. The latter species serve as the starting material for the sandwiches **342** ($R = \text{Me}, \text{Ph}$), triple-deckers **343** ($R = \text{Me}, \text{Ph}$) and **344**, binuclear complexes **345**, and salts **346** ($M = \text{Na}, R = \text{Me}, \text{Ph}; M = \text{NMe}_4, R = \text{Me}, \text{Ph}; M = \text{Cs}, R = \text{Ph}$) [87JOM(319)9]. Anions of the salts easily form the triple-deckers **347** ($M = \text{Mn}, R = \text{Me}, \text{Ph}; M = \text{Cr}, R = \text{Ph}$) and **348**, or tetradecker complexes **349**. The complexes **347** ($M = \text{Mn}, R = \text{Me}, \text{Ph}; M = \text{Cr}, R = \text{Ph}$) undergo regiospecific H/D exchange in the α position relative to the boron atom and acetylation with $\text{MeCOCl}/\text{SnCl}_4$ followed by formation of the 2-acetyl derivative. The triple-decker **350** with an amino substituent at the boron atom is also known [87JOM(324)57].



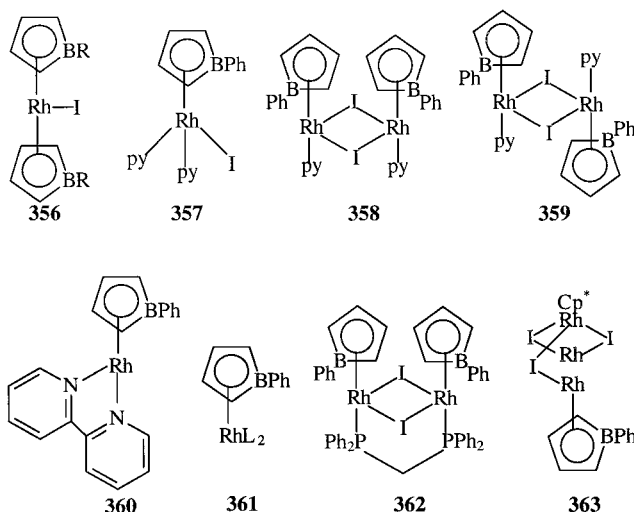
Complex formation with the Wilkinson catalyst $\text{RhCl}(\text{PPh}_3)_3$ gives **351** [87OM(319)311]. The reaction with $[\text{Rh}(\eta^2\text{-C}_2\text{H}_4)_2\text{Cl}]_2$ gives the triple-decker



complexes **353** (R = Me, Ph) [83AG(E)996; 86JOM(312)13]. The interaction of the latter with sodium cyclopentadienyl gives the neutral species **352** (R = Me, Ph), undergoing fast exchange at the α position relative to the boron atom. The synthetic method used for the η^5 complexes of boroles [83AG(E)1024] is applied for the boroles containing amino substituents at the boron atom. Thus, the complexes **354** and **355** are known [87JOM(324)57].

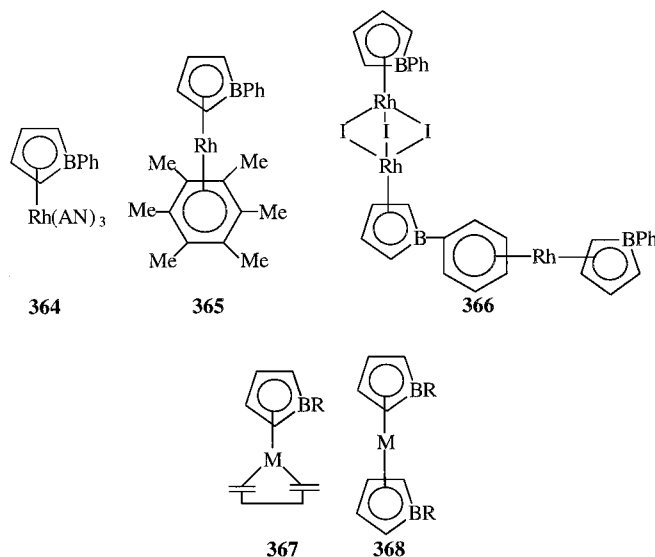


Triple-deckers **352** with I_2 give $[Rh(\mu_3-I)(C_4H_4BR)]_4$ ($R = Me, Ph$) and **356** (97OM4292). Species $[Rh(\mu_3-I)(C_4H_4BPh)]_4$ with pyridine gives **357**, **358** (*cis* isomer), and **359** (*trans* isomer). The product **357** enters ligand substitution with 2,2'-bipyridine to yield **360**. The other adduct formation of $[Rh(\mu_3-I)(C_4H_4BPh)]_4$ studied was with acetonitrile to afford **361** ($L = AN$) and with carbon monoxide to form **361** ($L = CO$). The latter, upon removal of the solvent, gives *cis* and *trans* isomers similar to **358** and **359**, respectively, with CO instead of py. Further adduct-forming agents were triphenylphosphine, giving **361** ($L = PPh_3$), bis(diphenylphosphino)methane (the product is **362**), nbd yielding **361** ($L_2 = nbd$), and $[RhI_2Cp_2^*]$ leading to **363**.



The tetrameric species $[Rh(\mu_3-I)(C_4H_4BPh)]_4$ (97OM4292, 97OM4800) on dissolution in acetonitrile gives $[RhI(AN)_2(C_4H_4BPh)]$ (98OM519). Silver tetrafluoroborate causes abstraction of the iodide ions from the latter to yield the salt **364**. With hexamethylbenzene or mesitylene, ligand displacement occurs, producing, for example, **365**. The starting $Rh_4(\mu_3-I)_4(C_4H_4BMe)_{4-x}(C_4H_4BPh)_x$ ($x = 0-4$) on standing gives the trinuclear species **366**.

Pentaphenylborole with $Ni(CO)_4$ yields $(OC)_2(\eta^5\text{-pentaphenylborole})$ nickel [77AG(E)42]. The ammonia adducts of 1*H*-boroles react with $M(cod)_2$ ($M = Ni, Pd, Pt$) followed by formation of the mixed-ligand complexes **367** ($M = Ni, R = Me, Ph$; $M = Pd, Pt, R = Ph$) [88JOM(350)51]. Thermolysis of the latter gave the bis(ligand) complexes **368** ($M = Ni, R = Me, Ph$; $M = Pd, Pt, R = Ph$). A platinum π complex of pentaphenylborole similar to **367** has been reported [87JOM(324)283].



VII. Conclusion

1. Organometallic chemistry of pyrrole is characterized by a delicate balance of the $\eta^1(\text{N})$ - and η^5 -coordination modes. Azacymantrene is an illustration of the considerable nucleophilicity of the heteroatom. However, azaferrocene can be alkylated at C2 and C3 sites. Ruthenium and osmium, rhodium, and iridium chemistry revealed the bridging function of pyrroles, including zwitterionic and pyrrolyne complex formation. The $\eta^2(\text{CC})$ coordination of osmium(2+) allows versatile derivatizations of the heteroring.
2. The η^6 coordination via the carbocycle prevails for indole and carbazole, although the η^5 species were also found in organomanganese and -iridium chemistry. Osmium carbonyls tend to produce the species with the bridging indole function. Some illustrations of the $\eta^1(\text{N})$ coordination exist.
3. Phospholes and analogs offer a wide variety of coordination modes and reactivity patterns, from the $\eta^1(\text{E})$ ($\text{E} = \text{P}, \text{As}, \text{Sb}, \text{Bi}$) through η^4 -dienic to η^5 -donor function, including numerous and different mixed coordination modes. Electrophilic substitution at the carbon atoms and nucleophilic properties of the phosphorus atom are well documented. In the η^5 -coordinated species, group V heteroles nearly acquire planarity and features of the π -delocalized moieties (heterocymantrenes and -ferrocenes).
4. Organometallic chemistry of silole and germole, recently dominated by the η^4 species, tends to enhancement of the range of the η^5 representatives. Cases of the $\eta^1(\text{Si}, \text{Ge})$ coordination in the pure and mixed situations are known as well.

5. Boroles are the η^5 -complex-forming ligands. However, in the presence of the π -donor substituents at the heteroatom, they tend to give η^4 or η^2 species, especially for the low-valent early transition metals.

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ADVANCES IN HETEROCYCLIC CHEMISTRY, VOL. 79

The Literature of Heterocyclic Chemistry, Part VII: 1997–1999

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I. Introduction

This survey is a sequel to the six other surveys already published in *Advances in Heterocyclic Chemistry* [66AHC(7)225; 79AHC(25)303; 88AHC(44)269; 92AHC(55)31; 98AHC(71)291; 99AHC(73)295]. It includes monographs and reviews published during the period 1997–1999 as well as some work published earlier but omitted in Part VI.

Like Parts III–VI, this survey is based mainly on short bibliographic papers published by the authors in *Khimiya Geterotsiklicheskikh Soedinenii* since 1998 (98KGS1277; 99KGS707; 99KGS844; 99KGS1264; 00KGS121; 00KGS997). Sources not only in English but also in Russian, Japanese, Chinese, Czech, and other languages are surveyed and classified. This feature of the survey should cause no problem because some of the sources are available in English translations, and practically all the others have informative English abstracts as well as quite understandable and useful schemes and lists of references. As before, carbohydrates are not covered. Such compounds are mentioned only in general cases (e.g., anomeric

effect) as well as when they serve as starting compounds for the synthesis of other heterocycles or are present as fragments of a complex system including another heterocyclic moiety such as nucleosides.

II. General Sources and Topics

A. GENERAL BOOKS AND REVIEWS

1. Textbooks and Handbooks

A general handbook: 98MI67; 99MI44.

Textbook on organic chemistry with three chapters devoted mainly to natural and synthetic physiologically active heterocycles: 98MI14.

2. Annual Reports

a. *Comprehensive Reports*. 97PHC1; 98PHC1; 99PHC1.

b. *Specialized Reports Devoted to Basic Series of Heterocycles*.

Three-membered heterocycles: 97PHC43, 98PHC49, 99PHC66.

Four-membered heterocycles: 97PHC64, 98PHC70, 99PHC87.

Pyrrole and its benzo derivatives: 97PHC97, 98PHC109, 99PHC124.

Furan and its benzo derivatives: 97PHC117, 98PHC129, 99PHC144.

Thiophenes, selenophenes, and tellurophenes: 97PHC77, 98PHC87, 99PHC102.

Five-membered heterocycles with more than one N atom: 97PHC148, 98PHC153, 99PHC163.

Five-membered heterocycles with N and S (Se) atoms: 97PHC190, 98PHC172, 99PHC184.

Five-membered heterocycles with O and S (Se, Te) atoms: 97PHC192, 98PHC195, 99PHC200.

Five-membered heterocycles with O and N atoms: 97PHC207, 98PHC209, 99PHC213.

Pyridine and its benzo derivatives: 97PHC222, 98PHC226, 99PHC230.

Diazines and their benzo derivatives: 97PHC249, 98PHC251, 99PHC256.

Triazines, tetrazines, and fused polyaza systems: 97PHC268, 98PHC275, 99PHC276.

Six-membered heterocycles with O and/or S atoms: 97PHC312, 98PHC292, 99PHC299.

Seven-membered heterocycles: 97PHC318, 98PHC320, 99PHC319.

Heterocycles with eight-member and large rings: 97PHC334, 98PHC335, 99PHC338.

c. *Reports Devoted to Individual Problems.*

Aromatic carbon–heteroatom coupling reactions with participation and formation of heterocycles: 98JCS(P1)2615.

Asymmetric reactions with formation and participation of heterocycles: 98JCS(P1)1151, 98JCS(P1)1439, 98JCS(P1)3101, 99JCS(P1)1109.

Carboxylic acids and esters of heterocyclic series including lactones: 98JCS(P1)2451, 99JCS(P1)3537.

Cycloaddition reactions of nitro compounds, nitrones, and nitrile oxides: 99JCS(P1)749.

Enantioselective desymmetrization of achiral or *meso* compounds with formation of enantiomerically enriched products, among them heterocycles: 99JCS(P1)1765.

Formation and transformations of cyclic amines and lactams: 98JCS(P1)2959, 99JCS(P1)2209.

Formation of heterocycles in reactions with participation of stoichiometric transition metal complexes: 98JCS(P1)819, 99JCS(P1)223.

Intramolecular Pd(0)-catalyzed Stille reaction of organotin reagents with electrophiles leading to C–C σ -bond formation in synthesis of heterocycles, particularly, macrocyclic lactones: 99JCS(P1)1235.

Main group organometallics in synthesis and transformations of heterocycles: 98JCS(P1)1343.

Nitro- and nitroso-substituted heterocycles and related compounds: 99JCS(P1)749.

Protecting groups in syntheses of heterocycles: 98JCS(P1)4005, 99JCS(P1)1589.

Saturated and unsaturated lactones: 98JCS(P1)1869, 99JCS(P1)1377.

Saturated nitrogen heterocycles: 98JCS(P1)3493, 99JCS(P1)2553.

Solid-phase synthesis and transformations of heterocycles: 97T5643, 98JCS(P1)3293, 98T15385.

Strategies for stereoselective synthesis of molecules with remote stereogenic centers across a double bond of fixed configuration: in particular, for synthesis of heterocycles, especially unsaturated macrocyclic lactones: 99JCS(P1)1899.

Synthesis of aromatic heterocycles: 98JCS(P1)615, 99JCS(P1)2849.

Synthesis of heterocyclic analogs of quinones: 99JCS(P1)2409.

Synthesis of heterocyclic halides: 98JCS(P1)1577, 99JCS(P1)737.

Synthesis of thiols, selenols, sulfides, selenides, sulfoxides, sulfones, and selenones of heterocyclic series: 98JCS(P1)1973, 99JCS(P1)641.

Synthetic modifications and applications of silacycles: 98JCS(P1)2209, 99JCS(P1)81.

Transformations of heterocycles in stoichiometric asymmetric processes: 99JCS(P1)357.

Transition metals in synthesis and transformations of heterocycles: 98JCS(P1)3637, 99JCS(P1)2645.

3. *Nomenclature*

Extension and revision of the von Baeyer system for naming polycyclic compounds (including bicyclic compounds), IUPAC recommendations for naming heterocycles: 99PAC513.

IUPAC recommendations for nomenclature of spiro compounds including spiro heterocycles: 99PAC531.

IUPAC recommendations for nomenclature of natural products and related compounds: 99PAC587.

Nomenclature of organometallic compounds of the transition elements (IUPAC recommendations): 99PAC1557.

4. *History of Heterocyclic Chemistry, Biographies*

Contribution of Prof. D. A. Evans to stereoselective synthesis of heterocycles with C—C bond formation (self-review): 99T8589.

Contribution of Prof. K. Nakanishi to heterocyclic chemistry: 98H(47)47.

Contribution of Prof. V. Prelog to the synthesis of drugs and study of antibiotics: 96CLY451.

Investigations of Prof. N. J. Leonard on N-heterocycles: 97T2325.

Investigations of Prof. A. Padwa in the field of photochemistry and heterocyclic chemistry (self-review): 99JHC1349.

Obituary of Professor Sir D. H. P. Barton (September 8, 1918 to March 16, 1988): 98T8847.

Reminiscences on Arthur Luttringhaus (1906–1992): 98EJO735.

Reminiscences on Rafael' Matevosyan (1926–1985): 99MI45.

Scientific biography of Karl Dimroth (1910–1995): 97LA(4)xxiii.

Self-review by Prof. S. Danishevsky concerning, in particular, the synthesis of natural heterocycles: 97T8689.

Studies of Prof. T. Mukayama in the field of complex natural heterocyclic compounds (self-review): 99T8609.

Studies of Prof. S. Tobinaga in the field of O-heterocycles derived from plants (self-review): 99YZ185.

5. *Bibliography of Monographs and Reviews*

a. *Specialized Surveys.* 98KGS1277; 99KGS707; 99KGS844; 99KGS1264; 00KGS121.

B. GENERAL TOPICS BY REACTION TYPE

Application of phase-transfer catalysis in heterocyclic chemistry: 99MI33.
Chalcogen heterocyclic chemistry: 98AHC(71)115.
Formation and transformation of heterocycles on solid surfaces: 95MI9.
Syntheses and transformations of heterocycles using zeolites and zeolite-type materials: 99T12657.

1. *Structure and Stereochemistry*a. *Theoretical Aspects.*

Asymmetric reactions using nonnatural chiral auxiliaries with participation and formation of heterocycles: 98YGK386.
Five- and six-member heteroaromatic compounds as σ and π ligands: 99AHC(72)1.
Heterocycles as chemosensors in molecular wire approach to sensory signal amplification: 98ACR201.
Heterocyclic bridging ligands in controlling electronic and magnetic properties in polynuclear complexes: 98ACR842.
Macroheterocycles as components of systems capable of molecular recognition in lamellar solids and thin films: 98ACR209.
Molecular recognition and the quest for new catalysts in combinatorial syntheses with participation and formation of heterocycles: 97LA637.
Ring-chain tautomerism of hydrazones of 1,3-dicarbonyl compounds: 99MI21.
Ring-chain tautomerism via intramolecular reversible addition reactions to the C=O group: 95AHC(64)251.
Ring-chain tautomerism via intramolecular reversible addition reactions to the C=N, C=N, C=C, and C $\equiv$ C groups: 96AHC(66)1.
Self-regeneration of stereocenters (SRS); particularly, the scope and limitations of the SRS synthetic principle in heterocycles: 97AG(E)2708.
Syntheses and transformations of various heterocycles in new asymmetric reactions utilizing carbanions: 99YZ114.
Use of Lewis acids in free-radical reactions of heterocycles and heterocyclizations: 98AG(E)2562.

b. *Molecular Dimensions.*

Classification and analysis of crystallographic and structural data for Sn-complexes with heterocyclic ligands: 98MI65.
Molecular structures of chalcogenadiazaheterocycles with $-N=Z=N-$ fragments ($Z = S, Se, Te$): 97ZSK988.
Structure of tropones, tropolones, and tropylium salts with fused heterocyclic rings: 95AHC(64)81.

c. Stereochemical Aspects.

Anomeric effect of monosaccharides and their derivatives; insights from the new QVBMM (quantized valence bond molecular mechanisms) molecular mechanics force field: 98H(48)2389.

Catalytic asymmetric synthesis with participation and formation of heterocycles (including asymmetric phase transfer reactions and asymmetric reactions with chiral Lewis catalysts): 93MI1.

Chiral heterocycles as ligands in asymmetric catalysis: 99JHC1437.

Derivatives of pyridine, azoles and macroheterocycles with C_3 symmetry in asymmetric catalysis and chiral recognition: 98AG(E)248.

Heterocycles as ligands in asymmetric catalytic metal carbene transformations: 98CRV911.

Heterocycles as ligands in complex catalysts for asymmetric cyclopropanation: 98T7919.

Nonlinear dependence between *ee* values of chiral auxiliary compound or ligand and product of reaction with participation or formation of heterocycle: 98AG(E)2922.

Stereochemistry of complexes with heterocyclic ligands: 95MI1.

Stereoelectronic interaction between heteroatoms in 2-hetero-substituted 1-azaadamantanes: 99PAC385.

Stereoselective asymmetric synthesis with participation of diazocarbonyl intermediates in the presence of catalysts possessing chiral heterocyclic ligands: 97CC983.

Topologically nonplanar centrohexasyclic heterocycles: 97LA1043.

d. Betaines and Other Unusual Structures.

Azafullerenes: 99ACR795.

Catenanes and rotaxanes including macroheterocyclic fragments: 99T5265.

Construction of one-dimensional multicomponent molecular arrays, transition metal complexes with terpyridines and/or porphyrins as ligands: 98EJI1.

Dendrimers including heterocyclic fragments: 97CRV1681, 98CSR233, 98T8543.

Electrochemistry of supramolecular systems with heterocyclic fragments as host molecules (porphyrinoids, complexes on the basis of 2,2'-bipyridine and 2,2',2''-terpyridine, hetero- and heteracyclophanes): 98AG(E)216.

Fullerenes fused with heterocycles: 99UK979.

N-Heterocyclic carbenes: 97AG(E)2162.

Interpenetrating nets with heterocyclic fragments: 98AG(E)1460.

Large, rigid molecules with functional heterocyclic units (effectors) located on a carbocyclic framework and taking part in 1,3-dipolar cycloaddition and heterodiene reactions: 98SL566.

Molecular rods including heterocyclic fragments, in particular pyridine and porphyrine residues: 99CRV1863.

Preparation of heterocyclic betaines using hypervalent Te-, I-, and Xe-reagents: 99JHC1573.

Structure and reactivity of cycloimmonium ylides: 99H(51)863.

Supramolecular chemistry of fullerenes linked or fused with heterocyclic fragments: 99CSR263.

Supramolecular systems including heterocycles: 98MI31.

e. *Miscellaneous Substituted Heterocycles.*

Chemistry of fully and partly saturated five- and six-member O- and N-heterocycles: 97MI44.

Generation and reactions of nonstabilized α -aminocarbanions, derivatives of saturated N-heterocycles: 98T2647.

Hetaryl thiocyanates: 99T7957.

Heterocyclic *ortho*-quinodimethanes: 98PHC25.

Luminescent polynuclear d^{10} -metal complexes with heterocyclic ligands: 99CSR323.

Mono- and polymetallic lanthanide-containing functional assemblies with heterocyclic ligands: 99CSR347.

Pentamethinic systems with heterocyclic substituents: 97CLY387.

Racemic and optically active α -aminophosphonic acids, derivatives of various heterocycles: 97ZOR1605.

Reactions and applications of organocobaloximes in synthesis and transformations of heterocycles: 98Y GK544.

Spiro compounds containing adamantane and heterocyclic fragments: 97KGS435.

Synthesis, reactions, and metal-chelating properties of functionalized heterocycles possessing N-hydroxyamide moiety: 97Y GK524.

Synthesis, structure, and properties of aromatic biheterocycles: 97AHC(67)1.

Synthesis, structure, transformations, and applications of sulfoxylic acid (HOSO₂H) derivatives containing heterocyclic fragments: 98ZOR1433.

Technetium-chelates with heterocyclic ligands: 98CSR43.

Transition metal complexes containing allenylidene, cumulenylidene, and related ligands with heterocyclic fragments: 98CRV2797.

2. Reactivity

a. *General Topics.*

Allylic protection groups and their removal through catalytic palladium π -allyl methodology in transformations of heterocycles: 98T2967.

