UTILIZATION OF AZOMETHINES IN SYNTHESES OF HETEROCYCLIC COMPOUNDS (REVIEW)

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Methods for obtaining nitrogen-containing heterocycles in which one of the starting compounds is an azomethine are examined.

Azomethines, which were discovered almost 125 yr ago by Schiff and are obtained in many cases in quantitative yields from aldehydes, ketones, and compounds that contain a primary amino group, have found wide application in organic chemistry, industry, and medicine [1, 2]. They occupy a special place in syntheses of three-five-membered nitrogen-containing heterocycles and heterocycles that contain nitrogen, oxygen, and sulfur atoms.

Despite the significant number of publications devoted to the chemistry of Schiff bases, reactions involving addition to the exocyclic azomethine bond that lead to the formation of various heterocycles have not been adequately correlated. There are only a few examples of such reactions in a previous review [3]. Primarily reactions involving the cycloaddition of azomethine ylids and azomethine imines to alkenes and reactions involving the photocyclization of Schiff bases and the cycloaddition of azadienes are examined in subsequent reviews [4-7]. These questions have also been previously illuminated in [8-10].

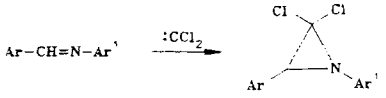
A considerable number of new methods for the synthesis of heterocyclic compounds on the basis of imines have appeared in recent years. Methods for the one-step synthesis of a number of heterocyclic compounds that have practically useful properties from azomethines have been developed, and some of them are being used with success in syntheses of alkaloids.

In the present review principal attention was directed to research on the utilization of azomethines in the synthesis of heterocycles that has been carried out in recent years.

Three-Membered Heterocycles

[2 + 1]-Cycloaddition reactions of carbenes, oxygen, and nitrenes at the azomethine bond constitute a general method for the synthesis of aziridines, oxaziridines, diaziridines, and diazirines.

Aziridines. The addition of carbenes to azomethines is an important method for obtaining aziridines [11]. This reaction was realized for the first time in the case of the reaction of N-arylideneanilines with dichlorocarbene [12]:



Dichloro-, dibromo-, chlorobromo-, and fluorochlorocarbenes, which were generated from the corresponding trihalomethanes, have been used in syntheses of geminal dihalo-substituted aziridines. Depending on the nature of the substituents attached to the carbon and nitrogen atoms of the azomethine bond, the yields of dihalo-substituted aziridines range from 30% to 90% [13]. Under interphase-catalysis conditions [14] this method made it possible to synthesize aziridines from imines obtained from aliphatic amines and aldehydes, which are usually readily converted to condensation products. Aziridines were obtained in 67-69% yields under these conditions from arylideneanilines containing elec-

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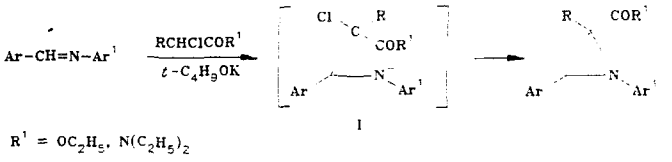
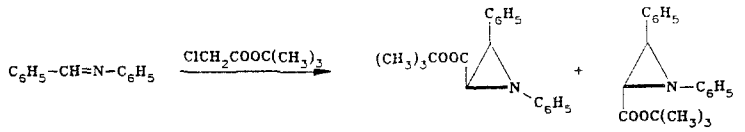
tron-acceptor substituents in aromatic rings [15, 16]. Phenyldiazomethane in the presence of zinc iodide (the Simmons–Smith method) [17] and sulfur ylids – sulfurans – [18] have been used for the generation of carbenes in reactions with imines.

Functionally substituted aziridines were obtained in 70-90% yields from aromatic imines and α-halo-substituted carboxylic acids and their esters, nitriles, and amides (the Darzens reaction) in the presence of bases [19, 20]. It is assumed that anion I with the charge on the nitrogen atom is formed as an intermediate in the reaction of imines with chloroacetic acid diethylamide in the presence of potassium tert-butoxide [19].



$R^1 = OC_2H_5, N(C_2H_5)_2$

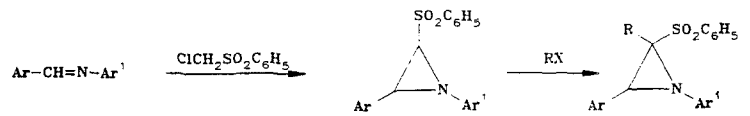
The basicity of the condensing agent affects the ratio of the cis and trans isomers of 1,2-diphenyl-3-tert-butyloxy-carbonyl aziridines formed in the condensation of benzylideneaniline with tert-butyl monochloroacetate.



Thus the ratio of the cis and trans isomers is 3:7 in presence of potassium tert-butoxide, whereas it is 19:1 in the case of lithium bis(trimethylsilyl)amide [20].

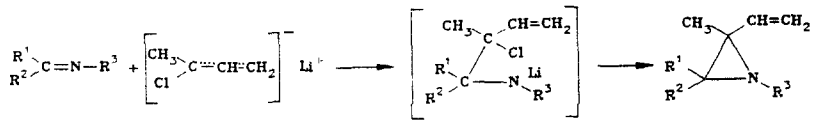
Geminal chloroalkoxycarbonyl-substituted aziridines were first obtained in up to 90% yields from aromatic azomethines and dichloroacetic acid esters in the presence of potassium isopropoxide [21]. Aliphatic imines do not undergo this reaction. The yields of aziridines from dibromoacetic acid esters do not exceed 50%.

Sulfonyl-substituted aziridines were obtained in 72-100% yields by reactions of diarylazomethines with chloromethyl phenyl sulfone in the presence of lithium diisopropylamide in THF at −78°C [22].



Stabilization of the resulting carbanion by the sulfonyl group promotes the occurrence of this reaction. The sulfone substitutent can be removed under mild conditions (Na/Hg, Na_2HPO_4, −20°C to 0°C) [23]. The sulfonyl-substituted aziridines readily form carbanions (lithium diisopropylamide, THF, −78°C), the alkylation of which gives 2,2-disubstituted aziridines in high yields.

A method for the synthesis of substituted 2-vinylaziridines from aromatic and aliphatic aldimines and ketimines and 1-lithio-3-chlorobutene, which is formed in the reaction of 1,3-dichloro-2-butene initially with triphenyllead bromide and then with butyllithium, has been developed [24]. The reaction is sensitive to steric factors. Aziridines could not be obtained from azomethines with bulky substituents attached to the carbon or nitrogen atom.

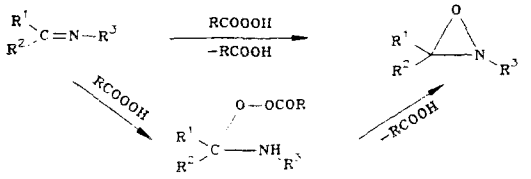


However, the synthesis of substituted 2-vinylaziridines by a different method generally includes several steps, and the starting compounds are difficult to obtain [25].

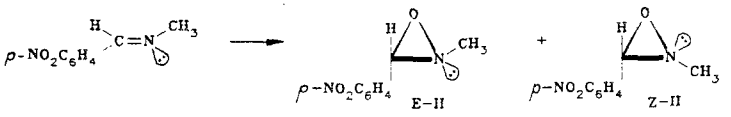
The production of 1-difluoromethyl-2,2-diphenyl-3-ethoxycarbonylaziridine by the reaction of ethyl N-dipehnylmethyliminoglycinate with difluorochloromethane has been described [26].

Oxaziridines. Oxaziridines are obtained in 50-90% yields by oxidation of imines with peracids. The instability of some amines, as well as oxaziridines, in an acidic medium limits the use of this method.

Two alternative mechanisms of the oxidation of imines have been examined [27]. A one-step mechanism involves direct nucleophilic reaction with the participation of the π-electrons of the imine bond [28, 29]. A two-step mechanism (a reaction of the Baeyer–Villiger type) assumes the addition of the peracid to the azomethine bond with the subsequent elimination of the acid and ring formation [30].

