SYNTHESIS OF HETEROCYCLES ON THE BASIS OF ALIPHATIC NITRO COMPOUNDS (REVIEW)

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The data on the synthesis of nitrogenous heterocycles on the basis of mononitroalkanes, polynitroalkanes, and alkenes have been generalized in this review. Only reactions in which the oxygen and nitrogen atoms of a nitro group appear in the composition of the heterocycle formed have been considered.

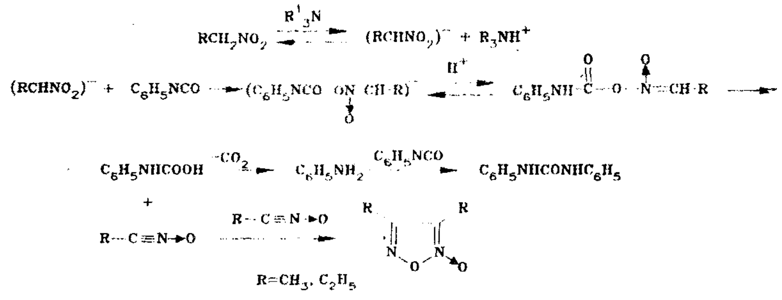
Among the numerous chemical conversions of nitro compounds, the reactions which result in the formation of heterocyclic structures occupy a fairly significant place. However, despite the fact that the first reports of the synthesis of heterocycles from nitro compounds date back to the end of the nineteenth century, only in the last 2-3 years have these reactions come to be regarded as convenient general methods for obtaining nitrogen-containing heterocycles rather than special cases in the chemistry of nitrocarbons.

The high and specific reactivity of aliphatic nitro compounds, their accessibility, and the practically unrestricted possibilities for the variation of their structure make these compounds some of the most convenient starting compounds for obtaining a broad spectrum of heterocyclic compounds. Moreover, it should be noted that in a number of cases, aliphatic nitro compounds are the only possible starting compounds for obtaining the desired heterocyclic compounds.

Out of the large variety of reactions of nitro compounds which give heterocycles, in the present review we shall consider only conversions in which the nitrogen and oxygen atoms enter the structure of a heterocycle. Our main attention will be focused on the work carried out in the period from 1960 to 1983. Unless it seemed necessary for some reason, the information on the synthesis of furoxans from aliphatic nitro compounds will be omitted, since this question was illuminated in a practically exhaustive manner in a recently published monograph [1]. We shall likewise not consider the numerous reactions resulting in the formation of cyclic structures following the reduction of ω-nitro carbonyl compounds (reductive cyclization), since in this case, the cyclization is caused by the conversions of functional groups formed as a result of the preliminary reduction of a nitro group.

MONONITROALKANES AND THEIR DERIVATIVES

In 1960 Mukaiyama and Hoshino discovered that the reactions of primary nitroalkanes with phenyl isocyanate in the presence of catalytic amounts of tertiary amines gives disubstituted furoxans, rather than the corresponding addition products, i.e., nitroanilides, and proposed the following scheme for the process [2, 3]:



When this reaction was carried out in the presence of alkenes, the corresponding isooxazolines were obtained.

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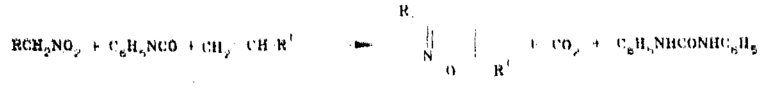
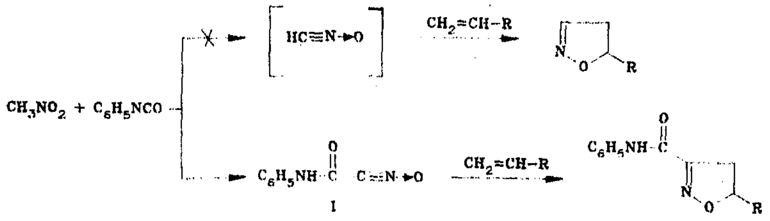


$R^1 = CH_3COO, C_6H_5$

This synthesis method was subsequently used fairly widely by many investigators for obtaining various N-containing heterocycles [4-15]. Such methods were used, for example, to carry out the synthesis of condensed heterocycles of the naphthoquinone series [4], cyclopentane-isoxazole derivatives which can be used in the synthesis of prostglandins [5], ferrocene-containing isoxazoles and isoxazolines [7, 8], oxo-1,2,3,5-oxathiadiazoles [11], and indolyl-substituted isoxazolines [14].

