

Synthesis, Reactions, and Properties of ONO Systems

Vladimir F. Rudchenko

N. N. Semenov Institute of Chemical Physics, Russian Academy of Sciences, 117977 Moscow, Russian Federation

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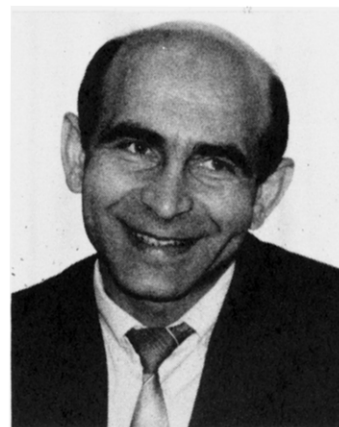
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I. Introduction

The first report (Posner, 1906)^{1,2} on the preparation of compounds with ONO fragment, namely 2-hydroxy- and 2-alkoxy-5-isoxazolidinones, was disproved 80 years later.³ The studies by Kohler^{4,5} and Sokolov^{6,7} on synthesis of 2-hydroxyisoxazolidines and 2-hydroxy-4-isoxazolines, respectively, also were found to be erroneous.⁸⁻¹¹ Knunyants and co-workers were probably the first to obtain such compounds, when in 1960 they published the synthesis of 2-hydroxydihydro-1,2,4-oxadiazete derivative.¹² In 1964, Tartakovskii et al.^{13,14} found a facile and universal method of ONO systems synthesis by 1,3-dipolar cycloaddition of nitronic esters to olefins. This reaction has been studied in detail and widely applied in organic synthesis due to the easy transformation of its products, 2-alkoxy- or 2-(silyloxy)-isoxazolidines into compounds of various types.¹⁵⁻²³

Some other trends in this field were successfully developed along with the above mentioned, and at the present time almost all possible types of acyclic and cyclic compounds of general formula XN(OR)₂, where X = alkyl, RO, H, Hal, (RO)₂N, RCO or unpaired electron have been synthesized. They are of special interest mainly due to the fact that, for example, the first two in this series represent the nitrogen analogues of such well-studied compounds as acetals, orthoesters of carboxylic acids or phosphonous diesters and trialkyl phosphites, respectively. They also possess some



Vladimir F. Rudchenko was born in Ukraine, in 1951. He graduated from Dniepropetrovsk Institute of Chemical Technology in 1973. From 1973 to 1976 he was a postgraduate student of the Institute of Chemical Physics, Academy of Sciences of the USSR. He then served as Junior Researcher (1977-1985), Senior Researcher (1985-1989), and Principal Researcher (1989-1992) at this Institute. He is currently working at Konica Corporation in Japan. He received his Candidate Degree (Ph.D.) in 1977 and his Doctor Degree (D.Sc.) in 1986 (both from the Institute of Organic Chemistry, Academy of Sciences of the USSR). His scientific interests include the problems of an asymmetric nitrogen atom.

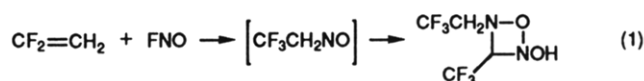
common chemical properties. The names recently suggested for them, nitroso acetals²⁴ or nitrosals²⁵ reflect the correlation between dialkoxyamines and acetals. The *O*-alkyl-*N*-alkoxyhydroxylamines are the orthoesters of nitrosyl hydride, HNO,²⁶ and *O*-alkyl-*N,N*-dialkoxyhydroxylamines are the orthoesters or nitrous acid, orthonitrites.

A high pyramidal stability is the unique feature of these compounds. The first separation of enantiomers with an asymmetric nitrogen atom in the open chain was carried out with these compounds.²⁷⁻²⁹

Data on all compounds containing the ONO fragment have been classified for the first time in this review, covering the literature up to the middle of 1992. Only the general aspects of the previously reviewed¹⁵⁻²³ material, mainly concerning 2-alkoxy- and 2-(silyloxy)-isoxazolidines, is presented here.

II. Synthesis

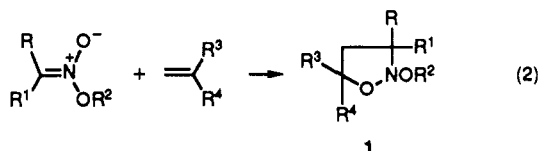
The first compound of this type was obtained by the reaction of vinylidene fluoride with nitrosyl fluoride (eq 1).¹² However, its structure has not been strictly proved, and the reaction has not been widely used.



A. [3 + 2]-Cycloadditions of Nitronic Esters

1. Cycloadditions of Open-Chain Nitronic Esters

Nitronic esters readily undergo 1,3-dipolar cycloaddition to alkenes (eq 2),^{13,14} which proceeds regio-



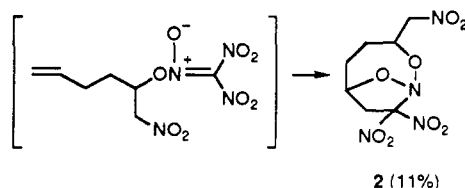
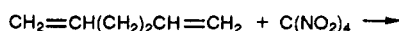
1	R	R ¹	R ²	R ³	R ⁴
a	CO ₂ Me	CO ₂ Me	Me	H	H
b	CF ₃	CF ₃	Me	H	CO ₂ Me
c	CO ₂ Me	CO ₂ Me	Me	H	NO ₂
d	H	Ph	Me	H	CO ₂ Me
e	H	CO ₂ Et	Me	H	Ph
f	CO ₂ Me	CO ₂ Me	Me	H	CN
g	CO ₂ Me	CO ₂ Me	Me	CO ₂ Et	CO ₂ Et

cifically and stereoselectively and leads to a 5-substituted isoxazolidines for monosubstituted olefins.^{13,14,25,30-64} 1,2-Disubstituted alkenes such as methyl crotonate or crotonitrile give products with the electron-withdrawing group in position 5.^{54,57} Trisubstituted olefins react with formation of 5,5-disubstituted products;^{65,66} 1,3- and 1,4-dienes react with the participation of each double bond separately.⁶⁷⁻⁷⁰ Nitronic esters obtained from mononitro-,^{13,25,30,32,37,38,43,46,51,54-58,60-65,67,71-75} 1,1-dinitroalkanes³³ and their derivatives, as well as 1,1,1-trinitrocompounds^{13,31,34,39-41,49,51-53,59,68,76,77} and tetranitromethane^{35,36,42,44,45,47,48,50,66,69,70,78} have been used in this reaction. Some of them are very unstable and may be generated conveniently in situ. The reactions are conducted under mild conditions. Yields are good to excellent in most examples. The influence of the structure of the nitronic ester upon its reactivity in 1,3-dipolar cycloaddition and the stereoselectivity of the reactions have been extensively studied by Carrie and his group.^{46,56,58,61,65,71-73,79}

In 1972 Ioffe and co-workers⁸⁰ suggested using the silyl esters of nitronic acids as a 1,3-dipolar component, which unlike their alkyl analogues is stable and retains the ability of cycloaddition to olefins.⁸¹⁻⁹²

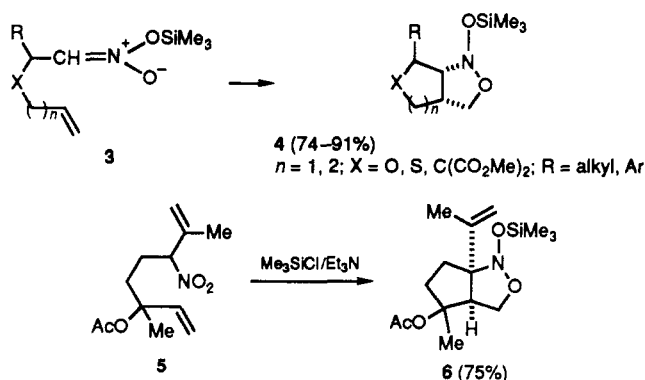
The intramolecular reactions of the nitronate group with double bonds are known too. For instance, the interaction of tetranitromethane with one of the double bonds of hexa-1,5-diene gives a nitronic ester which is then added to the other double bond forming bicyclic compound 2 (Scheme 1).⁷⁰ In this case the alkene

Scheme 1

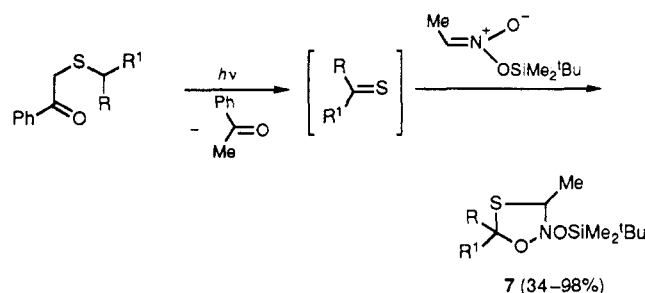


terminal carbon atom participates in C-C bond formation. However, the mode of cycloaddition is changed during cyclization of 3⁹³ and nitronic ester, which is

Scheme 2



Scheme 3

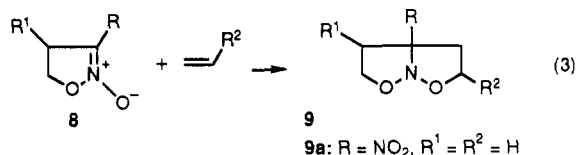


generated in situ from nitro compound 5⁹⁴ (Scheme 2). These reactions lead to fused products 4 and 6.