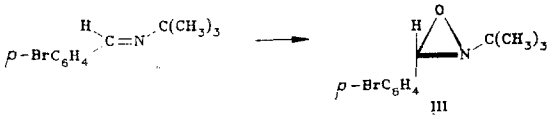


A two-step mechanism is confirmed by the formation of a mixture of the E and Z isomers of oxaziridine II from the E isomer of p-nitrobenzylidenemethylamine in its oxidation with m-chloroperbenzoic acid [31, 32].



Synthesis of substituted oxaziridines from aromatic and aliphatic-aromatic imines were described in [33-40]. The synthesis of oxaziridines by oxidation of Schiff bases with ozone [41], as well as with hydrogen peroxide [42, 43], is accompanied by side processes.

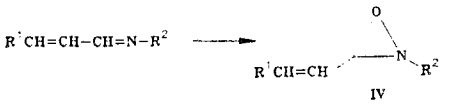
Oxaziridines are used as synthons in syntheses of macrolide antibiotics, pheromones, and other biologically active compounds [13]. In this connection chiral oxaziridines, the asymmetric synthesis of which was described in [44-53], are of considerable interest. In the oxidation of p-bromobenzylidene-tert-butylamine with a chiral oxidizing agent – monoperoxycamphoric acid – the corresponding optically active oxaziridine III was obtained with an optical purity of 60% [45].



Mixtures of diastereomeric oxaziridines with preponderance of one isomer are formed in the oxidation with m-chloroperbenzoic acid of optically active Schiff bases obtained from α-phenylethylamine and acetone, cyclopentanone, cyclohexanone, and 2-norbornenone [46-48]

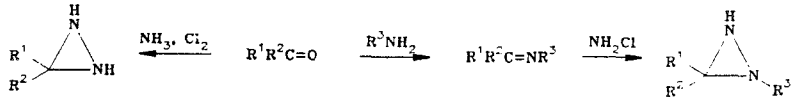
Optically active oxaziridines with a nitrogen chiral center were obtained by oxidation of benzophenone imines with optically active peracids [51], as well as by oxidation of diphenylketimines with m-chloroperbenzoic acid in the presence of chiral solvents such as R-(+)-1-cyclohexyl-2,2,2-trifluoroethanol [49]. However, this sort of partial asymmetric synthesis from achiral compounds could be carried out with an optical purity of 3-34%. This was demonstrated in the case of the synthesis of esters of N-alkyloxaziridine-3,3-dicarboxylic acids by oxidation of N-methyl(ethyl)iminomalonic esters with optically active peracids in chiral solvents [50].

3-Vinyloxaziridines were obtained by oxidation of 1-aza-1,3-dienes with m-chloroperbenzoic acid. At 20°C the olefinic bond is not involved, and 2-alkyl-3-(β-alkylvinyl)oxaziridines IV are formed in 20-87% yields [54].

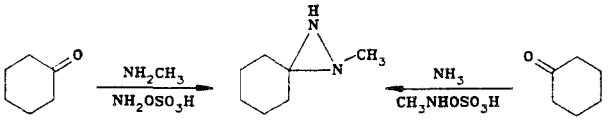


The development of the chemistry of oxaziridines is linked with azomethines – the principal starting substances for their synthesis.

Diaziridines. On reaction with chloroamine, azomethines obtained from primary aliphatic amines and ketones are converted to diaziridines [55]. A modification of this method is the direct reaction of carbonyl compounds with ammonia and chlorine or with chloroamine at reduced temperatures [55-64]. The yields of diaziridines range from 18% to 71%.

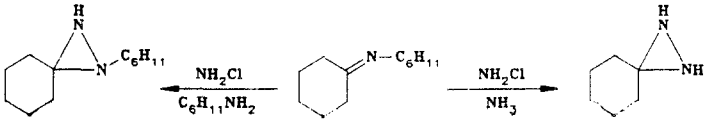


The production of diaziridines from imines and hydroxylamine O-sulfonic acid has been described [55, 57, 61, 62]. 1'-Methylspirocyclohexane [3']diaziridine (V) was obtained in greater than 60% yield in the reaction of cyclohexanone, methylamine, and hydroxylamine O-sulfonic acid. It was also obtained by condensation of cyclohexanone with ammonia, and N-methylhydroxylamine O-sulfonic acid (in 45% yield) [61].



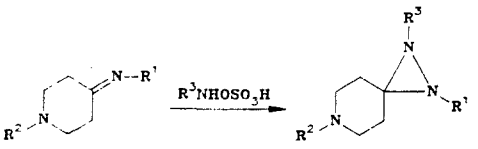
V

Spirocyclohexane [3']diaziridine (59% yield) was obtained in the condensation of cyclohexylidenecyclohexylamine with chloramine and ammonia, while 1'-cyclohexylspirocyclohexane-[3']-diaziridine (70% yield) was obtained in the condensation with chloramine and cyclohexylamine [57].

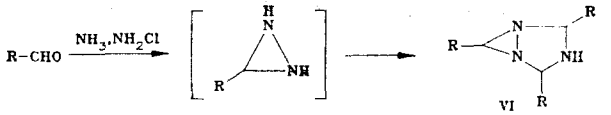


Antineoplastic activity has been observed for spiro compounds with a diaziridine fragment [65].

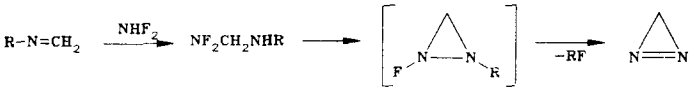
Spiro compounds with piperidine and diaziridine fragments, which were studied as analgesics and hypotensive agents, were obtained from γ-piperidone imines and N-alkylhydroxylamine O-sulfonic acids [66].



Azomethines formed in situ from aldehydes in the presence of chloramine and ammonia are not converted to diaziridines but give the condensed system of 1,2,4-triazolidine and diaziridine. In the case of butyraldehyde the corresponding diaziridine – triazolidine VI – was isolated in 80% yield [56, 62]:



Diazirine. Diazirine was obtained in the reaction of difluoro(dichloro)amine with azomethines formed in the condensation of formaldehyde with tert-butylamine, as well as tert-octylamine [67].



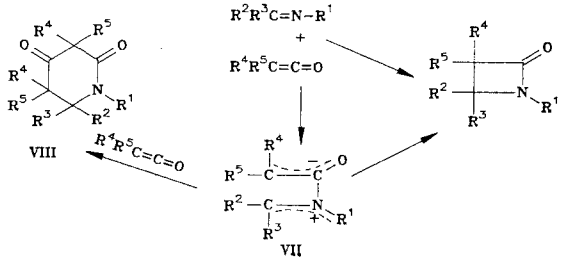
The condensation of tert-octylamine with chloramine is carried out in a formate buffer in the presence of sodium hypochlorite and aluminum chloride [68].

Four-Membered Heterocycles

Imines are convenient synthons in syntheses of azetidines and azetidin-2-ones. The intensive development of research involving the synthesis and study of azetidin-2-ones has been stimulated by investigations in the field of penicillin and cephalosporin, the molecules of which contain a β-lactam fragment. Substances that have a broad spectrum of biological activity (anitbiotics, β-lactamase inhibitors, fungicides, etc.) have been found among synthetic β-lactams [69].

1,3,3,4-Tetraphenylazetidin-2-one was obtained for the first time in 72% yield from benzylideneaniline and diphenylketene by Staudinger [70, p. 42]. Syntheses from azomethines and ketenes of numerous substituted β-lactams obtained prior to the start of the nineteen-sixties were described in [71]. This method is also widely used at the present time [72-75].