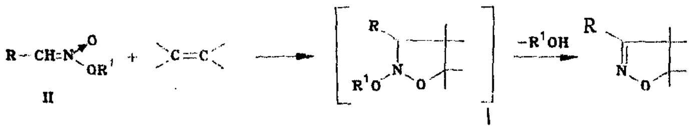
It is interesting that the use of nitromethane in these reactions resulted in the formation of products of the addition of more complicated nitrile N-oxide I (which is believed by Paul and Tshelitcheff [16] to be formed as a result of the interaction of nitromethane with phenyl isocyanate) to alkenes, rather than the expected isoxazolines without a substituent in position 3:



In the last few years it was shown that reagents such as carbodiimides [17], phosphoryl chloride [18-20], acetic anhydride [18, 21, 22], trifluoroacetic anhydride [19], acetyl chloride [23-26], and benzoyl chloride [19] can be used instead of phenyl isocyanate.

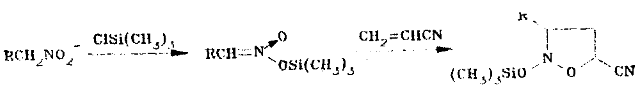
In all these reactions the formation of the heterocycles upon the interaction of the primary nitro compounds with the unsaturated compounds in the presence of phenyl isocyanate or other reagents is usually viewed as the result of the cycloaddition of the nitrile N-oxides formed from the aci form of the respective nitroalkanes under the action of these reagents to the dipolarophiles present in the reaction mixture. It should, however, be noted that nitrile N-oxides were not isolated in any of the investigations in this area and that no direct evidence of their formation has been obtained.

It seems more likely that nitronic esters II, which can themselves act as 1,3-dipoles, rather than nitrile N-oxides, form from primary nitro compounds.



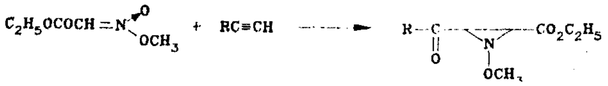
$R^1 = C_6H_5NHCO, CH_3CO, C_6H_5NHC{=}NC_6H_5$

Novikov et al. developed a convenient method for obtaining five-membered nitrogenous heterocycles on the basis of primary nitroalkanes, which was subsequently used by many investigators. According to this method, nitroalkanes are converted under the action of diazomethane [27-31], Lornblum's reagent [32-35], alkyl halides [36], trimethylchlorosilane [37-42], or bis(trimethylsilyl)acetamide [37, 38] into the corresponding O esters, which react with dipolarophiles according to a 1,3-dipolar cycloaddition scheme. In this case, the O esters can either be isolated and then used or utilized in an *in situ* reaction:

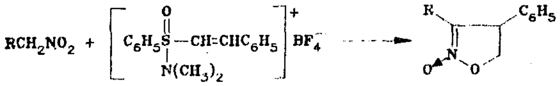


The mechanism of the cycloaddition of nitronic esters was thoroughly studied in [43-50].

