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Selenium-containing heterocycles

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Abstract. The literature data, mainly of the last ten years, on the synthesis, reactions, and practical utilisation of selenium-containing heterocycles are analysed, treated systematically, and surveyed. The bibliography includes 516 references.

I. Introduction

Organoselenium compounds have continued to attract the attention of a wide range of investigators owing to a series of their unique properties. At the Second International Symposium on the Chemistry and Biochemistry of Organic Compounds of Selenium and Tellurium, it was noted that the deficiency of selenium in the human and animal organisms constitutes the basis of various chronic diseases, in particular the necrosis of the liver.[1,2] There are data showing that organoselenium compounds are capable of sensitising processes in the living organism,[3] which includes the inhibition of anomalous oxidation processes with the aid of the Se^{2-} containing glutathioneperoxidase active group.[1] Four proteins containing a selenium atom are known among natural products.[4] Therefore, despite the high toxicity of many selenium compounds,[5–7] antitumour,[8–10] anticancer,[11,12] and other medicinal preparations,[13–21] as well as biologically active substances exhibiting antiviral,[22–26] antimicrobial,[27–30] antiarrhythmic,[31] and fungicidal[32–36] activities have been produced on the basis of organic derivatives of selenium. Furthermore, superconducting materials[37] as well as dyes,[38–48] including those suitable for electrophotography,[49–53] have been prepared on their basis.

Heterocycles containing a selenium atom occupy a special place among organoselenium compounds. Although numerous reviews on various aspects of the chemistry of organoselenium compounds have been published during the last ten years,[54–74] the survey of literature data on organoselenium heterocycles is restricted to pre-1985.[74–77] It therefore appears useful to analyse the data on selenium-containing heterocycles, concentrating attention on studies of recent years.

The size of the heterocycle and not the method of synthesis has been selected as the criterion for the systematic arrangement of data on selenium-containing heterocycles. To achieve a more compact exposition and to eliminate the possibility of duplicating data, the chemical and other properties of the compounds considered are quoted together with the methods of their synthesis.

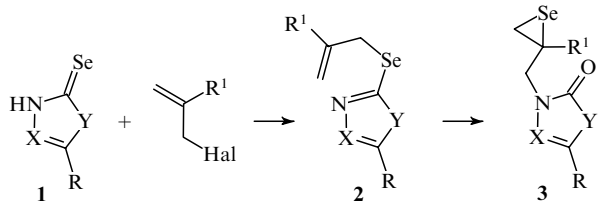
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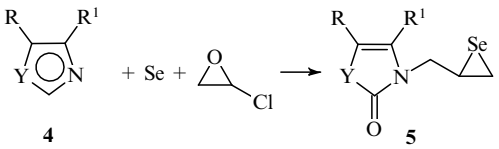
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II. Three-membered heterocycles

1. Seleniranes

Until recently, seleniranes (episelenides) had not been obtained in a pure form, in contrast to their sulfur analogues (thiiranes),[78] although their formation in the flash photolysis of CSe_2 had been reported,[79–81] which was confirmed in a mass-spectrometric study.[82] Later the formation of seleniranes as intermediates was postulated in reactions involving the specific conversion of oxiranes,[83–86] the cyclisation of *para*-substituted allyl phenyl selenides,[87] the conversion of bromohydrins into alkenes on treatment with potassium selenocyanate[88] or of 1,2-dibromoethane into ethene in the presence of the selenodithiocarbonate anion,[89] and in the desulfurisation of thiiranes with 3-methyl-2-selenoxobenzothiazole.[90] The decomposition of the intermediate seleniranes with liberation of selenium was noted in all the examples quoted above. It was not until the end of the 1990s that reports of the synthesis of substituted seleniranes by the alkylation of heterocyclic selenones **1** and the subsequent transformation of the selenides **2** into substituted seleniranes **3**[91,92] or by the interaction of the azoles **4** with selenium and epichlorohydrin with formation of the seleniranes **5**[93] appeared although without the specification of experimental data.



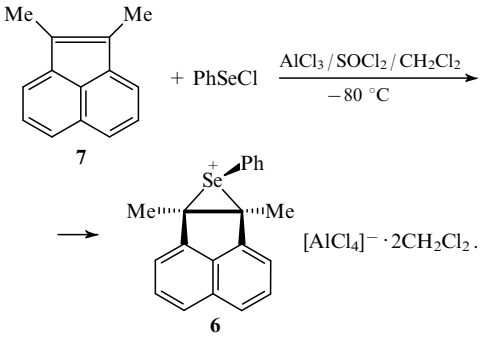


4 **5**

R = Alk, Ar; R^1 = H, Me; X = CR, N; Y = NR, S; Hal = Br, I.

2. Seleniranium salts

The formation of seleniranium (episelenonium) salts as intermediates has been noted in a series of studies,[94–104] but it was not until 1986 that data concerning the synthesis of the stable episelenonium salt **6** as a result of the treatment of 1,2-dimethylacenaphthylene **7** with PhSeCl in the $AlCl_3-SOCl_2-CH_2Cl_2$ system at −80 °C were published.[105] According to X-ray (XRD) data, a characteristic feature of the structure of the episelenonium ring in the salt **6** is the appreciable lengthening of the Se^+-C bond compared with the corresponding bonds in trimethylselenonium,[106] which causes their lower stability. It has also been shown that the benzene and acenaphthene rings in the salt **6** are in the *cis*-position not only in the solid state but also in solution.

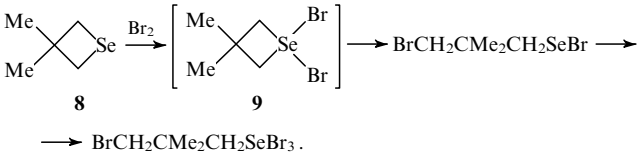


7 **6**

III. Four-membered heterocycles

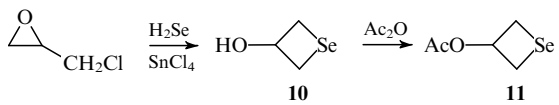
1. Selenetanes

Unsubstituted selenetane was first obtained in a low yield in 1930[107] by treating 1,3-dibromopropane with alkali metal selenides. Selenetane readily polymerises and can be stored only at reduced temperatures in the dark. 3,3-Dimethylselenetane **8** is more stable (up to 140 °C),[108,109] whereas its dibromo-derivative **9** is stable only at temperatures below −20 °C; when the temperature is raised, the selenetane ring is opened.[108,110]



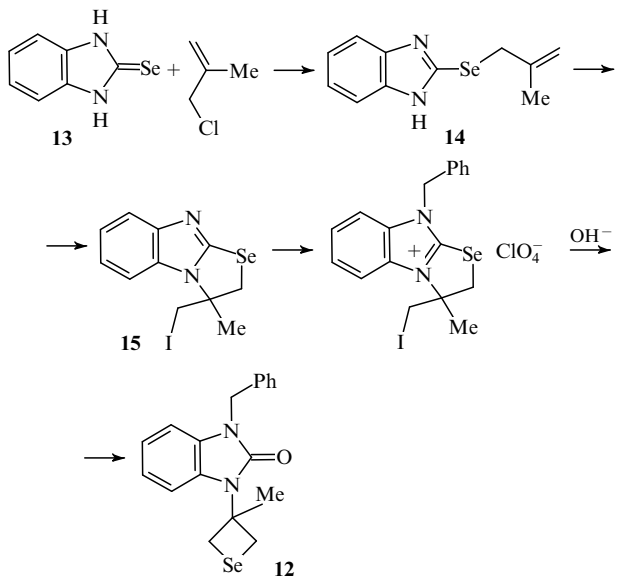
8 **9**

Selenetan-3-ol **10** is a stable compound. It is formed in 58% yield as a result of the reaction of epichlorohydrin with hydrogen selenide at 60 °C in the presence of tin chloride or toluene-*p*-sulfonic acid[111] or on decomposition of the salt Hg(II) 1,3-diselenylpropan-2-olate.[112] Treatment of the selenetanol **10** with acetic anhydride leads to the acetate **11**.[110]



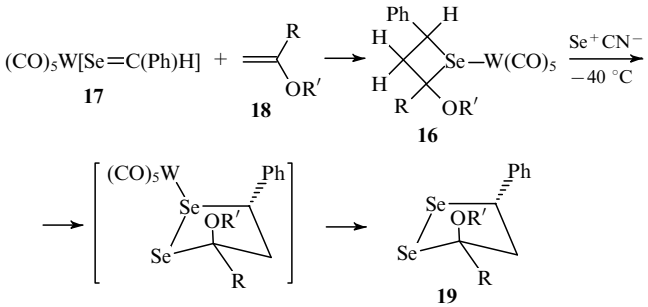
10 **11**

The substituted selenetane **12** is obtained by means of a scheme involving in the first stage the alkylation of the heterocyclic selenone **13** with formation of the selenide **14**, which affords benzimidazo[2,1-*a*]selenazolidine **15** on iodocyclisation. As a result of a series of reactions, involving quaternisation and recyclisation, compound **15** gives rise to the selenetane **12**.[113]



13 **14** **15** **12**

The pentacarbonyltungsten complexes of the selenetane **16** are formed when complex **17** is treated with an excess of vinyl ethers.[114,115] Complex **16** reacts with an excess of selenocyanate already at −40 °C in the course of several minutes with formation of the diselenolanes **19**. The structure of one of them (R = R′ = Me) has been confirmed by XRD data.[115]

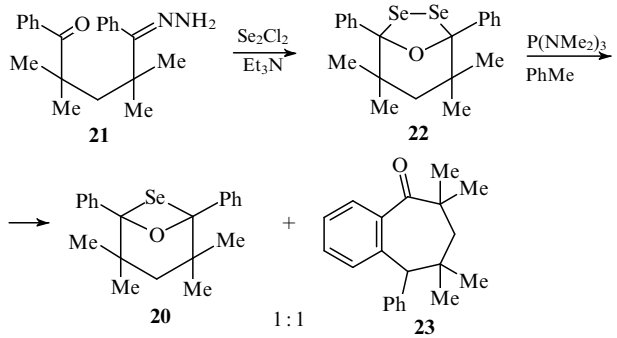


17 **18** **16** **19**

R = H, R′ = Et; R = R′ = Me.

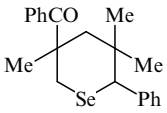
2. Oxaselenetanes

Four-membered heterocycles containing one oxygen atom and one selenium atom (oxaselenetanes) are known intermediates in the oxidation reactions of exocyclic alkenes by means of SeO_2/H_2O_2.[116] The bicyclic 1,3-oxaselenetane derivative **20** was obtained by refluxing 2,2,4,4-tetramethyl-1,5-diphenylpentane-1,5-dione monohydrazone **21** with Se_2Cl_2 in the presence of triethylamine.[117–119] The reaction proceeds *via* a stage involving the formation of the 1,3,4-oxadiselenolane **22**, the treatment of which with $P(NMe_2)_3$ or PPh_3 leads to a 1 : 1 mixture of oxaselenetane and benzosuberone derivatives **20** and **23** respectively.



21 **22** **20** **23**

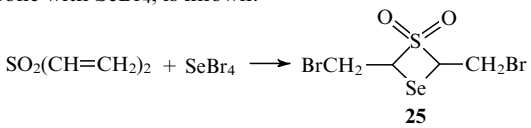
After refluxing the reaction mixture for 10 h, the substituted selenacyclohexane **24** is also formed.[119]



24

3. Thiaselenetanes

Among four-membered heterocycles containing sulfur and selenium atoms, the thiaselenetane **25**, formed on treating divinyl sulfone with $SeBr_4$, is known.[120]

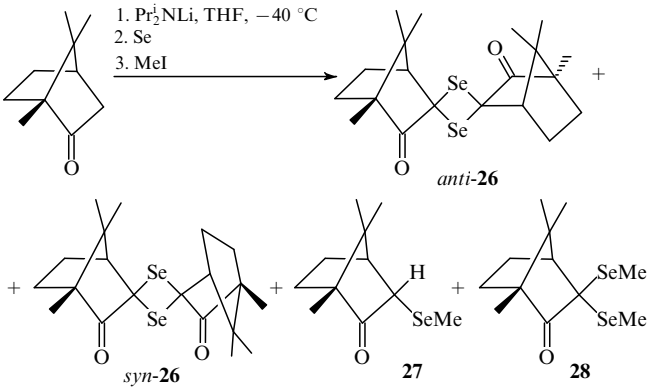


25

4. Diselenetanes

Many examples of four-membered heterocycles with two selenium atoms are known. Thus 3,4-bis(trifluoromethyl)-1,2-diselenetane has been obtained in 25% yield on pyrolysis of a mixture of bis(trifluoromethyl)ethyne and selenium.[121,122] 1,3-Diselenetanes are synthesised by various methods: the pyrolysis of 1,2,3-selenadiazole [123] and the reactions of dimethyl malonate with CSe_2,[124] of acetylacetone with $SeCl_4$,[125] of acetyl chloride with H_2Se and $AlCl_3$,[126] and of hexafluoroacetone with triphenylphosphine selenoxide.[127]

It has been observed recently that the reaction of the lithium enolate of camphor with selenium and subsequent treatment of the reaction mixture with methyl iodide lead to a mixture of the *syn*-(32%) and *anti*-(10%) isomers of 1,3-diselenetane **26** together with methyl selenide **27** (12%) and the diselenoacetal **28** (15%).[128] The structure of the *anti*-isomer **26** was confirmed by XRD.



anti-**26**

syn-**26** **27** **28**

IV. Five-membered heterocycles

The vast majority of studies on the chemistry of selenium-containing heterocycles have been devoted to five-membered heterocycles owing to their relative resistance to the action of various kinds of reagents (by virtue of their aromaticity, the small Bayer strain, and the absence of Pitzer strain) as well as their great practical importance.

Compounds with the properties of semiconductors,[129,130] polymethine dyes,[131–134] photosensitisers,[135–144] as well as substances exhibiting various types of biological activity (antimicrobial,[145] antiviral,[146] antitumour,[147–150] glutathioneperoxidase,[151] spasmolytic,[152,153] antiallergic,[154] antiherpetic,[155] and fungicidal [156–158]) are known among derivatives of this class.

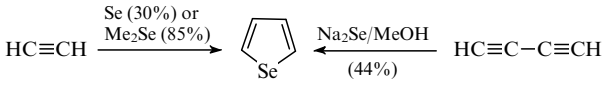
1. Five-membered heterocycles with one heteroatom

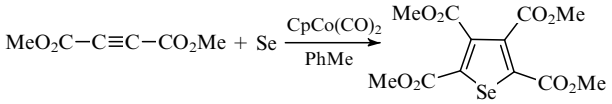
a. Selenophenes

Selenophene and its derivatives have been most thoroughly investigated among five-membered selenium-containing heterocycles. The continued interest in selenophene has been reflected periodically in published reviews.[159–162]

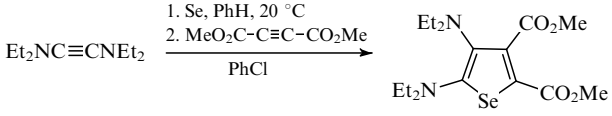
Fairly numerous new methods of synthesis of selenophene and its derivatives have been developed during the last decade. One of them involves the interaction of acetylene and its derivatives with elemental selenium. Selenophene has been obtained in this way in 30% yield [163] together with its derivatives **29** (4%) [164] and **30** (87%).[165]

Selenophene may be obtained in 85% yield by the reaction of acetylene with dimethyl selenide in the gas phase [166] and also by treating diacetylene with sodium selenide in methanol (yield 44%).[167] In addition, it has been shown that 2% of selenophene is also obtained on heating dimethyl selenide in a quartz tube at 470 °C.[168]



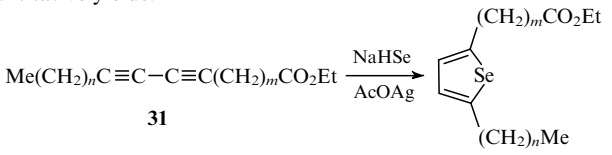


29 (4%)



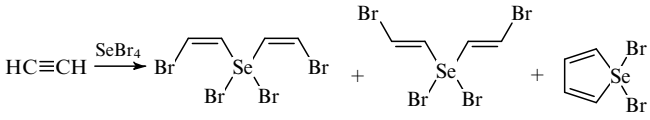
30 (87%)

The interaction of sodium hydrogenselenide with diethynes **31** results in the formation of 2,5-disubstituted selenophenes **32** in quantitative yields.[169]



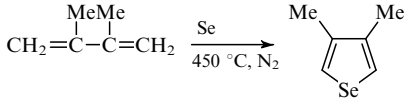
m, *n* = 1–8.

