

OXIMES OF FIVE-MEMBERED HETEROCYCLIC COMPOUNDS WITH TWO HETEROATOMS. 1. SYNTHESIS AND STRUCTURE (REVIEW)

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Data on the production and structure of oxazole, thiazole, and imidazole aldoximes, ketoximes, and amidoximes and their derivatives are reviewed.

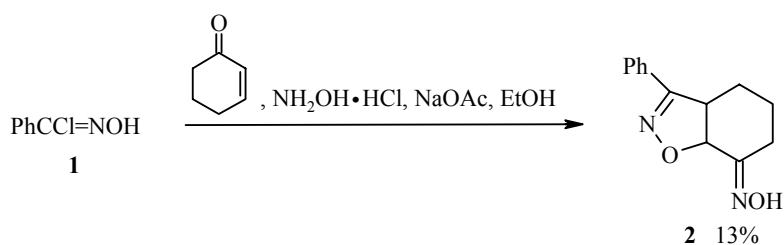
Keywords: imidazole, oxazole, oxime, pyrazoles, thiazole

The oximes of five-membered heterocyclic compounds with two heteroatoms are widely used as intermediates in fine organic synthesis. The principal methods for the production of oxazole, thiazole, and imidazole aldoximes, ketoximes, amidoximes, and their derivatives are summarized in this review. The principal methods of investigation of the structure of the oximes of five-membered heterocyclic compounds with two heteroatoms in relation to their isomerism are examined briefly. The characteristics of the reactions and biological activity of the oximes of five-membered heterocyclic compounds with two heteroatoms will be presented in the second part of the review.

1. SYNTHESIS

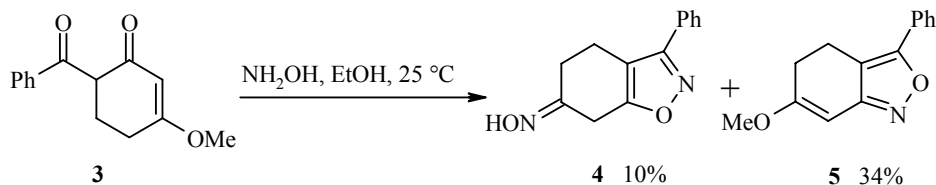
1.1. Synthesis of Oxazole, Isoxazole, Benzoxazole, and Benzisoxazole Aldoximes and Ketoximes

The classical method for the synthesis of oxazole, isoxazole [1], isoxazoline [2], benzoxazole [3], and benzisoxazole [4] aldoximes and ketoximes is based on reaction of the aldehyde or ketone with hydroxylamine hydrochloride in the NaOH/H₂O/EtOH [5] or NaOAc/MeOH/H₂O [6] system. The isoxazole **2** is formed from the oxime **1** and 2-cyclohexenone in the NH₂OH·HCl/NaOAc/EtOH system under similar conditions [7].



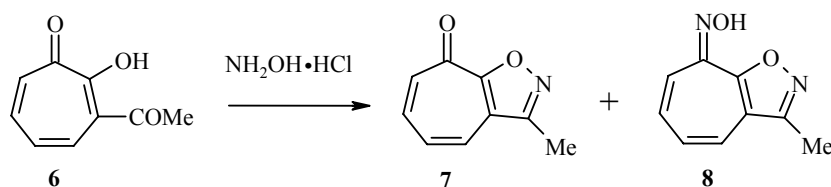
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The synthesis of bactericidal tetrahydrobenzoxazole oximes was described in [8-10]. For example, the reaction of 6-benzoyl-3-methoxy-2-cyclohexen-1-one (**3**) with hydroxylamine led to a mixture of the oxime **4** and the ether **5** [10].

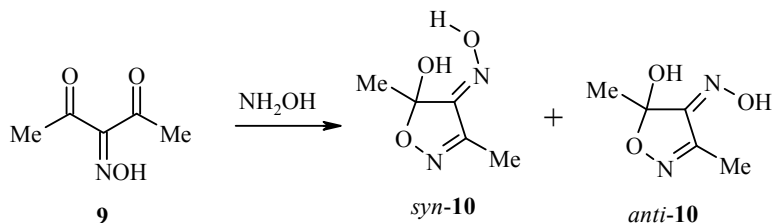


Reaction of the anion of glutacetaldehyde with NH_2OH gives (4,5-dihydroisoxazol-5-yl)acetaldehyde oxime [11].

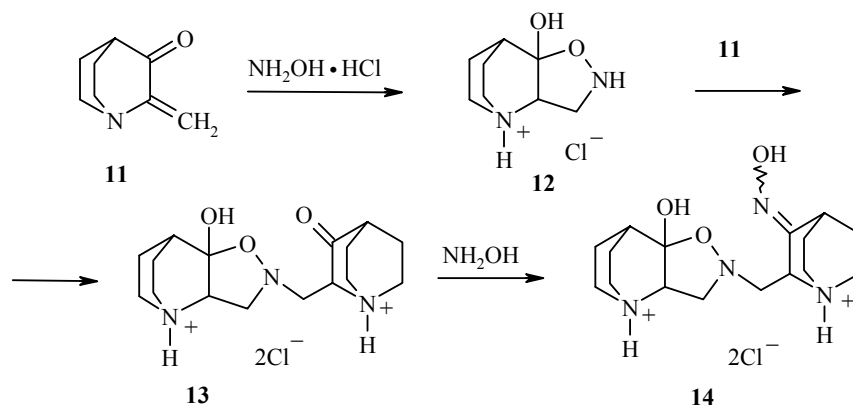
The reaction of 3-acetyltropolone (**6**) and $\text{NH}_2\text{OH}\cdot\text{HCl}/\text{MeOH}$ leads to a mixture of the ketone **7** (82%) and its oxime **8** (11%) [12].



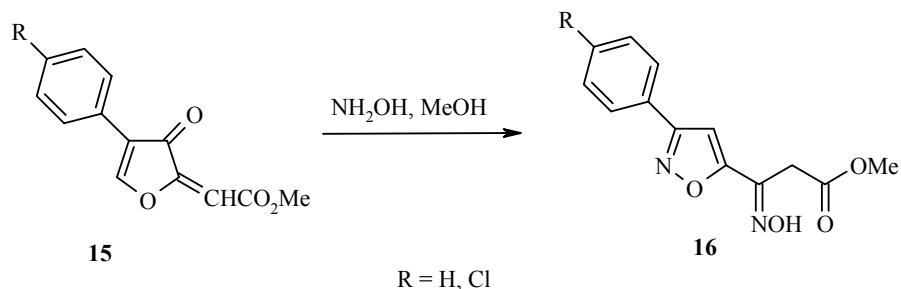
Acetyl(hydroxyimino)acetone (**9**) reacts readily with one equivalent of hydroxylamine, forming a mixture of the isomers of the oxime **10** with a yield of 80% [13]. The structure of the compound *syn*-**10** was confirmed by X-ray crystallographic analysis.