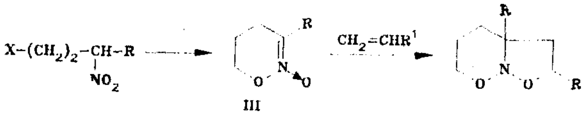
The reaction of the O-methylnitroacetic ester with substituted alkynes has a peculiar course: instead of the expected 1,3-dipolar cycloaddition products, i.e., isoxazolines, the corresponding aziridines were isolated [51]:



A way to obtain isoxazoline N-oxides from nitroalkanes and vinyloxosulfonium fluoborate was recently described [52]:

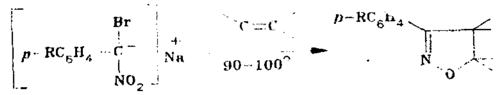


The presence of other functional groups in the molecule of a nitroalkane expands the possibilities for the synthesis of heterocycles. For example, it was shown in [53-55] that γ-halonitroalkanes readily form cyclic nitronic esters III as a result of the intramolecular nucleophilic attack of the carbon atom bonded to the halogen by the oxygen atom of the nitro group. Esters of type III can then be subjected to a 1,3-dipolar cycloaddition reaction.



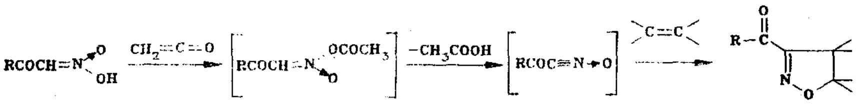
The synthesis of 3-nitroisoxazoline from the substituted propanes $X(CH_2)_3Y$ (X = I, Br, Cl, NO_2; Y = I, Br, Cl) and KNO_2 or $NaNO_2$ was recently patented [56]. The reaction probably takes place according to a scheme similar to that described above.

The sodium salts of the compounds of the α-bromophenylnitromethane series give isoxazolines in the presence of dipolarophiles upon heating [57].

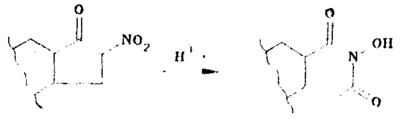


Different heterocycles can be obtained from nitro ketones, depending on their structure, as well as the reaction conditions. For example, disubstituted furoxans were synthesized by reacting benzenesulfonic acid with α-nitro ketones [58].

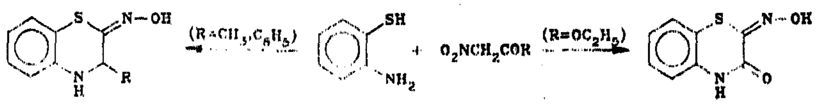
α-Nitroacetophenone reacts in an acidic medium with isopropenyl acetate to form 3-benzoyl 5-methylisoxazole with a yield greater than 60% [59]. At the same time, the corresponding isoxazole is obtained with a yield of only ~5% from α-nitroacetone under similar conditions. Nelson, Karparian, and Trager suggested that ketene is initially formed from isopropenyl acetate and then reacts with the nitro compound.



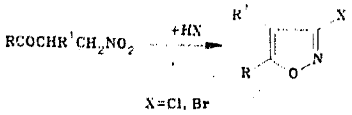
In the presence of acids, cyclic α-nitro ketones can undergo a rearrangement with ring expansion [60, 61]:



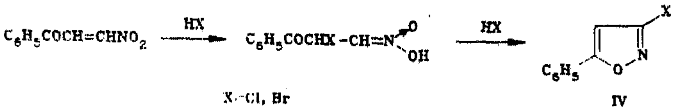
The reaction of α-nitro ketones with o-aminothiophenols gives condensed S,N-containing heterocycles [62, 63]:



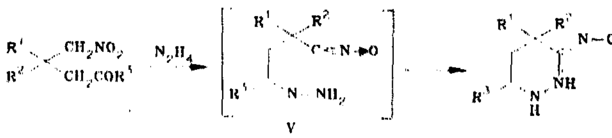
3-Halo-4,5-disubstituted isoxazoles were obtained in [64] by heating β-nitro ketones with concentrated hydrohalic acids. It was noted that the reaction takes place more readily with HBr:



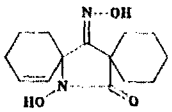
3-Halo-5-phenylisoxazoles (IV) form as a result of the reaction of phenyl β-nitrovinyl ketone with gaseous hydrogen chloride or hydrogen bromide [65].