On interaction with selenium tetrabromide, unsubstituted acetylene affords a mixture of bromine-substituted selenides, including selenophene dibromide **33**.[170]

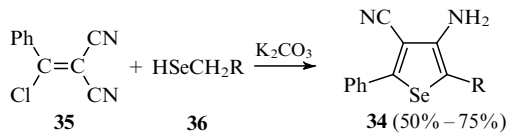


33

Other unsaturated compounds have also found application in the synthesis of selenophenes. Thus buta-1,3-diene reacts with selenium dioxide in the presence of Zeokar 2 as the catalyst with formation of selenophene in 45% yield.[171] 3,4-Dimethylselenophene is formed in up to 80% yield from 2,3-dimethylbuta-1,3-diene on interaction with powdered selenium in the presence of sand or glass fragments at 450 °C under a nitrogen atmosphere.[172]

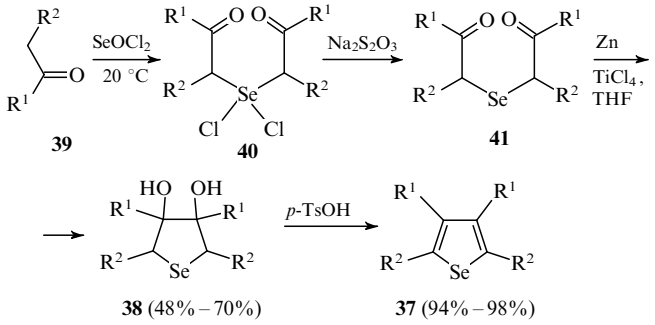


The synthesis of functionally substituted selenophenes **34** on interaction of the nitrile **35** with alkaneselenols **36** has also been reported.[173]



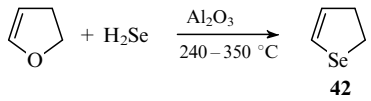
R = Ac, PhCO, CN, NO_2, CHO, COOH, CO_2Et.

The method of synthesis of substituted selenophenes **37** by the dehydration of selenolanediols **38** has been described.[174] The diols were obtained in accordance with the following scheme: the interaction of selenium oxychloride with the ketones **39** to form the chlorinated selenides **40** which are readily reduced by sodium thiosulfate in benzene to the selenide **41**, and subsequent treatment with $TiCl_4$ and Zn in tetrahydrofuran (THF) at 0 °C.

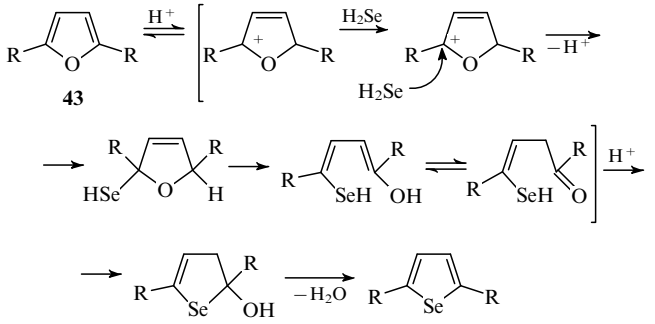


R^1 = Ar, Het; R^2 = H, Me.

The transformation of furan and thiophene into selenophene on treatment with hydrogen selenide or elemental selenium, used for the synthesis of 2,3-dihydroselenophene **42**, is well known.[175]

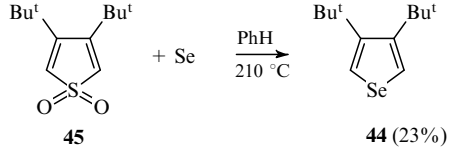


The mechanism of this reaction has been investigated relatively recently. It has been established in relation to the interaction of the furans **43** with hydrogen selenide that the reaction proceeds *via* a mechanism involving specific acid catalysis including a stage with a doubly protonated substrate.[176, 177]



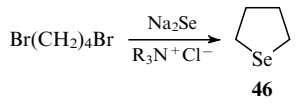
R = H, Me.

3,4-Di(*tert*-butyl)selenophene **44** has been obtained in 21% yield by treating 3,4-di(*tert*-butyl)thiophene 1,1-dioxide **45** with powdered selenium in benzene in an autoclave at 210 °C.[178]

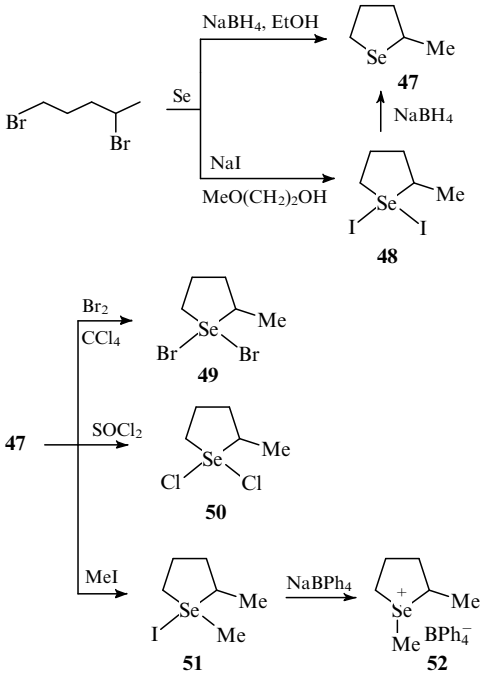


b. Selenolanes

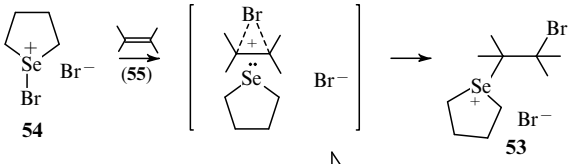
A simple method of synthesis of selenolane **46**, by refluxing 1,4-dibromobutane with sodium selenide under a nitrogen atmosphere under the conditions of phase-transfer catalysis, has been developed. Alkyl(C_8-C_{10})ammonium chlorides were used as the catalysts.[179]

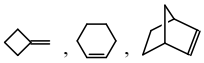


2-Methylselenolane **47** has been obtained [180] in two ways. The refluxing of 1,4-dibromopentane with a 1 : 1 mixture of Se and NaI in solution in the monomethyl ether of ethylene glycol leads to the diiodide **48** in 27% yield, which is reduced with $NaBH_4$ to 2-methylselenolane. The reaction of 1,4-dibromopentane with elemental selenium in alcohol in the presence of sodium tetrahydroborate under an argon atmosphere leads to compound **47** in 78% yield. Certain reactions of 2-methylselenolane have been investigated. Thus, on interaction with bromine in CCl_4, it gives rise to the dibromide **49**, while its interaction with $SOCl_2$ affords the dichloride **50** in 85% yield. Treatment of 2-methylselenolane **47** with MeI leads to compound **51** which on reaction with $NaBPh_4$ is transformed into the salt **52**.

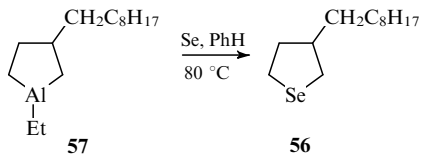


The interaction of tetramethylenebromoselenonium bromide,[54] obtained from selenolane and bromine, with the alkenes **55** in acetonitrile at 20 °C leads to the β-bromoselenonium salts **53** in yields ranging from 50% to 75%.[181] The reaction proceeds in accordance with the Markovnikov rule as stereospecific electrophilic addition with formation of *trans*-adducts.



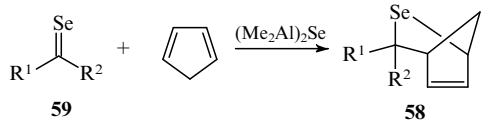
55: $C_5H_{11}CH=CH_2$, .

The interaction of 3-nonylaluminacyclopentane **57** with elemental selenium on refluxing in benzene has been used in the synthesis of 3-nonylselenolane **56**.[182]



57 **56**

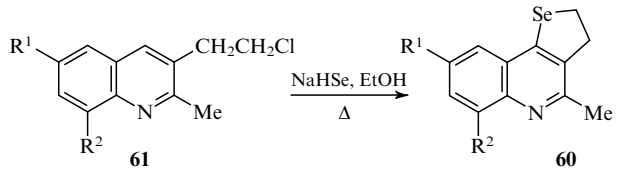
A method based on the interaction of selenocarbonyl compounds **59** with cyclopentadiene has been proposed for the synthesis of bicyclic selenolane derivatives — selenabicycloheptenes **58**.[183–187]



59 **58**

R^1, R^2 = H, Me, Ph, $SiMe_3$; $R^1–R^2$ = $(CH_2)_5$.

The interaction of 2-chloroethyl derivatives of the quinolines **61** with sodium hydrogenselenide in ethanol has been used in the synthesis of condensed derivatives of the selenolanes **60** containing a quinoline fragment; the yield of the selenolanes **60** then reaches 45%–58%.[188]

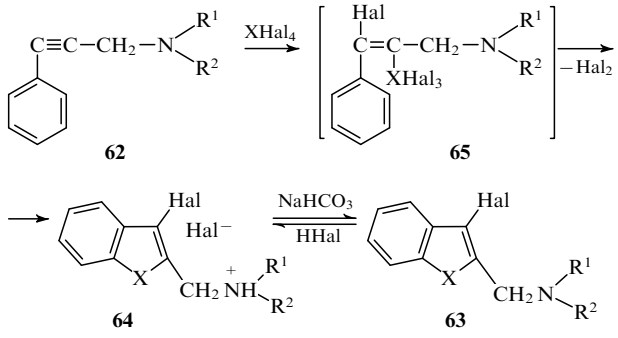


61 **60**

R^1, R^2 = H, Me, OMe, Cl.

c. Condensed selenophenes

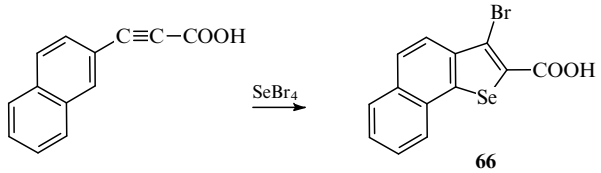
Substituted acetylenes, which give rise to both benzo- and naphtho-selenophenes on interaction with selenium tetrahalides, have been used successfully in the synthesis of condensed selenophenes. Thus a preparative method of synthesis of aminomethyl derivatives of benzo[*b*]thio(seleno, telluro)phenes **63** and their hydrohalides **64** has been developed on the basis of the interaction of chalcogen halides with 3-phenylpropynylamines **62**.[189] The reaction proceeds with formation of the intermediates **65**.



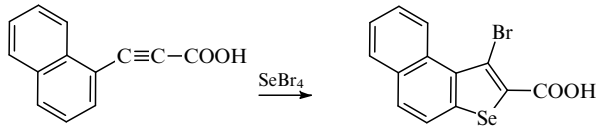
62 **65** **64** **63**

R^1 = H, Me, Et; R^2 = Me, Et; R^1,R^2 = $(CH_2)_5$, $(CH_2)_2O(CH_2)_2$; X = S, Se, Te; Hal = Br, Cl.

The similar treatment of 3-(2-naphthyl) and 3-(1-naphthyl)-propiolic acid derivatives with selenium tetrabromide leads to the substituted naphthoselenophenes **66** and **67**.[190]

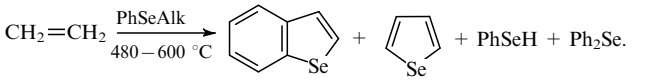


66

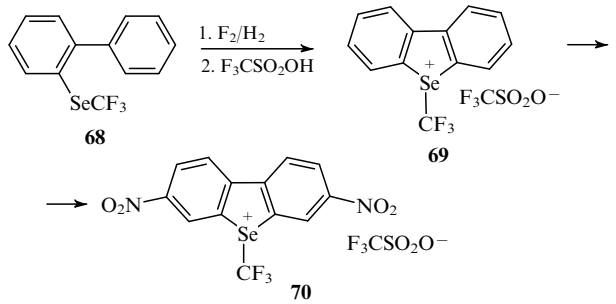


67

Ethene reacts with alkyl phenyl selenides at high temperatures (480–600 °C) to form benzoselenophene (yield 25%), selenophene, and other organo-selenium compounds.[191]

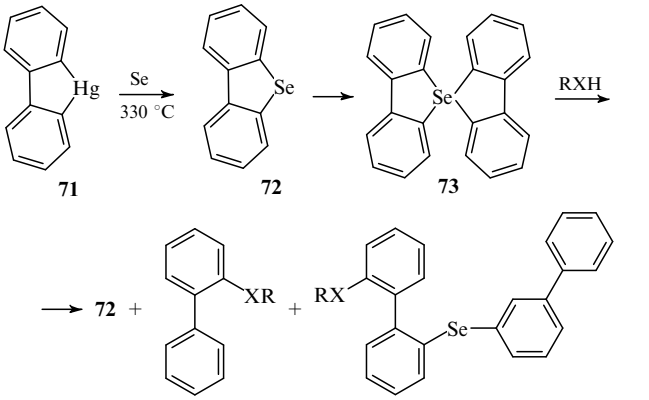


2-Biphenylyl trifluoromethyl selenide **68** has been used to obtain the dibenzoselenophene salt **69**, which is readily converted into the corresponding dinitro-derivative **70**.[192]



68 **69** **70**

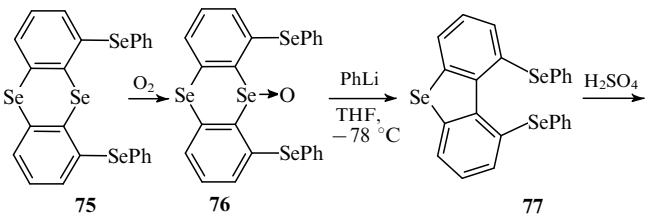
On heating with elemental selenium, 9-mercurafluorene **71** affords dibenzoselenophene **72**, which readily dimerises to the selenurane **73**[193] and the latter reacts with alcohols, thiols, and selenols in THF at −78 °C under an argon atmosphere to form a mixture of products, one of which is dibenzoselenophene **72**.[194]



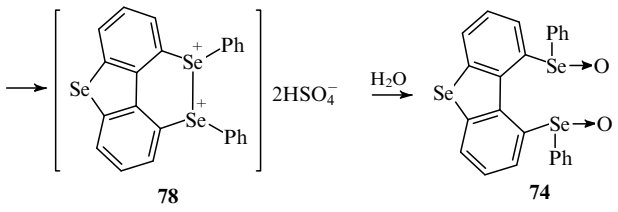
71 **72** **73**

R = Me, Et, Pr, Pr^i, Ph;
X = O, S, Se.

A method of synthesis of the disubstituted dibenzoselenophene **74** *via* a scheme involving the oxidation of selenanthrene **75** and the reduction of the resulting selenoxide **76** to compound **77** has also been described. Treatment of the latter compound with sulfuric acid leads to the dioxide **74**. It has been established by NMR that the reaction proceeds *via* the dication **78**.[195]

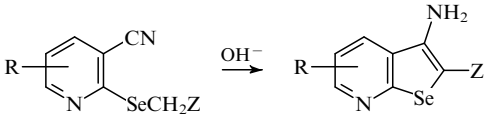


75 **76** **77**



78 **74**

The Thorpe – Ziegler cyclisation of pyridine-series selenides **80** has been used successfully in recent years for the synthesis of functionally substituted selenopheno[2,3-*b*]pyridines **79**.[69, 196 – 221]

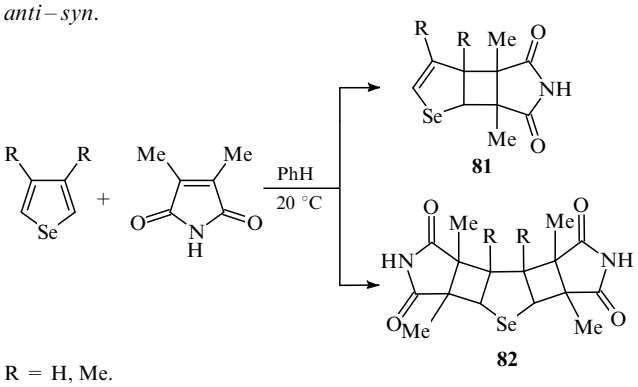


80 **79**

R = H, Ar, Het, Alk, NH_2, CO_2Alk, OH, CN;
Z = CN, CO_2Alk, COAr, COHet, CONHAr, $CONH_2$.

d. Certain properties of selenophenes

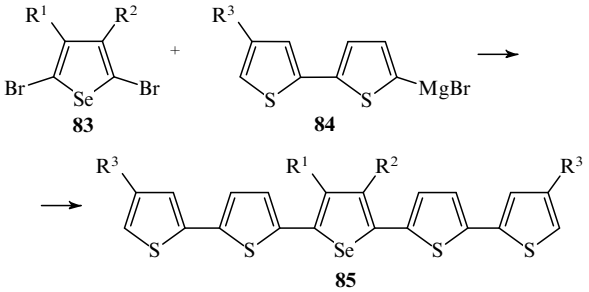
The photochemical [2 + 2]cycloaddition of selenophene and 3,4-dimethylselenophene to maleimide with a 1 : 1 reactant ratio leads to the monoadducts **81** in quantitative yields.[222] When a twofold excess of maleimide is used in this reaction, the bisadducts **82** are formed in 60% – 70% yields. According to ^{13}C NMR data, they exist in the form of three stereoisomers: *syn* – *anti*, *anti* – *anti*, and *anti* – *syn*.



81 **82**

R = H, Me.