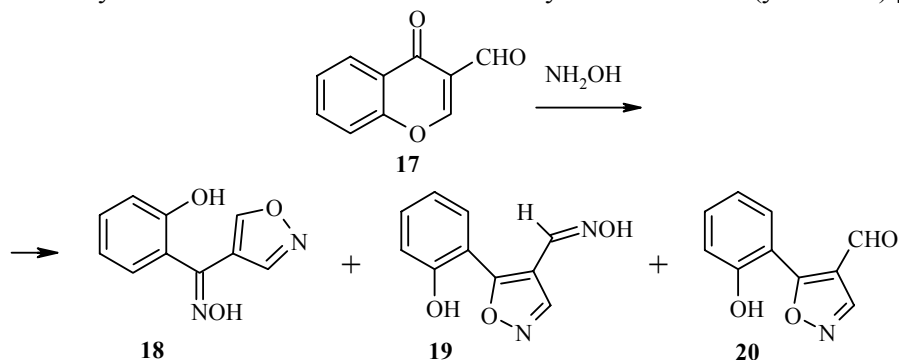
The reaction of 2-methylene-3-oxoquinuclidine (**11**) with $\text{NH}_2\text{OH}\cdot\text{HCl}$ gives the oxime **14** with a yield of 83% [14]. Compound **14** is formed through 4a-hydroxyisoxazolidino[4,5-*b*]quinuclidine (**12**), which then reacts with the initial ketone **11** to form the intermediate product **13**. Oximation of the latter leads to the oxime **14**.



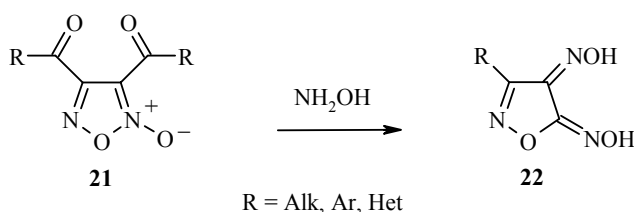
In the presence of hydroxylamine furanones [15, 16] and 4-acetylidene-2,6-dimethylpyran [17] give derivatives of isoxazole oximes. For example, the reaction of compound **15** with NH_2OH in methanol leads to the oximes **16** [15].



The reaction of substituted 3-formylchromones with hydroxylamine hydrochloride was studied by several authors [18-20]. It was shown that the reaction of the aldehyde **17** and NH_2OH under various conditions (pH and the reagent ratio) gave a mixture of products, mostly compounds **18-20**. For example, boiling of compound **17** with 1.5 eq. of hydroxylamine hydrochloride in ethanol leads selectively to the oxime **18** (yield 60%) [19].

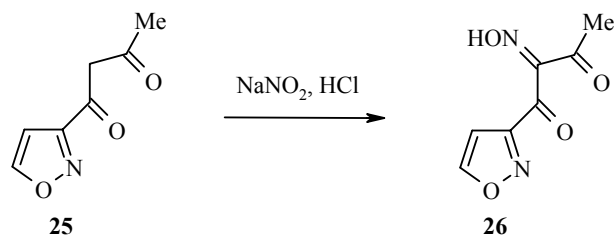
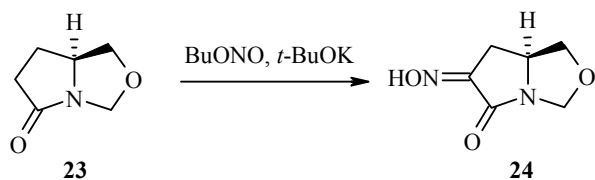


3,4-Diacylfuroxans **21** and hydroxylamine in pyridine rearrange to the 3-substituted 4,5-bis(hydroxyimino)-4,5-dihydroisoxazoles **22** as the main products [21-23]. The structure of compound **22** (R = *t*-Bu) was confirmed by X-ray crystallographic analysis.

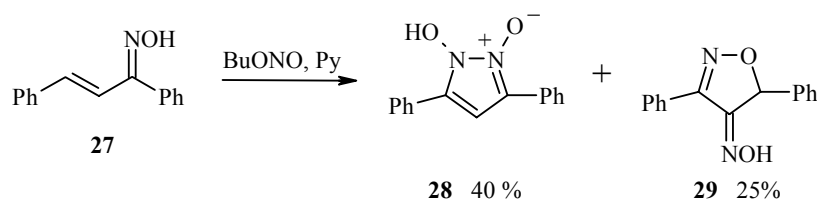


The rearrangement of furoxan-4-nitrolic acid in water leads to the dioxime of 3-nitroisoxazole-4,5-dione [24].

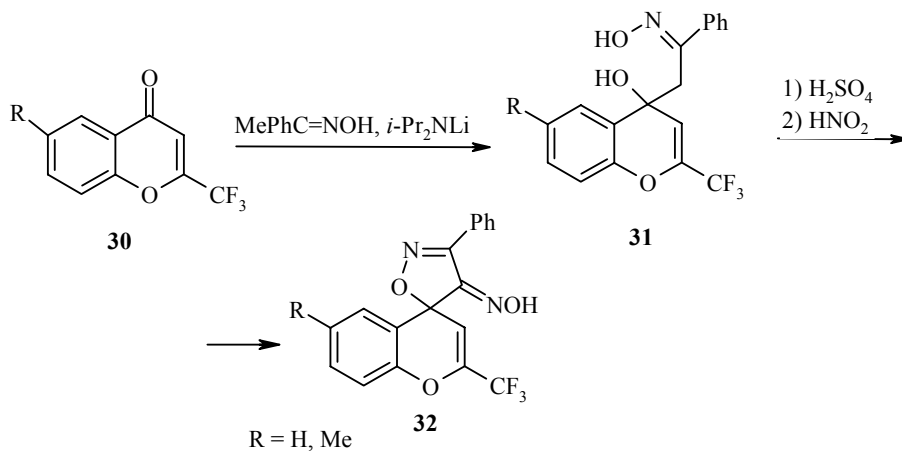
A series of methods for the synthesis of oxazole and isoxazole oximes are based on nitrosation of the alkyl side chain in the corresponding alkyl derivatives. The $\text{MeONO}/\text{NaOH}/\text{EtOH}$ [25, 26], $\text{BuONO}/\text{NaNH}_2/\text{NH}_3$ [27], alkyl nitrite/ NaNH_2 or KNH_2 [28], amyl nitrite/ $\text{NH}_3/\text{NaNH}_2$ [29], $\text{BuNON}/\text{NaOH}/\text{EtOH}$ [30], and $\text{EtONO}/\text{H}_2\text{O}/\text{HCl}$ [31] systems were used as nitrosating systems. In the $\text{BuONO}/\text{tert-BuOK}/\text{THF}$ system the bicyclic derivative of oxazole **23** gives the oxime **24** with a yield of 58% [32]. The reaction of the isoxazole ketone **25** with NaNO_2 in HCl leads to the formation of compound **26** as the only product [33].



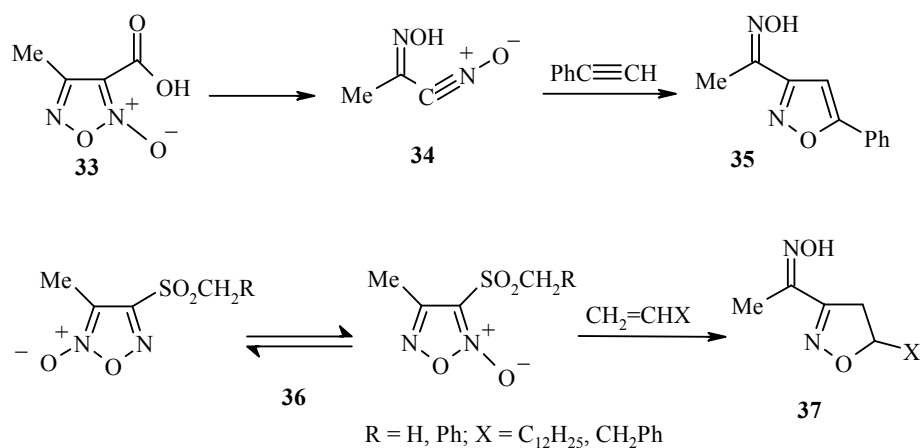
The reaction of the chalcone oxime **27** and butyl nitrite in the presence of metal ions [Cu(II) or Co(II)] leads to a mixture of 1-hydroxypyrazole 2-oxide **28** and the oxime **29** [34].



