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HETARYLCYANAMIDES. (REVIEW)

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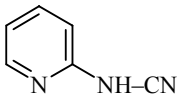
The nomenclature and the synthesis of hetarylcyanamides and their reactions that take place at the cyanoamino group are discussed.

Keywords: hetarylcyanamides, synthesis, chemical properties.

The development of simple methods for the synthesis of stable Het–NHCN compounds and the possibility of producing of new heterocyclic systems with useful practical characteristics based on them have recently increased the interest of chemists in compounds with such structures. By analogy with alkyl- and arylcyanamides they have been called hetarylcyanamides.

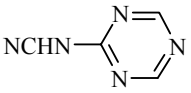
R–NHCN	Ar–NHCN	Het–NHCN
alkylcyanamides	arylcyanamides	hetarylcyanamides

In the IUPAC rules the cyanamide function (unlike the structurally similar –OCN or –SCN) does not have a separate name. In this respect hetarylcyanamides can probably be regarded more correctly as nitrile derivatives of carbamic acid and called hetarylcarbamonitriles. Sometimes such compounds are called substituted amines, for example,

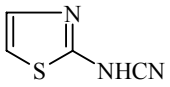


N-cyano-2-pyridylamine or N-(cyano)-N-(2-pyridyl)amine

During the formulation of names for such compounds some authors give priority to the heterocycle and for this reason place the cyanoamino group in the prefix. If its location in the heterocycle is ambiguous, it is indicated by a locant. For example,



cyanoamino-*sym*-triazine



2-cyanoaminothiazole

In order to present the material consistently we will call these compounds hetarylcyanamides.

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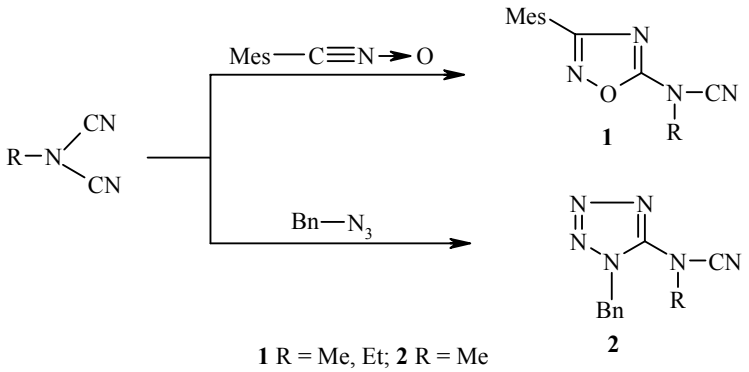
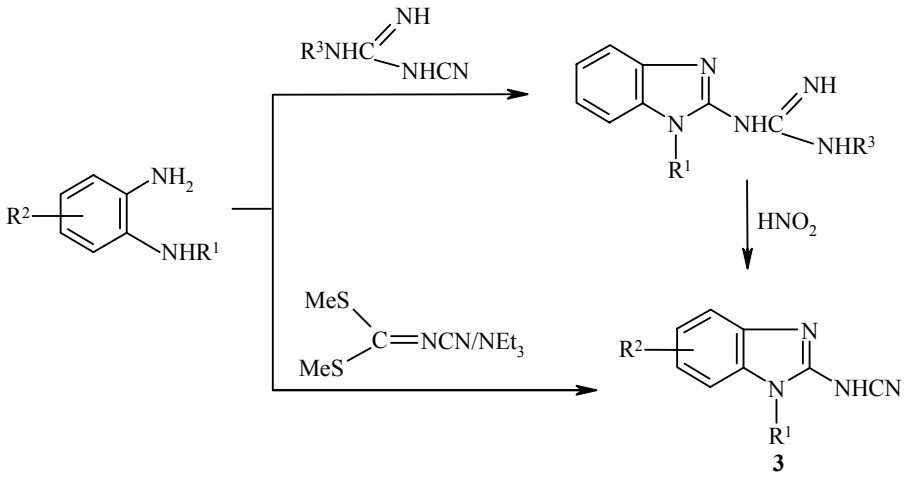
1. METHODS OF SYNTHESIS

There are various methods for the synthesis hetarylcyanamides involving the construction of the heterocycle from functionalized N-cyano compounds with retention of the cyanamide function or the introduction of a cyanoamino group into the heterocycle.

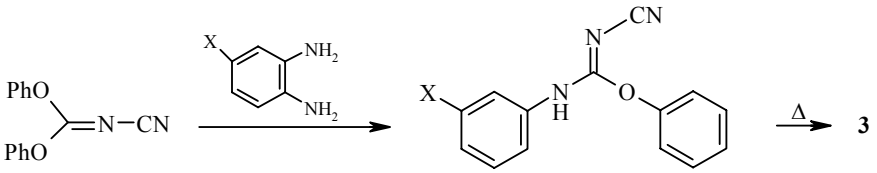
Thus, the 1,3-dipolar cycloaddition of methyl- or ethyldicyanamide with mesitylnitrile oxide or benzyl azide gives the corresponding oxadiazolyl- and tetrazolylcyanamides **1** and **2** [1].



The reaction of *o*-phenylenediamines with cyanoguanidine leads to the formation of 2-benzimidazolylguanidines, which are readily converted by the action of nitrous acid into 2-benzimidazolylcyanamides **3** [2-6].

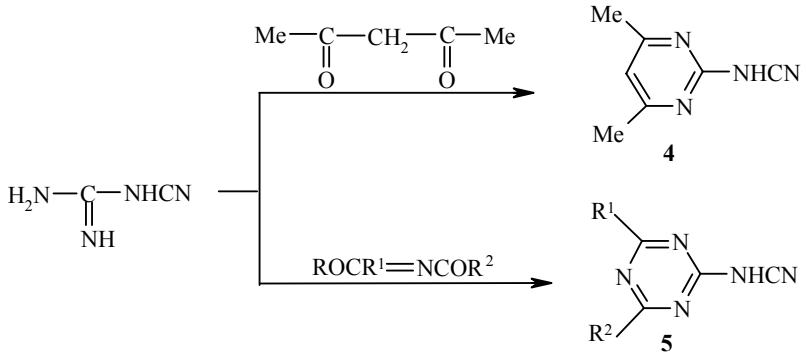


Analogous cyanamides **3** were obtained from *o*-phenylenediamines and dimethyl N-cyanodithioimidocarbonate in the presence of triethylamine. Electron-donating substituents in the benzene ring promote the reaction, while electron-withdrawing substituents hinder it. In the absence of the catalyst different reaction products are formed [7]. The reaction of diphenyl cyanocarbamate with *o*-phenylenediamine at room temperature leads to derivatives of N-cyano-O-phenylisourea, which undergo cyclization to the cyanamides **3** when boiled in 2-propanol. In an analogous reaction *o*-aminophenol immediately forms 2-benzoxazolylcyanamide [8].