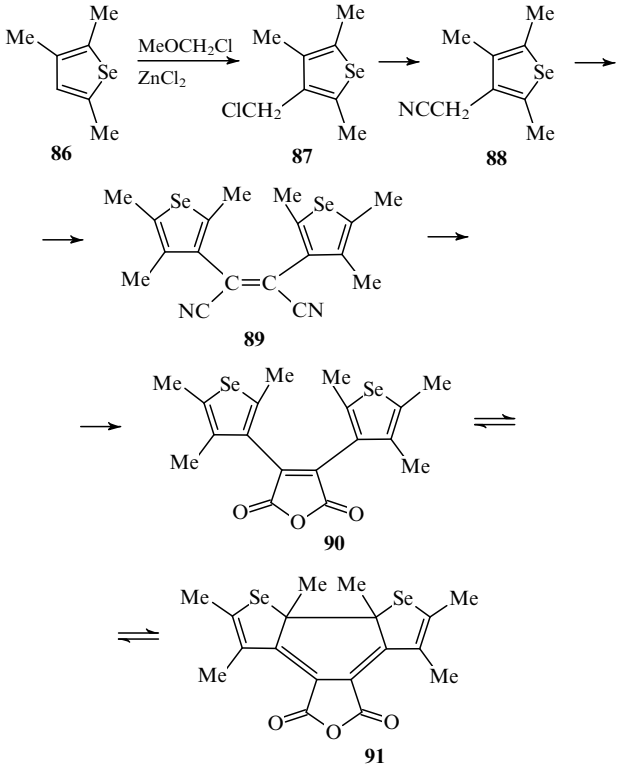
On interaction with dithienylmagnesium bromides **84**, the bromoselenophenes **83** afford the oligomeric heteroarylenes **85**.[223, 224]



83 **84** **85**

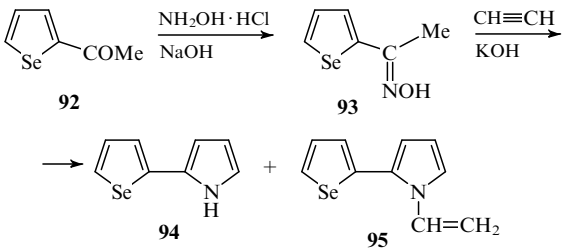
R^1, R^2, R^3 = H, Me.

The alkylation of 2,3,5-trimethylselenophene **86** with chloromethyl methyl ether in the presence of zinc chloride has been investigated. The resulting chloromethyl derivative **87** is readily transformed into the nitrile **88**, which dimerises to the substituted ethene **89**. Treatment of the latter with KOH in aqueous $EtOCH_2CH_2OH$ gives rise to the anhydride **90**, which on irradiation in benzene at 20 °C affords a photostationary mixture with the deeply coloured cyclic form **91** capable of being fully and reversibly converted into the anhydride **90** on irradiation with light at a wavelength $\lambda > 520$ nm.[225]



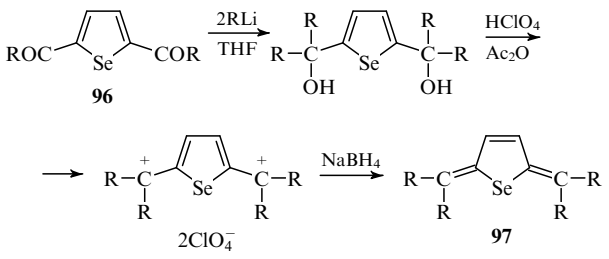
86 **87** **88** **89** **90** **91**

2-Acetylselenophene oxime **93** reacts with ethyne to form a mixture of the bisheterocycles **94** (10%) and **95** (2%).[226]



92 **93** **94** **95**

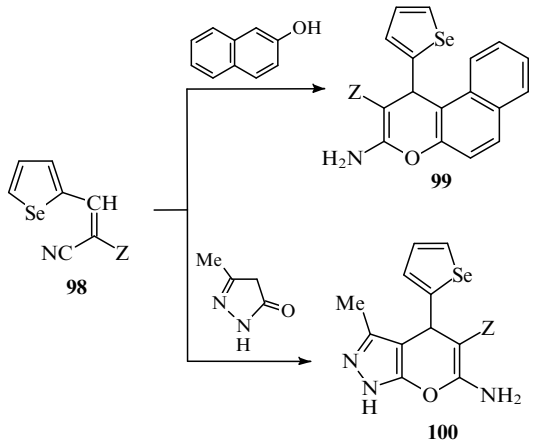
The triene heterocyclic systems **97** have been obtained from 2,5-diacylselenophenes **96** in 46% – 71% yields in accordance with the following scheme:[227]



96 **97**

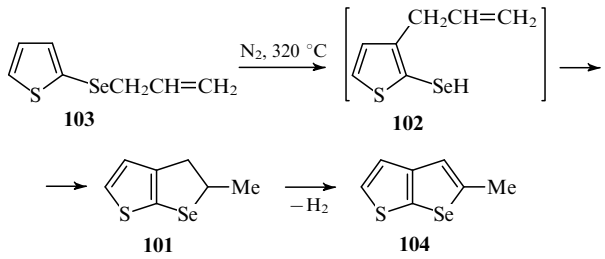
R = Ph, .

The selenophene-substituted acrylonitriles **98** have been used successfully in the synthesis of the naphthopyrans **99**[228] and the pyrazolopyrans **100** containing a selenophene ring.[229]

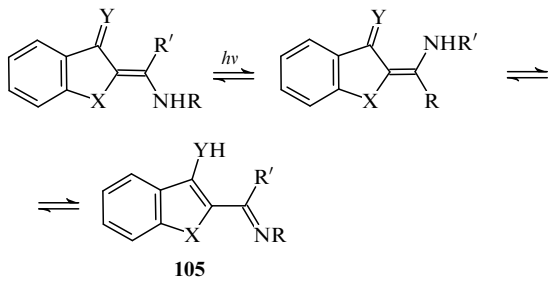


Z = CN, CO_2Et, $CONH_2$, $CSNH_2$.

2-Methyl-2,3-dihydroselenopheno[2,3-*d*]thiophene **101**, formed *via* the intermediate **102** on heating allyl 2-thienyl selenide **103**, is dehydrogenated to 2-methylselenopheno[2,3-*b*]thiophene **104**.[230]

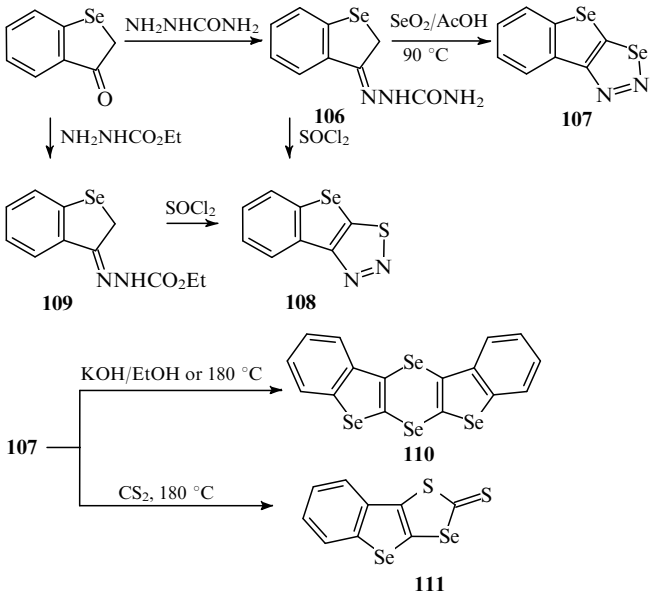


The benzenoid–quinoid tautomerism, which is a thermodynamically controlled process, has been investigated in a series of substituted benzo[*b*]heterocycles **105**. These compounds can be used for the accumulation and transformation of solar energy.[231,232]

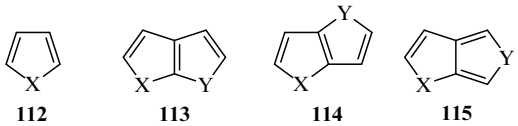


X = O, S, Se, Te; Y = O, S; R, R′ = H, Alk, Ar.

Benzo[*b*]selenopheno[3,2-*d*]-1,2,3-selenadiazole **107** has been obtained in 82% yield by the reaction of benzo[*b*]selenophen-3-one semicarbazone **106** with SeO_2 in glacial acetic acid.[233–235] Its sulfur analogue — benzo[*b*]selenopheno[3,2-*d*]-1,2,3-thiadiazole **108** — is formed on treatment with thionyl chloride of the semicarbazone **106** (yield 90%) or the ethoxycarbonylhydrazone **109** (yield 75%).[234,235] On treatment with an alcoholic solution of alkali, the selenadiazole **107** is converted in 94% yield into dibenzo[*b*]selenopheno[2,3-*b*:2,3-*e*]-1,4-diselenine **110**, which is also formed as the sole product in the thermolysis of the selenadiazole **107** at 180 °C. On the other hand, if the selenadiazole **107** is heated at the same temperature in an autoclave in the presence of CS_2, then benzo[*b*]selenopheno[2,3-*d*]-1,3-thiaselenole-2-thione **111** is formed as the main reaction product (25%).[234,235]



The dependence of the transfer of the influence of the substituents in the five-membered sulfur- and selenium-containing heterocycles **112** and their annelated derivatives **113**–**115** on the position of the reaction centre, the nature of the heteroatom, and the type of annelation has been analysed with the aid of quantum-chemical methods and the empirical constants of the substituents.



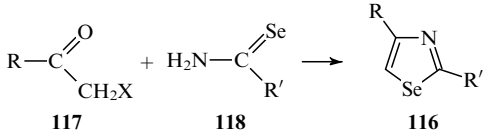
X, Y = S, Se.

In the series of isomeric selenophenothiophenes **113**, **114** (X = S, Y = Se), and **115** (X = S, Y = Se; X = Se, Y = S), it has been demonstrated by the 'pseudoatoms' method that the increase in the yield of the product of substitution in the α-position of the selenophene fragment with increase in the size of the attacking electrophile is associated with the stabilising interaction, specific to heterocycles, between the heteroatom and the attacking electrophile in the σ-complex formation stage.[236–238] The ^{77}Se chemical shifts in the series of isomeric selenophenothiophenes and thienothiophenes **113**–**115** have also been subjected to quantum-chemical analysis and correlations have been established between the shifts and the characteristics of the distribution of electron density obtained from quantum-chemical calculations.[238–240]

2. Five-membered heterocycles with two heteroatoms

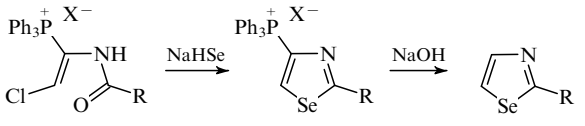
a. Selenazoles

A convenient method of synthesis of substituted selenazoles **116** is the interaction of the halo ketones **117** with compounds **118** containing a selenoamide fragment; the yields of the final products are then quantitative.[241–248]



X = Cl, Br; R = Alk, Ar, Het; R′ = NH_2, Alk, Ar.

A method based on the treatment of the phosphonium salts **120** with sodium hydrogen selenide has been used successfully in the synthesis of monosubstituted selenazoles **119**.[249–253]

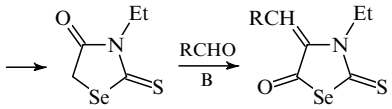


120 **119**

R = Alk, Ar, Het; X = Cl, ClO_4.

Sodium hydrogenselenide reacts with monochloroacetic acid and ethyl isothiocyanate to form the oxoselenazolethione **121**. In the presence of a base, it interacts with aldehydes, forming the substituted oxothionoselenazoles **122**, which are used in the synthesis of polymethine dyes.[254]

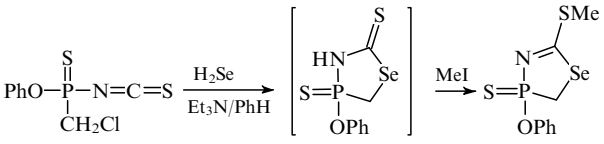
$$ClCH_2COOH + NaHSe + EtNCS \longrightarrow$$



121 **122**

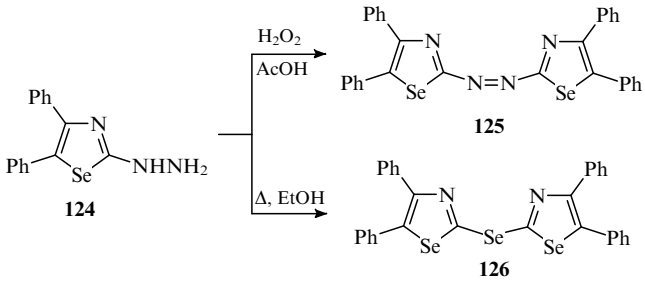
R = Ph, CH=CHPh, $CH{=}CHC_6H_4NMe_2$-4.

The five-membered heterocycle **123**, in which N, Se, and P atoms are present simultaneously, has been obtained in 26% yield as a result of the interaction of chloromethylisothiocyanatothiophosphonate with hydrogen selenide in benzene in the presence of an excess of triethylamine and subsequent treatment of the intermediate with MeI.[255]



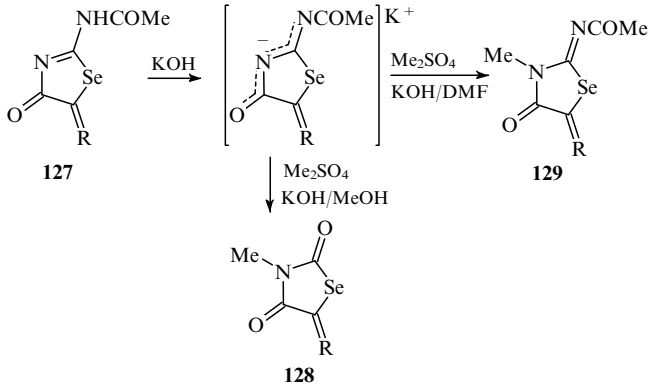
123

It has been reported [256] that the azoselenazole **125** is formed on oxidation of the hydrazinoselenazole **124** with a 30% solution of H_2O_2 in acetic acid, whilst refluxing in alcohol unexpectedly afforded the selenide **126**.[256]



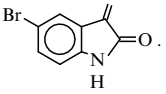
124 **125** **126**

The alkylation of 2-acetamidoselenazolinones **127** with methyl iodide or dimethyl sulfate in the presence of a 20% KOH solution has been investigated [257, 258] When the reaction is carried out in ethanol in the presence of a tenfold excess of the alkylating agent, it involves the nitrogen atom of the selenazolidine with formation of compounds **128**, while in the case where dimethylformamide (DMF) is used as the solvent, only compound **129** is formed regardless of the amount of the initial alkylating agent.

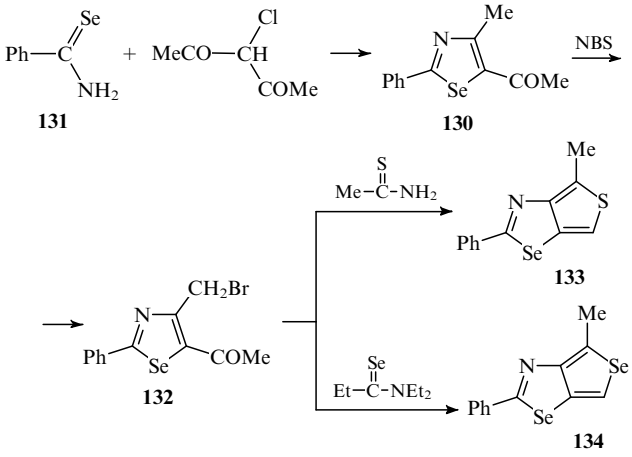


127 **129** **128**

R = H_2, PhCH, 4-$NO_2C_6H_4CH$, 4-$MeOC_6H_4CH$, 2-ClC_6H_4CH, 2,4-$Cl_2C_6H_3CH$,



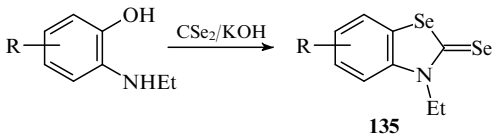
Methyl 4-methyl-2-phenyl-5-selenazolyl ketone **130**, obtained by the reaction of selenobenzamide **131** with 3-chloroacetylacetone, is brominated by *N*-bromosuccinimide with formation of the bromomethyl derivative **132**, which was used to obtain the condensed systems **133** and **134** containing the selenazole ring.[259]



131 **130** **133** **132** **134**

b. Condensed selenazoles

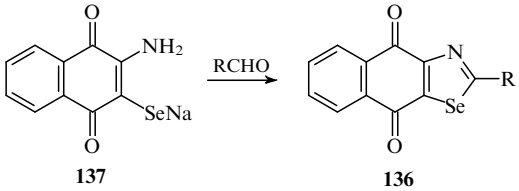
The benzoselenazoles **135**, which are used in the manufacture of photographic materials and dyes, have been obtained by the reaction of *o*-aminophenols with carbon diselenide.[260]



135

R = H, Alk, Ar, Ac, CN, OH, NH_2.

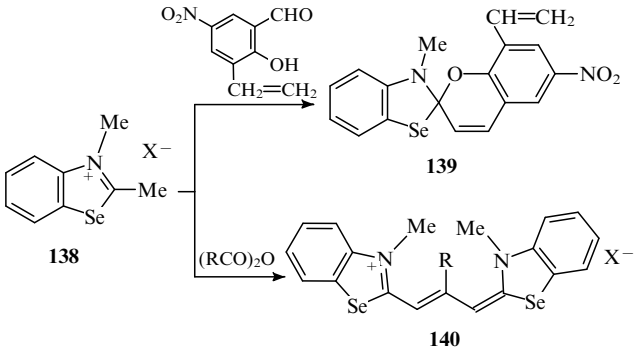
The dioxonaphthoselenazoles **136** have been obtained in 54%–68% yield by the reactions of the corresponding aminoselenolates **137** with aldehydes probably *via* a stage involving the formation of the corresponding azomethines.[261]



137 **136**

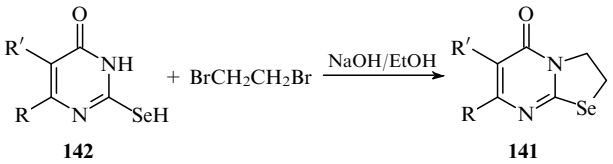
R = Alk, Ar, Het.