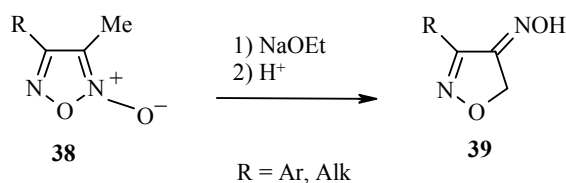
The oximes of β -hydroxy ketones **31** were obtained as a result of the addition of acetophenone oxime to 2-trifluoromethylchromones **30** in the presence of lithium diisopropylamide. In an acidic medium the compounds underwent intramolecular cyclization with the formation of spiroisoxazolines. The reaction of the nitrosyl cation with isoxazoline leads to the oximes **32** with yields of 59-78% [35].



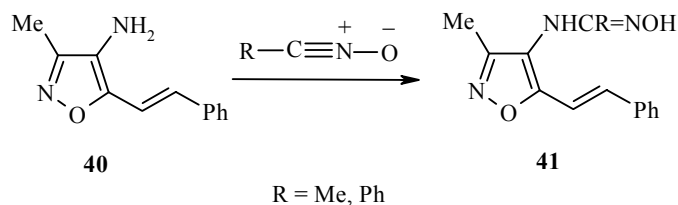
The nitrile oxide **34**, generated from the furoxan **33**, reacts with phenylacetylene and gives the isoxazole oxime **35** [36]. In the reaction of 4-alkylsulfonyl-3-methylfuroxans **36** with alkenes the isoxazoline oximes **37** are formed with yields of 9-14% [37].



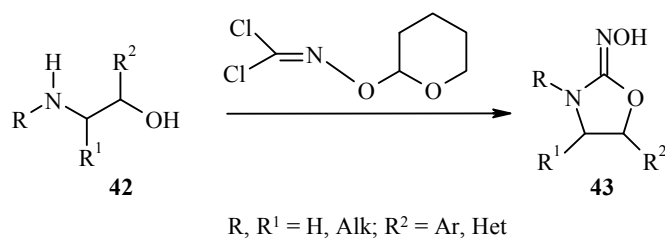
The reaction of 3-methyl-substituted furoxans **38** with sodium ethoxide followed by hydrolysis leads to the oximes of 2-isoxazolin-4-one **39** [38].



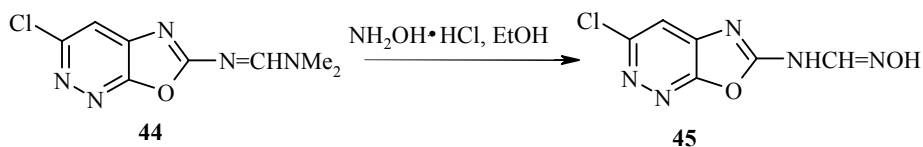
The reaction of acetonitrile or benzonitrile oxides with 4-amino-3-methyl-5-styrylisoxazole **40** leads to the formation of isoxazole oximes **41** instead of the expected cycloaddition products – isoxazolines [39].



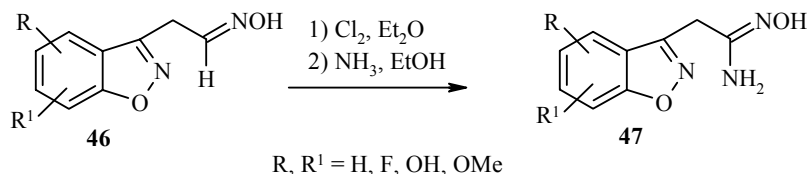
The cyclization of aminoethanols **42** in the presence of the O-tetrahydropyranyl ether of phosgene oxime gave the oximes of oxazolidin-2-ones **43** [40]. Compounds **43** were investigated as antidepressants.



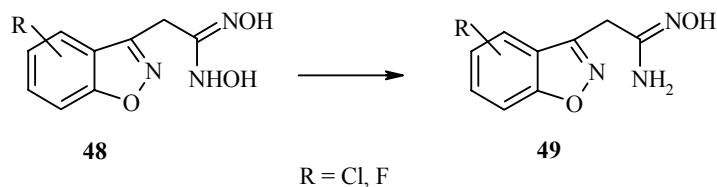
The reaction of the dimethylaminomethyleneamino derivative of oxazole **44** with hydroxylamine hydrochloride in ethanol leads to the oxime **45** with a yield of 86% [41].



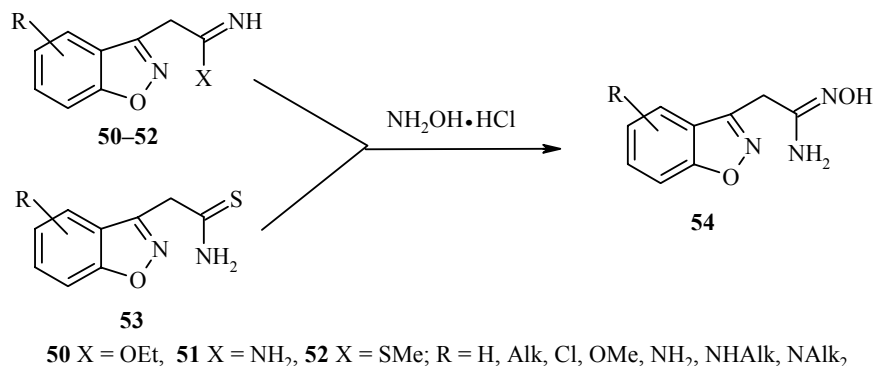
The classical method for the synthesis of benzisoxazole amidoximes is based on the reaction of the nitriles of benzoic acids with hydroxylamine hydrochloride in the presence of sodium or Na_2CO_3 [42], $\text{Na}_2\text{CO}_3/\text{H}_2\text{O}/\text{EtOH}$ [43], or $\text{Na}_2\text{CO}_3/\text{EtOH}$ [44, 45]. Amidoximes of the 1,2-benzisoxazole series **47** were obtained by a two-stage reaction from the corresponding aldoximes **46** [46].



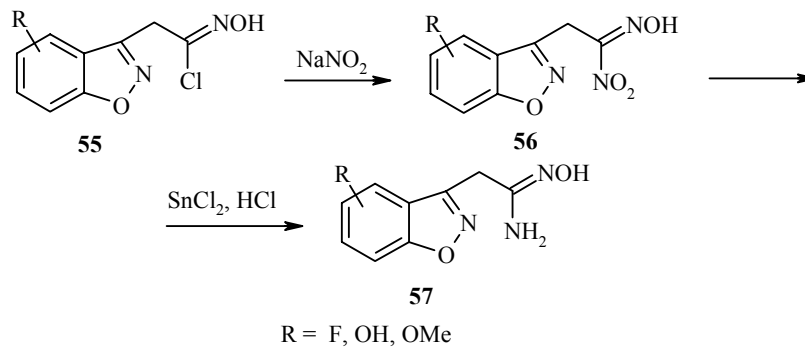
Reduction of the oximes of hydroxamic acids **48** gave the amidoximes **49** as the only products [47].