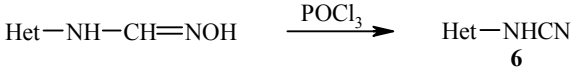
X = H, Cl

Cyanoguanidine reacts with acetylacetone, forming 4,6-dimethyl-2-pyrimidylcyanamide (**4**) [9].



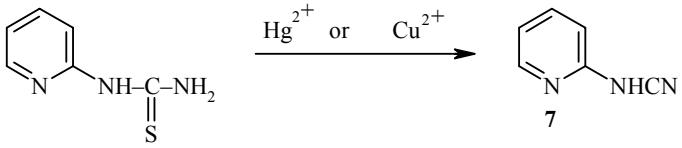
The 1,3,5-triazinylcyanamides **5** were synthesized by the cyclocondensation of cyanoguanidine with imidic esters [10].

Methods for the production of hetarylcyanamides based on the modification of substituents in the heterocycle to a cyanamide function are more varied. Thus, for example, the hydroxyiminomethyleneamino group is modified by the action of a dehydrating agent in the presence of a base [11, 12].

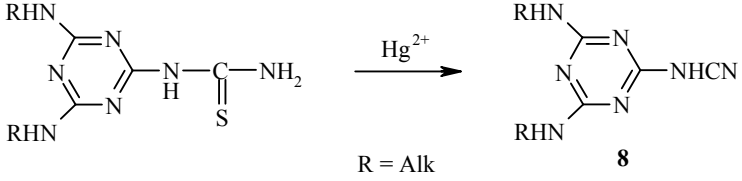


Het = 2-pyridyl, 2-pyrimidyl, 2-pyrazinyl, 3-pyridazinyl, 6-chloro-3-pyridazinyl, 2-thiazolyl

Oxidative desulfurization of 2-pyridylthiourea by the action of mercuric oxide or cupric hydroxide gives 2-pyridylcyanamide (**7**) [13].

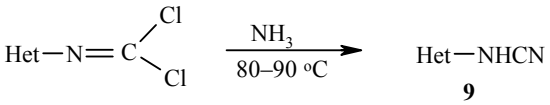


Substituted 1,3,5-triazinylcyanamides **8** can be obtained in a similar way [14].



R = Alk

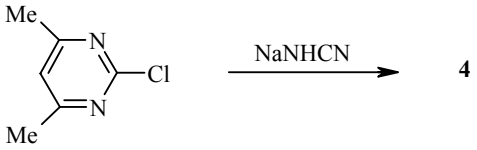
The reaction of hetarylphosgene imines with ammonia can probably be used as a general method for the production of hetarylcyanamides **9** [15].



9

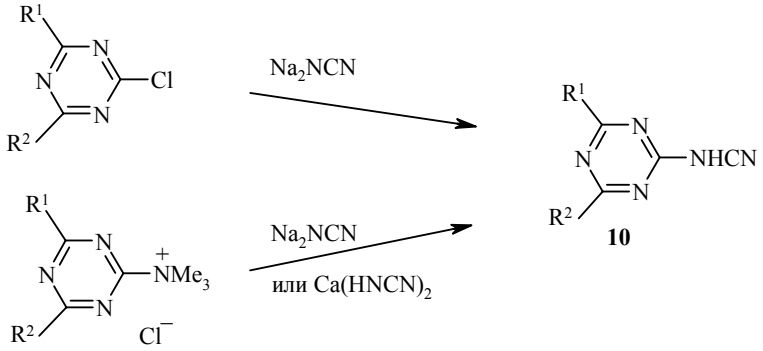
Het = 4-R^1-6-R^2-1,3,5-triazinyl

Substitution of the chlorine in 2-chloro-4,6-dimethylpyrimidine by a cyanamide group gives the corresponding hetarylcyanamide **4** [8].



4

Monochloro-substituted *sym*-triazines also undergo cyanoamination with sodium cyanide to form triazinylcyanamides **10** [16-19].

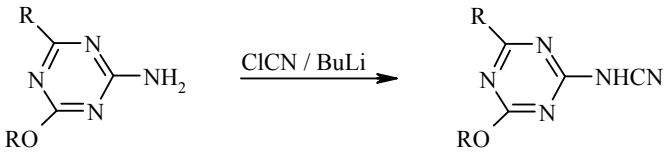


10

R^1R^2 = alkyl(aryl)amino; R^1 = alkylamino, R^2 = MeO, MeS

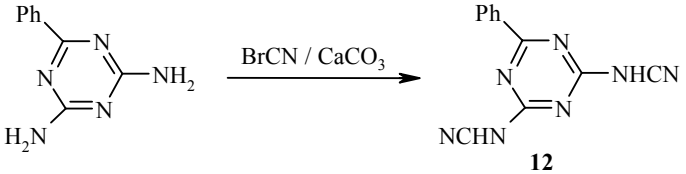
Trimethyl-*sym*-triazinylammonium chlorides undergo cyanoamination considerably more readily, and this makes it possible to conduct the reaction not only with sodium cyanide but also with calcium cyanamide [20-24].

The widely used method involving cyanation of alkyl- and arylamines with cyan halides has limited application in the case of hetarylamines. Only in the presence of a strong base such as butyllithium was it possible to realize this reaction with 1,3,5-triazinylamine with the formation of the corresponding cyanamide **11** [25].



11

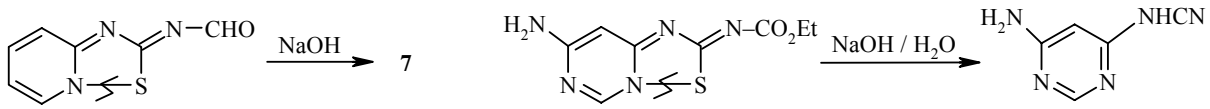
If there are two amino groups in the triazine ring, cyanation takes place under milder conditions. In this case cyanogen bromide in the presence of calcium carbonate is used as cyanation agent [26].



