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CHEMISTRY OF HETEROCYCLIC COMPOUNDS AT THE A. E. FAVORSKY IRKUTSK INSTITUTE OF CHEMISTRY, SIBERIAN BRANCH OF THE RUSSIAN ACADEMY OF SCIENCES, OVER 50 YEARS (REVIEW)

N. A. Nedolya

The review contains a concise historical account and information on the most significant researches undertaken by the staff at the A. E. Favorsky Irkutsk Institute of Chemistry, Siberian Branch of the Russian Academy of Sciences on the Chemistry of Heterocyclic Compounds.

Keywords: azasilinanes, azepines, diazepines, dioxanes, dioxolanes, diselenetanes, dithietanes, dithiolanes, imidazoles, oxazoles, oxasilinanes, oxasilolanes, oxiranes, pyrazoles, pyrans, pyridines, pyrimidines, pyrroles, selenazines, selenasilafulvenes, selenophenes, silatranes, tellurasilafulvenes, tellurophenes, thiadiazoles, thiazines, thiazoles, thiasiletanes, thiasilinanes, thiasilolanes, thietanes, thiopyrans, thiophenes, triazoles, furans, quinolines, cyclosilaalkynes, synthesis, reactions, properties.

The A. E. Favorsky Irkutsk Institute of Chemistry, Siberian Branch of the Russian Academy of Sciences (before 1997 Irkutsk Institute of Organic Chemistry), one of the first academic institutes of Siberia, was founded in 1957. The establishment and development of the Institute are closely connected with the names of its organizer and first director, the eminent student and nearest associate of the classic of organic chemistry Academician A. E. Favorsky – a Corresponding Member of the Academy of Sciences of the USSR M. F. Shostakovskii – and its second director (from 1970) and leading authority in the chemistry of organic compounds of silicon and sulfur – Academician of the Russian Academy of Sciences M. G. Voronkov. M. F. Shostakovskii laid the foundation for the dynamic development of the major Russian chemical school (the Favorsky school) on Siberian soil. Under his leadership the Institute immediately moved forward to leading positions in world chemical science in the field of the chemistry of acetylene, polymers, and silicon (particularly silicoacetylene compounds). Under the leadership of M. G. Voronkov pioneering researches unrolled in the field of the chemistry of hypervalent silicon giving the Institute worldwide renown, carbofunctional and biologically active organosilicon compounds, and the high-temperature synthesis of organic derivatives of sulfur.

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* Dedicated to Academician of the Russian Academy of Sciences B. A. Trofimov on his 70th jubilee.

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At the present time two leading officially recognized scientific schools – those of Academicians of the Russian Academy of Sciences B. A. Trofimov and M. G. Voronkov – have been successfully established and developed at the Institute. Certain historical landmarks and most interesting moments from the life of the Institute in the recollections of its members were described in the monograph "The A. E. Favorsky Irkutsk Institute of Chemistry – 50 Years" [1]. Information on the Institute and its scientific work can also be gathered from the collected papers [2, 3].

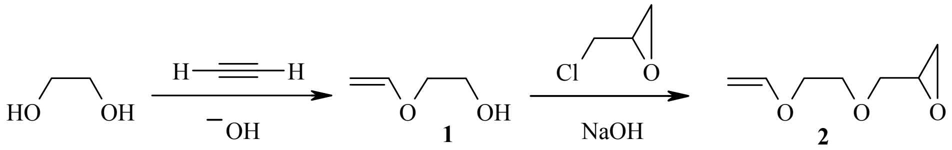
The advances in the chemistry of heterocyclic compounds are closely connected with the chemistry of acetylene, silicon and sulfur – the main directions of the fundamental and applied researches at the Institute. They include heterocyclic synthesis based on the use of such compounds as building blocks for the construction of a heterocyclic nucleus or a macromolecule and functionalization or annulation of heterocycles. Much attention has been paid to systematic study of the reactivity and physicochemical characteristics of the fundamental heterocycles leading to the discovery of new important reactions and to an understanding of their mechanisms. There is hardly a single scientific worker at the Institute who is not concerned with heterocyclic chemistry to some degree in his researches. It is worth mentioning that both named reactions (Voronkov and Trofimov) discovered* and developed at the Institute relate to the synthesis of heterocycles. Many of the biosubjects isolated (from vegetable material) and studied at the Institute, also have heterocyclic structures as illustrated by dihydroquercetin, maltol, arabinogalactan, etc.

The author of the present review has tried to map the contribution of the Irkutsk Institute of Chemistry to the development of the chemistry of heterocyclic compounds. The most important achievements of recent years and more recent papers that have not lost their significance up to the present day are covered. The author recognizes that to cover all or even the most interesting and significant results obtained at the Institute in a single review is an impracticable task and hopes for the understanding of his colleagues on this matter. In no way should the results not covered in the review be regarded as less important. The review is constructed in the traditional way; the information contained in it is classified according to the size of the heterocycle and the number of heteroatoms in it.

1. THREE- AND FOUR-MEMBERED HETEROCYCLES

1.1. Oxiranes

The perfection of methods for the production of 2-(vinyloxy)ethanol **1** [4-7] and 2-(vinyloxy)-ethoxymethyloxirane (vinylox) **2** based on it [7-13] has provided the motivation for comprehensive and systematic study of the characteristics and synthetic potential of oxiranes functionalized by vinyloxy groups. Various methods have been developed for synthesis of the various analogs of vinylox – the condensation of alkanediols and also of diethylene and triethylene glycols with epichlorohydrin [6-10], the selective addition of glycidol to the divinyl ethers of glycols [14-17], the successive treatment of 1,3-dioxolan-2-ones first with 2-(vinyloxy)ethylamine and then with an epichlorohydrin/base system [15, 18] or with the base alone [15, 19, 20].



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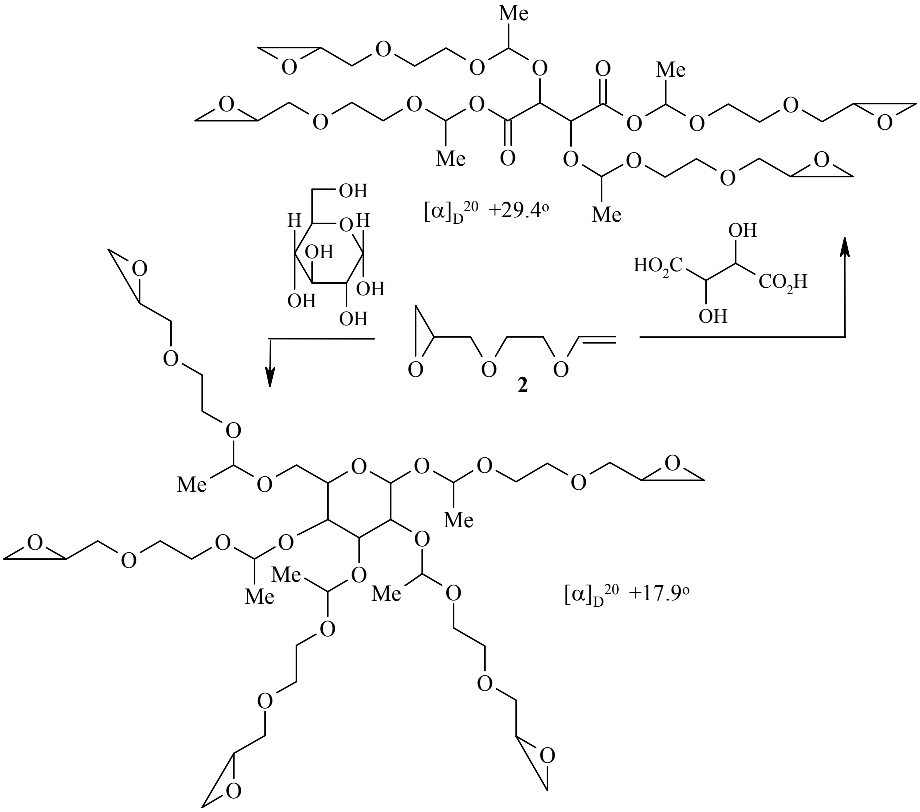
* The Voronkov reaction (the reaction of sulfur with arylchloroalkanes and arylchloroalkenes) was discovered at the Institute of Organic Synthesis of the Latvian SSR (now the Latvian Institute of Organic Synthesis) – Editor's remark.

A fundamentally new atom-saving strategy for the synthesis of epoxide resins, based on the use of vinylox as a volatile oxirane containing an "anchor" function (the vinyloxy group) capable of being involved readily, selectively, and quantitatively in the addition of bis- or polyprotogenic compounds (polyatomic alcohols, phenols, carboxylic acids, sugars, thiols, thiokols) while retaining the oxirane ring completely unaffected was created (B. A. Trofimov, N. A. Nedolya) [14, 15, 26-30] as a result of researches on the kinetics and mechanism of the electrophilic addition of alcohols and carboxylic acids to vinyl ethers [7, 14, 15, 21-25]. Such a volatile anchor oxirane can be purified easily by distillation, making it possible to produce epoxide resins with a specified degree of purity. Perfluorocarboxylic acids [14, 15, 27-32] or their acylals [14, 15, 30-32] and triphenylchloromethane [14] were first proposed as specific mildly acting catalysts for the regioselective addition of hydroxyl-containing compounds to the vinyloxy group.

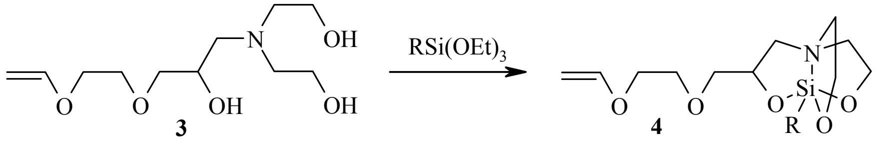
By using natural optically active hydroxy carboxylic acids [e.g., L-(+)-tartaric, $[\alpha]_D^{20}$ +(12-12.4)°] and sugars [e.g., L-(+)-glucose, $[\alpha]_D^{20}$ +(52.5-53)°] in reaction with vinylox it was possible to produce optically active epoxide resins readily and quantitatively for the first time [15].

Other anchor oxiranes were also used successfully in the synthesis of epoxide resins of a new generation [14, 15, 26]. This general approach, by which it was possible to synthesize hundreds of new epoxide resins not having analogs (either in structure or in properties) and including sulfur-, nitrogen-, silicon-, fluorine-, bromine-, and boron-containing compounds, has now received new recognition in connection with the practical involvement of epoxide resins in the design of nanostructural materials [33-35].

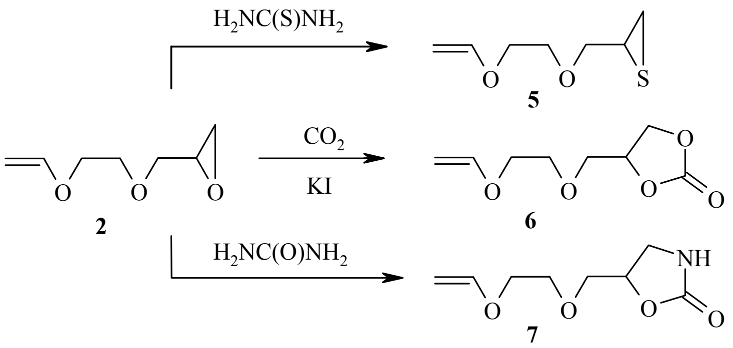


Vinylox has opened up a path not only to new epoxide resins but also to a series of new functional monooxiranes synthesized by highly selective addition reactions at its reactive double bond [7, 14, 15, 31, 32, 36-51]. In the case of vinylox the initiation of the free-radical addition of thiols to the vinyloxy group by typical cationic catalysts ($C_3F_7CO_2H$, *p*-TSA) was discovered for the first time [15, 43, 44]. The reaction mechanism involves electron transfer from the thiol to the carboxonium intermediate with the formation of radical particles, which then initiate a radical chain process.

The oxirane ring in vinylox and its analogs is easily opened by various nucleophilic reagents [7, 14, 15, 52-64] with retention of the vinyloxy group, representing a new general approach to the synthesis of the most widely varied functionalized vinyl ethers. After treatment with organyltrialkoxysilanes the adduct of vinylox with diethanolamine **3** [63] gives "anchor" silatranes **4** unusually readily and with almost quantitative yields [64].

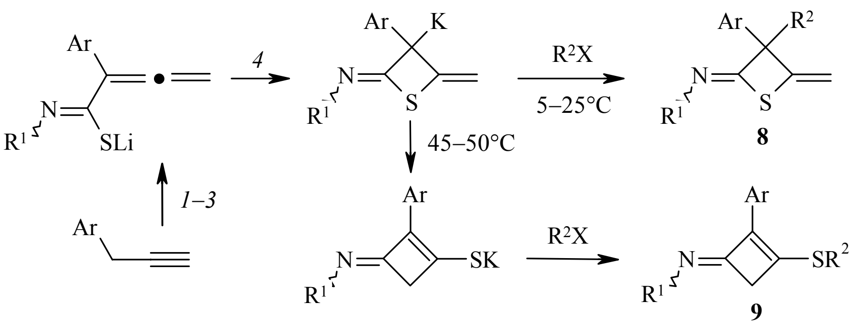


Exchange reaction between vinylox and thiourea gives the thiirane **5** [7, 15, 65], which was used as key compound for the production of biologically active derivatives of aminothiols [7, 15, 66], other functional thiols [7, 15], and thiiranes [15, 67, 68], including thiirane resins – previously unknown sulfur analogs of epoxide resins. Cycloaddition reactions with carbon dioxide [7, 15, 69, 70] and urea involving participation of the oxirane ring made such "anchor" heterocycles as 1,3-dioxolan-2-one **6** and 1,3-oxazolidin-2-one **7** accessible; these compounds are convenient reagents for the selective functionalization of various compounds [15, 71-73], including polymers [74, 75].



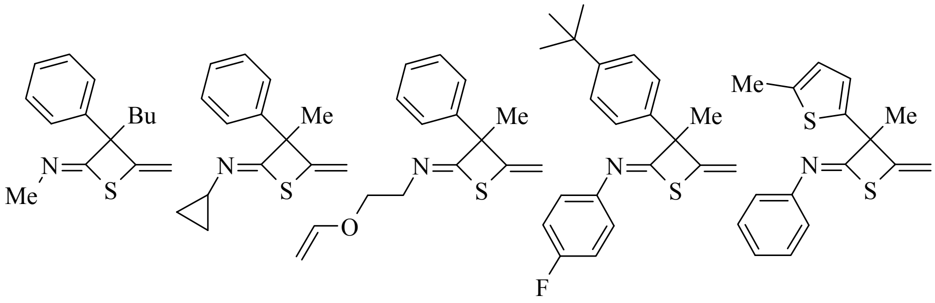
1.2. Thietanes

In Nedolya's group the low-temperature reactions of the 1,3-dianions of propargylarenes with isothiocyanates were discovered and studied systematically as a fundamentally new and effective path to functionalized four-membered systems – 4-imino-2-methylenethietanes **8** and 3-iminocyclobutenes **9** [76-79]. The strict temperature dependence of the reaction path makes it possible to realize a controlled synthesis of the thietane **8** or the isomeric cyclobutane **9** from one and the same precursor [78]. The process is carried out in one preparative stage.



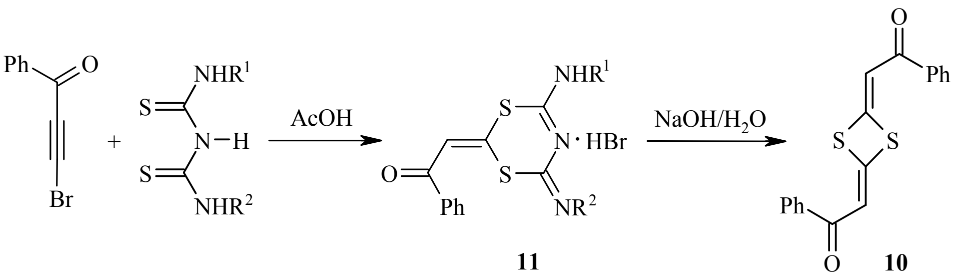
1: 2 BuLi/THF/C_6H_{14}; *2*: $R^1N{=}C{=}S$; *3*: *t*-BuOH; *4*: *t*-BuOK/DMSO

Iminothietanes with alkyl, cycloalkyl, heteroalkyl, vinyl, aryl, and hetaryl substituents became accessible as a result of this reaction, and some examples are presented below.



1.3. Dithietanes, Diselenetanes, and Thiasiletanes

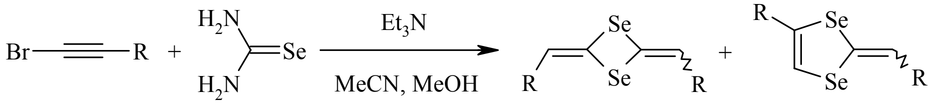
1,3-Dithietane **10** was unexpectedly produced (T. E. Glotova, N. I. Protsuk, M. Yu. Dvorko) by hydrolysis of the hydrobromides of 4H-1,3,5-dithiazines **11**, synthesized by the reaction of 2-benzoyl-1-bromoacetylene with 2,4-dithiobiurets [80-82].



11 **10**

R^1 = H, Me, Ph; R^2 = H, Ph

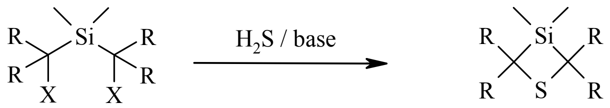
The reaction of bromoacetylene ketones with selenourea (S. V. Amosova et al.) leads to the 1,3-diselenetanes **12** and 1,3-diselenoles **13** [83].



12 **13**

R = Bz, 2-thenoyl

The action of S-nucleophiles on bis-α-haloalkyl-substituted silanes leads to the formation of 1,3-thiasiletanes **14** (M. G. Voronkov, E. N. Suslova, S. V. Kirpichenko) [84, 85].