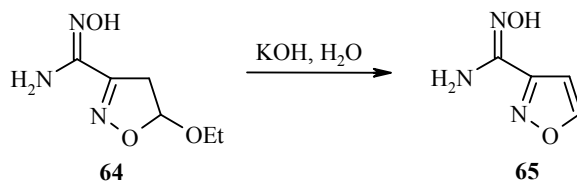
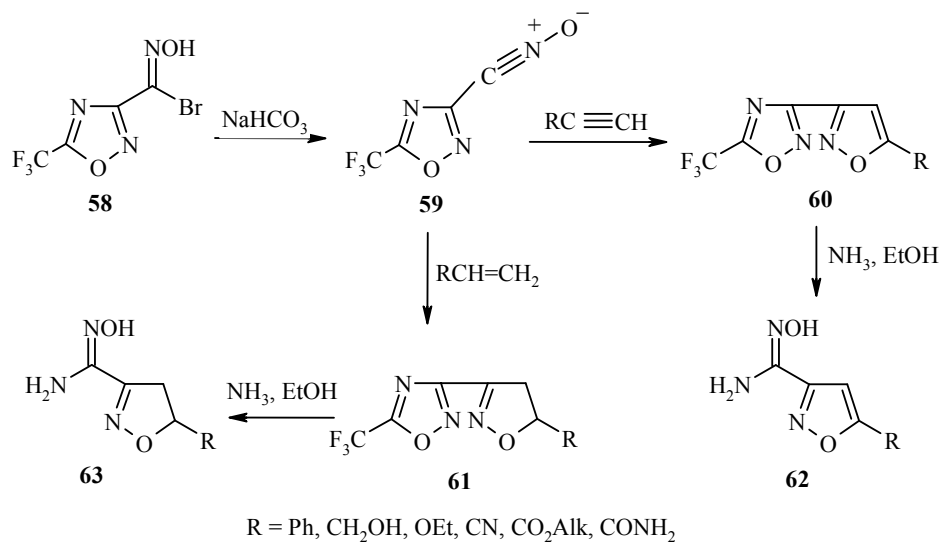
The amidoximes of 1,2-benzisoxazole **54** were obtained from the imines **50-52** or the thioamide **53** and $\text{NH}_2\text{OH}\cdot\text{HCl}$ in the presence of Na or NaCO_3 [42, 48].



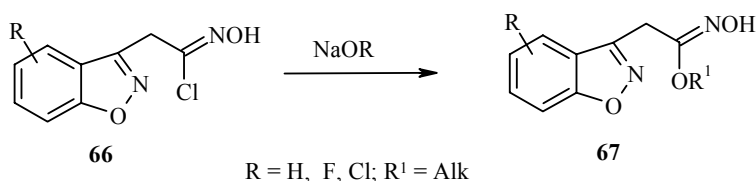
Reduction of the nitro derivatives of the oximes **56**, produced in the reaction of the chlorides of hydroxamic acids **55** and NaNO_2 , in the $\text{SnCl}_2/\text{HCl}/\text{H}_2\text{O}$ system also leads to the formation of amidoximes **57** [49].



It was shown recently that the nitrile oxide **59**, generated from the hydroxamic acid bromide **58**, and alkynes or alkenes form the corresponding 3-(3-isoxazolyl)-5-trifluoromethyl-1,2,4-oxadiazoles **60** or 4,5-dihydroisoxazoles **61**. The reaction of compounds **60** or **61** and ammonia leads to the amidoximes of isoxazole **62** and 4,5-dihydroisoxazole **63** respectively with yields of up to 82%. Alkaline hydrolysis of the ether **64** leads to the formation of the isoxazole amidoxime **65** with a yield of 85% [50].

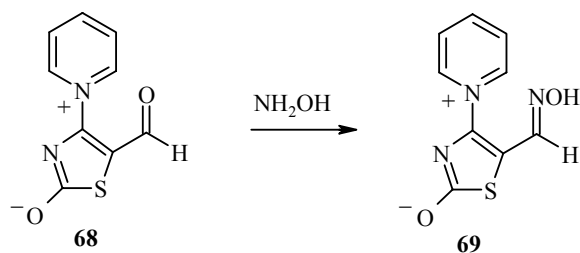


The esters of hydroxamic acids **67** were obtained in the reaction of hydroxamic acid chlorides **66** and alkali-metal alkoxides [51].



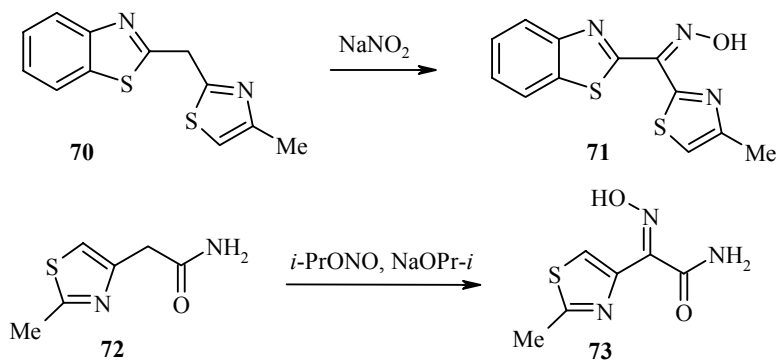
1.2. Synthesis of the Oximes of Thiazole, Isothiazole, Benzothiazole, and Benzoisothiazole Derivatives

The aldoximes and ketoximes of thiazoles [52], isothiazoles, benzothiazoles, and benzoisothiazoles are usually obtained by reaction of the aldehyde or ketone with hydroxylamine hydrochloride in the NaOAc/EtOH [53], pyridine/EtOH/H₂O [54], NaOEt [55], or NaHCO₃/H₂O [56] systems. The synthesis of the salts of benzothiazole and benzimidazole oximes was described in [57]. The aldoxime **69** is formed selectively in the reaction of the betaine **68** with hydroxylamine [58].

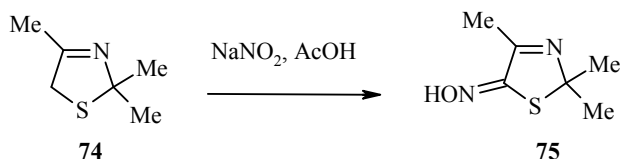


2-Acetylthiazole oxime was obtained as a side product in the reaction of 2-thiazolylacrylic acid with hydroxylamine [59].