Monomeric aliphatic thioketones and thioaldehydes have been found to behave⁹⁵⁻⁹⁷ as 1,3-dipolarophiles toward silyl nitronates to form stable 1,4,2-oxathiazolidines 7 (Scheme 3).

2. Cycloadditions of Cyclic Nitronic Esters

Isoxazoline N-oxides can be used as 1,3-dipoles. They add to alkenes forming bicycles 9¹³ (eq 3). The same



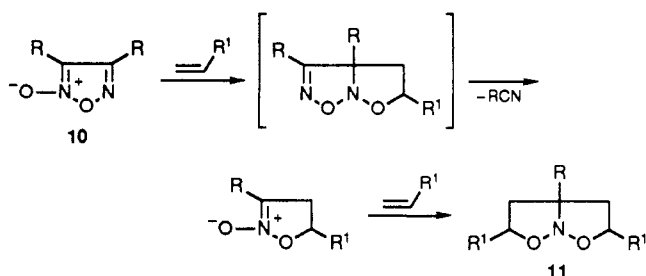
regularities as for the cycloaddition of the open-chain nitronates are observed for this reaction.⁹⁸⁻¹⁰⁵ The data on it, up to 1972, are summarized in the review of Takeuchi and Furusaki.¹⁶ The cycloaddition of 8 to silyl-substituted alkenes,¹⁰⁶ styrene,¹⁰⁷ allylic alcohol,¹⁰⁸ methyl acrylate,¹⁰⁹ as well as the stereochemistry of 3-methyl-5-phenylisoxazoline N-oxide reaction with different mono- and disubstituted olefins has also been studied.¹¹⁰

In 1983 Shimizu and co-workers suggested^{111,112} using 1,2,5-oxadiazole N-oxides (furoxanes) 10 as 1,3-dipoles. Their cycloadducts with alkenes are unstable and decompose into isoxazoline N-oxides, the reaction of which with the second mole of olefin gives the final products 11 (Scheme 4).

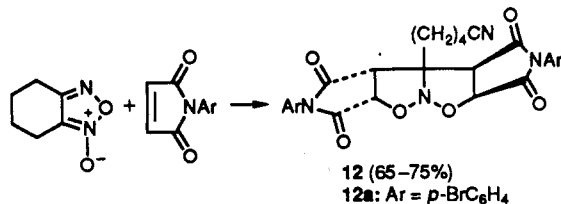
The stereospecific reaction in Scheme 5 proceeds according to similar a scheme.¹¹³

Furoxanes can also undergo the intramolecular cycloaddition which has been used for the synthesis of heterocyclic compound 13 (Scheme 6).¹¹²

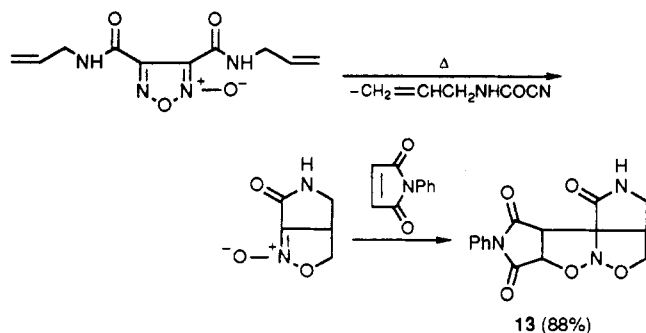
Scheme 4



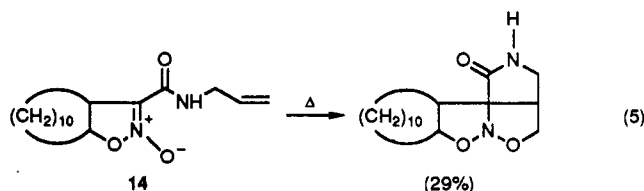
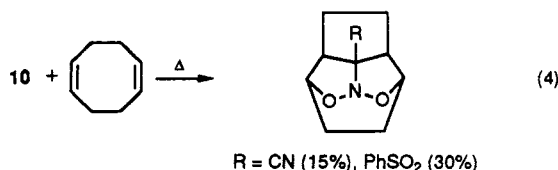
Scheme 5



Scheme 6

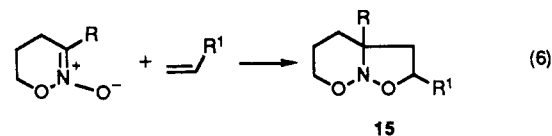


The intramolecular cycloadducts are formed in the reaction of furoxanes **10** with cycloocta-1,5-diene (eq 4)¹¹² as well as by the cyclization of isoxazoline *N*-oxide **14** (eq 5).¹¹¹



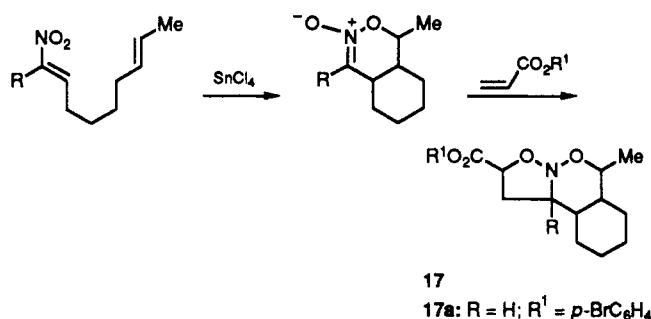
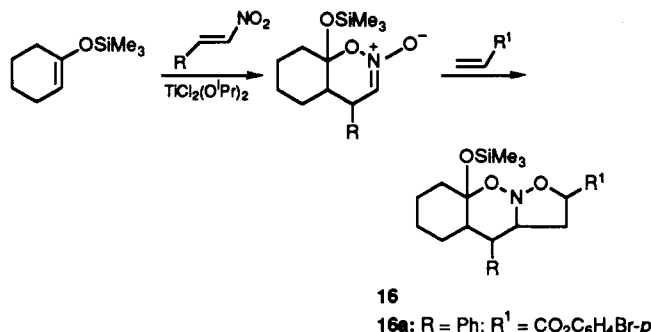
The reactions with various olefins have been described for 6-membered cyclic (eq 6)^{114–117} and bicyclic nitronates as well. The latter are readily available from nitro alkenes via inter-^{24,118} or intramolecular¹¹⁹ reactions of [4 + 2]-heterodiene synthesis (Scheme 7).

The 6-membered nitronates easily form the intramolecular [3 + 2]-cycloadducts. It has been demonstrated¹²⁰ for the synthesis of tricyclic compounds **18** (Scheme 8) via the tandem [4 + 2]/[3 + 2]-cycloadditions, which can also be effectively triggered with a chiral vinyl ether. The reactions proceed in good yield with high stereoselectivity.