14

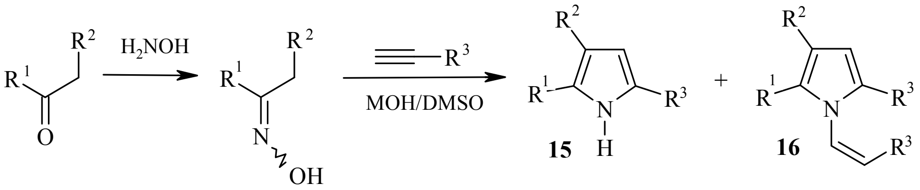
R = H, X = Cl; R = Ph, X = Br

2. FIVE-MEMBERED HETEROCYCLES WITH ONE HETEROATOM

2.1. Pyrroles

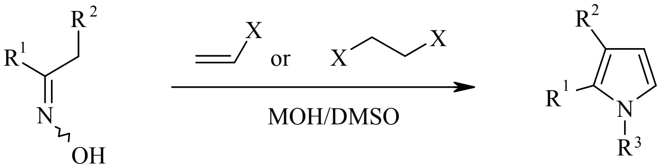
The reactions of acetylene with ketoximes [7, 86-98], of allene and acetylene carbanions with isothiocyanates [98-101], and of allyl isothiocyanate with the amides of alkali metals [99, 100, 102], which were discovered at the Institute, became the basis of fundamentally new strategies for the construction of a pyrrole ring.

A new general reaction of ketoximes with acetylenes, taking place in the presence of superbasic systems of the MOH/DMSO type (where M is an alkali metal) and leading to NH- and N-vinylpyrroles **15** and **16**, was discovered and systematically developed under the guidance of B. A. Trofimov [7, 86-98, 103-128]. With an excess of the acetylene it is possible to obtain the N-vinylpyrroles directly.



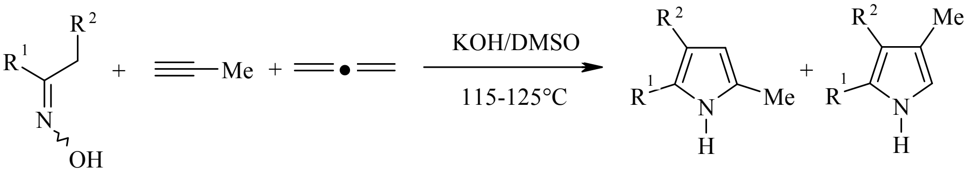
R^1, R^2 = Alk, Ar, Het; R^3 = H, Me, Ph; M = Li, Na, K, Rb, Cs

When necessary the synthesis can be conducted in one pot, by treating the ketone in the MOH/DMSO system first with hydroxylamine (or its hydrochloride) and then with the acetylene [116, 117]. In place of acetylene it is possible to use its synthetic equivalents – vinyl halides [86, 121, 122] and 1,2-dihaloethanes [123-125],

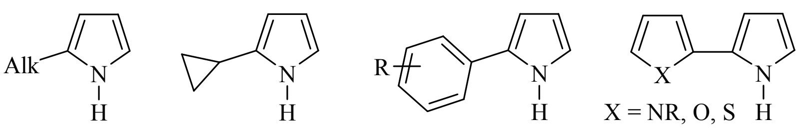


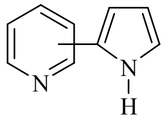
R^1 = Alk, R^2 = H, Alk; R^3 = H, Vin; X = Cl, Br; M = Na, K, Cs

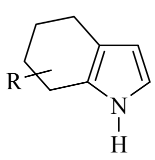
and also a propyne–allene mixture [126].

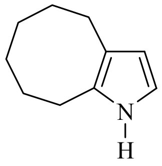


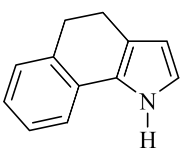
Pyrroles with alkyl, cycloalkyl, aryl, and hetaryl substituents and also pyrroles annulated with carbocycles and condensed systems have become accessible [87, 94, 95, 118] as a result of this reaction, known as the Trofimov reaction (e.g., see [97, 129] and the references therein).



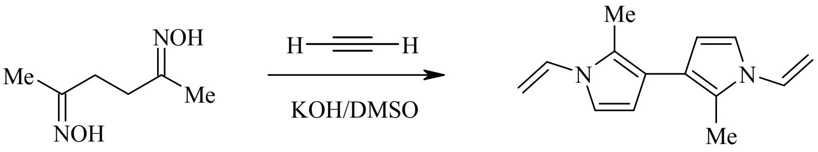




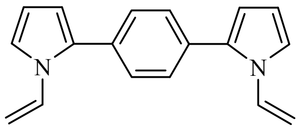


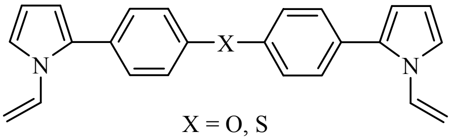


The reactions of the dioximes of alkanediones such as 2,5-hexanedione with acetylene lead to dipyrroles [110, 127].



Dipyrroles separated by aromatic spacers were obtained from acetylene and the dioximes of aromatic ketones [113, 114, 128].

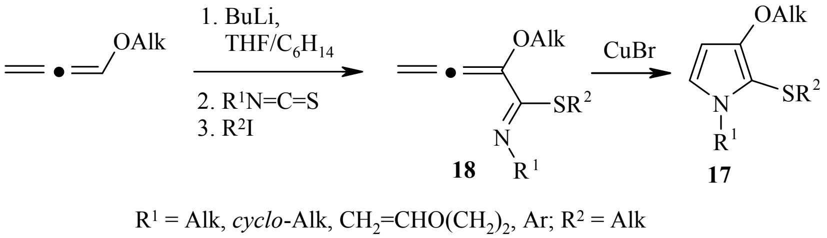




X = O, S

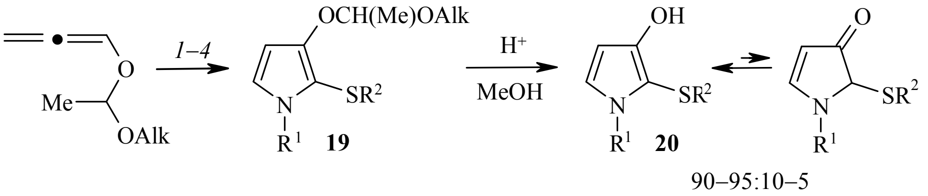
In the laboratory (the group from 2001) of N. A. Nedolya (in collaboration with Prof. L. Brandsma, Utrecht University, the Netherlands) a fundamentally new general strategy was discovered and developed systematically for the synthesis of a pyrrole ring [99-101, 130-149] and other fundamental aza-, oxa-, and thiaheterocycles (pyrrolines, pyridines, dihydropyridines, quinolines, azepines, dihydroazepines, dihydrofurans, thietanes, thiophenes, dihydrothiophenes, dihydrothiopyrans) [76-79, 99, 101, 148-150]. Essentially it involves the use of heterocumulenes (isothiocyanates, isocyanates) and carbanions readily generated *in situ* from available alkynes or 1,2- and 1,3-dienes, as key building blocks. The results of these investigations were summarized and analyzed in dissertations [98, 148, 149] and in recently published reviews and monographs [99, 101, 150-165].

A large series of 1,2,3-substituted pyrroles **17** with the rare 3-alkoxy- and 2-alkylsulfanyl substituents were obtained by the Cu(I)X-catalyzed cyclization (X = Cl, Br, I) of 1-aza-1,3,4-trienes **18**, formed during the reaction of α-lithiated alkoxyallenes and isothiocyanates followed by S-alkylation of the adducts [99-101, 131]. The reaction is conducted in a single preparative stage, although the intermediates **18** can be easily isolated when necessary.



R^1 = Alk, *cyclo*-Alk, CH_2=CHO$(CH_2)_2$, Ar; R^2 = Alk

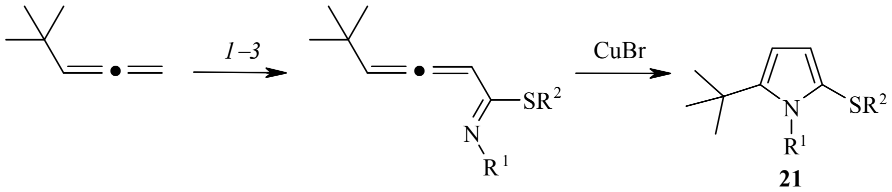
The 3-(1-alkoxyalkoxy)-2-(alkylsulfanyl)pyrroles **19** and the pyrrol-3-ols **20** stable in the hydroxy form were first obtained as a result of this reaction [99, 132].



90–95:10–5

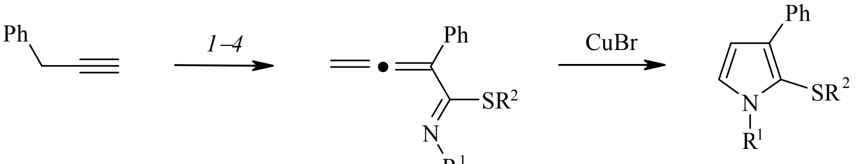
1: BuLi/THF/C_6H_{14}; *2*: $R^1N=C=S$; *3*: R^2I; *4*: CuBr

The introduction of γ-lithiated *tert*-butylallene into reaction with isothiocyanates leads to 5-*tert*-butylpyrroles **21** [133] while the 1,3-dilithiated propargylbenzene gives 3-arylpyrroles **22** [134].



21

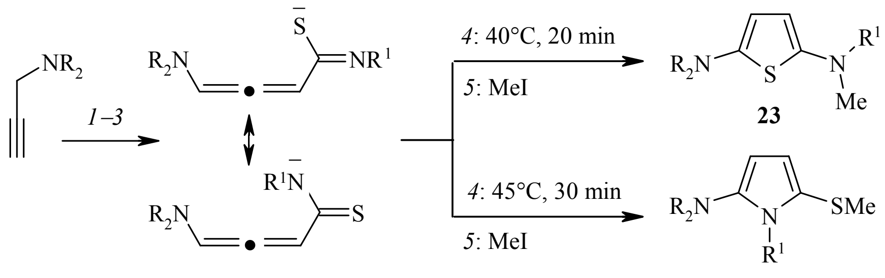
1: BuLi/THF/C_6H_{14}; *2*: $R^1N=C=S$; *3*: R^2I



22

1: 2 BuLi/THF/C_6H_{14}; *2*: $R^1N=C=S$; *3*: *t*-BuOH; *4*: R^2I

By the reaction of monolithiated propargylamines with isothiocyanates it is possible to realize a highly selective synthesis of 2,5-bis(amino)thiophenes **23** and 2-aminopyrroles **24** from one and the same precursors. In contrast to the reactions examined above the S- or N-centered azatriene anions in this case undergo intramolecular heterocyclization [135].

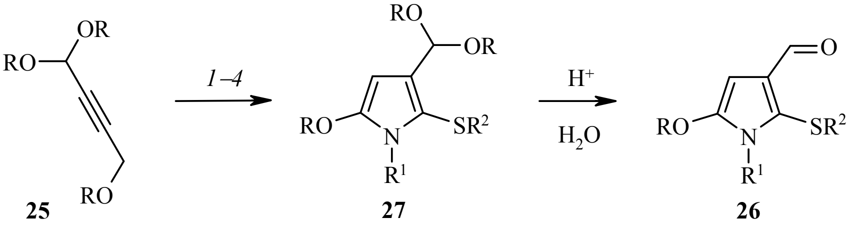


24

1: 2 BuLi/THF/C_6H_{14}; *2*: $R^1N=C=S$; *3*: *t*-BuOK/*t*-BuOH/DMSO

Representative series of 1,2,3,5-tetrasubstituted pyrroles containing alkyl, alkoxyalkyl, aminoalkyl, cycloalkyl, alkenyl, alkynyl, aryl, hetaryl, acetal, alkoxy-, hydroxy-, and alkylsulfanyl, amino, formyl, and other substituents were obtained from isothiocyanates and metallated alkynes in a single preparative stage [99, 136-146].

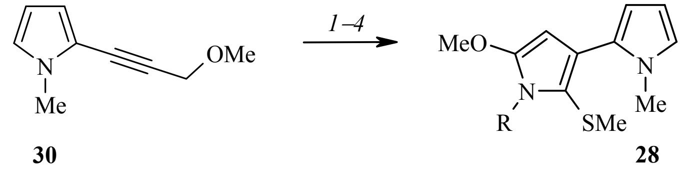
The use of lithiated 1,1,4-trialkoxy-2-butynes **25** in reaction with isothiocyanates as a source of the carbanions of blocked 4-alkoxy-2,3-butadienals opens up a direct route to 3-formylpyrroles **26** (through the pyrroles **27**) [139, 140].



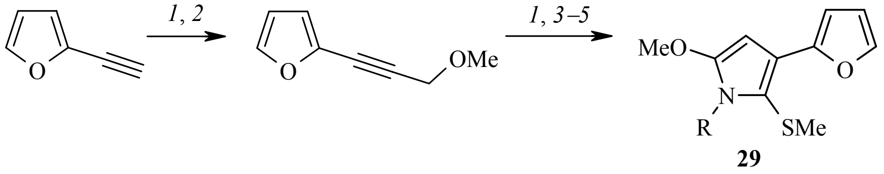
1: BuLi/THF/C_6H_{14}; *2*: $R^1N{=}C{=}S$; *3*: R^2I; *4*: CuBr

R = Me, Et; R^1 = Alk, *cyclo*-Alk, $MeOCH_2$, $CH_2{=}CHO(CH_2)_2$, Ar; R^2 = Alk

The uncommon hetarylpyrrole assemblies 2,3'-bipyrrole **28** and 3-(2-furyl)pyrrole **29** were obtained in a single preparative stage from isothiocyanate and lithiated 2-alkynylpyrrole **30** [143] and 2-ethynylfuran [144] respectively.

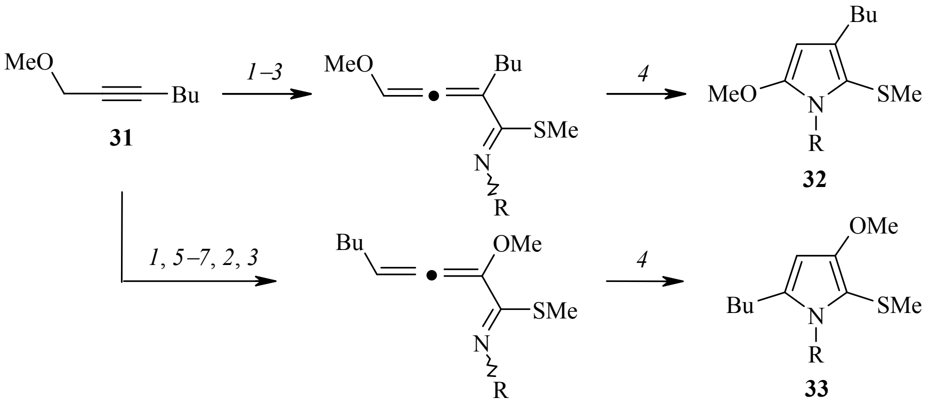


1: BuLi/THF/C_6H_{14}; *2*: RN=C=S; *3*: MeI; *4*: CuBr



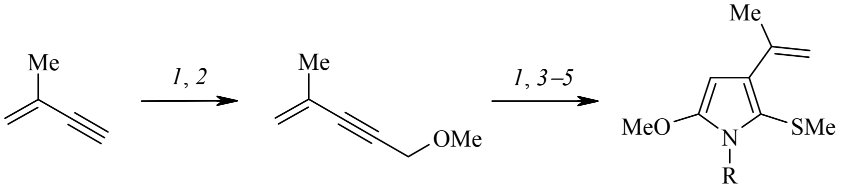
1: BuLi/THF/C_6H_{14}; *2*: $ClCH_2OMe$; *3*: RN=C=S; *4*: MeI; *5*: CuBr

The structure of the final product can be determined effectively at the stage of deprotonation of the 2-alkyne. Thus, for example, 3-butyl-5-methoxypyrroles **32** were obtained from 2-heptyne **31** lithiated with BuLi and isothiocyanates. The use of the BuLi/*t*-BuOK/HMPA as deprotonating agent led to 5-butyl-3-methoxypyrroles **33** [99, 141].

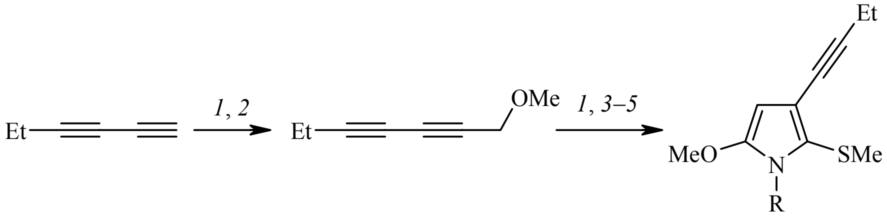


1: BuLi/THF/C_6H_{14}; *2*: RN=C=S; *3*: MeI; *4*: CuBr; *5*: *t*-BuOK; *6*: HMPA; *7*: LiBr

The use of conjugated enynes and diynes in the reaction leads to simultaneous assembly and C-3-vinylation [99] or C-3-alkynylation of the pyrrole ring [99, 146].

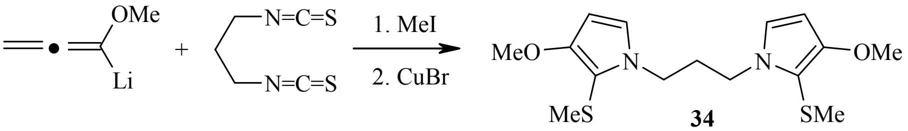


1: BuLi/THF/C_6H_{14}; *2*: $ClCH_2OMe$; *3*: RN=C=S; *4*: MeI; *5*: CuBr



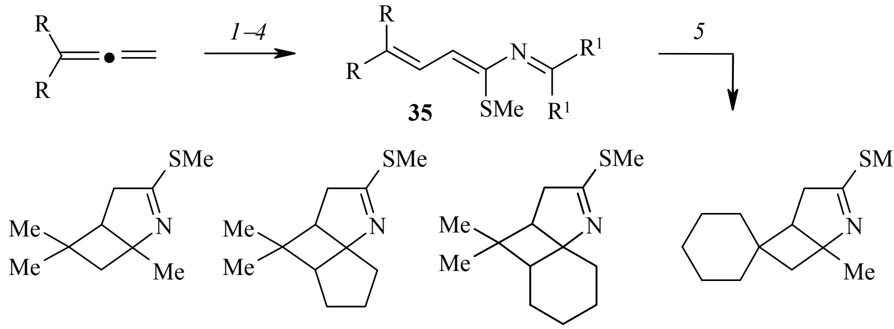
1: BuLi/THF/C_6H_{14}; *2*: $ClCH_2OMe$; *3*: RN=C=S; *4*: MeI; *5*: CuBr

The use of diisothiocyanates in reaction with lithiated allenes leads to 1,1'-(1,ω-alkanediyl)bis(pyrroles) such as **34** [99, 147].



