SYNTHESIS OF NITROGEN HETEROCYCLES BY (4 + 2)π-CYCLOADDITION FROM NITROGEN-CONTAINING HETERODIENES ("AZADIENE SYNTHESIS") (REVIEW)

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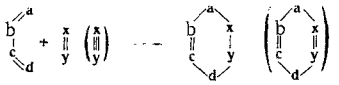
UDC 547.83.07+547.521

The review is devoted to the (4+2)π-cycloaddition with conjugated nitrogen-containing dienes. The participation in the synthesis of 1-azadienes, 2-azadienes (Schiff bases, oxazoles, and the acridizinium ion), 1,4-diazadienes (dehydroindigo, etc.), 2,3-diazadienes (tetrazines, 4H-pyrazoles, and triazines), α,β-unsaturated azo compounds, 5,6-dihydrotriazines, formazans, and azadienes containing more than one heteroatom in the conjugation chain is examined. The azadiene synthesis is a method for the preparation of pyridines, quinolines, pyridazines, oxazines, oxadiazines with different degrees of saturation, and several other heterocycles.

(4+2)π-Cycloaddition, where the components contain at least one nitrogen atom, is a universal method for the synthesis of six-membered nitrogen heterocycles.



Variants of the reactions with the application of heterodienophiles (with C=N, N=N, N=O, N=S and other bonds) have been discussed in detail [1-4] and are therefore not considered in this review. The subject of this review is the Diels–Alder reaction with the participation of azadiene systems. 1,4-Cycloaddition for heterocumulenes (a=b=c and a=b=c–d=e) is not included here, since, first, this problem has already been examined [5], and, second, heterocumulenes react as 1,2- or 1,4-dipoles [6], and the reaction with them is consequently not (4+2)π-cycloaddition.*

In spite of the previously formulated opinion regarding the low reactivity of 1,3-azadienes in 1,4-cycloaddition [10], more and more attention is currently being extended to both the theoretical and practical aspects of the azadiene synthesis. In discussing the problem, one should take into account a number of general assumptions that are characteristic for the Diels–Alder reaction as a whole.

First, it is necessary to consider the compliance of the synthesis with specific steric principles (conformational requirements for the reaction components, steric factors, etc.) [11].

Second, in accordance with the Woodward–Hoffman rule [12], this reaction is a thermally allowed (4s+2s) process, as a consequence of which it is highly stereospecific (cis principle and endo rule) [11].

Third, the azadiene components should have low 1,4-localization energies ($E_{1,4}$) [13], and it should therefore be expected that heterocyclic systems with strongly expressed aromatic character are passive in the azadiene synthesis.

*See also [7-9] for individual interesting examples of reactions with heterocumulenes.

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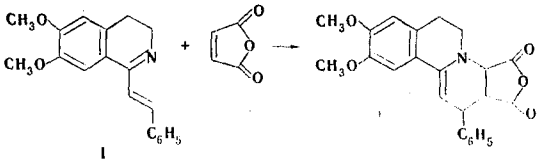
Fourth, the introduction of nitrogen atoms into the conjugated system should lead to an increase in the role of polar factors for the unsymmetrical azadiene components and, in extreme cases, to a polar two-step mechanism. The character of the azadiene component may change appreciably under the influence of acidic agents, and the appearance of acid catalysis is therefore possible during the reaction.

Fifth, donor-acceptor relationships for the diene and dienophile are essential. The introduction of nitrogen atoms into a conjugated system should lower its nucleophilicity and consequently suppress the reactivity with respect to the usual electron-acceptor dienophiles (tetracyanoethylene, maleic anhydride, etc.). However, two different variants should be distinguished. In terminal azadiene components (1-aza and 1,4-diaza-1,3-dienes), at least one of the ends of the conjugated chain retains nucleophilic character ($\overset{\delta+}{C}=\overset{\delta-}{N}$) when their nucleophilicity is lowered as a whole. Consequently, one should expect low activity in 1,4-cycloaddition reactions for such systems. On the other hand, the nucleophilicity of both the entire system and its ends is reduced for 2-aza- and 2,3-diaza-1,3-dienes. The manifestation of reactivity in condensations with electron-donor dienophiles (inversion of the diene synthesis) is more likely for these azadiene components.