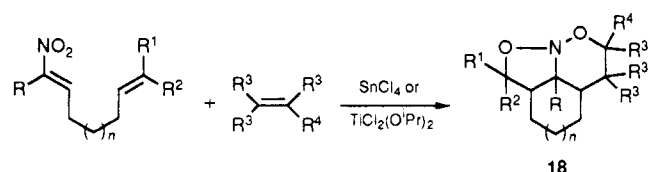


15	R	R ¹
a	COMe	Ph
b	Ph	CO ₂ Me
c	CO ₂ Me	Ph
d	Me	Ph
e	Me	CO ₂ Me
f	NO ₂	Ph

Scheme 7



Scheme 8

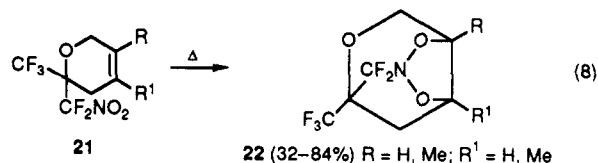
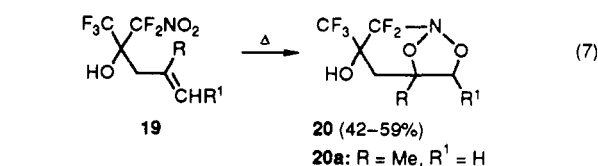


18	n	R	R ¹	R ²	R ³	R ⁴
a	0	Me	CO ₂ Et	H	Me	Me
b	1	Me	CN	H	Me	Me

B. Cycloadditions of Nitro Compounds

The nitro group, according to the Huisgen's classification,¹²¹ belongs to the allylic type 1,3-dipoles, which are well stabilized by resonance and should show little tendency to cycloadditions. The calculations predict¹²² that the nitro group should participate in cycloadditions as a 2 π -partner rather than 1,3-dipole. In accordance with that, the mechanism of intramolecular thermal cyclization of nitro olefins **19** and **21** was suggested,¹²³ which involves initial [2 + 2]-cycloaddition with formation of 2*H*-1,2-oxazete *N*-oxides followed by their rearrangement into 1,3,2-dioxazolidines **20** (eq 7) and **22** (eq 8).

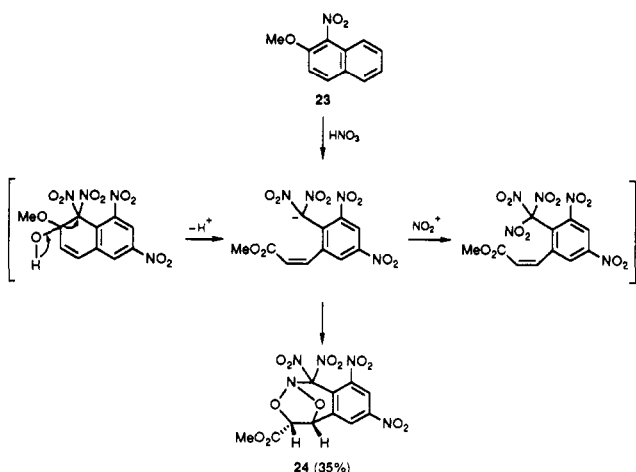
Nevertheless, it is assumed¹²⁴ that the intramolecular 1,3-cycloaddition of nitro group to the double bond is



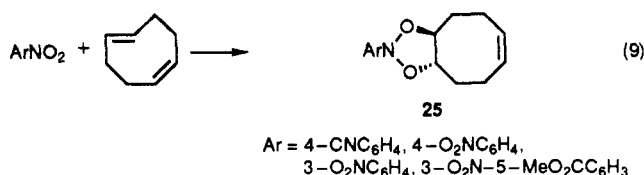
one of the steps (Scheme 9) in the unusual nitration reaction of 1-nitronaphthalene **23** with formation of compound **24**.

There is only one example of thermal intermolecular cycloaddition of the nitro compounds to alkenes, which

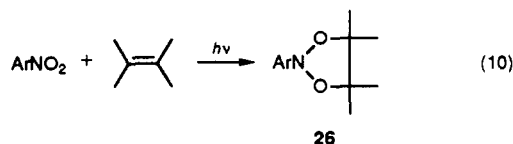
Scheme 9



was carried out with nitrobenzene derivatives bearing electron-withdrawing groups and the highly reactive (*E,Z*)-cycloocta-1,5-diene (eq 9).¹²⁵



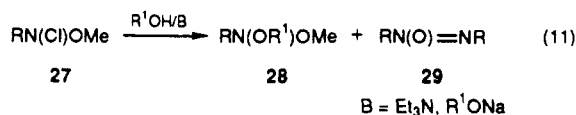
For the nitro compounds it is more common to undergo the photochemical cycloaddition to olefins (eq 10)¹²⁶ via a biradical intermediate with formation of unstable adducts **26**.



C. Nucleophilic Substitution of Chlorine in ONCl Compounds

The high anionic mobility of chlorine is the characteristic feature of *N*-chloro-*N*-alkoxy-substituted nitrogen-containing compounds. It is caused by $n_{\pi(\text{O})} \rightarrow \sigma^*_{\text{N-Cl}}$ interaction, which kinetically destabilizes the N-Cl bond and facilitates its heterolysis. Thus,

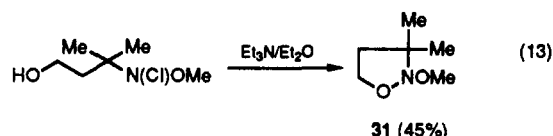
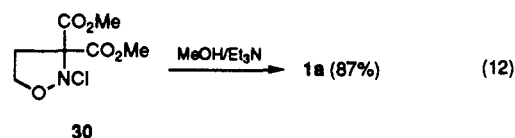
N-chloro-*N*-alkoxyamines **27**¹²⁷ readily react with a number of alcohols under mild conditions, giving the stable dialkoxyamines **28** (yields 21–88%), which are in most cases the only reaction products (eq 11).^{28,127,128}



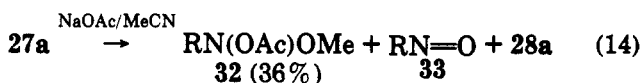
28	R	R ¹
a	MeO ₂ CCMe ₂	Me
b	MeO ₂ CCMe ₂	PhCH ₂
c	MeO ₂ CCH ₂ Me ₂	Me
d	MeO ₂ CCH ₂ Me ₂	Et
e	MeO ₂ CCH ₂ Me ₂	PhCH ₂
f	MeO ₂ CCH ₂ C(CF ₃)Me	Me

However, reactions of **27** with sterically hindered alcohols (*i*PrOH, *t*BuOH) in the presence of triethylamine also yield diazene oxides **29** due to the reduction of **27** into *N*-alkoxy aminyl radicals and their further transformations.¹²⁹ This method is suitable only for preparation of *N*-*tert*-alkyl-substituted compounds, because *N*-chloroamines **27** with primary and secondary *N*-alkyl substituents are unstable and are easily dehydrochlorinated.²⁸

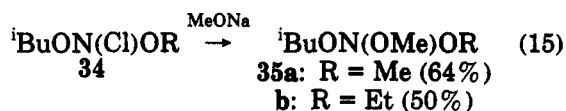
This reaction has been used in two of its modifications for synthesis of isoxazolidines **1a** (eq 12)^{130–132} and **31** (eq 13).^{28,133}



The nucleophilic substitution of chlorine in **27a** also proceeds under the action of sodium acetate, leading to *N*-acetoxy-*N*-methoxyamine **32** and its decomposition products **33** and **28a** (eq 14).¹³⁴

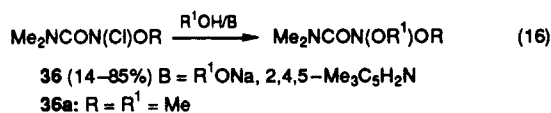


The N-Cl bond in chloro compounds **34** more likely undergoes heterolysis than in **27** due to two-electron-donating *N*-alkoxy substituents. For this reason **34** are highly unstable. Their reaction in situ with sodium methylate remains so far the only method of ortho-nitrites **35** synthesis (eq 15).^{135–137}



The nucleophilic substitution of chlorine proceeds in *N*-chloro-*N*-alkoxyureas under the action of alcohols (eq 16)^{138–141} and in *N*-chloro-*N*-alkoxybenzamides when treated by silver acetate (Scheme 10).^{142,143}

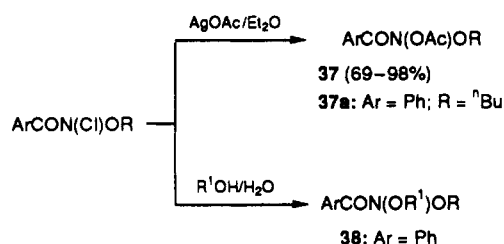
The solvolysis of the latter compounds in aqueous alcohols with the formation of dialkoxyamides **38** has



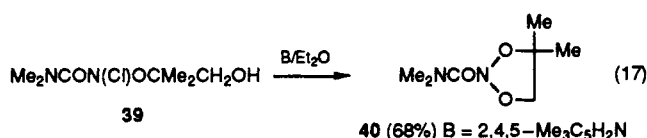
also been reported (Scheme 10).¹⁴² Although, these products have not been characterized in this paper.