34

Unusual thermal rearrangements in the azatriene systems **35**, obtained from γ-lithiated 1,1-disubstituted allenes and isothiocyanates with *sec*-alkyl or cycloalkyl substituents, lead to complex di- and tricyclic structures of the pyrrolines series **36** [166-170].

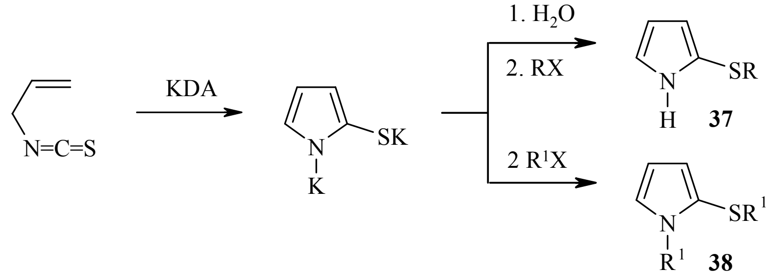


35

36a (87%) **36b** (85%) **36c** (75%) **36d** (90%)

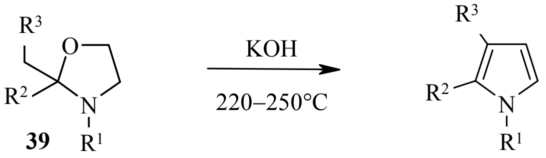
1: BuLi/THF/C_6H_{14}; *2*: $R^1{}_2CHN=C=S$; *3*: MeI; *4*: [1,5]-H shift; *5*: 220–255°C, ~10–15 min
$R = R^1 = Me$; R–R = $(CH_2)_5$; R^1–R^1 = $(CH_2)_n$, $n = 4, 5$

A reaction leading to the reorganization of allyl isothiocyanate to NH- and 1-alkyl-2-(organylsulfanyl)pyrroles **37** and **38** by the action of potassium diisopropylamide was discovered (N. A. Nedolya et al.) [99, 102, 171-173].



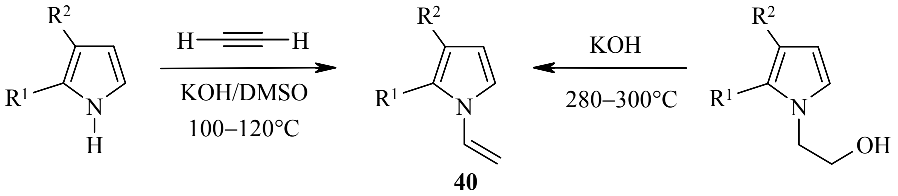
KDA = *t*-BuOK + *i*-Pr_2NLi (LDA)
R = Alk, All, $HC\equiv CCH_2$; R^1 = Alk; X = Cl, Br, I

Another approach to the synthesis of pyrroles based on nonacetylene precursors was developed (B. F. Kukharev, A. S. Atavin, V. K. Stankevich) as a result of a systematic study of the catalytic transformations of 1,3-oxazolidines **39** [174-177].

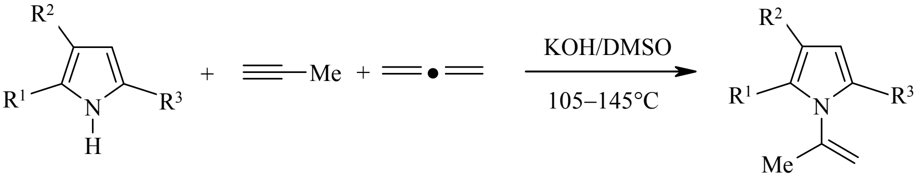


R^1 = Alk, *cyclo*-Alk, $(CH_2)_2OH$, Bn, Ph;
R^2, R^3 = H, Alk, Ph; R^2–R^3 = $(CH_2)_n$, $n = 3–5$; $(CH_2)_3CHMe$

Much attention has been paid to the search for convenient methods for functionalization of the pyrrole ring [98, 178-196]. Thus, the processes involved in the vinylation of indole and other azoles by acetylene (under pressure, at 180-220°C, in the presence of KOH, alkali metals, and their salts with azoles) and also the characteristics of the synthesized N-vinylazoles and their derivatives were actively studied from the very early years (G. G. Skvortsova, E. S. Domnina et al.) [171-181]. Present-day methods for the production of N-vinylpyrroles **40** (B. A. Trofimov et al.) are based on the use of superbases as catalysts for the direct vinylation of pyrroles with acetylene, making it possible to conduct the process under milder conditions – at atmospheric pressure and relatively low temperature [87, 95]. Another potentially useful method for the production of N-vinylpyrroles is the dehydration of N-(2-hydroxy-ethyl)pyrroles, synthesized from oxazolidines **39** [177].

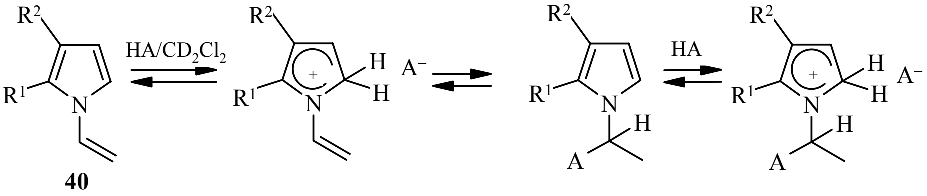


The propyne–allene/KOH/DMSO system was used successfully for the direct N-isopropenylation of various azoles (pyrroles, pyrazoles, imidazoles, 1,2,4-triazoles) at atmospheric pressure and in an autoclave [182, 183].



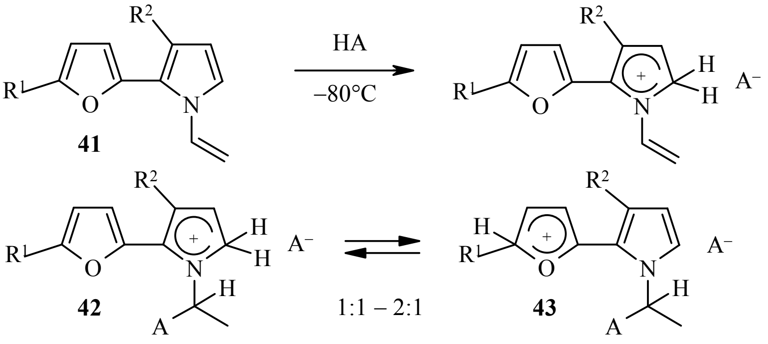
Since they become accessible N-vinylpyrroles have been studied as starting materials and monomers for the production of novel or little-known types of pyrrole compounds [184-196] and also as highly informative models for theoretical investigations of the electronic and conformational effects of various types of interacting π-systems separated by a nitrogen atom [196-201].

In consideration of the exceptional role of pyrrolium and indolium ions as key intermediates in many important reactions the protonation of N-vinylpyrroles was studied comprehensively and systematically (B. A. Trofimov et al.) [98, 196-205]. It was shown that N-vinylpyrroles **40** are protonated at the α-position of the pyrrole ring, at the β-position of the vinyl group, or at both centers simultaneously depending on the conditions [205].



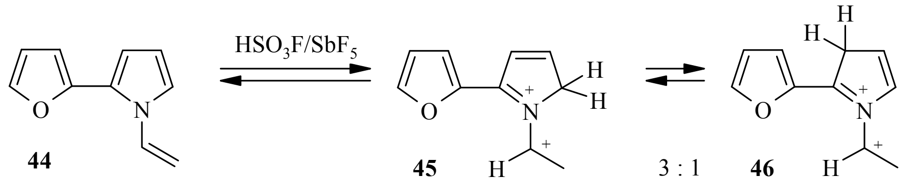
R^1 = H, Alk, Ar, Het; R^2 = H, Alk; A = Cl, Br, $AlCl_4$, $AlBr_4$, CF_3CO_2, SO_3F, SbF_6

At −80 to 0°C 2-(2-furyl)-1-vinylpyrroles **41** are protonated by acids at the C-5 atom of the pyrrole ring without affecting the vinyl group, whereas with hydrogen halides at −30°C furanium cations **43** are unexpectedly formed in addition to pyrrolium cations **42** [198, 205]. At the same time the hydrogen halide adds to the vinyl group. When the α-position of the furan ring is occupied by a methyl group the hydrogen halides only add to the vinyl group [205].

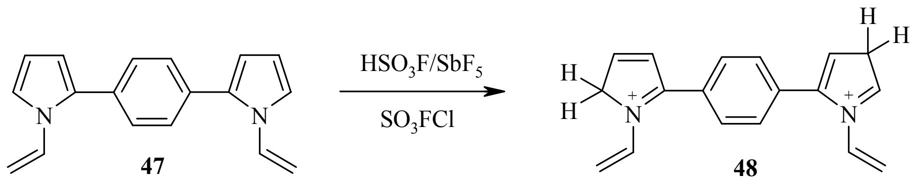


R^1 = H, Me; R^2 = H, Me; A = Cl, Br, CF_3CO_2, SO_3F, SbF_6

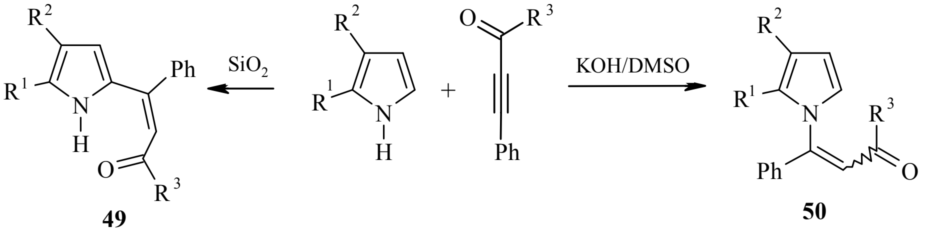
The reaction of 2-(2-furyl)-1-vinylpyrrole **44** with a superacid leads to the formation of an equilibrium mixture of the dications **45** and **46** [204].



Bis(1-vinyl)-2,2'-(1,4-phenylenepyrrole) **47** is protonated by a superbasic acid to the unsymmetrical dication **48** – with α- and β-protonated pyrrole rings [205].



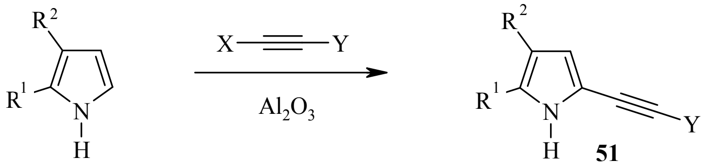
The reactions of pyrroles with disubstituted acetylenes were investigated systematically (B. A. Trofimov, L. N. Sobenina, A. I. Mikhaleva, et al.) [206-215]. On the surface of SiO_2 the reaction of pyrrole and 2-phenylpyrrole with 2-acyl-1-phenylacetylenes takes place stereo- and regioselectively with the formation of (*Z*)-2-ethenylpyrroles **49** [206], whereas the N-adducts **50** are formed in the KOH/DMSO system [207].



R^1 = H, Ph; R^2 = H; R^1–R^2 = $(CH_2)_4$;
R^3 = Et, CCl_3, Ph, 2-furyl, 2-thienyl

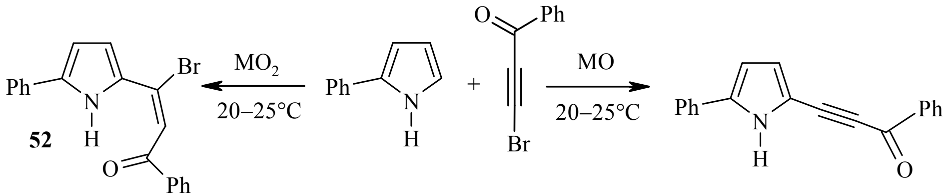
R^1 = R^2 = H; R^1 = H, R^2 = Ph;
R^3 = Ph, 2-furyl

Cross-coupling of NH-pyrroles with haloacetylenes on Al_2O_3 leads to 2-ethynylpyrroles **51** [208-210]. N-Vinylpyrroles, tetrahydroindoles, and indoles were used successfully in this reaction [211-213].



R^1, R^2 = Alk, Ar, Het; X = Br, I; Y = $R^3C(O)$, $R^3OC(O)$; R^3 = Alk, Ar, Het

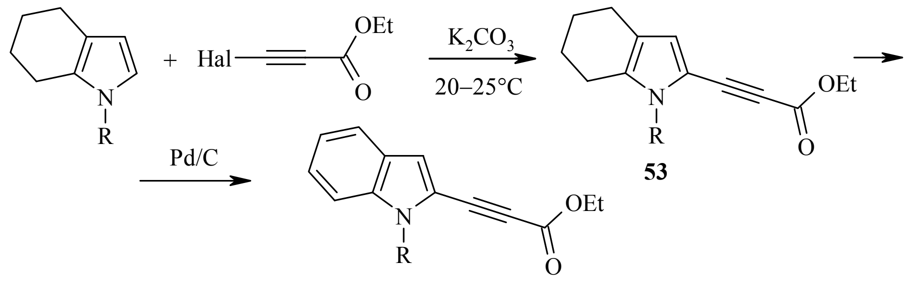
It was established for the first time that the oxides of Ca, Mg, Zn, and Ba promote the ethynylation of 2-phenylpyrrole by bromobenzoylacetylene, and ZnO and BaO are more active than Al_2O_3 [214]. The oxides of Ti, Zr, and Si, which are inactive in cross-coupling, were specific catalysts for the regio- and stereoselective addition of pyrroles to the triple bond of bromobenzoylacetylene with the formation of 2-ethenylpyrrole **52** [214].



M = Ti, Zr, Si

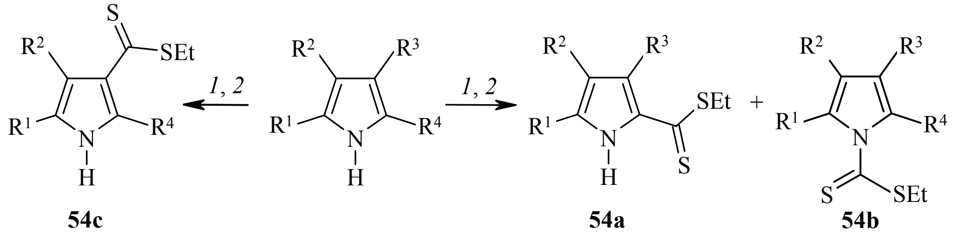
M = Ca, Mg, Zn, Ba

The general idea of C-2-functionalization of the indole skeleton is based on the ethynylation of derivatives of 4,5,6,7-tetrahydroindole by halopropionates on K_2CO_3 followed by aromatization of the tetrahydroindole **53** [215].



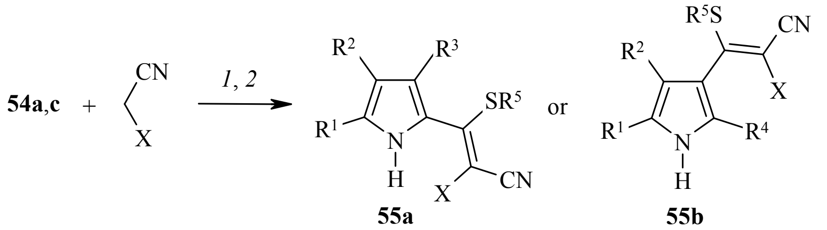
R = H, Me, Bn, Vin, $EtS(CH_2)_2$, $PrS(CH_2)_2$, *i*-PrOCH(Me), BuOCH(Me)

Esters of pyrrole-2-carboxylic acids **54a** were obtained by the reaction of pyrroles with CS_2 and alkyl halides in the KOH/DMSO system (B. A. Trofimov, L. N. Sobenina, A. I. Mikhaleva, et al.) [184, 185, 216-221]. Unsubstituted pyrrole gives only the N-isomer **54b**. When R^1 and $R^4 \neq H$, pyrrole-3-carbodithioates **54c** are formed exclusively [219-221].



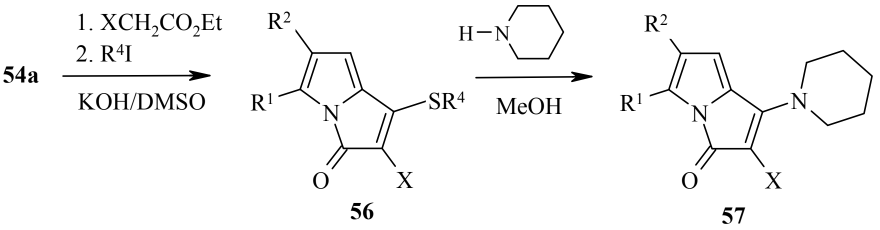
1: CS_2/KOH/DMSO; *2*: EtI
R^1 = H, Alk, Ar; R^2 = H, Alk; R^3, R^4 = H, Me

An effective method for the production of 2- and 3-ethenylpyrroles **55** was developed on the basis of the reaction of pyrrolecarbodithioates **54a**,**c** with methylene-active nitriles in the KOH/DMSO system [222, 223].



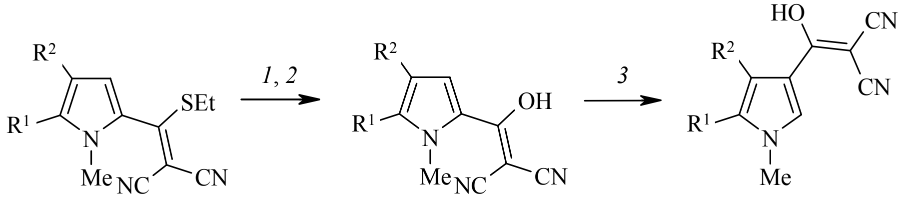
1: KOH/DMSO; *2*: R^5I
X = CN, $C(O)NH_2$; R^5 = Alk, All

The condensation of pyrrole-2-carbodithioates **54a** with CH acids containing ester groups in the KOH/DMSO system gave 3H-pyrrolizin-3-ones **56** [224-226], which were transformed into 1-amino-3H-pyrrolizin-3-ones **57** when treated with secondary amines [226].