[3+3]Benzannulation of heteroaromatics consisting of successive nucleophilic

- attack; cation, anion or radical cyclization; and aromatization with elimination and/or oxidation: 99T8263.
- Carbene transfer reactions with participation of organometallic compounds bearing heterocyclic substituents: 99CSR315.
- Chiral selenium compounds in transformations of heterocycles: 99T1.
- Combinatorial syntheses with participation of heterocycles: 99AG(E)2494.
- Competitive coordination of ambident heterocyclic ligands in metal complexes: 97UK434.
- Controlled racemization of optically active heterocycles: 97T9417.
- Control of chemical activity and selectivity by catalytic antibodies in reactions of heterocycles: 95MI11.
- Desulfonation reaction (reductive and oxidative desulfonation, nucleophilic substitution, elimination, and SO₂ extrusion) in synthesis and transformations of heterocycles: 99T10547.
- Direct carbonylation of heterocycles at C—H bond catalyzed by transition metal complexes: 98YGK443.
- Electrochemistry of fullerene derivatives with heterocyclic fragments: 98ACR593.
- Enantioselective oxidation of cyclic dithioacetals to monosulfoxides catalyzed by bacterial cyclohexanone monooxygenases: 96CC2303.
- Enzymatic reactions of heterocycles: 97MI28, 97MI29.
- Enzyme-controlled transformations of fluorinated heterocycles: 97MI32.
- High pressure in enzyme-catalyzed organic reactions: 98H(48)1023.
- Introduction of fluorinated alkyls into heterocycles: 97MI33.
- Medium effects on charge transfer in metal complexes with heterocyclic ligands: 98CRV1439.
- Metal-assisted cycloaddition with participation of heterocycles: 97CRV523.
- Microwave activation of reactions of heterocycles: 98S1213, 99T10851.
- New *gem*- and *vic*-disubstituent effects on cyclization with participation of heterocycles: 99SL843.
- Preparative biotransformations of heterocycles: 99JCS(P1)1.
- Pummerer reaction in transformations of heterocycles: 98MI62.
- Reactions of fullerenes with heterocycles: 99UK979.
- Reactions of glutaconic acid derivatives with heterocycles: 98OPP657.
- Reactions of heterocycles catalyzed by antibodies: 98UK1099.
- Reactions of heterocycles under high pressure: 97LA623, 97T2669, 98H(47)1135.
- Reactions of some heterocyclic carbanions and N-anions with CCl₄ and CBr₄: 99OPP359.
- Reactivity of tropones, tropolones, and tropylium salts with fused heterocyclic rings: 96AHC(66)285.
- Reactivity patterns of radical cations in transformations of heterocycles: 97AG(E)2550.

Recyclization reactions of heterocycles with participation of malononitrile and its derivatives: 99UK45.

Regioselectivity in reactions of heterocyclic anions of allyl-type with electrophiles: 99CRV665.

Skeleton transformations of heterocage compounds involving heterofunction: 97UK1015.

Solid-phase reactions of heterocycles: 98MI38.

Solid-state photoreactions of heterocycles in two-component crystals: 98S1.

Stereoselectivity of chiral homoenolate equivalents in reactions of heterocycles: 99S365.

Transformations of heterocycles on solid-phase catalysts: 97T5643.

Transformations of heterocycles with participation of functionalized polymers including heterocyclic fragments: 97S1217.

Use of bis(trimethylsilyl)acetamide and bis(trimethylsilyl)urea for protection and as control reagents in reactions with participation of heterocycles: 98S357.

b. *Reactions with Electrophiles and Oxidants.*

Alkynylcarbenium, alkynylpyridinium, and alkynylphosphonium ions in transformations of heterocycles: 98UK899.

Arylboron compounds as acid catalysts in transformations of heterocycles: 99EJO527.

Chemical transformations of heterocycles induced by hypervalent iodine reagents: 97T1179.

Electrochemical fluorination of heterocycles: 97MI34.

Electrophilic fluorination of heteroaromatic compounds: 99T12431.

Fluorination of heteroaromatic compounds and introduction of fluorine-containing substituents: 96MI6.

Fluorination of saturated and aromatic heterocycles with elemental fluorine: 97MI30.

Heterocycles in metal-catalyzed cross-coupling reactions: 98MI32.

α -Metallocenylalkylation of heterocycles: 97UK677.

Organocerium compounds in transformations of heterocycles: 99T3803.

Organoiron compounds in reactions of heterocycles: 99S727.

Prereactive dihalogen complexes with O- and S-heterocycles as Lewis bases in the gas phase: 99AG(E)2686.

Reactions of aliphatic nitro alcohols with heterocycles: 98UK39.

Reactions of heterocycles in aqueous media with participation of indium compounds as reagents or catalysts: 99T11149.

Reactions of metallated hetarenes with carbonyl compounds and their imino derivatives: 99PHC21.

Reactions of SO_2Cl_2 with heterocycles: 97ZOR327.

Reactions of sulfonic acid *N*-sodium *N*-chloroamides with heterocycles in organic synthesis, in particular in synthesis of natural compounds: 99ZOR503.

Reactivity and selectivity of organometallic reagents addition to C=N bond with participation and formation of heterocycles: 98CRV1407.

Recent advances in Cr(II)- and Cr(III)-mediated reactions of heterocycles: 99S1.

Selective oxidation of heterocycles catalyzed by peroxidases: 97T13183.

Ti(III)-mediated reactions of heterocycles: 99S2001.

Transformations of heterocycles with C—C bond formation, involving organochromium(III) reagents: 99CRV991.

Transformations of heterocycles with participation of organozinc compounds: 98T8275.

Transition metal-catalyzed cyclizations of heterocycles accompanied by multiple bonds migration: 98SL1.

Transition metal-mediated transformations of heterocycles: 98MI26, 98MI27.

Use of Sc(OTf)₃ as Lewis acid in reactions of heterocycles: 99EJO15.

c. Reactions with Nucleophiles and Reducing Agents.

Chain reactions of heterocycles with participation of silicon and germanium hydrides: 98AG(E)3072.

Cobalt-catalyzed C—C and C-heteroatom bond formation in transformations of heterocycles: 97SL876.

Heterocycles in reactions with organozinc reagents: 96MI8.

Hydroboration of heterocycles catalyzed by transition metal complexes: 97T4957.

Metallation of heterocyclic compounds: 90MI1.

Metal-catalyzed hydrometallation of heterocycles: 98PAC1059.

Nucleophilic aromatic substitution of hydrogen in heteroaromatic compounds, reactivity and reaction mechanisms: 94MI2.

Organometallics in coupling reactions of π -deficient azaheterocycles: 95AHC(62)305.

Palladium(0)-catalyzed allylation of ambident nucleophilic aromatic heterocycles: 96AHC(66)73.

Reactions of heterocycles with α -aminoalkylphosphonates: 98UK940.

Reactions of heterocycles with fluorinated organometallic compounds: 97MI31.

Reduction and desulfurization of hetarenes with participation of nickel boride: 97OPP1.

Selective transition metal-promoted carbon—carbon bond formation in polyhalohetarenes: 97OPP137.

Vicarious nucleophilic substitution with participation of heterocycles: 97LA1805.

d. Reactions with Intermediacy of Free Radicals, Carbenes, etc.

Catalytic asymmetric cyclopropanation of heterocycles: 98T7919.

e. *Reactions with Cyclic Transition State.*

Conjugated nitroso alkenes in reactions with heterocycles: 98UK523.

Intramolecular cycloaddition reactions of allylic cations with participation and/or formation of heterocycles, mainly [4+3]-cycloaddition to furan system: 97T6235.

Metal-assisted cycloaddition in formation of heterocycles: 97CRV523.

Stereoselective intermolecular [2+2]-photocycloaddition reactions of unsaturated heterocycles with formation of fused systems: 98S683.

Tandem reactions of heterocycles combining Diels–Alder reactions with sigmatropic rearrangements: 98S227.

f. *Reactivity of Substituents.*

Design and synthesis of conformationally fixed amino acids of heterocyclic series as versatile building blocks: 97T12789.

Nonclassical Wittig reaction, olefination by phosphoranes interaction with carboxylic acid derivatives, in transformation of heterocycles: 99JCS(P1)3049.

g. *Heterocycles as Intermediates in Organic Synthesis.*

Asymmetric catalysis using chiral ligands, including cyclic phosphine or pyrazole fragments covalent-bonded with ferrocene system: 98PAC1477.

Asymmetric Sharpless dihydroxylation of olefins using catalysts supported by polymers with heterocyclic fragments: 98EJO21.

Atroposelective cleavage of configurationally unstable lactone cycle in biaryl derivatives as effective route to chiral natural products and useful reagents: 99S525.

Chiral bicyclic lactams as useful precursors and templates for asymmetric syntheses: 97CC1.

Chiral cyclic *N,O*-acetals of glyoxal and their use for preparation of α -amino acids: 98SL449.

Heterocycles as reagents and substrates in modern modifications of the Mannich reaction: 98AG(E)1045.

N-Fluoropyridinium salts and their analogs as fluorinating agents: 96MI6.

N-Fluoro-substituted *N*-heterocycles as fluorinating reagents: 98MI40, 99T12431, 99UK725.

Heterocycles in syntheses of natural products with C–P bond: 99T12237.

Heterocycles in synthesis of supramolecular structures: 92MI1.

Levogluconone as an optically active starting material: 97YGK1074.

Making and breaking the dioxygen O–O bond with participation of synthetic copper complexes with heterocyclic ligands: 97ACR227.

Mechanism and selectivity of free-radical halogenation with cyclic *N*-haloimides: 99MI19.

Pd-catalysts with heterocyclic ligands as catalysts for Heck reaction: 98CSR427.

Reactions of acetoacetic ester with aryl- and hetarylamines: 97KGS579.
Recent advances in applications of chiral amino alcohols including heterocyclic fragments and chiral oxazaborolidines in asymmetric catalysis: 98MI63.
Ring opening of heterocycles in syntheses of pentamethinic systems: 97CLY387.
Syntheses, structures, and reactions of heterocycles capable of serving as halogenating reagents: 99MI42.
Vinylogous aldol addition of heterocyclic silyloxydienes in organic synthesis: 99SL1333.

3. Synthesis

a. *General Topics, Nonconventional Synthetic Methodologies.*

Absolute asymmetric syntheses of heterocycles under physical fields: 98CRV2391.
Arylboron compounds as acid catalysts in syntheses of heterocycles: 99EJO527.
Asymmetric synthesis of heterocycles: 99YGK581.
Ru-catalyzed syntheses of heterocycles: 98CRV2599.
Chain reactions with participation of silicon and germanium hydrides in syntheses of heterocycles: 98AG(E)3072.
Cobalt-catalyzed C—C and C-heteroatom bond formation in synthesis of heterocycles: 97SL876.
Combinatorial synthesis of heterocycles: 96MI7, 97CRV449, 99AG(E)2494, 99CSR1.
Coupling of organic halides with carbonyl compounds promoted by SmI_2 in synthesis of heterocycles: 99CRV745.
Disposable formation of heterocycles in complex organic syntheses: 98T2289.
Enzymatic reactions in synthesis of heterocycles: 97MI28, 97MI29.
Formation of N-, O-, and S-heterocycles in reactions with participation of hypervalent iodine (25 years of development at University of Thessaloniki): 98SL221.
Fullerene-based donor–acceptor complexes and ion-radical salts with tetrathiafulvalenes, metalloporphyrins, and cyclic amines as donors: 99UK23.
High pressure and selectivity in reactions with formation of O- and N-heterocycles: 97T2669.
Lipases as acylation catalysts, mechanism and preparative applications, particularly in heterocyclic chemistry: 98AG(E)1608.
Metal-catalyzed hydrometallations in formation of heterocycles: 98PAC1059.
Microwave activation in phase-transfer catalysis of formation of heterocycles: 99T10851.
New catalysts and methods for enantioselective metal carbene reactions in syntheses of O- and N-heterocycles: 98PAC1123.
New *gem*- and *vic*-disubstituent effects on heterocyclizations: 99SL843.

Novel solvent-free organic reactions using focused microwaves with formation of heterocycles: 98S1213.

Ring-chain transformations and their application in the synthesis of heterocyclic compounds: 99MI31.

Screening of new microbial enzymes for preparation of biologically and chemically useful compounds: 97MI46.

Solid-phase synthesis of heterocycles: 98MI37, 98OPP489, 98YGK2.

Solid-state photoreactions in two-component crystals with formation of heterocycles: 98S1.

Solvent-free synthesis of heterocyclic compounds using microwaves: 99JHC1565.

Synthesis of heterocycles on solid-phase catalysts: 97S1217, 97T5643.

Synthesis of heterocycles under high pressure: 97LA623, 98H(47)1135.

Transition metal-mediated syntheses of heterocycles: 98MI26, 98MI27.

b. *Synthetic Strategies and Individual Methods.*

Alkene metathesis for the synthesis of carbo- and heterocyclic compounds: 97YGK1101.

Allylic protection groups and their removal through catalytic palladium π -allyl methodology in synthesis of heterocycles: 98T2967.

Annulation of carbocycle to heterocycle and cyclizations with simultaneous formation of carbo- and heterocyclic fragments: 98JCS(P1)983.

Annulation of chromium carbene complexes in synthesis of heterocycles: 99CSR187.

Application of Pummerer reaction in the synthesis of complex carbo- and heterocycles: 97S1353.

Asymmetric Birch reduction and reduction-alkylation in synthesis of natural products: 99CC1263.

Asymmetric Heck reaction in synthesis of heterocycles: 97T7371.

Asymmetric Pauson-Khand reaction in syntheses of heterocycles fused with five-member carbocyclic fragment: 98OPP121.

Asymmetric syntheses of heterocycles by conjugate addition reactions: 98EJO2051.

Asymmetric syntheses of heterocycles using carbohydrates as chiral auxiliaries: 97T14823.

Directed *ortho*-metallation—transition metal-catalyzed reaction symbiosis in heteroaromatic synthesis: 99JHC1453, 99PAC1521.

Electrochemical syntheses of fluorinated heterocycles: 99MI22.

Enantioselective synthesis with lithium/(-)-sparteine carbanion pairs: 97AG(E)2282.

Facile approach to highly functionalized N-, O-, and S-heterocycles using cascade syntheses with cumulated phosphorus ylides: 98MI25.

Formation of heterocycles by intramolecular catalytic asymmetric cyclopropanation: 98T7919.

Formation of N- and N,O-heterocycles from fluoro-substituted nitrogen ylides: 99MI23.

Formation of heterocycles in reactions using organozinc reagents: 96MI8.

Formation of 4- to 6-membered heterocycles in cycloaddition reactions: 98JCS(P1)3873.

Heteroatom radical addition–cyclization and its synthetic application: 99H(50)505.

Heterocyclizations by reactions of electrophilic carbenes with α -amino acids derivatives: 97T3425.

Intramolecular methathesis of α,ω -diolefins containing a heteroatom in the chain: 97AG(E)2036.

Inverse Diels–Alder reaction as synthetic tool in the synthesis of annelated heterocycles: 95F419.

Metalloacycles as intermediates in synthesis of heterocycles by transition metal-catalyzed coupling reactions under C–H bond activation: 99AG(E)1698.

Methods for synthesis of α,β -unsaturated trifluoromethyl ketones and their use in the synthesis of trifluoromethyl-substituted heterocycles: 99UK483.

Pummerer-type rearrangements in synthesis of heterocycles: 97YZ282, 98MI62.

Reactions of unsaturated carbanions with isothiocyanates in synthesis of heterocycles: 99JHC1469.

Recyclization reactions in the synthesis of small heterocycles: 99MI8.

Regioselective and stereoselective template synthesis in chemistry of fullerenes annelated with heterocycles or linked with heterocyclic fragments: 99ACR537.

Regioselectivity of syntheses of heterocycles in reactions of allyl-type anions with electrophiles: 99CRV665.

Stereocontrolled synthesis of spirocyclic compounds with heterocyclic fragments: 99T9007.

Stereoselectivity of chiral homoenolate equivalents in synthesis of heterocycles: 99S365.

Strategies of organic synthesis; in particular, synthesis of heterocycles: 98MI20, 98MI21, 98MI28.

Syntheses of ketones of heterocyclic series from acid chlorides and organometallic compounds: 99T4177.

Synthesis and applications of optically active cyclic nitroxides: 98T667.

Synthesis of chiral cyclic N,O-acetals of glyoxal useful for preparation of α -amino acids: 98SL449.

Synthesis of 1,3-diheteracycloalkanes (1,3-dioxa-, benzo-1,3-dioxa-, 1,3-oxaza-, 1,3-diaza-, 1,3-oxathia-, and 1,3-dithiacycloalkanes): 98MI24.

Synthesis of heterocycles, among them macroheterocycles, using olefin metathesis: 98T4413.

Synthesis of heterocycles by iodocarbocyclization and iodoaminocyclization with metal reagent participation: 99SL1191.

Synthesis of heterocycles by transition metal-catalyzed coupling reactions under C—H bond activation: 99AG(E)1698.

Synthesis of heterocycles using the intramolecular Heck reaction involving a “formal” *anti*-elimination process: 99H(51)1957.

Synthesis of nonracemic heterocyclic phosphonates: 97T16609.

Synthesis of tropones, tropolones, and tropylium salts with fused heterocyclic rings: 95AHC(64)81.

Use of bis(trimethylsilyl)acetamide and bis(trimethylsilyl)urea for protection and as control reagents in reactions with formation of heterocycles: 98S357.

Wolframacyclobutadienes as intermediates in syntheses of heterocycles: 99MI24.

c. Versatile Synthons and Specific Reagents.

Carbenes and carbenoids in synthesis of heterocycles: 96AHC(65)93.

Chiral acetylenic sulfoxides and related compounds in synthesis of heterocycles: 97MI39.

Chiral selenium compounds in syntheses of heterocycles: 99T1.

N,N-Dimethylhydrazine in synthesis of heterocycles: 99MI1.

Enamines as synthons in the synthesis of heterocycles: 99AHC(72)283.

Enaminones in heterocyclic synthesis: 97AHC(67)207.

Iminophosphoranes: versatile tools in heterocyclic synthesis: 95AHC(64)159.

Organocerium compounds in synthesis of heterocycles: 99T3803.

Organoiron compounds in syntheses of heterocycles: 99S727.

Phosphorus ylides in synthesis and transformations of heterocycles: 94MI4.

Recent advances in Cr(II)- and Cr(III)-mediated syntheses of heterocycles: 99S1.

Silylketenes as useful building blocks for heterocycles: 99JHC1555.

2-Substituted 3-dimethylamino- and 3-cyanopropenoates in the synthesis of heterocyclic systems: 99JHC1581.

Sulfones in synthesis and transformations of heterocycles: 93MI4.

N-Sulfonylimines as useful synthons in stereoselective synthesis of heterocycles: 97MI40.

Syntheses of heterocycles, among them carbazole alkaloids, with participation of tricarbonyl(η^4 -diene)iron complexes: 99CSR151.

Synthesis of heterocycles by sequenced reactions with participation of SmI₂: 98T3321.

Synthesis of heterocycles with participation of organozinc compounds: 98T8275.

Synthesis of heterocyclic compounds using hypervalent iodine reagents: 98AHC(69)1.

Thiocyanates in synthesis of heterocycles: 99T7957.

Use of $\text{Sc}(\text{OTf})_3$ as Lewis acid in formation of heterocycles: 99EJO15.

d. *Ring Synthesis from Nonheterocyclic Compounds.*

Amino acids as synthons for heterocyclic compounds: 95AHC(64)1.

Catalytic enantioselective addition to imines, in particular, aza-Diels–Alder reaction: 99CRV1069.

Catalytic asymmetric hetero-Diels–Alder addition of carbonyl compounds: 99ACR605.

Chiral $\text{Cu}(\text{II})$ -complexes as catalysts in hetero-Diels–Alder reaction: 99PAC1407.

Conjugated nitroso alkenes in reactions leading to heterocycles: 98UK523.

Cyanoacetamides and their $\text{C}=\text{S}$ and $\text{C}=\text{Se}$ analogs in synthesis of heterocycles: 99UK817.

Cyclizations initiated by C -acylnitrilium ions in synthesis of heterocycles: 99T9947.

Electrophilic heterocyclizations onto $\text{C}-\text{C}$ bonds mediated by halogen or chalcogen atoms: 97OPP33.

Electrophilic heterocyclizations onto $\text{C}-\text{C}$ bonds mediated by metal ions: 97OPP63.

Fluorinated 2,4-dioxo acids in synthesis of heterocycles: 99UK227.

Formation of O- and N-heterocycles in cyclization reactions mediated by organometallic compounds: 98MI39.

Formation of heterocycles in [4+2], [3+2], [4+1], [2+2], and [2+1] cycloaddition reactions to methylene- and alkylidenecyclopropane derivatives: 96MI9.

Heteroyl radicals in radical heterocyclization: 99CRV1991.

Heterocycles via Pd-catalyzed molecular quenching process: 98PAC1047.

Heterocyclization of quinoneimides: 99MI5.

Heterocyclization of vinylic selenides and tellurides: 97S373.

Heterocyclization under Vilsmeier conditions and its possibilities in drug research: 95F439.

Heterocyclization with participation of organomagnesium compounds: 99JOM(575)1.

Hetero-Diels–Alder reactions in organic chemistry: 97MI35.

Highly selective synthesis of polyfunctionalized heterocycles based on ring expansion of squaric acid derivatives: 97Y GK785, 98SL1167.

Interaction of carbonyl compounds with α,β -unsaturated nitriles as a convenient route to carbo- and heterocycles: 98UK442.

Intramolecular reactions of nitrile imines as a fruitful source of heterocycles: 98H(47)541.

Metal-mediated iodocarbocyclization and iodoaminocyclization reactions leading to heterocycles: 99SL1191.

Metal-promoted synthesis of heterocycles from heterocyclic compounds: 99JHC1523.

Palladium(II)-catalyzed cyclization of N-alkylation of allyl alcohols by uretanes and its application to the synthesis of natural saturated heterocycles: 98YGK34.

Palladium-catalyzed heteroannulation: 99PAC1436.

Polyfunctional fluoroalkyl-containing carbonyl compounds in the synthesis of heterocycles: 98IZV1279.

Reactions of 1,4-dichlorobut-2-yne derivatives leading to heterocycles: 99H(50)1227.