The intramolecular cyclization of *N*-chlorourea **39** is

Scheme 10

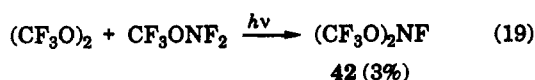
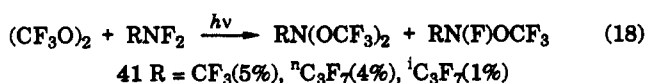


a simple method for 1,3,2-dioxazolidine **40** synthesis (eq 17).¹⁴⁴

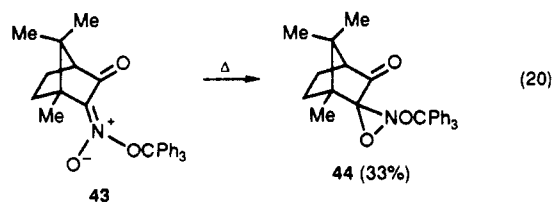


D. Other Synthetic Methods

In 1965, a patent was registered on the preparation of perfluorinated dialkoxyamines **41** (eq 18) and **42** (eq 19) by a photochemical reaction with difluoroamines.¹⁴⁵

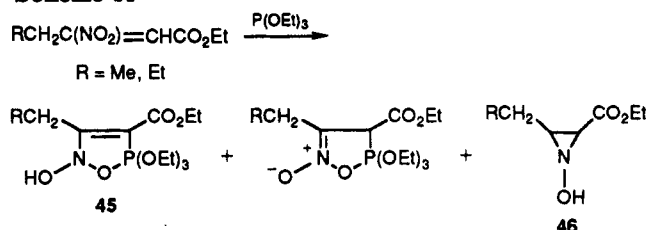


So far, the only known *N*-alkoxyoxaziridine **44** has been synthesized by the thermal isomerization of nitronic ester **43** (eq 20).¹⁴⁶

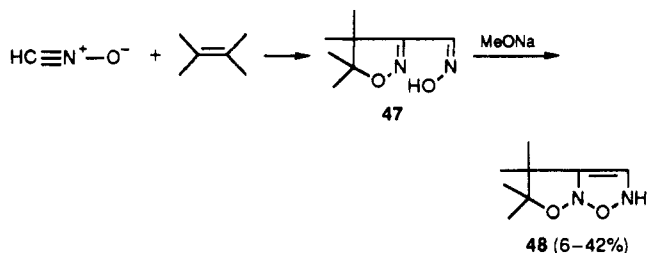


The reaction of ethyl α,β-unsaturated-β-nitrocarboxylates with triethyl phosphite gives an *N*-hydroxy derivative of a O,N,P-containing heterocycle **45** as one of the products (Scheme 11).¹⁴⁷ Unfortunately, its structure has not been strictly proved, while it was shown¹⁴⁸ that *N*-hydroxyaziridine structure of **46** was erroneous.

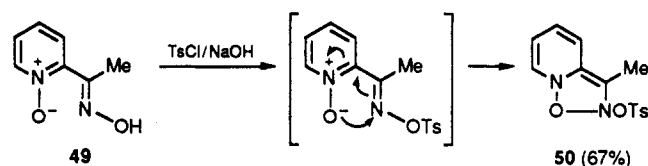
Scheme 11



Scheme 12



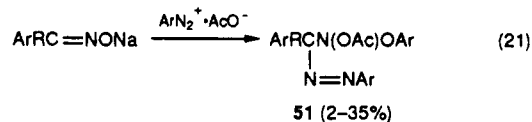
Scheme 13



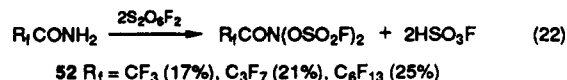
The cyclization of *anti*-oximes **47**, produced in the reaction of formonitrile oxide with olefins (ethylene, cyclopentene, 1-acetylcyclopent-1-ene, 3β-acetoxy-5,6-pregnadiene-20-one), leads to a new heterocyclic system **48** (Scheme 12).¹⁴⁹

The tosylation of the *E* form of oxime **49** gives the corresponding oxime *O*-ester which undergoes an unusual intramolecular cyclization producing bicycle **50** (Scheme 13).¹⁵⁰

N-(Acyloxy)-*N*-(aryloxy)amines **51** were obtained by the arylation of benzaldoxime and ketoximes salts with aryl diazonium salts (eq 21).^{151,152} A wide range of other products is also formed. However, in this paper there is no evidence for structure **51**.



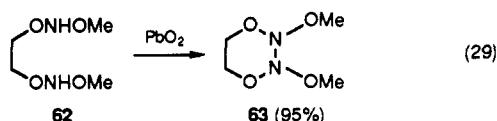
The first amides with a nitrogen atom covalently bounded with two oxygen atoms were obtained by reaction of perfluorocarboxylic acids with peroxydisulfuryl difluoride (eq 22).¹⁵³ The low yields of compounds **52** may be interpreted by their instability under the reaction conditions at –20 to –30 °C.



The *N,N*-bis[(trialkylsilyl)oxy]-1-alken-1-amines were synthesized predominantly (95%) in the form of *E* isomers **54** by the complete silylation of nitro compounds **53** (eq 23).¹⁵⁴

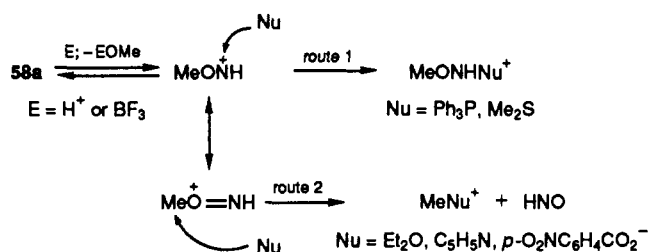
27), which can be isolated in a pure state.¹⁴¹ These radicals can recombine with the 2-cyanoisopropyl radical as well.¹⁴¹

The oxidation of 1,2-bis[(methoxyamino)oxy]ethane (62) leads to a new heterocycle 63, for which no equilibrium with the corresponding diradical has been observed (eq 29).¹⁷²



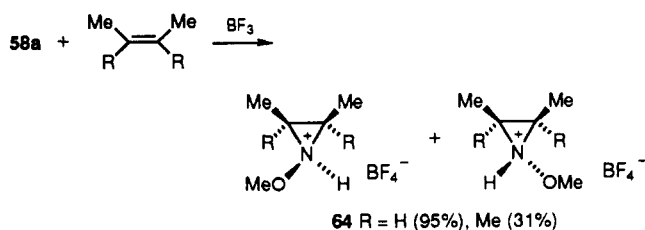
O-Methyl-*N*-methoxyhydroxylamine (58a) easily undergoes electrophilically initiated cleavage of the N–O bond, affording *N*-methoxy nitrenium ion, which is an ambident cation and takes part in reactions with nucleophiles as methoxyaminating or methylating agent (Scheme 17).^{159,160}

Scheme 17



This ion can be added to alkenes producing 1-methoxyaziridinium salts 64 (Scheme 18).^{159,160} The stereo-

Scheme 18



reospecificity of *N*-methoxy nitrenium ion cycloaddition to *cis*-but-2-ene was considered as evidence of its singlet ground electronic state.

C. *N,N*-Dialkoxy-*N*-alkylamines

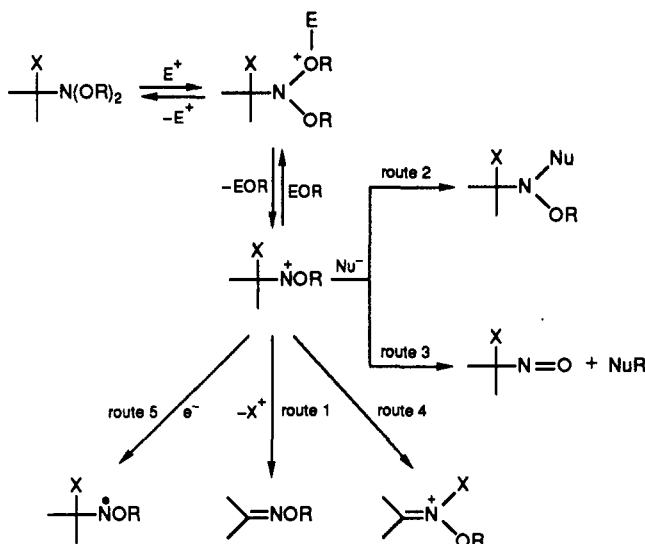
The properties of acyclic and cyclic compounds of this series are considered here.