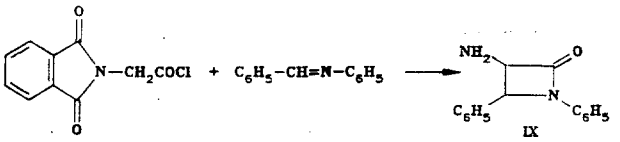
Two mechanisms for the formation of β-lactams from azomethines and ketones have been proposed: concerted [2 + 2]-cycloaddition [76] and a two-step mechanism through dipolar intermediate VII [76, 77].



The two-step mechanism is evidently more likely, since a 2:1 adduct – substituted 2,4-dioxopiperidine VIII, which is formed in the reaction of intermediate VII with the ketene – was isolated in some cases. A product of condensation of the imine with the dimer of the starting ketene was also isolated in these experiments [76, 77].

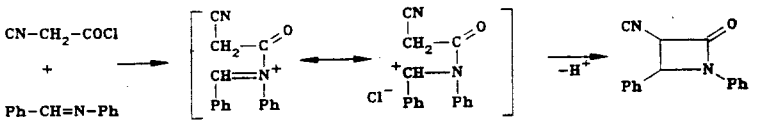
Products of [2 + 2] and [4 + 2] cycloaddition were obtained in the reaction of α,β-unsaturated imines with diphenyl- and dichloroketenes [78]. Ketenes generated in the photolysis [79] of diazo ketones or in their thermal decomposition [80] are used to obtain β-lactams. The use of monosubstituted ketenes is limited because of their facile dimerization [81].

In connection with the synthesis of penicillins and cephalosporins, methods for obtaining amino-substituted β-lactams on the basis of azomethines have been developed [71, 82-89]. 3-Amino, 1,4-diphenylazetidin-2-one (IX) was obtained from benzalaniline and phthalamidoacetyl chloride in 25% yield. The reaction is carried out in the presence of triethylamine in inert solvents at 20°C [82]:



IX

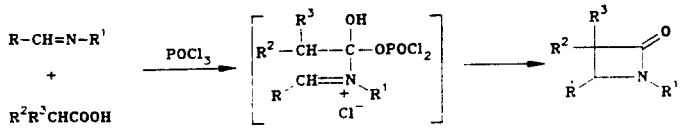
It is assumed [82] that the β-lactam is formed not through the ketene generated from the phthalamidoacetyl chloride but rather through an intermediate – the iminium salt – as demonstrated in the case of the synthesis of 3-cyano-1,4-diphenylazetidin-2-one [90]:



The occurrence of the reaction via one or another mechanism depends on the structures of the reagents, the order of their addition, and the reaction time [76, 77]. A ketene–imine reaction is more likely when the acid chloride is added to a solution of the imine and triethylamine. If, however, the triethylamine is added to a solution of the imine and acid chloride, the iminium salt undergoes dehydrohalogenation.

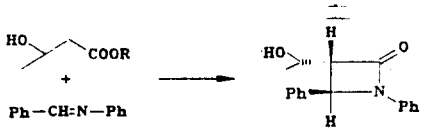
α-Azidoketenes formed in situ from α-azido acid chlorides are used to obtain 3-azido-substituted β-lactams [91-93]. The azido group is readily reduced to an amino group [94]. A large number of substituted azetidin-2-ones have been obtained from imines and acid chlorides [96-108].

A method for the synthesis of azetidin-2-ones from azomethines using carboxylic acids and phosphorus oxychloride is widely used [109-113]. It is assumed that the reaction proceeds via the following scheme [77, 96]:



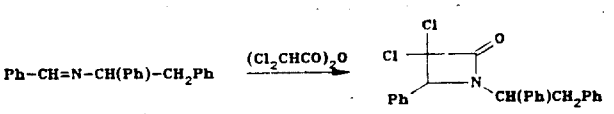
3-N-(alkylamino)azetidin-2-ones were obtained in 35-45% yields by this method from imines and α-alkylaminoacetic acid esters [114, 115]. Many substituted β-lactams were obtained from imines and derivatives of carboxylic acids [97, 98, 116, 117].

The stereoselective synthesis of 1,4-diphenyl-3-(α-hydroxyethyl)azetidin-2-one from benzalaniline and a β-hydroxybutyric acid ester (THF, –20°C in the presence of lithium diisopropylamide and hexametapol) has been realized [118].

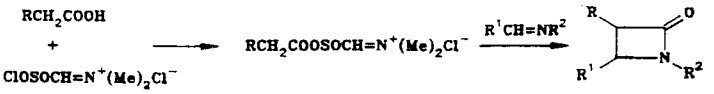


Azetidin-2-ones are formed in the reaction of imines with acyl diethyl phosphates in the presence of triethylamine [87, 119, 120].

3,3-Dichloro-β-lactams were obtained from imines containing bulky substituents attached to the carbon and nitrogen atoms (a necessary condition) and dichloroacetic acid anhydride [121, 122].

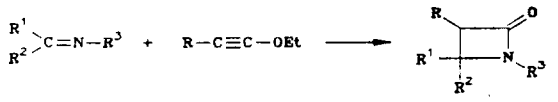


β-Lactams are obained by condensation of Schiff bases with intermediates formed from DMF, thionyl chloride, and carboxylic acids [123-126].



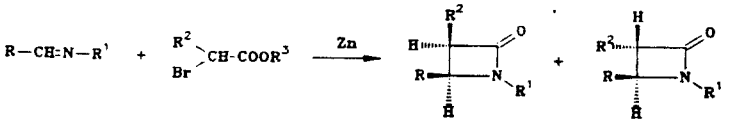
The production of β-lactams from imines and carboxylic acids in the presence of p-toluenesulfonyl chloride in methylene chloride at 25°C [127] and under the influence of triphenylphosphine in CBr_4 at −78°C [128] has been described.

At elevated temperatures benzylideneaniline reacts with ethoxyacetylene to give β-lactams. The reaction is accompanied by splitting out of ethylene [129]:



This method and the synthesis of β-lactams by the reaction of imines with diazo ketones have not undergone further development.

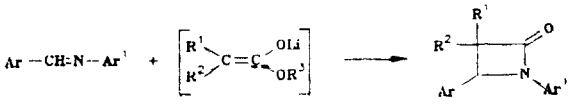
The reaction of imines with esters of α-bromo carboxylic acids under the conditions of the Reformatskii reaction is a general method for the synthesis of β-lactams [130-136]. The cis and trans isomers of 3,4-disubstituted azetidin-2-ones were obtained for the first time by this method [137].



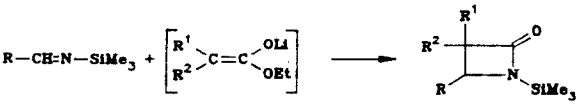
The stereoselectivity of the reaction depends on the structures of the starting compounds and the nature of the solvent [138-141].

The use of ultrasound in carrying out the reaction of p-methoxybenzylidene-p-anisidine with ethyl bromoacetate and zinc made it possible to increase the yield of 1-(p-anisyl)-3-(p-anisyl)azetidin-2-one to 95% [142, 143].

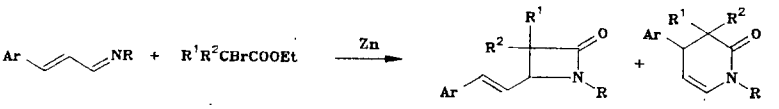
Carboxylic acid esters enolized in the presence of lithium diisopropylamide have been used in place of bromo-substituted esters [144].



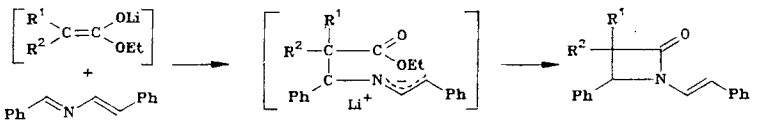
N-Trimethylsilyl-substituted β-lactams were obtained in 53-74% yields under similar conditions from N-trimethylsilylimines [145, 146]. Since the N-trimethylsilyl group is easily removed, this method for the synthesis of NH-azetidin-2-one is often used in syntheses of β-lactam antibiotics.