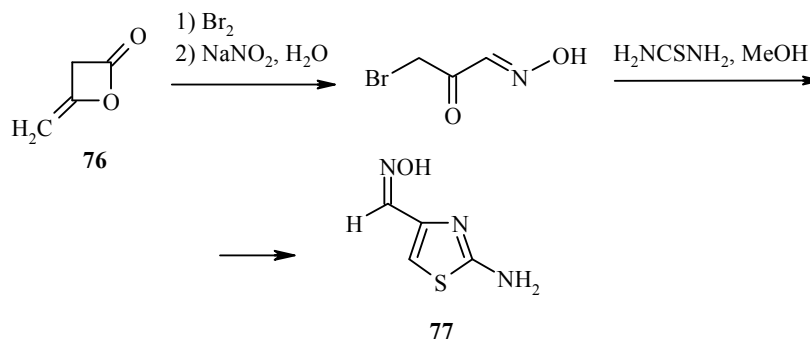
Thiazole and benzothiazole oximes were obtained as a result of nitrosation of the alkyl side chain in the corresponding alkyl derivatives. Thus, in the reaction of 2-benzothiazolyl(4-methyl-2-thiazolyl)methane (**70**) with NaNO_2 the oxime **71** is formed with a yield of 89% [60]. In the *i*-PrONO/ NaOPr-*i* system 2-methyl-4-thiazolylacetamide (**72**) is transformed into the oxime **73**. The structure of compound **73** was confirmed by X-ray crystallographic analysis [61].



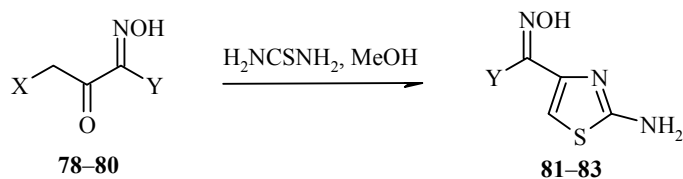
The reaction of 2,2,4-trimethyl-3-thiazoline (**74**) with sodium nitrite in acetic acid leads to the thiazoline oxime **75** [62].



2-Amino-4-formylthiazole oxime **77** was obtained from the diketene **76** in a three-stage synthesis. The reaction of compound **76** with bromine and then with sodium nitrite gave 3-bromo-2-oxopropionic aldehyde oxime, which readily underwent cyclization in reaction with thiourea, forming the oxime **77** with a yield of 39% [63].

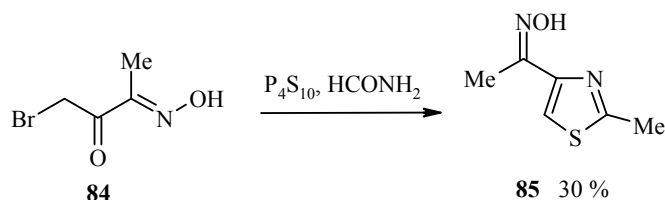


The nitrile [64] and ether [65-71] derivatives of thiazole oximes have been widely investigated as intermediates for the synthesis of cephalosporin antibiotics. For example, the reaction of the oximes **78** [64], **79** [68], and **80** [72] with thiourea in methanol or ethanol gives the thiazole oximes **81-83** respectively.

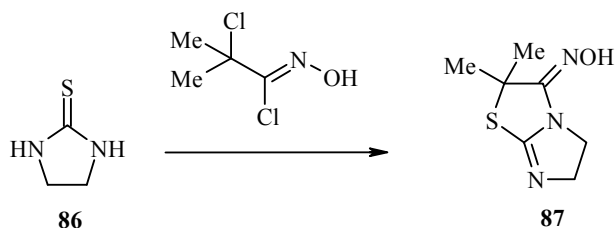


78, 81 Y = CN; **79, 82** Y = CO₂Et; **80, 83** Y = ArCO; X = Cl, Br

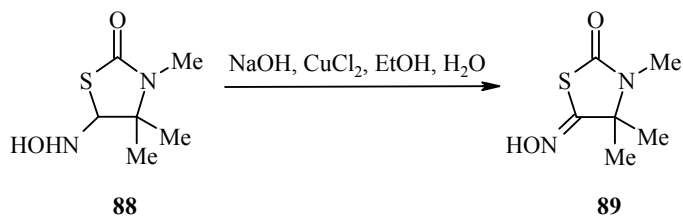
4-Acetylthiazole oxime **85** was obtained in the reaction of the oxime **84** with P₄S₁₀ and formamide [73].



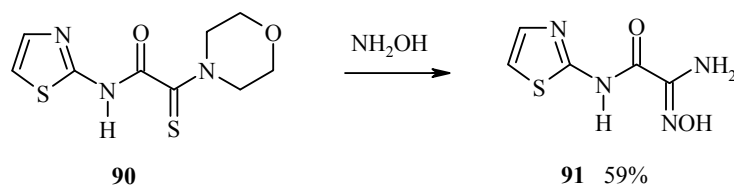
2,2-Dimethyl-5,6-dihydroimidazo[2,1-*b*]thiazole-3-one oxime (**87**) can be obtained by the reaction of 2-imidazolidinethione (**86**) with 2-methyl-1,2-dichloropropionic aldehyde oxime [74].



The transformations of 4- and 5-(hydroxyamino)thiazolidine-2-ones in the presence of alkali was studied in detail in [64]. For example, in the NaOH/CuCl₂/H₂O/EtOH system the thiazolone **88** gives the oxime **89** with a yield of 60%.

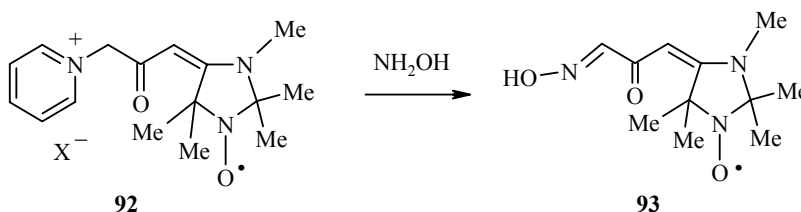


Thiazole amidoximes were obtained in the reaction of nitriles with NH₂OH·HCl in ethanol in the presence of NaHCO₃ [75] or Na₂CO₃ [76]. The thiazole carbamoylamidoxime **91** was synthesized successfully from the morpholine derivative **90** and hydroxylamine in pyridine [77].