Terminal Azadienes

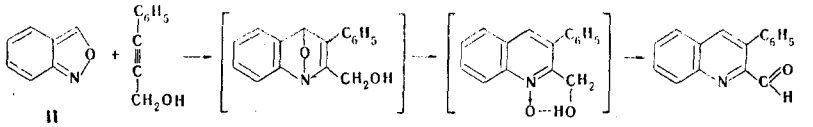
1-Aza-1,3-dienes. 1,4-Cycloaddition reactions are unknown for noncyclic derivatives, i.e., α,β-unsaturated imines and Schiff bases. The products of the addition of them to maleic anhydride are apparently molecular complexes [14].

1-Styryl-6,7-dimethoxy-3,4-dihydroisoquinoline (I) reacts with maleic anhydride [15] in the same way as 1-(1-cyclohexenyl)-6,7-dimethoxy-3,4-dihydroisoquinoline [16].



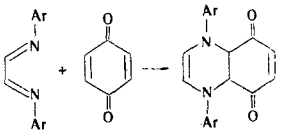
I

Isoxazoles and pyrazoles [17] are inactive in the diene synthesis because of their strongly expressed aromatic properties (high $E_{1,4}$ value). However, anthranil (II) gives 1,4-cycloaddition products with maleic anhydride [18], maleinimide [19], and some acetylenes [20], and the latter reaction is a method for the synthesis of 2,3-disubstituted quinolines.



II

1,4-Diaza-1,3-dienes. Diimines and dioximes of α-dicarbonyl compounds do not react via the scheme of the Diels–Alder reaction, with the exception of the bis(4-dimethylaminoanil) of glyoxal (III) in the reaction with p-benzoquinone [21].

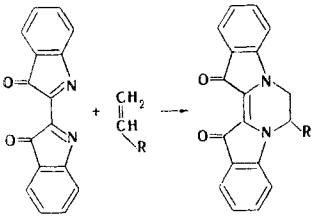


III

Ar = p-$(CH_3)_2NC_6H_4$

The electron-donor effect of the dimethylamino group, which increases the nucleophilicity of the 1 and 4 positions of the conjugated chain, is probably exerted here.

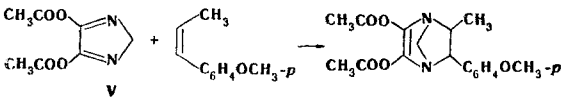
The opposite effect is observed when electron-acceptor substituents are introduced into the 2 and 3 positions of the diene component. Because of this, dehydroindigo (IV) reacts with the dienophiles with nucleophilic character – styrene, safrole, isosafrole, and isoeugenol methyl ester [22].



IV

Products of the 1,4-cycloaddition of IV to methyl acrylate [23], acrylonitrile [22], and methyl propiolate [24] are formed in low yields under severe conditions.

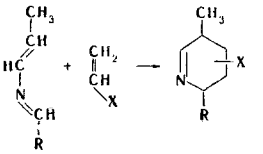
Dimethyl imidazole-4,5-dicarboxylate in the tautomeric form (V) reacts with anethole [25].



V

Azadienes with Nonterminal Nitrogen Atoms

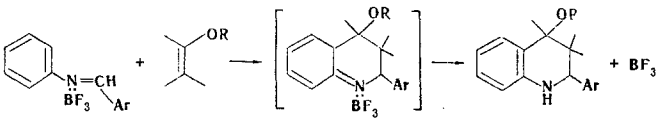
2-Aza-1,3-dienes. The recently synthesized [26] aliphatic derivatives with a system of C=C−N=C bonds have proved to be extremely reactive with respect to both electrophilic (maleic anhydride and acrolein) and nucleophilic (ethyl vinyl ether) dienophiles. The reactions lead to Δ^1-tetrahydropyridines [27].



Another variant of this condensation, which is carried out over K_2O/Al_2O_3 at 400-450°C, has proved to be a promising method for the synthesis of pyridines, which are obtained as a result of aromatization of their tetrahydro derivatives [28, 29].

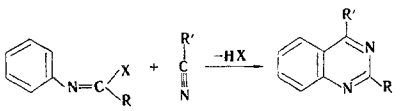
Schiff bases are also active 2-azadiene components in reactions with nucleophilic dienophiles [30, 31]. The peculiarities of this condensation and its mechanism were discussed in an earlier review [32].

The reaction is characteristic for anils of both aromatic and aliphatic aldehydes in the presence of Lewis acids (BF_3 and $AlCl_3$) with respect to vinyl ethers and thioethers of various structures, as well as acetylenic dienophiles [33]. A catalyst is necessary in this case, and its role is to polarize the C=N bond, which leads to the strongly electrophilic character of the diene component, as well as to create the favorable planar configuration of the diene system [32].