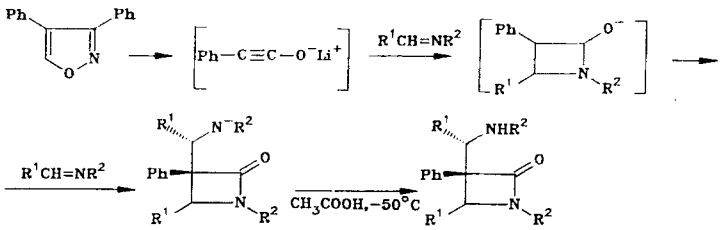
Adducts of both 1,2- and 1,4-cycloaddition are obtained under the conditions of the Reformatskii reaction from α-bromo-substituted acid esters and α,β-unsaturated imines [147-149].



β-Lactams are formed in quantitative yields in the reaction of 2-aza-1,3-butadienes with lithium enolates of carboxylic acid esters; the following scheme of this reaction is proposed [150]:



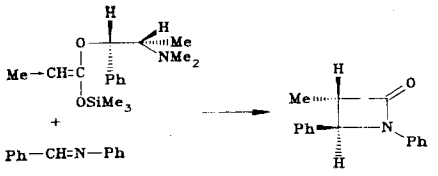
The reaction of imines with lithium alkoxides of phenylhydroxyacetylene, formed by cleavage of 3,4-diphenyloxazole with butyllithium in THF at −78°C, has been described. In the first step the enolate of azetidin-2-one, to which a second molecule of the imine adds, is formed as a result of anionic cyclization [151].



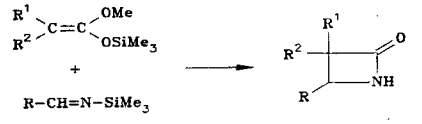
The formation of 4-aryl-3-phenyl-3-α-arylaminomethylazetidin-2-ones proceeds with high selectivity: the cis isomer of 4-(p-nitrophenyl)-3-phenyl-3-[α-(p-nitrophenyl)-α-(p-nitrophenylamino)methyl]azetidin-2-one was obtained in 89% yield [151].

β-Lactams are often synthesized by condensation of imines with ketenesilylacetals (20°C, dichloroethane, $TiCl_4$). Esters of β-amino acids are formed as side products [152-155].

This reaction was used to obtain optically active derivatives of azetidin-2-ones; both optically active azomethines [156, 157] and ketenes of silylated esters such as ephedrine derivatives [158, 159] were used.



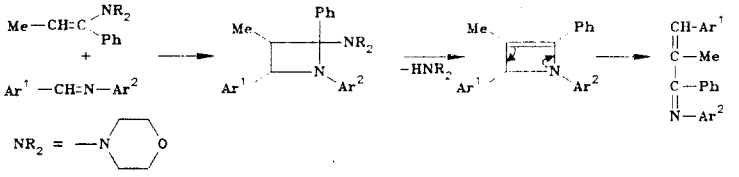
NH-Azetidin-2-ones are obtained from N-trimethylsilylimines.



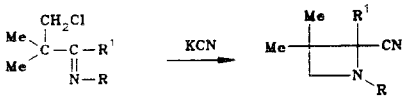
In carrying out the one-step synthesis of NH-β-lactams a mixture of the complex of the imine with zinc iodide, the silylketeneacetal, and tert-butyl alcohol is maintained at 20°C for 2-3 h, after which three equivalents of methylmagnesium bromide are added; the yields range from 27% to 82% [160].

The reaction of imines with enamines with the formation of substituted 2-aminoazetidines proceeds via a [2 + 2]-cycloaddition mechanism [161-164]. Azetidines of this sort are obtained in quantitative yields by carrying out the reaction at high pressures ($12 \cdot 10^5$ kPa) [162].

In acetic acid at 25°C the reaction is complicated by rapid deamination of the resulting azetidines. Subsequent opening of the ring of the resulting azetidin-2-enones leads to substituted 1-aza-1,3-butadienes [163].



Substituted 2-cyanoazetidines were obtained from β-chloro-substituted imines and KCN in 76-96% yields by refluxing in methanol [165].



2-Methoxyazetidine X was obtained by similar intramolecular cyclization [76].

The production of 2-aryl-N-sulfonylazetidin-2-ones from aryl-N-arylsulfonylimines and dimethylsulfoxonium methylid (the Corey reagent) was described in [166, 167].

The reaction of imines with pentacarbonylmethoxyalkyl(aryl)carbenechromium complexes proceeds regio- and stereo-specifically [168-170].

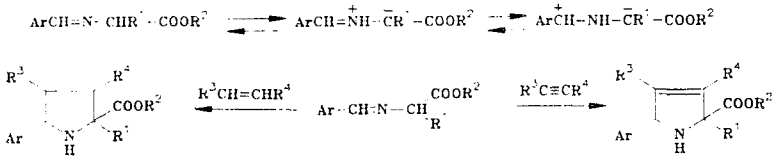
Azetidin-2-ones were obtained in the reaction of imines with trimethylsilyl tribromoacetate [171], substituted formamides [172], cyclohexyl benzoylformate [173], and ketene imines [174].

The reaction of imines with thioketenes leads to azetidine-2-thiones [175], whereas the reaction with acyl isothiocyanates leads to 1,3-thiaazetidines [176].

Five-Membered Heterocycles

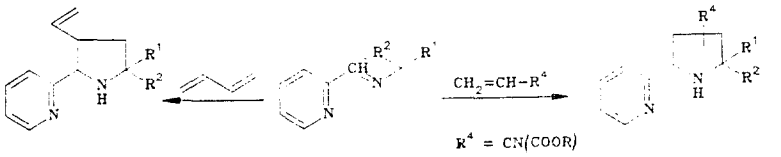
Functionally substituted Schiff bases are potential 1,3-dipoles [177, 178] that react with alkenes and alkynes to give [3 + 2]-cycloaddition products – substituted pyrrolidines

and pyrrolenines [179]. When the reaction was carried out under interphase-catalysis conditions [trimethylbenzylammonium (TEBA) ion, K_2CO_3, CH_3OH, 20°C), pyrrolidines were obtained in 80-90% yields [180].



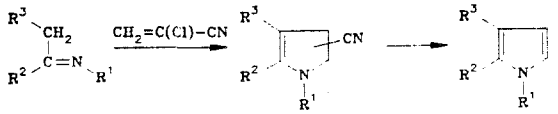
The condensation of N-benzylaldimines and N-benzylketimines with olefins in the presence of sodium also leads to substituted pyrrolidines [181-183].

α-Nornicotines with, respectively, a vinyl or cyano (alkoxycarbonyl) group in the pyrrolidine ring were synthesized in the condensation of ketimines obtained from 2-formylpyridine in the presence of triphenylphosphine and nickel with butadiene, as well as with acrylonitrile (acrylates) [184, 185].

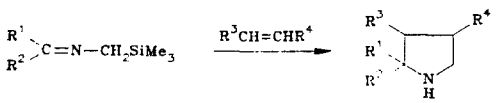


N-Phenyl-2-(2-pyridyl)pyrrolidine was obtained in the electrolysis of a solution of α-formylpyridine in 1,3-dibromopropane in the presence of tetrabutylammonium iodide [186].

The condensation of azomethines with 2-chloroacrylonitrile (triethylamine, benzene, 20°C) has been described. The resulting cyano-substituted pyrrolines split out HCN on heating and are converted to substituted pyrroles. It is assumed that the reaction proceeds via a condensation of the Michael type with the participation of the enamine form of the azomethine [187]. Satisfactory yields (50-60%) were obtained in the case of ketimines of cyclic ketones but not in the case of aldimines.

