

SYNTHESIS AND PROPERTIES OF THE S-, O-, AND N-ALLYL DERIVATIVES OF AROMATIC AZINES (REVIEW)

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Published data on the synthesis and properties of S-, O-, and N-allyl derivatives of aromatic azines are classified and reviewed.

1. SYNTHESIS OF S-ALLYL DERIVATIVES

As a rule, allylthioazines are obtained by the reaction of mercaptoazines or metal thiolates with allyl halides in organic solvents. The following compounds of the pyridine series were obtained in this way: 2-Allylthiopyridine [1-3]; 2-allylthio-3-hydroxy-6-methylpyridines [4, 5]; 2-allylthio-6-methyl(aryl)-3-cyano(carboxy)pyridines [6-9]; 6-(1-adamantyl)-3-cyano-2-allylthiopyridine [7]; 2-allyl(2-cyclohexenyl)thionicotinic acids [8]; 2-allylthio-6-amino-3,5-dicyanopyridine [10]; 2-allylthio-6-amino-4-aryl-3,5-dicyanopyridine [11]; 2-allyl(2-cyclohexenylthio)-1,5-dinaphthylpyridines [12]; 3-amino- and 3-nitro-2-allylthiopyridines [13]. The following compounds of the quinoline series were obtained by this method: 2-allyl- and 4-allylthioquinolines and their substituted derivatives [14-18]; 3-allylthioquinoline [19]; 2-allylthio-4-aryl-3-cyano-5,6,7,8-tetrahydroquinolines [20]; 2-allylthio-4-aryl-7,7-dimethyl-5-oxo-3-cyano-6,8-dihydroquinolines [20]; 8-allylthioquinoline [22-25]; 1-allylthioisoquinoline [26, 27]. The following compounds of the pyrimidine series were obtained: 2-allylthiopyrimidine [28]; 2-allylthio-4,6-dimethylpyrimidine [22, 29]; 6-allylthio-2-amino-4-methylthio-5-cyanopyrimidine [30, 31]; 2-allylthiopyrimidin-4(3H)-ones [32-36]; 2-allylthio-3-phenyl-4-oxothieno[2,3-d]pyrimidines [37, 38]. Mercaptothiazines, which are capable of thione-thiol tautomerism, react with alkyl halides with the exclusive formation of the S-allyl derivatives. 9-Allylthioacridine, 3-chloro-9-allylthioacridine [39], and 2-allylthiopyrimidine [40] were obtained by the reaction of the respective mercaptothiazines with allyl halide in a two-phase system in the presence of phase-transfer catalysts.

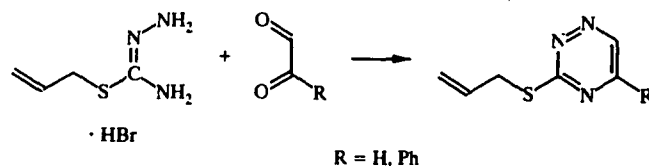
Sodium 8-mercaptoquinolate reacts with allyl bromide in water in the presence of a surfactant with the formation of 8-allylthioquinoline [41].

2-Allylthiopyridine was obtained by the reaction of S-allylthiopyridinium bromide with 2-bromopyridine [1] and by the reaction of allyl bromide with 2-pyridineisothiuronium halide [22, 42].

The reaction of ethyl 5-amino-2-allylthio-7-methylthiothieno[2,3-d]pyrimidine-6-carboxylate with allyl isocyanate gave 3-allyl-2-allylthio-7-methylthiothieno[2,3-d;4,5-d']dipyrimidin-4(3H)-one. The latter is formed as a result of allylation of the intermediately formed 3-allyl-2-mercapto-7-methylthiothieno[2,3-d;4,5-d']dipyrimidin-4(3H)-one [43].



3-Allylthio-1,2,4-triazines were obtained by the reaction of S-allylthiosemicarbazide hydrobromide with glyoxal and phenylglyoxal [44].