Stereocontrolled synthesis of polycyclic ethers and related heterocycles via intramolecular reactions of allylstannanes: 97YGK619.

Successive nucleophilic addition/ring closure (NARC) in stereocontrolled synthesis of heterocycles: 97MI38.

Synthesis of heterocycles by ring closure of *ortho*-substituted *t*-anilines: 96AHC(65)1.

Synthesis of heterocycles from glutaric acid derivatives: 98OPP657.

Synthesis of heterocycles on the basis of β,β -bifunctionalized ketene *N,N*-acetals: 98UK1013.

Syntheses of heterocycles on the basis of levulinic acid: 99UK80.

Synthesis of heterocycles via ring-closing heterodiene metathesis: 98JCS(P1)371.

Synthesis of heterocycles with C—C bond formation, involving organochromium(III) reagents: 99CRV991.

Synthesis of *p*-quinones, derivatives of fused systems including heterocyclic fragments through oxidation of phenol derivatives: 98OPP603.

Tandem reactions combining Diels–Alder reactions with sigmatropic rearrangements in syntheses of heterocycles: 98S227.

Transformations of nitro compounds, nitrones, nitrates, hydroxylamines, and amino-*N*-oxides into heterocycles: 98SL939.

Transition metal-catalyzed cyclizations accompanied by multiple bond migration with formation of heterocycles: 98SL1.

Trimethylenemethanediyl trapping in synthesis of heterocycles: 98EJO1.

e. *Syntheses by Transformation of Heterocycles.*

Alkynylpyridinium ions in synthesis of heterocycles: 98UK899.

Catalytic cross-coupling reactions in synthesis of bihetarenes and arylhetarenes: 98T263.

Reactions of heterocycles with α -aminoalkylphosphonates: 98UK940.
Stereoselective intermolecular [2+2]-photocycloaddition reactions of unsaturated heterocycles with formation of fused systems: 98S683.
Syntheses of natural heterocycles with C—P bond in side chain: 99T12237.
Synthesis of hetarylethylenes using novel methods for C=C double-bond formation: 98ACR584.
Synthesis of heterocyclic amines by hydrogenative amination of aldehydes and ketones: 99MI29.
Transition metal-catalyzed synthesis of hetaryl amines and hetaryl ethers from triflates and aryl/hetaryl halides or heterocyclic amines: 98AG(E)2046.

4. *Properties and Applications (Except Drugs and Pesticides)*

a. *Dyes and Intermediates.*

Derivatives of N-heterocycles with reactive substituents as dyes for animal fibers and synthetic polyamides: 97CLY149.

b. *Substances with Luminescent and Related Properties.*

Zn-complexes with heterocyclic ligands as fluorescent probes for biological investigations: 98CSR179.

Fluorescent derivatizing reagents with benzofuran skeleton for HPLC with spectrometric detection: 98YZ483.

Heterocycles as fluorescent sensors and switches: 97CRV1515.

Heterocycles as photochromes in photochemical processes for information recording systems: 98MI61.

Macroheterocycles as components of photo- and redox-active host ensembles: 98PAC2359.

Photoinduced transformations of photochromes (spiropyrans, furan-derived fulgides, dithienylethenes) in polymers: 98PAC2157.

c. *Organic Conductors (Except Polymers).*

peri-Fused S-hetarenes forming conducting molecular complexes: 97SL544.

Oligopyridine “two-dimensional” ligands connected by alkyne spacers with 1–4 ethynyl groups as photoactive conductors of molecular size: 96CC1707.

Polypyrrole, polythiophene, polyfuran, polycarbazole, polystyrene with tetrathiafulvalene substituents, polyethylene with carbazole substituents, and polyoxyphenazine as electrochemically active polymers for rechargeable batteries: 97CRV207.

Porphyrin and its isomers, porphycene, hemiporphycene, corphycene, and isoporphycene, as high-technological dyes: 99CLY767.

Tetrathia- and tetraselenafulvalenes, polythiophenes and related compounds, polypyridines as organic electronic conductors: 97YGK410.

d. *Coordination Compounds.*

Bimetallic catalysis by binuclear complexes of Cu, Fe, Mn, Ni, Pd, and Rh with heterocyclic ligands: 98T12985.

Borane complexes of P-heterocycles as versatile precursors for the synthesis of chiral phosphine ligands used for asymmetric catalysis: 98S1391.

Carbenoid complexes with heterocyclic ligands as catalysts in enantioselective cyclopropanation of olefins: 97S137.

Cationic complexes of *trans*-chelating tridentate ligand, (*R,R*)-4,6-dibenzofurandiyl-2,2'-bis(4-phenyloxazoline), with transition metal(II) perchlorates as effective catalysts for asymmetric cycloaddition of nitrones: 98YGK368.

Chiral carbenoid complexes with 2,2'-bipyridine ligands for asymmetric synthesis: 98YGK764.

Complexes with chiral heterocycles possessing P-containing substituents as P-mono- and P,N-bidentate ligands and their use in homogeneous asymmetric catalysis: 98KK883.

Complexes of *d*-elements with heterocyclic ligands as promising components of non-silver photographic systems: 97UK735.

Transition metal complexes with 2,2'-bipyridine ligands in anion-selective recognition and optical/electrochemical sensing: 96CC689.

e. *Polymers.*

Catalytic asymmetric dihydroxylation of alkenes with participation of insoluble polymer-bound cinchonine alkaloids: 99SLI181.

Electroluminescent conjugated polymers with heterocyclic fragments: 98AG(E)402.

Interface control of light-emitted devices based on pyridine-containing conjugated polymers: 99ACR217.

Linear monodisperse π -conjugated oligopyrroles and oligothiophenes as model compounds for polymers: 99AG(E)1350.

Poly(arylene oxides) with heterocyclic fragments based on new types of activated difluoroaromatic compounds: 99MI18.

Poly(phenylquinoxalines) containing acetylene groups in main chains of macromolecules: 99MI28.

Solvation, complexation, and reactivity of poly(*N*-vinyl lactams): 98MI29.

Synthesis of polyperileneimides and polynaphthylimides: 99MI4.

Synthesis, properties, and applications of polypyrrole as a conducting polymer: 97UK489.

Synthetic principles for bandgap control in π -conjugated systems of poly(thiophene) and related systems with fused thiophene rings: 97CRV173.

f. *Miscellaneous.*

Applications of tropones, tropolones, and tropylium salts with fused heterocyclic rings: 96AHC(66)285.

Azorhodanines and azothiopropiorhodanines as analytical reagents: 98UK236.
Chemistry of fragrant compounds (lactones and other O-heterocycles): 98T7633.

Copper(I) halide supramolecular networks linked by N-heterocyclic donor bridging ligands: 98PAC2351.

Derivatives of 4,4'-bipyridinium dication in electrochromic techniques: 96CLY135.

Derivatization of heterocycles for their detection by HPLC, GLC and GLC-MS: 97YZ647.

Heterocycles as fluorescent probes: 96MI11.

Heterocycles as herbicides: 94MI3.

Heterocycles as ligands in ionophores for potentiometric and optical sensors: 98CRV1593.

Heterocycles as perfumes: 94MI1.

Heterocycles as photoconductive materials used for electrophotographic organic photoreceptors: 99YGK541.

Heterocycles in industrial synthesis of fragrant and flavoring substaces in Czech Republic: 99CLY412.

Heterocycles in processes of color photography: 97CRV83.

Heterocycles in synthesis of herbicide glyphosate; heterocyclic derivatives and analogs of glyphosate: 97PHC17.

Macroheterocycles as components of molecular ensembles for metal ion binding: 98PAC2345.

Metal ion extraction with crown ethers containing linear lateral groups capable of acid ionization: 98PAC2393.

New reagents for ^1H NMR determination of enantiomeric excess in alcohols and amines, (R)-(-)- and (S)-(+)-O-coumarinylmandelic acids: 97YZ786.

Polysaccharide derivatives for chromatographic separation of heterocyclic enantiomers: 98AG(E)1021.

Use of heterocycles, in particular tetrathiafulvalenes and pyridinium zwitterions, in unimolecular electronic devices: 99ACR950.

C. SPECIALIZED HETEROCYCLES

1. Nitrogen Heterocycles (Except Alkaloids)

a. General Sources and Topics.

The aza-Wittig rearrangement in synthesis and transformations of N-heterocycles: 97S497.

Design of self-adapting N-heteroaromatic-substituted claw ligands as E^-/M^+ (E = p-block element, M = main group metal) charged spacers: 97CB1365.

One-pot preparation and stereochemistry of saturated polyazapolyheterocycles: 99YGK280.

Syntheses, conformations, electronic structures, and radical polymerization of *N*-vinylactams: 98MI29.

Versatile supramolecular helicated metal complexes with *N*-heterocyclic ligands: 97CRV2005.

b. *Structure and Stereochemistry.*

Alkyl(alkoxy)silylalkyl-derivatives of *N*-heterocycles: 99UK318.

Chiral crystallization of achiral *N*-heterocycles—generation of chirality without chiral environment: 98YGK268.

N-Heterocycles as fragments of artificial supramolecules capable of multiple interactions: 97YGK357.

Isolation and characterization of stereoisomers in di- and trinuclear complexes with *N*-heterocyclic ligands: 98CSR185.

c. *Reactivity.*

Complexes of *N*-heterocycles with boron compounds: 99JOM(581)129.

Heminal-activated haloolefins with heterocyclic fragments in reactions with *N*-heterocycles as nucleophiles: 98UK317.

Homogeneous and heterogeneous catalytic activation (in the dark and in the light) of cyclic 1,2-diazenes: 97SL1335.

Lewis acid complexes of saturated cyclic tertiary amines and related compounds: 97CRV721.

Nucleophilic aromatic amination by the action of unsaturated *N*-heterocycles: 99T11399.

Oxidative addition reactions of platinum(II) complexes with *N*-heterocyclic ligands: 97CRV1735.

Selective hydrogenation of fused *N*-heterocycles on Re-catalysts: 98UK175.

d. *Synthesis.*

Cyclization of α - and β -aminoketones to 3–6-member *N*-heterocycles: 99KGS723.

Electrophilic cyclizations of unsaturated amides to form 5- and 6-member lactams: 98T13681.

Free-radical cyclization in the synthesis of *N*-heterocycles: 97T17543.

N-Heterocycles based on aromatic unsaturated ketones: 98MI13.

N-Heterocycles, formation in transition metal-catalyzed enyne metathesis: 98YGK433.

Heterocyclization by catalytic formation of $N-C_{ar}$ bond to give indoles, aminopyridines, and *N*-aryl-substituted saturated heterocycles: 98ACR805.

Metal carbenoid-mediated tandem processes in synthesis of azapolycycles: 97MI36.

New synthetic pathways to nitrogen heterocycles: 97LA1.

Nitrogen heterocycles from furans by aza-Achmatovicz reaction: 98SL105.

Syntheses and use of vicinal diamines with one or two N atoms included in heterocycle as chiral auxiliaries: 98AG(E)2580.

Synthesis of N-heterocycles using ureas and related compounds: 98UK333.

Synthesis of N-heterocycles with C—N bond formation catalyzed by transition metal catalysts: 97SL749.

Transition metal-catalyzed heterocyclizations with formation of N-heterocycles: 98AG(E)2046.

2. Oxygen Heterocycles

a. *Chemistry of Individual Classes of O-Heterocycles.*

Infrared carbonyl frequencies of heterocyclic lactones: 98H(47)559.

Saturated O-heterocycles: 98JCS(P1)4175.

b. *Reactivity.*

Asymmetric induction of radicals with neighboring ester fragment and O-heterocyclic stereogenic center: 98SL213.

Cycloadditions of oxa-aromatics and their reactions with nucleophiles: 96AHC(65)283.

O-Heterocycles in synthesis of drimane sesquiterpenoids from labdane diterpenoids: 97IZV896.

Reactions of cyclic acetals with organosilicon compounds: 97MI42.

Ring opening of oxabicyclic compounds as a strategy in organic synthesis: 97MI37.

Transformations of cyclic acetals and their heteroanalogs under the action of organosilicon reagents: 97MI1.

[1,2]-Wittig rearrangement with participation of O-heterocycles: 97LA1275.

c. *Synthesis.*

Electrophilic cyclizations of unsaturated amides to form five- and six-member lactones: 98T13681.

Formation in saturated O-heterocycles via carbon-heteroatom bond-forming reductive elimination: 98ACR852.

Halocyclization in formation of O-heterocycles: 98YGK661.

Low-valent chromium compounds in synthesis of natural O-heterocycles: 99CSR169.

Mechanism of ozonide formation: 99MI9.

Polymer-supported synthesis of natural macrocyclic lactones and other O-heterocycles: 99AG(E)1903.

Stereocontrolled synthesis and recyclizations of C-acetylenylglycosides in the presence of dicobalt hexacarbonyl with formation of fused saturated O-heterocyclic systems: 98CC2665.

Synthesis of bis-spiroacetal cyclic systems: 99T7661.

Transformation of cyclic ketones into lactones by Baeyer–Villiger oxidation: 99EJO737.

Transition metal catalysis in Baeyer–Villiger oxidation of cyclic ketones with formation of lactones: 98AG(E)1198.

Transition metal-catalyzed heterocyclizations with formation of O-heterocycles: 98AG(E)2046.

3. Sulfur Heterocycles

a. Chemistry of Individual Classes of S-Heterocycles.

peri-Fused S-heteroarenes forming conducting molecular complexes: 97SL544.

Natural cyclic disulfides, disulfides, and polysulfides: 97UK901.

Saturated S-heterocycles; synthesis and properties: 98MI15.

Synthesis, properties, and reactions of cyclic trisulfides and their oxides: 98OPP551.

Thiophene, benzo[*b*]thiophene and dibenzo[*b,d*]thiophene as precursors to highly conjugated organosulfur compounds formed at Rh- and Ir-mediated thiophene ring opening: 97SL643.

b. Structure and Stereochemistry.

Synthesis and stereochemical study of reactions of optically pure cyclic sulfuranes, S(IV)-derivatives and their Se(IV)- and Te(IV)-analogs: 99Y GK587.

c. Reactivity.

New reactions utilizing features of sulfur atom in chiral sulfoxides, derivatives of heterocycles: 99YZ126.

Ring contraction of heterocycles by sulfur extrusion: 96AHC(65)39.

d. Synthesis.

Homolytic addition of dithiols to alkynes as a new approach to construction of dithiacranes and thiacyclobutanes: 97IZV1256.

D. NATURAL AND SYNTHETIC BIOLOGICALLY ACTIVE HETEROCYCLES

1. General Sources and Topics

Progress in chemistry of organic natural products: 96FOR(68)1; 96FOR(69)1; 97FOR(70)1; 97FOR(71)1; 97FOR(72)1; 98FOR(73)1; 98FOR(74)1; 98FOR(75)1; 99FOR(76)1; 99FOR(77)1; 99FOR(78)1.

a. *Biological Functions of Natural and Synthetic Bioactive Heterocycles.*

Carbohydrate mimetics including heterocyclic fragments and a new strategy for tackling the problem of biological recognition with participation of carbohydrates: 99AG(E)2300.

Chemical studies on nyctinastic leaf movement regulated mainly by heterocycles: 99H(51)927.

Comparative QSAR analysis of derivatives of heterocycles as nonsteroid ligands of estrogen receptors: 99CRV723.

Complexes with macroheterocyclic ligands as synthetic analogs of metallo-biosites: 99CSR159.

Ecological danger of polychlorinated dibenzodioxines and dibenzofurans: 97MI26, 97MI45, 98MI33, 98MI34.

Functionalized supramolecular systems based on monolayers of bioactive compounds (macrocyclic ionophoric peptides, crown ethers, nucleoside derivatives): 98MI12.

Gene-directed biologically active substances (non-sense oligonucleotides and their derivatives): 97MI17.

Harmful natural heterocycles (including petroleum components and products of oil, coal, and shale processing): 98MI23.

Heterocycles as agents for the cleavage of phosphodiester bonds in RNA: 98CRV961.

Heterocycles controlling leaf movement of plants: 99YGK571.

N-Heterocyclic fragments as active centers of biomolecules with hydrogen-bonding receptors: 98PAC2371.

Heterolignans, synthetic analogs of lignans containing heterocyclic fragments: 99H(51)1443.

Interdependence of redox and molecular recognition of natural and synthetic heterocycles: 99ACR44.

Macroheterocycles as components of supramolecular structures in carbohydrate recognition through noncovalent interactions: 99AG(E)2978.

Mechanism of formation of methemoglobin, heme with oxidized Fe atom not capable for binding and transfer of oxygen: 99MI16.

Mechanism of RNA decoding in protein biosynthesis: 97YZ749.

Metal complexes with heterocyclic ligands in medicine: 99AG(E)1512.

Model compounds in the study of repair of UV-light-induced DNA lesions: 98EJO1245.

Naturally occurring bromo derivatives of heterocycles: 99CSR335.

Naturally occurring halo derivatives of heterocyclic compounds: 98ACR141.

Novel natural fused O-heterocycles from marketed plants of eastern and southern Africa: 99PAC919.

Participation of nucleotides in NO formation processes in mammalian organisms: 97CCC1355.

Prebiotic chemistry of natural heterocycles, among them purines, pyrimidines, sugars, and riboflavin: 97T11493.

Silatrane and germatrane as new bioactive compounds: 98MI60.

Spiropyran and fulgides bound with biomolecules as photoswitchable biomaterials (a route to optoelectronic systems): 97ACR347.

Synkinetic chemistry of natural molecular ensembles including heterocyclic components: 97SL1015.

Theoretical methods of pattern recognition in determination of the activity type of some heterocycles: 97ZOR9.

b. *General Approaches to Syntheses of Biologically Active Heterocycles.*

Advances in total synthesis and development of natural products using carbohydrates: 97Y GK970.

Alkylation of chiral triflates with organomagnesium or organolithium reagents for construction of side chains of naturally occurring heterocycles, antibiotics, and enzyme inhibitors: 99Y GK334.

Allenylmethylsilanes in synthesis of natural N- and O-heterocycles: 97Y GK793.

Catalytic synthesis of natural fused O- and N-heterocycles: 98JHC1057.

Chemoenzymatic synthesis of chiral biologically active heterocycles: 97F307.

Combinatorial synthesis of multicomponent mixtures, among them mixtures of heterocycles, for screening of bioactive compounds: 99JMC3743.

Cyclitols as novel chiral building blocks in synthesis of heterocyclic natural products: 97CC807.

Cycloaddition approaches to O-heterocycles in synthesis of natural products: 99JHC1391.

Cyclohexadiene-*cis*-diols as versatile intermediates in the synthesis of natural heterocycles: 96CC1993.

Development of base-catalyzed Diels–Alder reaction of 3-hydroxy-2-pyrone and its application to synthesis of bioactive compounds: 99Y GK84.

Fluorination of O- and N-heterocycles with molecular fluorine in the synthesis of fluorine-containing bioactive compounds: 98Y GK107.

Methods for the synthesis of naturally occurring heterocycles: 94MI5.

Novel indole synthesis and its application to natural product synthesis: 98JHC1043.

Syntheses of natural O- and N-heterocycles via stereocontrolled Pd-catalyzed reactions: 99PAC1065.
Synthesis of azasugars and alkaloids via ring-closing metathesis: 99EJO959.
Synthesis of natural compounds by intramolecular attack of N-nucleophile on electrophilic center: 99CSR61.
Synthesis of natural products containing bis-spiroacetal fragment: 99JHC1373.
Synthesis of tritium-labeled biologically important diazines: 99UK254.
Total syntheses of naturally occurring molecules possessing 1,7-dioxaspiro[4,4]nonane skeletons: 99EJO1757.
Total syntheses of pyrrole alkaloids, furanoterpenes, macrolide antibiotics, and carbohydrates based on transformations catalyzed by transition metals: 99SL1523.
Total synthesis of complex bioactive natural N-, O-, and N,O-heterocycles, alkaloids, enzyme inhibitors, and herbicides: 99YGK736.
Wittig and related reactions in the synthesis of natural O-heterocycles: 97LA1283.

2. Alkaloids

a. General.

Acid-catalyzed C-3 epimerization of reserpine and other indolo[2,3-*a*]quinolizidines: 98H(48)1275.
Dehydro- β -agarofuran sesquiterpenes, *ansa*-macrolide alkaloids, mantansioids, and triazacyclotridecane alkaloids as important compounds of *Celastraceae* plants: 99CLY690.
Ligand recognition in the opioid receptors by modeling methods and design of opioids: 98YZ1.
Plant and animal steroid alkaloids: 99KPS131.
Synthesis of coordination compounds and catalysis with participation of fluoro derivatives of alkaloids: 98KK3.

b. Structure.

Conformational study of geissoschizine isomers and their model compounds (geissoschizine is the indolo[2,3-*a*]quinolizidine derivative considered to be an important participant of indole alkaloids biogenesis): 99H(51)649.
Mass spectrometry in structural investigation of diterpene alkaloids: 97IZV1096.
Structures of marine sponge alkaloids manzamines and related compounds: 97H(46)765.

c. Synthesis.

Alkaloid syntheses by cascade cycloaddition/cyclization reactions of *N*-acyliminium ion: 98CC1417.

Biomimetic formation and interconversion in the heteroyohimbine series: 98H(48)1483.

Development and applications of new reactions for construction of basic structures of natural products, in particular proaporphine alkaloids and lactones: 99YZ357.

Enaminones as versatile intermediates for alkaloid synthesis: 99PAC979.

Heterocyclic and acyclic azadiene Diels–Alder reaction in total synthesis of nothapodyne B, alkaloid with antiviral activity: 98JHC1003.

Manzamine alkaloids (β -carboline derivatives from sponges), syntheses and synthetic approaches: 98T6201.

New synthetic methodology on the route to asymmetric piperidine alkaloids: 99JHC1549.

Pictet–Spengler syntheses of tryptamines containing hydroxy group at N atom of the side chain, and their transformation to tri-, tetra-, and pentacyclic systems related to eudistomins: 98H(49)499.

Progress in total synthesis of marine alkaloids, aaptamines: 99H(50)549.

Racemic and chiral syntheses of some indolo[2,3-*a*]quinolizidine alkaloids through a lactim ether route: 98H(47)525.

Regio- and stereocontrolled radical cyclizations in syntheses of alkaloids: 97YZ973.