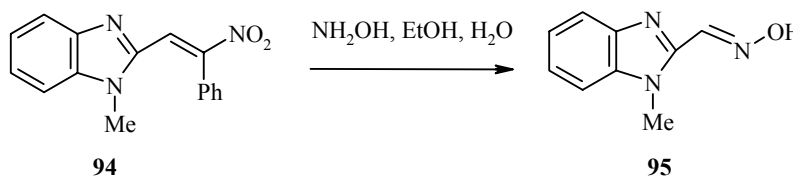


1.3. Synthesis of the Oximes of Imidazole, Pyrazole, Benzimidazole, and Benzindazole Derivatives

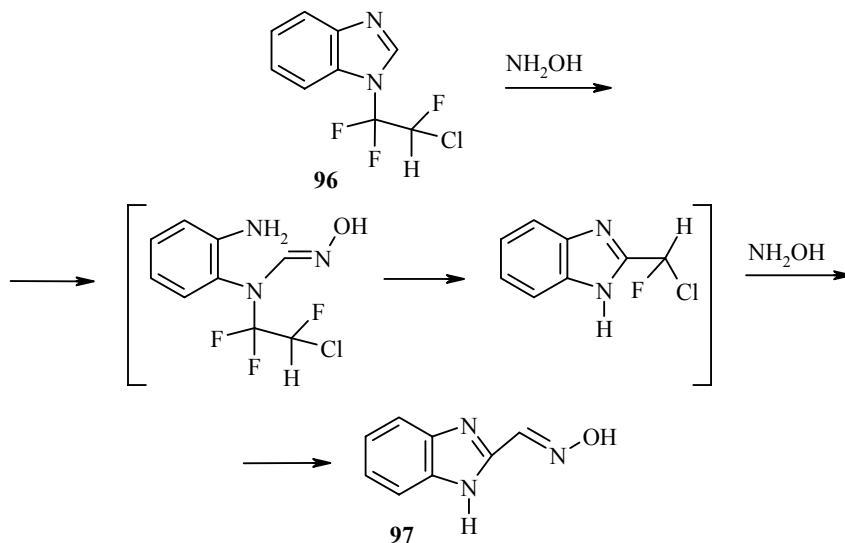
The classical method for the synthesis of imidazole [78-80] and pyrazole [81, 82] derivatives of aldoximes and ketoximes is based on the reaction of the aldehyde or ketone with hydroxylamine hydrochloride in the NaOAc/MeOH [83], $\text{NaOAc}/\text{H}_2\text{O}/\text{MeOH}$ [84], MeOH [85], $\text{EtOH}/\text{pyridine}$ [86], or $\text{NaHCO}_3/\text{EtOH}$ [87] systems. In a few papers the synthesis of aldoximes and ketoximes imidazolium salts was described [87, 88]. It was established that the reaction of the nitron **92** with hydroxylamine leads to the oxime **93** with a yield of 30% [89].



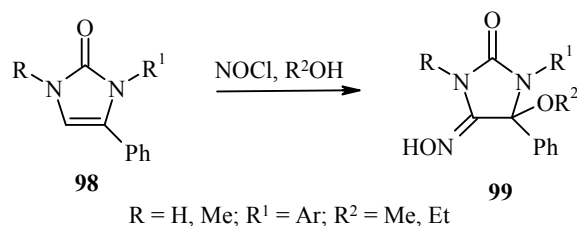
The only product in the reaction of 1-methyl-2-(β -nitro- β -phenylvinyl)benzimidazole (**94**) with hydroxylamine in aqueous alcohol is the benzimidazole aldoxime **95** [90].



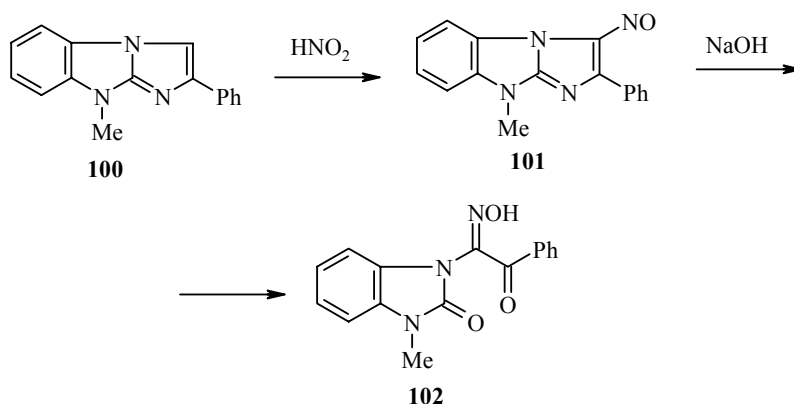
The benzimidazole aldoxime **97** was obtained in the reaction of N-(2-chloro-1,1,2-trifluoroethyl)benzimidazole (**96**) with hydroxylamine in pyridine [91, 92].



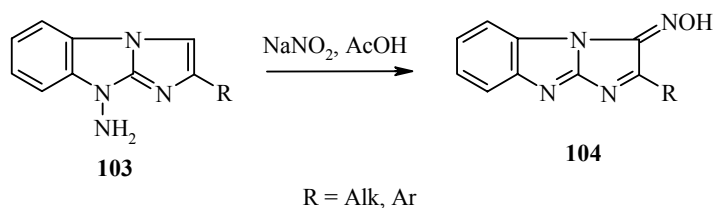
A series of methods for the synthesis of imidazole and pyrazole oximes are based on nitrosation of the alkyl side chain in the corresponding alkyl derivatives. The isoamyl nitrite/AcOH [93, 94], $\text{NaNO}_2/\text{AcOH}/\text{HCl}$ [95], amyl nitrite/EtONa [96], and isoamyl nitrite/NaOMe/EtOH [97] systems were used as nitrosation agents. In many cases such syntheses are accompanied by other transformations. In many cases such synthesis is accompanied by other transformations. Thus, the reactions of 1,5-diarylimidazolin-2-ones **98** with nitrosyl in alcohols lead to 5-alkoxy-4-hydroxyiminoimidazolidin-2-ones **99** with yields of 73-85% [98].



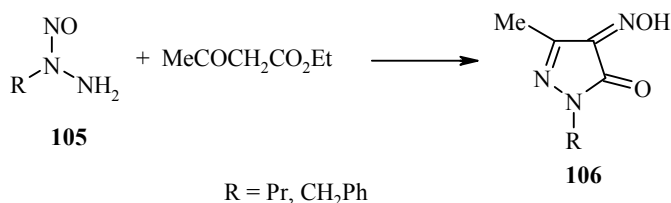
In the $\text{NaNO}_2/\text{AcOH}$ system the imidazo[1,2-*a*]benzimidazole **100** gives the nitroso derivative **101**, the treatment of which with an alcohol solution of alkali leads to the oxime **102** with a yield of 47% [99].



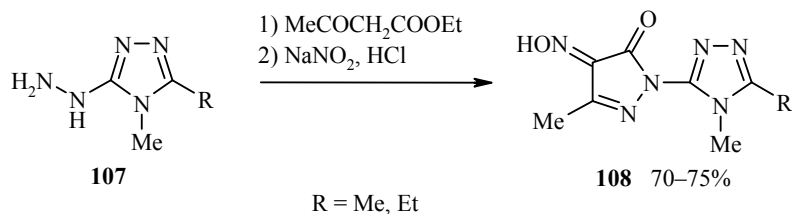
However, in the $\text{NaNO}_2/\text{AcOH}$ system the imidazo[1,2-*a*]benzimidazoles **103** form 3-hydroxyimino derivatives of imidazobenzimidazoles **104** with yields of up to 85% [100].



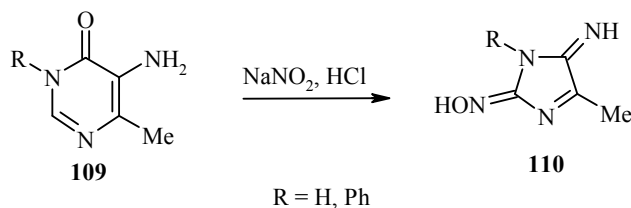
The reaction of the nitrosohydrazines and ethyl acetoacetate leads to pyrazole oximes with yields of up to 41% [101].



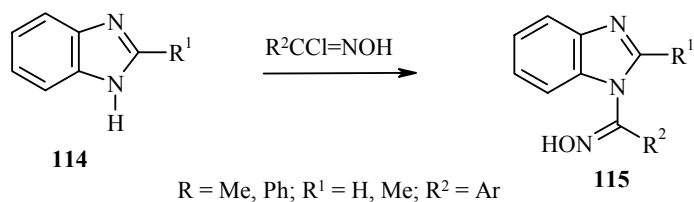
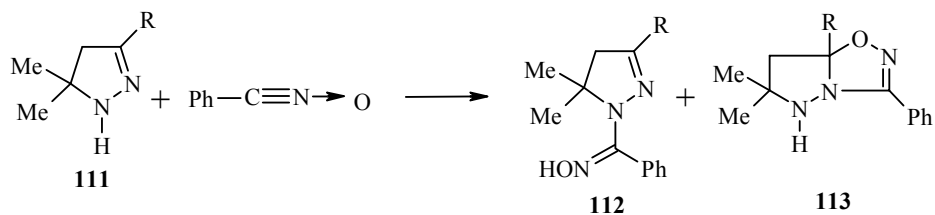
The pyrazolone oximes **108** were prepared successfully in a two-stage synthesis from the corresponding hydrazines **107** [102].