4-Allylthio-2(1H)-pyrimidinone was obtained by the silylation of 4-thiouracil with di(trimethylsilyl)amine in DMFA followed by treatment with allyl halide in acetonitrile [45].

2. SYNTHESIS OF O-ALLYL DERIVATIVES

Allyloxazines are produced by the reaction of halogenoazines with sodium allyl oxide. 2-Allyloxypyridine [46, 47], 2- and 4-allyloxyquinolines [48, 49], 1-allyloxyisoquinoline [50], and 4-allyloxypyrimidines [51-53] were obtained in this way.

Triallyl cyanurate was obtained with a high yield in the reaction of cyanuric chloride with allyl alcohol in the presence of alkali in an aqueous medium [54, 55].

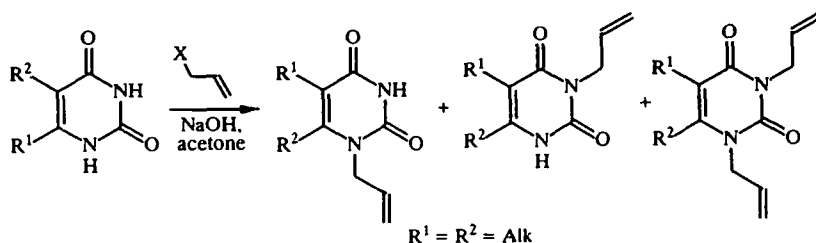
As a rule, hydroxyazines, which are capable of prototropic tautomerism, are alkylated by allyl halides with the preferential formation of N-allyl derivatives, but 6-(1-adamantyl)-3-cyano-2-hydroxypyridine [6], 5,6-disubstituted 2-hydroxy-3-phenyl-4-oxothieno[2,3-*d*]pyrimidines [38], and 4-hydroxy-2-quinolone [56] react with allyl halides with the formation of the O-allyl derivatives.

8-Allyloxyquinoline was obtained by allylation with allyl halide in the presence of sodium ethoxide [57] and in a two-phase system (sodium hydroxide—water—methylene chloride) in the presence of tetrabutylammonium bromide [58]. In [59] it was shown that a mixture of 8-allyloxyquinoline and 7-allyl-8-allyloxyquinoline is formed during the reaction of 8-hydroxyquinoline with an excess of allyl bromide in acetone and alcohol.

3-Allyloxyisoquinoline was obtained by the reaction of 3-hydroxyisoquinoline with allyl bromide in DMFA in the presence of silver carbonate [50].

3. SYNTHESIS OF N-ALLYL DERIVATIVES

A comparatively large number of papers have been devoted to the synthesis of the N-allyl derivatives of uracil and its derivatives. In [60] it was shown that the allylation of uracils by allyl halide in the presence of sodium hydroxide in acetone gives a mixture of $N_{(1)}$ -, $N_{(3)}$ -, and $N_{(1),N_{(3)}}$ -allyl derivatives; in [61] the $N_{(1)}$ -allyl and $N_{(1),N_{(3)}}$ -diallyl derivatives were obtained in the presence of potassium carbonate in DMFA, while in [62] the $N_{(1),N_{(3)}}$ -diallyl derivative was obtained in a two-phase system.



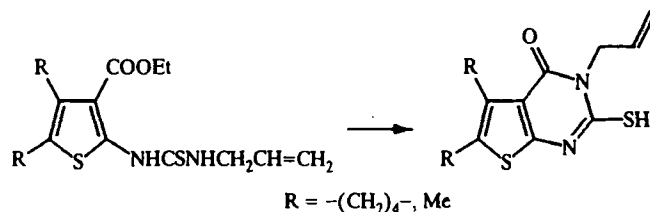
The allylation of 5-fluorouracil with allyl bromide in crown ether in the presence of potassium carbonate gave 1-allyl-5-fluorouracil [63]. Allylation of the potassium salt of 6-methyluracil in DMFA in the presence of potassium bicarbonate gave 1-allyl-6-methoxyuracil [64].