1. Reactions with Electrophilic Reagents

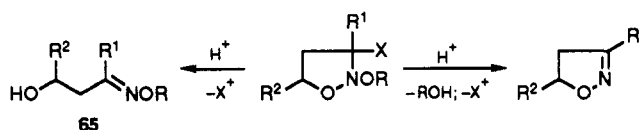
The main directions of these reactions are presented in Scheme 19.¹⁷³

a. Route 1. This type of transformations predominates if there is electrofuge (H, NO₂, CONHMe, Ac) at the α -carbon atom in *N*-substituent of isoxazolidines 1,^{30–33,36,37,41,62,67,83,85–92,156} 1,4,2-oxathiazolidines 7,⁹⁶ as well as bicyclic dialkoxyamines 4,⁹³ 9,^{98,101,106,174} and 15.^{175,176} HCl, H₂SO₄, *p*-TsOH, ZnCl₂, BF₃, and HOAc were used as electrophiles. The acid hydrolysis of isoxazolidines 1 has been investigated most thoroughly (see also reviews found in refs 15, 20, and 22). It was found to proceed under the stereoelectronic control,^{156,157} and the preference of one of its directions

Scheme 19



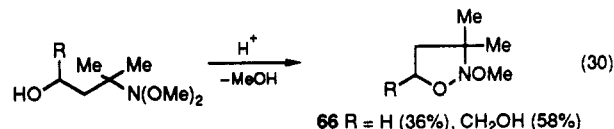
Scheme 20



(Scheme 20) depends on the acid used^{32,33} and the nature^{32,37} and mutual orientation³⁷ of R¹ and R² substituents. The β -imino alcohols 65^{32,37} under some reaction conditions are transformed further into β -hydroxy ketones,³⁰ β -hydroxy acids (for compounds with R¹=NO₂),^{31,33} or α,β -unsaturated acids.^{31,36,41,51}

The hydrolysis of 2-(silyloxy)isoxazolidines gives exclusively 2-isoxazolidines.^{83,85–92}

b. Route 2. This includes the substitution of *N*-alkoxy group for chlorine during interaction of dialkoxyamines 28^{129,130} with MeCOCl, CF₃COCl, SOCl₂, Me₃SiCl, HCl, or isoxazolidine 1a^{130,131} with SOCl₂ as well as acid-catalyzed "transesterification" of these compounds under the alcohols action.^{156,158} The latter reaction is used for synthesis of isoxazolidines 66 (eq 30)¹⁵⁸ as well as *N*-alkyl-substituted heterocycles with

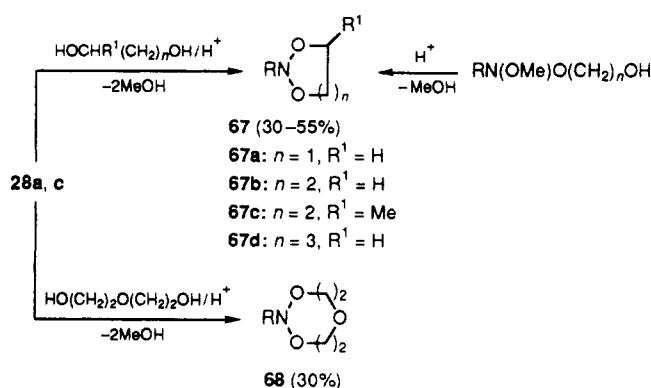


endocyclic fragment ONO 67^{158,177–179} and 68¹⁸⁰ (Scheme 21).

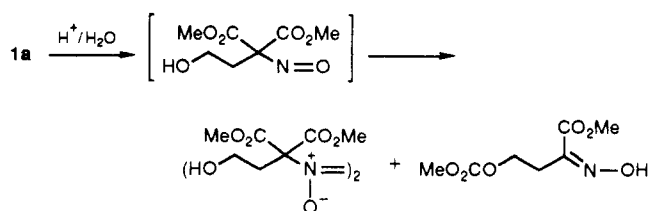
c. Route 3. The nitroso compounds are formed by acid-catalyzed alcoholysis¹⁵⁸ or hydrolysis^{25,64} of dialkoxyamines 28a,c,f. The acid hydrolysis of isoxazolidine 1b leads to the corresponding γ -nitroso alcohol.^{25,64} Isoxazolidine 1a gives a nitroso alcohol dimer and the product of its rearrangement (Scheme 22).^{25,64}

d. Route 4. Dialkoxyamines readily undergo a BF₃-induced 1,2-rearrangement with migration of various groups to the nitrogen atom. The 1,2-shift of the ester group was observed in the dialkoxyamine 28a^{129,158} and isoxazolidine 1a^{156,161} as well as the methyl group in dialkoxyamine 28c.¹⁵⁸ The 1,2-rearrangement of bicyclic 15 with migration of acetyl (in 15a^{175,176}) or phenyl (in 15b¹⁸¹) groups leads to spirocyclic products. In the

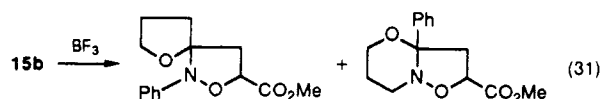
Scheme 21



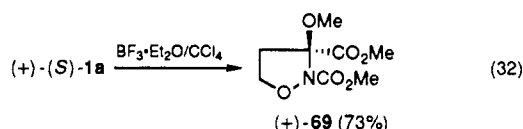
Scheme 22



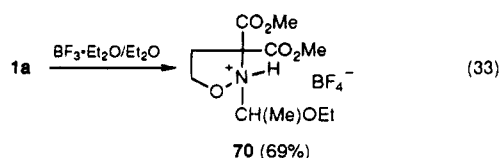
latter case a competitive 1,2-shift of the alkyl group has also been observed (eq 31).¹⁸¹



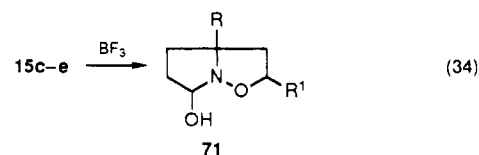
It is assumed that the rearrangement of isoxazolidine **1a** proceeds according to synchronous mechanism. The isolation of optically active product (+)-**69** of isoxazolidine (+)-(*S*)-**1a** rearrangement (eq 32) was considered as one of the evidences of that.^{181,161}



e. Route 5. This direction is realized if dialkoxyamines are treated by an electrophile in the presence of a reducing reagent. The intermediately formed *N*-alkoxy aminyl radicals undergo further transformations. Thus, in reactions of **28a,c** with triethylamine hydrochloride they are dimerized into 1,2-dialkoxyhydrazines, fragmentation of which gives the final products, 1,2-dialkyldiazene oxides.^{129,158} In the reaction of isoxazolidine **1a** with BF_3 etherate (eq 33),¹⁶¹ the diethyl ether acts as a reducing agent and product **70** is formed by recombination of aminyl radical and the radical produced by oxidation of ether.



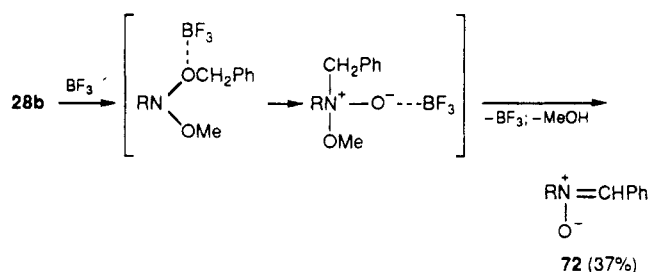
The red-ox mechanism is proposed¹⁶¹ for the following rearrangement (eq 34) of bicycles **15c-e** which leads to products **71**.¹⁸¹⁻¹⁸³



Besides the above-discussed reactions, which were summarized in Scheme 19, the electrophilically initiated addition of **28a**¹²⁹ and **1a**¹⁶¹ to isobutylene, the acid-catalyzed reduction of **28a** by cathechol,¹⁵⁸ acid hydrolysis of oxaziridine **44**,¹⁴⁶ and 2-(2-nitro-1-arylethoxy)isoxazolidines¹⁸⁴ with formation of 2-isoxazoline *N*-oxides were also studied.

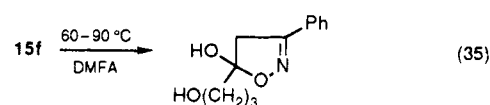
It should be mentioned that reactions of dialkoxyamines according to routes 1-3 (Scheme 19) are also typical for their carbon analogues, acetals. At the same time there is an obvious difference in the properties of dialkoxyamines and their phosphorous analogues, phosphonous diesters, in the reactions of which an electrophilic attack at the phosphorous atom is realized.¹⁸⁵ The dialkoxyamines are inert under the conditions of Arbuzov reaction and are converted according to routes 3 or 4 (Scheme 19) when treated by methyl triflate.¹⁷³ Nevertheless, to explain the formation of nitron **72** in the reaction of **28b** with BF_3 ,¹⁵⁸ Scheme 23 was suggested.¹⁷³ It involves the isomerization of

Scheme 23



28b into derivative of *O*-methylhydroxylamine *N*-oxide, which is similar to Arbuzov rearrangement.