12

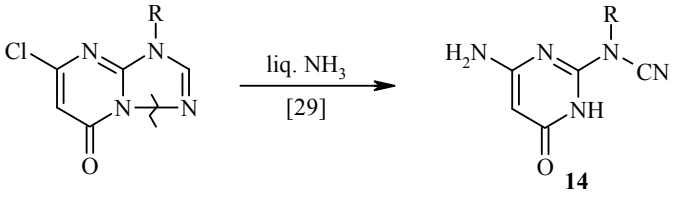
The reaction takes place at both amino groups with the formation of triazinedicyanamide **12**.

Some azinylcyanamides, such as **7** [27] or **13** [28], can be obtained by the cleavage of thiazoloazines with alkali.



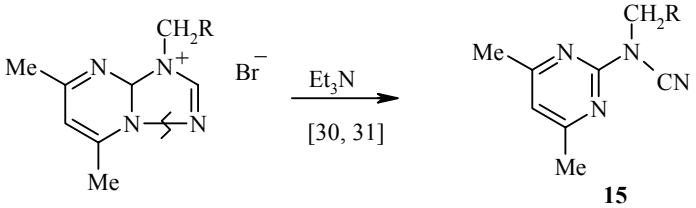
13

Derivatives of 1,3,4-triazoloazines unsubstituted at position 2 are easily cleaved by the action of bases with the formation of the cyanamides **14-17**.



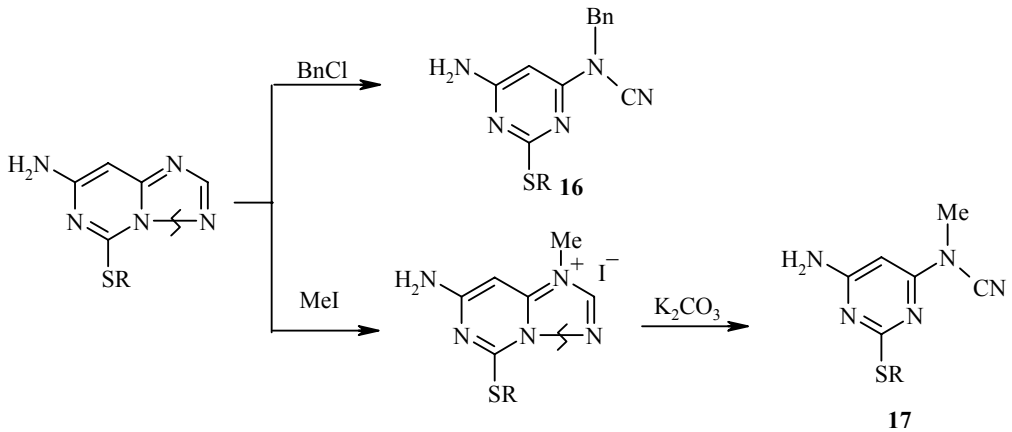
14

R = β-*D*-ribofuranosyl



15

R = H, CN, COOMe



17

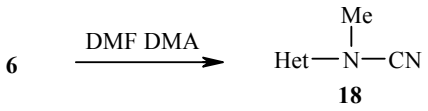
R = Me, $PhCH_2$ [32]

2. CHEMICAL PROPERTIES

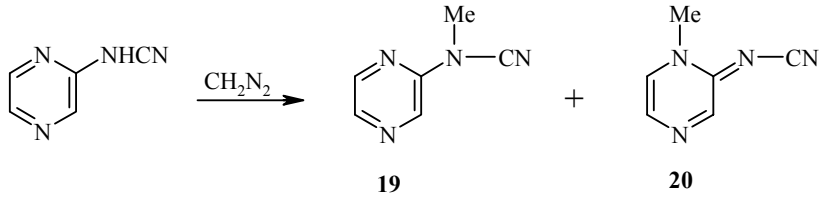
Hetarylcyanamides enter into reaction with a wide range of reagents, since their molecules contain two substantially different reaction centers, i.e., the nucleophilic nitrogen atom of the amino group and the electrophilic carbon atom of the nitrile group. The molecules of such compounds are capable of substitution at the amino group or addition and cycloaddition at the triple bond of the nitrile group. Many of the reactions affecting the amino group are catalyzed by bases or require the participation of an anion. The reactions at the nitrile group take place in the presence both of acids and of bases. The hetaryl substituent has the greatest effect on the amino group. On account of the fact that the amino and cyano groups are linked to each other the effect of the heterocycle extends indirectly to the cyano group. This makes the hetarylcyanamides (in contrast to the aliphatic and aromatic analogs) more resistant to di- and trimerization.

2.1. Reactions of the Amino Group

The hetarylcyanamides **6** are easily methylated by treatment with dimethylformamide dimethyl acetal (DMF DMA) with the formation of the corresponding N-methylhetarylcyanamides **18** [11].

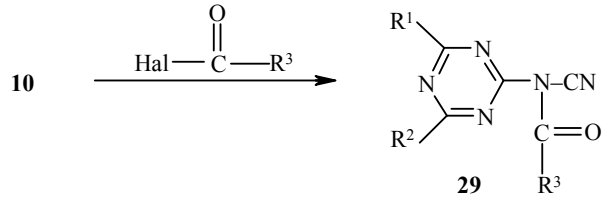


The methylation of 2-pyrazinylcyanamide with diazomethane leads to N-methylation in the side chain (compound **19**) and N-methylation at the $N_{(1)}$ atom in the heterocycle (compound **20**) [12].