X = CN, C(O)Me, CO_2Et

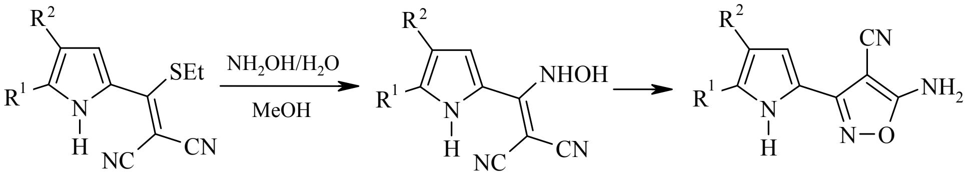
It was found that 2-(2,2-dicyano-1-hydroxyethenyl)pyrroles **58**, formed during the solvolysis of the pyrroles **59**, are transformed into the 3-isomers **60** when heated [227].



59 **58** **60**

1: NaOH/MeOH/H_2O; *2*: H^+/H_2O; *3*: 80–140°C; $R^1 = R^2 = H$, $R^1–R^2 = (CH_2)_4$

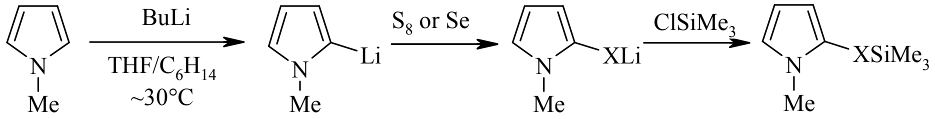
3-(2-Pyrrolyl)isoxazoles **61** were synthesized by the reaction of the pyrroles **55a** (R^3 = H, R^5 = Et, X = CN) with hydroxylamine [187, 228].



55a **61**

R^1 = Bu, R^2 = Pr; $R^1–R^2 = (CH_2)_4$; R^1 = Ph, R^2 = H

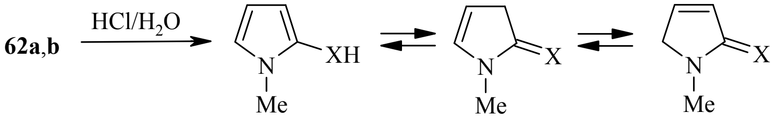
Fundamental information on the metallation of N-methyl- [229], N-isopropenyl- [230], N-allenyl- [231-233], N-propargyl- [233], and N-ethynylpyrroles [234] with BuLi or BuLi/*t*-BuOK and on the reactions of the obtained carbanions with various electrophiles was obtained in conjunction with Prof. L. Brandsma. Thus, α-lithiated N-methylpyrrole was used successfully for the synthesis of silylated pyrroles **62** (by successive reactions with chalcogen and trimethylchlorosilane) [229].



62a,**b**

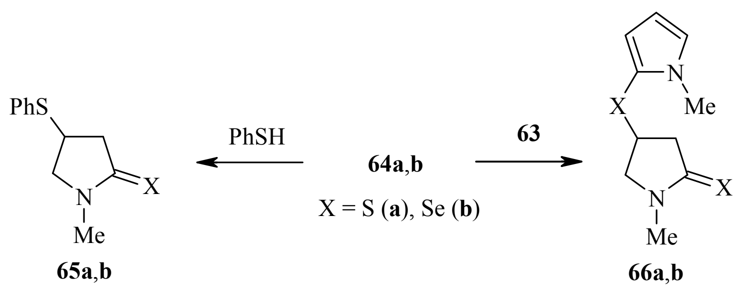
X = S (**a**), Se (**b**)

Mild hydrolysis of the pyrroles **62** leads to 1-methylpyrrole-2-thiol **63a** and 1-methylpyrrole-2-selenol **63b**, which exist in dilute solutions predominantly in the form of 1,5-dihydro-2H-pyrrole-2-thione **64a** and 1,5-dihydro-2H-pyrrole-2-selenone **64b** (NMR).

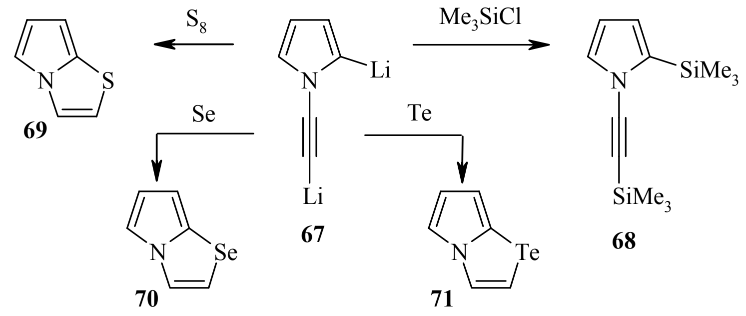


63a,**b** **64a**,**b**

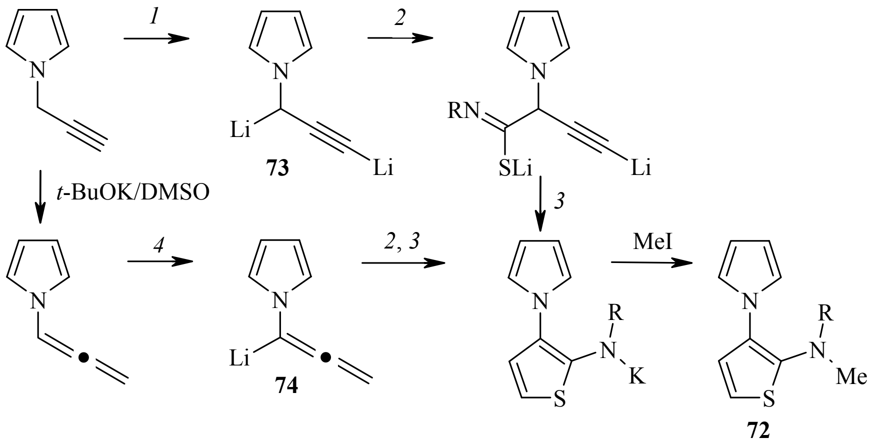
The addition of thiols and selenols to 1,5-dihydro-2H-pyrroles **64**, opening up access to 4-substituted 2-pyrrolidinethiones **65a** and **66a** and 2-pyrrolidineselenones **65b**, **66b**, was discovered [229].



The functionalization of 2-lithio-1-(lithioethynyl)pyrrole **67**, produced *in situ* from N-(1,2-dichlorovinyl)pyrrole [234] or N-ethynylpyrrole [235] and BuLi, with trimethylchlorosilane or chalcogens led to the silylated pyrrole **68**, pyrrolo[2,1-*b*][1,3]thiazole **69**, and the corresponding selenazole **70** and tellurazole **71**.



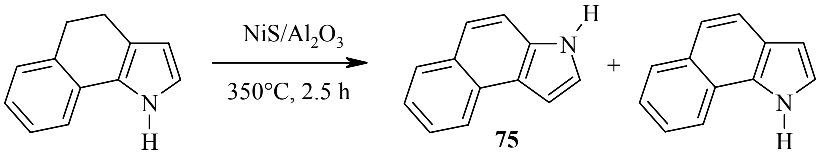
1-(3-Thienyl)pyrroles **72** became accessible thanks to the reactions of the *in situ* generated N-(1,3-dilithiopropargyl)pyrroles **73** or N-(1-lithioallenyl)pyrroles **74** with isothiocyanates [233].



1: 2 BuLi/THF/C_6H_{14}; *2*: RN=C=S; *3*: *t*-BuOH/*t*-BuOK/DMSO; *4*: BuLi/THF

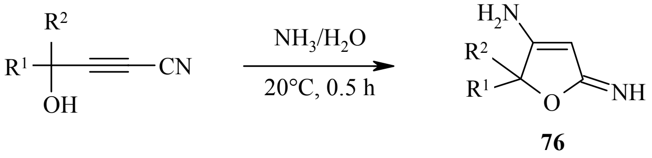
R = Alk, Ar: Ph, XC_6H_4, X = 4-F, 4-Cl, 4-Me, 3-Cl, 3-MeO, 3-CF_3

A far-reaching skeletal rearrangement leading mostly to the formation of 3H-benzo[*e*]indole **75** at the initial stages of the process, taking place during the catalytic dehydrogenation of 4,5-dihydrobenzo[*g*]indole, produced from tetralone oxime and acetylene, was discovered (B. A. Trofimov et al.) [236].



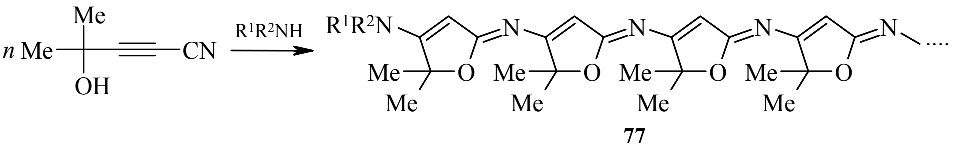
2.2. Furans

Systematic investigations in the chemistry of α,β-acetylenic γ-hydroxy acids and their derivatives (B. A. Trofimov, Yu. M. Skvortsov, A. G. Mal'kina, A. N. Volkov) made both the subjects of the investigation themselves and the various heterocyclic structures based on them accessible [237-241]. The 4-amino-2(5H)-furanimines **76** were synthesized from the nitriles of α,β-acetylenic γ-hydroxy acids in aqueous ammonia. Other primary amines were also used successfully in the reaction [237, 238].



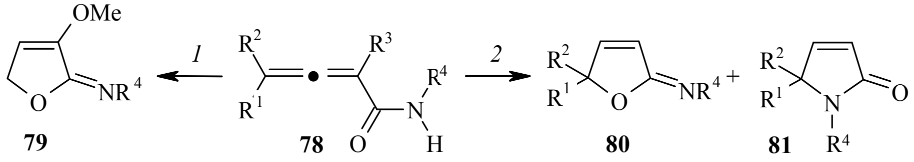
$R^1 = R^2 = Me$; $R^1 = Me$, $R^2 = Et$; $R^1–R^2 = (CH_2)_5$

By varying the ratio of the reagents and the reaction conditions it is possible to bring about the one-pot assembly of polyconjugated heterocyclic systems **77** having unusual electrophysical characteristics [242-247].



$R^1 = H$, Me; $R^2 = Ph$, 2-naphthyl

The heterocyclization of allenylamides **78**, produced from allene carbanions and isothiocyanates, to 3-monosubstituted (**79**) and 5,5-disubstituted (**80**) 2(5H)-furanimines and their structural isomers – 2H-pyrrol-2-ones **81** – was realized for the first time (N. A. Nedolya, N. I. Shlyakhtina, L. Brandsma) [149, 248-250].

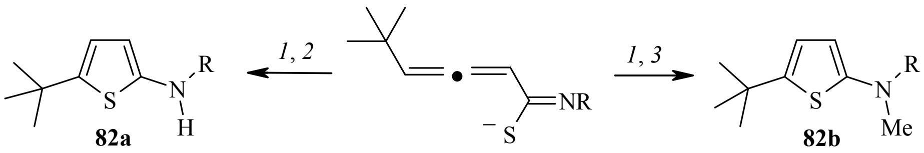


MeCOMe: AgOAc (*1*), $AgNO_3$

R^1, $R^2 = H$, $R^3 = OMe$; R^1, $R^2 = Me$, $R^3 = H$; $R^1 = H$, $R^2 =$ *t*-Bu, $R^3 = H$; $R^4 =$ Alk

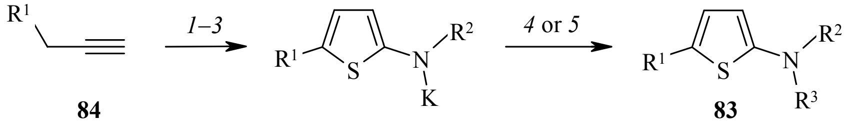
2.3. Thiophenes, Selenophenes, and Tellurophenes

The reaction of metallated allenes or alkynes with isothiocyanates was discovered and developed systematically (N. A. Nedolya, O. A. Tarasova, et al.) as a new general approach to the synthesis of 2-thiopheneamines [79, 99, 101, 233, 251-256]. 5-*tert*-Butyl-2-thiopheneamines **82** were synthesized for the first time by intramolecular cyclization of the adducts of lithiated *tert*-butylallene and isothiocyanates [99, 251].



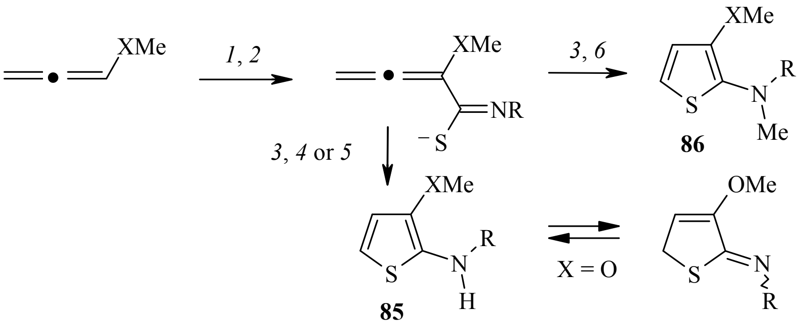
1: *t*-BuOH/*t*-BuOK/DMSO; *2*: H_2O; *3*: MeI

Unsubstituted and 5-substituted 2-thiopheneamines **83** were obtained likewise from isothiocyanates and 1-lithiated 1-propynes **84** [99, 251].



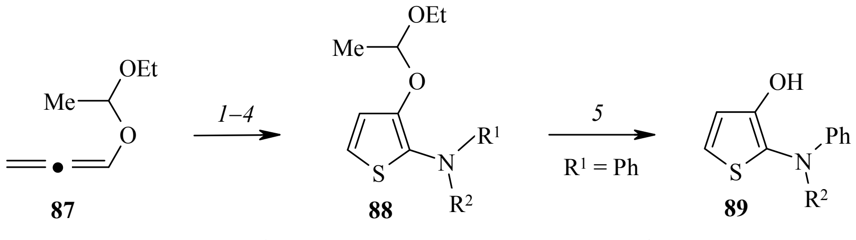
1: BuLi/THF/C_6H_{14}; *2*: $R^2N{=}C{=}S$; *3*: *t*-BuOH/*t*-BuOK/DMSO; *4*: H_2O; *5*: AlkI
R^1 = H, Me, MeO, Me_2N; R^2 = Alk, Ar; R^3 = H, Alk

The reactions of α-lithiated methoxy- and (methyl sulfanyl)allenes with isothiocyanates lead to N-alkyl-**85** and N,N-dialkyl-3-methoxy(methylthio)-2-thiopheneamines **86** [99, 252]. In the case of 3-methoxy-2-thiopheneamines **85** experimental evidence was obtained for amine–imine tautomerism in 2-thiopheneamines for the first time [99, 252, 253].



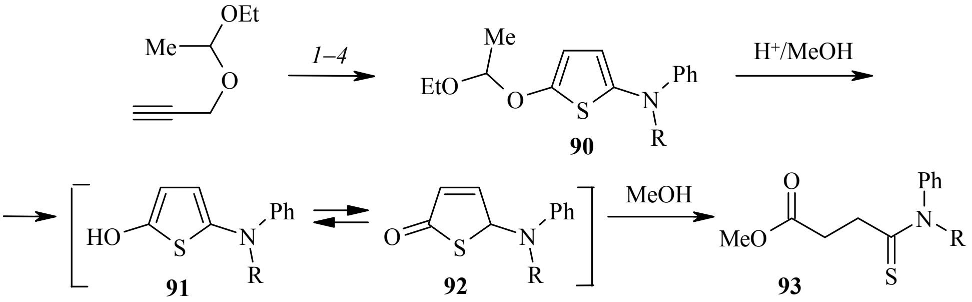
1: BuLi/THF/C_6H_{14}; *2*: RNCS; *3*: *t*-BuOH/*t*-BuOK/DMSO or *t*-BuOK/HMPA; *4*: H_2O; *5*: H_3O^+; *6*: MeI; X = O, S; R = Alk, Ar

Starting from the blocked allenol **87** it is easy to arrive both at the 3-acetal **88** and at the 3-hydroxy-2-thiopheneamines **89**, which exist almost exclusively in the hydroxy form [254].



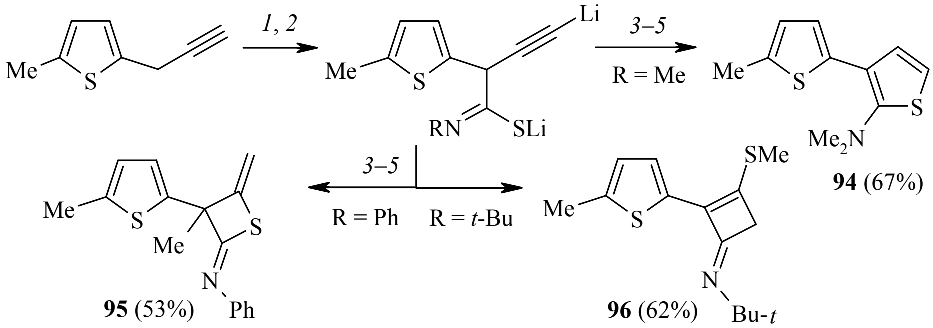
1: BuLi/THF/C_6H_{14}; *2*: R^1NCS; *3*: *t*-BuOH/*t*-BuOK/DMSO; *4*: R^2I; *5*: H^+/MeOH;
R^1 = Alk, Ar; R^2 = Alk

Alcoholysis of the acetal **90** instead of 5-hydroxy-2-thiopheneamine **91** or its keto tautomers **92** led unexpectedly to the ester of 4-amino-4-thioxobutyric acid **93** [254].



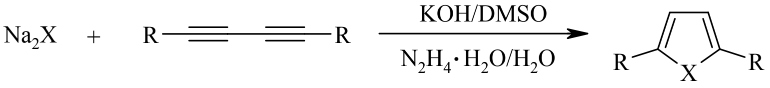
1: BuLi/THF/C_6H_{14}; *2*: PhNCS; *3*: *t*-BuOH/*t*-BuOK/DMSO; *4*: RI; R = Alk

The use of 2-methyl-5-propargylthiophene as propargyl component leads to 2,3'-bithiophenes **94**, 2-(3-thietanyl)thiophenes **95**, and 2-(1-cyclobutenyl)thiophenes **96** [79].