Uracil and its derivatives can also be allylated by triallyl phosphate with the formation of the $N_{(1)}$ -allyl derivatives [65].

The 1- and 3-allyl-2-thiouracils and their 6-methyl derivatives were synthesized by the rearrangement of 2-allylthiopyrimidin-4(3H)-ones in the presence of palladium [32]. 3-Allyl-6-methyl-2-thiouracil was also obtained by the reaction of ethyl β -aminocrotonate with allyl isocyanate [66].

The authors in [67, 68] erroneously supposed that 1-allyl-2-thiouracils were formed in the reaction of 2-thiouracil and 6-methyl-2-thiouracil with allyl bromide in the presence of hexamethyldisilazane. More recently it was shown that 2-allylthiopyrimidin-4(3H)-ones are in fact formed [35].

Substituted 3-allyl-4-oxo-2-mercapto-3,4-dihydrothieno[2,3-*d*]pyrimidines were obtained by the cyclization of 2-(N-allylthioureido)-3-ethoxycarbonylthiophene [69].



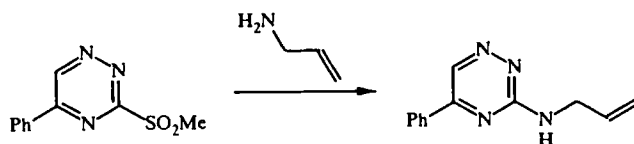
The reaction of 2-benzylthio-4-hydroxypyrimidine with allyl bromide in an alkaline medium leads to the formation of a mixture of 1-allyl- and 3-allyl-2-benzylthio-4-pyrimidinone [51].

2-Allyl-3-pyridazone and its 6-methyl, 6-phenyl, and 6-alkoxycarbonyl derivatives were obtained by the allylation of the sodium salts of 3-pyridazones with allyl bromide in organic solvents [55, 70, 71]. 2-Allyl-6-allyloxy-3-pyridazone was formed during the allylation of 6-hydroxy-3-pyridazone with an excess of allyl bromide [70]. 2-Allyl-6-hydroxy-3-pyridazone was also obtained in the reaction of maleic anhydride with allylhydrazine in the presence of hydrochloric acid [70].

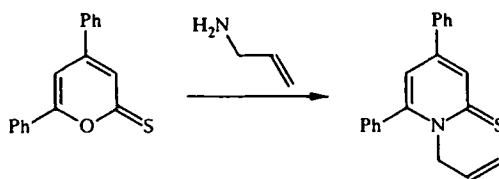
1-Allyl-4-methyl-2-quinolone was synthesized by the allylation of 4-methyl-2-quinolone with allyl bromide in the presence of alkali [72, 73] and by the cyclization of N-allylacetacetanilide under the influence of sulfuric acid [74].

1-Allyl-2-pyridone [75], 1-allyl-2-quinolone [76], and 2-allyl-1-isoquinolone [77, 78] were obtained by the oxidation of pyridine, quinoline, and isoquinoline bromoallyl derivatives with potassium hexacyanoferrate in an alkaline medium. 1-Allyl-2-pyridone was also obtained by the reaction of the sodium salt of 2-pyridone with allyl chloride [46].

3-Allylamino-5-phenyl-1,2,4-triazine was obtained by substitution of the methylsulfonyl group in 3-methylsulfonyl-5-phenyl-1,2,4-triazine by the action of allylamine [44].



Under the influence of allylamine 4,6-diphenyl-2-pyranthione undergoes recyclization by the ANRORC mechanism with the formation of 1-allyl-4,6-diphenyl-2-pyridinethione [79].



The allylation of 2-aminopyridine by allyl halide takes place at the ring nitrogen atom in a neutral medium [80] and at the ring and exocyclic nitrogen atoms in an alkaline medium [81]. The allylation of 8-aminoquinoline takes place at the amino group [59, 82].