According to the data in [34], iron and cobalt carbonyls can also be used as catalysts. The stereochemical peculiarities of the synthesis were studied in [35, 36]. This reaction, which is a convenient method for the synthesis of quinolines, was recently used to obtain a series of new heterocyclic structures in which the quinoline ring is condensed with other rings [37].

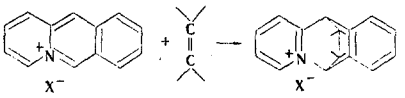
N-Arylacylimido chlorides and N-arylacylimino esters, which condense with nitriles in the presence of $AlCl_3$ [38], behave like Schiff bases.



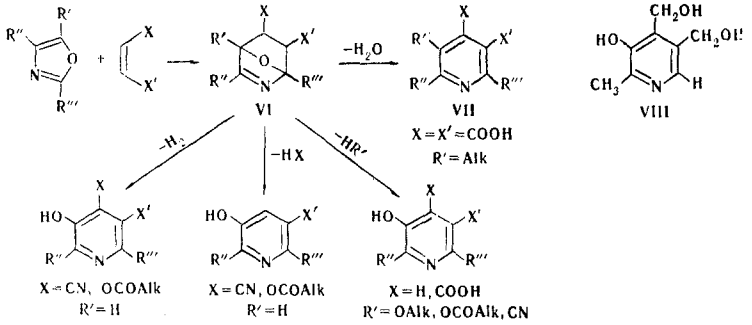
X = Cl, OCH_3

The favorable cissoid conformation, the low 1,4-localization energy, the electrophilic character, and the strong polarization of the conjugated system lead to high reactivity in the 1,4-cycloaddition of the acri-

dizinium ion and some of its condensed analogs, primarily in reactions with nucleophilic dienophiles [39-43]. The two-step mechanism of this condensation was proved in [43].

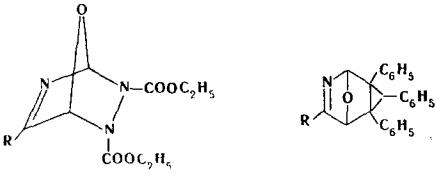


Oxazoles can be used as the 2-azadiene component [44]. Data on the reaction up to 1969 are discussed in reviews [45, 46], while the two-step mechanism was proved in a communication [47]. The paths for the aromatization of intermediate adducts VI (see the scheme below), which are usually unstable and are isolated only in a few cases [48-50], are extremely diverse. As a result, one obtains various pyridine derivatives – cinchomeronic acids (VII) and their derivatives, pyridoxine (VIII) and its analogs, as well as other types of compounds.

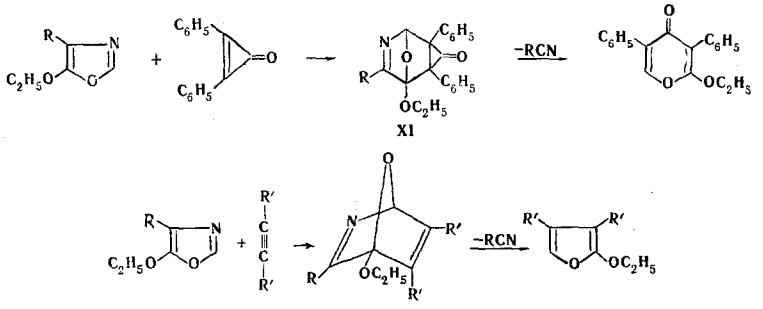


The reaction was also accomplished for benzoxazole [19]. The condensation of oxazoles with fumaronitrile to give nitriles of the pyridine series was recently described [51]. The reaction of vinylpyridines with oxazoles gives dipyridyls [52].

Diethyl azodicarboxylate and 1,2,3-triphenylcyclopropene [53, 54] were also used as dienophiles, as a result of which adducts IX and X were isolated.

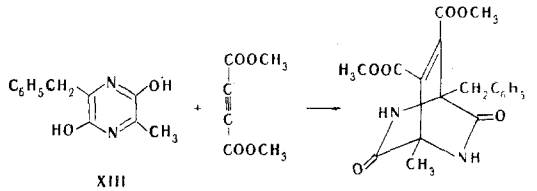


An interesting variant of the retrodiene synthesis, which leads to oxygen-containing heterocycles and is accompanied by the liberation of a nitrile molecule, was observed in [53-55] for the adducts of several oxazoles with diphenylcyclopropene (XI) and acetylenic dienophiles (XII).