1: 2 BuLi/THF/C_6H_{14}; *2*: RN=C=S; *3*: *t*-BuOH; *4*: *t*-BuOK/DMSO; *5*: MeI

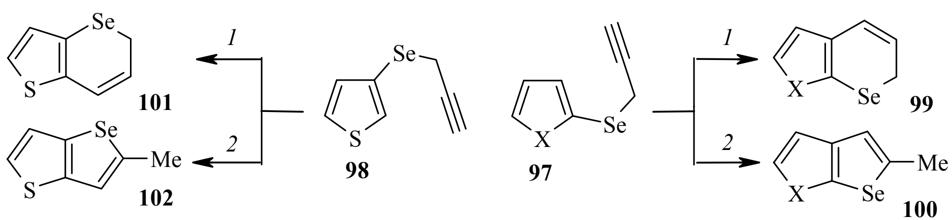
Effective methods for the production of thiophenes, selenophenes, and tellurophenes were developed (V. A. Potapov, S. V. Amosova, B. A. Trofimov) on the basis of the reaction of chalcogenide anions with diacetylenes [257-259].



X = S, Se, Te; R = H, Ph

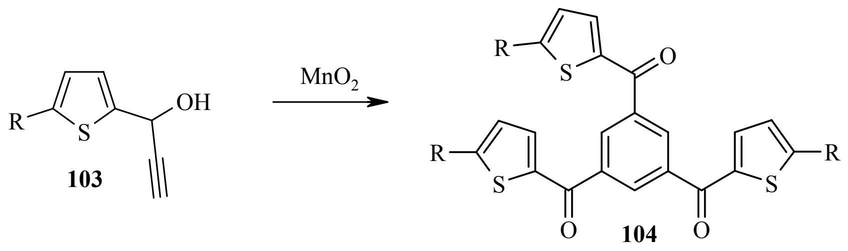
Thermal heterocyclization of divinyl chalcogenides also leads to high yields of chalcogenophenes [260, 261].

An effective approach to the construction of condensed heterocyclic systems **99-102** with the selenium atom at various positions in the ring was developed in conjunction with foreign authors (L. Brandsma, E. H. Morkved, O. Bjorlo) on the basis of the [3,3]-sigmatropic rearrangement of propargylselenochalcogenophenes **97** and **98** [262].

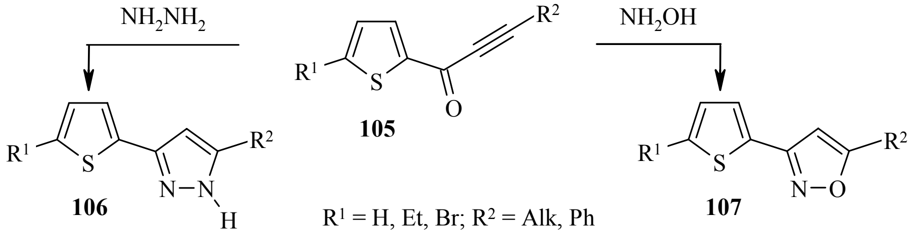


130–160 °C: HMPA (*1*), DMSO, *i*-Pr_2NH (*2*); X = S, Se, Te

An unusual oxidative trimerization of 1-(2-thienyl)-2-propyn-1-ol **103** to 1,3,5-trithenoylbenzene **104** was discovered (A. S. Nakhmanovich, V. I. Knutov) in the course of systematic investigations into the chemistry of unsaturated carbonyl compounds of the thiophene series [263].



3-(2-Thienyl)-1H-pyrazoles **106** and the corresponding isoxazoles **107** were obtained for the first time by the reactions of 1-(2-thienyl)-2-alkyn-1-ones **105** with hydrazine and hydroxylamine [263].

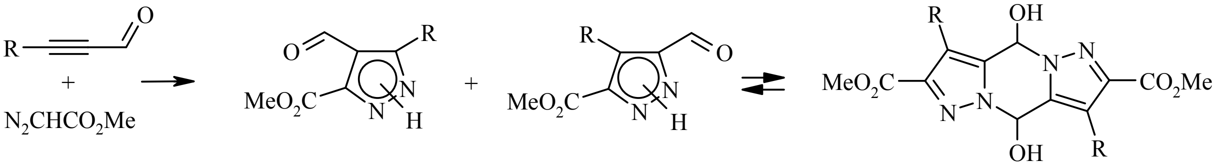


R^1 = H, Et, Br; R^2 = Alk, Ph

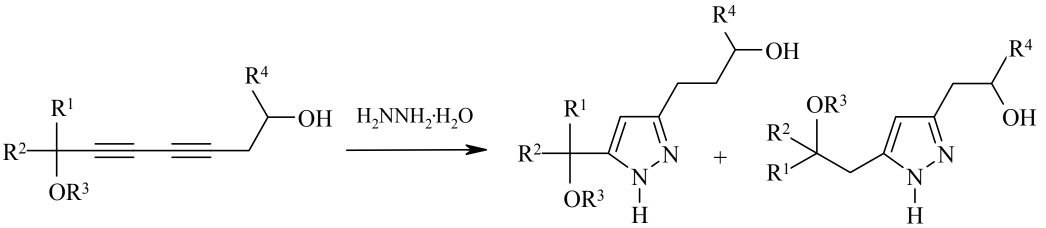
3. FIVE-MEMBERED HETEROCYCLES WITH TWO OR MORE HETEROATOMS

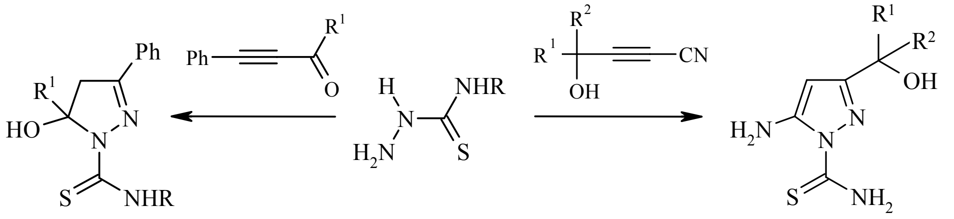
3.1. Pyrazoles, Imidazoles, Thiazoles, and Oxazoles

Methods were developed for the synthesis of polyfunctionalized pyrazoles by the reactions of various acetylene compounds with diazo compounds [264-267] or hydrazine hydrate [268] (A. S. Medvedeva et al.) and its derivatives (B. A. Trofimov, T. E. Glotova, M. Yu. Dvorko, et al.) [82, 269, 270], and also of α-chloro-β-ketodimethoxy acetals with hydrazines (A. N. Mirskova, G. G. Levkovskaya, et al.) [271, 272].

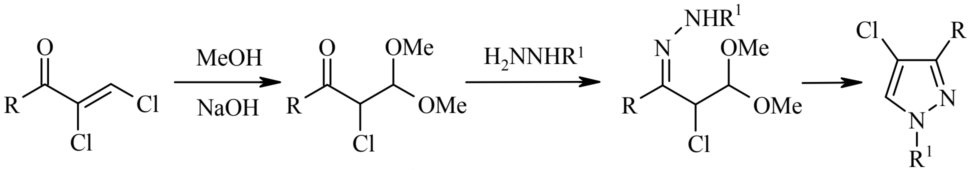


R = Bu, *t*-Bu, Ph, 4-$O_2NC_6H_4$, Et(Me)C(OH), Pr(Me)C(OH), Am(Et)C(OH), Me_3Si, Et_3Si, Et_3Ge, Ph_3Ge



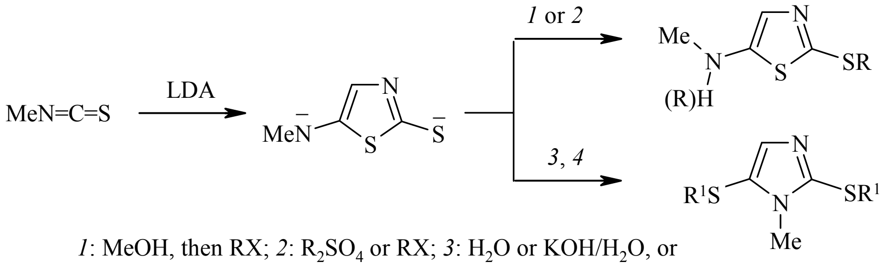


R = NH_2, N=CHPh; R^1 = Ph, 2-thienyl;   R = H; R^1 = R^2 = Me; R^1 = Me, R^2 = Et; R^1–R^2 = $(CH_2)_5$



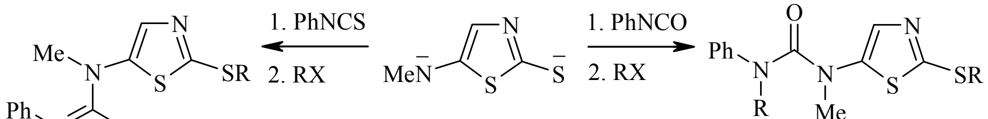
R = Alk; R^1 = Alk, Bn, Ph, 2,4-$(O_2N)_2C_6H_3$

Low-temperature reactions of isothiocyanates with lithium and potassium amides were discovered and studied systematically, and a fundamentally new strategy for the one-pot assembly of pyrroles [99, 102, 171-173], thiazoles [99, 273-278], and imidazoles [277-281] was developed from them.



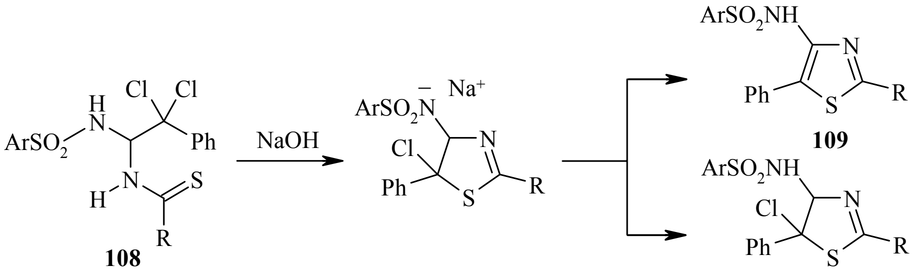
1: MeOH, then RX; *2*: R_2SO_4 or RX; *3*: H_2O or KOH/H_2O, or *t*-BuOK/THF, 15–40°C; *4*: R^1X, $R^1{}_2SO_4$; R, R^1 = Alk, Bn; X = I, Br

By using a mixture of heterocumulenes it is possible to achieve the controlled cascade assembly of polyfunctionalized thiazoles [99, 277].



R = Alk, Bn; X = I, Br

The treatment of the amides of arenesulfonic acids **108** with aqueous alkali leads to 1,3-thiazoles **109** or 4,5-dihydro-1,3-thiazoles **110** [282].

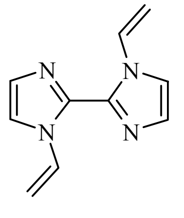


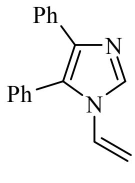
108

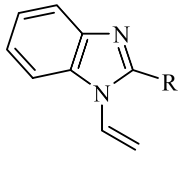
109

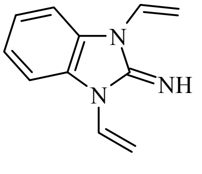
Ar = Ph, R = Me, R = NH_2; Ar = 4-ClC_6H_4, R = Me, R = NH_2 **110**

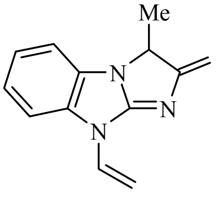
As a result of systematic investigations into the chemistry of mono-, di-, and triazole systems (G. G. Skvortsova, E. S. Domnina, L. V. Baikalova, et al.) methods were developed for the synthesis of various N-vinylimidazoles and their derivatives, and important information was obtained on their electronic and stereochemical structure [178-181, 283-290]. Extensive series of complexes of 1-vinyl-, 1-isopropenyl-, and 1-allenylazoles with the salts of metals of groups I, II, IV, VII, and VIII and HCl were synthesized and studied [178, 179, 286, 290]. Physiologically active substances that have been used in medicine were found among them.



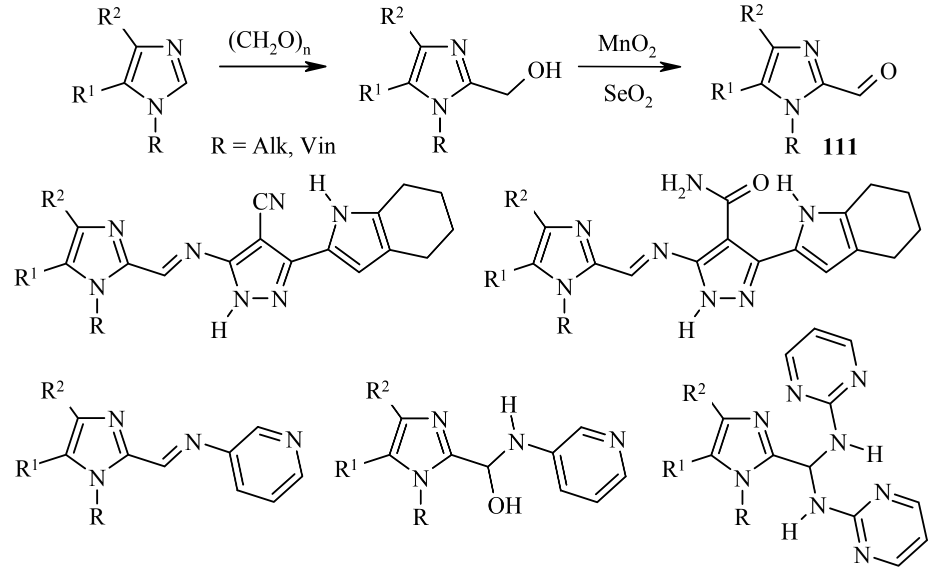




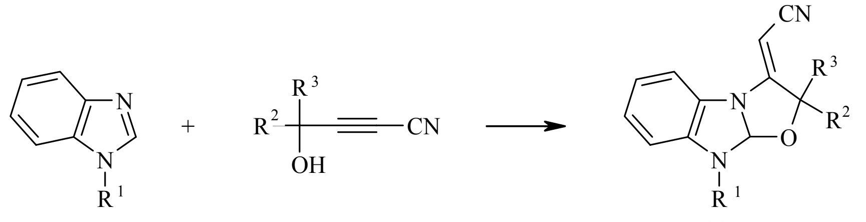




Approaches to the synthesis of imidazole-2-carbaldehydes **111** [284-286], and various hetarylazomethines and aminals, including those presented below, were obtained [286-290].

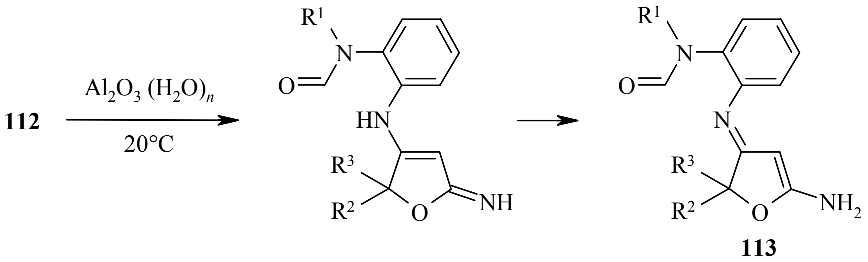


111

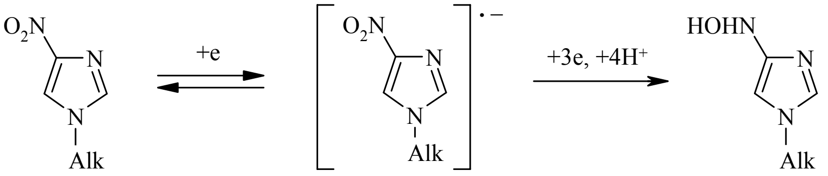


R^1 = Me, Et, Vin, All; $R^2 = R^3$ = Me; R^2–$R^3 = (CH_2)_5$ **112**

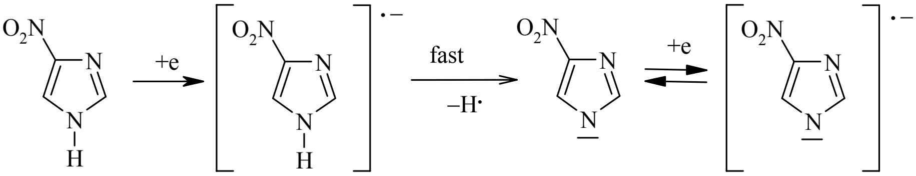
On neutral Al_2O_3 the annulation products undergo hydrolytic rearrangement with the formation of 4,5-dihydro-2-furanamines **113** [291].



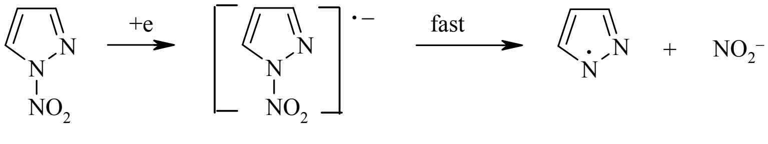
Extensive series of nitroazoles were investigated systematically by ESR and polarography (T. I. Vakul'skaya, V. A. Lopyrev, et al.), and the mechanisms of their electrochemical reduction and the structure and properties of the intermediate radical-ions were studied [292-298]. In contrast to NH-nitroazoles the transfer of the first electron is reversible for N-alkylnitroazoles, and at the first stage of reduction they form stable primary radical-anions that undergo further reduction to hydroxyamino derivatives.



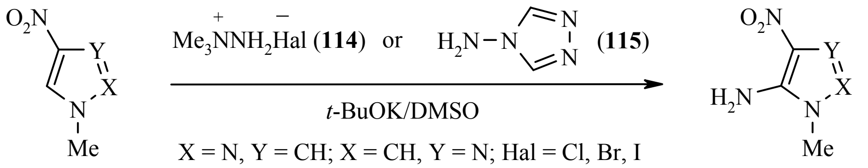
NH-Nitroazoles form stable radical-dianions at the second stage of reduction, corresponding to loss of the hydrogen attached to the nitrogen atom of the heterocycle.

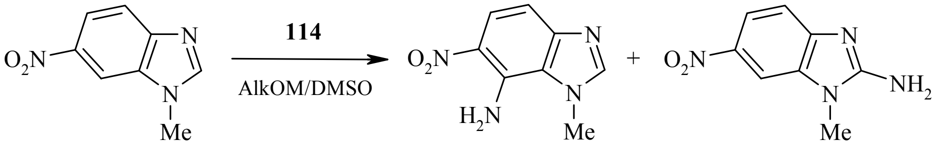


The presence of the N–NO_2 bond in the azoles greatly facilitates their electrochemical reduction. It was shown that the first wave on the polarogram of N-nitropyrazole corresponds to one-electron irreversible transfer while the second, which has a large drop before the discharge of the supporting electrolyte, corresponds to reduction of the NO_2^- anion.