Among the chemical properties of benzoselenazoles, the ability of the 1,2-dimethylbenzoselenazolium cations **138** to condense with substituted salicylaldehyde to form the spiro-derivatives **139** [262] or with carboxylic acid anhydrides to form the selenocarbocyanines **140** has been described.[263, 264]



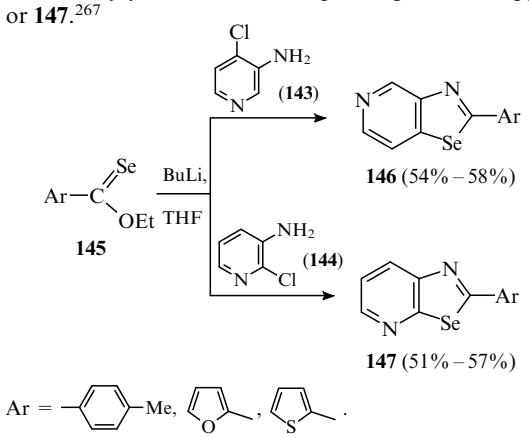
X = I, ClO_4, BF_4, OTs; R = H, Me, Et, CF_3, C_3F_7, Ph.

A promising method of synthesis of new condensed systems containing the selenazolidine ring **141**, involving the interaction of the selenols **142** with 1,2-dibromoethane, has been described.[265, 266]

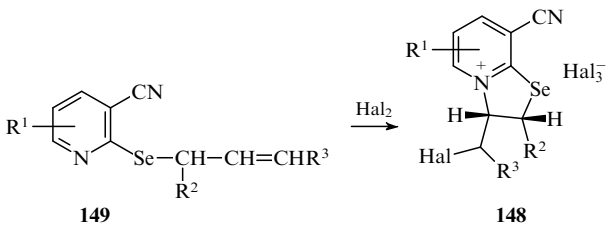


R = Me, R′ = H; R – R′ = $(CH_2)_4$.

The condensation of the chloroaminopyridine **143** or **144** with aromatic selenoesters **145** in the presence of BuLi in THF leads to satisfactory yields of the corresponding selenazolopyridines **146** or **147**.[267]

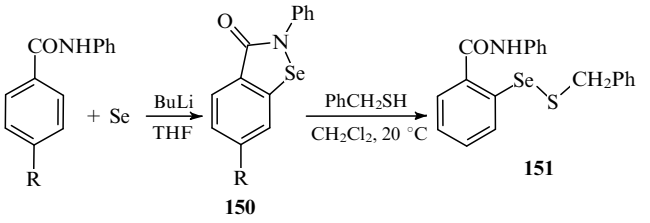


A general and convenient method of synthesis of selenazolopyridinium salts **148**, consisting in the cyclisation of substituted allyl 2-pyridyl selenides **149** on treatment with the halogens, has been developed.[268–277] It has been shown that this reaction proceeds *via* an intramolecular electrophilic heterocyclisation mechanism and is a highly stereoselective process: *trans*-quaternisation with formation of a *cis*-junction. The stereoselectivity is ensured by the synchronicity of the interaction in the transition state of the donor (electron pair of the pyridine nitrogen atom) and the acceptor (the halogen molecule) with the multiple bond.[278–283]



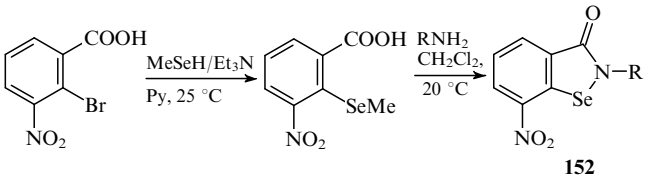
Hal = Br, I; R^1 = H, Alk, Ar, Het, OH, NH_2, CN; R^2, R^3 = H, Alk, cyclo-Alk.

The substituted benzoisoselenazolones **150**, which possess a high antitumour activity, have been obtained in yields ranging from 20% to 60% by treating arylamides with butyllithium and powdered selenium at –40 °C.[284, 285] On treatment with mercaptans, the benzoisoselenazole heterocycle of compound **150** (R = H) is ruptured with formation of compound **151**.[286]



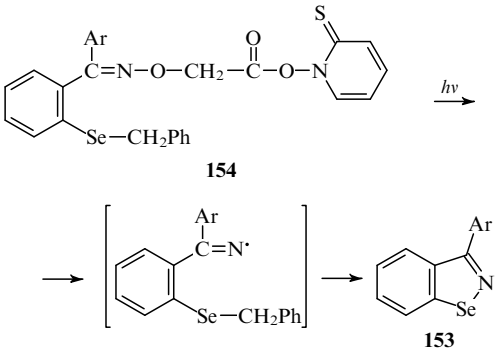
R = H, Me.

The benzoisoselenazolones **152** have been obtained from substituted benzoic acids in accordance with the following scheme:[287]

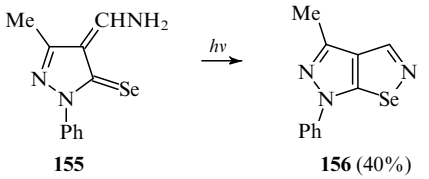


R = Bu^t, Ph, $PhCH_2$.

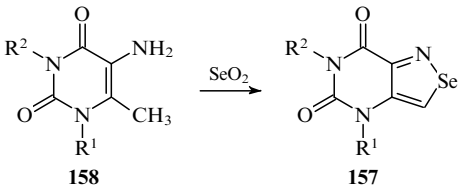
Arylbenzoisoselenazoles **153** are formed when compounds **154** are irradiated.[288]



Irradiation (λ = 330–480 nm) of the pyrazoloselenone **155** leads to a complex mixture of products, from which the condensed isoselenazole **156** was isolated in 40% yield.[289]



The synthesis of isoselenazolopyrimidines **157** by treating the substituted uracils **158** with selenium dioxide in dioxane at 110 °C has also been reported.[290]

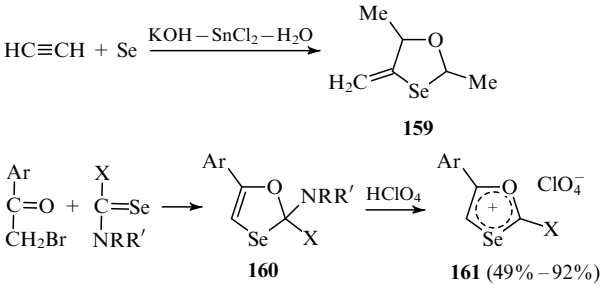


158 **157**

R^1, R^2 = Me, Et, Pr^i, Ph.

c. Oxaselenolanes

Two approaches to the synthesis of substituted 1,2-oxaselenolanes are known: the interaction of acetylene with elemental selenium in aqueous solution in the presence of tin chloride, which results in the formation of a mixture of seven selenium-containing products (one of them is 2,5-dimethyl-4-methylene-1,3-oxaselenolane **159**)[291] and the reaction of ω-bromoacetophenones with selenoamides leading to 1,3-oxaselenolanes **160**, characterised as the salts **161**.[292]

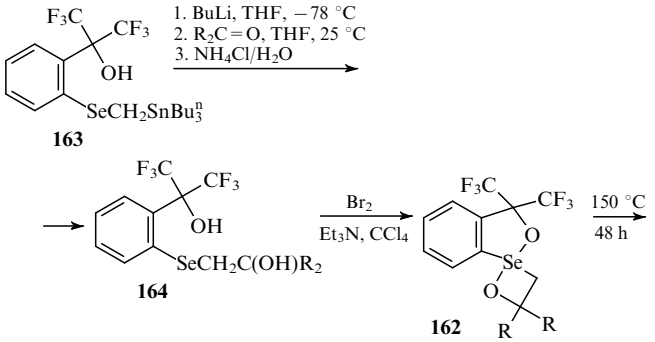


159

160 **161** (49% – 92%)

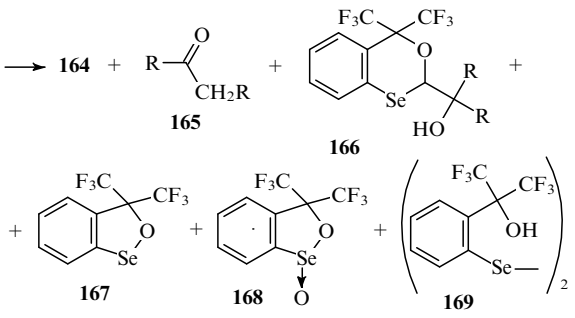
X = H, $N(Me)_2$, NHPh, OCH_2Ph, SPh; R,R′ = H, Alk;
Ar = Ph, 4-MeC_6H_4, 4-ClC_6H_4, 4-BrC_6H_4, 4-PhC_6H_4.

The 1,2-oxaselenolane system **162** has been obtained in accordance with the following scheme: treatment of the selenide **163** with 2.4 equiv. of BuLi (THF, −78 °C, 30 min) and subsequent reaction with a carbonyl compound leads to the β-hydroxyalkyl selenide **164**. The interaction of the latter with l equiv. of bromine in the presence of 2 equiv. of triethylamine (CCl_4, 25 °C, 3 h) gives compound **162**.[293] A complex mixture of products is formed in the thermolysis of the oxaselenolane **162**: the selenide **164** (12%), the ketone **165** (55%), the product **166** with an expanded ring (10%), the oxaselenolane **167** (16%), and 35% of a mixture of the selenoxide **168** and the diselenide **169**.



163

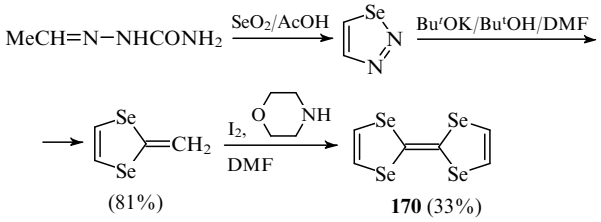
164 **162**



165 **166** **167** **168** **169**

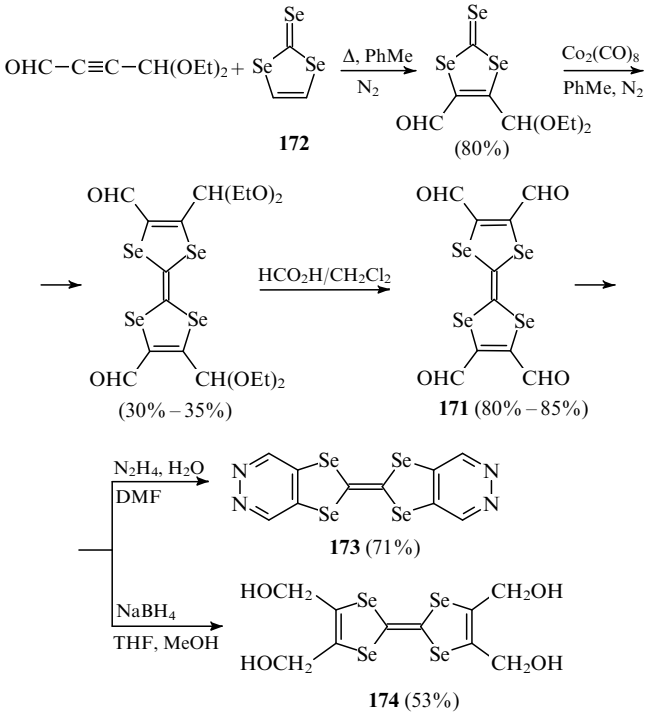
d. Selenafulvalenes

Tetrahetero(S, Se, Te)fulvalenes[294, 295] as well as their derivatives and analogues[296] have attracted much attention by investigators after the discovery of the conductivity[297] and superconductivity[297, 298] of their radical-cation salts and charge-transfer complexes. Cava and coworkers[299] described a simple and economical method of synthesis of tetraselenafulvalene **170** by means of the following scheme:



(81%) **170** (33%)

Later[300, 301] a method of synthesis of tetraformyltetraselenafulvalene **171** from 1,3-diselenole-2-selenone **172**[302] was developed. It is based on the thoroughly investigated coupling reaction of 1,3-dithioles or 1,3-diselenoles.[303–307] Treatment of the fulvalene **171** with hydrazine leads to the bispyridazino-derivative **173**[300, 308] whilst reduction with sodium tetrahydroborate affords the tetraselenafulvalene **174**.[300]



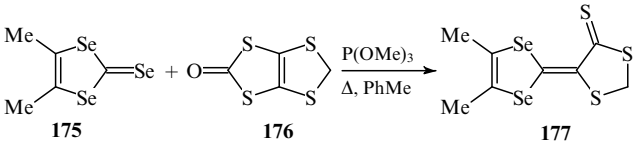
172 (80%)

(30% – 35%) **171** (80% – 85%)

173 (71%)

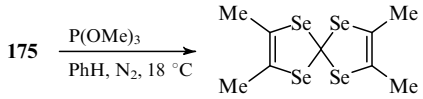
174 (53%)

Trialkyl phosphites are used fairly widely as condensing agents for the synthesis of asymmetric selenium-containing fulvalenes.[309–313] Thus the interaction of 4,5-dimethyl-1,3-diselenole-2-selenone **175** with the ketone **176** in boiling toluene in the presence of trimethyl phosphite under an argon atmosphere resulted in the formation of 4,5-dimethyl-2-(4-thioxo-1,3-dithiolan-5-ylidene)-1,3-diselenole **177** in 28% yield.[311]



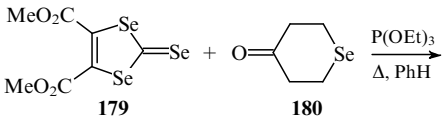
175 **176** **177**

In the absence of the ketone **176**, the interaction of the 1,3-diselenole-2-selenone **175** with trimethyl phosphite in benzene at 18 °C under an argon atmosphere leads to a spiro-compound — 2,3,7,8-tetramethyl-1,4,6,9-tetraselenaspiro[4,4]nona-2,7-diene **178** — the structure of which has been confirmed by XRD data.[313]

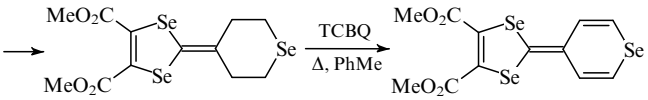


178

The reaction of 4,5-bis(methoxycarbonyl)-1,3-diselenole-2-selenone **179** with 4-oxoselenane **180** on refluxing in benzene in the presence of triethyl phosphite leads to the cross-coupling product **181** in 23% yield. The product is dehydrogenated on heating in toluene with tetrachlorobenzoquinone (TCBQ).[314]

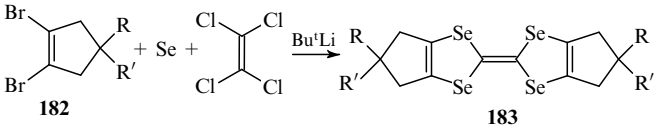


179 **180**



181

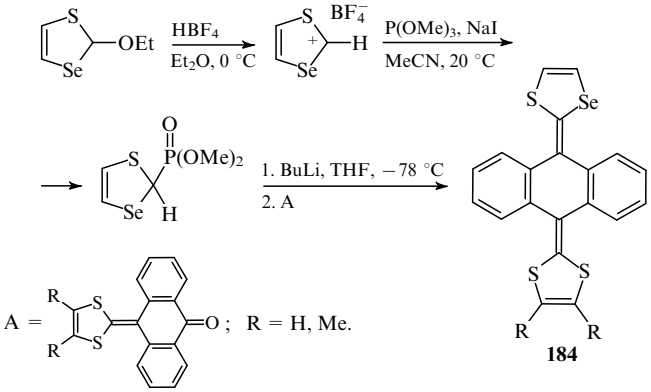
The substituted tetraselenafulvalenes **183** have been synthesised in 5%–10% yields by the interaction of the dibromocyclopentenes **182**, selenium, and tetrachloroethene in the presence of Bu^{t}Li.[310]



182 **183**

R, R′ = H, Me

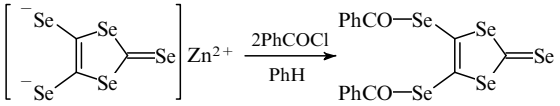
The preparation of highly conjugated selenatrithiafulvalene derivatives **184** *via* the following scheme has also been reported:[312]



A = ; R = H, Me.

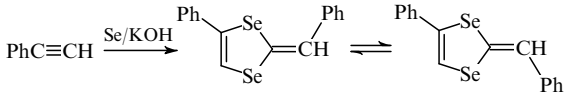
184

Acylation of compound **185** with benzoyl chloride in benzene leads to a 33% yield of the dibenzoyl derivative **186**.[315]



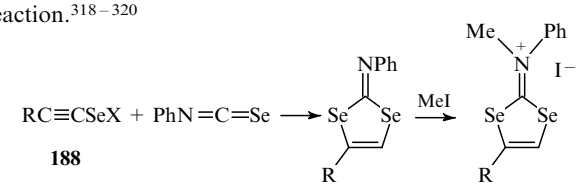
185 **186**

A convenient method of synthesis of the substituted 1,3-diselenole **187** in the form of a mixture of isomers by the interaction of phenylethyne with elemental selenium in the presence of an aqueous KOH solution in both aprotic dipolar solvents[316] and under the conditions of phase transfer catalysis[317] has been developed.