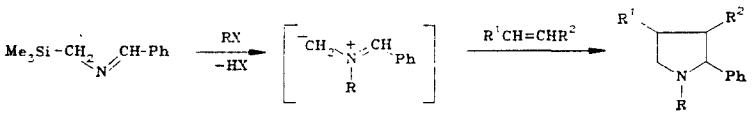


Imines obtained on the basis of trimethylsilylmethylamine have recently been used as potential 2-azaallyl anions; lithium fluoride was used for their generation [188]. The production of substituted pyrrolidines via this method has been described [189-193].



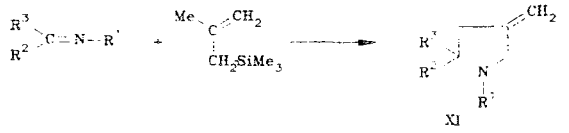
The condensation of silyl-containing benzylideneamines with olefins and acetylenes in the presence of acyl chlorides leads to N-acyl-substituted pyrrolidines and pyrrolenines [194].

A modification of the method of synthesis of pyrrolidines from alkenes and N-benzylidenetrimethylsilylmethylamine is conversion of the latter to an ylid (by heating with alkyl halides in hexametapol) [195].

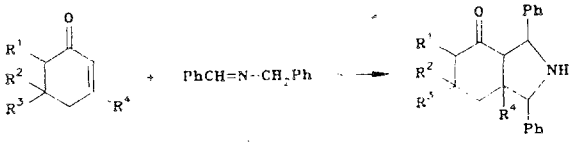


The condensation of azomethines with methylallyltrimethylsilane in the presence of nickel and triethyl phosphite with the formation of pyrrolidine XI has been described [196].

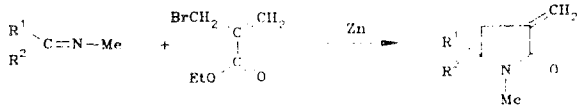
In this case an allyl anion is generated from the trimethylsilyl-substituted olefin.



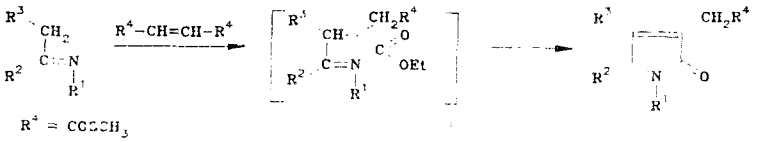
Perhydrooxoisoindoles were obtained in 41-64% yields by condensation of N-benzylidenebenzylamine with alkyl-substituted 2-cyclohexenones under interphase-catalysis conditions (NaOH, H_2O, DMSO, 20°C, 24 h) [197].



1-Methyl-5,5-dialkyl-3-methylene-2-oxopyrrolidines were obtained from ketimines, ethyl α-bromoacrylate, and zinc in THF (the Reformatskii reaction) [198, 199].

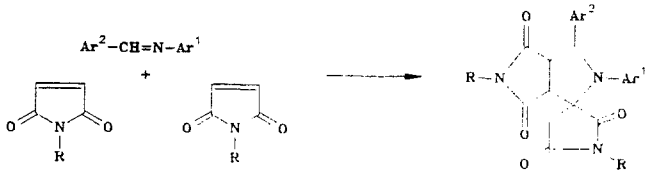


Substituted pyrroleninones [200] rather than tetrahydro-2-pyridinones [201] are formed in the reaction of azomethines with dimethyl maleate in the presence of aluminum chloride. It is assumed that a product of Michael condensation is the intermediate in this reaction

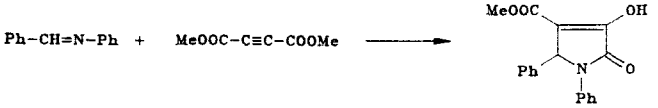


Similarly, N-substituted 2-oxacyclohexane[b]pyrrolidin-3-enes were obtained from N-benzyl(cylohexyl, isopropyl, methyl)-cyclohexylideneamines [202].

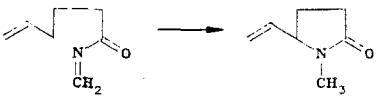
An interesting example of the synthesis of spiro heterocycles is the reaction of Schiff bases with N-substituted maleinimides — the so-called dipolar spiro bisaddition [203-205]. 4,1'-Dialkyl-1,6-diaryl-3,5,2',5'-tetraoxo-1,4-diazaspirobicyclo[3.3.0]octane[2,3']pyrrolidines (in 74-86% yields) were obtained from N-arylidenearylamines and N-alkylmaleinimides.



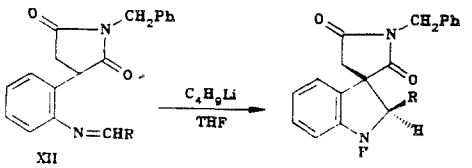
1,5-Diphenyl-3-hydroxy-4-methoxycarbonyl-2-oxopyrrolidin-3-ene was obtained in the condensation of N-benzylideneaniline with dimethyl acetylenedicarboxylate in an aqueous medium in 26% yield [206].



β-Allylpropionic acid N-methyleneamide undergoes intramolecular cyclization to give 1-methyl-5-vinyl-2-pyrrolidinone when it is heated [207].

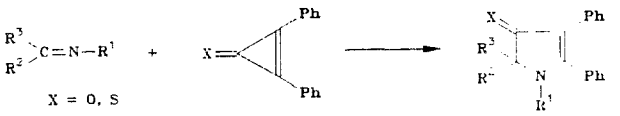


The synthesis of optically active indoles as a result of intramolecular addition to the azomethine bond of XII has been described [208-210].

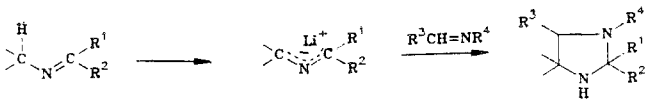


XII

Substituted 4-oxo(thio)pyrrolidin-2-enes were obtained in 70-90% yields from 1,2-diphenyl-3-oxo(thio)cyclopropene and azomethines [211, 212].

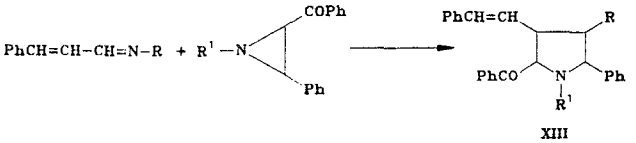


[3 + 2]-Cycloadducts – substituted imidazolidines – were obtained in 50% yield in the reaction of Schiff bases with 2-azaallyl anions formed by deprotonation of N-alkylated Schiff bases (lithium diisopropylamide, THF, –70°C) [213, 214]. 1,3-"Anionophilic" cycloaddition reactions of 2-azaallyl anions with various types of C=X (X = C, O, N, S) and C≡X bonds (X = C, N) were described in [213, 215].



1,3-Diaroylimidazolidines were obtained by treatment of bisalkylidene(arylidene)ethylenediamines with organic acid chlorides in polar solvents [216].

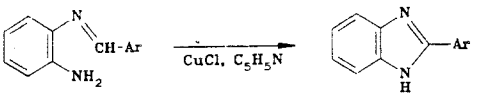
The condensation of 1-aza-1,3-dienes with substituted aziridines, as, for example, in the production of imidazolidine XIII (in up to 60% yield), has been described [217]:



XIII

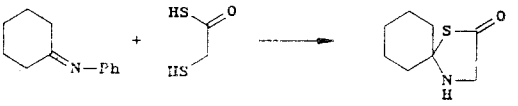
The reaction of imines with ethyl diazoacetate also leads to the formation of substituted imidazolidines [218].

The Schiff bases obtained from o-phenylenediamine and aromatic aldehydes are converted to 2-aryl-substituted benzimidazoles by catalytic dehydrocyclization [219]. This transformation is also realized by the action of lead tetraacetate [220].

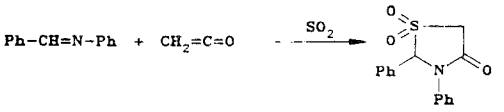


8-Phenyltheophylline was obtained in quantitative yield from 5-benzylideneamino-6-amino-1,3-dimethyluracil by heating in DMSO in the presence of mercuric chloride [221].