The vicarious nucleophilic substitution of hydrogen was first used by V. A. Lopyrev, G. V. Dolgushin, and coworkers for the C-amination of N-methylnitroazoles by 1,1,1-trimethylhydrazinium halides **114** or 4H-1,2,4-triazole-4-amine **115** in superbasic media [299-306].



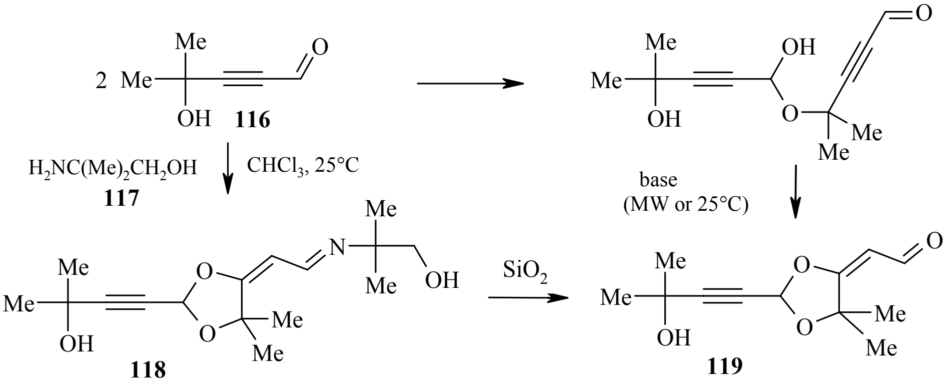


^{15}N NMR spectroscopy was used successfully to obtain evidence for the structure of the reaction products [299-303, 307, 308]. Both the features of the structure and tautomeric transformations of a wide range of substituted azoles and the structure of the products based on them were established by multinuclear and dynamic NMR spectroscopy (^{1}H, ^{13}C, ^{15}N, ^{29}Si) (L. I. Larina, V. A. Lopyrev, V. A. Shagun, et al.) [307-311].

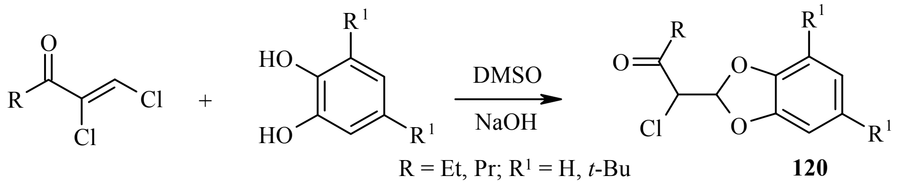
3.2. Dioxolanes and Dithiolanes

A large number of papers written under the guidance of A. S. Atavin and B. A. Trofimov were devoted to various aspects of the chemistry of the cyclic acetals and ketals of glycols and glycerol [4-7, 10, 312-324]. Suitable methods were developed for the synthesis of cyclic acetals [4-6, 312, 313] and also their various functional derivatives, including vinyl [312-315], propargyl, allenyl, and glycidyl ethers, which were subsequently used in syntheses as anchor 1,3-dioxolanes [316-322].

The reaction of the propynal **116** with 2-amino-1-propanol **117** leads to the 1,3-dioxolane **118**, which is transformed during chromatography on SiO_2 into (1,3-dioxolan-4-ylidene)acetaldehyde **119** [325]. The same product was obtained (I. A. Novokshonova, A. S. Medvedeva) by dimerization of the propynal **116**.

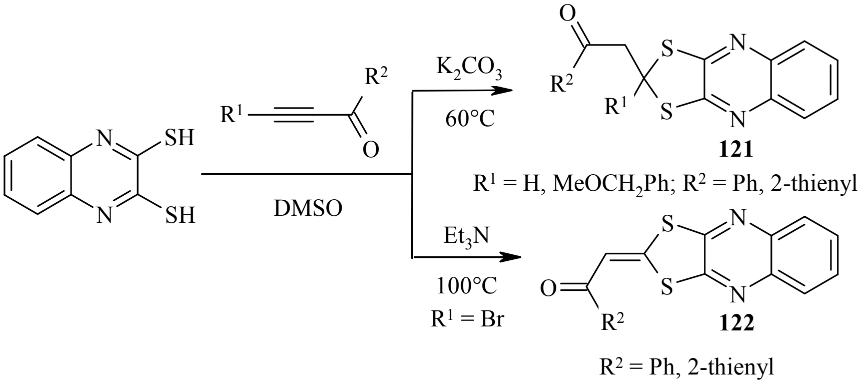


Apart from the production of pyrazoles alkyl 1,2-dichlorovinyl ketones were also used (A. N. Mirskova, G. G. Levkovskaya, et al.) in the synthesis of chlorine-substituted ketones of the dioxolane series **120** [271].



R = Et, Pr; R^1 = H, *t*-Bu **120**

Derivatives of [1,3]-dithiolo[4,5-*b*]quinoxaline **121** and **122** were synthesized from α-acetylenic ketones and 2,3-quinoxalinedithiol (A. S. Nakhmanovich, G. G. Skvortsova, et al.) [326].


121

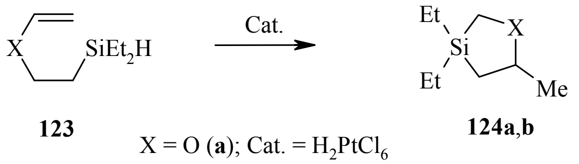
R^1 = H, $MeOCH_2Ph$; R^2 = Ph, 2-thienyl

122

R^2 = Ph, 2-thienyl

3.3. Oxa- and Thiasilolanes, Selenasila- and Tellurasilafulvenes

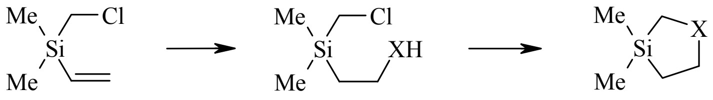
M. G. Voronkov and S. V. Kirpichenko showed that the β-monoadducts **123**, produced by the hydrosilylation of divinyl ether or divinyl sulfide, undergo catalytic intramolecular cyclization and are converted into five-membered 1,3-Si,O- (**124a**) or 1,3-Si,S-heterocycles **124b** respectively [327, 328].


123 **124a**,**b**

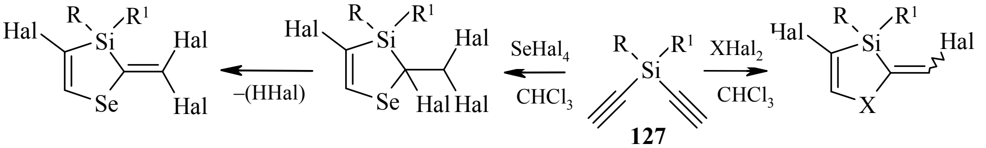
X = O (**a**); Cat. = H_2PtCl_6

X = S (**b**); Cat. = $(Ph_3P)_3RhCl$

An effective method for the production of the analogous unsubstituted heterocycles **125** is intramolecular nucleophilic cyclization of dicarbofunctional silanes **126** [329, 330].

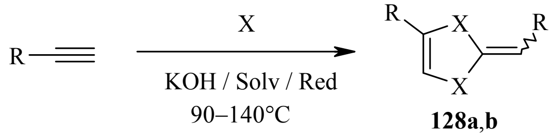

126 X = O, S **125**

Starting from the diethynylsilanes **127** and selenium and tellurium halides V. A. Potapov, S. V. Amosova, O. G. Yarosh, and coauthors developed approaches to the synthesis of 2,3-dihydro-1,3-selenasiloles and 1,4-selenasila- and 1,4-tellurasilafulvenes [331-335].


127

X = Se, $TeCl_2$; Hal = Cl, Br; R = R^1 = Me; R–R^1 = $(CH_2)_n$, *n* = 4, 5; R = Me, R^1 = $(CH_2)_2SiMe_3$

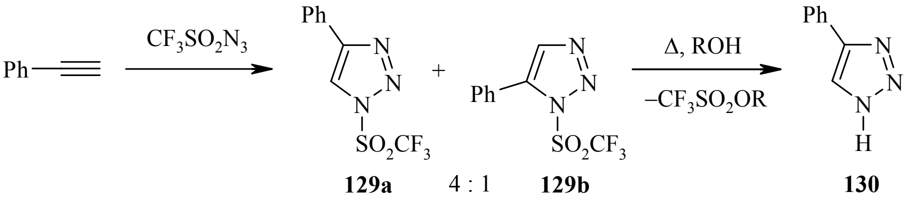
In the presence of water the ethyneselenolate anions generated from acetylenide anions and elemental selenium undergo dimerization with the formation of 1,4-diselenafulvenes **128a** [336]. The reaction of elemental tellurium with phenylacetylene in the KOH/HMPA/$SnCl_2/H_2O$ system leads to 1,4-ditellurafulvene **128b** (R = Ph), whereas with acetylene either 1,4-ditellurafulvenes ($SnCl_2$) or 1,4-ditellurins ($N_2H_4{\cdot}H_2O$) are formed [337].



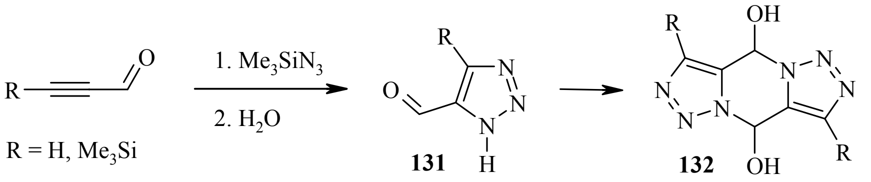
X = Se (**a**), Te (**b**); R = H, Ph; Solv = DMSO, HMPA; Red = $N_2H_4 \cdot H_2O$, $SnCl_2$

3.4. Triazoles and Thiadiazoles

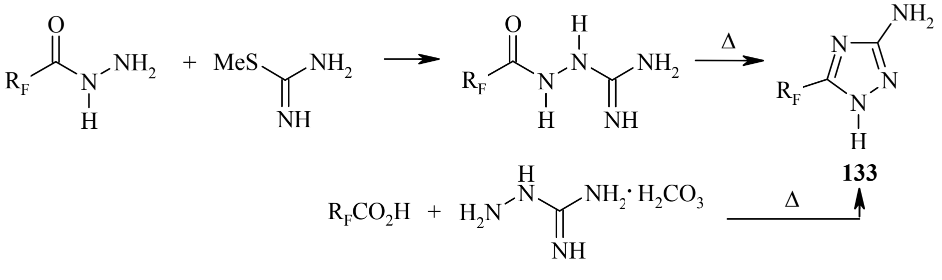
The 1,3-cycloaddition of trifluoromethanesulfonyl azide to phenylacetylene, which leads to isomeric 1'-trifluoromethanesulfonyl-4(5)-phenyl-1H-1,2,3-triazoles **129** (B. A. Shainyan, V. I. Meshcheryakov), can serve as a convenient method for the production of N-unsubstituted 1H-1,2,3-triazole **130** on account of the simple elimination of the highly nucleofugic CF_3SO_2 group [344].



N-Unsubstituted 1H-1,2,3-triazole-5-carbaldehydes **131**, which dimerize to tricyclic bis(hemiaminals) **132** with increase in the temperature and polarity of the medium, were produced by the 1,3-dipolar cycloaddition of trimethylsilyl azide to propynals (A. S. Medvedeva et al.) [345].

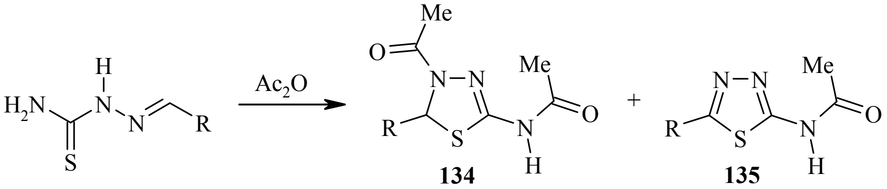


V. A. Lopyrev and coworkers realized a targeted synthesis of functionally substituted 1,2,4-triazoles and studied their acid–base characteristics and synthetic potential [346-356]. Methods were developed for the synthesis of various derivatives of amino- and diamino-1,2,4-triazoles, including fluorine-substituted 1H-1,2,4-triazole-3-amines **133** [346, 354].



R_F = CF_3, C_2F_5, C_3F_7, C_6F_{13}, $CF_3OCF_2CF_2$

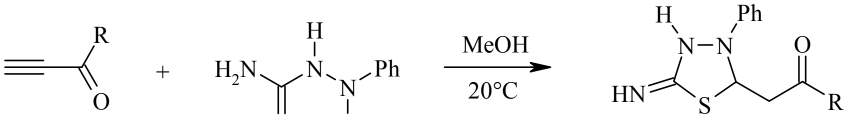
Polyfluoroalkyl-1,3,4-oxadiazoles, which are of great practical interest, were synthesized and investigated [346, 355-358]. N-(4,5-Dihydro-1,3,4-thiadiazol-2-yl)- and N-(1,3,4-thiadiazol-2-yl)acetamides **134** and **135** were obtained during the acetylation of thiosemicarbazides with acetic anhydride [308, 359].



134 **135**

R = 4-$MeOC_6H_4$, 2-, 3-, 4-Py, 2-thienyl, 2-furyl, indol-3-yl, 3-methyl-2-oxo-2,3-dihydroindol-3-yl

A. S. Nakhmanovich and T. E. Glotova synthesized 1,3,4-thiadiazolidines **136** by the reaction of α-acetylenic ketones with 1-phenylthiosemicarbazide [360].

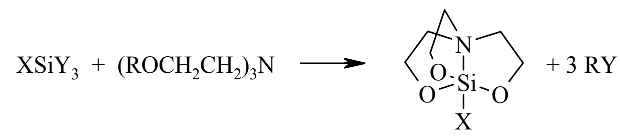


R = Ph, 2-thienyl

136

3.5. Silatranes

As a result of fundamental investigations by M. G. Voronkov's school in the chemistry, physical chemistry, biology, and pharmacology of silatranes (chelated tricyclic silicon ethers of triethanolamine) and also other compounds of hypervalent silicon, silicon has become the basis of a new branch of science – bio-silicon organic chemistry [361-369]. During this period hundreds of silatranes have been synthesized and investigated, and new theoretical data initiating vigorous development in the chemistry of hypervalent silicon were obtained [64, 308, 365, 369-372]. The wide variety of methods used for the production of silatranes [64, 361-364] is represented in general form in the following scheme.



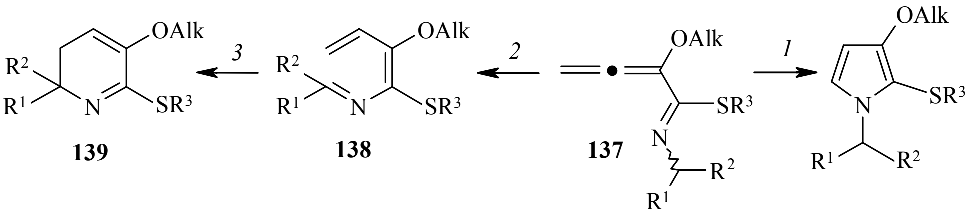
X = organyl, OR'; Y = OR^1, F, Cl, H, NR_2, $OC(O)R^2$; R = H, $SiMe_3$, $SnMe_3$

Numerous derivatives and analogs of silatranes have been synthesized [364, 273]. Organic chelate compounds of pentacoordinated germanium (1-organylgermatranes) were also discovered by M. G. Voronkov's school, and their physicochemical characteristics and biological activity were studied [374].

4. SIX- AND SEVEN-MEMBERED HETEROCYCLES

4.1. Pyridines and Quinolines

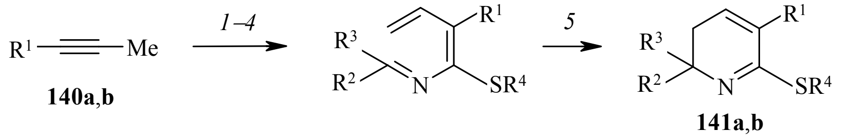
The low-temperature reactions of metalloalkynes and metallodienes with isothiocyanates made not only the pyrroles, thiophenes, and thietanes discussed above but also such valuable nitrogen-containing heterocycles as 2,3- and 1,2-dihydropyridines, pyridines, dihydropyridinones, and pyridinethiones accessible [99, 101, 130, 142, 375-392]. When heated in the absence of a catalyst (salts of monovalent copper) 1-aza-1,3,4-trienes **137**, formed during the reaction of lithiated alkoxyallenes with aliphatic isothiocyanates, go through two competing processes – intramolecular cyclization to pyrrole and isomerisation (through a [1,5]-H sigmatropic shift) to 2-aza-1,3,5-trienes **138**, the electrocyclization of which leads to a new type of 5,6-disubstituted 2,3-dihydropyridines **139** [99, 130, 375-378]. All stages of the process – from lithiation of the allene to the formation of the heterocyclic structures – are carried out in one reactor without isolation of the intermediates.



1: Δ, 1,5-cyclization; *2*: Δ, [1,5]-H shift; *3*: Δ, 6π-electrocyclization

R^1, R^2 = H, Alk; R^1 = H, R^2 = Alk, $VinOCH_2$, MeO; $R^1–R^2$ = $(CH_2)_n$, *n* = 4, 5; R^3 = Alk

The only products from the reaction of the carbanions generated from 1-(alkysulfanyl)-1-propynes **140a** and 2-alkynes **140b** with aliphatic isothiocyanates are 5,6-di(alkylsulfanyl)- [379] and 5-alkyl-6-(alkylsulfanyl)-2,3-dihydropyridines [99, 377, 380] **141a** and **141b,** respectively.