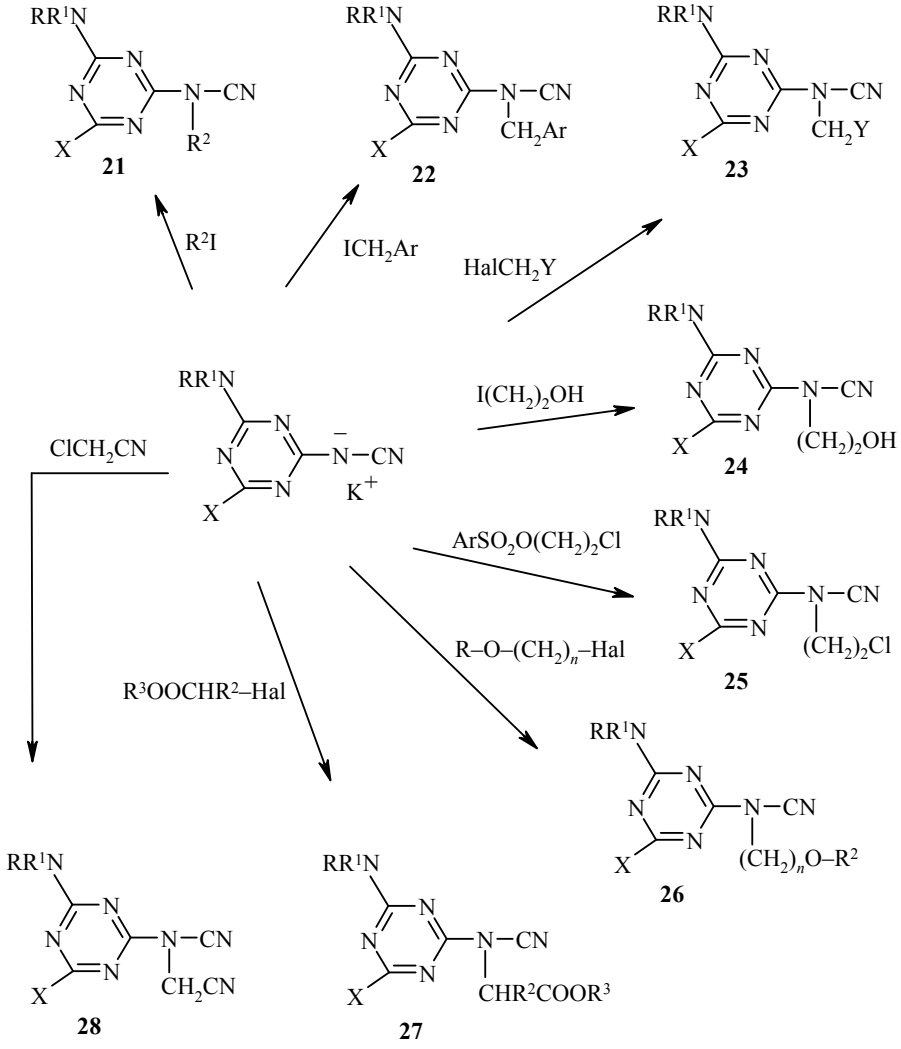


The hydrogen atom in the NH group of hetarylcyanamides **10** has acidic character, and this makes it possible to obtain stable potassium salts. These salts react readily with alkyl halides [33-36], arylalkyl halides [37], halohydrins [38], chloroethyl tosylates [39], haloalkyl ethers [40], chloroacetic, propionic, and bromomalonic esters [41-43], haloacetonitriles [41], and others. The reaction products are N-alkylhetarylcyanamides **21**-**28** (Scheme 1).

The hetarylcyanamides **10** are acylated by the Schotten–Baumann procedure with the formation of the derivatives **29** [44].

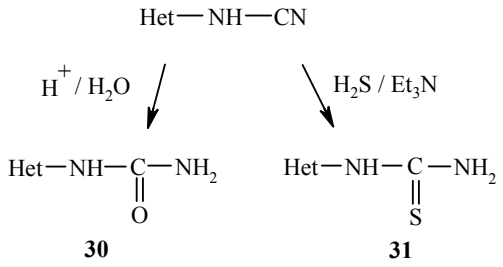


Scheme 1

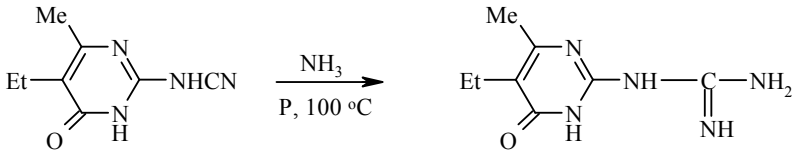


2.2. Addition at the Cyano Group

Hydrolysis of the cyano group of hetarylcyanamides in sulfuric acid (1:1) gives the corresponding N-hetarylureas **30** [11, 12, 45]. The hetarylthioureas **31** are formed when hydrogen sulfide is passed through a solution of the hetarylcyanamide **6** in the presence of triethylamine or ammonia [12, 14, 46-48].



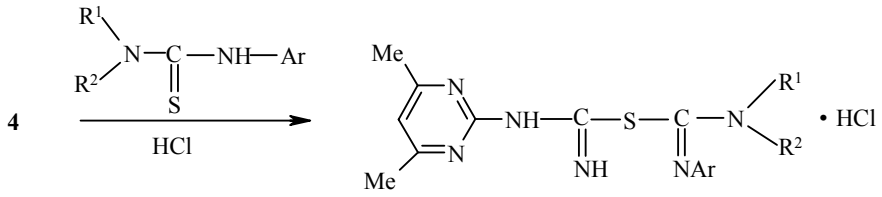
Aminolysis of the hetarylcyanamide **32** leads to the formation of the hetarylguanidine **33** [46].



32 **33**

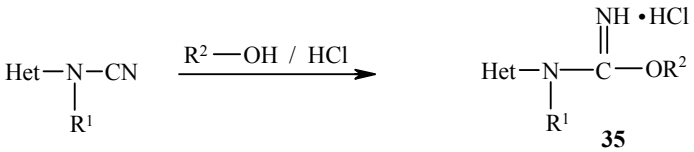
Analogous compounds are formed during addition to the cyano group of substituted amines [47, 48].

Pyrimidylcyanamide **4** reacts with trisubstituted thioureas in a hydrochloric acid medium. The reaction products in this case are thiobisformamidines **34** [46].



34

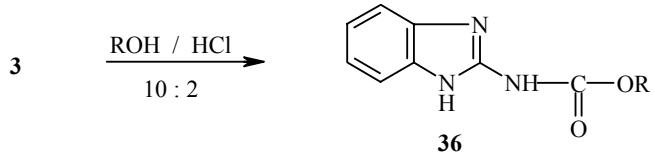
Alcoholysis of hetarylcyanamides in the presence of gaseous hydrogen chloride leads to the formation of the hydrochlorides of hetaryl-O-alkylisoureas **35** [49].