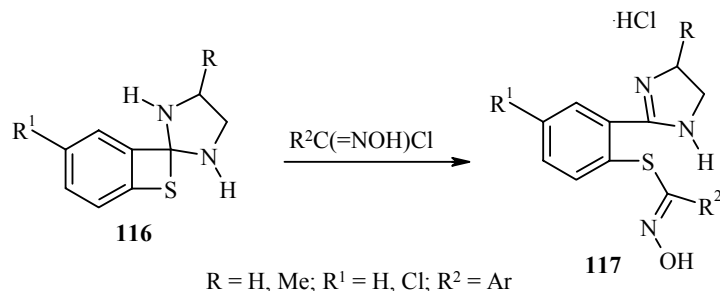
The transformation of 5-amino-4(3H)-pyrimidones **109** in the NaNO_2/HCl system leads to the imidazole oximes **110** with yields of up to 97% [103].



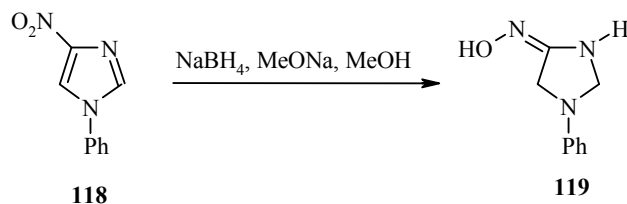
In the literature there are also methods for the synthesis of the oximes of pyrazole [104-107], imidazole [104, 105, 108], and benzimidazole [109-111] derivatives by the reaction of the respective heterocycles with the halogen derivatives (or sulfates) of oximes or with nitrile oxides. For example, 5,5-dimethyl-4,5-dihydropyrazoles **111** and benzonitrile oxide give a mixture of the oximes **112** and the pyrazolooxadiazoles **113** [106]. The benzimidazoles **114** and hydroxamic acid chlorides form the benzimidazole oximes **115** with yields of 70-96% [109].



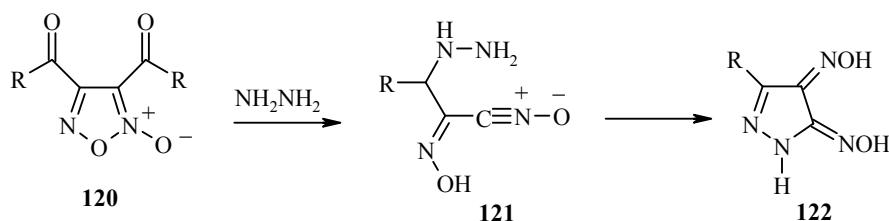
Spirobenzothietanes **116** and hydroximoyl chlorides give the oximes **117** with yields of up to 98% [112].



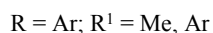
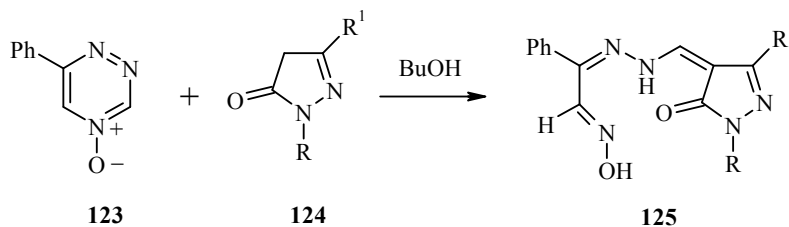
Nitroimidazoles are reduced to the corresponding oximes by lithium or sodium borohydrides [113] or by irradiation with γ -rays in the presence of sodium formate or 2-propanol [114]. For example, in the $\text{NaBH}_4/\text{MeONa}/\text{MeOH}$ system 4-nitro-1-phenylimidazole (**118**) gives the oxime **119** with a yield of 94%.



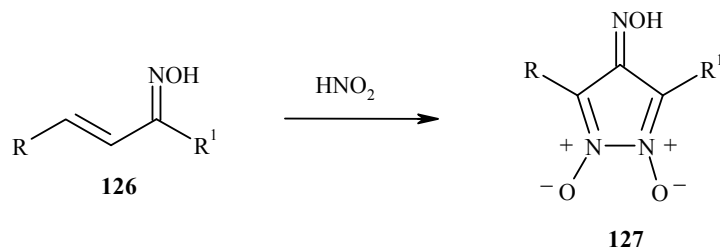
The reaction of 3,4-diacylfuroxans **120** with hydrazine takes place through the intermediates **121** [22, 115] and gives good yields of pyrazole dioximes **122**.



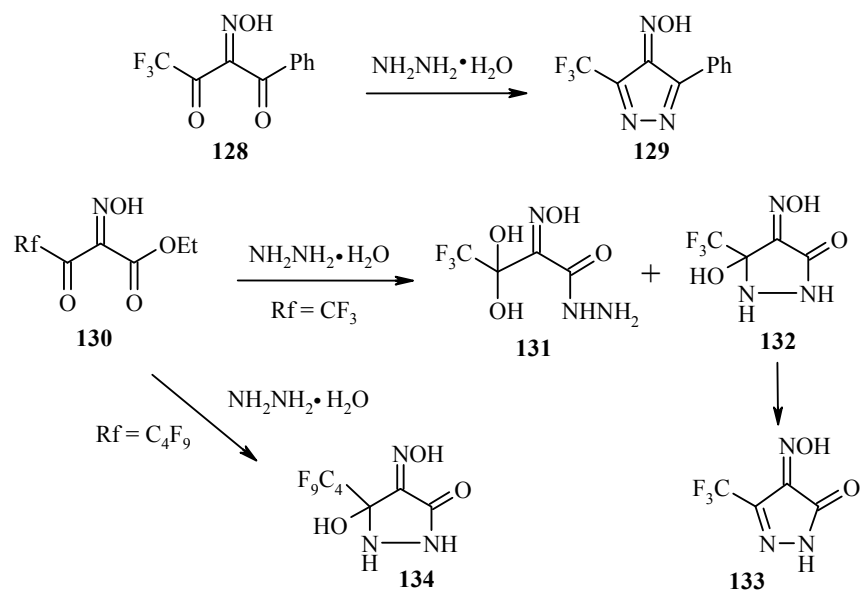
The reaction of 6-phenyl-1,2,4-triazine 4-oxide (**123**) with pyrazolones **124** gives the oximes **125** with yields of 8-35% [116].



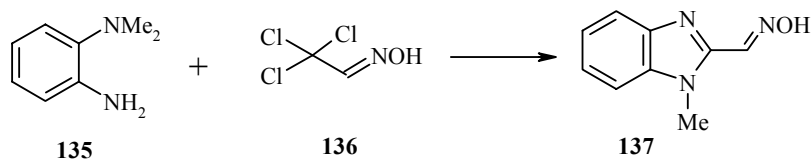
A number of methods for the synthesis of pyrazole and imidazole aldoximes are based on cyclization. By the cyclization of the oximes of α,β -unsaturated ketones **126** in the presence of HNO_2 it is possible to obtain the pyrazole oximes **127** selectively [117].