Synthesis of quaternary benzo[*c*]phenanthridine alkaloids and their analogs: 97AHC(67)345.

Synthesis of some alkaloids using allyl boron derivatives: 99KGS1015.

Synthetic studies on alkaloid pumiliotoxin C using Pd-promoted cyclization: 98YGK818.

Tandem cyclization–cycloaddition of diazoketones in the synthesis of some alkaloids: 97F303.

Total syntheses of alkaloids containing spiro fragments: 97YZ468.

Total synthesis of 2-(6-chloro-3-pyridyl)-7-azabicyclo[2.2.1]heptane (epibatidine): 97MI21.

d. *Individual Groups of Alkaloids.*

Alkaloids from Australian flora: 96MI2.

3-Alkylpiperidine alkaloids from marine sponges of Haplosclerida order: 96MI4.

Biogenesis of manzamine alkaloids (β -carboline marine alkaloids): 97H(46)765, 97YGK1114.

Biosynthesis of tropan alkaloids hyosciamine and scopolamine by isomerization of alkaloid littorine in *Datura stramonium* and related species: 98CSR207.

β -Carboline and isoquinoline alkaloids from marine organisms: 96MI5.

Isolation, synthesis, and possible mechanism of action of anticancer alkaloid camphotecin and related alkaloids: 99YGK181.

Production of morphinone as a metabolite of morphine and its physiological role: 99YZ249.

Pyridine and piperidine alkaloids: 96MI3.

Pyrrolidine alkaloid transformations in insect organisms: 98EJO13.

Quinazoline alkaloids: 97KPS297.

Recent advances on antitumor-active benzo[c]phenanthridine alkaloids: 99H(50)627.

Structures and functions of marine bioactive alkaloids: 99YGK105.

Studies on the alkaloids of *Erythrina* plants: 99YZ340.

3. Antibiotics

a. General.

Antibiotics and macroorganisms: 99MI36.

b. Antitumor Antibiotics.

Chemical biology of epitolones A–E, 16-member macrolide antitumor antibiotics: 98AG(E)2015.

Design and synthesis of analogs of dynemycin, antitumor antibiotic with anthraquinone, 10-member enediyne, tetrahydropyridine, and oxirane fragments: 99EJO1.

Enediynes with ethylene carbonate fragment in the synthesis of dienediyne models of the biradical-forming and DNA-cleaving natural neocarzinostatin chromophore: 99SL657.

Studies on asymmetric total synthesis of antitumor antibiotic, fredericamycin A: 98YGK963.

Syntheses of duocarmycins and related antibiotics: 97CRV787, 98YZ353.

Synthesis of the densely functionalized heterocyclic system of antitumor antibiotics azinomycins A and B, azirino [1,2-*a*]pyrrolidine derivatives: 98SL1031.

Synthetic and mechanistic studies of bleomycin: 99AG(E)448.

Total synthesis of antitumor antibiotic FR900482, 3,9-epoxy-3*H*-azirino[2,3-*c*]-l-benzazocine. 97YGK946.

c. β -Lactam Antibiotics.

Asymmetric synthesis of 3-amino β -lactams via Staudinger ketene–imine cycloaddition reaction: 98KGS1448.

Chiral 2,2-disubstituted 1,3-benzoxazin-4-ones as auxiliaries in the synthesis of carbapenem antibiotics: 97YGK858.

Development of efficient synthetic methods for 1 β -methylcarbapenems: 98YZ353.

β -Lactam antibiotic amoxycillin in therapy of infections: 97MI14.

Molecular mechanisms of penicillins binding with biopolymers and membranes: 98ZOR655.

New processes for manufacturing the key intermediates for the synthesis of β -lactam antibiotics: 99YGK387.

Novel chiral auxiliary, 2'-isopropyl-5'-methylbenzoxazine-spiro-[2.1']cyclohexan-4-one, and its application to the synthesis of carbapenem antibiotics: 97YGK858.

Penicillin biosynthesis and Pummerer reaction: 98KGS1463.

Recent advances in carbapenem chemistry: 98KGS1475.

Regio- and stereocontrolled radical cyclizations in syntheses of β -lactams: 97YZ973.

Stereocontrolled synthesis of oxabicyclic β -lactam antibiotics via [2+2] cycloaddition of isocyanates to sugar vinyl ethers: 96CC2689.

d. *Macrocyclic Antibiotics.*

Avermectins biosynthesis: 97CRV2591.

Biosynthesis of erythromycin and rapamycin: 97CRV2611.

Biosynthesis of macrolide antibiotic avermectins: 97MI11.

Chemical modification of glycopeptide antibiotics with macrocyclic aglycones: 98MI48.

Chemical modification of monensin, polyether ionophoric antibiotic with bound tetrahydropyran, two tetrahydrofuran, and octahydrospiro-2,2'-furofuran fragments: 97YZ583.

Chemistry, biology, and medicine of glycopeptide antibiotics, in particular synthesis of vancomycin: 99AG(E)2096.

Configurational assignment of polyene macrolide antibiotics using the [^{13}C]acetone analysis: 98ACR9.

Discovery and development of a novel immunosuppressant, Tacrolimus hydrate (FK-506), macrolide antibiotic with piperidine and tetrahydrofuran fragments: 97YZ542.

Macrolides and phagocytosis: 99MI10.

Macrolides with azine fragments: 98KGS867.

Mechanism of resistance to macrolides of their producers and eubacteria: 98MI4.

Progress in the synthesis of chiral natural macrolide antibiotics epothione B and tartrolon B: 99JHC1421.

Properties, structure, and derivatives of macrocyclic polyenic lactone amphotericin B: 97MI12.

Stereostructure and synthesis of aplyronine A (macrolide with 24-member lactone ring), an antitumor substance of marine origin: 96YGK1077.

Total synthesis of antitumor macrolide rhizoxin: 97YZ486.

Total synthesis of brefeldins, 13-member lactones with four asymmetric centers: 97YGK110.

Total synthesis of macrolide immunodepressants using 1,3-dithiane aldol couplings and σ -bond olefin constructions: 98ACR35.

Vancomycin group of antibiotics and the fight against resistant bacteria: 99AG(E)1172.

e. *Miscellaneous Antibiotics.*

Aminoglycoside antibiotics: 97MI16.

Antibiotics from *Xenorhabdus* and *Photorhabdus* spp. (Enterobacteriaceae): 98KGS1561.

Antimicrobial action peculiarities and pharmacokinetics of novel fluoroquinolones: 98MI6.

Antimicrobial spectrum, pharmacodynamic and pharmacokinetic characteristics of rifabutin, a long-acting antituberculosis semisynthetic antibiotic of rifamicin group: 99MI13.

Comparative pharmacokinetics of lomefloxacin and other fluoroquinolones: 98MI8.

Fluoroquinolone antibiotic ciprofloxacin: 97MI13, 99MI12.

Fluoroquinolone antibiotic ofloxacin in therapy of mycobacterioses: 97MI15.

Fluoroquinolones in clinic praxis: 98MI22, 98MI46, 98MI47.

Fungicide antibiotic micothiazoles (4,4'-bithiazole derivatives with β -methoxyacrylic acid fragment as a substituent) as cell respiration inhibitors: 98UK595.

Lomefloxacin, difluoroquinolone with wide spectrum of antimicrobial activity: 98MI3.

Macrocyclic peptides as antibacterial glycopeptide antibiotics: 98KGS1605.

Mass spectrometry of ergot alkaloids: 98CLY538.

Novel synthetic strategies for DNA binding pyrrolobenzodiazepine antibiotics: 98KGS1588.

Pharmacokinetic interaction between fluoroquinolones and other drugs: 98MI5.

Pharmacokinetic interaction of fluoroquinolones and methylxanthines: 99MI11.

Phosphomycin, (1R,2S)-(1,2-epoxypropyl)phosphonic acid: 97YGK229.

Synthetic studies on heteroanthracyclines, heteroanalogs of anthracycline anti-tumor antibiotics: 97H(46)705.

Total synthesis and development of some antibiotics and enzyme inhibitors: 98YGK714.

Total synthesis of (+)- and *ent*-(-)-duocarmycin: 95MI8.

Unified approach to peptin antibiotics containing pyridazine, quinoline, and benzopyridazine fragments): 99JHC1409.

4. *Vitamins*

Anaerobic pathway of vitamin B₁₂ biosynthesis: 98H(47)1051.

Biosynthesis of thiamine: 97AG(E)1032.

Biotinyl diazirine photophore for probing receptor-ligand interface: 98H(47)625.

Electroorganic reactions mediated by vitamin B₁₂ modeling complexes: 96YGK859.
Metabolic interrelations between S-adenosylmethionine, folates, and vitamin B₁₂: 96CLY165.
Some approaches to the directed search of drugs on the basis of nicotinic acid: 99KFZ(4)6.
Total syntheses of biotin: 97CRV1755.
The unexpected role of vitamin E (α -tocopherol) in peroxidation of human low-density lipoprotein: 99ACR27.

5. *Drugs*

a. *General.*

Biosterism-based approach in drug design: 96CRV3147.
Biotechnology and synthetic chemistry as routes to clinically important compounds, in particular terpenolactones: 99PAC1025.
Chemistry and molecular biology of transmissible spongiform encephalopathies: 97AG(E)1674.
N-Heterocycles as neuronal acetylcholine receptors in drug discovery: 97JMC4169.
Molecular recognition of protein–ligand complexes and drug design: 97CRV1369.
Preparation of clinically important natural products using plant cell culture and synthetic chemistry: 97G293.
The role of hydrogen bonding and hydrophobic interactions in medicinal effect of heterocycles: 99AG(E)736.

b. *Definite Types of Activity.*

Antibacterial agents, mainly heterocycles: 97CC2333.
Anticancer pyrimidine acyclonucleosides: 95F395.
Antihypoxic properties of some N- and S-heterocycles: 97KFZ(2)3.
Antitumor compounds, among them cyclic peptides, terpenoids, and alkaloids isolated from higher plants: 99YZ529.
Avidin–biotin system as the aid of address delivery of antitumor compounds: 98MI54.
Benzimidazole and benzodiazepines derivatives as retinoid antagonists: 96YZ928.
Benzo- and tetrahydrobenzofurocoumarins as photochemotherapeutic drugs: 97F289.
Bioactive O- and N-heterocycles from Indonesian medicinal plants: 96YZ911.
Bioactive polyphenol acids and lignans with O-heterocyclic fragments and terpenoid δ -lactones as components of traditional Chinese drugs: 98PAC547.
Central and peripheral analgesic agents from *kappa*-opioid agonists: 95F405.

Combined chemotherapy/gene therapy of tumors based on activation of antitumor preparations with cytochromes P450: 99MI38.

Compounds of B₁₂ series combined with ascorbic acid as potential antitumor agents: 98MI56.

Cyclic sulfoxides, lactones, lactams, and other heterocycles as transdermal penetration enhancers: 99CLY107.

Design and synthesis of glutathione analogs containing heterocyclic fragments: 98F721.

Design and therapeutic application of matrix metalloproteinase heterocyclic inhibitors: 99CRV2735.

Design of DNA minor-groove binders as new antitumor agents: 99F15.

Development of large-scale synthesis of antitumor agent EO9, an indoloquinone derivative: 99YGK401.

DNA cross-linking agents (mainly heterocycles) as antitumor drugs: 98CRV2723.

Ferrocenylalkylazoles as a new class of low-toxic compounds with antitumor activity: 98MI58.

Heterocycles as anti-aggregation agents: 97KFZ(3)3.

Heterocycles as drugs for diabetes of type II treatment: 97KFZ(11)5.

N-Heterocycles as drugs for the treatment of benign prostatic hyperplasia: 97JMC1293.

Heterocycles as drugs in pharmacological treatment of obesity: 99JMC181.

Heterocycles as inhibitors of tumor necrosis α -factor: 99JMC2295.

Heterocycles as new drugs in antiglaucoma therapy: 97JMC2793.

Heterocycles as nonnucleoside reverse transcriptase inhibitors for therapy of HIV-1 infection: 99F26.

Heterocycles as pharmacological means acting on insulin resistance: 99KFZ(7)13.

Heterocycles as selective antagonists at α_1 -adrenoreceptors: 98F278.

Heterocycles as synthetic estrogens in emerging therapies for the prevention or treatment of postmenopausal osteoporosis: 99JMC1.

Heterocycles, calcium channels blockers, as auxiliary agents in resistant tumor therapy: 97YZ455.

O-Heterocycles of microbial origin as preparations for atherosclerosis therapy: 98MI7.

Heterocyclic analogs of *N*-nitrosoureas as anticancer drugs: 97CRV829.

Heteropentalenes, heterocycles with antiphlogistic properties: 98CLY390.

Improved understanding of antitumor chemistry of Pt-cycles: 96CC801.

Indolo-, benzofuro- and benzothenopyrimidinediones as selective ligands of α_1 -adrenoreceptors: 95F471.

Interaction of biomolecules with ginghamosu (artemisinin) and its derivatives in the presence of Fe(II) ion—an exploration of antimalarial mechanism: 99PAC1139.

Investigation of cytotoxic antitumor activity of phthalocyanin conjugates with epidermal growth factor: 98MI55.

Metal complexes with heterocyclic ligands as enzyme inhibitors: 99CRV2711.

Monoclonal antibodies against naturally occurring cannabinoids, partly saturated annelated pyrans, marichuana components: 99YGK708.

Natural and synthetic heterocycles as DNA cross-linking agents and antitumor drugs: 98CRV2723.

Natural organic compounds (among them heterocycles) affecting microtubule functions: 98YZ111.

New antibacterial agents: 96JMC3853.

Novel bioreductive anticancer agents based on indolequinones: 97F271.

Nucleic acid recognition by metal complexes of bleomycin: 99CRV2797.

Octasodium salt of octa-4,5-dicarboxyphthalocyanin cobalt(II) combined with ascorbic acid (teraphthal) as a new drug for binary catalytic therapy of malignant tumors: 98MI57.

Pharmacology, chemistry, and technology of medicines on the basis of synthetic antioxidants, derivatives of 3-hydroxypyridine and 5-hydroxynicotinic acid: 98MI59.

Principles and recent developments in chelation treatment of metal intoxication: 99CRV2683.

Recent advances in the discovery and development of plant-derived natural products and their analogs, in particular polycyclic O-heterocycles, as anti-HIV agents: 99PAC1045.

Recent developments in chemotherapy of HIV infections: 98PAC567.

Recognition and reaction of metallointercalators with DNA: 99CRV2777.

Saturated N-heterocycles with serotonin 5-HT₃ and 5-HT₄ receptor binding affinities: 98YGK851.

Sleep and its modulation by N- and N,O-heterocyclic drugs that affect γ -aminobutyric acid (GABA) receptor function: 99AG(E)2852.

Structure-activity relationship for ginkgolides (lactones) as PAF-antagonists: 99PAC1153.

Synthetic and natural antimalarials, among them heterocyclic compounds: 99JHC1599.

c. Individual Substances and Groups of Compounds.

β -Aminomethylated N-aryl- and N-azaheteroaryl-2,5-dimethylpyrroles, compounds with potential biological activity: 95F431.

Antimalarial drug artemisinin, sesquiterpenic δ -lactone with 1,2,5-trioxane (endoperoxide) fragment: 98CSR273.

Arbidol (ethyl 6-bromo-4-dimethylaminomethyl-5-hydroxyindole-3-carboxylate), a new immunomodulator: 99KFZ(3)3.

Asymmetric total synthesis of antitumor styryl lactones and related natural compounds: 97H(45)367.

- Azolophenanthridines as antineoplastic agents: 95F365.
- Biochemistry and pharmacology of melanine pigments, indole derivatives: 99MI14.
- Biological and medicinal chemistry of bi-heterocycles and bi-chelates: 97CB669.
- Cephempime (cephalosporine antibiotic of IV generation) in the treatment of patients with severe hospital infections: 99MI37.
- Chemical composition and perspectives of using in medicine of lactones and benzofuran derivatives from *Cetraria islandica* (L.) ACH: 99MI15.
- Chemistry, biology, and medicine of tubulin polymerizing macrocyclic lactones: 99PAC989.
- Chemistry, pharmacology, and clinical efficacy of the chinese nootropic agent huperzine A, alkaloid from *Huperzia serrata* with annulated 2-pyridone and 1-amino-3-methyl-9-ethylidenebicyclo[3.3.1]nona-2,6-diene fragments: 99ACR641.
- Combined biotechnological and synthetic methods for preparation of polycyclic γ -lactones, clinically important compounds: 98PAC2093.
- Development of a new indolocarbazole anticancer agent (NB-506): 97YGK566.
- Development of hypoglycemic agent troglitazone (CS-045). 6-hydroxy-2-[p-(2,4-dioxothiazolidin-5-ylmethyl)phenoxy-methyl]-2,5,7,8-tetramethylchromane: 97YZ597.
- Development of novel response modifiers derived from thalidomide (3-phthalimidopyridine-2,6-dione) and other phthalimide derivatives: 99YGK94.
- Discovery and development of *N*-benzyl-4-(5,6-dimethoxy-1-oxo-indan-2-ylmethyl)piperidine hydrochloride (donepezil hydrochloride) for treatment of Alzheimer's disease: 98YGK320, 99YZ101.
- Fluoroquinolones in the treatment of pneumonia in elderly patients: 99MI34.
- Fluoroquinolones in therapy of bacterial infections: 99MI35.
- Gadolinium(III) chelates as magnetoresonance imaging (MRI) contrast agents: 99CRV2293.
- B-Heterocycles in tumor therapy: 98CRV1515.
- N-Heterocycles as antitumor agents: 98CSR251.
- N-Heterocycles as inhibitors of dimerization of HIV-1 protease: 98SL1040.
- Heterocyclic sulfone derivatives with anti-HIV activity: 97F321.
- Lethal drug interactions of new antiviral, sorivudin [1- β -D-arabinofuranosyl-(*E*)-5-(2-bromovinyl)uracil], with anticancer prodrugs of 5-fluorouracil structure: 97YZ910.
- Naphthalocyanins, phthalocyanins, and porphyrins as sensitizers in photodynamic cancer therapy: 98IZV836.
- Natural N-heterocycles as novel drugs against tuberculosis: 98PAC365.
- New chemistry of qinghao, a marvelous herb of antiquity, and antimalarial trioxane qinghaosu: 97ACR73.

- New natural γ - and δ -lactones from medicinal plants of Pakistan: 98PAC385.
- Nucleosides, nucleotides, peptidomimetics, and reverse transcriptase inhibitors in HIV infection therapy: 98CCC449.
- Oxazolidinones and dihydrofuranones as inactivators and substrates of monoamine oxidase B, approaches to the design of antiparkinsonian agents: 97F343.
- Pharmacological activity spectrum and toxicological properties of benzimidazole derivatives: 99KFZ(5)6.
- Phenothiazines, benzo-1,2,4-thiadiazine 1,1-oxides, fluoroquinolones, nicotinic acid, and nitrofurantoin derivative as potentially dangerous photosensitizers: 98MI11.
- Phthalimides with α -TNF production regulating activity as novel biological response modifiers (α -TNF is α -tumor necrotic factor): 97YZ91.
- Physiological and pharmacological activity of simple alkyl- and arylpyrazines: 97YZ435.
- Porphyrin-based photosensitizers for use in photodynamic therapy: 98T4151.
- Porphyrins, modified natural chlorophylls, chlorins, phthalocyanins, xanthenes, phenothiazine, and phenoxazine dyes as new sensitizers for photodynamic therapy: 98MI50, 98MI51, 98MI52, 98MI53.
- Preparation of calcium antagonist diltiazem, derivative of 2,3-dihydro-4*H*-1,4-benzothiazepinone: 99YGK394.
- Pt-complexes with heterocyclic ligands and Pt-chelates as antitumor drugs: 99CRV2451.
- Pyrazino[2,3-*c*]-1,2,6-thiadiazine 2,2-dioxides useful in medicinal chemistry: 97F283.
- Pyridazino[4,5-*b*]quinolinediones as novel glycine-*N*-methyl-D-aspartate antagonists for the treatment of stroke: 98JHC1171.
- Recognition of DNA sequence and molecular design of artificial repressors, pyrrole-imidazole polyamides, and calicheamycin derivatives: 97YGK384.
- Research and development of Beraprost sodium, a stable modified prostaglandin PGI₂ possessing cyclopenta[*b*]benzofuran fragment: 96YGK1055, 97YZ509.
- Structure, recognition, and processing of cisplatin-DNA adducts: 99CRV2467.
- Structures of new terpenoids with N- and O-heterocyclic fragments from Chinese herbal medicine: 98PAC431.
- 5-Substituted *N,N*-dialkyl(cycloalkyl)furfurylamines and their quaternary salts, 2-substituted 1,3-dioxolanes, oxathiolanes, and -dithiolanes, as antagonists and muscarinic receptors: 98F1.
- Synthesis and antiproliferative activity of furocoumarin isomers (pyrrolo-, thieno-, oxazolo-, and triazolocoumarins, 8- and 4- azapsoralenes): 95F479.
- Synthesis and biological activity of glycosidopyrrole analogs of gancyclovir: 97F281.
- Synthesis, pharmacology, and molecular modeling of 2-pyridone derivatives as inotropic agents: 97F331.

Synthesis of boron heterocycles for neutron capture therapy: 94YGK1044.
Synthesis of imidazo[1,2-*b*]pyridazines and their interaction with central and peripheral-type (mitochondrial) benzodiazepine receptors: 98JHC1205.
Synthetic, computer, spectroscopic, and biological studies on new chemotherapeutic O-heterocycles: 98PAC539.
Synthetic study of 4-thiazoline derivative curacin A, a novel antimitotic agent isolated from cyanobacterium *Lyngbya majuscula*: 99YGK552.
Technetium chelates as potential small molecule radiopharmaceuticals: 99CRV2205.
Therapeutic potential of cidofovir, (S)-1-[3-hydroxy-2-(phosphonomethoxy)propyl]cytosine (HPMPC) for the treatment of DNA virus infections: 98CCC480.
Thiophene, 2,2-bithiophene, and 2,2',5',2''-terthiophene derivatives from Chinese medicinal plants as oncogene signal transduction inhibitors (protein kinase C inhibitors): 99PAC1101.
Toxicity of pharmaceutically important sesquiterpenic lactones and pyrrolizidine alkaloids from *Asteraceae* sp.: 99CLY320.