The above material illustrates the diverse synthetic possibilities of 1,4-cycloaddition with oxazoles as the diene component.

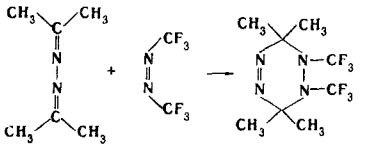
The diene synthesis with dimethyl acetylenedicarboxylate and norbornene was recently accomplished for 2,5-dihydroxypyrazine (XIII) [56].



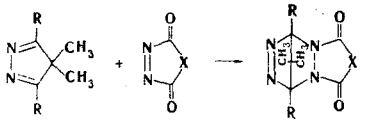
XIII

1,4-Cycloaddition for the 2,5-dihydroxypyrazine system is proposed [56] as an intermediate step in the biosynthesis of brevianamide A.

2,3-Diaza-1,3-dienes. Azines are inactive in 1,4-cycloaddition because of their tendency to add two molecules of olefin in the 1,3 position via a "crosswise" scheme. The known examples of this reaction are presented in reviews [10, 57], while later studies [58-60] confirm this property. The single exception is the reaction of isopropylideneazine with perfluoroazomethane [61].



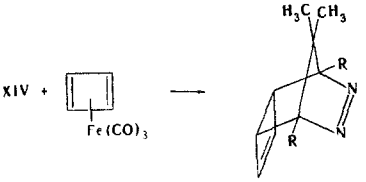
(4H)-Pyrazoles (XIV) have been successfully introduced into the reaction with 1-phenyl-1,2,4-triazoline-2,5-dione [62, 63] and 4,4-dialkylpyrazoline-3,5-dione [62, 64].



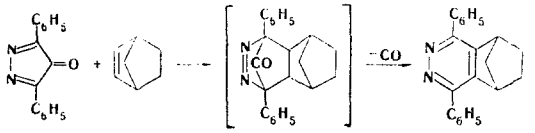
XIV

X = NC$_6$H$_5$, CR$_2$

According to [65], (4H)-pyrazoles do not react with hydrocarbon dienophiles, but they have been made to undergo cycloaddition to cyclobutadiene [66].

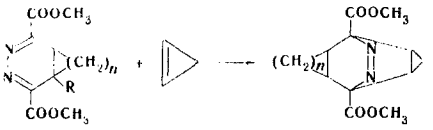


1,4-Cycloaddition was recently observed for 3,4-diazacyclopentadienone (XV) [67] on reaction with norbornene.



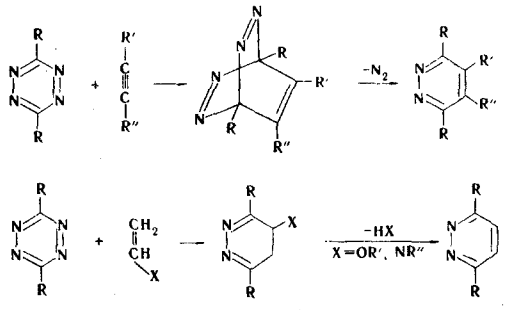
XV

2,3-Diaza-1,3-dienes are also reactive in the six-membered ring [68, 69].



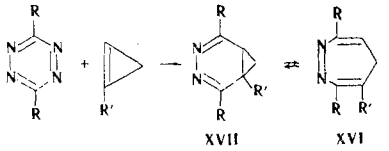
n = 1, 2

In contrast to noncyclic azines, sym-tetrazines are extremely active in the diene synthesis. This property of them, which was observed in 1959 [70], subsequently became the subject of systematic investigations [71-75]. A characteristic peculiarity of the behavior of tetrazines is their increased activity with respect to nucleophilic dienophiles (inversion of the diene synthesis) [71]. The reaction of tetrazines with olefins [70-72, 76-78] is a method for the synthesis of 1,4-dihydropyridazines, while pyridazines are synthesized by the reaction of tetrazines with acetylenes [70, 72, 78, 79], vinyl ethers, and enamines [75].



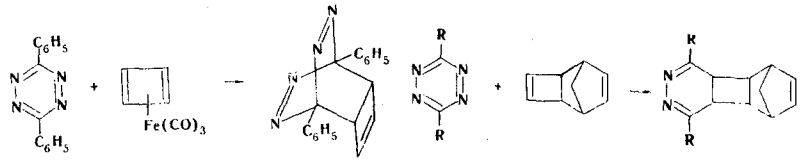
The reaction has been proposed as a quantitative method for the analysis of olefins [76].