10-Allylphenothiazine and 10-allylphenoxazine were obtained by the allylation of phenothiazine and phenoxazine by allyl halide in the presence of a base [83, 84] and in a two-phase system in the presence of phase-transfer catalysts [85, 86].

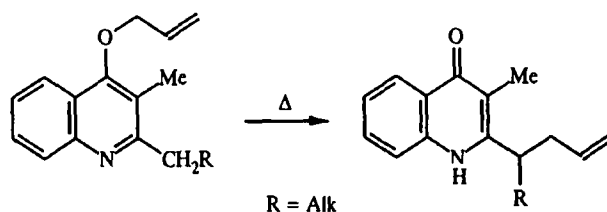
4. PROPERTIES OF THE S-, O-, AND N-ALLYL DERIVATIVES OF AROMATIC AZINES

4.1. Rearrangements

3,3-Sigmatropic rearrangements of allylthiazines were summarized in [87]. Thermal thio-Claisen rearrangements of 3- and 4-allylthioquinolines do not stop at the formation of *o*-allylquinolinethiols, but further cyclization occurs with the formation of thiophene and thiopyran fragments condensed with the quinoline ring [18].

When 8-allyloxyquinoline is heated, a Claisen rearrangement leads to the formation of 7-allyl-8-hydroxyquinoline [57]. On heating, 7-allyloxyquinoline gives 8-allyl-7-hydroxyquinoline [88], and 4-allyloxyquinoline gives a mixture of 3-allyl-4-quinolines and 2,3-dihydrofuro[2,3-*c*]quinolones [18, 49].

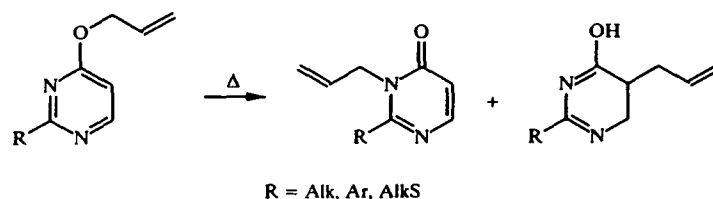
The rearrangement of 4-allyloxy-2-alkyl-3-methylquinolines takes place in a unique manner. The allyl group migrates to the α -methylene group of the alkyl substituent at the *m* position [89].



The thermal rearrangement of 2-allyloxyquinoline gives a high yield of 1-allyl-2-quinoline [48], while the thermal rearrangement of 2-allylthioquinoline gives only 1% of 1-allyl-2-quinolinethione [45].

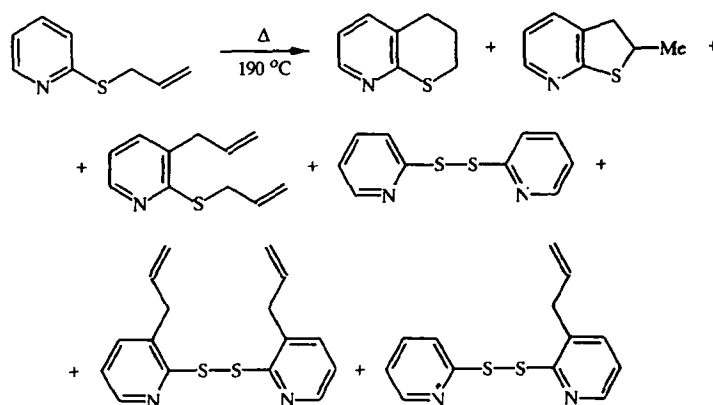
The 3,3-sigmatropic rearrangement of 2-allylthiopyrimidin-4(3H)-ones and 3-allylthio-1,2,4-triazin-5(2H)-ones in the presence of bis(benzonitrile)palladium leads to the formation of products from migration at both nitrogen atoms [32, 90, 91].

The thermal rearrangement of 2-substituted 4-allyloxypyrimidines takes place both at the nitrogen atom and at the carbon atom [51-53].