In the opinion of Mack [66], γ-nitro ketones interact with hydrazine according to a scheme, which includes the intermediate formation of nitrile N-oxides (V):

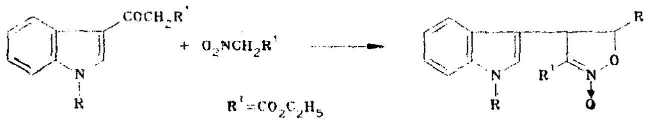


The synthesis of heterocycles from nitroalkanes and carbonyl compounds has been described. The reaction of nitromethane with cyclohexanone in the presence of pyridine is somewhat unusual in that the structure of the dispirane formed was established only many years after its synthesis [67-69]:

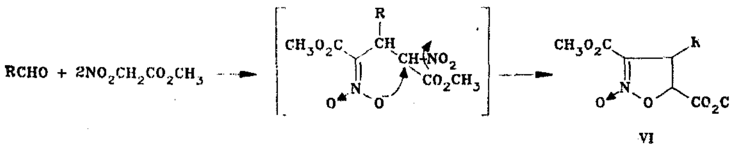


The reaction of nitromethane with cyclopentanone produces a similar structure [70].

Ethyl nitroacetate reacts with keto esters of the indole series in the presence of $POCl_3$ to form isoxazolinylindoles [71].

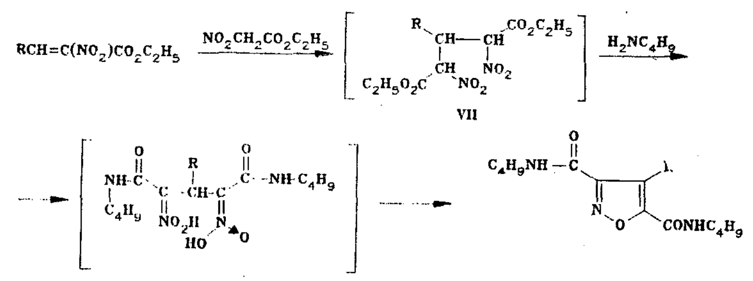


In a number of cases, the nitroalkenes formed as a result of the reaction of nitroalkanes with carbonyl compounds undergo an additional reaction with another molecule of the nitroalkane, giving β-dinitro compounds, which are readily converted into heterocycles under the conditions of the reaction. A number of investigators have suggested that the intramolecular cyclization occurs as a result of the nucleophilic replacement of one nitro group by the oxygen atom of an ambidentate nitronate ion arising from another nitro group. The possibility of the occurrence of such a reaction was pointed out by Kornblum [72]. The reaction of methyl nitroacetate with aldehydes [73], which produces isoxazoline N-oxides VI clearly takes place according to the scheme

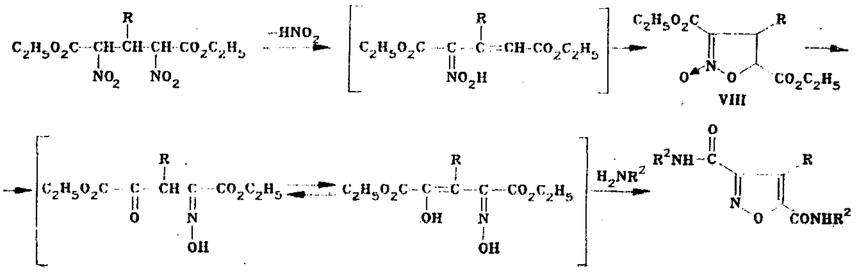


Nielsen and Archibald [74, 75] described the synthesis of 3,4,5-triphenylisoxazole from benzaldehyde and phenylnitromethane in an alkaline medium. They believe that the reaction involves the intermediate formation of 1,3-dinitro-1,2,3-triphenylpropane. Besides isoxazole, 3,4,5-triphenylisoxazoline forms with a small yield.