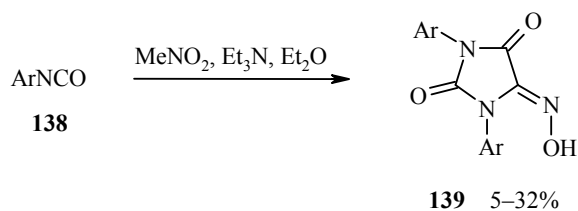
In reaction with hydrazine the fluorine-containing 1,3-diketone oxime **128** gives the oxime **129**. The hydroxyimino-substituted 1,3-oxo esters **130** and NH_2NH_2 form the oximes **131-134**, depending on the substrate [118].



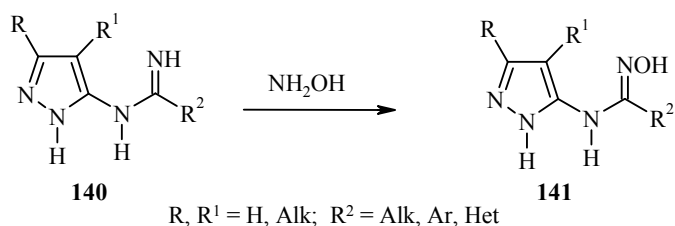
One of the methods for the synthesis of benzimidazole oximes is based on the cyclization of *o*-phenylenediamines with chloral oxime [119-122]. For example, the diamine **135** and the oxime **136** form 1-methyl-2-formylbenzimidazole (**137**) with a yield of 80%.



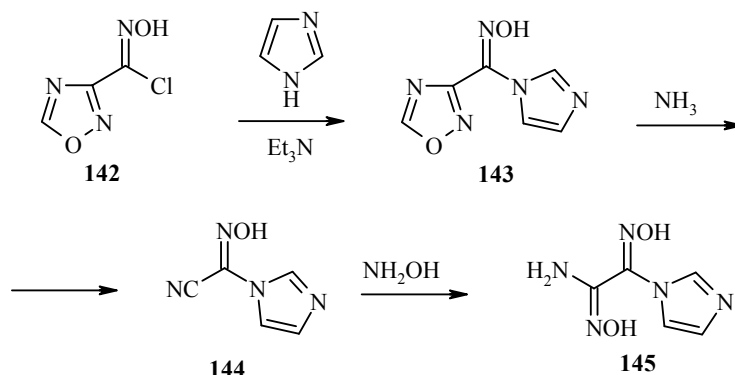
1,3-Diaryl-5-(hydroxyimino)imidazolidine-2,4-diones **139** were obtained by the cyclization of aryl isocyanates **138** in the nitromethane/ Et_3N / Et_2O system [123].



Imidazole and pyrazole amidoximes were synthesized from the respective nitriles and $\text{NH}_2\text{OH} \cdot \text{HCl}$ in ethanol in the presence of $\text{NaOH}/\text{MeOH}/\text{H}_2\text{O}$ [124, 125], EtOH [126], or $\text{Et}_3\text{N}/\text{dioxane}$ [127]. The imine derivatives of pyrazole and hydroxylamine also form amidoximes [128-131]. For example, the reaction of amidines **140** and NH_2OH leads to the oximes **141** [131].



The N-substituted amidoxime **143** was obtained from 1,2,4-oxadiazole-3-carbohydroximoyl chloride (**142**) and imidazole. Compound **143** and ammonia form cyanamide oxime **144** (yield 64%). The reaction of the oxime **144** with hydroxylamine leads to 1-amino-2-(1-imidazolyl)glyoxime (**145**) with a yield of 74% [132].



2. STRUCTURE

One of the most reliable methods for determining the structure of the isomeric oximes of five-membered heterocyclic compounds with two heteroatoms is NMR. The ^1H and ^{13}C NMR spectra of the oximes of dihydroisoxazole [133, 134], thiazole and benzothiazole [135, 136], pyrazole [137-140], imidazole [127, 141-144], and benzimidazole [145, 146] have been investigated in greatest detail. The rule according to which the H_0 protons in the *E*-isomers are deshielded in relation to H_0 in the *Z*-isomers was used during analysis of the spectra of pyrazole and imidazole aldoximes ($\text{RCH}_0=\text{NOH}$) [137].

Reaction of the same type between the oximes and metals was also used to obtain evidence for their structure. The possibility of ring formation during the formation of salts of pyrazole oximes with various metals was discussed from the theoretical point of view [147] and the formation of complexes of thiazole and benzothiazole oximes with various metals [Fe(II), Pd(II), Cu(II), Co(II), Au(III), and others] was examined [147-150]. The imidazole [151, 152] and benzimidazole [153, 154] oximes are used as analytical reagents in the determination of metals.

The structure of *Z*- α -(3-chlorooxazol-5-yl)- α -methoxyiminoacetic acid was confirmed by X-ray crystallographic analysis [155]. The characteristic bond lengths and angles of the oxime group in this compound were determined. (The length of the $\text{C}=\text{N}$ double bond was 1.377 Å, which is typical of this type of compound.)

The structures of the oximes of 1-(4-fluorophenyl)-2-[5-(4-fluorophenyl)-4,5-dihydro-3-isoxazolyl]-ethanone [134], 1,2-benzisothiazol-3-yl methyl ketone [156], and 4-benzoyl-5-methyl-2-phenyl-1,2-dihydro-3H-pyrazol-3-one [157], 1-(4-chlorophenyl)-4-phenyl-2,5-dihydro-1H-imidazol-2-ylphenylmethanone oxime 3-oxide [158], 2-(imidazol-1-yl)-1-(2-naphthyl)-ethanone O-(2-propene)oxime hydrochloride [159], and 6-{[(hydroxyimino)phenyl]methyl}-1-[(1-methylethyl)sulfonyl]-1H-benzimidazole-2-amine [145] were also confirmed by X-ray crystallographic analysis.

The structure of the imidazole oxime was also confirmed by the data from investigations of the kinetics of the $\text{S}_{\text{N}}\text{Ar}$ reaction of the oximate anion with 2,4-dinitrofluorobenzene [160]. The rate constants of the dissociation of benzimidazole oximes were determined [161, 162]. The parameters of the acid-base and tautomeric equilibrium in the *anti* isomers of benzimidazole, imidazole, and pyrazole aldoximes in aqueous solutions were calculated by the potentiometric method [163]. The acid-base characteristics of 5-substituted benzimidazole oximes were studied in detail [164], and the O-nucleophilicity of imidazole amidoxime was investigated [165].

UV [143] and IR [142,144, 166] spectroscopy were used to study the structure of imidazole oximes. The structure of thiazole oximes was also confirmed by data from the UV and IR spectra[167], and the tautomers of 6-hydroxy-7-nitrosobenzothiazole were investigated in the crystalline form and in solutions. It was shown that this compound exists simultaneously in the quinone oxime and nitrosophenol tautomers [168].

In the presence of acid the *syn* isomers of 3-(piperidinocarbonyl)isoxazole oximes isomerize to the more stable *anti* isomers [169]. In turn the *anti* isomers of benzimidazole oximes are transformed exclusively into the *syn* isomers during photolysis (350 nm) [170]. Data on the possibility of separating the isomers of 2-amino-1-sulfonylbenzimidazoles by crystallization are presented in [171].

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