A number of studies [73-75, 80] have been devoted to the reaction of tetrazines with cyclopropenes, particularly in connection with the problem of the valence isomerization of (4H)-1,2-diazepines to dihydropyridazines (XVI⇌XVII).



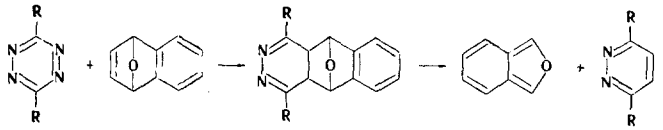
XVII XVI

Examples of the reactions of tetrazines with cyclobutadiene (as a complex with iron tricarbonyl) [81] and with cyclobutene derivatives [69, 82] are interesting.



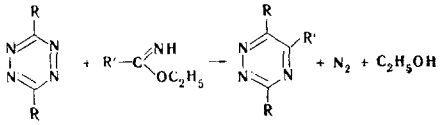
R=2-pyridyl

A variant of the reaction with tetrazines has been proposed for the preparation of isobenzofuran [83].

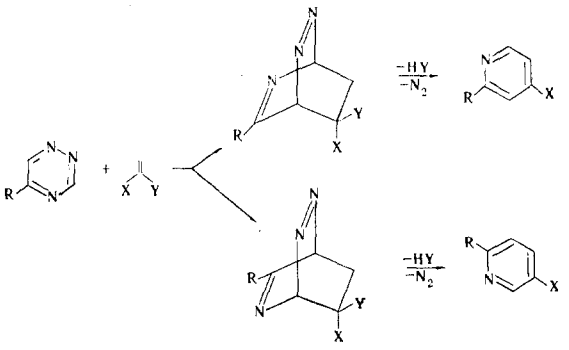


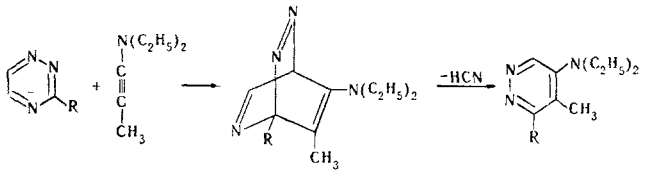
R=2-pyridyl

The reaction of tetrazines with imino esters serves as a new method for the synthesis of 1,2,4-triazines [78].

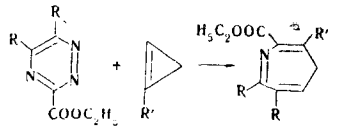


It was recently established that the 2,3-diaza-1,3-diene system is also active in 1,2,4-triazines, although the reactions proceed with more difficulty than with tetrazines. The 1,4-cycloaddition of 1,2,4-triazines to nucleophilic olefins leads to pyridines [84, 85], while the addition to acetylenes gives pyridazines [84].

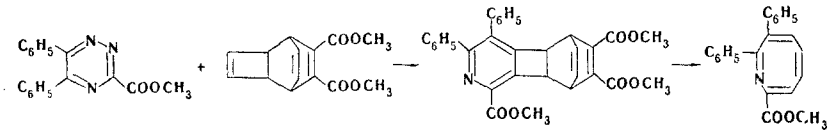




The action of cyclopropenes on 1,2,4-triazines is a method for the synthesis of (4H)-azepines [85].



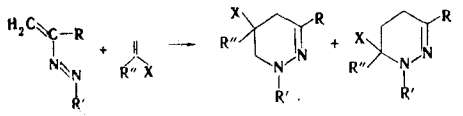
Azocines are similarly obtained from cyclobutene derivatives [86].



Azadienes with Nitrogen Atoms at the Ends and in the Middle of the Chain

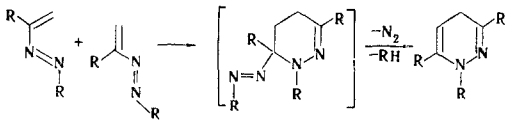
α,β-Unsaturated Azo Compounds. The Diels–Alder reaction with aliphatic α,β-unsaturated azo compounds was first realized in 1968 [87]. 1,4-Cycloaddition is a general property of aliphatic α,β-unsaturated azo compounds when they react with electrophilic dienophiles (dienophiles of the acrylic type, maleic anhydride, and dimethyl fumarate) [88-91]. The structural specificity in condensations with unsymmetrical dienophiles, as a result of which one obtains a mixture of structural isomers of Δ^2-tetrahydropyridazines, was studied in [91], while the stereospecificity of the process was proved in [92, 93].