(*Z*)-**187** (*E*)-**187**

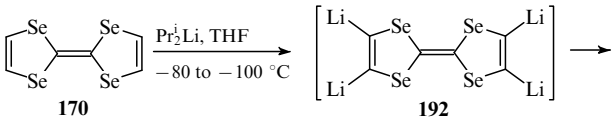
It has also been shown that the ethynylselenolates **188** enter into an anionic 1,3-cycloaddition reaction with phenyl isoselenocyanate to form the 1,3-diselenoles **189**, electron-donating substituents accelerating and electron-accepting ones retarding the reaction.[318–320]



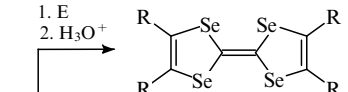
188 **189**

R = C_6H_5, 4-MeC_6H_4, 4-$MeOC_6H_4$, 4-ClC_6H_4, 4-$NO_2C_6H_4$;
X = Li, K.

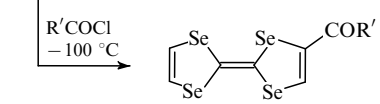
Changes permitting the synthesis of only monosubstituted tetraselenafulvalenes **191** were subsequently introduced into this reaction. For this purpose, the tetraselenafulvalene **170** is treated at −100 °C with 4 equiv. of lithium diisopropylamide in THF and the resulting tetralithium derivative **192** is then treated with a deficiency of an electrophile.[321]



170 **192**



190



191

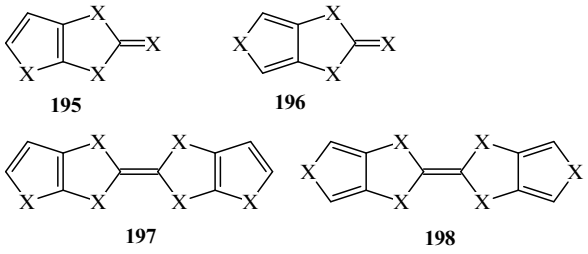
R = SPh, SePh, CO_2Me, COOH;
R′ = Me, Pr^n, n-$C_{13}H_{27}$, n-$C_{15}H_{31}$.

The metallation of the tetraselenafulvalene **170** with lithium diisopropylamide at temperatures between −80 and −100 °C followed by the addition of an excess of an electrophile (Ph_2S_2, Ph_2Se_2, $ClCO_2Me$, or CO_2) leads to the corresponding tetrasubstituted tetraselenafulvalenes **190**.[322]

It is of interest that treatment of compound **170** with Bu^{n}Li leads to the overall degradation of the molecule (only compounds **193** and **194** were detected mass-spectrometrically).[322]

$BuCH_2{=}CH_2SeBu$ **193** $BuSeCH{=}CHSeBu$ **194**

It has been shown by CNDO/CI quantum-chemical calculations with a modified set of spectroscopic parameters that heteroaromatic asymmetric thiones and selenones **195** and **196** as well as the heterofulvalenes **197** and **198** based on them have smaller ionisation potentials and energies of the lowest electronic transitions than their symmetrical isomers. An increase in the size of the thiones and selenones by their annelation to heterocyclic fragments alters the ionisation potentials and the electronic transition energies insignificantly, which can be explained by the small contributions of the atomic orbitals of these fragments to the highest occupied molecular orbital, whereupon the main contribution to the ionisation potentials comes from the $4p\pi$ orbitals of the heteroatoms in the 2,3-positions of the five-membered heterocycle as well as the $2p\pi$ orbitals of the carbon atoms forming the double bond.[323–332]

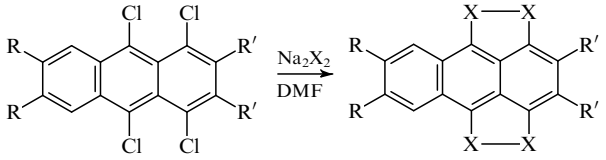


195 **196** **197** **198**

X = S, Se.

e. Other five-membered heterocycles with two selenium atoms

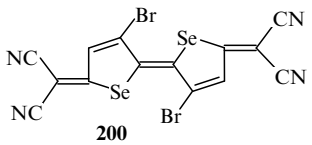
In order to obtain 'organic metals' with a high electrical conductivity, a method of synthesis of new types of electron donors — dimethyl- and tetramethyl-anthra[1,9-*cd*:4,10-*c′d′*]bis-1,2-dithioles and the corresponding diselenoles and ditelluroles **199** — *via* the following scheme has been developed:[333, 334]



199

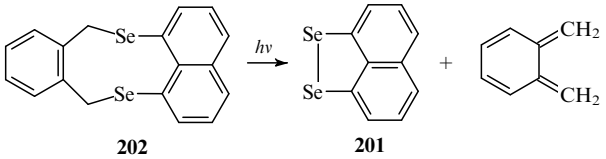
X = S, Se, Te; R, R′ = H, Me.

Using 7,7,8,8-tetracyanoquinodimethane as well as its 2,3,5,6-tetrafluoro-, 2,5-dimethyl-, and 2,5-dimethoxy-derivatives, 2,5-bis(dicyanomethylene)-$\Delta^{2,2'}$-di(3-thiolene), and 3,3′-dibromo-5,5′-bis(dicyanomethylene)-$\Delta^{2,2'}$-di(3-selenolene) **200** as electron acceptors, charge-transfer complexes with electrical conductivities ranging from 8 to 10^{-9} S cm^{-1} were obtained from compounds **199**.[333]



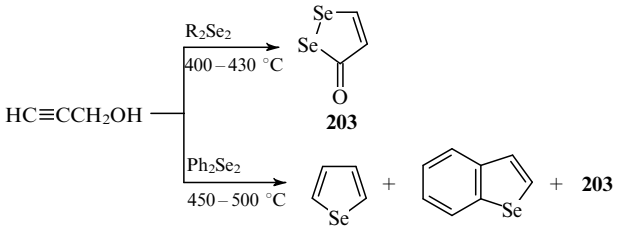
200

The synthesis of naphtho[1,8-*c*,*d*]-1,2-diselenole **201** on irradiation of 8,13-dihydrobenzo[*g*]naphtho[1,8-*b*,*c*]-1,5-diselenonine **202** has been reported.[335]



202 **201**

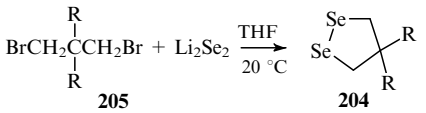
1,2-Diselenol-3-one **203** is formed in a high yield as a result of the gas-phase reaction of propargyl alcohol with dialkyl diselenides at 400–430 °C.[336, 337] The replacement of the dialkyl diselenide by diphenyl diselenide in this reaction lowers the yield of the diselenolone **203** and leads to the formation of selenophene and benzoselenophene together with the latter.[337]



203

R = Me, Et, Pr^i.

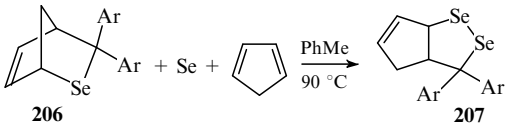
The cyclic five-membered 1,2-diselenides **204** have been obtained in a high yield (76%–98%) as a result of the treatment of 1,3-dibromoalkanes **205** with lithium diselenide in THF.[338, 339]



205 **204**

R = H, Me.

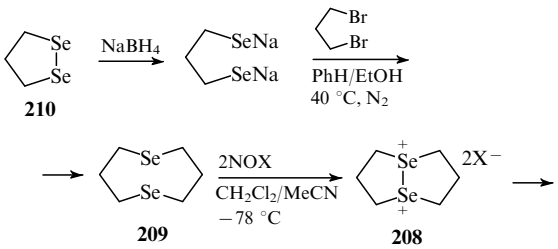
The bicyclic diselenides **207** have been obtained by treating 3,3-diaryl-2-selenabicyclo[2.2.1]hept-5-enes **206** with elemental selenium and cyclopentadiene in toluene.[340]



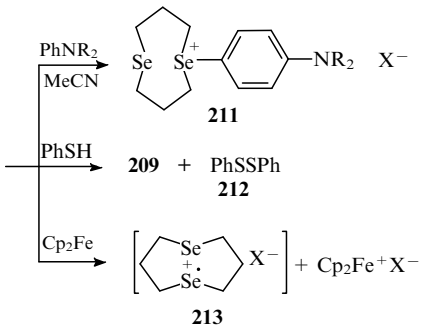
206 **207**

Ar = Ph, 4-MeC_6H_4, 4-FC_6H_4, 4-ClC_6H_4.

A series of studies have been devoted to the reactivity of the salts of the dication **208** obtained by the two-electron oxidation of 1,5-diselenacyclooctane **209** with $NOPF_6$ (or $NOBF_4$).[341–344] The initial bisselenide **209** was obtained by treating 1,3-diselenolane **210** with sodium tetrahydroborate and by the subsequent reaction of the resulting sodium propane-1,3-diselenolate with 1,3-dibromopropane in a benzene–ethanol mixture at 40 °C under a nitrogen atmosphere using the high dilution technique. The interaction of the dication **208** with aniline or *N*,*N*-dimethylaniline in anhydrous MeCN at room temperature under an argon atmosphere leads to the corresponding *para*-substituted selenonium salt **211**, whereas the reaction with thiophenol is accompanied by the formation of the disulfide **212** (92%) and 1,5-diselenacyclooctane **209**.[341, 343] It has also been shown that one-electron reduction with formation of the radical-cation **213** and the ferrocenium cation occurs in the interaction of the dication **208** with ferrocene in anhydrous MeCN at −20 °C under an argon atmosphere.[342]



210 **209** **208**



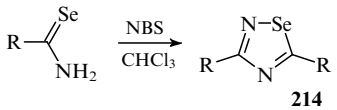
X = PF_6, BF_4; R = H, Me.

3. Five-membered heterocycles with three heteroatoms

a. Selenadiazoles

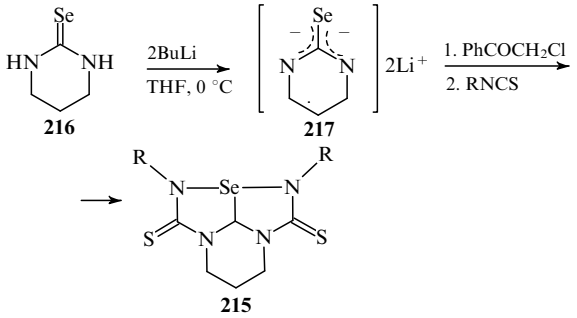
Various methods have been used to synthesise isomeric selenadiazoles.

Thus 1,2,4-selenadiazoles **214** have been obtained in 32%–87% yield by treating selenoamides with *N*-bromosuccinimide in chloroform.[345]



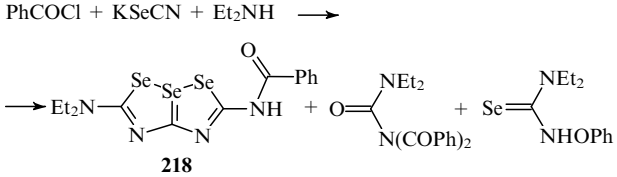
R = Ph, 4-MeC_6H_5, 4-ClC_6H_5, $MeCH_2CH_2$, $Me(CH_2)_3CH_2$, $Me(CH_2)_5CH_2$, Me_2N, $PhCH_2S$.

Condensed derivatives of 1,2,4-selenadiazoles — selenatetraazapentalenes **215** — have been obtained by treating selenourea **216** with 2 equiv. of butyllithium in THF at 0 °C under a nitrogen atmosphere and the subsequent reaction of the resulting dianion **217** with phenacyl chloride and alkyl isothiocyanate.[346–348]

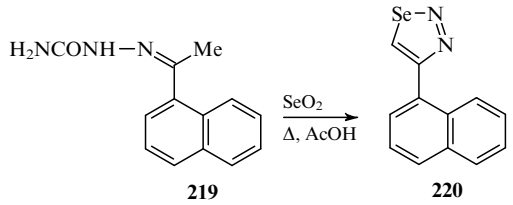


R = Me, Et, $CH_2{=}CHCH_2$.

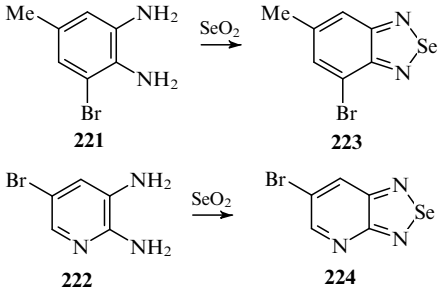
The substituted triselenadiazapentalene **218** has been synthesised in a low yield (4.8%), together with other products, by the interaction of benzoyl chloride, potassium selenocyanate, and diethylamine in acetone at room temperature.[349]



A convenient method of synthesis of 1,2,3-selenadiazoles has been developed. It consists in the treatment of the semicarbazones of aromatic and carbocyclic ketones with selenium dioxide.[350–354] Thus treatment of the semicarabazone **219** with SeO_2 in glacial acetic acid results in the formation of 4-(1-naphthyl)-1,2,3-selenadiazole **220** in 65% yield.[350]

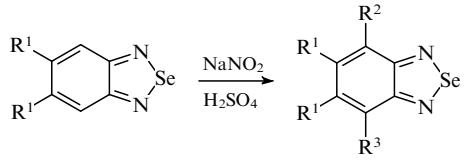


Selenium dioxide is a convenient reagent also in the synthesis of 2,1,3-selenadiazoles from aromatic and heteroaromatic diamines.[355–358] For example, treatment of the substituted phenylenediamine **221** and 5-bromo-2,3-diaminopyridine **222** with selenium dioxide in dioxane leads to the 2,1,3-selenadiazoles **223**[355] and **224**[356], respectively.



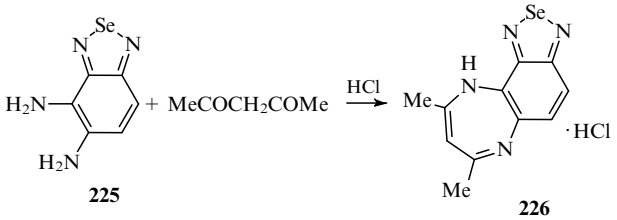
In the synthesis of condensed 2,1,3-selenadiazoles, H_2SeO_3[359–361] and $SeCl_4$[362] have also been used as sources of selenium.

In the nitration of 5,6-disubstituted benzo-2,1,3-selenadiazoles with sodium nitrite in H_2SO_4, an increase in the deactivating influence of the substituent on their reactivity is observed in the series Me < Cl < NO_2.[357]

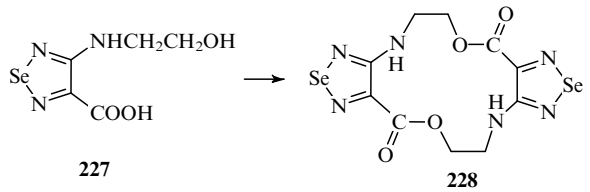


R^1 = Me, $R^2 = R^3 = NO_2$; R^1 = Cl, $R^2 = NO_2$, R^3 = H.

4,5-Diaminobenzo-2,1,3-selenadiazole **225** reacts with acetylacetone in the presence of concentrated HCl to form the hydrochloride of 2,4-dimethyl-1,5-diazepino[5,4-*e*]benzo-2,1,3-selenadiazole **226** in 88% yield.[363]



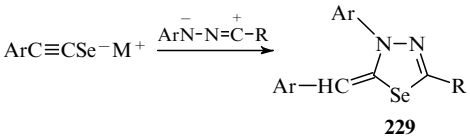
The dimerisation of 2,1,3-selenadiazole **227** to the 14-membered lactone **228** has been reported.[364]



Photoelectron He(I) spectra of benzo-2,1,3-selenadiazole and its perfluoro-derivatives have been measured and interpreted on the basis of calculations by the MNDO method, the π-fluoro-effect, and the analysis of the vibrational structure and relative

band intensities.[365] The reduction of benzo-2,1,3-selenadiazoles with 57% HI [366] and NaH,[367] as well as the ability of 4- and 5-(β-amino-β-carboxy)ethylbenzo-2,1,3-selenadiazole to form complexes with Cu(II) and Pt(II) salts have been investigated.[368]

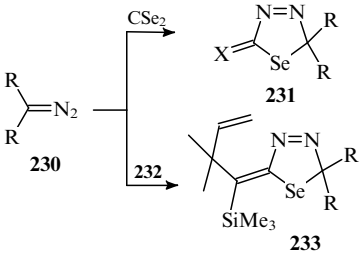
Derivatives of quaternary isomeric selenadiazole — 1,3,4-selenadiazolidines **229** — have been obtained in 70% – 80% yields by the interaction of acetylenic alkali metal selenolates with nitrilimines.[369 – 375]



229

M = Li, Na, K; R = Ar, Ac, CO_2Et.

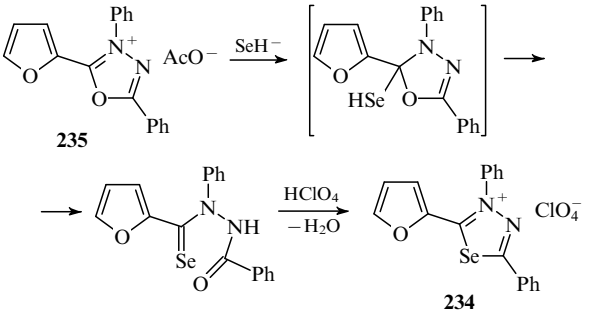
Diazoalkanes **230** react with an excess of carbon diselenide at 80 °C in toluene to form a mixture of substituted 1,3,4-selenadiazolines **231**, which have been separated by thin-layer chromatography (TLC).[376] The introduction of selenoketene **232** into the reaction with the diazoalkanes **230** leads to a complex mixture of products, among which substituted 1,3,4-selenadiazolines **233** have been detected.[377]



230 **231** **233**

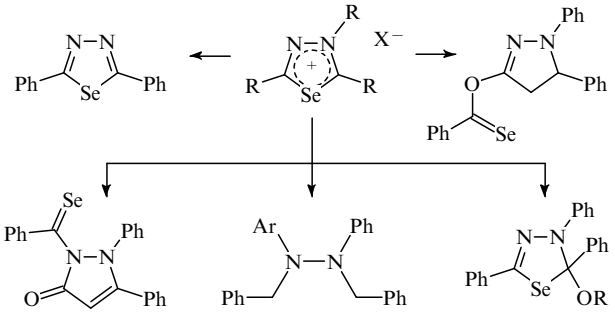
R = Bu^t, Ar; X = R_2C, $R_2C{=}NN$, RCH;
232 = $CH_2{=}CHCMe_2C(SiMe_3){=}C{=}Se$.