6. Pesticides

Development of paddy field rice herbicide "Thenylchlor," *N*-(2,6-dimethylphenyl)-*N*-(3-methoxy-2-thenyl)chloroacetamide: 98YGK221.
Enantioselective synthesis of bioactive O-heterocycles related to plant protection and physiology: 98YGK884.
Polymeric fungicides including heterocyclic fragments: 99MI39.
Pyrrole, pyridine, pyrazole, and imidazole derivatives as insecticides and acaricides: 99UK773.

7. Miscellaneous

a. General.

Analysis of alkaloids, barbiturates, and other drugs and poisons in forensic science: 97AC123R.
Clinical chemistry: 97AC165R.
Environmental analysis: 97AC251R.
Microanalysis of tryptophan metabolites: 97YZ657.
Pharmaceuticals and related drugs: 97AC145R.

b. Enzymes, Coenzymes, and Their Models.

Azacrown ethers as enzyme mimics of ATPase: 99MI17.
Biomimetic oxidation and asymmetric reduction with coenzyme NAD analogs: 99YGK512.

Chemistry of glycosylases and endonucleases involved in base-excision repair: 98CRV1221.

Design, synthesis and evaluation of azapeptides, oxapeptides and related heterocycles as inhibitors of D,D-peptidase and β -lactamase: 95F455.

Development of (Z,E)-5-(2-methyl-3-phenyl-2-propenylidene)4-oxo-2-thioxothiazolidine-3-acetic acid (Epalrestat, Kinedak), an aldose reductase inhibitor: 97YGK651.

2,5-Disubstituted 4-dimethylaminopyridines as artificial inhibitors of transcription factors involved in HIV replication: 97YGK697.

Effect of alteration of heterocyclic nucleus of indolactam V on its isoform selectivity for protein kinase C: 95F425.

Enantiomeric recognition of amino compounds by chiral macrocyclic receptors including pyridine or triazole subunits: 97CRV3313.

Energy, life, and adenosine triphosphate (Nobel lecture): 98AG(E)2296.

Enzymology and molecular biology of aflatoxin biosynthesis: 97CRV2537.

Formation of artificial receptors by interaction of metal with macroheterocycles as templates: 97CRV1669.

Heme-containing oxygenases: 96CRV2841.

Heme/copper terminal oxidases: 96CRV2889.

N-Heterocycles as antagonists of tachikinin receptors: 97MI18.

Heterocycles as enzyme inhibitors: 98AG(E)688.

N-Heterocycles as ligands in dioxygen activation by enzymes containing binuclear nonheme iron clusters: 96CRV2625.

N-Heterocycles as ligands in dioxygen activation by enzymes with mononuclear nonheme iron-containing active sites: 96CRV2607.

N-Heterocycles as receptor models based on molecular recognition at membrane surfaces: 97YGK436.

P,O-Heterocycles with P-atom hexacoordinated owing to donor interaction, participation in enzymatic reactions: 98ACR535.

Heterocyclic templates and nonpolyglutamatable inhibitors of thymidylate synthase as potential antitumor agents: 99JHC827.

Human ribonucleases: 98MI49.

Inhibition of configuration-retaining glycosidases by lactones, piperidines, and tetrazoles: 99AG(E)750.

Ligands of the histamine H_3 -receptors, new potent antagonists of the 2-thioimidazole type: 97F295.

Macroheterocycles as synthetic receptors for steroids in molecular recognition: 97CRV1567.

N-Macroheterocycles and derivatives of pyrrolidine and piperidine as inhibitors of immunophilin enzymes: 98JMC5119.

Mechanism of catalysis and the inhibition of β -lactamases: 98CC1609.

Mechanism of heme oxygenase action: 98ACR543.

Metal chemistry relevant to the multinuclear molybdenum and tungsten pterine enzymes: 97CC1251.

Molecular bases for interactions between β -lactam antibiotics and β -lactamases: 97ACR162.

Mononuclear molybdenum pterine-based enzymes: 96CRV2757.

Natural and semisynthetic monocomponent flavocytochromes as catalytically self-sufficient monooxygenases: 97MI19.

Pterin-dependent amino acid hydroxylases: 96CRV2659.

Role of Na^+/K^+ -ATPase as energy convertor (Nobel lecture): 98AG(E)2320.

Role of polyketide synthases in biosynthesis of some heterocycles, in particular macrolides: 97CRV2465.

Structural basis of protein kinase C activation by heterocyclic tumor promoters: 98ACR163.

Structures and functions of ribonucleases: 97YZ561.

Studies on the oxygen activation mechanisms by new heme enzymes using hemoprotein mutants and synthetic heme models: 96YGK1046.

Syntheses on the basis of 4- and 3-hydroxyprolines using regio- and stereospecific proline hydroxylases: 99YGK523.

Synthesis and reactions of NADH and NADPH model compounds with interconversion of central and axial chirality: 97YGK132.

Synthesis of adenosine triphosphate by means of cyclic catalysis (Nobel lecture): 98AG(E)2308.

c. Amino Acids and Peptides.

Biological function and chemistry of endothelin, vasoactive peptide with macrocyclic fragments formed by disulfide bridges between cysteine residues: 99CCC1211.

Biomimetic syntheses of isodithyrosine natural products, and an approach to chemistry and molecular recognition of secoaglucovancomycin and related oligopeptides: 97YGK1029.

Chemical synthesis of natural cyclic peptides: 97CRV2243.

Chemistry of photoproteins (modified proteins with luminescent covalent-linked heterocyclic fragments) as interface between bioactive molecules and protein function: 98PAC2085.

Chemistry, structure–activity relationships, and prospects of semisynthetic glycopeptides (including cyclic ones): 97F313.

Combinatorial synthesis of cyclic peptides: 97CRV411.

Conformationally constrained peptides with Phe, Tyr, Trp, and His units annelated with heterocycles: 99T585.

Design and synthesis of conformationally fixed amino acids, particularly those of heterocyclic series, as peptidomimetics: 97T12789.

Formation of functionalized cyclic nitrones via a new reaction intermediate, nitrosoketene, and its development for the synthesis of nonproteinogenic amino acids: 99YGK116.

Formation of helical-like peptides stabilized due to heterocyclic bridges formed between coils by natural and artificial amino acids: 99T11711.

Heteroarylalanines: 97JHC1067.

Introduction of heme residues and different artificial receptors in protein molecules in chemical modification of structures and functions of proteins by the cofactor reconstruction method: 99EJO539.

Investigation of immune recognition segments in presynapsis and postsynapsis neurotoxins (macrocyclic peptides): 98KPS22.

Medicinal chemistry of fulleroproline and its N-substituted derivatives: 99CC663.

Molecular identification of cytokinin-specific binding protein: 99YZ612.

Regulator activity of the simplest proline-containing peptides PG, GP, PGP, and GPGG, and possible sources of their biosynthesis: 98MI10.

Structural determination of macrocyclic peptides cyclosporins by mass spectrometry: 97CLY2.

Structure of insulin: 98CLY294.

Studies on the synthesis of vancomycin and related cyclic peptides: 98PAC391.

Synthesis, transformations, and structural investigation of derivatives of dehydroamino acids containing heterocyclic ring as a substituent: 97CLY-610.

d. *Plant Metabolites.*

Bioactive sesquiterpenic γ -lactones and flavonoids in plants of *Inula* L. genus: 97MI20.

Biologically active terpenoids, phenols, O-macroheterocycles, N-heterocycles, γ - and δ -lactones of Japanese inedible mushrooms: 98H(47)1067.

Chemical synthesis of vitanolides, steroid δ -lactones: 97KPS178.

Chemistry and biological activity of artemisinin (sesquiterpene γ -lactone with oxepane fragment and transannular peroxide bridge) and related antimalarials: 99H(51)1681.

Chemistry and biosynthesis of isoprenylated flavonoids from moraceous plant: 99PAC1116.

Chemistry of anti HIV-1 active *Calophyllum* coumarins: 98YGK116.

Chemistry of taxol, anticancer diterpenoid with oxethane cycle as a part of fused system: 98PAC331.

Chromatographic analysis of some mycotoxins, in particular O-heterocycles: 97KFZ(7)49.

Content and composition of essential oils from *Nepeta* L. species, most of them containing diastereomeric nepetalactones, unsaturated δ -lactones with annelated cyclopentane ring: 98KPS84.

Flavonolignans from *Silybium marianum*: 96CLY859.

Furosclerodanes (diterpenoids including furan or γ -butyrolactone cycle) from *Teucrium* genus: 98H(48)2185.

Glycosylated macrocyclic lactones from resins of Ipomoea family plants: 97MI23.

Heterocyclic compounds found in bryophytes: 97H(46)795.

Isoprenoid-substituted flavonoids from *Artocarpus* (Moraceae) plants: 98H(47)1179.

Limonoids (tetranorterprenoids with furan fragments) from *Melia toosendan* (Meliaceae) and their antifeedant activity: 99H(50)595.

Plant coumarins, structure and properties: 98KPS560.

Recent works on antitumor lactones from Annonaceae plants in China: 99PAC1119.

Structure and properties of plant coumarins: 98KPS250, 98KPS384.

Synthesis of Annonaceae acetogenins with long aliphatic chain including tetrahydrofuran fragments: 97YGK877.

Synthesis of germacrane sesquiterpenoid lactones and related compounds: 99T2115.

Syntheses of natural antifungal bis- γ -lactone avenaciolide and related bis-lactones: 98OPP328.

Synthesis of paclitaxel and resiniferatoxin analogs, diterpenoids with oxetane fragments: 97G461.

Synthesis of polyoxysteroids of *Achlya* (steroid γ - and δ -lactones): 98KPS3.

Synthetic studies on limonoids: 98YGK1014.

Synthetic studies on sesquiterpenoids from D-glucose, total syntheses of (+)-eremantholide A and (–)-verrucarol: 98YGK1026.

Tandem cyclization–cycloaddition of diazoketones in the synthesis of sesquiterpenes, possessing 7-oxabicyclo [2.2.1]heptane fragment: 97F303.

Terpenoids with heterocyclic fragments as bioactive taxoids from Japanese yew *Taxus cuspidata* and taxol biosynthesis: 98H(47)1111.

Total synthesis of fumitremorgins and verruculogens, tremorgenic mycotoxins—pentacyclic systems with β -carboline fragments: 97H(46)673.

e. Heterocycles Produced by Marine Organisms.

Amfidinolides (unique macrolides from marine dinoflagellates): 97H(44)543.

Antitumor N-, N,S-, and S-heterocycles and macrocyclic lactams from ascidians: 97MI22.

Biogenesis of manzamine alkaloids (β -carboline marine alkaloids): 97YGK1114.

Biological phenomena displayed by marine microalgae and related bioactive saturated O-heterocycles: 98Y GK651.

Chemical realization of the biogenetic pathways proposed for fused polycyclic ethers of marine origin: 99H(50)561.

Complete structure of maitotoxin molecule, which includes 32 6–8-member O-heterocycles as parts of fused systems linked by C–C bonds and 2–4-member carbon chains: 98PAC339.

Heterocyclic metabolites of Luthistid sponges and from microorganisms: 98AG(E)2162.

New bioactive O- and N-heterocycles from Chinese marine organisms: 99PAC1147.

Pteridine, pyrimidine, pyrrole, and imidazole derivatives as natural compounds of marine origin influencing larval settlements and metamorphosis of marine sessile organisms: 99YZ457.

Review of oxygenated 2,11-cyclized marine cembranoids, tri- and tetracyclic bioactive O-heterocycles with cembrane skeleton: 98H(49)531.

Synthetic studies on biological activities of marine-origin polycyclic bis-steroid systems with O-heterocyclic and pyrazine fragments or spirooxazoline fragment: 99PAC1095.

Synthetic study on marine polycyclic ethers and total synthesis of hemibrevetoxin B: 98Y GK940.

Total synthesis of brevetoxin B, a fused polycyclic marine-derived saturated O-heterocycle: 97Y GK686.

Total synthesis of taxoids, diterpenoids with O-heterocyclic fragments: 97H(46)727.

f. Cyclodextrins.

Applications of computational chemistry to the study of cyclodextrins: 98CRV1829.

Biomimetic reactions catalyzed by cyclodextrins and their derivatives: 98CRV1997.

Complexation of cyclodextrins: 97CC141, 98CRV1875.

Cyclodextrin-based catenanes and rotaxanes: 98CRV1959.

Cyclodextrin drug carrier systems: 98CRV2045.

Cyclodextrins as stereodifferentiating agents: 98CRV1803.

Industrial applications of cyclodextrins: 98CRV2035.

Introduction and general overview of cyclodextrin chemistry: 98CRV1743.

Methods for selective modifications of cyclodextrins: 98CRV1977.

NMR studies of cyclodextrin and cyclodextrin complexes: 98CRV1755.

Organic reactions mediated by cyclodextrins: 98CRV2013.

Structures of the common cyclodextrins and their larger analogs: 98CRV1785.

Synthetic cyclic oligosaccharides: 98CRV1919.

g. *Other Topics.*

Amides with N-heterocyclic fragments as peptidomimetic growth hormone secretagogues: 98JMC3103.

Biomimetic synthesis of natural nonfused polycyclic ethers by metal oxide-induced *syn*-oxidative polycyclizations of hydropolyenes: 98PAC355.

Chemical synthesis of cytochrome P-450-dependent metabolites (epoxyeicosatriene acids and other metabolites possessing heterocyclic fragments): 98MI9.

Complete structure of maitotoxin, polycyclic saturated O-heterocycle: 97Y GK535.

5,8-Endoperoxides of sterols: 98KPS599.

Heterocycles as commercial synthetic nonnutritive substances with sweet taste or intermediates in their synthesis: 98AG(E)1802.

Heterocycles as exogenic NO donors and inhibitors of its formation: 97F339, 97F627, 97UK792.

Natural cyclic bisulfides, disulfides, and polysulfides: 97UK901.

Natural peroxides and pharmacological preparations possessing oxirane fragment: 99MI3.

Nonplanar porphyrins and their significance in proteins: 98CSR31.

Occurrence and sensorial properties of alkoxy pyrazines contained in foodstuffs and wines: 98CLY402.

Pathways and biocatalysts of bacterial degradation quinolines: 98AG(E)577.

Polychlorinated dibenzo-*p*-dioxins and polychlorinated dibenzofurans in marine products and estimation of exposure through fishes and shellfishes: 97YZ850.

Stereocontrolled synthesis of multifunctional bioactive 7–10-member O-heterocycles: 97Y GK44.

Steroids with O-heterocyclic fragments (pyrans and O-macroheterocycles) as bioactive metabolites from echinoderms and porifera: 97G771.

Structure–odor relationships: 96CRV3201.

Synthesis, stereochemistry, and bioactivity of heterocyclic pheromones: 97CC1153, 98CCC899, 98EJO1479.

Thin-layer chromatography of antibiotics, drugs, alkaloids, purines, pyrimidines, nucleic acids, toxins, and vitamins: 98AC7R.

III. Three-Membered Rings

A. GENERAL TOPICS

Asymmetric epoxidation and aziridination of ylides: 97CRV2341.

Catalytic asymmetric epoxidation and aziridination mediated by sulfur ylides: 98SL329.

Enantioselective synthesis and transformations of oxirane and aziridine derivatives: 99PAC423.
Homochiral thiiranium and aziridinium ion intermediates formed by Lewis acid-induced rearrangement of 1-hetero-2, 3-epoxides: 97SL11.
Oxiranyl anions and aziridinyll anions: 96CRV3303.
Reactivity of three-member heteryladamantanes: 97ZOR1447.
Stereoselective synthesis of epoxides and aziridines via ylide routes: 99PAC369.

B. ONE HETEROATOM

1. *One Nitrogen Atom*

Aza-Payne rearrangement of optically active *N*-aryl(alkyl)sulfonyl-substituted 2-aziridinemethanols to corresponding epoxysulfonamides: 98CSR145.
Aziridines in the synthesis of thiagazole, *Polyangium* sp. metabolite, containing oxazoline and thiazoline fragments: 97SL1.
Reactive intermediates from *N*-aziridinyllimines: 98EJO201.
Ring opening of *N*-(4-oxoquinazolin-3-yl)aziridines: 99T1519.
Synthesis and chemistry of substituted 1-azabicyclo[1.1.0]butanes: 97SL1029.
Synthesis of aziridines via stereoselective reactions with imines: 99PAC1033.

2. *One Oxygen Atom*

Steric strain and reactivity of 3-oxatricyclo[3.2.1.0^{2,4}]octanes (epoxynorbornanes): 99ZOR661.
Synthesis and properties of 1,2-epoxyalkylphosphonates: 99S207.

a. *Reactivity of Oxiranes.*

Chemoselective reduction of α,β -epoxy carbonyl compounds to aldols and their analogs by organoseleniums and its application to natural product synthesis: 98YKG736.
Development of the technology of ethylene and propylene oxide processing: 98MI43.
Direct metallation of oxirane cycle leading to carbenoid species: 98SL337.
Epoxide-hydrolases as asymmetric catalysts for ring opening of oxiranes: 97T15617.
Phosphine in reactions with oxiranes: 99UK240.
Polymerization of terpene epoxides over solid catalysts: 97UK376.
Reactions of oxiranyl anions as nucleophilic epoxides: 97YKG176.
Reductive ring opening of epoxides in radical reactions in presence of titanocenes as electron transfer catalysts: 98SL801.

Ring opening of epoxides and some other transformations of heterocycles on aluminum oxide: 97T7999.

Spiroepoxycyclohexa-2,4-dienones in organic synthesis: 99ACR324.

b. *Synthesis of Oxiranes.*

Asymmetric catalytic epoxidation of allylic alcohols: 93MI2.

Asymmetric catalytic epoxidation of nonfunctionalized olefins: 93MI3, 98MI1.

Aza-Payne rearrangement of optically active *N*-aryl(alkyl)sulfonyl-substituted 2-aziridinemethanols to corresponding epoxysulfonamides: 98CSR145.

Directing influence of functional groups and geometry of reactant molecules on the peroxide epoxidation of alkenes: 99UK206.

Hydroxy group directivity in the epoxidation of chiral allylic alcohols: 99ACR703.

Natural peroxides and pharmacological preparations possessing an oxirane fragment: 99MI3.

Oxiranes, synthesis, bioactivity, and mechanism of peroxide epoxidation of alkenes: 99MI3.

Stereochemical aspects of epoxidation of substituted norbornenes and accompanying intramolecular transformations: 98UK299.

3. *One Sulfur Atom*

Phosphine in reactions with thiiranes: 99UK240.

Recent developments in Ramberg–Backlund rearrangement and episulfone chemistry: 99CC217.

Thiiranium and thiirenium ions as reaction intermediates and building blocks in organic synthesis: 97G177.

C. TWO HETEROATOMS

1. *Two Oxygen Atoms*

Chemistry and chemiluminescence of dioxiranes: 99MI2.

Development of chiral, nonracemic dioxiranes for the catalytic enantioselective epoxidation of alkenes: 99SL847.

2. *Two Sulfur Atoms*

Synthesis and properties of dithiiranes: 97YGK897.

IV. Four-Membered Rings

A. GENERAL TOPICS

Reactivity of four-member heteryladamantanes: 97ZOR1447.
Silylketenes in formation of β -lactones and β -lactams: 98JCS(P1)2105.
Syntheses of β -lactams, β -lactones, and 1,3- and 1,4-diazetidinediones by photochemically induced cycloaddition reactions of chromium carbene complexes with imines, aldehydes, and azo compounds: 97T4105.

B. ONE HETEROATOM

1. *One Nitrogen Atom*

Asymmetric syntheses of β -lactams by Staudinger ketene–imine cycloaddition reaction: 98KGS1448, 99EJO3223.
The azomethine ylide strategy for β -lactam synthesis: 99JHC1365.

2. *One Oxygen Atom*

Methods for the synthesis of optically active β -lactones (2-oxetanones): 99T6403.
Paterno–Büchi reaction in preparation and transformations of 3-oxetanols and 3-aminooxetanes: 97LA1627.
Preparation of oxetanes by [2+2]-photocycloaddition reactions of ketones and dienes: 98S683.

V. Five-Membered Rings

A. GENERAL TOPICS

The concept of transient chirality in stereoselective synthesis of five-membered heterocycles using the retro-Diels–Alder methodology: 99CRV1163.
Five-member heteryladamantanes: 99ZOR183.
Formation of five-member N,S- and N-heterocycles by sigmatropic rearrangements of functionalized allenes: 97LA2005.
Fragmentation of five-membered rings: 99AHC(72)361.
Generation and reactions of carbonyl ylides, nonstabilized 1,3-dipolar reagents: 98YKG681.

Heterocyclic [3,2-*b*]-fused pentalenes and their benzoannelated derivatives: 97CLY547.

Heteropentalenes with annelated imidazole or 1,2,4-triazole ring and one bridge nitrogen atom: 98AHC(69)271.

Highly stereoselective introduction of N-containing functions to carbon skeletons via cycloaddition with formation of 5-member N,O-heterocycles: 96Y GK836.

Hydroxymethylation and alkylation of furans, thiophenes, and pyrroles in the presence of H⁺ cations: 98KGS3.

The origin of stereoselection in 1,3-dipolar cycloadditions to chiral alkenes: 97G167.

Radical reactions of Hoffmann–Löffler–Freitag type with participation of iodine(III) compounds leading to five-member N-heterocycles: 97Y GK90.

Stable heterocyclic five-member azomethine imines, azolium *N*-imides, triazolium and pyrazolidinium ylides: 98H(49)587.

Substituent effect on the ¹⁵N NMR parameters of azoles: 97MRC35.

Synthesis of amino derivatives of five-membered heterocycles by Thorpe–Ziegler cyclization: 99AHC(72)79.

Synthesis of pyrrole, pyrazole, and thiazole derivatives using conjugated azoalkenes and related compounds: 97SL1128.

Unsaturated germanates and stannates in the synthesis of N-heterocycles by [2+3] cycloaddition: 98KGS1155.

B. ONE HETEROATOM

1. General

Activation of pyrrole, thiophene, and furan molecules with pentaammineosmium(II): 97CRV1953.

Bipyrroles, furyl- and thienylpyrroles: 99UK506.

Chemistry of conjugated heterocycles built from furan pyrrole, or thiophene ring fused with bicyclic (norbornadiene, bornene, or azanorbornene) skeletons: 98Y GK192.