It is assumed¹⁷³ that such a type of isomerization might also be one of the steps of the acid-catalyzed or thermal rearrangement of bicycles **15** ($R = OH, OMe$; $R^1 = Ph$)^{181,186} or **15f**,¹⁸⁶⁻¹⁸⁸ respectively (eq 35).

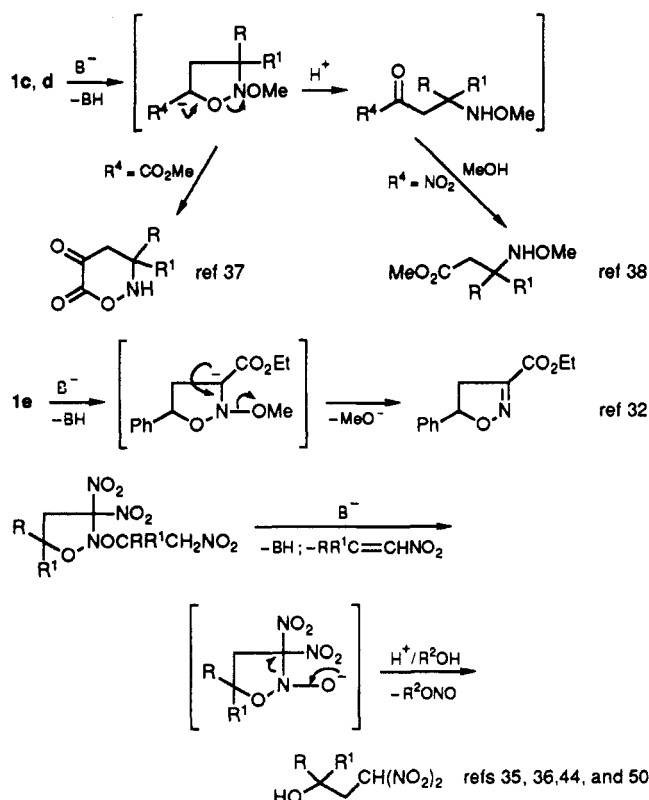


2. Reactions with Nucleophiles

These reactions can proceed either with retention or with cleavage of dialkoxyamine ONO fragment. This fragment is not affected by the nucleophilic substitution of nitro group in bicycles **15** ($R = NO_2$) for OH ,¹⁸⁹ OR ,^{190,191} N_3 ,¹⁹² or CN ,^{193,194} as well as by amidation or alkaline hydrolysis of some isoxazolidines **1**^{38,74,195-197} and dialkoxyamines **28**²⁷⁻²⁹ or **67**.^{179,198}

The reactions of the second type are observed for isoxazolidines **1** with an electron-withdrawing (NO_2 or CO_2Me) substituent in the cycle or the *N*-alkoxy group, which promotes the formation of an anion at the carbon atom bearing that substituent. Further transformations of this carbanion depend on its location and are

Scheme 24

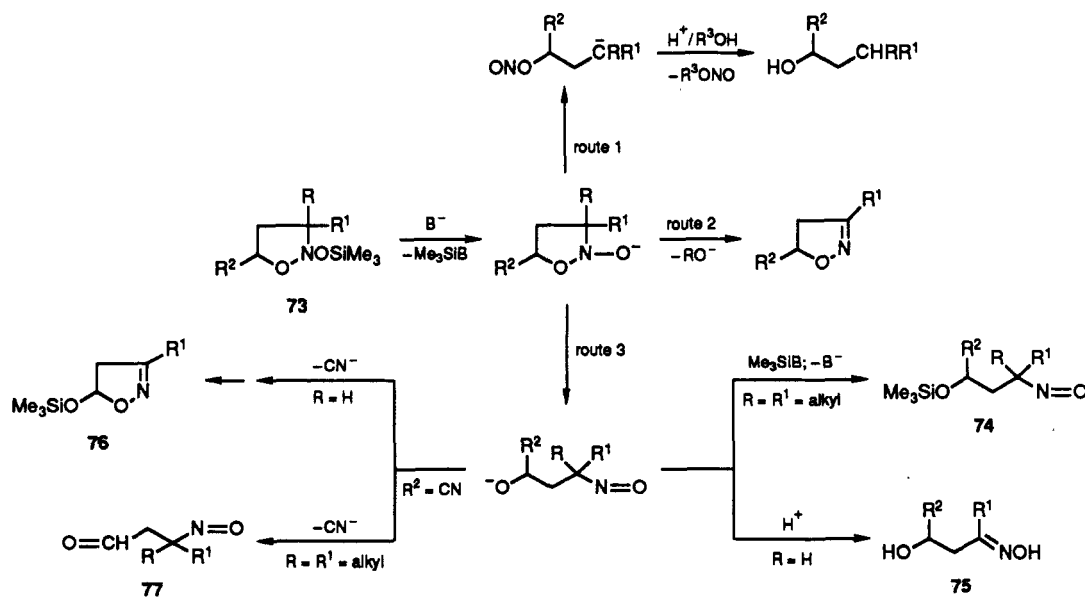


accompanied by cleavage of one of the nitrogen atom bonds (Scheme 24).^{32,35-38,44,50}

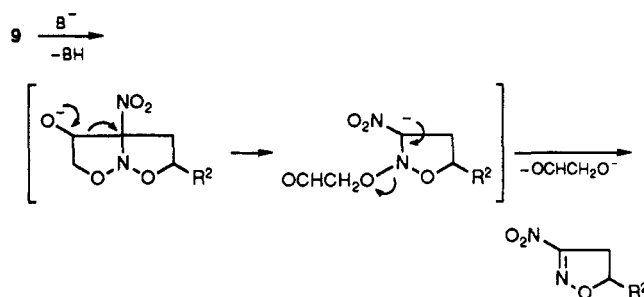
The reactions of 2-(silyloxy)isoxazolidines **73** with nucleophiles (see also the reviews found in refs 22 and 23) always occur via the cleavage of the Si-O bond. The anion formed can be then transformed according to the three routes in Scheme 25.

3,3-Dinitro-substituted isoxazolidines **73** react with alcoholic solution of alkali or alcoholates by route 1.^{49,81,82} For some of them⁸¹ route 2 is also partially realized, which becomes the main one for isoxazolidines **73**, where $\text{R} = \text{R}^1 = \text{CO}_2\text{Me}$.⁸⁰ In all of the rest of cases the reactions proceed with the cleavage of the cyclic N-O

Scheme 25



Scheme 26



bond (route 3). Depending on the type of substituents in the cycle and the nucleophile used, the final products are nitroso compounds **74**,⁸⁸ oximes **75**,^{83,85,86} 2-isoxazolidines **76**,^{87,89} and nitroso aldehydes **77**.⁸⁸

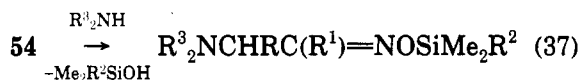
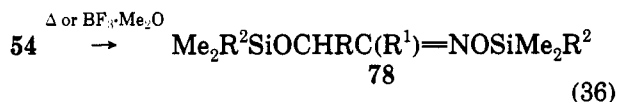
The F⁻-induced transformation of 1,4,2-oxathiazolidines **7** into carbonyl compounds⁹⁶ and the fragmentation of bicycle **9** ($\text{R} = \text{NO}_2$, $\text{R}^1 = \text{OH}$) according to the Scheme 26¹⁰⁰ are related to this reaction type as well.