In the presence of bis(benzonitrile)palladium in THF 2-allylthiopyrimidine undergoes rearrangement with the formation of a complex of 1-allyl-2-pyridinethione with palladium chloride [92].

In [3] it was shown that a mixture of six compounds, including the products of thio-Claisen rearrangement, is formed when 2-allylthiopyridine is heated without a solvent or in tetralin or DMFA at 190°C.



At 420-460°C, 2-allylthiopyridine isomerizes almost quantitatively (85-90%) to 2-(1-propenylthio)pyridine. Thio-Claisen rearrangement is not observed here [42].

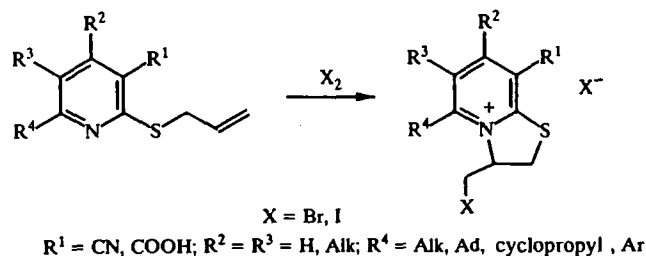
The prototropic rearrangement of 2-allylthiopyridine [1, 2], 2- and 8-allylthioquinolines [14, 15, 93], 1-allyl-2-pyridone [94], and 10-allylphenothiazine and 10-allylphenoxazine [83, 84, 86] under the influence of bases leads to the formation of the 1-propenyl derivatives.

2-Allyloxypyridine isomerizes under the influence of palladium or platinum compounds to 1-allyl-2-pyridone [95, 96], while in dimethylaniline at 250°C it isomerizes to 1-allyl-2-pyridone and 3-vinyl-2-pyridone [47].

4.2. Reaction with Halogens

Allyl compounds containing an electron-donating group react with halogens with intramolecular ring formation [97-101]. Data on the halogenocyclization of the allyl compounds of the heterocyclic series were reviewed in [102].

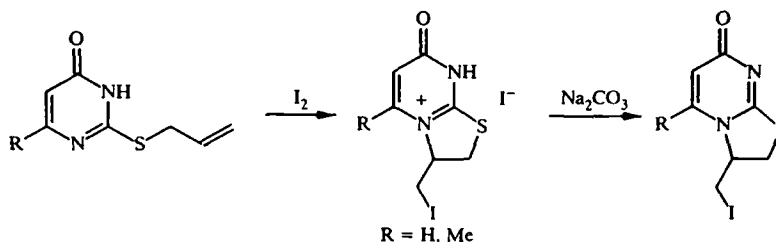
Systematic investigations into the halogenocyclization of various substituted 2-allylthiopyridines were undertaken by a group of authors in [6-12, 103]. They showed that the halogenation reactions take place regioselectively with the formation of thiazolo[3,2-*a*]pyridinium and not thiazino[3,2-*a*]pyridinium salts or the adducts from the addition of the halogens to the allylic double bond.



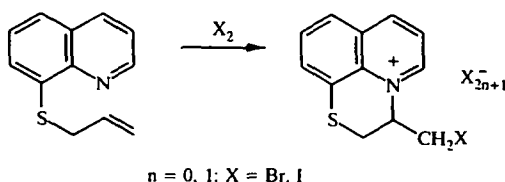
In the opinion of the authors, the high regioselectivity of the halogenocyclization of 2-allylthiopyridines results from the simultaneous action of the electron acceptor (the halogen molecule) and electron donor (the nitrogen atom) on the double bond of the allylic fragment (a synchronous mechanism) [8]. Depending on the reaction conditions, the halogenocyclization of olefins can also take place by an asynchronous mechanism through the formation of ion pairs [100].