3,4-Dimethylenefuran-, 3,4-dimethylenethiophene- and 3,4-dimethylenepyrrolediyl radicals as non-Kekule molecules with tunable singlet–triplet energy spacings: 97ACR238.

1,3-Dipolar cycloadditions of five-member cyclic nitrones to α,β -unsaturated acid derivatives: 99H(50)1213.

Fischer-type carbene complexes in the synthesis of furan, pyrrole, 5*H*-furanone, and 5*H*-pyrrolone derivatives: 98Y GK413.

Regiospecific synthesis of 3,4-disubstituted furans and thiophenes: 97LA459.

Remote asymmetric induction using chiral sulfoxides, derivatives of furan, thiophene, and pyrrole: 98YGK798.

Stoichiometric closure of furan and pyrrole cycles on McMurry reaction induced by low-valent transition metals: 98PAC1071.

Toward a unitary description of the photochemical isomerization of furan, thiophene, pyrrole, isoxazole, imidazole, and pyrazole derivatives: 99H(50)1115.

2. One Nitrogen Atom

Synthesis of *N*-arylpyrroles and *N*-arylpyrrolidines via carbon-heteroatom bond-forming reductive elimination: 98ACR852.

a. Monocyclic Pyrroles.

Development of classical and modern methods for the synthesis of arylpyrroles: 98ZOR967, 98ZOR1767.

Reactions of metal-stabilized carbenoids with pyrroles: 95MI2.

b. Hydropyrroles.

N-Acyliminium precursors in tetracarbonyliron-mediated stereoselective alkylations of 5-(*R*)-isopropoxy-3-pyrrolin-3-ones: 98EJO1729.

Chiral *N,N*-disubstituted 2-(aminomethyl)pyrrolidines as catalysts for asymmetric acylation of alcohols: 99YGK598.

Design of chiral catalysis and asymmetric autocatalysis for diphenyl-(1-methylpyrrolidin-2-yl) methanol-catalyzed enantioselective additions of organozinc reagents: 97YGK994.

Diastereoselective synthesis of pyrrolidine derivatives using chiral and non-racemic *N*-cyanomethyloxazolidines: 99CSR383.

Dirhodium tetra(*N*-arylsulfonylprolinates) as chiral catalysts for asymmetric transformations of vinyl and aryldiazoacetates: 99EJO2459.

Reactions of iminyl and related radicals in formation of pyrrolines and fused pyrrolines: 96SL1148.

Ring-opening reactions of succinimides: 98H(48)2677.

Synthesis and photophysical properties of electro- and photoactive fulleropyrrolidines: 97G779.

c. Porphyrins and Related Systems.

Binding of organic nitroso compounds to metalloporphyrins: 99ACR529.

Design and applications of chiral porphyrins: 98YGK201.

Expanded porphyrins and their heterologs: 97CRV2267.

Fullerenes linked with one or two porphyrin residues as novel acceptors in photosynthetic electron transfer: 99EJO2445.

- Functionalization of porphyrins via Wittig reactions using intermediates with $\text{CH}_2\text{PPh}_3\text{Cl}$ groups as building blocks: 98SL1297.
- Functionalized calix[4]pyrroles ([1,1,1,1]-2,5-pyrrolophanes): 98PAC2401.
- Macrocyclic effect and specific character of complex formation with porphyrins: 97MI8.
- Mechanism of chlorophyll breakdown: 99ACR35.
- Metalloporphyrins and their analogs as photo- and radiosensitizers: 99CRV2379.
- Multifunctional and chiral porphyrins as model receptors for chiral recognition: 98ACR81.
- "Oxo-hydroxo tautomerism" as a useful tool in oxygenation reactions catalyzed by water-soluble metalloporphyrins: 98CC2167.
- Peculiarities of electrophilic substitution in *meso*-tetraazaporphyrins: 98ZOR647.
- Photoinduced electron transfer reactions in supramolecular model systems based on metalloporphyrins: 97YGK557.
- Polyhaloporphyrins as unusual ligands for metals and metal-catalyzed oxidations: 97ACR251.
- Porphyrin-based diboronic acids as artificial receptors for saccharides: 98YGK831.
- Porphyrin-based synthetic receptors for ubiquinone and cytochrome C: 98YGK745.
- Porphyrin derivatives as models in bacterial photosynthesis imitation: 98PAC2189.
- Porphyrinic co-complexes as novel multinuclear catalysts for the reduction of dioxygen directly to water: 97ACR437.
- Porphyrinogen-porphyrin relationship: the discovery of artificial porphyrins: 96CC1257.
- Progress in investigation of ruthenium porphyrin complexes: 97MI10.
- Progress in porphyrin chemistry: 97MI43.
- Quantum chemical studies of porphyrins: 98ACR189.
- Recent progress in multiporphyrin synthesis: 99YGK749.
- Reinvestigation of iron porphyrins by Mossbauer spectroscopy using synchrotron radiation: 98PAC917.
- Ruthenium oxo- and tosylimidoporphyrin complexes for epoxidation and aziridination of alkenes: 99PAC281.
- Semicorrins as ligands in asymmetric catalysis: 99SL837.
- Solvation effects and coordination properties of porphyrins and metalloporphyrins in solutions: 98MI19.
- Synthesis and physical properties of hemiporphyrazines as targets for the preparation of molecular materials: 98CRV563.
- Synthesis and properties of (dimethylaminomethyl)porphyrins: 97KGS1299.

Synthesis, characterization, and chemistry of core-modified porphyrins and their nickel complexes: 97NJC691.

Synthesis of chlorins, bacteriochlorins, isobacteriochlorins, and higher reduced porphyrins: 98PHC1.

Synthesis of porphyrins with exocyclic rings from cycloalkenopyrroles: 95MI3.

Synthetic and structural chemistry of heme derivatives with nitrogen oxide ligands: 99ACR350.

Synthetic models of hemoglobin and myoglobin: 99ACR455.

d. *Indoles, Carbazoles, Related Systems, and Hydrogenated Derivatives.*

Chemistry and biological properties of polyfunctional indole-3-carbinol derivatives [1-(indol-3-yl)glycerols and related compounds, β -hydroxytryptamines, and ascorbigens]: 95F369.

Cycloaddition reactions of indole derivatives: 95MI5.

Effect of C1, C2, and C3 substituents on the conformational equilibrium of indolo[2,3-*a*]quinolizidines: 99H(51)2227.

Fulleropyrrolidines: 98ACR519.

Heterocyclization of indolin-2-one derivatives: 97KFZ(12)20.

1-Hydroxyindoles: 99H(50)1157.

Indoledione-indole rearrangement: 98KGS1170.

New reactions of indole nucleus and their synthetic applications: 99YZ35.

Nucleophilic substitution of C-hydrogen in five-member ring of indoles: 99PHC45.

Pd-catalyzed reactions of indole coupling: 95MI4.

Synthesis of [*b*]-annelated indoles by thermal electrocyclic reactions: 95MI7.

Transition metal-mediated syntheses of carbazole derivatives: 95MI6.

Vilsmeier-Haack and Friedel-Crafts reactions, bromination, debromination, debenzoylation in indole series and their synthetic application: 99YZ35.

e. *Isoindoles (Including Phthalocyanins and Porphyrazines).*

Chiral N,N-disubstituted 1-(aminomethyl)isoindolines as catalysts for asymmetric acylation of alcohols: 99YGK598.

Functional supramolecular materials, formation by self-assembly of phthalocyanins and porphyrazines: 96CC2385.

Macrocyclic effect and specific character of complex formation with phthalocyanins: 97MI8.

Metallophthalocyanins as photo- and radiosensitizers: 99CRV2379.

Novel aerobic oxidation method using *N*-hydroxyphthalimide as a catalyst (transformations of hydrocarbons to alcohols and/or carbonyl compounds): 99YGK24.

Oxidation of pollutants catalyzed by metallophthalocyanins: 97ACR470.
Recent developments in the use of *N*-phthaloyl-amino acid derivatives in synthesis: 98SL457.
Star porphyrazines and related multimetal macrocycles: 98JHC1013.

f. Polycyclic Systems Including Two Heterocycles.

Novel pyrroloimidazoles: 95F401.
Pyrrolizidine and indolizidine syntheses involving 1,3-dipolar cycloadditions: 99S905.
Pyrrolo[2,1-*a*]isoquinolines: 97KGS291.
Synthetic, structural, reactive, and biological aspects of novel tetraazabicyclooctanes: 95F379.

3. One Oxygen Atom

a. Furans.

Furan derivatives of group VII elements: 97KGS151.
Furan derivatives of group VIII elements: 97KGS154.
Furyl(aryl)methanes and their analogs: 99KGS867.
Reductive amination of furan derivatives: 99UK61.
Regioselective synthesis of substituted furans: 98T1955.
Regiospecific synthesis of 3,4-disubstituted furans: 97LA459.
Synthesis of multisubstituted furan rings using silyl protection: 99CSR209.
Synthetic applications of furan Diels–Alder chemistry: 97T14179.
Transformation of furans to *N*-heterocycles by aza-Achmatovicz reaction: 98SL105.
Use of furans in natural product synthesis: 99PAC1041.

b. Hydrofurans.

Acetylene-based functionalized dihydrofuranones and related biomimetic assemblies: 99H(51)2485.
2,3-Dihydrofurans in synthesis of heterocyclic compounds: 97UK151.
Hydrofurans in syntheses of heterocycles: 97KGS723.
Reductive amination of tetrahydrofuran derivatives: 99UK61.
Synthesis of annelated furanoses and their transformation to carbocycles: 98SL693.

c. Annelated Furans.

Derivatives of 7-oxabicyclo[2.2.1]heptane in nature and as useful synthetic intermediates: 99T13521.
Diels–Alder reactions of benzo[*b*]furan-4,5-diones and benzo[*b*]furan-4,7-diones: 99H(50)1137.

Methods for the synthesis of benzofuran derivatives: 97KGS1444.
Recent advances in the chemistry of benzo[*c*]furans and related compounds: 99AHC(73)1.
Synthesis and properties of furopyridines: 97H(45)975.
Synthesis of annelated furanoses and their use for preparation of carbocycles: 98SL693.
Thermal [3,3] Claisen rearrangement of the 3-substituted phenyl allyl and propargyl ethers; synthesis of 4-halobenzo[*b*]furans: 98H(48)2173.
X-Ray diffraction analysis of phthaleine pigments: 97YZ764.

d. *Five-Member Lactones.*

Enantiocontrol in syntheses of γ -lactones catalyzed by metal carbenes: 99IZV16.
Pd-catalyzed intermolecular coupling reactions of allyl alkynoates with formation of bioactive γ -lactones: 98SL115.
Photochemical transformations of 2(5*H*)-furanones: 98OPP401.
Reactions of 2(3*H*)-furanones: 99AHC(73)275.
Regio- and stereoselective synthesis of γ -alkylidenebutenolides and related compounds: 97T6707.
Synthesis of heterocyclospirobutanolides: 98MI66.
Synthesis of γ -lactones using tungsten alkynyl and propargyl compounds: 98PAC1111.

4. *One Sulfur Atom*

a. *Thiophenes.*

2-Aminothiophenes by Gewald reaction: 99JHC333.
Nucleophilic substitution on thiophene derivatives: 97G753.
Pd-catalysis in synthesis of thienylacetylenes: 99SL1704.
Progress and prospects in hydrogenation, hydrogenolysis, and desulfurization of thiophenes with soluble metal complexes: 98ACR109.
Regiospecific synthesis of 3,4-disubstituted thiophenes: 97LA459.
Thiophene, synthesis and properties: 98MI17.
Thiophene as precursor of highly conjugated organosulfur compounds formed at Rh- and Ir-mediated thiophene ring opening: 97SL643.
Thiyl radicals and their analogs in gas-phase syntheses and transformations of thiophenes: 99ZOR11.

b. *Annelated Thiophenes.*

Benzo[*b*]thiophene and dibenzo[*b,d*]thiophene as precursors to highly conjugated organosulfur compounds formed at Rh- and Ir-mediated thiophene ring opening: 97SL643.

S-(Perfluoroalkyl)dibenzothiophenium salts: 95AHC(64)323.
 Synthesis, chemistry, and biological properties of thienopyrimidines:
 96AHC(65)235, 96AHC(66)193.

c. Hydrothiophenes.

Catalytic hydrogenation of thiolene 1,1-dioxides to thiolane 1,1-dioxides:
 97UK463.

Sulfolane as a selective solvent: 98MI30.

Synthesis and properties of dihydrothiophenes: 98KGS1299.

C. TWO HETEROATOMS

1. General

Asymmetric cycloaddition of functionalized alkenes to nitrile oxides and nitrones: 98YGK11.

Asymmetric 1,3-dipolar cycloaddition reactions: 98CRV863.

1,3-Dipolar cycloaddition in the synthesis of fullerene C₆₀ derivatives containing heterocyclic fragments: 98KGS291.

Reactions of *N*-aminoazolinethiones: 97KGS1155.

2. Two Nitrogen Atoms

a. Pyrazoles.

Chemistry of transition metal complexes supported by hydrotris(pyrazolyl) borates and chemistry of dioxygen complexes based on these ligands: 99YGK619.

Chemistry, syntheses, and applications of thallium(I) hydrotris(pyrazolyl)borate [TpT1(I)]: 98MI44.

High-temperature gas-phase pyrolysis of pyrazole and indazole derivatives: 99MI27.

Metal basicity and cooperative effects in the reactions of dinuclear pyrazolato rhodium complexes: 98PAC779.

Progress in the chemistry of nitropyrazoles: 98ZOR1124.

Recent advances in complexes of group VIB metals (Mo, W) with poly-(pyrazolyl)borate ligands: 98MI2.

Study on poly(pyrazol-1-yl)alkane complexes: 99MI32.

Synthesis of pyrazoles: 98JHC489.

Synthesis of pyrazoles and fused pyrazoles: 99JHC321.

Synthesis of pyrazolines in reactions of α,β -enones with diazomethane and hydrazines: 97KGS747.

Synthesis of pyrazolo[1,5-*a*]quinolines and related reduced derivatives: 97H(45)1839.

b. *Imidazoles*.

Imidazol-2-ylidene and its *N,N'*-disubstituted derivatives as stable carbenes: 99ACR913.

Room-temperature ionic liquids, salts with *N,N*-dialkylimidazolium cations in synthesis and catalysis: 99CRV2071.

c. *Annelated Imidazoles*.

Advances in chemistry of Troeger bases (1,5-methanodibenzo[*b,f*]-1,5-diazocines): 99PHC1.

Molecular and supramolecular objects from glycoluril, 2,4,6,8-tetraazabicyclo-[3.3.0]octan-3,7-dione: 99ACR995.

Novel pyrrolo- and thiazoloimidazoles: 95F401.

Synthesis of 2,3-dihydroimidazo[1,2-*a*]pyridines and their salts: 99KGS1299.

3. *One Nitrogen Atom and One Oxygen Atom*

a. *1,2-Heterocycles*.

Application in organic synthesis of optically active isoxazolidones obtained by asymmetric cycloaddition of nitrones with allenes: 97T403.

Base-catalyzed rearrangements of 5-oxodihydroisoxazoles: 99H(51)3013.

1,4- and 1,3-Cycloreversions of 2-isoxazolines: 97UK31.

High-temperature gas-phase pyrolysis of isoxazole derivatives: 99MI27.

Isoxazole derivatives in synthesis of prostanoids: 99ZOR1749.

Steroidal isoxazoles, isoxazolines, and isoxazolidines: 98JHC731.

Syntheses of isoxazolinyl and isoxazolidinyl nucleoside analogs: 98T6587.

b. *1,3-Heterocycles*.

Annelated oxazolidines and oxazolines on the basis of *cis*-1-aminoindan-2-ol: 98S937.

Asymmetric bias generated by protected vicinal diol controller and its application to asymmetric nitron-olefin cycloaddition reactions: 98YGK86.

Chiral and nonracemic *N*-cyanomethyloxazolidines in diastereoselective synthesis, particularly of pyrrolidine and piperidine derivatives: 99CSR383.

Chiral Cu(II)-complexes of bis-oxazolines as Lewis acids for catalyzed cycloaddition, carbonyl addition, and conjugate addition reactions: 99PAC1407.

Chiral 4,5-disubstituted oxazolidin-2-ones in stereoselective synthesis of β -hydroxy- α -amino acids: 97G475.

Chiral oxazolines in asymmetric synthesis: 98JHC991.

4,5-Disubstituted 2-oxazolidines as chiral auxiliaries; synthesis from 2-oxazolones: 97YZ339.

Enantioselective free-radical reactions of oxazolone-2-ones: 99ACR163.

Oxazol-5(4*H*)-ones as intermediates in the formation of macrolides, cyclodepsipeptides, and cyclopeptides: 99JHC1539.

Photolysis, luminiscent properties, and laser activity of oxazole derivatives: 97MI27.

Reactions of 2-oxazolines and their utilization: 98CLY475.

Synthesis and properties of 2-oxooxazolopyridines: 99KGS168.

Synthesis of oxazoles from diazocarbonyl compounds: 97PHC1.

Synthesis of oxazolines as efficient reagents in organic syntheses and monomers for macromolecular chemistry: 98CLY175.

Synthetic versatility of 2-oxazolone heterocycle for stereocontrolled construction of 2-amino alcohols: 97YGK1018.

4. *One Nitrogen Atom and One Sulfur Atom*

3-(2,6-Diisopropylphenyl)-4,5-dimethylthiazol-2-ylidene as a stable carbene: 99ACR913.

Novel thiazoloimidazoles: 95F401.

Synthesis of 2-(*N*-cyanoimino)thiazolidines and use of their 3-acyl derivatives as efficient N-, O-, and S-acylating agents: 98YGK182.

Synthesis of 2-R-substituted thiazoles and transformation of a thiazole heterocycle into aldehyde group by successive N-methylation, reduction, and subsequent hydrolysis: 98S1681.

5. *Two Oxygen Atoms*

The synthesis of 1,3-dioxolanes in combined reaction-rectification process: 98MI42.

6. *Two Sulfur Atoms*

Advances in synthesis and research of oligomeric tetrathiafulvalenes: 97MI9.

Enantioselective oxidation of 1,3-dithiolanes to corresponding S-oxides and S,S-dioxides by designer yeast: 99JHC1533.

Macromolecular tetrathiafulvalene chemistry: 98CC945.

Reactivity of tetrathiafulvalenes: 95AHC(62)249.

Synthesis of tetrathiafulvalene derivatives for the creation of new organic functional materials: 98YGK755.

Tetrathiafulvalene thiolates as building blocks for macrocyclic and supramolecular chemistry: 97SL1211.

Two- and three-dimensional tetrathiafulvalene macrocycles: 97LA2177.

D. THREE HETEROATOMS

1. *Three Nitrogen Atoms*a. *Monocyclic Systems.*

Ferrocenyltriazaoles: 97MI25.

Spin transfer in Fe(II) complexes of 1,2,4-triazaoles: 98KK403.

1,2,4-Triazole derivatives as high-energy compounds: 97MI4.

1,2,4-Triazoline-3,5-diones: 97AHC(67)119.

b. *Annelated Triazoles.*

Advances in synthetic methodology using benzotriazole: 99JHC1501.

Application of benzotriazole in synthesis and transformations of heterocycles:

98JHC1123.

Chemistry of 1,2,4-triazolo[4,3-*a*]pyrimidines: 99AHC(73)131.

Michael additions of benzotriazole-stabilized carbanions: 98CCC599.

Properties and synthetic utility of N-substituted benzotriazoles: 98CRV409.

1,2,3-Triazolo[4,5-*d*]pyridazines: 97F205.

1,2,3-Triazolo[*x,y-z*]pyrimidines: 98AHC(71)57.

1,2,4-Triazolo[*x,y-z*]pyrimidines: 99AHC(72)127.

s-Triazolo[3,4-*b*]-1,3,4-thiadiazole derivatives: 98H(48)561.

2. *Two Nitrogen Atoms and One Oxygen Atom*

Chemistry of furazanes fused with six- and seven-membered heterocycles with one heteroatom: 99UK154.

Nitro- and nitroaminofurazanes: 97MI2.

Synthesis and reactivity of amino- and nitrofuroxanes: 97MI3.

3. *Two Nitrogen Atoms and One Sulfur Atom*

s-Triazolo[3,4-*b*]-1,3,4-thiadiazole derivatives: 98H(48)561.

4. *Three Oxygen Atoms*

1-Alkoxy-2,3,7-trioxabicyclo[2.2.1]hept-5-enes as useful synthons for multi-functional compounds: 98SL17.

5. *Two Oxygen Atoms and One Sulfur Atom*

1,3,2-Dioxathiolane oxides as epoxide equivalents and versatile synthons: 98AHC(68)89.

6. *Three Sulfur Atoms*

Synthesis and reactions of benzotrithiole derivatives: 99PAC489.

E. FOUR HETEROATOMS

Chemistry of dithiadiazolium and dithiadiazolyl rings: 95AHC(62)137.

Epoxy derivatives of vinyltetrazole: 97MI6.

Ferrocenyltetrazoles: 97MI25.

High-energy tetrazoles: 97MI5.

Radiolysis of tetrazolium salts solutions: 98UK745.

Spin transfer in Fe(II) complexes of tetrazoles: 98KK403.

1,2,4-Tetrazolo[x,y-z]pyrimidines: 99AHC(72)127.

VI. Six-Membered Rings

A. GENERAL

Chemistry of furazanes fused with six-membered heterocycles with one heteroatom: 99UK154.

Conformational flexibility of six-member dihydro N-, O- and S-heterocycles: 97IZV2095.

Cyclization reactions of quaternary salts of azaaromatic compounds: 97KGS23.

Development and application of hetero Diels–Alder reactions with participation of amino acid-derived chiral acylnitroso compounds: 98T1317.

1,3-Dipolar cycloadditions of six-member cyclic nitrones to α,β -unsaturated acid derivatives: 99H(50)1213.

Formation of 7-hetera- and 7,8-diheterabicyclo[2.2.2]octa-2,5-dienes using synthetic equivalents of cyclohexatriene in [4+2] cycloaddition reactions: 97SL1327.

Formation of six-member N-heterocycles in radical reactions of Hoffmann–Löffler–Freitag type with participation of iodine(III) compounds: 97YKG90.