The 1,3,4-selenadiazolium salt **234** has been obtained by treating the oxadiazolium acetate **235** with alkali metal hydrogen selenides and subsequent acid cyclodehydration of the intermediate.[378, 379]



235 **234**

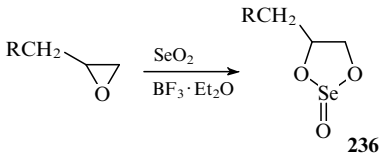
On treatment with potassium acetate in acetic anhydride, 1,3,4-selenadiazolium salts recyclise to various derivatives.[379 – 382]



X = I, ClO_4, AcO, TsO; R = Alk, Ar, Het.

b. Other five-membered heterocycles with three heteroatoms

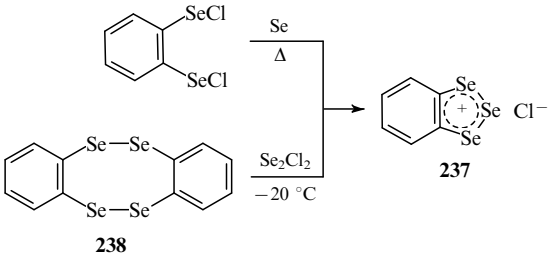
The interaction of equimolar amounts of selenium dioxide and epichlorohydrin or epoxypropane in the presence of boron trifluoride etherate in dioxane at 20 °C under a nitrogen atmosphere is a convenient method of synthesis of substituted 1,3,2-dioxaselenolanes **236**.[383]



236

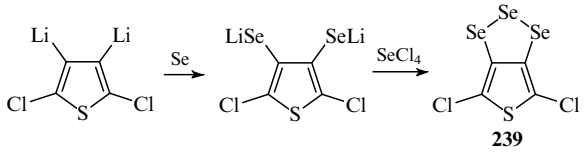
R = H, Cl.

Benzo-1,2,3-triselenolium chloride **237** has been obtained as a result of the interaction of 1,2-di(chloroseleno)benzene with elemental selenium and also by treating dibenzotetraselenocine **238** with Se_2Cl_2 in dichloromethane.[384]



237 **238**

Thieno-1,2,3-triselenole **239** has been obtained from 2,5-dichloro-3,4-dilithiothiophene in accordance with the following scheme:[385]



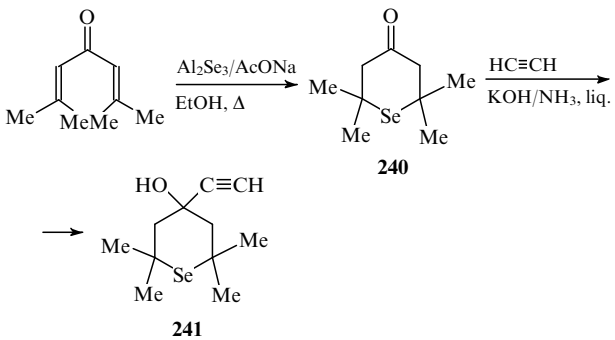
239

V. Six-membered heterocycles

Hydrogen selenide or compounds which readily hydrolyse in the reaction medium to hydrogen selenide, as well as alkaneselenols, selenium dioxide, selenium tetrahalides, and, to a lesser extent, elemental selenium are most often used as sources of selenium in the synthesis of six-membered selenium-containing heterocycles.

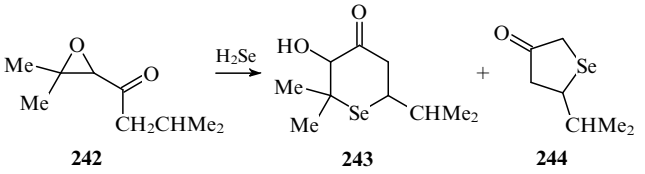
1. Selenanes

2,2,6,6-Tetramethylselenan-4-one **240** has been obtained by the reaction of phorone with a mixture of aluminium selenide and sodium acetate in 90% ethanol. Its ethynylation under the conditions of the Favorskii reaction in liquid ammonia with powdered potassium leads to the selenanol **241**.[386]



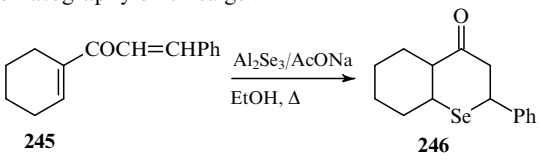
240 **241**

The interaction of the epoxy ketone **242** with hydrogen selenide in the presence of $SnCl_4$ or toluene-*p*-sulfonic acid in acetonitrile at 20 °C leads to a mixture of selenane derivatives **243** (55%) and tetrahydroselenophene **244** (12%).[111]

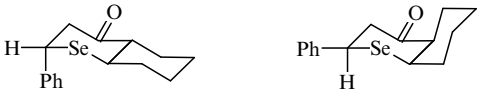


242 **243** **244**

The condensation of the dienone **245** with hydrogen selenide, formed on heating a mixture of aluminium selenide and sodium acetate in 90% ethanol, leads to 2-phenyl-1-selenabicyclo[4.4.0]-decan-4-one **246** as a mixture of four stereoisomers, from which the two individual isomers **246a** and **246b** were isolated by column chromatography on silica gel.[387–389]

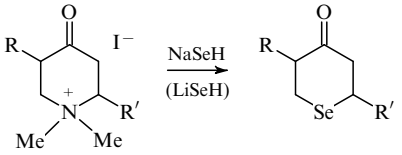


245 **246**



246a **246b**

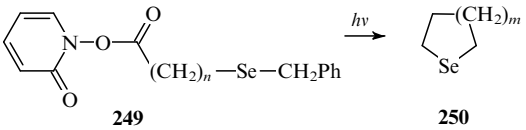
Substituted selenan-4-ones **248** have been synthesised by treating the methiodides of *N*-methylpiperidinones **247** with sodium or lithium hydrogenselenide in alcohol.[390–392]



247 **248**

R, R′ = H, Me.

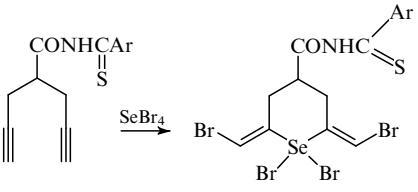
The irradiation of a benzene solution of the selenides **249** with light from a tungsten lamp leads to the selenanes **250** in 50%–79% yields.[393]



249 **250**

n = 3–5; m = 1–3.

On treatment with selenium tetrabromide, compound **251** gives rise to the selenane dibromide **252**.[394]

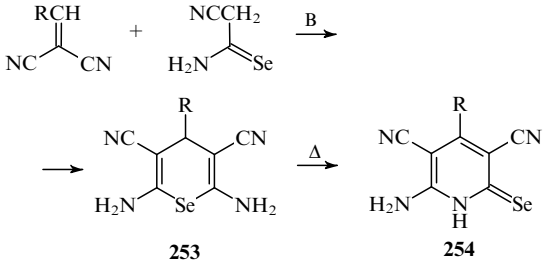


251 **252**

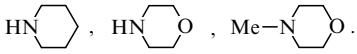
Apart from the ethynylation of the selenanone **240** indicated above,[386] among the chemical reactions the reduction of 2,5-dimethylselenan-4-one with lithium tetrahydroborate[395] as well as the phosphorylation of selenan-4-one with dialkyl phosphites[396, 397] have been described.

2. Selenopyrans

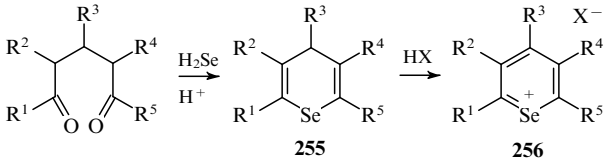
A convenient method has been developed in recent years for the synthesis of functionally substituted 4-aryl(heteroaryl)-2,6-diamino-3,5-dicyano-(4*H*)-selenopyrans **253**. It consists in the interaction of cyanoselenoacetamide with aryl(heteroaryl)idenemalononitriles at 20 °C in the presence of organic bases.[398–405] On refluxing in alcohol, the selenopyrans **253** recyclise to the pyridineselenones **254**.



253 **254**

R = Ar, Het; B = 

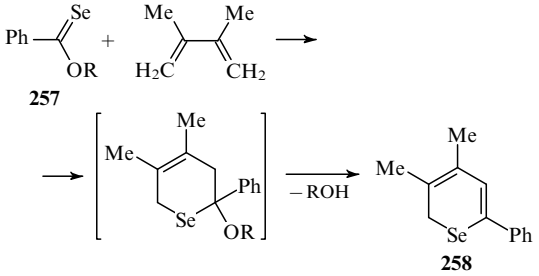
Another method of synthesis of substituted (including condensed) selenopyrans **255**, which readily form the selenopyrylium salts **256**, involves the interaction of 1,5-diketones with hydrogen selenide in acid media.[406–413]



255 **256**

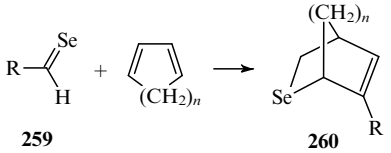
R^1–R^2 = $(CH_2)_3$, $(CH_2)_4$; R^1, R^5 = Ar; R^2, R^4 = H, Me; R^3 = H, Ar; X = Cl, BF_4, ClO_4.

The cycloaddition of selenocarbonyl compounds to 1,3-dienes has also been used in the synthesis of alkyl(aryl)seleno- pyrans.[414–419] Thus the interaction of the selenoesters **257** with 2,3-dimethylbuta-1,3-diene leads to the 3,4-dimethyl-6-phenyl-(2*H*)-selenine **258**,[117] while the interaction of selenoaldehydes **259** with cyclic dienes affords the bicyclic adducts **260**.[420]



257 **258**

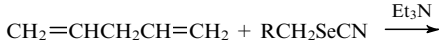
R = Et, Bu;

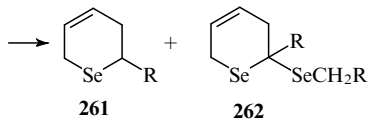


259 **260**

R = CO_2Me, COPh, CN; n = 1, 2.

Selenocyanates react with 1,4-dienes, forming dihydroselenopyrans **261** with a small admixture of the selenides **262**.[419]

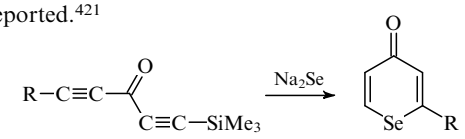




261 **262**

R = CN, COAr.

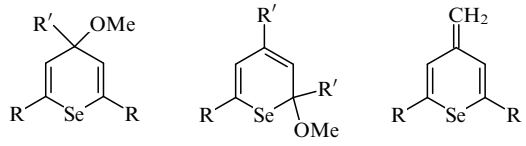
The synthesis of the selenopyranones **263** by the interaction of the unsaturated ketones **264** with sodium selenide has been reported.[421]



264 **263**

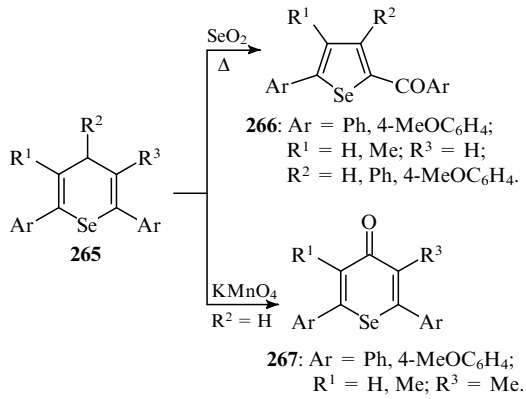
R = Ph, $SiMe_3$.

It has been demonstrated by ^{1}H NMR spectroscopy that, depending on the number and nature of the substituents in the heteroaromatic cation, the interaction of selenopyrylium salts **256** with sodium methoxide results in the formation of various reaction products: (4*H*)-selenopyrans, a mixture of (4*H*)- and (2*H*)-selenopyrans with various proportions of the isomers, or 4-methyleneselenopyrans.[422]



R, R′ = H, Me, Bu^t, Ph, 4-$MeOC_6H_4$;
X = BF_4, Cl, Br.

The oxidation of the selenopyrans **265** (R^3 = H) with selenium dioxide in pyridine leads to the substituted selenophenes **266**.[423, 424] On the other hand, the oxidation of the selenopyrans **265** (R^2 = H) with potassium permanganate in acetone or acetonitrile results in the formation of the 4-selenopyranones **267** in a high yield.[423, 425, 426]



265

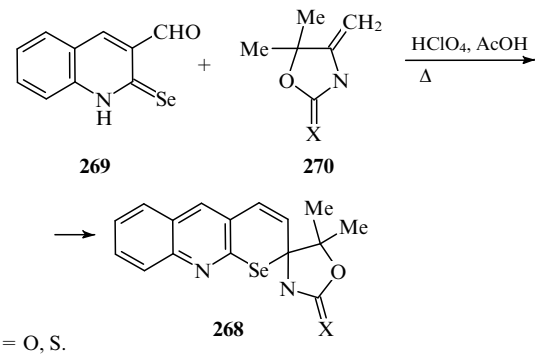
266: Ar = Ph, 4-$MeOC_6H_4$;
R^1 = H, Me; R^3 = H;
R^2 = H, Ph, 4-$MeOC_6H_4$.

267: Ar = Ph, 4-$MeOC_6H_4$;
R^1 = H, Me; R^3 = Me.

The electrochemical reduction of the selenopyrans **255**[427, 428] as well as the dimerisation[429] and dissociation of the salts **256** in aqueous solutions[430] have also been investigated. In addition, a study has been made of the nucleophilic substitution in the series of selenopyrans[422, 431, 432] and of certain other reactions.[433–436]

3. Condensed selenopyrans

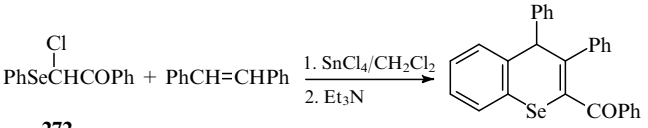
The spiroselenopyrans **268**, incorporating a quinoline fragment condensed with the selenopyran ring, are formed when a mixture of 3-formyl-(1*H*)-quinoline-2-selenone **269** and the methylene bases **270** is refluxed in glacial acetic acid in the presence of catalytic amounts of perchloric acid.[437–439]



269 **270** **268**

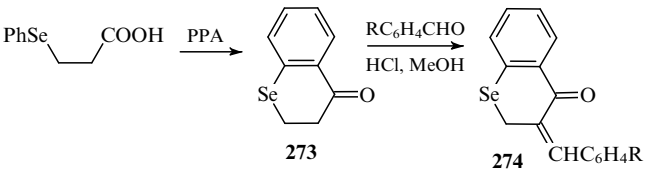
X = O, S.

The trisubstituted selenochroman **271** has been obtained by the reaction of α-chlorophenacyl phenyl selenide **272** with *trans*-stilbene in anhydrous CH_2Cl_2 at −20 °C under an argon atmosphere in the presence of zinc chloride and subsequent treatment of the reaction mixture with triethylamine.[440]



272 **271**

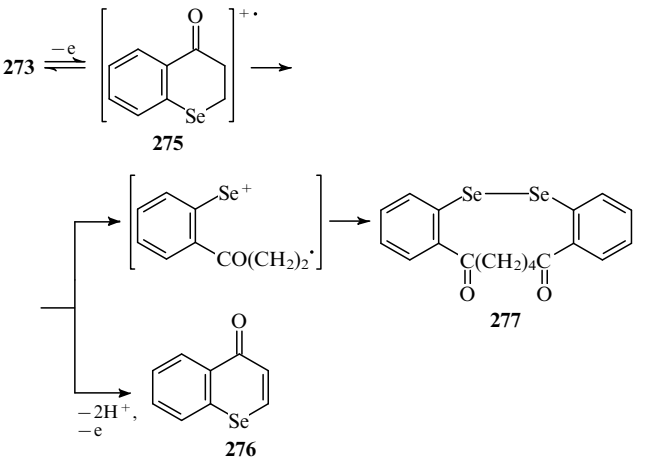
In the presence of polyphosphoric acid (PPA), 3-phenylselenopropionic acid affords the selenochroman-4-one **273**, the interaction of which with aromatic aldehydes in the presence of concentrated HCl in methanol leads to 3-arylideneselenochroman-4-ones **274** in 30% – 60% yield.[441, 442]



273 **274**

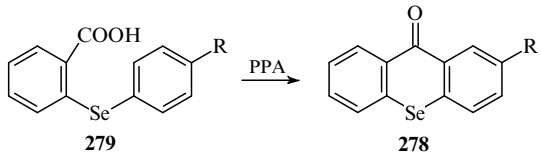
R = 4-MeC_6H_4, 4-$MeOC_6H_4$, 4-$N(Me)_2C_6H_4$, 4-ClC_6H_4, 4-BrC_6H_4, 4-$NO_2C_6H_4$.