35

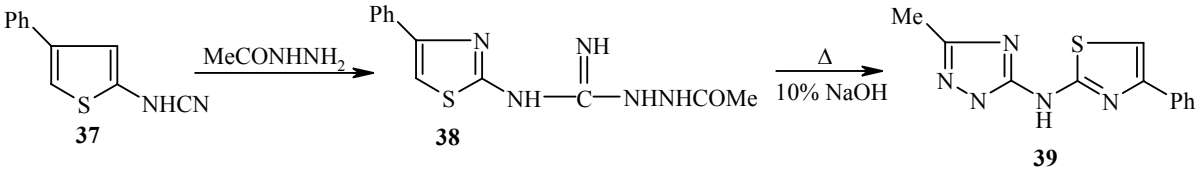
Het = 1,3,5-triazinyl

Alcoholysis in dilute hydrochloric acid is accompanied by hydrolysis of the intermediately formed O-alkylisoureas to the carbamic esters **36** [50].



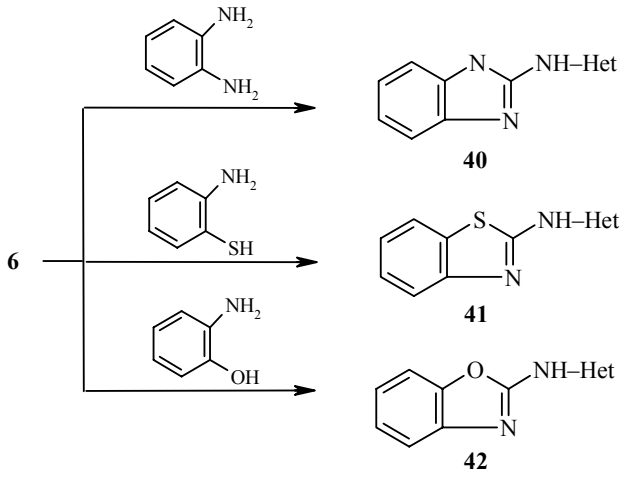
36

3-Methyl-5-(4-phenyl-2-thiazolylamino)-1,2,4-triazole (**39**) was obtained from thiazolylcyanamide **37** and acetylhydrazine through derivatives of acetylaminoguanidine **38** [51].

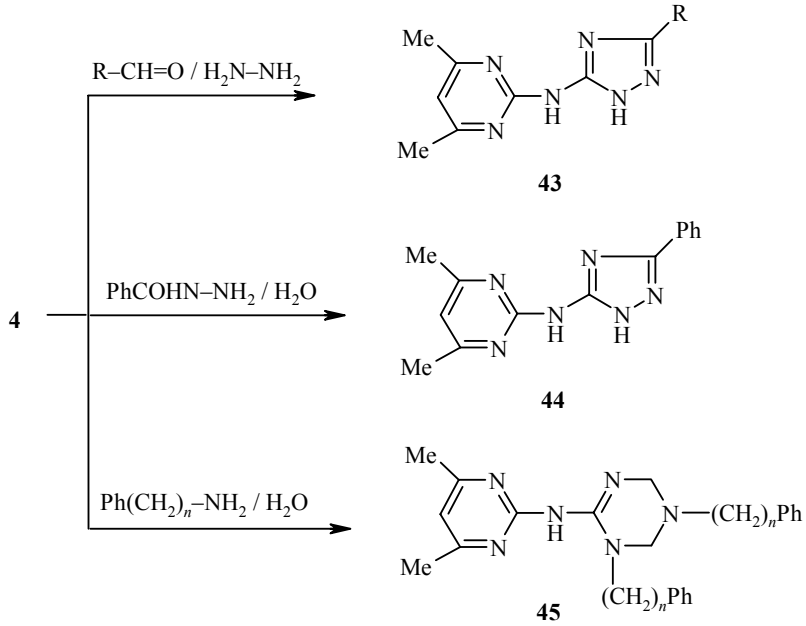


37 **38** **39**

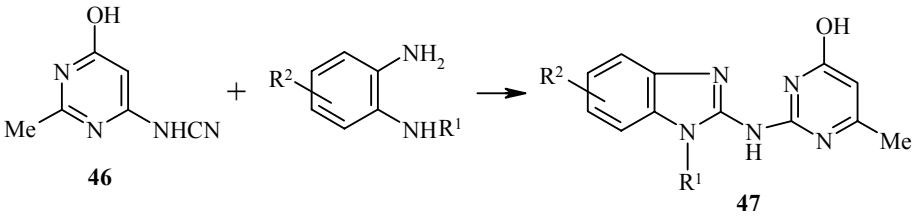
The reaction of *o*-phenylenediamine and *o*-aminothiophenol with hetarylcyanamides **6** takes place with the immediate formation of the corresponding derivatives of hetarylaminobenzimidazole **40** and hetarylaminobenzothiazole **41**. *o*-Aminophenol reacts under harsher conditions and with more prolonged reaction time, leading to the formation of hetarylaminobenzoxazoles **42** [52].



The pyrimidylaminotriazole **43** is formed when pyrimidylcyanamide **4** is treated with hydrazine hydrate and formaldehyde. The analogous reaction with benzaldehyde as carbonyl component leads to the formation of compounds **44**. The three-component condensation of cyanamide, formaldehyde, and benzylamine or phenylethylamine gives pyrimidylaminotriazole **45** [53].

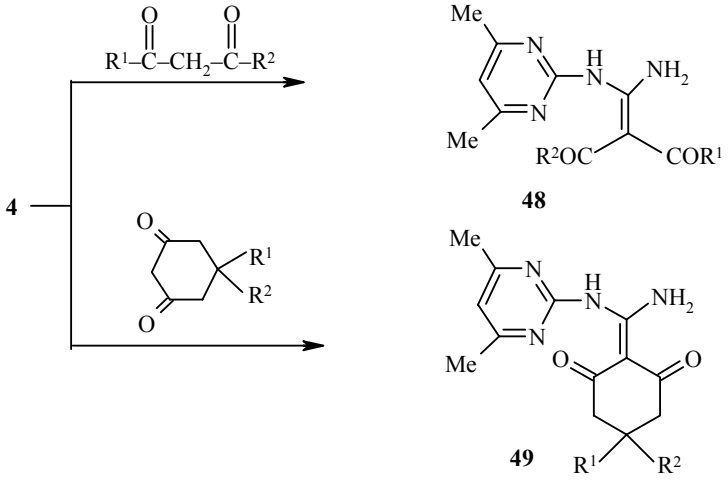


2-(2-Benzimidazolylamino)-4-hydroxypyrimidines **47** were synthesized by the cyclization of hetarylcyanamide **46** with substituted *o*-phenylenediamines [54].