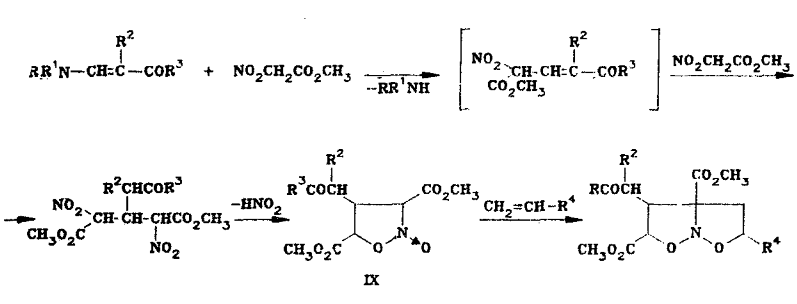
Several reports [76-79] of the synthesis of isoxazoles from nitroalkanes and α,β-unsaturated α-nitro esters have been published. The reaction clearly takes place according to a scheme which includes the intermediate formation of dinitro derivative VII:



Ethyl nitroacetate reacts also with several azomethines to form disubstituted isoxazoles Zen and Umezawa [78] suggested that the reaction also involves the intermediate formation of a dinitro derivative. An alternative scheme for the synthesis of an isoxazole ring from β-dinitro compounds, which includes the intermediate formation of cyclic nitrone ester VIII, was proposed in [79]:

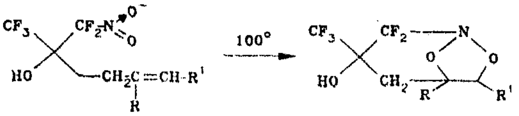


Such a course of the reaction is indirectly confirmed by the fact that in the case of the reaction of ketoenamines with ethyl nitroacetate, intermediately formed nitronic ester IX was isolated in the form of adducts with olefins [80, 81]

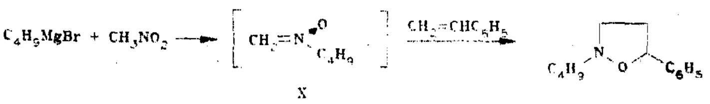


When 1-nitropropane was reacted with 2-nitro-1-butene, 3,5-diethylisoxazole, which probably forms as a result of the intramolecular cyclization of dinitroalkane, was isolated along with the expected 3,5-dinitroheptane [82, 83].

An unusual case of intramolecular heterocyclization with the participation of both oxygen atoms of the nitro group was described in [84]:

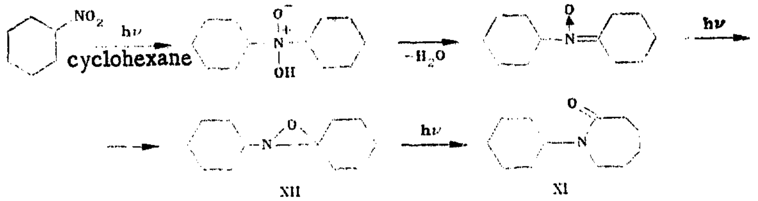


The reaction of butylmagnesium bromide with nitromethane in the presence of styrene gives isoxazolidine. It has been postulated [85] that the reaction involves the preliminary formation of nitrone X:



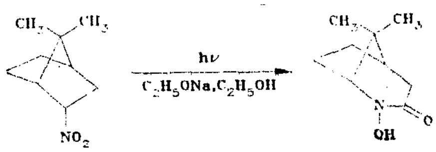
The reaction of 1-chloro-1-nitrocycloalkanes with triphenylphosphine results in the formation of lactams after a fairly complex series of conversions [86]:

N-cyclohexylcaprolactam (XI) was obtained as a result of the UV irradiation of nitrocyclohexane [87]:

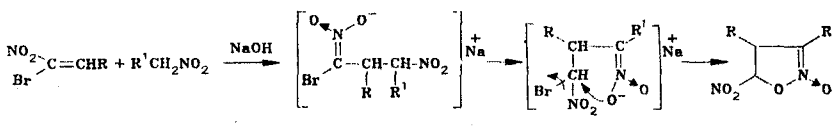


In order to confirm such a course for the reaction, oxaziridine XII obtained by an independent method was converted practically quantitatively into N-cyclohexylcaprolactam under irradiation [87].