The production of thiazolidines from azomethines and sulfhydryl derivatives [100, 222-232] is widely used in the synthesis of biologically active compounds such as fungicides [225]. The synthesis of spiro compounds with thiazolidine and cyclohexane fragments is a development of these studies [233].

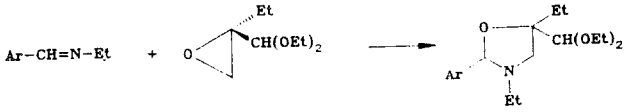


1,2-Diphenyl-5-thiazolidinone S,S-dioxide was obtained in the reaction of benzylideneaniline and ketene in liquid sulfur dioxide in 52% yield [234].

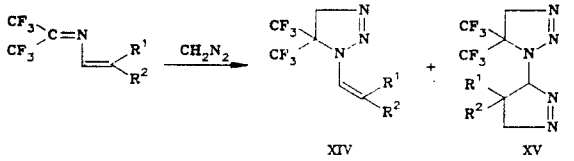


Anils that contain a hydroxy group in the ortho position are oxidized by lead tetraacetate to 2-substituted oxazoles [220].

Oxazolidines are formed in the reaction of azomethines with oxiranes in the presence of Lewis acids [235]. 2-Aryl-3,5-diethyl-5-diethoxymethyloxazolidines were obtained in 30-70% yields in the form of a mixture of geometrical isomers from 2-ethyl-2-formyloxirane acetal and N-arylideneethylamine by heating in ethanol [236].

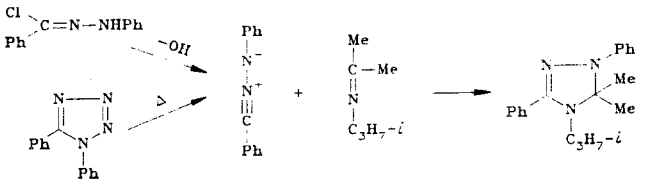


The synthesis of Δ^2-1,2,3-triazolines from azomethines and diazoalkanes has been known for a long time [237-241]. Data [242] that indicate that the reaction of Schiff bases with diazo compounds proceeds via a concerted mechanism involving 1,3-dipolar cycloaddition through a transition state in which maximum stabilization is achieved through overlapping of the highest occupied molecular orbital (HOMO) in the dipole with the lowest vacant molecular orbital (LVMO) in the dipolarophile have been presented. An example of synthesis by this method was described in [243]; the ratio of products XIV and XV depends on the nature of the substituent attached to the $C_{(4)}$ atom in the imine.



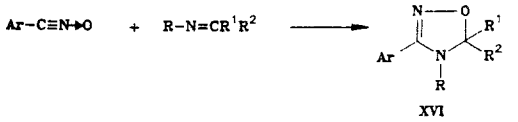
XIV XV

A general method for the synthesis of Δ^2-1,2,4-triazolines is 1,3-dipolar cycloaddition of azomethines to nitrile imines. The latter are generated in situ by the action of bases on hydrazonoyl halides or by thermolysis of 1,5-disubstituted tetrazoles [244, 245].



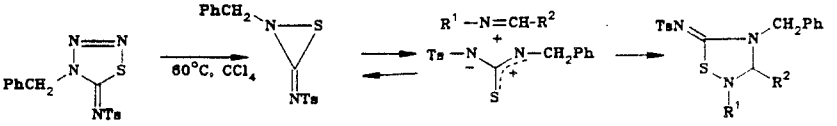
A number of substituted Δ^2-1,2,4-triazolines were obtained in 80-90% yields by this method.

Azomethines react readily with nitrile oxides to give Δ^2-1,2,4-oxadiazolines XVI in high yields [242, 246]



XVI

The synthesis of 1,2,4-thiazolidines by the [3 + 2]-cycloaddition of azomethines to thiaziridine imines formed in situ by pyrolysis of N-tosyliminothiatriazole has been described [247].

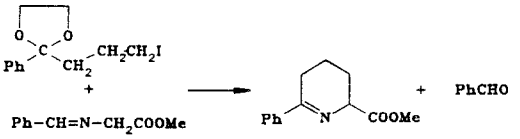


The cycloaddition of p-methoxybenzonitrile sulfide to imines also leads to 1,2,4-thiadiazolidine derivatives [248, 249]

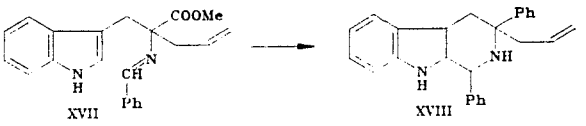
Six-Membered Heterocycles

2,5-Divinylpiperidines have been obtained by condensation of aliphatic imines with divinyl in the presence of a palladium catalyst in 73% yield [250].

An example of the synthesis of piperidin-1-enes is the condensation of ω-iodobutyrophenone with methyl N-benzylideneaminoacetate. The oxalane protection of the starting ketone prevents the formation of a cyclopropane [251].

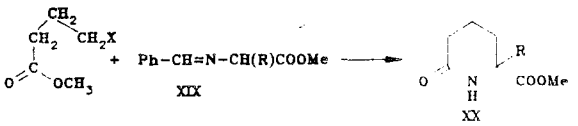


Under acid-catalysis conditions the azomethine based on tryptamine undergoes intramolecular cyclization to tetrahydro-β-carboline [252]. The cyclization of azomethine XVII, obtained from L-tryptophan, takes place similarly. Synthesized tetrahydro-β-carbolines XVIII have been used in syntheses of alkaloids [253].

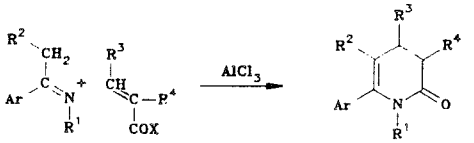


XVII XVIII

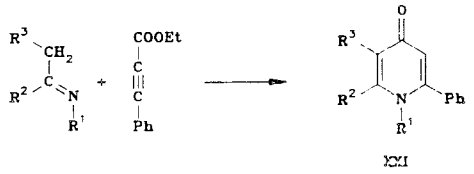
Similar to the condensation presented above, substituted 2-piperidones XX were synthesized from the methyl ester of a γ-halo-substituted butyric acid and azomethine XIX [254].



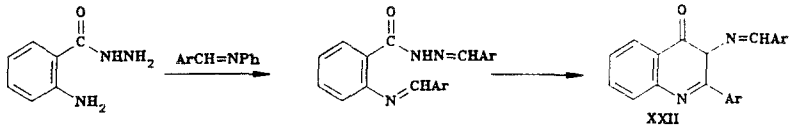
The reaction of imines with acrylamide (methylacrylamide), which is called aza-annelation, leads to 2-oxo-1,2,3,4-tetrahydropyridines. Similar compounds are obtained by condensation of azomethines with esters of α,β-unsaturated acids in 25-90% yields [255-262].



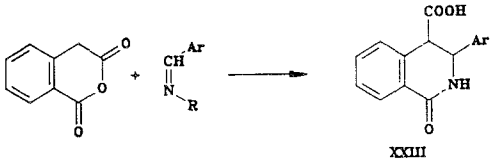
The reaction of ketimines with phenylpropiolic acid leads to 4-oxodihydropyridines XXI [261], which are herbicides and antiphlogistic agents [263-265].



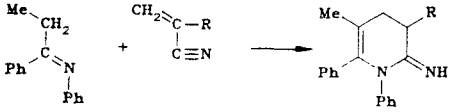
3-Arylideneamino-4(3H)-quinazolinones XXII were obtained via the scheme presented above using anils of aromatic aldehydes [266].