3. Other Reactions

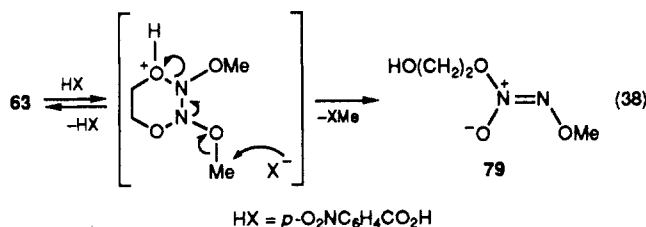
Reduction of isoxazolidines **6** and **73** (B_2H_6 ,⁸⁵ Li/NH_3 ,⁹⁴ $\text{H}_2/\text{Raney Ni}$,⁸⁶⁻⁸⁸) as well as of bicycles **16**²⁴ and **18**¹²⁰ ($\text{H}_2/\text{Raney Ni}$) is accompanied by the cleavage of the cyclic N-O bond and formation of corresponding amino alcohols, which in some cases may undergo further transformations. Reduction of 3-phenyl-substituted isoxazolidines **1** by LiAlH_4 leads to isoxazolidines.³⁰ However, ONO fragment remains intact with LiAlH_4 reduction of **28**,¹⁵⁸ KMnO_4 oxidation of **1**,⁶⁸ and bromination of **9**.¹⁰²

D. Miscellaneous ONO Systems

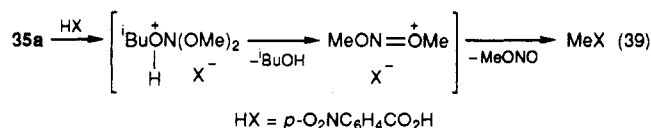
Chemical properties of compounds considered in this section are presented by some certain reactions. Thus, hydrogenation of **25**¹²⁵ and **26**¹²⁶ on Pd-C produces corresponding 1,2-diols. Enamines **54** undergo thermal or BF_3 -catalyzed rearrangement to silyl esters **78** (eq 36).¹⁵⁴ They can also add amines to their double bond with the loss of one molecule of trialkylsilanol (eq 37).¹⁵⁴



Regio- and stereospecific stereoelectronically controlled reaction of hydrazine 63 with *p*-nitrobenzoic acid produces an equimolar mixture of methyl *p*-nitrobenzoate and dialkoxydiazene oxide 79 in the form of the *E* isomer (eq 38),¹⁷² which is the first representative of this type compounds.



The decomposition of orthonitrite 35a occurs on *p*-nitrobenzoic acid treatment and leads to the methyl ester of this acid in 74% yield (eq 39).¹³⁷ This product



is also obtained in 91% yield by reaction of 35a with *p*-nitrobenzoic acid chloride.¹³⁷ These two reactions obviously demonstrate the similar properties of orthonitrites and their carbon analogues, orthoesters of carboxylic acids.

IV. Properties

A. Structure and Stereochemistry

Topomerization of ONO systems as well as of some other compounds with an N–O bond¹⁹⁹ is an inversion-rotation process. However, while, for instance, the acyclic trialkyl-substituted hydroxylamines have close values of barriers to nitrogen inversion and N–O bond rotation ($\Delta G^\ddagger$ 9–12 kcal/mol),¹⁹⁹ for compounds with ONO fragment the inversion of the nitrogen pyramid is the rate-determining step in their topomerization. The latter has been related to the effect of oxygen atoms and might be explained as in the following.^{43,200} The nitrogen inversion proceeds via a planar transition state in which its lone electron pair occupies the p_z orbital. Electronegative ligands increase the s character of nitrogen nonbonding orbital and thereby destabilize the inversion transition state, increasing the inversion barrier. The 6-electron interaction of lone electron pairs of heteroatoms provides a considerable contribution into destabilization of this state as well.^{43,201} It has been suggested¹⁵⁷ that the pyramidal state might be stabilized due to vicinal $n_{\pi(\text{O})}-\sigma^*_{\text{N-O}}$ interaction.

Configurational stability of acyclic compounds $\text{XN}(\text{O-R})\text{OR}^1$ (Table I) to a great extent depends on the nature of the substituent X. *N,N*-Dialkoxy-*N*-alkylamines 28 and *O*-alkyl-*N*-alkoxyhydroxylamines 58 have close

values of inversion barriers. It is assumed¹⁶⁵ that the $n_{\text{N}}-\sigma^*_{\text{C-R}}$ interaction, stabilizing the planar transition state of inversion, affects the pyramidal stability of compounds with $\text{X} = \text{CH}_2\text{R}$, where $\text{R} = \text{OH}$, Me_2N , or Me_3N^+ . The conjugation of dialkoxyamine nitrogen atom with carbonyl group leads to a considerable decrease of its configurational stability. Therefore low inversion barriers have been found for dialkoxyureas 36 (Table I). The restricted rotation around the $\text{Me}_2\text{N}-\text{CO}$ bond ($\Delta G^\ddagger$ 13.3–14.5 kcal/mol) has been observed for these compounds as well.^{140–202}

An extremely high configurational stability among acyclic nitrogen-containing compounds is expected for orthonitrites 35, although it cannot be evaluated because of their low thermostability.¹³⁷ The pyramidal configuration of the nitrogen atoms was found in hydrazines 61, which, however, undergo homolytic cleavage of N–N bond already at room temperature.

Configurational stability of the nitrogen pyramid increases when an ONO fragment is incorporated into the cycle because that destabilizes the planar inversion transition state to a great extent.²⁰⁰ Thus, inversion barriers in isoxazolidines 1 ($\Delta G^\ddagger$ 26.9–29.3 kcal/mol)^{43,58,73,74} are ca. 5 kcal/mol higher (all data are collected in ref 157) than in their acyclic analogues 28. Anomalous low inversion barriers among isoxazolidines 1 (E_a 14.6²⁰³ and 16.1 kcal/mol²⁰⁴) were found for their 3,3-dinitro derivatives, which were interpreted²⁰⁴ by the increase of the ground-state energy due to nonvalent interactions in *hem*-dinitro groups. Inversion barriers for 1,3,2-dioxazines 57b (9.8 kcal/mol) and 59b (21.9 kcal/mol) as well as 1,3,2-dioxazolidine 57a (15.9 kcal/mol) have also been estimated.¹⁶⁴

It is assumed^{73,156,157,198,205,206} that the stereochemical properties of ONO systems are mainly caused by the existence of $n_{\pi(\text{O})}-\sigma^*_{\text{N-O}}$ vicinal interaction. That is why such compounds adopt the most favorable for $n-\sigma^*$ overlapping geometry (anomeric effect²⁰⁷). They also display a tendency to populate the rotamers with a maximum number of gauche interactions between adjacent electron pairs and polar bonds ("gauche" effect²⁰⁸). In accordance with that, *O*-methyl-*N*-methoxyhydroxylamine 58a exists²⁰⁵ in the crystalline and gaseous state as well as in solution in (+sc, +sc) conformation (symmetry C_2) with dihedral angles CONO (Figure 1) of 78.7° (X-ray). A substantial shortening of the N–O bonds in 58a (1.384 Å) in comparison, for instance, with MeONHMe (1.496 Å),²⁰⁹ is considered to be the second geometrical consequence of $n-\sigma^*$ overlapping. It is interesting that ab initio calculations²¹⁰ of dihydroxylamine $(\text{HO})_2\text{NH}$ predict an energy minimum for its (+ac, -ac) conformer.

The advantage of synclinal conformations around N–O bonds is preserved upon incorporation of one of these bonds into a 5-membered cycle. Thus, by NMR^{46,55,58,61,71–73,75,105,203,204,211,212} and X-ray analysis^{74,90b,156,157,197,201,213–216} it has been established that isoxazolidines 1 exist both in solution and in the crystalline state as anomers with a pseudoaxial orientation of *N*-alkoxy group. Isoxazolidine ring has the envelope type conformation. The values of dihedral angles CONO for *endo*- and *exo*-CON fragments equal to 65.5–79.4° and 81.4–98.8°, respectively. (Selected structural parameters of isoxazolidines 1 are collected in ref 157.)

Table I. Inversion Barriers (kcal/mol) in XN(OR)OR¹ Compounds

X	R	R ¹	solvent	T, °C	ΔG [‡] , T	ref
H	ⁱ Bu	Me	toluene- <i>d</i> ₈	25	20.4	165
H	ⁱ Bu	Et	toluene- <i>d</i> ₈	25	20.0	165
H	PhCH(Me)NHCOCMe ₂	Et	toluene- <i>d</i> ₈	64	18.9	140
MeO ₂ CCMe ₂	Et	Me	Ph ₂ O	20	23.6	28
MeO ₂ CCMe ₂	CF ₃ CH ₂	Me	toluene- <i>d</i> ₈	20	22.6	28
MeO ₂ CCMe ₂	ⁱ Pr	Me	Ph ₂ O	20	21.9	28
MeO ₂ CCMe ₂	HO(CH ₂) ₂	Me	Ph ₂ O	20	22.4	28
MeO ₂ CCH ₂ CMc ₂	Et	Me	toluene- <i>d</i> ₈	20	24.6	28
MeO ₂ CCH ₂ CMc ₂	ⁱ Pr	Me	Ph ₂ O	20	21.7	28
MeO ₂ CCH ₂ CMc ₂	PhCH ₂	Me	Ph ₂ O	20	22.6	28
MeO ₂ CCH ₂ CMc ₂	PhCH ₂	Me	MeOH	20	23.5	28
(EtO ₂ C) ₂ CHCMe ₂	ⁱ Pr	Me	Ph ₂ O	20	22.2	28
HOCH ₂	PhCH ₂	Me	DMSO- <i>d</i> ₆	25	20.7	165
Me ₂ NCH ₂	PhCH ₂	Me	DMSO- <i>d</i> ₆	25	21.2	165
Me ₃ N ⁺ CH ₂	PhCH ₂	Me	DMSO- <i>d</i> ₆	25	15.4	165
Me ₂ NCO	ⁱ Bu	CF ₃ CH ₂	toluene- <i>d</i> ₈	25	10.5	202
Me ₂ NCO	CH ₂ =CHCH ₂	CF ₃ CH ₂	toluene- <i>d</i> ₈	25	9.8	202
Me ₂ NCO	PhCH(Me)NHCOCMe ₂	Et	toluene- <i>d</i> ₈	-30	12.8	140

Figure 1. Newman projections along N-O bonds of *O*-methyl-*N*-methoxyhydroxylamine (58a).