46 **47**

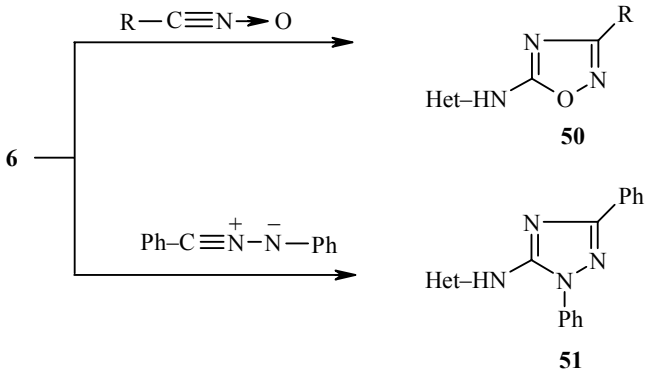
The hetarylcyanamide **4** reacts abnormally with β-diketones and β-keto esters in the presence of catalytic amounts of nickel acetylacetate with the formation of the N,N-acetals of diacyl- and alkoxycarbonyl(acyl)ketenes **48** and **49** [55, 56].



48 **49**

R^1 = Alk or Ph, R^2 = Me or EtO

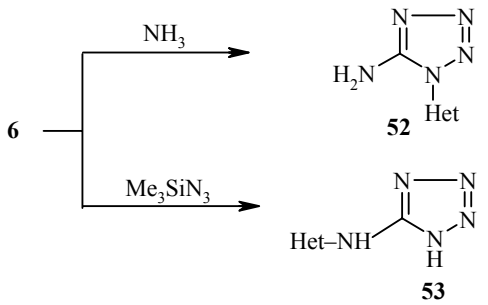
2.2.1. Cycloaddition at the Cyano Group. Nitrile oxides and nitrile imines react at the cyano group according to a 1,3-dipolar cycloaddition mechanism. In the first case hetarylamino-1,2,4-oxadiazoles (**50**) are formed, and in the second hetarylamino-1,2,4-triazoles (**51**) [46].



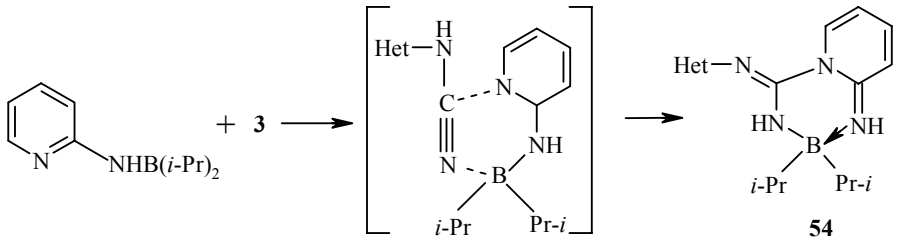
50 **51**

Het = pyrimidyl, 1,3,5-triazinyl

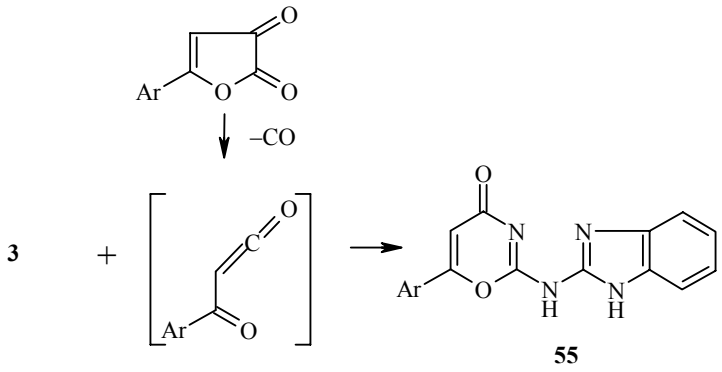
The hetarylcyanamides **6** react with azides in two directions, forming 5-amino-1-hetaryltetrazoles **52** or 5-hetarylaminotetrazoles **53**. Compounds of the first type are formed under mild conditions, whereas in higher-boiling solvents or with azidotrimethylsilane the thermodynamically more stable compounds **53** are formed [52].



2-Isopropylborylaminopyridine adds smoothly to the cyanamide **3** with the formation of the [4+2] cycloadduct **54** [57].