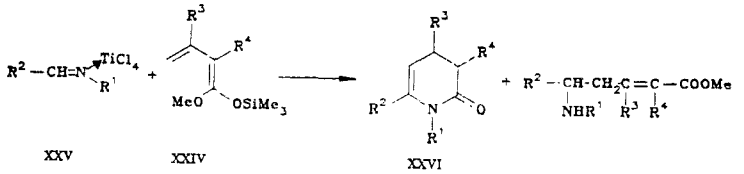
Substituted 3,4-dihydro-1-isoquinolone XXIII is formed in the reaction of N-arylideneamines with homophthalic anhydride [4, 267]. The mechanism of the reaction has been studied [268].



Unsaturated cyclic amidines are obtained from ketimines and acrylonitrile (methylacrylonitrile) in the presence of Lewis acids ($SnCl_4$, $FeCl_3$, $ZnCl_2$) [269].

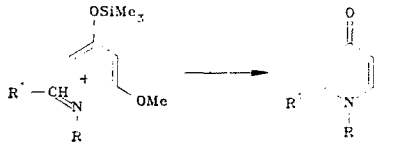


Piperidein-2-ones XXVI were obtained by the reaction of vinyl ketene acetals XXIV with azomethine–titanium tetrachloride complexes XXV in 20-86% yields. Esters of ω-alkylamino α,β-unsaturated acids are formed simultaneously [270].



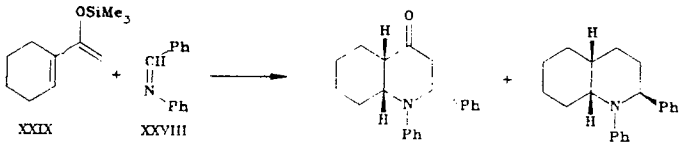
1-Benzyl-4-methyl-6-phenyl-2-oxopiperidin-3-ene was obtained from N-benzylidenebenzylamine and 1-methoxy-1-trimethylsilyloxy-3-methyl-1,3-butadiene (one of the "Danischefsky dienes") in the presence of $TiCl_4$ in 92% yield [271].

Substituted piperidein-4-ones XXVII were obtained via the scheme in [271-273].



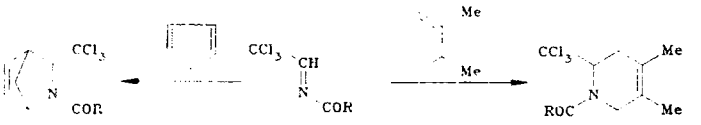
XXVII

A mixture of diastereomers of cis-fused 1,2-diphenyl-4-oxodecahydroquinoline is formed in the reaction of benzaldehyde anil (XXVIII) with 1-(α-trimethylsilyloxy)vinylcyclohexene (XXIX) in 72% yield; the ratio of the trans and cis isomers is 7:3 [274]



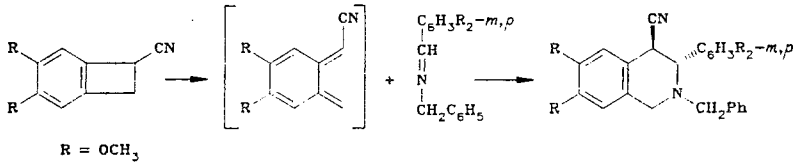
XXIX XXVIII

A number of piperideines were obtained in 80-90% yields by reactions involving the diene synthesis [275-279]. Activation of azomethines as dienophiles is achieved by the introduction of strong electron-acceptor substituents [275].



The same condensation of N-(dicyanomethylidene)tosylamine with 2,3-dialkylbutadiene and cyclopentadiene has been described [280].

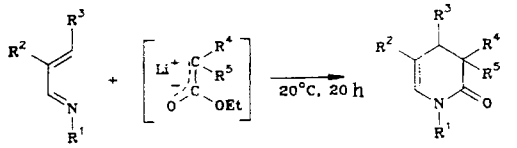
The cycloaddition of azomethines to o-quinone methides generated in situ by opening the cyclobutane fragment is realized regio- and stereospecifically [281, 282].



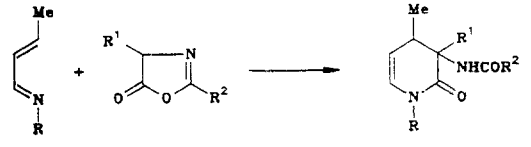
R = OCH_3

The stereochemistry of the addition of aldimines to conjugated dienes was examined in [283-286].

The azadiene synthesis is discussed in detail in previous reviews [5-10]. Substituted piperidein-2-ones are obtained in 24-78% yields by reactions of azadienes with carbanions formed from carboxylic acid esters by treatment with lithium diisopropylamide [287].



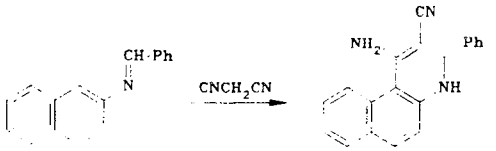
Similar compounds XXX were synthesized via the scheme in [288].



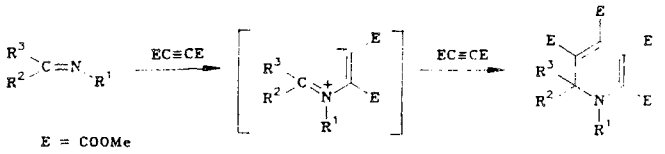
XXX

In the presence of Lewis acids anils of aromatic and aliphatic aldehydes react with olefins (cyclic olefins) to give tetrahydroquinolines [289-293].

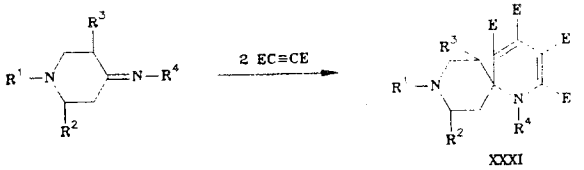
Malononitrile adds smoothly to an azomethine with subsequent cyclization [294].



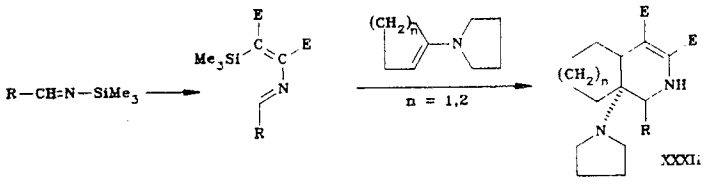
The 1,4-dipolar cycloaddition of dimethyl acetylene-dicarboxylate to azomethines is used to obtain dihydropyridines [295-299].



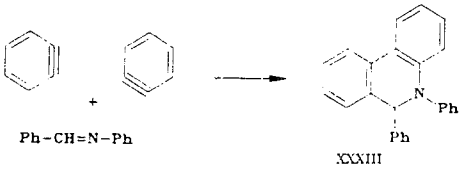
The condensation of N-(4-piperidylidene)arylamines with dimethyl acetylenedicarboxylate leads to spiro compounds XXXI [300-304].



The condensation of 2-aza-1,3-butadienes, formed in the reaction of N-trimethylsilyl-imines with dimethyl acetylenedicarboxylate, with enamines of cyclic ketones has been described. The yields of condensed piperideines XXXII range from 70% to 85% [305].

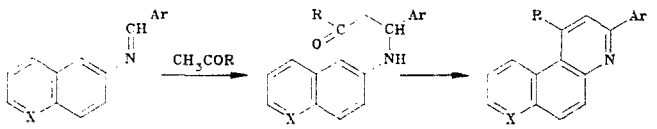


5,6-Diphenyl-5,6-dihydrophenanthridine (XXXIII) is formed from benzalaniline and dihydrobenzene [306]. The reaction is accompanied by the formation of a complex mixture of substances [307], from which N-(o-anilinobenzhydryl)aniline (16%) and 5,10-diphenyl-5,10-dihydroacridine (5%) were isolated.



In the presence of oxide catalysts (aluminum potassium oxide, aluminum chromium potassium oxide, and chromium oxide) at 350°C N-alkylideneallylamines undergo heteroaromatization to give α(β)-alkyl- (up to 5%) and α,β-dialkylpyridines (up to 60%) [308-313]. Similarly, substituted quinolines were obtained from N-arylideneallylamines. In the case of N-(m,p-dimethoxybenzylidene)allylamine the yield of 4-methyl-6,7-dimethoxyisoquinoline is 37% [313].