Derivatives of the selenochromanone **273** are also formed from oxo-derivatives of benzo-1,3-selenazine on treatment with iron or zinc in acetic acid.[443] The anodic oxidation of the selenochromanone **273** has been studied and it has been shown that the resulting radical-cation **275** can react *via* two parallel routes: deprotonation with formation of selenochromone **276** and homolytic dissociation of the C – Se bond with subsequent dimerisation and formation of the diselenide **277**.[444]



275 **277** **276**

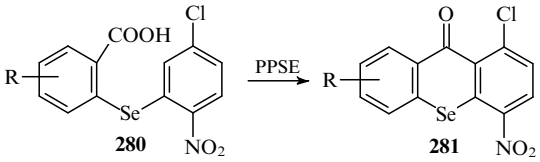
It has also been shown that the reactions of quaternised selenochromans, isoselenochromans, selenochromanones, and isoselenochromanones with Grignard reagents or metallic magnesium are accompanied by the homolytic dissociation of the C–Se bond and the formation of selenides of the benzene series.[445,446] The intramolecular Friedel–Crafts cyclisation of 2-arylselenobenzoic acids **279** in the presence of PPA has been used for the synthesis of the selenoxanthenones **278**.[447,448]



279 **278**

R = H, Cl.

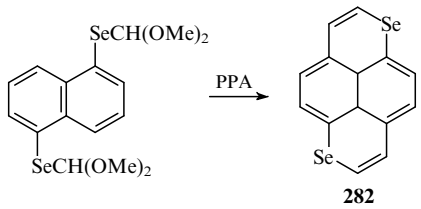
Later a series of substituted selenoxanthenones **281** were obtained by the analogous cyclisation of the arylselenobenzoic acids **280** using trimethylsilyl polyphosphate (PPSE) as the catalyst.[449]



280 **281**

R = H, 4-MeO, 5-MeO, 3,4-OCH_2O.

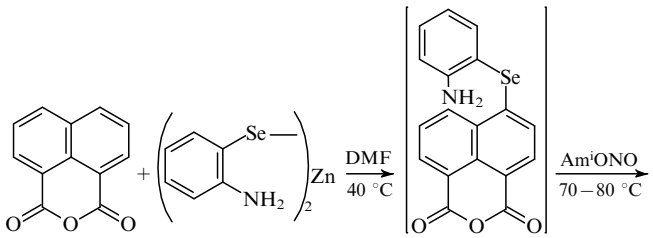
Polyphosphoric acid has been used as a catalyst also in the synthesis of 1,6-diselenapyrene **282**.[450]



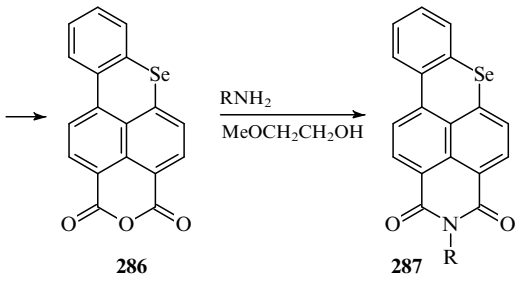
282

Certain properties of selenoxanthene derivatives have been studied: carboxylation,[451] interaction with nucleophiles,[452] and electrochemical reduction.[453]

The reaction of naphthalic anhydride **283** with the zinc salt of *o*-aminophenyl selenide **284** in DMF leads to the anhydride **285**, which is treated, without isolation, with isoamyl nitrite and the anhydride of benzo[*k*,*l*]selenoxanthene-3,4-dicarboxylic acid **286** is obtained in 60% yield.[454] A series of imides **287** have been obtained in 64%–94% yield in the interaction of the anhydride **286** with aliphatic amines on refluxing in 2-methoxyethanol.



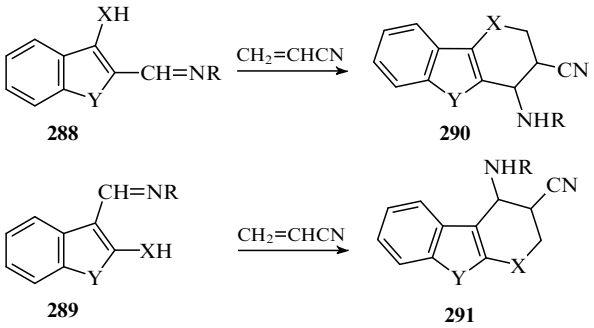
283 **284** **285**



286 **287**

R = Bu, C_6H_{11}, $C_{18}H_{37}$, CH_2CH_2COOH.

Benzo[*b*]furan and benzo[*b*]thiophene thiolo- and selenoloaldimines **288** and **289** react with acrylonitrile to form the condensed thio- and seleno-pyrans **290** and **291** in yields up to 87%.[455–459]

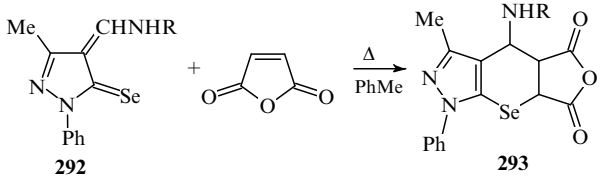


288 **290**

289 **291**

X = S, Se; Y = O, S; R = Ph, 4-MeC_6H_4, 4-$MeOC_6H_4$.

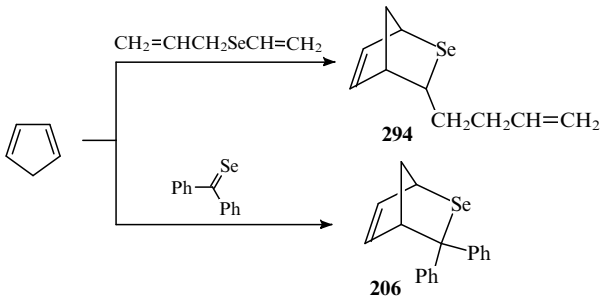
The cycloaddition of maleic anhydride to the pyrazoloselenones **292** on refluxing in toluene leads to the anhydrides of 4-arylamino-3-methyl-1-phenyltetrahydroselenopyrano[2,3-*d*]-pyrazole-5,6-dicarboxylic acid **293** in yields up to 98%.[460]



292 **293**

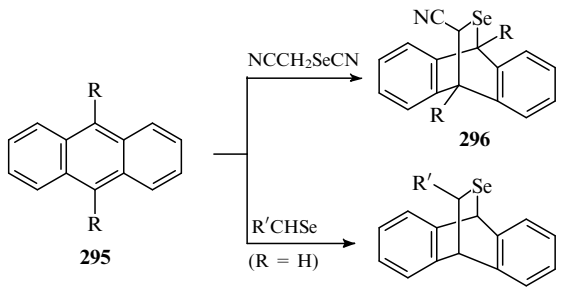
R = H, 3-MeC_6H_4, 4-MeC_6H_4, 3-$MeOC_6H_4$, 4-$MeOC_6H_4$, 3-$NO_2C_6H_4$.

The cycloaddition of cyclopentadiene to selenium-containing dienophiles has been used in the synthesis of bicyclic derivatives of 2-selenabicyclo[2.2.1]octene **206**[461] and **294**.[462]



294 **206**

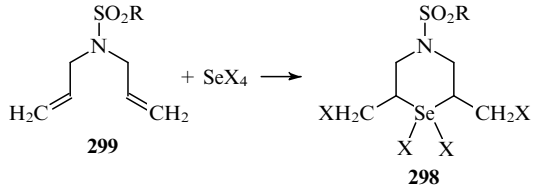
The selenopyran derivatives **296**[463] and **297**[464] have been synthesised from substituted anthracenes **295** and selenium-containing dienophiles.



R = H, Me; R′ = Alk_F.

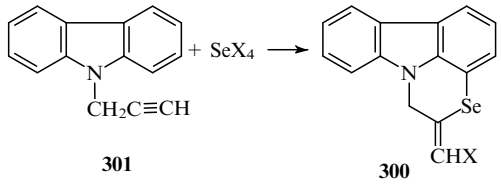
4. Six-membered heterocycles with two heteroatoms

Substituted perhydro-1,4-selenazines **298** have been synthesised by the reaction of selenium tetrahalides or phenylselenium trihalides with 1,6-heterodienes **299**.[465, 466]



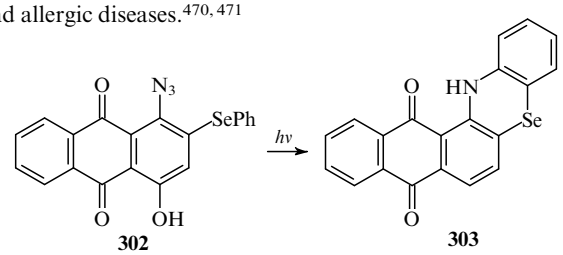
R = Ph, 4-MeC_6H_4; X = Br, Cl.

Selenium tetrahalides have also been used to obtain condensed analogues of 1,4-selenazines — 2-halomethylidene-1,4-selenazino[2,3-*gh*]carbazoles **300**.[467, 468] It has been shown that the reaction proceeds *via* the electrophilic addition of SeX_4 to the triple bond and the subsequent electrophilic attack on the nucleus of the carbazole **301** with formation of the condensed system **300**.

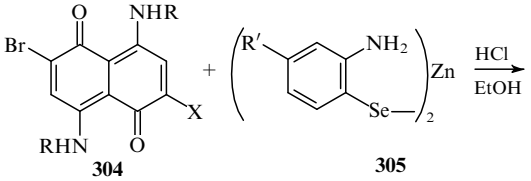


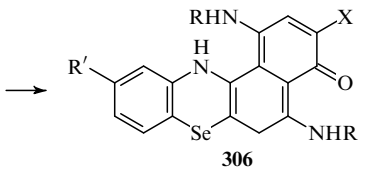
X = Br, Cl.

The 1,4-selenazine **303** has been obtained by irradiating the anthraquinone derivative **302**.[469] Compounds suppressing the biosynthesis of leucotrienes have been detected among its derivatives; preparations based on them have been proposed for the treatment of asthma, inflammatory processes, and cardiovascular and allergic diseases.[470, 471]



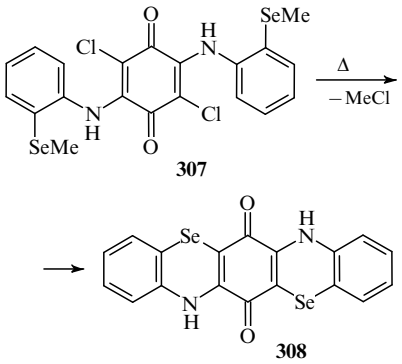
1,5-Bis(alkylamino)-(4*H*)-benzo[*a*]phenoselenazin-4-ones **306**, which absorb in the near infrared and can find application in laser techniques, have been synthesised by condensing the 1,5-naphthoquinones **304** with the zinc salts **305**.[472]



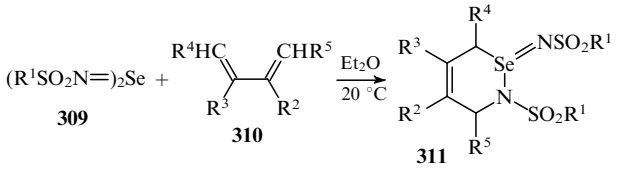


R = Me, Et, Pr^i; R′ = H, Cl, OMe; X = H, Br.

Heat treatment of compound **307** leads to di(benzoselenazino)quinone **308** in 41% yield.[473]

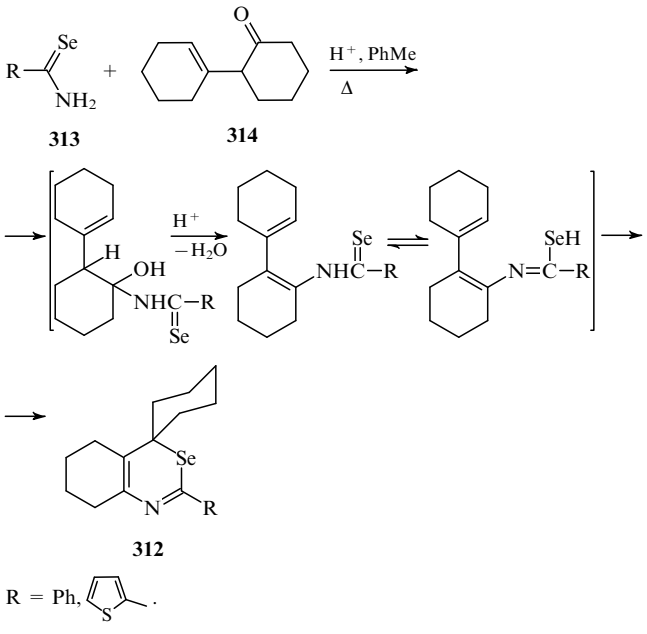


Selenium *N*,*N*′-di(arenesulfonyl)diimides **309** undergo [2 + 4]-cycloaddition to the 1,3-dienes **310** and afford 3,6-dihydro-1,2-selenazines **311** in 88% – 94% yields.[474]



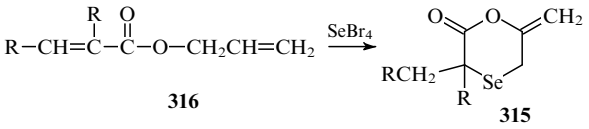
R^1 = Ph, R^2 = R^4 = R^5 = H, R^3 = Me; R^1 = Ph, R^2 = R^3 = Me, R^4 = R^5 = H; R^1 = Ph, R^2 = R^3 = H, R^4, R^5 = $(CH_2)_2$; R^1 = 4-MeC_6H_4, R^3 = R^4 = R^5 = H, R^3 = Me; R^1 = R^3 = Me, R^2 = R^4 = R^5 = H.

The 1,2-selenazine derivatives **312** have been obtained by condensing carboxylic acid selenoamides **313** with 2-(cyclohex-1-enyl)cyclohexanone **314** in the presence of catalytic amounts of naphthalene-β-sulfonic acid.[475]



R = Ph, .

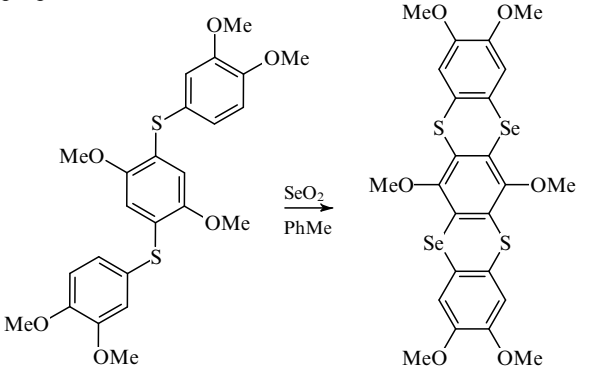
The substituted 1,4-selenoxane **315** has been synthesised by treating the ester **316** with selenium tetrabromide.[476]



316 **315**

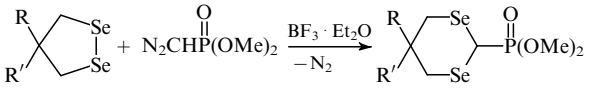
R = H, Me.

The interaction of the disulfide **318** with selenium dioxide afforded 2,3,6,9,10,13-hexamethoxy-5,12-dithia-7,14-diselena-5,7,12,14-tetrahydropentacene **317**, which posesses semiconducting properties.[477]



318 **317**

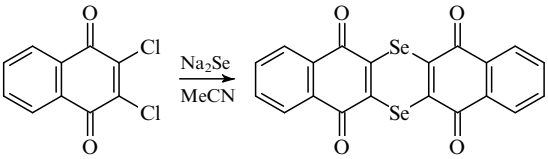
A method of synthesis of phosphorus-substituted 1,3-diselenanes **319** involving the interaction of 1,2-diselenolanes **320** with dimethyl diazomethylphosphonate in dichloromethane at room temperature in the presence of boron trifluoride etherate has been reported.[478]



320 **319** (11% – 18%)

R = R′ = Me; R = H, R′ = Bu^t.

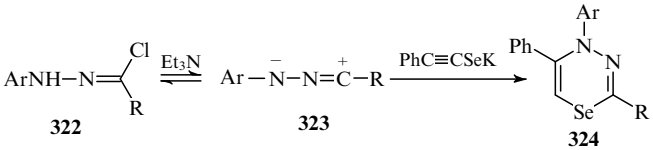
The condensed 1,4-diselenoxane derivative **321** has been obtained by treating 2,3-dichloro-1,4-naphthoquinone with sodium selenide in acetonitrile.[479]



321

5. Six-membered heterocycles with three heteroatoms

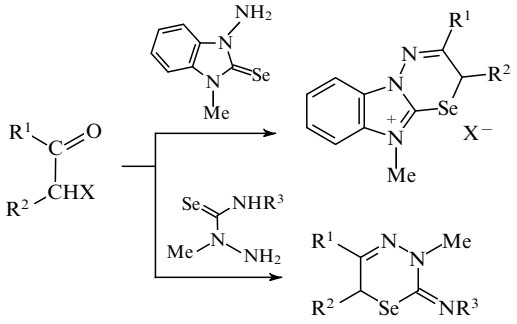
The nitrilimines **323**, obtained by treating *N*-aryl-*C*-chlorohydrazones **322** with an organic base, undergo [3 + 3]-cycloaddition to potassium phenylethyneselenolate, forming 2,4,5-trisubstituted (4*H*)-1,3,4-selenadiazines **324** in 89% – 100% yields.[480] The interaction of the nitrilimines **323** with dialkylselenamides of monosubstituted acetic acids also leads to derivatives of 1,3,4-selenadiazines.[481]



322 **323** **324**

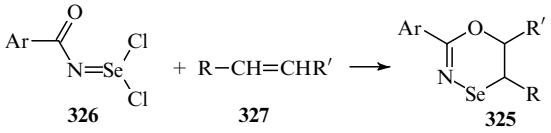
Ar = Ph, 4-MeC$_6$H$_4$, 4-BrC$_6$H$_4$; R = COMe, CO$_2$Et.