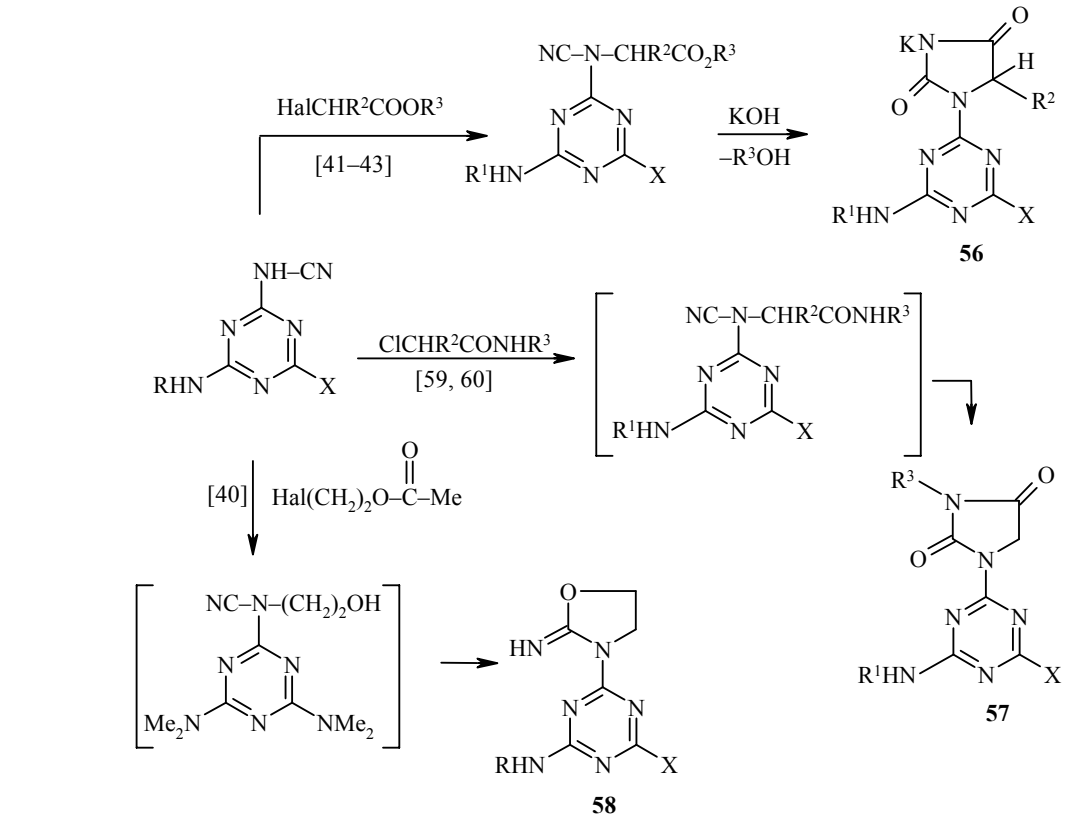
The benzimidazolylcyanamide **3** enters into [4+2]-cycloaddition with aroylketenes, generated during thermolysis of 5-aryl-2,3-dihydrofuran-2,3-diones. The products are benzimidazolylaminooxazinones **55** [58].



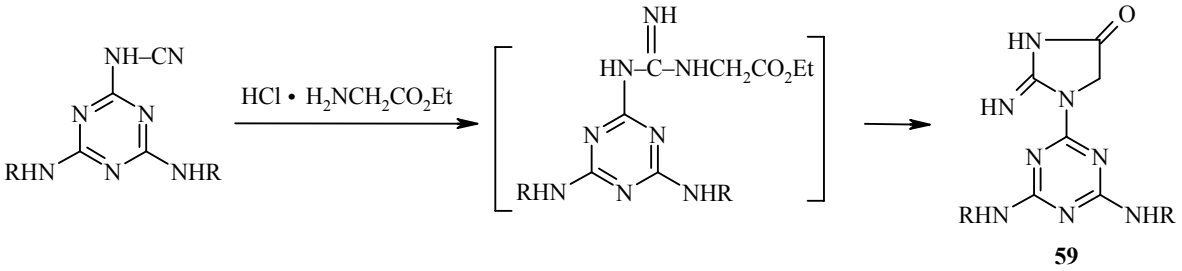
The pyrimidylcyanamide **4** does not enter into this reaction.

2.3. Reactions with the Simultaneous Participation of the Amino and Cyano Groups

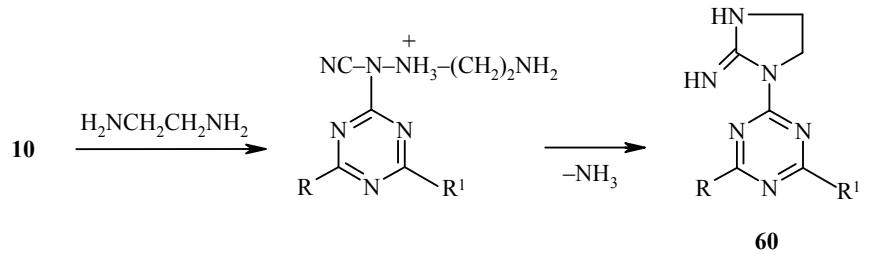
As already mentioned above, the hetarylcyanamides **10** can react at both groups simultaneously; at the first stage substitution by the functionalized alkyl halide occurs in the amino group, and at the second intramolecular addition of the introduced function occurs at the multiple bond of the cyano group. This is illustrated by the scheme.



In some cases the order of the stages may be reversed [61]



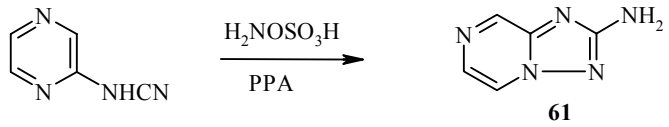
On account of the acidic characteristics of the NH group in the hetarylcyanamide **10** 2-aminoethylammonium salts are formed in its reaction with ethylenediamine, and on heating they release ammonia to form compounds **60** [62].



Oxirane and aziridine react with the hetarylcyanamides **10** with the formation of compounds **58** [62] and **60** [63].

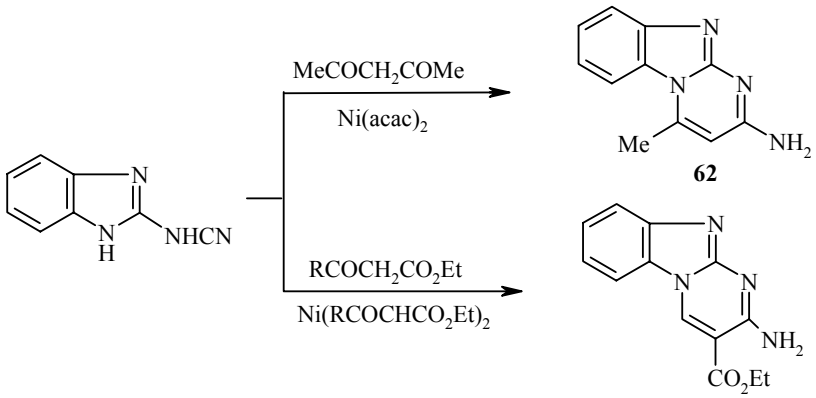
2.4. Reactions with the Participation of the Cyanoamino Group and the Heterocycle

As a rule condensed heterocyclic systems are formed in reactions that take place with participation of the cyanoamino group and the heterocycle. Thus, treatment of 2-pyrazinylcyanamide with hydroxylaminesulfonic acid in the presence of polyphosphoric acid gives the triazolopyrazine **61** [52].



61

With acetylacetone in the presence of nickel acetate the benzimidazolylcyanamide **3** undergoes cyclocondensation to form the pyrimido[1,2-*a*]benzimidazole **62** [64]. Acylacetic esters enter into this reaction with the formation of pyrimido[1,2-*a*]benzimidazoles **63** [65].