Metal-catalyzed asymmetric hetero Diels–Alder reactions of unactivated dienes with glyoxylates: 98PAC1117.

Polynitroazaadamantanes: 97MI24.

Preparation and use of amphiphilic oximes, derivatives of pyridine, pyridazine, pyrimidine, and pyrazine: 98CLY469.

Properties and applications of aryl-substituted azines: 97KGS1011.

Reactions of *N*-aminoazinethiones: 97KGS1155.

Syntheses and properties of allylthio-, allyloxy-, and allylamino-substituted azine derivatives: 97KGS1603.

Unusual photophysical properties of polyazaanthracenes and polyazapentacenes having low values of calculated singlet-triplet energy gap: 99PAC295.

B. ONE HETEROATOM

1. *One Nitrogen Atom*

Enthalpic increments for a group additivity scheme in thermochemistry of pyridine, quinoline, and their substitution products: 99PAC1257.

a. *Pyridines.*

Functionalization of pyridines via formation of carbon-heteroatom bond with elements of groups IV, V, and VI: 99KGS437.

Platinum(II) complexes of pyridines: 99CCC435.

Pyridinophanes: 98KGS579.

Reactions of potentially tautomeric methyl and methylene derivatives of pyridine with N-electrophiles: 98KGS147.

Reductive amination with pyridine bases: 99UK61.

Structural chemistry of pyridonate complexes of late 3*d* metals: 97ACR89.

Synthesis and properties of 3-cyanopyridine-2(1*H*)-chalcogenones: 99KGS579.

Synthesis of acylpyridines, pyridinecarboxylic acids and their derivatives: 98KGS1013.

Synthesis of 3-aminomethyl-6-chloropyridine by combining microbial and chemical reactions: 99YGK466.

Transition metal complex-mediated hydroacylation of pyridine derivatives with aldehydes: 99SL1.

b. *Pyridinium Compounds, Ylides, Pyridine N-Oxides.*

N-Fluoropyridinium salts: 95AHC(62)1.

Pyridinium ylides in nucleophilic Ad-E reactions: 97ZOR975.

Room-temperature ionic liquids, salts with *N*-alkylpyridinium cations in synthesis and catalysis: 99CRV2071.

c. *Applications of Pyridines.*

Complexing extraction of pyridine bases from coal coking products with organic solvents: 97KGS3.

Novel amine chemistry based on 4-(dimethylamino)pyridine-catalyzed acylation: 98ACR494.

Pyridines and pyridine *N*-oxides as additives in asymmetric catalysis: 99AG(E)1570.

d. *Bipyridines and Related Systems.*

Asymmetric synthesis using chiral carbenoid complexes with 2,2'-bipyridine ligands: 98YGK764.

Chemiluminescence in reactions of 9,9'-biacridinium salts: 97YZ864.

Metallodendrimers with 2,2',2''-terpyridines as ligands: 97CC1073.

Multipurpose reagents from ethynyl-grafted oligopyridines: 99S1839.

Transition metal complexes with 2,2'-bipyridine ligands in anion-selective recognition and optical/electrochemical sensing: 96CC689.

Vectorial transfer of electronic energy in rod-like ruthenium-osmium complexes with bis-2,2',2''-terpyridine ligands: 97CC333.

e. *Hydropyridines.*

Asymmetric routes to substituted piperidines: 98CC633.

Asymmetric synthesis and synthetic utility of 2,3-dihydro-4-pyridones: 99JHC1491.

Chemistry of 2,3,4,5-tetrahydropyridine (Δ' -piperidine) and its derivatives: 98UK1133.

Chemistry of 2,3,4,5-tetrahydropyridine 1-oxides: 98KGS435.

Diastereoselective synthesis, particularly of piperidine derivatives using chiral and nonracemic *N*-cyanomethyloxazolidines: 99CSR383.

Partially hydrogenated pyridinechalcogenones: 98IZV2123, 99KGS147.

Use of enzymes in preparation of enantiopure 1,4-dihydropyridines: 98H(48)1943.

f. *Biologically Active Pyridines and Hydropyridines.*

Complexes of O₂ with [$\{\text{TMPA}\}\text{Cu}^{\text{I}}(\text{RCN})\}^+$ [TMPA = tris(2-pyridylmethyl)-amine] as functional models for proteins: 98PAC855.

g. *Pyridines Annelated with Carbocycles.*

Acridines fused with heterocycles: 98AHC(70)89.

Asymmetric nucleophilic addition of dialkylzinc to 3,4-dihydroisoquinoline 1-oxides: 98YGK11.

Methylpyridines and other methylazines as precursors of bi- and polycycles: 98AHC(68)181.

Polycyclic fluoroquinolones: 99ZOR1447.

Some aspects of chemistry of hydrogenated quinolines: 98JHC761.

Synthesis and stereochemistry of perhydroacridines: 97KGS867.

Synthesis and transformations of azafluorenes: 97UK131.

Synthesis of 2,3-dihydroimidazo[1,2-*a*]pyridines and their salts: 99KGS1299.

Synthesis of 3(2)-isoquinolinones and related compounds: 96MI1.

Transition metal complex-mediated hydroacylation of quinolines with aldehydes: 99SL1.

h. Pyridines Annelated with Heterocycles.

Chemistry of benzologs of pyrido[1,2-*a*]-pyrimidines: 99AHC(73)177.

Chemistry of pyrido[1,2-*b*]-1,2-oxazines, pyrido[1,2-*b*]-1,2-thiazines, pyrido[1,2-*b*]pyridazines, and their benzologs: 98AHC(69)89, 98AHC(70)1.

Chemistry of pyrido[2,1-*b*]-1,3-oxazines, pyrido[2,1-*b*]-1,3-thiazines, and their benzologs: 99AHC(72)225.

Chemistry of pyrido[2,1-*c*]-1,4-oxazines, pyrido[2,1-*c*]-1,4-thiazines, pyrido[1,2-*a*]pyrazines, and their benzologs: 98AHC(71)145.

Effect of C1, C2, and C3 substituents on the conformational equilibrium of indolo[2,3-*a*]quinolizidines: 99H(51)2227.

Indolizidine syntheses involving 1,3-dipolar cycloadditions: 99S905.

Pyrrolo[2,1-*a*]isoquinolines: 97KGS291.

Synthesis and properties of furopyridines: 97H(45)975.

Synthesis and properties of 2-oxooxazolopyridines: 99KGS168.

Synthesis of 2,3-dihydroimidazo[1,2-*a*]pyridines and their salts: 99KGS1299.

Synthesis of pyrazolo[1,5-*a*]quinolines and related reduced derivatives: 97H(45)1839.

2. One Oxygen Atom

Conformation analysis of saturated six-member O-heterocycles: 98AHC(69)217.

a. Pyrans and Hydropyrans.

I(III) Compounds in carbohydrate-based synthesis of 2,3-dihydro-4*H*-pyranones as starting compounds for the preparation of *C*-saccharides, glycosylstananones, and analogs of tromboxane A₂: 98EJO2267.

Dienone-2*H*-pyran valence isomerization: 99KGS1443.

New developments in the chemistry of pyrans: 95AHC(62)19.

Synthesis and reactivity in aqueous medium of pyronoids, mono- or polycyclic compounds with 2- or 4-pyrone fragments: 99H(50)611.

Synthesis of δ -lactones using tungsten alkynyl and propargyl compounds: 98PAC1111.

Synthesis of α -pyrone meroterpenoids, pyropyropens: 98YGK478.

Synthesis of spiro sugars and C-nucleosides using anomeric free radicals: 98SL700.

Synthesis, structure, and transformations of α -pyrone derivatives, viz., xanthyrone, glaucyrone, and chelated magnesium enolates: 98T8243.

b. *Annelated Pyrans and Pyrylium Salts.*

¹H NMR spectroscopy in investigation of flavan-3-ols and their derivatives: 97KPS16.

¹H NMR spectroscopy of proantocyanidines: 97KPS135.

¹³C NMR spectroscopy of flavan-3-ols and proantocyanidines: 97KPS545.

Photochrome flavylium compounds as multistate/multifunction molecular-level systems: 99CC107.

Photolysis, luminescent properties, and laser activity of coumarin derivatives: 97MI27.

Role of the bite angle (DA–M–DA angle, where DA is donor atom and D is metal atom) in catalytic properties of catalysts with xanthene-based bis-phosphine ligands: 99PAC1443.

Synthesis and reactions of isoflavone analogs, 3-hetarylchromones: 99KGS3.

Synthesis of cyclopenta[*b*]pyran skeleton of natural lactones: 97T14507.

Synthesis of isocoumarins: 97OPP631.

3. *One Sulfur Atom*

Recent progress in the synthesis and reactions of isothiochromans: 98OPP243.

Thiopyran and thiopyrylium salts; synthesis and properties: 98MI18.

C. TWO HETEROATOMS

1. *Two Nitrogen Atoms*

Chemistry of conjugated heterocycles built from pyridazine or pyrazine ring fused with bicyclic (norbornadiene, bornene, or azanorbornene) skeletons: 98Y GK192.

Reactions of potentially tautomeric methyl and methylene derivatives of diazines with N-electrophiles: 98KGS147.

Synthesis of tritium-labeled biologically important diazines: 99UK254.

a. *1,2-Heterocycles.*

Asymmetric dipolar cycloaddition of azomethine imines derived from diazoalkane–pyridazine cycloadducts: 98JHC1187.

Chemical and pharmacological properties of aminopyridazines: 98JHC1091.

Chemistry and biology of pyridazinones and their tricyclic analogs: 98JHC1161.

Chemistry of pyrido[1,2-*b*]pyridazines and their benzologs: 98AHC(69)89, 98AHC(70)1.

1,3-Dipolar cycloadditions of diazoalkanes to pyridazines: 98JHC1187.

3(2*H*)-Pyridazinones in modern synthetic and medicinal chemistry: 98JHC1075.

Synthesis and biological activity of 1,5-dihydropyridazino[3,4-*b*]quinoxalines: 98JHC1101.

Synthesis and chemistry of pyridazines functionalized in positions 3 and 5 with heteroatoms: 98JHC1111.

Synthesis of polycyclic pyridazinediones as chemiluminescent compounds: 98JHC1219.

Synthesis of substituted pyridazines using novel cyclization reactions of mono- and dichloroazodienes: 99JHC301.

1,2,3-Triazolo[4,5-*d*]pyridazines: 97F205.

b. *1,3-Heterocycles: Monocyclic Pyrimidines and Hydropyrimidines (Except Pyrimidine Nucleoside Bases and Nucleosides).*

Nitrodihydropyrimidines: 97KGS1587.

Cl $\rightarrow$ R Stille replacement on interaction of 4-chloropyrimidines fixed on an amide resin with RSnBu: 99JHC1595.

c. *Annelated Pyrimidines (Except Purines, Pteridines, and Flavins).*

3-Acetoxyaminoquinazolinones as aziridination agents: 99T1519.

Chemistry of 1,2,4-triazolo[4,3-*a*]pyrimidines: 99AHC(73)131.

Chemistry of benzologs of pyrido[1,2-*a*]pyrimidines: 99AHC(73)177.

Synthesis, chemistry, and biological properties of thienopyrimidines: 96AHC(65)235, 96AHC(66)193.

Synthesis, stereochemistry, and transformations of cyclopenta-, cyclohexa-, cyclohepta-, and cycloocta-annelated pyrimidines: 98AHC(69)349.

1,2,4-Triazolo- and tetrazolo[*x,y-z*]pyrimidines: 99AHC(72)127.

1,2,3-Triazolo[*x,y-z*]pyrimidines: 98AHC(71)57.

d. *Pyrimidine Nucleoside Bases and Purines.*

Acid dissociation constants, UV and NMR spectral data for mono- and poly-N-methylated adenines: 99H(51)2255.

Chemistry of N^x, N^y, N^z -trimethyladenines and more highly N-methylated adenines: 99H(51)1141.

Chemistry, physicochemical properties, and biological activities of isomeric 9-, 7-, 3-, 1- and *N*(6)-methyladenines: 98H(48)1673.

Chemistry, physicochemical properties, and biological activities of N^x -oxygenated adenines: 99H(51)1971.

Chemistry, physicochemical properties, and biological activities of the 11 positional isomers of N^x, N^y -dimethyladenine: 99H(51)393.

Chemistry, synthesis, and biological evaluation of purine 7-*N*-oxides relative to nucleic acids: 97H(44)573.

Creating regular arrangement of nucleobases through metal ion coordination and H-bond formation: 98PAC977.

- Dimroth rearrangement in the adenine series: 98H(48)359.
- Guanine derivatives, the effects of N(7)-coordinated *cis*-diammine-platinum(II) on the acid–base properties of: 98PAC845.
- Occurrence, chemistry, synthesis and cytokinin activity of 1'-methyl-*trans*-zeatin and its analogs (glycosylated adenine derivatives): 97H(46)659.
- e. Nucleotides and Nucleosides.*
- C-Alkenylation of pyrimidine nucleosides and their analogs: 99UK532.
- Chemistry of C-nucleosides of fused heterocyclic bases: 98AHC(70)163.
- Chemistry of C-nucleosides of heteromonocyclic bases: 98AHC(68)223.
- Chemistry of 4',5'-unsaturated nucleosides: 99OPP379.
- Diheterocyclanes as synthons for the preparation of nucleoside and acyclonucleoside analogs: 97F263.
- Di-*seco*-nucleosides: 98AHC(68)1.
- Gene-directed biologically active substances (non-sense oligonucleotides and their derivatives): 97MI17.
- In vitro* selection of catalytic polynucleotides: 97CRV371.
- Molecular recognition of nucleobases and nucleotides at air–water interfaces (complementary hydrogen bonding and multisite interaction): 98ACR371.
- Mono-*seco*-nucleosides: 97AHC(67)391.
- New developments in the selective synthesis of cyclopentyl carbocyclic nucleosides: 98T9229.
- C-Nucleosides derived from simple aromatic hydrocarbons: 97SL341.
- Oxidative nucleobase modifications leading to strand scission: 98CRV1109.
- Practical synthesis of nontrivial nucleosides: 98PAC313.
- Pronucleotides as efficient tools for the delivery of biologically active nucleoside monophosphates: 98SL233.
- Role of nucleotides in nitrogenase catalysis: 97ACR260.
- Solute–solvent interactions in aqueous solutions of pyrimidine nucleic acid bases: 99PAC1286.
- Stereocontrolled synthesis of thiosugars from acyclic precursors and preparation of pseudonucleosides with thiosugar moiety: 97Y GK186.
- Synthesis and biological activity of azanucleosides: 99S1541.
- Synthesis and enzymatic stability of oligonucleotides consisting of isonucleosides: 99PAC1143.
- Synthesis and properties of azanucleosides: 98Y GK1036.
- Syntheses of isoxazoliny and isoxazolidiny nucleoside analogs: 98T6587.
- Syntheses of nucleosides labeled with stable isotopes and their application to structural biology: 99YZ299.
- Synthesis of 3'-C- and 4'-C-branched oligodeoxynucleotides and the development of locked nucleic acids (LNA): 99ACR301.

Synthesis of spiro sugars and C-nucleosides using anomeric free radicals: 98SL700.

Tri-, tetra-, and penta-*seco*-nucleosides: 98AHC(69)129.

f. *Nucleic Acids*.

A structural basis for RNA–ligand interactions: 97CRV1489.

Atomic determinants for aminoacylation of RNA minihelices and their relationship to genetic code: 99ACR368.

Chemical approach to the study of supramolecular biological structure of chromatin, nucleoproteid complex of DNA: 99UK365.

Cleavage of nucleic acids by bleomycin: 98CRV1153.

Conformational properties of DNA strands containing guanine and adenine: 98CLY530.

J-Coupling restrains in RNA structure determination: 99ACR614.

Dialdehyde-containing nucleic acids and their components, synthesis, properties, and affine modification of proteins: 99UK267.

DNA as components for molecular architecture: 97ACR357.

DNA-mediated electron transfer: 98PAC873.

Genesis of nucleic bases, nucleotides, and nucleosides; the problem of RNA origin and its role in life origin: 99T3141.

Mechanism of cleavage–transesterification of RNA: 97AG(E)432.

Mechanism of hydrolysis of DNA and RNA by lanthanide ions: 99CC1443.

Nanostructure and topology of nucleic acids: 98AG(E)3220.

Ni(II)-Xaa-Xaa-His metallopeptide-DNA/RNA interactions (Xaa = any α -amino acid residue): 99ACR827.

Nucleic acid analysis: 99AC425R.

Peptide nucleic acid, a molecule with two identities: 99ACR624.

Photocleavage of nucleic acids with participation of metal complexes with heterocyclic ligands: 98CRV1171.

Photoprocesses of RNA-bound copper complexes with macroheterocyclic ligands: 98CRV1201.

Preorganization of DNA and improving nucleic acid recognition by synthetic oligonucleotides: 97CRV1473.

Programmed material synthesis with DNA: 99CRV1849.

Recognition of DNA sequence and DNA bending and molecular design for artificial repressors: 97Y GK384.

Regulation of mRNA transcription and DNA bending by using triple helix forming oligonucleotide: 99Y GK194.

RNA-protein intermolecular recognition: 97ACR189.

Selection of nucleic acids and combinatorial chemistry: 97CRV349.

Shape-dependent catalysis for the CC-1065 and duocarmycin DNA alkylation reaction: 99ACR1043.

Site-specific photosensitized modification of nucleic acids by biradical and electrophilic reagents: 99UK1062.

Synthesis and application in molecular biology of double-helical nucleic acids with covalent-bonded chains: 98UK274.

Synthesis and properties of analogs of nucleic acids: 95MI10.

Synthesis of calicheamicin oligosaccharide dimers and their duplex DNA recognition: 97YGK600.

Synthesis of DNA-binding oligosaccharides: 97SL401.

Synthesis of DNA lesions and DNA-lesion-containing oligonucleotides for DNA-repair studies: 99S1085.

Synthesis of oxorhenium(V) and oxotechnetium(V) complexes as inhibitors of ribonucleases and for generation of catalytic antibodies: 97SL537.

Synthesis of reactive nucleic acids derivatives and their use for investigations of structure and functions of biopolymers: 98UK688.

Synthetic polynucleotides and analogs as models for DNA studies: 97G231.

Tertiary motifs in RNA structure and folding: 99AG(E)2326.

g. *1,4-Heterocycles: Pyrazines and Hydropyrazines.*

Free-radical reactions in the synthesis of diketopiperazines and other cyclic derivatives of α -aminoacids: 97CRV53.

Methods for the preparation of acetylpyrazines: 99CLY15.

Reactions and synthesis of pyrazines: 97YZ1.

Syntheses of naturally occurring pyrazines: 97YZ32.

Syntheses of piperazine derivatives by combining microbial and chemical reactions: 99YGK466.

Synthesis and biological activity of 1,5-dihydropyridazino[3,4-*b*]quinoxalines: 98JHC1101.

Synthesis of alkoxypyrazines: 98CLY622.

3-Ylidenepiperazine-2,5-diones as versatile organic substrates: 99CSR251.

h. *Annelated Pyrazines.*

Chemistry of pyrido[1,2-*a*]pyrazines and their benzologs: 98AHC(71)145.

Imidazo[1,2-*a*]pyrazines: 97UK207.

Pterins: 98CLY689.

Recent advances in selective synthesis of 6- and 7-substituted pteridines: 98H(48)1255.

2. *One Nitrogen Atom and One Oxygen Atom*

Asymmetric synthesis of α,α -disubstituted α -amino acids via an intramolecular Strecker synthesis using 1,4-oxazin-3-one derivatives as key intermediates: 97YGK982.

Chemistry of 4*H*-3,1-benzoxazin-4-ones: 99JHC563.

Chemistry of pyrido[1,2-*b*]-1,2-oxazines and their benzologs: 98AHC(69)89, 98AHC(70)1.

Chemistry of pyrido[2,1-*b*]-1,3-oxazines and their benzologs: 99AHC(72)225.

Chemistry of pyrido[2,1-*c*]-1,4-oxazines and their benzologs: 98AHC(71)145.

Methods for the synthesis of 4*H*-1,3-oxazines and their properties: 98KGS723. 1,3-Oxazinium and 3-azapyrylium salts: 95AHC(64)341.

Synthesis, stereochemistry, and transformations of cyclopenta-, cyclohexa-, cyclohepta-, and cycloocta-annelated 1,3-oxazines: 98AHC(69)349.

3. *One Nitrogen Atom and One Sulfur Atom*

Chemistry of pyrido[1,2-*b*]-1,2-thiazines and their benzologs: 98AHC(69)89, 98AHC(70)1.

Chemistry of pyrido[2,1-*b*]-1,3-thiazines and their benzologs: 99AHC(72)225.

Chemistry of pyrido[2,1-*c*]-1,4-thiazines and their benzologs: 98AHC(71)145.

Chemistry of 1,3-thiazin-4-ones and their derivatives: 96AHC(66)131.

Synthesis, stereochemistry, and transformations of cyclopenta-, cyclohexa-, cyclohepta-, and cycloocta-annelated 1,3-thiazines: 98AHC(69)349.

4. *Two Oxygen Atoms*

Chiral derivatives of 4,6-dimethyl-1,3-dioxane: 97Y GK517.

Cycloaddition reactions of *o*-benzoquinones with formation of bi- and tetracyclic systems including 2,3-dihydrobenzo[*b*]dioxine fragment: 96SL1143.

Synthesis and properties of 1,3-dioxanium salts: 99KGS291.

Thermal fragmentation of 1,3-dioxin-4-ones or acylated Meldrum acids with generation of α -oxoketenes, hetero Diels–Alder reactions of the latter, and their transformations into lactones and lactams, among them macrocyclic: 99Y GK76.

5. *One Oxygen Atom and One Sulfur Atom*

X-Ray diffraction analysis of phthaleine and sulfophthaleine pigments: 97YZ764.

6. *Two Sulfur Atoms*

1,3-Dithiane 1-oxide derivatives as chiral auxiliaries and asymmetric building blocks for organic synthesis: 98OPP145.

Enantioselective oxidation of 1,3-dithianes to corresponding *S*-oxides and *S,S*-dioxides by designer yeast: 99JHC1533.

D. THREE HETEROATOMS

1. *Three Nitrogen Atoms*

Cyanamino-sym-triazine: 98KGS17.

Synthesis and properties of nitrotriazines: 97MI7.

Synthesis, properties, and applications of nonsymmetric triazinones: 99MI26.