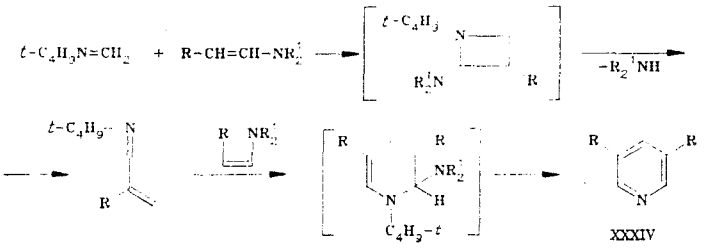
N-Arylidene-2-naphthylamines were used in syntheses of benzo[f]quinolines [314-318], while N-arylidene-6-quinolylamines were used in syntheses of 4,7-phenanthrolines [319-324]. The method of construction of the pyridine ring consists in condensation of imines with ketones and subsequent cyclodehydration of the β-amino ketones under acid-catalysis conditions.



X = CH_2, N

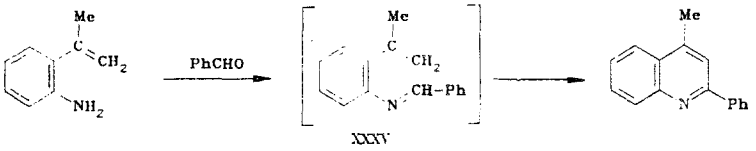
The cyclization of N-(1,2,5-trimethyl-4-piperidylidene)-α-aminonaphthalene on the K-16 catalyst at 410-420°C leads to the formation of 9-methylnaphtho[1,2-b][1,6]naphthyridine and 2,3-dimethyl-4-azaphenanthrene [325].

Methylidene-N-tert-butylamine reacts with enamines to give β,β'-substituted pyridines XXXIV in 67-87% yields [326, 327].



XXXIV

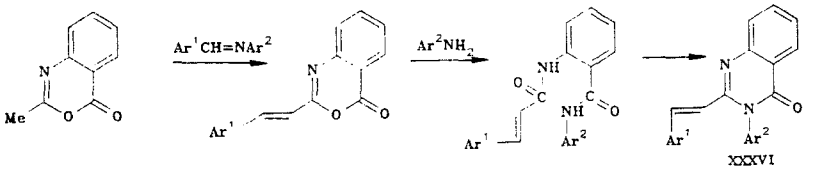
2-Phenyl-3-methylquinoline is formed when equimolar amounts of benzaldehyde and o-isopropenylaniline are refluxed. The reaction probably proceeds via dehydrocyclization of intermediate imine XXXV [328].



XXXV

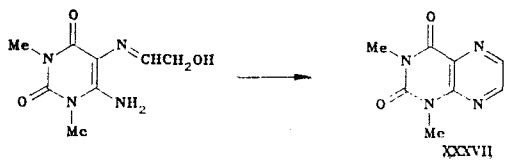
The photochemical transformations of imines to give pyridine bases were examined in [329, 330].

Syntheses of diheteroazines and their partially hydrogenated analogs from azomethines have been described. 3,4-Dihydro-4-oxobenzopyrimidines XXXVI were obtained in the reaction of 2-methyl-4-oxo-3,1,4-benzoxazine with N-arylideneamines in the presence of sodium acetate in acetic acid [331-333].



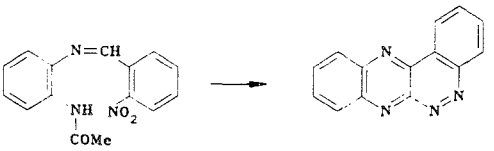
XXXVI

Hexahydropteridine system XXXVII is formed in quantitative yield from 6-amino-5-(β-hydroxyethylideneamino)-1,3-dimethyluracil by the action of mercuric chloride in DMSO [221].

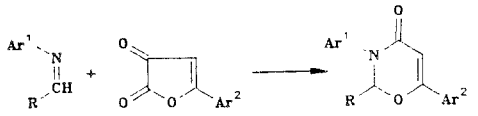


XXXVII

Quinoxalino[2,3-c]cinnoline (52% yield) was obtained by refluxing o-acetylamino-N-(o-nitrobenzylidene)aniline with KCN in methanol [334, 335].

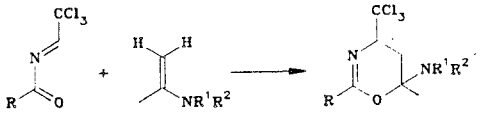


Substituted 2,3-dihydro-4-oxooxazines are formed when equimolar amounts of N-arylidene(alkylidene)arylamines are refluxed with 5-aryl-2,3-dihydrofuran-2,3-diones in benzene [336]. 3-Aryl(alkyl)-2,6-diaryl-2,3-dihydro-4H-1,3-oxazin-4-ones are obtained in 44-88% yields.

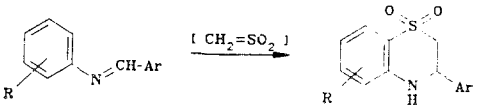


Dihydrooxazinones were similarly obtained by condensation of azomethines with 2,2,6-trimethyl-1,4-dioxan-4-ones [337].

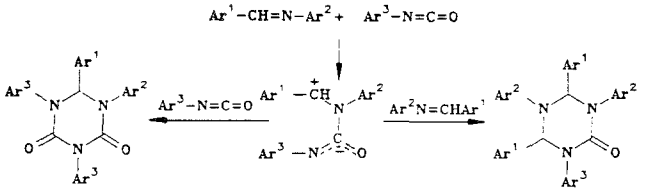
The [4 + 2]-cycloaddition of N-acylimines to enamines is a new method for the synthesis of amino-substituted 5,6-dihydro-4H-1,3-oxazines [338].



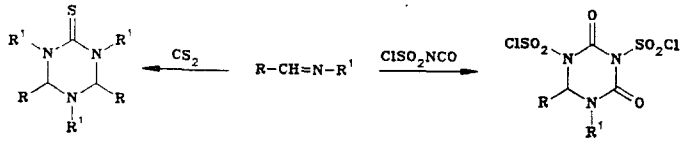
Substituted benzothiazines are formed in 70-80% yields in the condensation of methylenesulfene – the intermediate formed from methanesulfonyl chloride by the action of triethylamine – with N-arylidenearylamines [339-341].



Symmetrical perhydrotriazinones are obtained by condensation of azomethines with isocyanates or isothiocyanates. The direction of the reaction is determined by the stability of the zwitter-ion intermediate, as well as by kinetic and thermodynamic control. Electron-acceptor substituents in the para position of the arylidene fragment of the Schiff base and a relatively high temperature promote dipolar [4 + 2]-cycloaddition; the intermediate undergoes condensation with either the azomethine or the isocyanate to give, respectively, 2:1 or 1:2 adducts [342, 343].



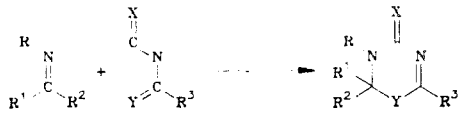
Condensation of Schiff bases with chlorosulfonyl isocyanate, as well as with carbon disulfide, has been described [344, 345].



1,6-Disubstituted sym-perhydrotriazine-2,4-diones were obtained in the hydrolysis of bis(chlorosulfonyl)-substituted triazines [346, p. 138].

Arylidene-N-methylamines react with acyl isocyanates [347] via the scheme

while other Schiff bases react with acyl and thioacyl isocyanates via a 1,4-cycloaddition mechanism [345, 347, 348].



X, Y = O, S

The data examined in this review provide evidence for the high synthetic potential of Schiff bases in the chemistry of heterocyclic compounds; this potential is far from having been exhausted.

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