The reaction of α-haloketones with selenosemicarbazide, its derivatives, or other compounds containing the selenosemicarbazide fragment has been used fairly successfully in the synthesis of 1,3,4-selenadiazines, including condensed ones.[482 – 487]



R^1, R^2 = H, Alk, Ar, Het; R^3 = H, Alk; X = Br, Cl.

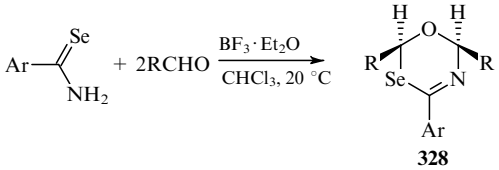
The 1,3,4-oxaselenazines **325** have been obtained by the reaction of *N*-aroyliminoselenyl chloride **326** with the alkenes **327** *via* the [4 + 2]-cycloaddition mechanism.[488]



326 **327** **325**

Ar = Ph, 4-MeC$_6$H$_4$, 4-BrC$_6$H$_4$, 4-ClC$_6$H$_4$, 4-NO$_2$C$_6$H$_4$;
R = H, R′ = Ph; R, R′ = (CH$_2$)$_4$.

A stereoselective method of synthesis of 1,3,5-oxaselenazines **328** by the interaction of selenoamides with aliphatic aldehydes in the presence of boron trifluoride etherate in chloroform at 20 °C has been developed (yields 69% – 97%).[489]



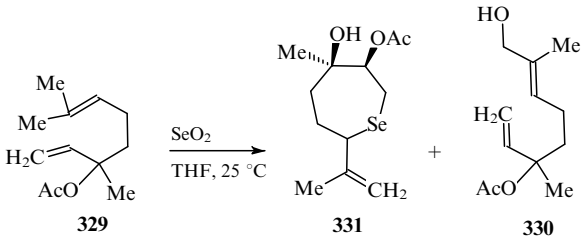
328

R = Me, C$_7$H$_{15}^n$, Bu^t, cyclo-C$_6$H$_{11}$; Ar = Ph, 3-ClC$_6$H$_4$, 4-MeOC$_6$H$_4$.

VI. Seven – nine-membered heterocycles

The number of publications in recent years on these selenium-containing heterocycles is small and the data presented in them are in most cases of a specific character and they are therefore described in order of increasing ring size of the heterocycle.

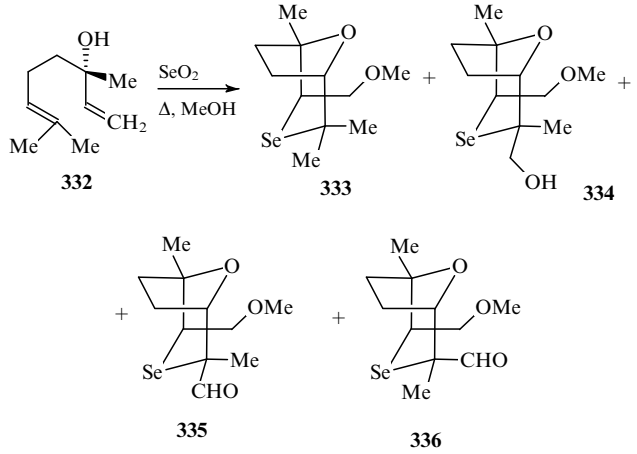
The interaction of freshly sublimed selenium dioxide with linalyl acetate **329** in THF at 25 °C leads to the alcohol **330** and the selenepane **331** in proportions of 7 : 3; the yield of the selenepane **331** is then 16% [490] while in the case where the reaction is carried out in methanol, it is 2%.[491] The structure of the selenepane **331** was established by XRD.[492]



329 **331** **330**

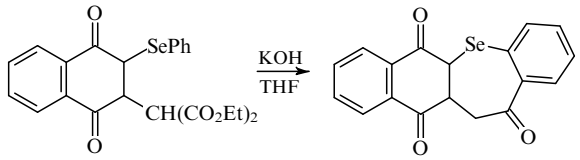
The analogous treatment of linalool **332** with selenium dioxide in methanol leads to a mixture of compounds containing the

8-oxa-3-selenabicyclo[3.2.1]octane skeleton (compounds **333**–**336**).[493] After flash chromatography on silica gel, 22.4% of compound **333**, 7.5% of compound **334**, 2.5% of compound **335**, and 3.1% of compound **336** was obtained.



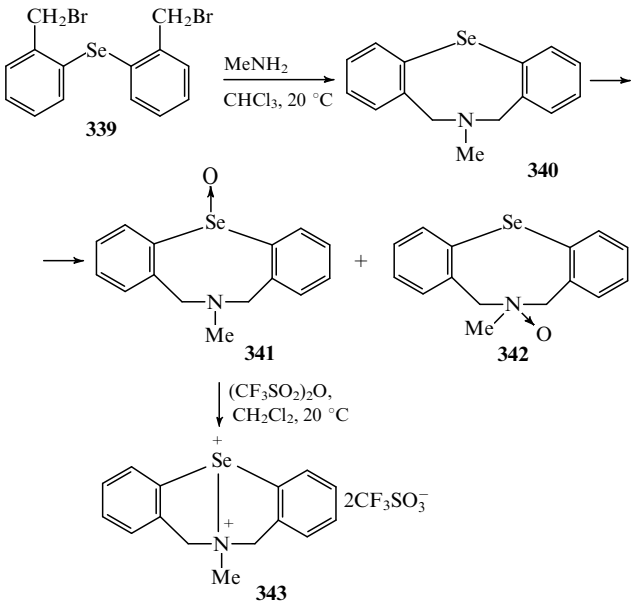
332 **333** **334** **335** **336**

The synthesis of the tetracyclic selenepinone **337** by the cyclisation of compound **338** on treatment with a KOH solution in THF has also been described.[494]



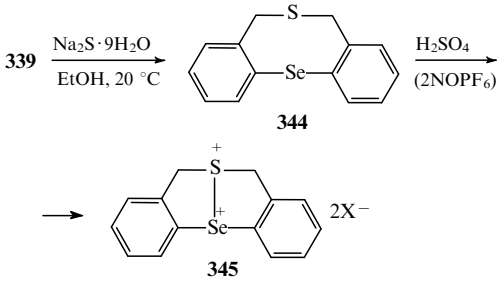
338 **337**

Dibenzoselenazocine **340**, the oxidation of which with *m*-chloroperbenzoic acid leads to the selenoxide **341** and the *N*-oxide **342**, has been obtained in 72% yield by the reaction of bis(2-bromomethylphenyl) selenide **339** with methylamine in chloroform at 20 °C under an argon atmosphere using the high dilution technique.[495] The interaction of the selenoxide **341** with trifluoromethanesulfonic acid anhydride [495] or thionyl chloride [496] results in the formation of the stable crystalline salt of the dication **343**, which exists as two conformers according to ^{1}H NMR data obtained in deuterochloroform at −50 °C: the boat-boat form (74%) and the boat-chair form (26%).[497]



339 **340** **341** **342** **343**

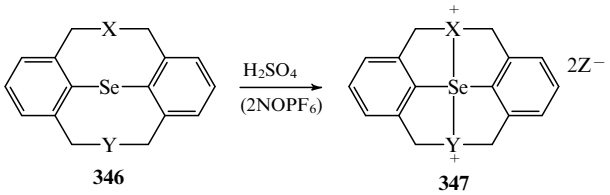
The analogous reaction of the selenide **339** with sodium sulfide in ethanol at 20 °C leads to a 90% yield of dibenzoselenathiocine **344**, which gives rise to the dication **345** on oxidation with concentrated H_2SO_4 or 2 equiv. of $NO^+PF_6^-$.[498]



344 **345**

X = HSO_4, PF_6.

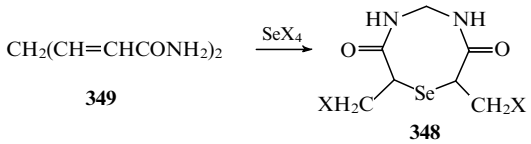
It has also been shown that salts of selenuranes **347** are formed on oxidation of dibenzodiselenocine **346** (X = Y = Se), dibenzoselenathiocine **346** (X = Y = S) or dibenzoselenazocine **346** (X = Y = NH, NCH_2Ph) with concentrated H_2SO_4 or 2 equiv. of $NO^+PF_6^-$.[499, 500]



346 **347**

X = Y = S, X = Y = Se; X = Y = NH; X = Y = NCH_2Ph; X = NH, Y = NCH_2Ph; Z = HSO_4, BF_6.

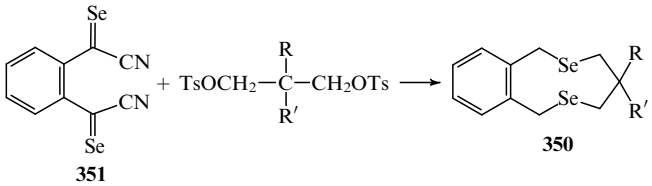
The synthesis of 2,8-dihalomethylperhydro-1,4,6-selenadiazocines **348** by the interaction of the diamide **349** with selenium tetrahalides has been reported.[501]



349 **348**

X = Br, Cl.

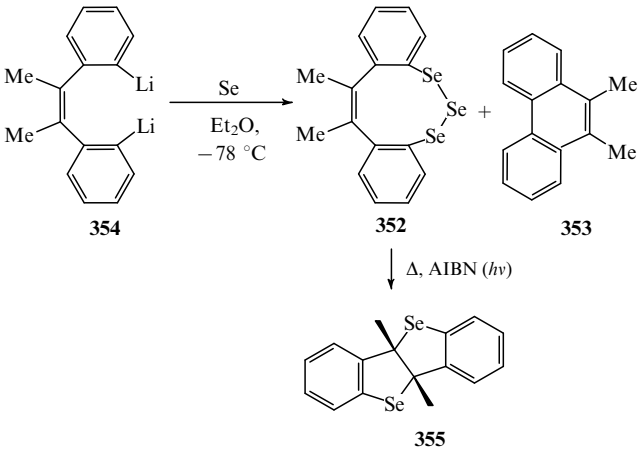
Nine-membered heterocycles containing two selenium atoms benzodiselenonines **350** — have been obtained by the interaction of 1,2-bis(cyanoselenocarbonyl)benzene **351** with the ditosyl derivatives of 2,2-bis(hydroxymethyl)alkanes or the corresponding cycloalkanes in THF.[502]



351 **350**

R = R′ = H; R, R′ = CH_2OCH_2, $(CH_2)_2$, $(CH_2)_4$.

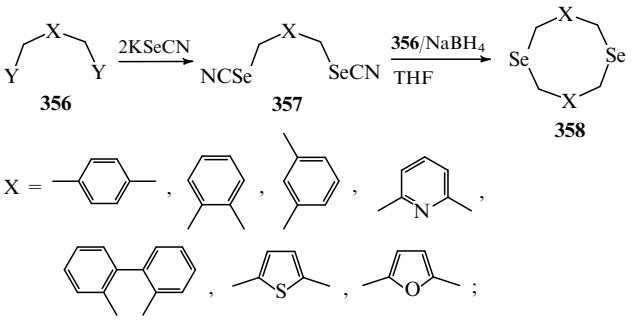
An analogous system containing three selenium atoms — 12,13-dimethylbenzo[*d*,*h*]-1,2,3-triselenonine **352** — is formed in 18% yield as a mixture with 9,10-dimethylphenanthrene **353** (5%) on interaction of (*Z*)-2,3-bis(2-lithiophenyl)but-2-ene **354** with 2–4 equiv. of selenium in ether at −78 °C.[503] On thermolysis (180 °C), on refluxing in benzene in the presence of azobisisobutyronitrile, or on photolysis (λ > 290 nm) in THF at 0 °C, the triselenonine **352** recyclises with loss of one selenium atom to 5*a*,10*a*-dimethyldibenzo[*b*,*f*]seleno[3,2*a*]selenophene **355**. The structures of compounds **352** and **355** were established by XRD.



354 352 353 355

VII. Selenacyclophanes and selenium-containing crown ethers

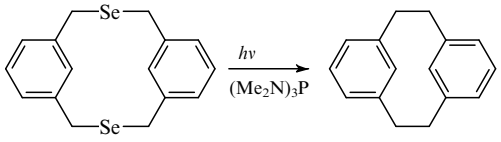
The methods of synthesis of various selenacyclophanes are based on the reactions of α,ω-dihaloalkanes or the corresponding cycloalkanes with sodium hydrogenselenide or potassium selenocyanate *via* α,ω-diselenocyanates, the yields of the final products approaching quantitative yields under these conditions.[504–510] For example, bis(halomethyl)-substituted aromatic and heteroaromatic compounds **356** interact with 2 equiv. of potassium selenocyanate to form the diselenocyanates **357**, which are converted into the diselenacycylophanes **358** in a high yield as a result of the reaction with the initial halide **356** in THF in the presence of sodium tetrahydroborate.[508]



356 357 358

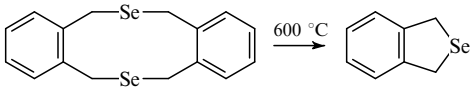
Y = Cl, Br.

On heat treatment or photoirradiation in the presence of tertiary phosphines, the diselenacyclophanes lose selenium and are converted into cyclophanes in a high yield.[504–507, 511, 512] For example, metacyclophane **360** has been obtained in 93% yield as a result of the photodeselenation of diselenacyclophane **359**.[513]



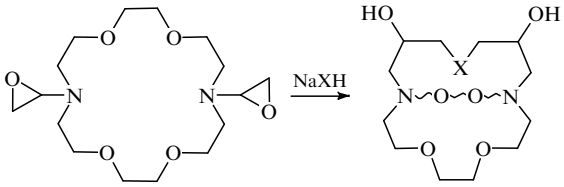
359 360

On the other hand, 1,3-dihydro-2-benzoselenophene **362** is formed in 92% yield in the pyrolysis of 2,11-diselena[3.3]-orthocyclophane **361** instead of the corresponding cyclophane.[513]



361 362

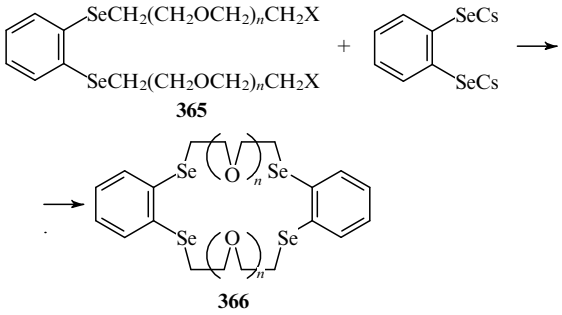
In conclusion, we shall consider data on crown ethers, cryptands and coronands containing selenium atoms and being of interest from the standpoint of the modelling of involved natural complexes. A simple method of synthesis of sulfur- and selenium-containing cryptands **363** by the interaction of bis(-methylene-epoxy)diaza-18-crown-6 **364** with sodium hydrogensulfide or hydrogenselenide in ethanol under an argon atmosphere has been developed.[514]



364 363

X = S, Se.

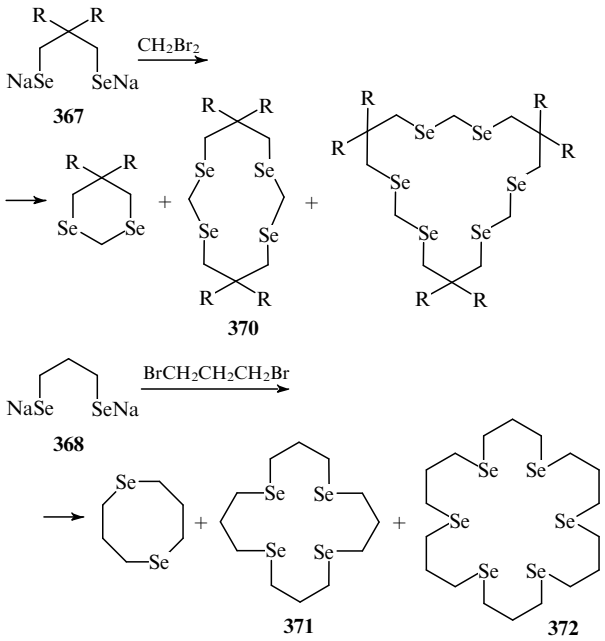
The reaction of α,ω-dihalides with cesium benzenediselenolate leads to the crown ethers **366** containing selenium atoms, in yields up to 40%.[515]



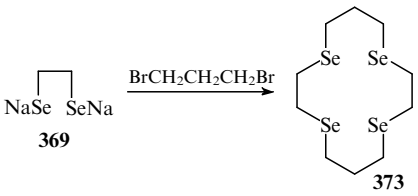
365 366

n = 1–3; X = Br, Cl.

A new class of coronands, containing selenium atoms, has been obtained in the reactions of the sodium bisselenolates **367**–**369** with dibromoalkanes in THF: tetraselenacyclododecane **370**, tetraselenacyclohexadecane **371**, hexaselenacyclotetracosane **372**, and tetraselenacyclotetradecane **373**.[516]



367 370 368 371 372



369 **373**

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