2-Allylthiopyridine [22, 104, 105], 2-allylthio-3-hydroxy-6-methylpyridine [4], 2-allylthioquinoline [16, 17, 106], substituted 2-allylthio-5,6,7,8-tetrahydroquinoline [20], 1-allylthioisoquinoline [26], 2-allylthio-4,6-dimethylpyrimidine [30, 31], and 2-allylthio-3-phenylthieno[2,3-*d*]pyrimidin-4-ones [37, 38, 107] react similarly with halogens with the formation of a thiazole ring.

The halogenocyclization of 3-allylthio-1,2,4-triazines takes place with the participation of the $N_{(2)}$ nitrogen atom [44], while the iodocyclization of 2-allylthio-4(3H)-pyrimidines takes place with the participation of the $N_{(1)}$ nitrogen atom [35, 36].



8-Allylthioquinoline reacts with halogens with the formation of thiazino[2,3,4-*i,j*]quinolinium systems [108].

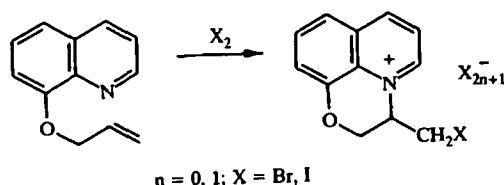


The authors in [109] supposed that the reaction of 2-allyloxypyridine with bromine in benzene led to the formation of 3-bromomethyl-2,3-dihydrooxazolo[3,2-*a*]pyridinium bromide, but the structure was not supported by spectral data.

In a more recent paper [105] it was shown that the halogenocyclization of 2-allyloxypyridine takes place in chloroform and ether with cooling. At room temperature an unstable product from the addition of bromine at the double bond is mainly formed.

The halogenocyclization of substituted 2-allyloxy-3-cyanopyridines [6, 11] and 5,6-disubstituted 2-allyloxy-3-phenyl-4-oxothieno[2,3-*d*]pyrimidines [38] takes place with the formation of an oxazole ring.

8-Allyloxyquinolines react with bromine and iodine with the formation of 3-halogenomethyl-2,3-dihydro-1,4-oxazino[2,3,4-*i,j*]quinolinium halides [59, 110, 111]. Chlorocyclization, which is encountered quite rarely in the literature, was also realized in the case of 8-allyloxy-5,7-dibromoquinoline.

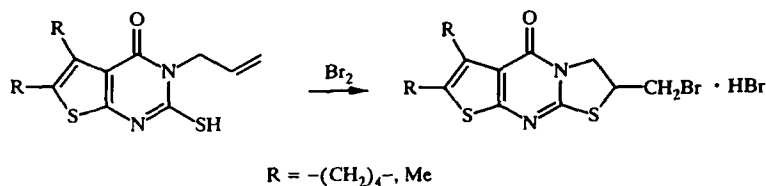


1-Allyl-2-pyridone and its hydrohalides react with bromine and iodine with the formation of 2-halogenomethyl-2,3-dihydrooxazolo[3,2-*a*]pyridinium halides [75, 112, 113].

Similarly, 1-allyl-2-quinolones [72, 73, 76, 114, 115] and 2-allyl-1-isoquinolone [77, 78] react with halogens according to the halogenocyclization scheme. The halogenocyclization of 1-allyl-2-pyridone and 1-allyl-2-quinolone can be realized by the halogen generated *in situ* from the hydrohalic acid and hydrogen peroxide (oxidative halogenocyclization) [72, 75, 76].

The halogenocyclization of 4,6-diphenyl-1-allyl-2-pyridinethione [79] and 1-allyl-2-thiouracil [32] leads to the formation of a thiazole ring. In [67, 68] the authors supposed erroneously that they were studying the reaction of 1-allyl-2-thiouracil with halogens. More recently it was established that they obtained not 1-allyl-2-thiouracil but 2-allylthio-4(3H)-pyrimidinone [35].