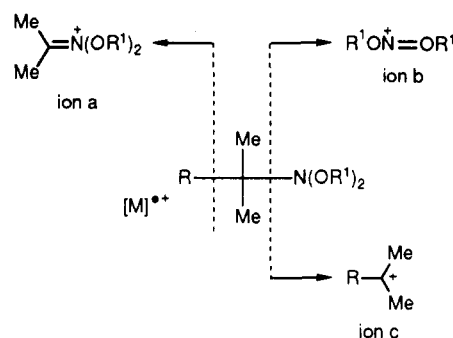
Conformation of ONO systems is greatly affected by steric effects. Thus, by NMR^{217,218} and X-ray analysis²¹⁹ it has been shown that bicycles 15 have cis-fused rings with the R group equatorial to the 6-membered ring and pseudoaxial to the 5-membered ring. Such a geometry is favorable for effective $n_{\pi(O)}-\sigma^*_{N-O}$ interaction where the oxygen atom of the 5-membered ring acts as an n donor (the average value of dihedral angles CONO equals to 75.9°).¹⁵⁷ At the same time almost orthogonal orientation of $n_{\pi(O)}$ orbital of 6-membered ring and σ^*_{N-O} orbital of the 5-membered ring (the average value of the angles between them equals to 88.6°)¹⁵⁷ actually excludes their overlapping. The consequences of that are the shortening of the N-O bond of the 5-membered ring in comparison with that one of the 6-membered ring and the selective cleavage of the latter bond in reactions (see section III.C.1) of bicycles 15 (kinetic anomeric effect).^{156,157}

The structure of such bicycles containing isoxazolidine cycle as 9a,²²⁰ 12a,¹¹³ 16a,²⁴ 17a,¹¹⁹ 18a,¹²⁰ and 18b¹²⁰ has been examined by X-ray analysis.

The conformational peculiarities of heterocycles with endocyclic ONO fragment have been studied in detail. It was established¹⁹⁸ that 1,3,2-dioxazine 67b, both in the crystalline state and in solution, exists in a chair conformation and its N-substituent has equatorial orientation. Geometry is the same for the unsubstituted at nitrogen 1,3,2-dioxazine 59b as well.¹⁶⁴ 1,3,2-Dioxazolidine ring of 59a¹⁶⁴ adopts a curved envelope conformation. N-H proton is pseudoaxial oriented. The structure of bicyclic 1,3,2-dioxazolidines 20a²²¹ and 24¹²⁴ has been determined by X-ray analysis.

Tetramethoxyhydrazine 61a in the crystalline state²⁰⁶ has an unusual, for hydrazines, geometry with anti-periplanar orientation of nitrogen lone electron pairs. The orientation of the methoxy groups is characterized by the dihedral angles CONO equal to 108.1° and -131.7°, i.e. the conformation of CONOC fragments is

Scheme 27



close to (+ac, -ac). It is assumed that such a geometry is the most favorable for simultaneous realization of two stabilizing $n_{\pi(O)}-\sigma^*_{N-O}$ and $n_{\pi(O)}-\sigma^*_{N-N}$ interactions. The latter is thought to be responsible for the lengthening of the N-N bond in 61a (1.484 Å) compared to that in N₂H₄ (1.446 Å).

Cyclic hydrazine 63 exists in solution as a mixture of ee and ae isomers. Parameters ($k = 188.7 \text{ s}^{-1}$, $\Delta G^{\ddagger} = 11.3 \text{ kcal/mol}$) for interconversion of the latter (ae $\rightleftharpoons$ ea) were determined by ¹³C NMR.¹⁷²

B. Spectral Properties

The decrease the p character of nitrogen lone electron pair in compounds with the ONO fragment (see sections III.B and IV.A) is reflected in their spectral properties as well. Thus, the first ionization potential of *O*-methyl-*N*-methoxyhydroxylamine 58a (9.49 eV)²²² is much higher than that of, for instance, dimethylamine (8.93 eV).²²³ In accordance with that, either the decrease of contribution or absence of amine-type fragmentation (Scheme 27, ion a) under the electron impact of acyclic 28²²⁴ and cyclic 67^{158,224} dialkoxyamines has been observed and attributed to a weak delocalizing ability of the dialkoxyamine nitrogen atom. In the mass spectra of these compounds ions b or c (depending on the type of R substituent) predominate. The amine-type fragmentation is not the main decomposition route under the electron impact of isoxazolidines 1a¹⁵⁶ and 66¹⁵⁸ as well. However, the heaviest ion in the mass spectra of bicycles 9 (R = NO₂) appears correct on α -cleavage with nitro group elimination.^{225,226}

The intensive absorption band at 1010–1060 cm^{-1} in the IR spectra of **1** and **9** was attributed to the ONO fragment.²²⁷

V. Optically Active Derivatives

ONO systems present one of those few types of compounds²²⁸ which pyramidal stability (section III.A) is sufficient for their resolution into geometrical and optical isomers with an asymmetric nitrogen atom. It was experimentally proved for the first time in 1969 by Muller and Eschenmoser, who managed⁴³ to separate the diastereomers of isoxazolidine **1f**. The first optically active compounds of this type were also obtained from isoxazolidines **1**,¹⁹⁵ for which many of those classical methods of antipode resolution, which are developed for the compounds with a chiral carbon atom, can be applied. Thus, the enantiomer-differentiating amidation of isoxazolidines **1a**⁷⁴ and **1g**¹⁹⁵ by 1-ephedrine leads to unreacted **1a** and **1g** enriched by one of the antipodes. The crystallization of isoxazolidine **1g** tetraamide either from 1-methyl lactate or from a mixture 1-methyl lactate– H_2O (3:5) gives either right- or left-rotating amides.¹⁹⁵ Heating of isoxazolidine **1a** diamide in 1-methyl lactate (100 °C, 6.5 h) followed by chiral solvent removal produces left-rotating diamide (the asymmetric reaction of nitrogen inversion).²²⁹ Methanolysis of 2-chloroisoxazolidine (+)-**30** occurs with the retention of optical activity and leads to isoxazolidine (–)-(2*R*)-**1a**.^{131,132} All these methods give the products only partially enriched by one of enantiomers. The complete separation of isoxazolidine **1a** bis(methylamide) into antipodes was accomplished via diastereomeric salts of (*S*)- and (*R*)-phenylethylamine with monoacids obtained from **1a**.^{196,197} The optical purity of antipodes (100 and 93%) was determined by NMR using $\text{Eu}(\text{tfc})_3$ as a chiral shift reagent, and the absolute configuration, by X-ray analysis of one of the diastereomeric salts.¹⁹⁷

The possibility of the existence of optical isomers with asymmetric nitrogen atom in the open chain was first demonstrated²⁷ for resolution of dialkoxamines **28d**^{27,28} and **28e**^{28,29} into antipodes. It has been carried out via diastereomeric salts of (*S*)- and (*R*)-phenylethylamine with corresponding acids obtained from **28d** and **28e**. However, these isomers undergo rapid racemization at room temperature ($t_{1/2} = 5.18$ h, 20 °C, MeOH).²⁸

Sufficiently high pyramidal stability ($\Delta G^\ddagger$ 18.9 kcal/mol) and considerably hindered exchange of NH proton provided the partial separation of diastereomers of compound **58b** by its single crystallization at normal conditions.¹⁴⁰ It is the first example of the separation of optical isomers with an asymmetric nitrogen atom in NH group. The complete resolution of diastereomers of this compound is possible, probably, only at low temperature, since its half-epimerization time equals to 8.5 s at 20 °C.

VI. Application

Mutagenic activity has been found for some isoxazolidines **1**²³⁰ and benzamides **37**.^{142,143} Compounds **57**, **59**, and **67** can be used for prevention of photofading in colored materials.²³¹ **56** acts as an effective chemiluminescence energy transducing agent.¹⁵⁵

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VIII. References

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