Transformations of 1,2,4-triazines by the action of C-nucleophiles: 98ZOR327.

1,2,4-Triazine *N*-oxides and their annelated derivatives: 98UK707.

2. *Two Nitrogen Atoms and One Oxygen Atom*

Synthesis of 1,3,4-oxadiazines: 98H(49)557.

3. *Two Nitrogen Atoms and One Sulfur Atom*

Synthesis of 1,3,4-thiadiazines: 98H(49)557.

E. FOUR HETEROATOMS

4,6-Disubstituted 1,2,3,5-oxathiadiazine-2,2-dioxides, synthesis, structural features, chemical properties: 97KGS1182.

Synthesis and reactions of benzotetrathiine derivatives: 99PAC489.

VII. Rings with More Than Six Members

A. GENERAL

Synthesis and stereochemistry of seven- and eight-membered S-heterocycles: 99MI7.

B. SEVEN-MEMBERED RINGS

1. *One Heteroatom*

Chemistry of furazanes fused with seven-membered heterocycles with one heteroatom: 99UK154.

Methods for synthesis of saturated seven-member O-heterocycles and bicyclic systems with O-bridge: 98ACR603.

Stereoselective azepine ring formation via intramolecular ene reaction: 97YGK725.

Synthesis of seven-member oxacycles: 98T12631.

2. Two Heteroatoms

1,7-Electrocyclizations of $\alpha,\beta;\gamma,\delta$ -unsaturated 1,3-dipoles: 99AHC(73)97.

1,5-Benzodiazepines and 1,5-benzodiazepinium salts: 98AHC(71)1.

3. Three or More Heteroatoms

Synthesis and reactions of benzopentathiepine, 1,2,5-benzotrithiepine, 1,2,5-benzodithiaselenepine, and 1,2,5-benzoselenadithiepine derivatives: 99PAC489.

C. MEDIUM RINGS

Free-radical syntheses of medium-size heterocycles: 99T9349.

Synthesis and reactions of 2,3,6-benzotrithiocine derivatives: 99PAC489.

D. LARGE RINGS

1. General Problems

a. Structure, Stereochemistry, Reactivity, Design.

Artificial supramolecules: studies on multiple interactions: 97YGK357.

Carceplexes and hemicarceplexes, host-guest complexes in which carcerands and hemicarcerands, in particular those including heterocyclic fragments, are hosts: 99CRV931.

Cavity design using calix[n]arenes with heterocyclic fragments: 97CRV1713.

Complexes of cations with macroheterocycles including unsaturated and aromatic fragments: 97CRV1303.

Macrocyclic effect and specific character of complex formation with rigid macrocyclic ligands such as porphyrins and phthalocyanins: 97MI8.

Macroheterocycles as self-assembling cavities and capsules: 97CRV1647.

[(2n)]-Metacyclophanes (homocalixarenes) and their hetero- and heteroanalogues, compounds related to calixarenes with aromatic and heteroaromatic cycles bound by two-atom chains: 99ACR729.

Metallo dendrimers with heterocyclic fragments as supersupramolecules with novel properties: 99CRV1689.

Peculiarities of electrophilic substitution in *meso*-tetraazaporphyrins: 98ZOR647.

Polyrotaxanes and polycatenanes as interlocked macromolecules 99CRV1643. Positively charged and electroneutral macroheterocycles as host molecules for anions: 97CRV1609.

Progress in molecular recognition of functionalized calixarenes with heteroatomic bridges as synthetic receptors: 99MI43.

Pyridinophanes: 98KGS579.

Rotaxanes with macroheterocyclic components as new architectures for photoinduced electron transfer and motions: 99CSR293.

Stabilization of highly reactive species in molecular bowls and capsules with an endohedral functionality: 97LA2393.

Syntheses and reactions of carbohelicenes, heterohelicenes, and related systems: 94Y GK1021.

Template-directed synthesis of catenanes: 97CCC527.

Template effect and incapsulation in macroheterocyclic supramolecular systems: 97T15911.

Template synthesis and chirality of catenanes, rotaxanes, and pretzelanes including N-macroheterocyclic lactams and related compounds as structure components: 99PAC247.

Transannular interactions in macroheterocycles including two chalcogen atoms: 97Y GK1006.

b. *Synthesis.*

Pyridinophanes: 98KGS579.

Syntheses of Archaeobacteria 36- and 72-member macrocyclic membrane lipids (di- and tetraethers): 99Y GK785.

Syntheses of macrocyclic musks, among them lactones, dilactones, and oxalactones: 99S1707.

c. *Applications.*

Anion binding with polyazamacroheterocyclic cations and neutral S,N- and B,O-macroheterocycles: 99CLY546.

Complexes of transition metals with macroheterocyclic ligands as host molecules in the construction of molecular containers: 98CSR289.

Design and application of macroheterocycles as neutral anion receptors: 98CC443.

Structure, physical properties, and applications of dendrimers including heterocyclic fragments: 99CRV1665.

2. *Crown Ethers and Related Compounds*

- Cavitands with heteroatom bridges: 99EJO1991.
Crown ethers as analytical reagents and extractants of radionuclides: 98MI36.
Crown ethers as selective ligating agents: 97ACR338.
Design and synthesis of crown ethers and other macroheterocycles as highly selective ionophores for chemical ion sensors: 98YGK291.
Facile syntheses of highly symmetric cage-type cryptands consisted of pyridine rings and their properties as host molecules: 98YGK604.
Homolytic addition of dithiols to alkynes: a new approach to construction of dithiacyclanes and thiacycrown ethers: 97IZV1256.
NMR investigation of molecular structure of paramagnetic lanthanide complexes with crown ethers in solutions: 98ZSK714.
Novel electron-transfer reactions mediated by alkali metals complexed with crown ethers as macrocyclic ligands: 98ACR55.
Self-assembly of polyiodide networks on templates with crown ethers, thia- and thiazacycrown ethers as ligands: 98CSR195.
Self-assembly of secondary dialkylammonium ions with macrocyclic polyethers and formation of pseudorotaxanes: 96CC1483.
Structure–chemical aspects of complex formation in metal halide–macrocyclic polyether systems: 99UK136.
Supramolecular organic photochemistry of crown-containing styryl dyes: 97IZV641.
Supramolecules and supramolecular assemblies including silsesquioxanes and azacycrown ethers fragments as liquid crystals with restricted molecular topology: 98CC2057.
Synthesis and properties of thiacycrown compounds: 99KGS1587.

3. *Miscellaneous Macroheterocycles*

- Calixarenes including heterocyclic fragments as a rich source for molecular receptors: 97G637.
Calixpyrroles: 98CC1.
Covalent and noncovalent combination of porphyrins as well as calix[4]arenes, resorcin[4]arenes including macroheterocyclic fragments, and cyclodextrins by construction of supramolecular artificial receptors: 98EJO2689.
Dibenzotetraaza[14]annulenes as versatile ligands for transition and main group metal chemistry: 98CSR105.
Dynamic anion recognition by macrocyclic polyamines in neutral pH aqueous solution: 98CC1495.
Intramolecular mobility of metal complexes of rotaxanes and catenanes with macroheterocyclic fragments: 98ACR611.

Macroheterocycles capable of forming binuclear complexes with a common ligand: 96CC457.

Macroheterocyclic systems with 1,1'-binaphthyl fragments in molecular recognition and asymmetric catalysis: 98CRV2405.

Metal-directed self-assembly of two- and three-dimensional synthetic receptors (macroheterocycles involving transition metal atoms, among them chelated atoms): 98CSR417.

Molecular movements in pseudorotaxanes, rotaxanes, and catenanes with macroheterocyclic fragments: 98ACR405.

New supramolecular transition metal catalysts bound with cyclodextrin residues: 98JHC1065.

Noncovalent synthesis of organic nanostructures, hydrogen-bonded ensembles including heterocyclic analogs of calix[4]arenes: 98PAC1459.

Nonionic template synthesis of amide-linked catenanes and rotaxanes with macroheterocyclic fragments: 97AG(E)930.

Self-assembly in construction of pseudorotaxanes, rotaxanes, and catenanes with crown ether fragments: 98PAC419.

Structure/chiroptics relationships of planar chiral and helical molecules, in particular of heteracyclophanes and heterohelicenes: 98EJO1491.

Sulfur-containing macroheterocycles; synthesis and properties: 98MI16.

VIII. Heterocycles Containing Unusual Heteroatoms

A. GENERAL

9,10-Dimetallaanthracenes and 9,10-dimetallatripticycenes (Si-, Ge-, Sn-, Zn-, P-, As-, and Sb-metallacycles): 99CSR17.

B. PHOSPHORUS HETEROCYCLES

1. *Chemistry of Individual Classes of P-Heterocycles*

Atropisomeric binaphthalene-core phosphacyclic derivatives in coordination chemistry and homogeneous catalysis: 97CB543.

Chiral cyclic esters of phosphonic acid in the synthesis of coordination compounds and homogeneous asymmetric catalysis: 99KK83.

Contribution of Kazan chemical school to chemistry of phosphorus heterocycles: 99MI30.

Diphosphirenium and diphosphirenylium salts, diheteroatom-containing cyclopropenium analogs: 99ACR561.

P-Heterocycles with several heteroatoms: 99ACR751.
Kabachnik–Fields reaction in synthesis and transformations of P-heterocycles: 98UK940.
Mechanism of phase-transfer phenolysis of chlorinated cyclophosphazenes: 99MI20.
Some aspects of thermochemistry of O,P-heterocycles: 99MI41.
Syntheses, valence isomerizations, and reactions of phosphalkyne cyclotetramers: 97CB823.

2. *Structure and Stereochemistry*

Calixarenes with P,O-heterocyclic fragments: 98UK995.
Chiral P-heterocycles as P-mono- and P,N-bidentate ligands in the synthesis of coordination compounds and homogeneous asymmetric catalysis: 98KK883.
Coordination compounds with three-, four-, and six-member phosphorus-containing heterocycles: 98PAC819.
Hypercoordinated derivatives of P,O-heterocycles with intramolecular coordination by donor groups: 98EJI1847.
Triple-decker complexes with pentaphospholyl cycle between Ru and Fe or Ni atoms and carboranes and carbacycles C_nH_n ($n = 4-7$) as terminal ligands: 99IZV1636.
Ylidic four π -electron four-member λ^3 -phosphorus heterocycles as electronic isomers of heterocyclobutadienes: 98AG(E)270.

3. *Reactivity*

Applications of oxazaphospholidine–borane complexes, cyclic phosphoramides, compounds with N–P=O function, and other P-heterocycles in enantioselective catalysis: 99SL377.
Asymmetric olefination using optically active P,O-heterocycles as reagents: 98Y GK521.
Borane complexes of P-heterocycles as versatile precursors for the synthesis of chiral phosphine ligands for asymmetric catalysis: 98S1391.
Cyclic esters of α -halo boronic acids in asymmetric synthesis: 98T10555.

4. *Synthesis*

Phosphine in syntheses of P-heterocycles: 99UK240.
Reactions of halophosphines with conjugated heterodienes leading to P,O-, P,N-, and P,N,O-heterocycles: 99UK167.
Syntheses of five- and six-member P-heterocycles from 1,4- and 1,5-diketones: 98IZV1905.

Synthesis of novel optically active cyclic phosphine ligands and their use in catalytic asymmetric reactions: 98YGK511.

C. BORON HETEROCYCLES

1. Chemistry of Individual Classes of B-Heterocycles

Advances in the chemistry of B,N-heterocycles: 99T1197.
Borane clusters: 99CCC747.
Chemistry and structure of semi-sandwich platinum metal metallacarboranes derived from *nido*-C₂B₉H₁₂[−]: 97JOM(536/537)51.
Chemistry of small carborane ligands: 99JOM(581)1.
Cobaltoborane clusters: 99CCC783.
B-Heterocycles as ligands in transition metal complexes: 98AG(E)1786.
Recent advances in the chemistry of heterocarborane complexes incorporating s- and p-block elements: 97CRV2421.
Recent advances in the chemistry of small cage main group metallocarboranes: 99JOM(581)13.
Synthesis and properties of carborane-containing organophosphorus compounds: 97UK1125.
Synthesis, reactivity, and structure of transition metal boryl compounds, derivatives of B,N- and B,O-heterocycles with B—M bond: 98CRV2685.

2. Structure and Stereochemistry

cis-1-Aminoindan-2-ol-based annelated 1,3,2-oxazaborolidines; preparation and application in asymmetric synthesis: 98S937.
Borafullerenes B₂C₅₈, B₁₀C₅₀, B₁₂C₄₈, B₁₆C₄₄, B₁₈C₄₂, B₁₀C₁₀, and fullerene-like B₃₂, B₄₂ clusters: 98KK896.
Carboranes as a new class of weakly coordinating anions for strong electrophiles, oxidants, and superacids: 98ACR133.
N→B Chetates: 99JOM(581)129.
Complexes of B-heterocycles with nitrogen-containing compounds: 99JOM(581)129.
Correlations between ¹¹B NMR chemical shifts and electronic structure in borane and metal-borane clusters: 99CCC767.
o-, *m*-, and *p*-Dicarbadodecaboranes (12) as icosahedral main group cage molecules: 99CC1153.
Five- and six-coordinated nitrogen in azaborane clusters (*closo*-NB₉H₁₀, *nido*-NB₁₀H₁₃, *closo*-NB₁₁H₁₂, and their derivatives): 98EJI143.
Nucleic acids and nucleosides containing carboranes: 99JOM(581)156.

3. Reactivity

Asymmetric aldol reaction promoted by chiral oxazaborolidinone: 97Y GK313.

Enantioselective reductions of prochiral ketones by means of oxazaborolidines: 97CLY9.

Selective reactions of minor tautomers of allylic type triorganoboranes, among them cyclic triorganoboranes: 99JOM(581)98.

4. Synthesis

Triple-decker complexes with pentaphospholyl or cyclopentadienyl cycle between Ru and Fe or Ni atoms and carboranes as terminal ligands: 99IZV1636.

5. Applications

Alkyl-9-borabicyclo[3.3.1]nonanes in oxidation reactions leading to polyolefins: 99JOM(581)176.

Boratabenzenes as promising catalysts: 99JOM(581)92.

Carboranes, azaboranes, and borane clusters in constructing liquid crystal materials: 99CCC895.

B-Heterocycles as hydroborating agents: 97T4957.

B,S- and B,Se-Heterocycles and novel polymeric materials: 98AG(E)3208.

Olefins with 9-borabicyclo[3.3.1]nonane residues in the synthesis of borane-containing polyolefins: 99JOM(581)176.

Organic derivatives of *closo*-boranes, a new class of liquid-crystal materials: 99JOM(581)28.

Recent advances in the asymmetric catalytic reduction of ketones using chiral oxazaborolidines as ligands: 98MI64.

Reduction of carbonyl compounds with chiral oxazaborolidine catalysts: 98AG(E)1987.

D. SILICON, GERMANIUM, TIN, AND LEAD HETEROCYCLES

1. Chemistry of Individual Classes of Heterocycles

Cyclic Si-, Ge-, Sn- and Pb-analogs of carbenes and alkenes: 99EJI373.

Germynes and germanium double-bonded species in heterocyclic organogermanium chemistry: 99KGS1155.

Si,O-Heterocyclic cages as precursors to new materials: 97JOM(542)141.
Synthesis and stereochemistry of seven- and eight-member Si-heterocycles: 99MI7.
Synthesis and structures of carbonyl complexes with Si-heterocyclic π -ligands: 97JOM(536/537)31.

2. *Structure and Stereochemistry*

Molecular structures of Si-, N,Si-, and S,Si-heterocycles: 98MI45.
Molecular structures of cyclic compounds with Si—Si bonds: 98MI41.
New aspects in the chemistry of multiple bonds to heteroatoms, considered in particular for 2-silanaphthalene and silabenzene stabilized with 2,4,6-tris[bis(trimethylsilyl)methyl]phenyl substituent at the Si atom: 99PAC405.
Organized Langmuir films and polylayers of mesomorph cyclolinear polysiloxanes: 98MI35.
Self-organization of polysiloxanes containing cyclic fragments into mono- and multilayer structures at interphase boundaries: 99MI40.
Stereochemistry of polysubstituted 1,3-dioxo-2-silacyclohexanes: 97MI42.

3. *Reactivity*

Direct spectroscopic studies and calculations of cyclic silylene-to-silene and germylene-to-germene interconversions: 99IZV2027.

4. *Synthesis*

Intramolecular reactions of temporarily silicon-tethered molecules in synthesis of O,Si-heterocycles: 97S813.
Synthesis and properties of silole derivatives: 98YGK500.
Synthesis of 1,3-dioxo-2-silacycloalkanes and their heteroanalogs: 97MI42, 98MI24.

E. SELENIUM AND TELLURIUM HETEROCYCLES

1. *General Sources and Topics*

Se-Containing heterocycles: 97UK1025.
Synthesis of optically active selenium and tellurium heterocycles and their application for asymmetric synthesis: 97OPP603.

2. Chemistry of Individual Classes of Heterocycles

B,Se-Heterocycles and novel polymeric materials: 98AG(E)3208.

Se- and Te-(Perfluoroalkyl)dibenzoselenophenium and -tellurophenium salts: 95AHC(64)323.

Reactivity of tetraselenafulvalenes: 95AHC(62)249.

Synthesis and reactions of 1,2,5-benzodithiaselenepine and 1,2,5-benzoselenadithiepine derivatives: 99PAC489.

Synthesis and stereochemical study on reactions of optically pure cyclic chalcogenuranes, Se(IV)-, and Te(IV)-derivatives: 99YGK587.

Synthesis, reactions and structures of complexes of metal carbonyls and cyclopentadienyl carbonyls with Te-heterocycles as ligands: 99UK454.

Thiyl radicals and their analogs in gas-phase syntheses and transformations of compounds of selenophene series: 99ZOR11.

F. OTHER UNUSUAL HETEROCYCLES

1. Metallacycles

Chemistry and structure of semi-sandwich platinum metal metallacarboranes derived from *nido*-C₂B₉H₁₂⁻: 97JOM(536/537)51.

Chemistry of As-Heterocycles: 99MI25.

Cobaltoborane clusters: 99CCC783.

Coordination as the motif in the rational design of supramolecular metallacyclic polygons and polyhedra: 97ACR502.

Copper-catalyzed or mediated carbon-carbon bond formation reactions of zirconacycles and alkenylzirconocenes: 97YGK958.

Cyclogallenes, Ga-heterocycles with Ga-Ga bond: 99ACR773.

Ferrocenophanes with all carbon bridges: 99JOM(578)31.

Formation of metallacycles in solid state: 98JOM(571)149.

Generation of dialkoxytitanacycles from olefins, acetylenes, and (η^2 propene)Ti (O-*i*-Pr)₂ and their new synthetic reactions: 99YGK492.

Al-Heterocycles, formation from olefins and acetylenes in a metallocomplex-catalyzed cycloaddition reaction and further transformations: 98IZV816.

Al-Heterocycles, structural data: 97JOM(543)1.

As-Heterocycles with several heteroatoms: 99ACR751.

Luminescent polynuclear metal acetylides with a metal as part of a cycle: 99JOM(578)3.

[*m*]Mercuracarborands-*n* (macrocyclic compounds with *m*-member cycle constructed from *n* *o*-carborane residues connected with the bridge Hg atoms) as

- host molecules in recognition of electron-donating guest molecules: 97ACR267.
- Metallacycles as intermediates in redox rearrangements of cyclopropane and cyclobutane derivatives: 96MI10.
- Metallacycles containing metal-heteroatom bond in their ring system: 98Y GK171.
- Molecular phosphonate cages with metal, P, and O atoms as model compounds and a source for phosphate materials: 99ACR117.
- Ni- and Pt-Cycles as intermediates in reactions of Ni(0)- and Pt(0)-complexes with benzyne or related small-ring alkynes: 97CB1029.
- Palladacycles as reactive intermediates: 97CB1567.
- Planar tetracoordination in metallocycles with group 4 and 5 metals: 99ACR494.
- Ring-opening polymerization of strained metallocenophanes, mainly ferrocenophanes with Si atom at the bridge, to give high-molecular-weight poly(metallocenes): 99PAC1471.
- Self-assembly of [2]catenanes containing metals in their backbones: 99ACR53.

2. Metal Chelates and Related Complexes

- Progress in investigation of ruthenium porphyrin complexes: 97MI10.
- Structure and reactivity of chelates due to intramolecular coordination of hypervalent type: 99MI6.
- Synthesis of chelates (including those with heterocyclic ligands) by direct interaction of ligands with metals or metal oxides, particularly using electrochemical or tribochemical activation: 97MI41.
- Technetium chelates with heterocyclic ligands: 98CSR43.

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Preface

Volume 79 of *Advances in Heterocyclic Chemistry* commences with an overview of Tellurium–Nitrogen-Containing Heterocycles by I. D. Sadekov and V. I. Minkin of Rostov State University, Russia, and represents an update of the review published by the same authors in Volume 58 of *Advances*, eight years ago. The field has expanded markedly in the recent past, and the compounds show promise in an increasing number of applications, particularly in the material science field.

Professor D'Auria (Basilicata University) summarizes the photochemical isomerization of pentaatomic heterocycles, providing a unified description in terms of the five conceivable mechanisms. R. Alan Aitken and Andrew W. Thomas of the University of St. Andrews, Scotland, summarize the substantial recent progress in heterocyclic acyl and formyl anion equivalents.

The fourth chapter of this volume comprises the second part of an ongoing series by Professor A. P. Sadimenko (Fort Hare University, South Africa) dealing with organometallic compounds of pyrrole, indole, carbazole, phospholes, siloles, and boroles. This follows the review in Volume 78 of *Advances* covering organometallic compounds of thiophene and furan. The enormous recent advances in this area are summarized and classified according to the nature of the heterocycle and of the metals.

This volume ends with part 7 of the ongoing series on the literature of heterocyclic chemistry, by Professor L. I. Belen'kii and Drs. N. D. Kruchkovskaya and V. N. Gramenitskaya, all of the Russian Academy of Sciences, Moscow. It is a sequel to six earlier parts published in *Advances in Heterocyclic Chemistry*, which started in 1966 in Volume 7 of *Advances*, and have also appeared in Volumes 25, 44, 55, 71, and 73. The present part covers monographs and reviews published during the three-year period, 1997–1999.

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