Theoretically, the halogenocyclization of 3-allyl-2-mercapto-3,4-dihydrothieno[3,4-*d*]pyrimidin-4-ones can lead to the formation of both thiazole and oxazole rings. In [69] it was shown that bromocyclization in carbon tetrachloride leads to the formation of a thiazole ring.



Imidazo[1,2-*a*]pyridines were obtained by the reaction of 2-allylaminopyridine [98, 109], 1-allyl-2-imino-1,2-dihydropyridine, and its hydrohalides [80] with halogens.

8-(Allyltosylamino)quinoline reacts with iodine with the formation of 3-iodomethyl-1-tosyl-2,3-dihydropyrazino[2,3,4-*i,j*]quinolinium iodide [59].

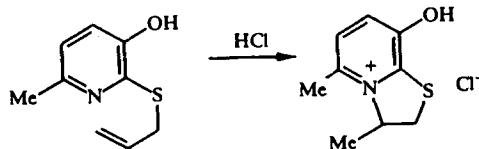
4.3. Other Reactions

8-Allylthioquinoline is oxidized to the sulfoxide [116] and sulfone [25, 41] by hydrogen peroxide. With the salts of metals it forms complexes [116-118], and it is hydrogenated in the presence of Raney nickel to 8-propylthioquinoline [119].

2-Pyridyl allyl sulfoxide was obtained by the oxidation of 2-allylthiopyridine with hydrogen peroxide [3].

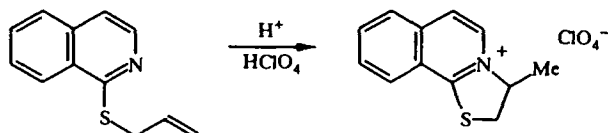
1-(2,3-Epoxypropyl)thymine is formed during the oxidation of 1-allylthymine with perbenzoic acid, and 1-(2,3-dihydroxypropyl)thymine is formed during oxidation with potassium permanganate [120].

2-Allylthio-3-hydroxy-6-methylpyridine reacts with hydrochloric acid in acetic acid with the formation of 3,5-dimethyl-8-hydroxy-2,3-dihydrothiazolo[3,2-*a*]pyridinium chloride [5].



The action of heat on 6-methyl-3-cyano-2-allylthiopyridine with perchloric acid in acetic acid leads only to protonation of the pyridine and the formation of the perchlorate, while heating in the presence of hydrobromic acid leads to hydrolysis of the cyano group [8].

The reaction of 1-allylthioisoquinoline with sulfuric acid and with hydrogen chloride in the presence of perchloric acid leads to the formation of 3-methyl-2,3-dihydrothiazolo[2,3-*a*]isoquinolinium perchlorate [26].



3-Allyl-2-mercapto-6-methyl-3,4-dihydropyrimidin-4-one [66] and 4-allyl- and 2-allyl-5-oxo-1,2,4-triazine-3-thiones undergo intramolecular cyclization in an acidic medium [91].

1-Allyl-2-quinolines [15] and 1-allyluracil [45, 121] react with phosphorus pentasulfide to form 1-allyl-2-quinoline-thiones and 1-allyl-4-thiouracil respectively. The methylation of 1-methyl-4-thiouracil with diazomethane leads to a mixture of 1-allyl-3-methyl-4-thiouracil, 1-allyl-4-methylthiouracil, and 1-allyl-2-methoxy-4-thiouracil. Methylation with methyl iodide leads to the formation of 1-allyl-4-methylthiouracil [45, 121]

Substituted 2-alkylthio-3-allylthieno[2,3-*d*]pyrimidin-4-ones react with tellurium tetrahalides and form complexes that undergo cyclization under the influence of carboxylic acids with the formation of an oxazole ring [122].

8-Allylaminoquinoline reacts with arenediazonium salts with the formation of azo dyes [82].

Data on the polymerization of 3-allylcyanurates and allylpyridazones are summarized in [55]. The radiation polymerization of allylpyridazones takes place with a low yield and is accelerated by the addition of phosphoric acid [71].

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