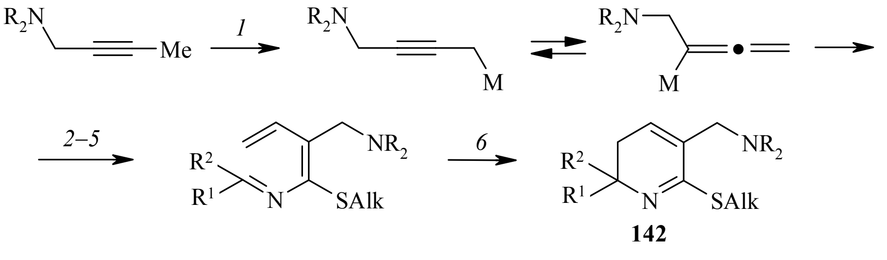


1: BuLi/THF/C_6H_{14}; *2*: $R^2R^3CHN{=}C{=}S$; *3*: R^4X; *4*: [1,5]-H shift;

5: Δ, 6π-electrocyclization;

R^1 = SAlk (**a**), Alk (**b**); R^4 = Alk; R^2, R^3 = H, Alk; $R^2–R^3$ = $(CH_2)_n$, *n* = 4, 5; X = Br, I

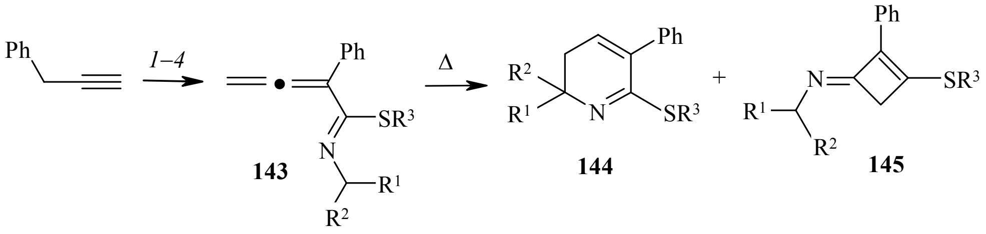
A new class of 5-(N,N-dialkylamino)methyl-2,3-dihydropyridines **142** was obtained from isothiocyanates and diethyl- and dipropylamino-2-butynes [99, 142].



1: BuLi/*t*-BuOK/THF/C_6H_{14}; *2*: LiBr/THF; *3*: $R^1R^2CHN{=}C{=}S$; *4*: AlkI; *5*: Δ, [1,5]-H shift;

6: electrocyclization; R = Et, Pr; R^1, R^2 = H, Alk, $R^1–R^2$ = $(CH_2)_5$; M = K/*t*-BuOLi

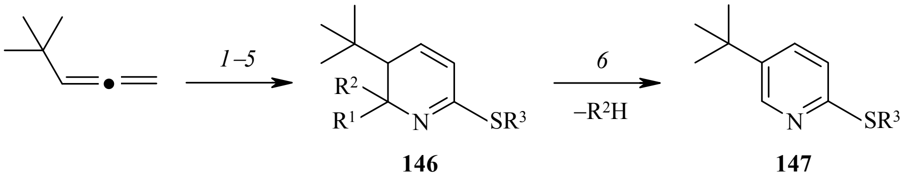
The transformation of the 1-aza-1,2,4-trienes **143**, generated from 1,3-dilithiopropargylbenzene and alkyl isothiocyanates, to 5-phenyl-2,3-dihydropyridines **144** is accompanied by an unusual thermal rearrangement to the structurally isomeric iminocyclobutenes **145** [381].



143 **144** **145**

1: 2 BuLi/THF/C_6H_{14}; *2*: $R^1R^2CHN=C=S$; *3*: *t*-BuOH; *4*: R^3I

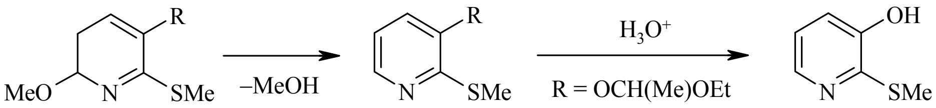
γ-Lithiated *tert*-butylallene and isothiocyanates were found to be suitable starting compounds for a one-pot synthesis of *tert*-butyl-substituted 2,3-dihydropyridines **146** and pyridines **147** [99, 382, 383].



146 **147**

1: BuLi/THF/C_6H_{14}; *2*: $R^1R^2CHN=C=S$; *3*: R^3X; *4*: [1,5]-H shift;
5: Δ, 6π-electrocyclization; *6*: 200–220°C, 10–15 min
$R^1 = H$: $R^2 = H$, Alk, $VinOCH_2$; $R^1 = R^2 = Me$; $R^1–R^2 = (CH_2)_n$, *n* = 4, 5; X = Br, I

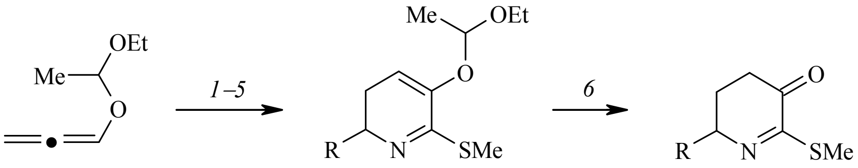
2,3-Dihydropyridines **148**, obtained from allene carbanions and methoxymethyl isothiocyanate, can be easily converted into pyridines **149** by the thermal or acid-catalyzed elimination of methanol [384, 385]. Removal of the acetal protection from the latter [when R = OCH(Me)OEt] leads to the first synthetic analog of natural 2-(methylsulfanyl)-3-pyridinol **150** [386, 387].



148 **149** **150**

R = Me, OMe, SMe, OCH(Me)OEt

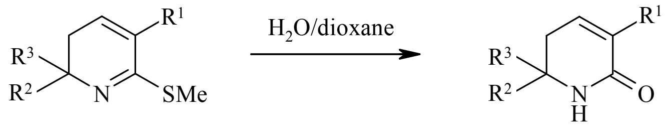
A simple method for the synthesis of acetals of the dihydropyridines series **151** and 5,6-dihydro-3(4H)-pyridinones **152** was found on the basis of carbo- and heterocumulene precursors [388].



151 **152**

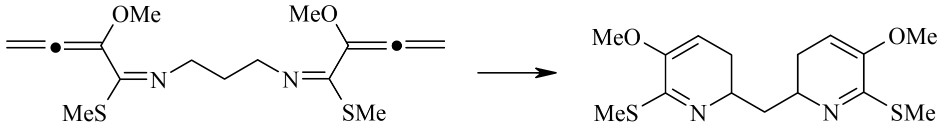
1: BuLi/THF/C_6H_{14}; *2*: $RCH_2N=C=S$; *3*: MeI; *4*: Δ, [1,5]-H shift; *5*: Δ, cyclization ; *6*: H^+/H_2O

An unusual 1,2-hydration of endocyclic 1-azadienes of the dihydropyridine series **153**, providing a simple route to 5,6-dihydro-2(1H)-pyridinones **154**, was discovered (N. A. Nedolya) [389].



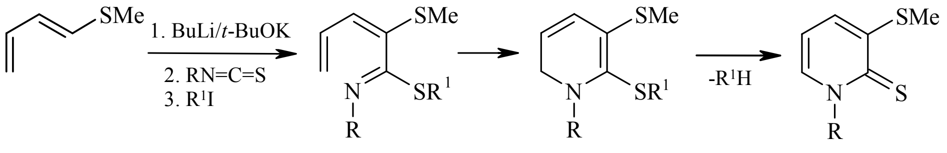
153 R^1 = Me, MeO, Ph; R^2, R^3 = H, Alk **154**

The action of heat on the bis(1-aza-1,3,4-triene) **155** (an S-alkylated diadduct of lithiated methoxyallene and 1,3-diisothiocyanatopropane) leads to the bis(2,3-dihydropyridine) **156** [147].



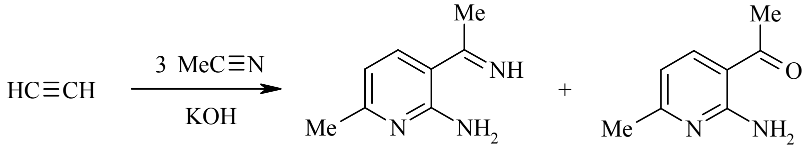
155 **156**

The reaction of α-metallated (1*E*)-1-(alkylsulfanyl)-1,3-butadienes with isothiocyanates leads to one-pot assembly of 1,2-dihydropyridines **157** [99, 391], the action of heat on which (180-200°C, 10-15 min) is accompanied by an unusual cleavage of the S–R^1 bond with elimination of the alkane leading to 1,2-dihydro-2-pyridinethiones **158** [392].



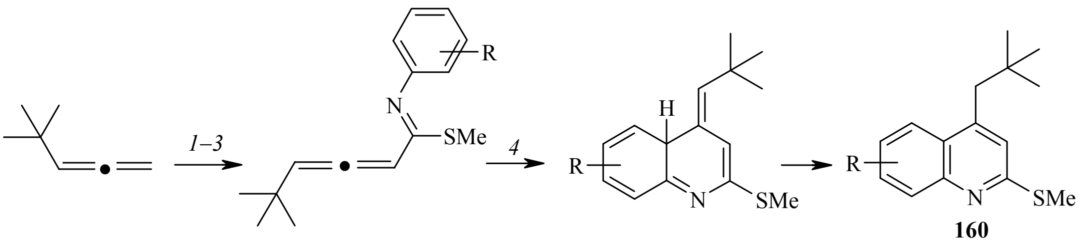
157 **158**

The cyclization of acetylene with three molecules of acetonitrile, leading to 2-aminopyridines **159a**,**b**, was discovered (B. A. Trofimov et al.) [393].



159a **159b**

A conceptually new method was found and developed (N. A. Nedolya and coworkers) for the synthesis of di- and polysubstituted quinoline structures – by the reaction of lithiated allenes or alkynes with aromatic isothiocyanates or isocyanates in a single preparative stage [99, 101, 381, 394-399]. 2-(Alkylsulfanyl)-4-neopentyl-quinolines **160** were obtained in this way from γ-lithiated *tert*-butylallene and aryl isothiocyanates [99, 395].

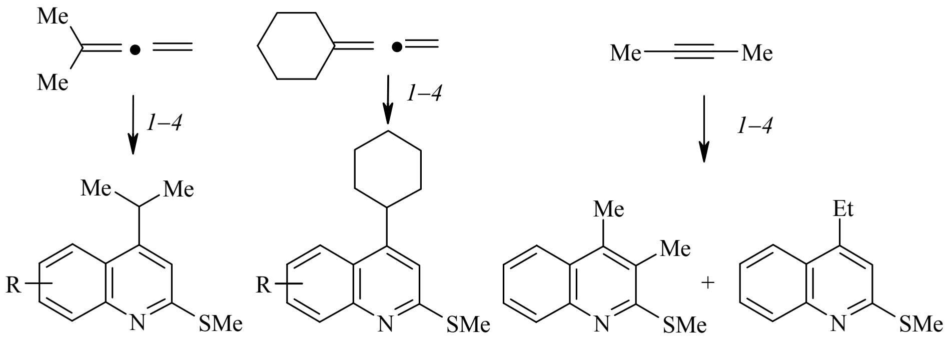


160

1: BuLi/THF/C_6H_{14}; *2*: RC_6H_4N=C=S; *3*: MeI; *4*: Δ, electrocyclization

R = H, 2-F, 3-F, 4-F, 4-Cl, 4-Br, 2-CF_3, 3-CF_3, 4-CF_3, 2-MeO, 4-MeO, 4-Me_2N, 4-Me

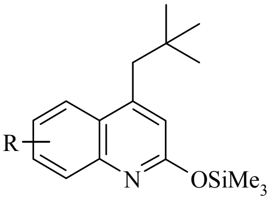
4-Isopropylquinoline [396] and 4-cyclohexylquinoline [397] **161** and **162** were synthesized similarly (from 1,1-dimethylallene and ethenylidenecyclohexane respectively), whereas a mixture of 3,4-dimethylquinoline (80%) and 4-ethylquinoline (20%) **163** and **164** was obtained from 2-butyne. This was due to the participation not only of allene but also of acetylene carbanions in reaction with the aryl isothiocyanate [99, 398].



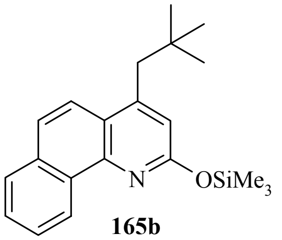
161 **162** **163** **164**

1: BuLi/THF/C_6H_{14}; *2*: $RC_6H_4N{=}C{=}S$; *3*: MeI; *4*: Δ, electrocyclization

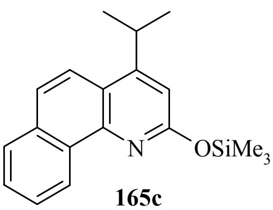
2-(Trimethylsilyloxy)quinolines **165a** and 2-(trimethylsilyloxy)benzo[*h*]quinolines **165b**,**c** were made accessible by the use of aromatic isocyanates in reaction with lithiated allenes [99, 399].



165a

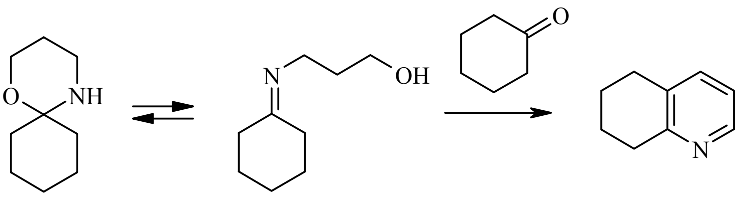


165b

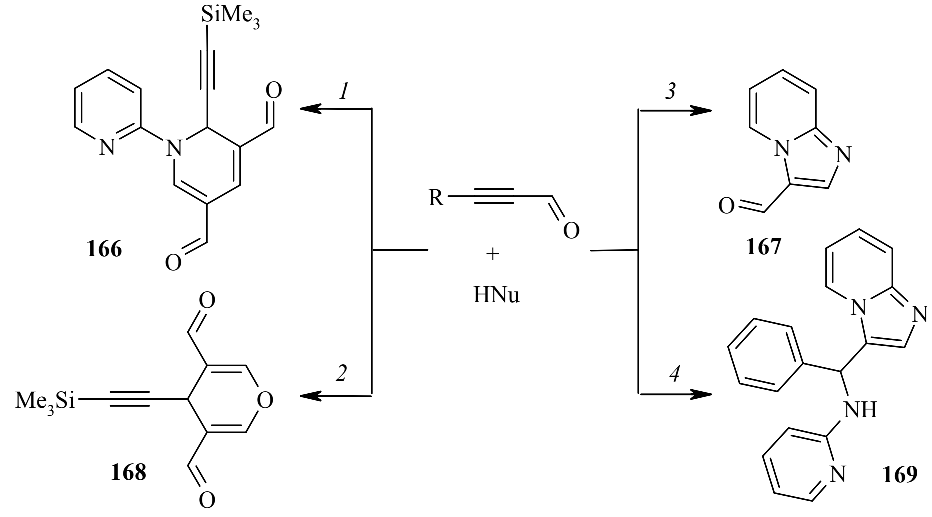


165c

In the case of 1-oxa-5-azaspiro[5.5]undecane it was shown (B. F. Kukharev, V. K. Stankevich) that the oxidation of 1,3-oxazinanes, unsubstituted at the nitrogen atom, by carbonyl compounds led to tetrahydroquinolines [400].

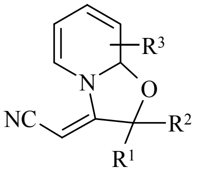


Unusual processes leading to the cascade assembly of polyfunctional nitrogen- and oxygen-containing heterocycles – 2H-1,2'-bipyridine **166**, imidazo[1,2-*a*]pyridine **167**, and 4H-pyran **168** [403] – from element-containing propynals and 2-pyridineamine or water were discovered (A. S. Medvedeva et al.) [401-403]. Imidazo[1,2-*a*]pyridine **169** is formed during the acid-catalyzed reaction of 2-pyridineamine with phenylpropynal.



1: R = Me_3Si, Nu = 2-H_2NPy; *2*: R = Me_3Si, Nu = H_2O; *3*: R = Me_3Si, Et_3Ge, $RC{\equiv}CCH_2OH$, MnO_2/SiO_2; *4*: R = Ph, Nu = 2-H_2NPy

The heterocyclization of the nitriles of α,β-acetylenic γ-hydroxy acids with pyridine, quinoline, 6-methyl-1,2,3,4,7,8,9,10-octahydrophenanthridine and quinoxaline gave [1,3]oxazolo[3,2-*a*]pyridines **170** [404], [1,3]oxazolo[3,2-*a*]quinolines **171**, [1,3]oxazolo[3,2-*f*]phenanthridines **172**, and bis[1,3]oxazolo[2,3-*c*:3',2'-*a*]quinoxalines **173** [405-410].



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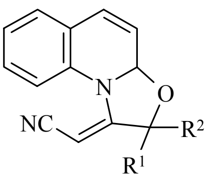

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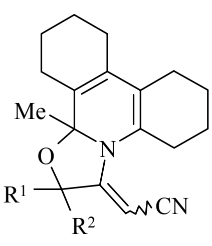

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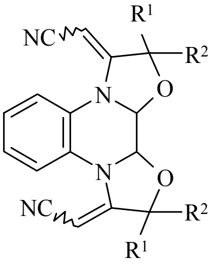
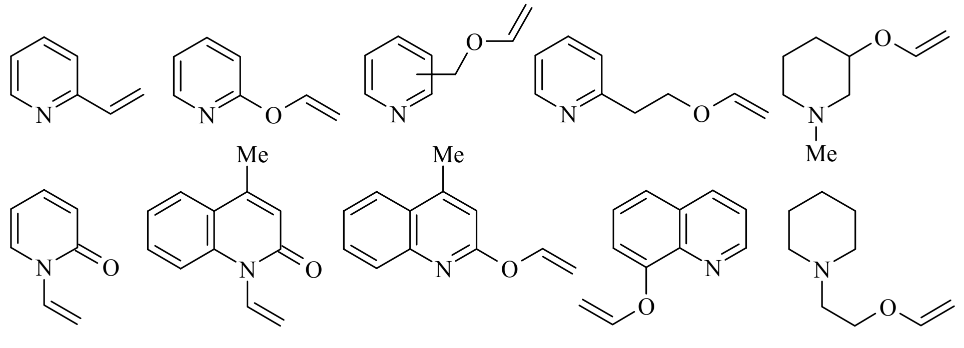


173

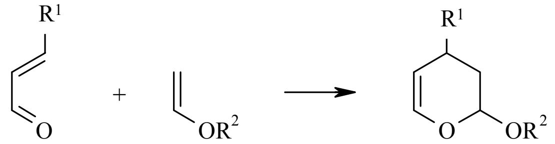
Pioneering investigations in the chemistry of vinyl derivatives of six-membered azaheterocycles – pyridylalkanols, 8-quinolinol, 1-methyl-3-piperidinol, 2-(1-piperidinyl)ethanol, 2(1H)-pyridinone, and 4-methyl-2-(1H)-quinolinone – were carried out by G. G. Skvortsova et al. [178]. The first representatives of new families of C-, N-, and O-vinyl derivatives of the investigated azaheterocycles were synthesized by direct vinylation with acetylene, and their properties were studied.