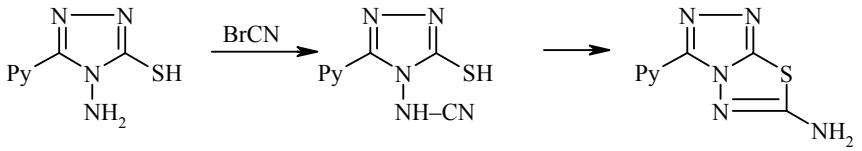


62 **63**

Annelation of the pyrimidine ring to the benzimidazole ring is promoted by activation of the C≡N group in the hetarylcyanamide by the catalyst. In the absence of the nickel salts it is not possible to synthesize compounds **62** and **63** even if acidic or basic catalysts are used.

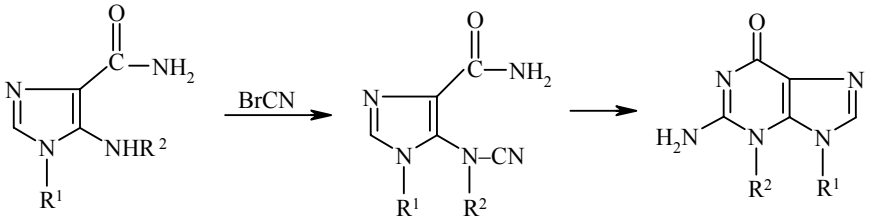
2.5. Intramolecular Reaction of the Cyanamide Group with Other Groups in the Heterocycle

The cyanation of 1-amino-2-mercaptotriazole **64** with bromocyanogen gives the hetarylcyanamide **65**, which undergoes cyclization under the reaction conditions to 6-amino-*sym*-triazolo[3,4-*b*]-1,3,4-thiadiazole **66** [66].



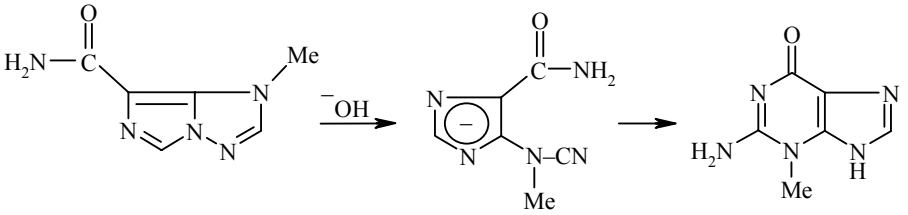
64 **65** **66**

The cyanation of 5-amino-4-imidazolecarboxamide **67** takes place in a similar way. The reaction of the amide and cyanamide groups in the intermediate compound **68** leads to the formation of the guanine derivatives **69** [67, 68].



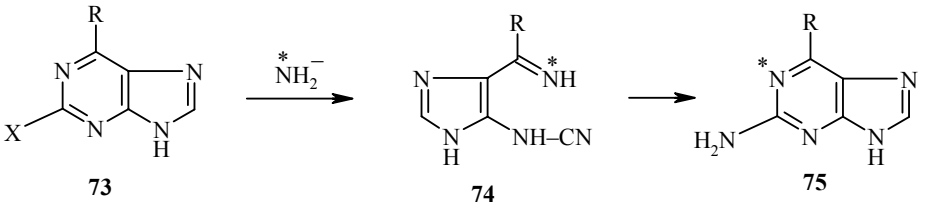
67 **68** **69**

The recyclization of imidazo[1,5-*b*]-*s*-triazole **70** to the guanine **72** also takes place with the participation of these groups [69].



70 **71** **72**

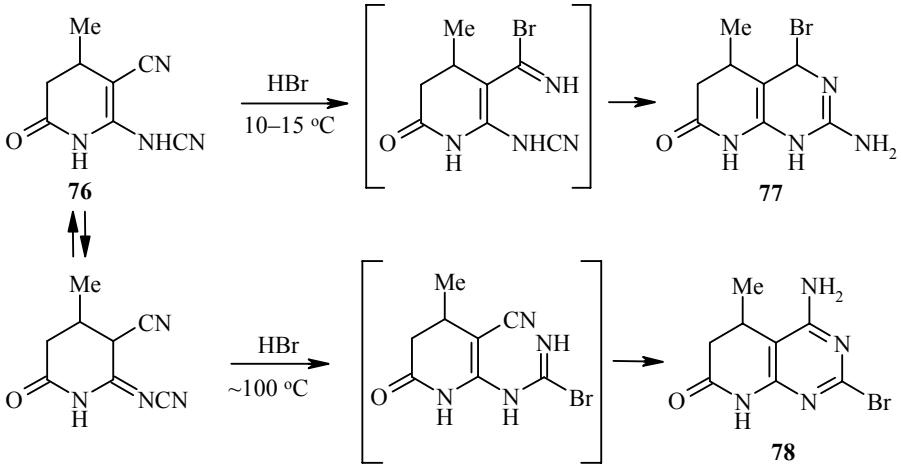
Amination of the purines **73** with a labeled amine showed that dehalogenation takes place by an S_N (ANRORC) mechanism with the intermediate formation of the hetarylcyanamide **74**. The final product of such a reaction is the aminopurine **75** [70].



73 **74** **75**

R = H, Ph; X = F, Cl, SMe

If there is a nitrile group at position 3 of the hetarylcyanamide **76**, cyclization takes place under the influence of hydrogen bromide. The nitrile group is more active at reduced temperature, and the cyanamide group at increased temperature. In the first case compound **77** is formed, and in the second its regioisomer **78** is formed [71, 72].



76 **77** **78**

In the presence of hydrogen chloride only one isomer **78** was isolated irrespective of the reaction conditions [72].

Thus, the published data on the synthesis and properties of hetarylcyanamides that we examined indicate that these compounds can be used as important synthons for the production of various azaheterocycles. In addition, these hetarylcyanamides exhibit specific biological activity. Thus, derivatives of *sym*-triazinylcyanamides have found use as pesticides [73]. Substances with sedative and tranquillizing activity have been found among the synthesized 1,3-oxazin-4-ones produced from hetarylcyanamides [74].

A series of papers devoted to the problems discussed in this review appeared in 2000-2001 [75-79].

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