The 1,4-cycloaddition of 1,2-diaza-1,3-dienes is a general method for the synthesis of Δ^2-tetrahydropyridazines.

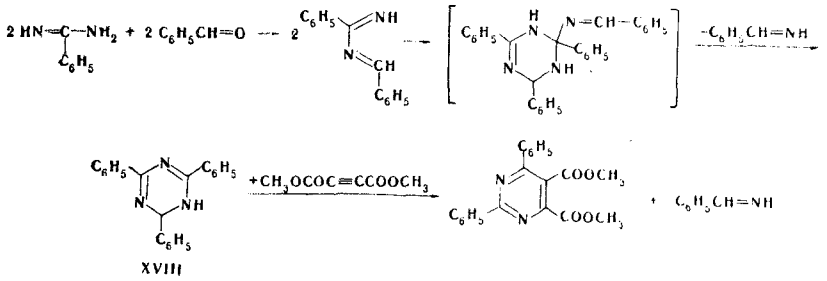


This 1,4-cycloaddition was recently observed for arylazocyclohexenes [94] and for carbohydrate derivatives with an α,β-unsaturated azo fragment [95].

It has been demonstrated that α,β-unsaturated azo compounds dimerize via the scheme of the diene synthesis [92, 96].

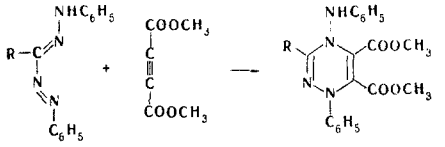


1,3-Diaza-1,3-dienes. 1,4-Cycloaddition only recently became known for the conjugated system of 1,3-diaza-1,3-dienes in the synthesis of sym-dihydrotriazine XVIII and also in its reaction with dimethyl acetylenedicarboxylate [97].



XVIII

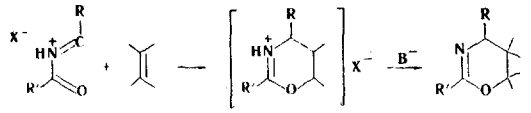
1,2,4-Triaza-1,3-dienes. As demonstrated in [98] in two cases, formazans react with dimethyl acetylenedicarboxylate to give, in addition to other products, 1,4-dihydro-1,2,4-triazine derivatives.



R=H, CH_3

Azadienes Containing More than One Heteroatom

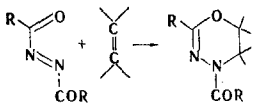
1-Oxa-3-aza-1,3-dienes. Acylimmonium salts XIX are active diene components that readily react with olefins (alkenes, dienes, and α,β-unsaturated carbonyl compounds) to form (4H)-5,6-dihydro-1,3-oxazines [99-101].



XIX

The reaction of XIX with acetylenes gives 1,3-oxazines [102], while (4H)-1,3,5-oxadiazines are obtained from XIX and nitriles [102, 103]. A detailed study of the peculiarities of this condensation demonstrated that it proceeds via a synchronous mechanism [99, 101].

1-Oxa-3,4-diaza-1,3-dienes. Azodicarbonyl compounds, which are active dienophiles with respect to nucleophilic dienes, can in turn act as diene components in reactions with nucleophilic olefins (indene, cyclopentene, norbornene, and vinyl ethers) [104-110] to form 5,6-dihydro-1,3,4-oxadiazines. 1,2-Cycloaddition to give 1,2-diazetidines competes with this transformation. Several factors that determine the direction of the process are examined in [105].



From the material presented above it follows that the rapidly developing azadiene synthesis has evolved into an independent area of the Diels–Alder reaction. The diverse variants of 1,4-cycloaddition lead to the production of numerous types of nitrogen heterocycles (pyridines, quinolines, pyridazines, triazines, oxazines, oxadiazines, etc.) with different degrees of saturation. In individual cases, there is a transition from one class of heterocycles to another, with the same number of atoms or with an increase or reduction in the number of links in the ring. At present, all types of aza- and diazadiene systems have been successfully introduced into the diene synthesis, and the reactions with some of them are not only promising but sometimes the most acceptable methods for the synthesis of some heterocyclic systems. The stereospecificity, structural orientation, and mild conditions of many of the condensations make it possible to realize directed syntheses. All of this creates grounds for further investigations in this field.

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