Various heterocyclic compounds were obtained (B. A. Trofimov, S. V. Amosova, et al.) from divinyl sulfide – a multidisciplinary intermediate of contemporary organic synthesis [411-413]. In the case of halogen-containing pyridines and divinyl sulfide a series of new (halo)vinylsulfanylpyridines were obtained [414-418], and their oxidation with an excess of hydrogen peroxide gave the corresponding sulfonylpyridines [417, 418].

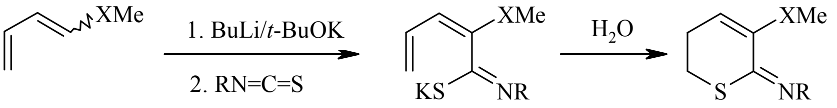
4.2. Pyrans, Thiopyrans

The condensation of α,β-unsaturated carbonyl compounds with the vinyl ethers of phenols, α,β-naphthols [178, 419-421], alcohols of the piperidine, pyridine, and quinoline series (G. G. Skvortsova et al.) [178, 422], polyfluoroalkanols (N. A. Nedolya, L. L. Dmitrieva) [16, 423, 424], and also with the mono- and divinyl ethers of diols (A. S. Atavin, V. I. Lavrov, V. K. Stankevich) [36, 425-428] leads to 3,4-dihydro-2H-pyrans. Involvement of the latter in various reactions with participation of the substituents or dihydropyran ring led to the production of new families of functionalized dihydropyrans [36, 427, 429-431] and tetrahydropyrans [178].



R^1 = H, Me, Ph; R^2 = Ar, Het, $(CH_2)_nOH$, $n = 2–4$, $CH_2(CF_2CF_2)_nH$, $n = 1–3$ etc.

The reaction of the carbanions generated *in situ* from 1,3-butadienes with isothiocyanates leads to 2-imino-5,6-dihydro-2H-thiopyrans **174** (N. A. Nedolya and coworkers) [99, 101, 432, 433].

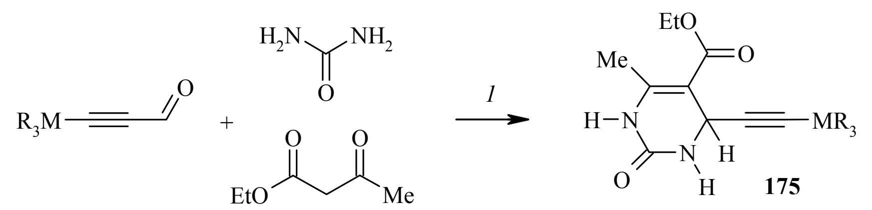


X = O, S; R = Alk, Ar

174

4.3. Pyrimidines, Thiazines, Selenazines, Dioxanes, Aza-, Oxa-, and Thiasilinanes

3,4-Dihydro-2(1H)-pyrimidinones **175** were first synthesized in the laboratory of A. S. Medvedeva under the conditions of the Biginelli reaction by boiling 3-(trimethylsilyl)- and 3-(triethylgermyl)-2-propynals with urea and acetoacetic ester in methanol in the presence of HCl [434].

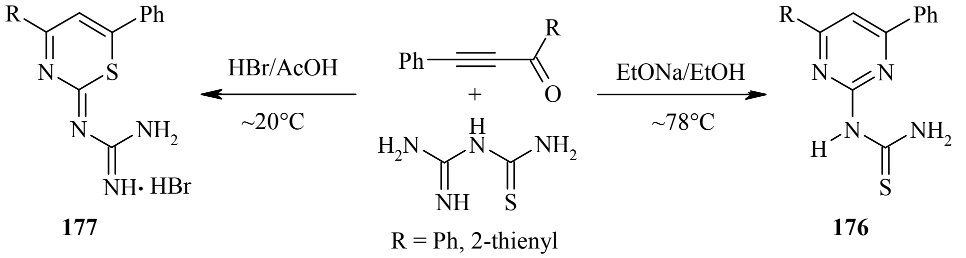


175

$R_3M = Me_3Si, Et_3Ge$

1: MeOH, HCl (5 mol %), 60°C, 25 h

New polyfunctional derivatives of pyrimidine **176** and 1,3-thiazine **177** were obtained from 2-acyl-1-phenylacetylenes and N-[amino(imino)methyl]thiourea (T. E. Glotova et al.) [435].

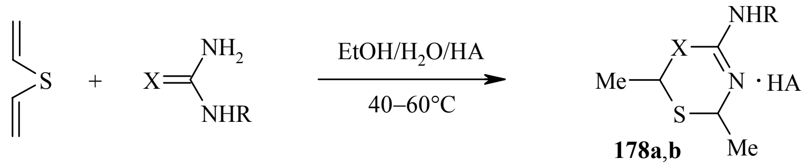


177

176

R = Ph, 2-thienyl

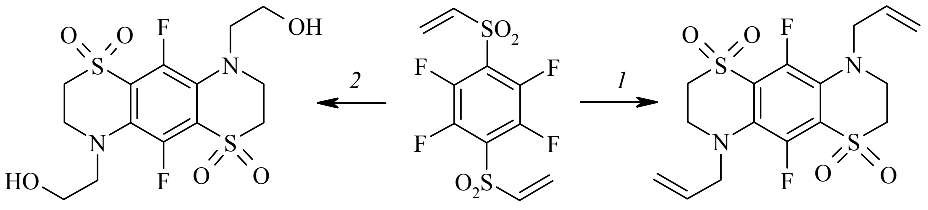
1,3,5-Dithiazinium **178a** and 1,3,5-thiaselenazinium **178b** salts were obtained by B. A. Trofimov, S. V. Amosova, and coworkers from divinyl sulfide and thiourea or selenourea in the presence of strong acids [436-440].



178a,**b**

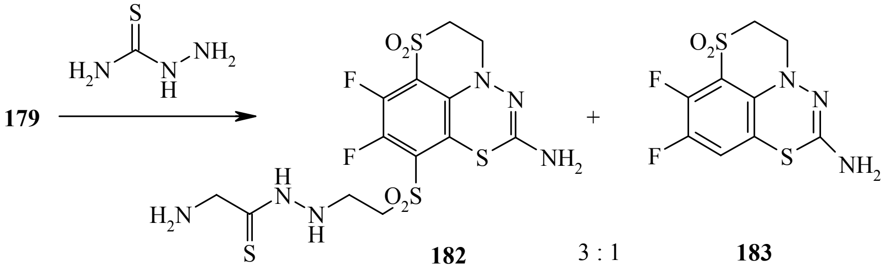
X = S (**a**), Se (**b**); R = H, Me, Et, Ph; HA = HCl, HBr, $HClO_4$, *p*-$TolSO_3H$

The reaction of 3,6-bis(vinylsulfonyl)-1,2,4,5-tetrafluorobenzene **179** with amines provides a simple path to [1,4]thiazino[2,3-*g*][1,4]benzothiazine 1,1,6,6-tetroxides **180** [441] and **181** [442], and the reaction with thiosemicarbazides leads to [1,4]thiazino[2,3,4-*ij*][4,1,2]benzothiadiazine 7,7-dioxides **182** and **183** (S. V. Amosova and coworkers) [443, 444].



181 **179** **180**

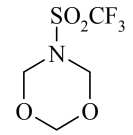
DMFA or EtOH, 50–55°C: $AllNH_2$ (*1*); $HO(CH_2)_2NH_2$ (*2*)

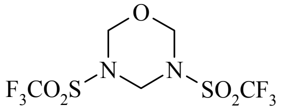


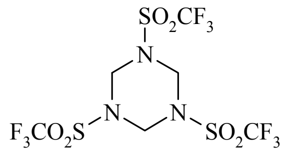
182 3 : 1 **183**

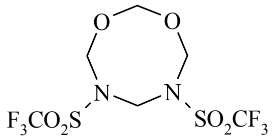
The reaction of triflamide with paraform in sulfuric acid at various temperatures with the reagents in various ratios led to the formation of a whole series of linear and heterocyclic condensation products presented below (B. A. Shainyan and coworkers) [445].

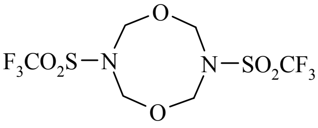
$CF_3SO_2NHCH_2NHSO_2CF_3$ $CF_3SO_2N(CH_2NHSO_2CF_3)_2$

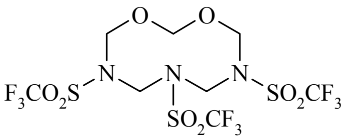




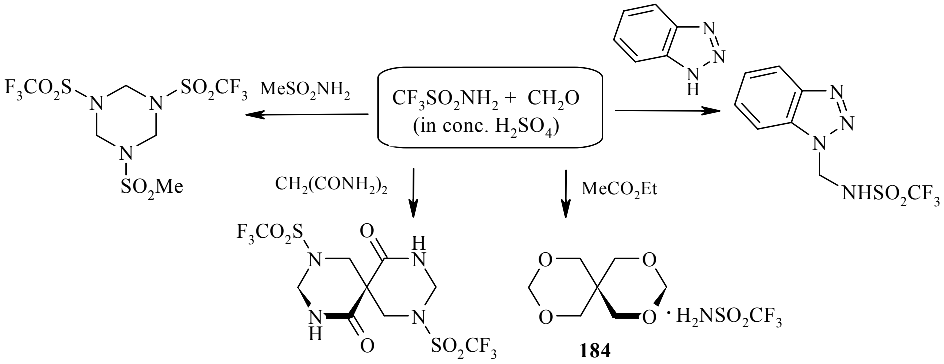






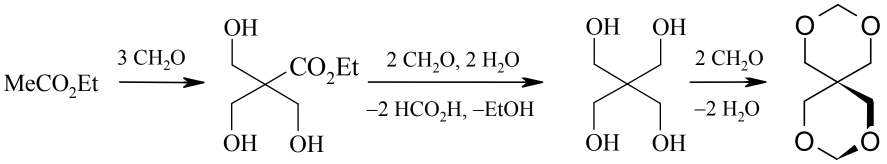


In a number of cases three-component condensations with paraform and triflamide also led to the products from heterocyclization [446].



184

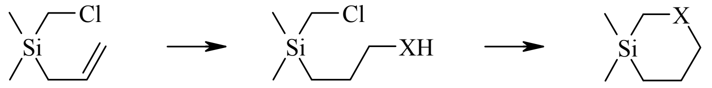
When the reaction was carried out in ethyl acetate the 1:1 complex of 2,4,8,10-tetraoxaspiro[5,5]undecane with triflamide **184** was unexpectedly obtained [447]. A reaction scheme confirming the formation of this spirocycle when paraform is heated with ethyl acetate in sulfuric acid was proposed.



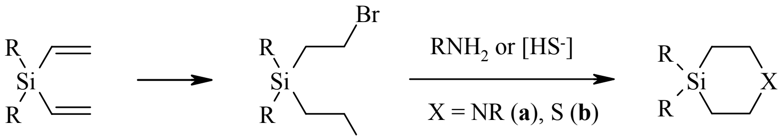
The discovered reaction is the first example of the reduction of a CO_2R group to CH_2OH by the action of formaldehyde.

The stereodynamic behavior was studied for all the obtained heterocycles by low-temperature multinuclear NMR, and the presence of rotamers with an *exo*- and *endo*-oriented CF_3SO_2 group was demonstrated [448-450].

In the same laboratory allylsilanes were converted easily into 1,3-oxa- and 1,3-thiasilinanes **185a** and **185b** [451], and 1,4-aza- and 1,4-thiasilinanes **186a** and **187b** were obtained by dihydrobromination of divinyl(diorganyl)silanes followed by cyclization by the action of N- or S-nucleophiles [452].

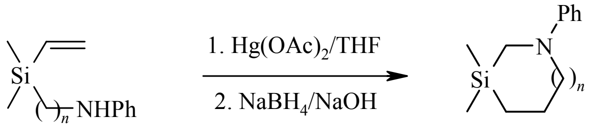


X = O (**a**), S (**b**) **185a**,**b**



186a,**b**

Electrophilic cyclization of aminoalkylvinylsilanes leads to five-, six-, and seven-membered Si,N-containing heterocycles [453].

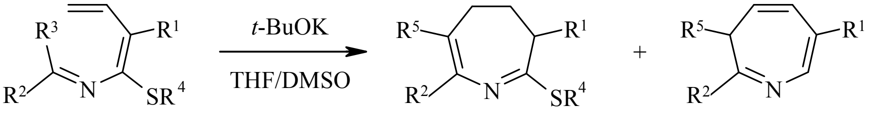


187

The analogous reaction of aminoalkylallylsilanes leads to α-substituted five- and six-membered Si,N-containing heterocycles [453].

4.3. Azepines, Diazepines

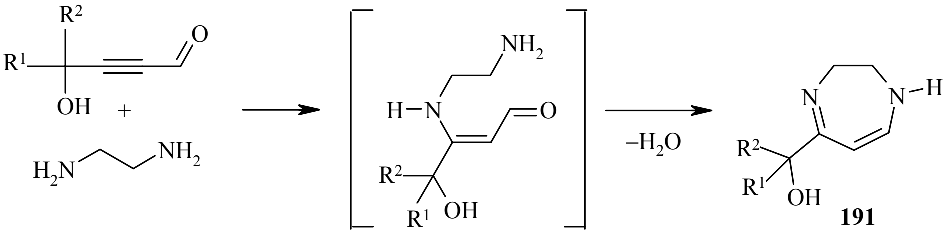
In N. A. Nedolya's group it was shown for the first time that conjugated 2-aza-1,3,5-trienes **188**, generated from polyunsaturated carbanions and isothiocyanates in a single preparative stage [99, 101], can provide a fundamentally new promising source of new families of azacycloheptadienes **189** and trienes **190** [454-456].



188 **189** **190**

R^1 = OAlk, OCH(Me)OAlk, Ph, 1-pyrrolyl; R^2 = H, Me, Et; R^3 = Me; $R^2–R^3 = (CH_2)_4$;
R^4 = Alk; R^5 = H, $R^2–R^5 = (CH_2)_3$

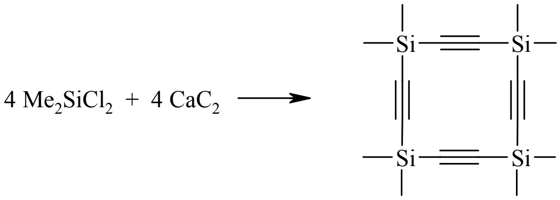
2,3-Dihydro-1H-1,4-diazepines **191** were obtained from acetylenic γ-hydroxy aldehydes and 1,2-ethylenediamine (A. S. Medvedeva and coworkers) [457].



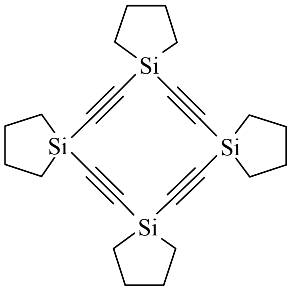
$R^1 = R^2$ = Me; R^1 = Me, R^2 = Et; R^1 = Me, R^2 = i-Pr; $R^1–R^2 = (CH_2)_4$

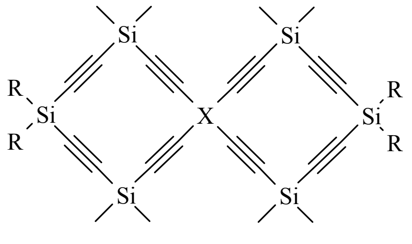
Macroheterocycles

New types of highly unsaturated 12-30-membered macrocyclic organosilicon systems, constructed from $–Si(R)_2CH{=}CH–$ and $Si(R)_2C{\equiv}C–$ units, and synthons for their synthesis were obtained and investigated under the leadership of M. G. Voronkov (O. G. Yarosh, L. V. Zhilitskaya) [458, 459]. The first macrocyclic silicohydrocarbon, constructed from four alternating Si(Me)$_2$C≡C– fragments, was synthesized by the reaction of dimethyldichlorosilane with calcium carbide in a molten LiCl/KCl eutectic mixture at 400°C [460].

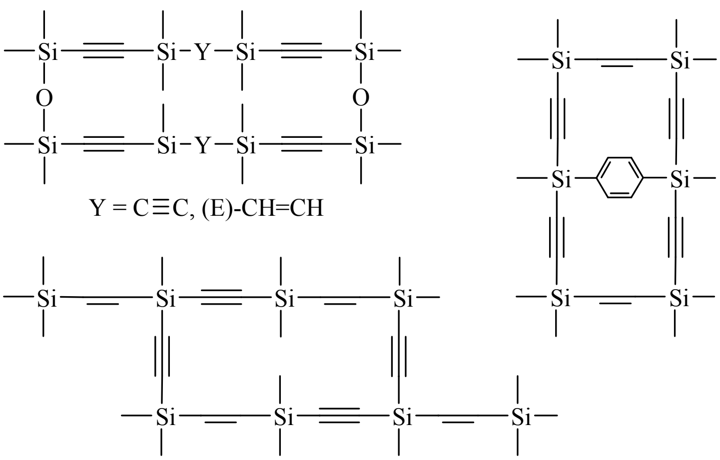


More recently obtained compounds are the homologous series of permethyl-, methyl(alkyl)-, methyl(vinyl)-, and methyl(aryl)cyclosilaethynes (R$_2$SiC≡C)$_n$ (R = Me, Alk, *cyclo*-Alk, Vin, Ar, n = 1-4) [461, 462] and also macroheterocycles simultaneously containing double and triple bonds, endocyclic fragments (CH$_2$)$_n$ (n = 1-4) [463-466] and endocyclic oxygen atoms [467, 468], and condensed and spiran bi- and tricyclic systems [458, 461, 466, 469, 470], individual representatives of which are presented below.





X = Si, Ge; R = Me, Et



Y = C≡C, (E)-CH=CH

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