The irradiation of a bicyclic nitroalkane results in ring expansion with the formation of an hydroxylactam [88].

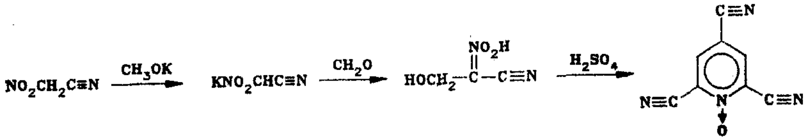


The reaction of nitroacetonitrile or methyl nitroacetate with 1-bromo-1-nitroalkenes produces 4-nitroisoxazoline N-oxides [89].

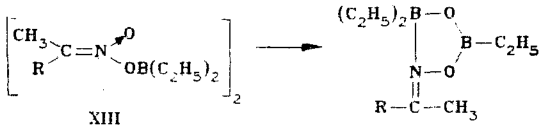


Novikov et al. [90, 91] obtained derivatives of 2-hydroxy-4,5-dihydro-1,2,3-oxadiazole as a result of the treatment of N-nitro amino compounds with bases.

A method -or the synthesis of 2,4,6-tricyanopyridine N-oxide on the basis of nitroacetonitrile was proposed in [92]:

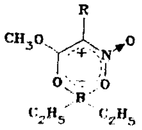


The reaction Na salts of secondary [93] or primary [94] nitroalkanes with diethylboron halides results in the formation of dimeric esters XIII, which are converted upon storage intc 2,5,5-triethyl-4-alkylidene-1,3-dioxa-4-aza-2,5-diboracyclopentanes:



XIII

α-Nitro esters of carboxylic acids react with diethylboron chloride to form heterocyclic compounds of type XIV [95]:

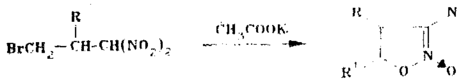


XIV

DINITROALKANES AND THEIR DERIVATIVES

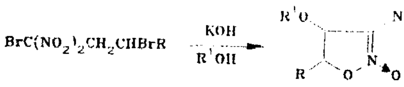
Compounds containing the $-C(NO_2)_2H$ grouping are converted fairly smoothly under the influence of various reagents into 3-nitroisoxazoline N-oxides, which are capable of undergoing a 1,3-dipolar cycloaddition reaction.

3-Nitroisoxazoline N-oxides were obtained with good yields when potassium acetate was reacted with γ-bromodinitro compounds in [96-99]. Aqueous potassium hydroxide or potassium methoxide can be used instead of potassium acetate [97]:



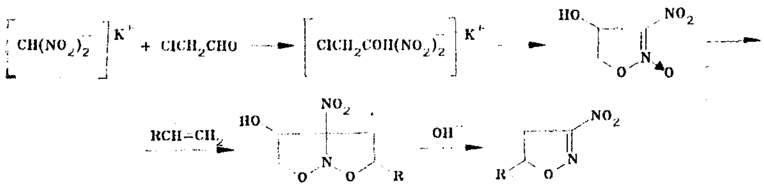
$R=H, CH_3, C_6H_5, R^1=H; R-R^1=-(CH_2)_n-$ $(n=3,4)$

Alkoxylated and carboxylated 3-nitroisoxazolines have been obtained from the corresponding α,γ-dibromo derivatives [100]:



$R=H, CO_2CH_3; R'=CH_3, C_2H_5$

The reaction of the K salt of dinitromethane with chloroacetaldehyde results in the formation of 4-hydroxy-3-nitroisoxazoline N-oxide [101].



XV

The adducts of this N-oxide with a number of olefins (XV) are easily split under the action of bases and converted into derivatives of 3-nitroisoxazoline.

Compounds of the 3-nitroisoxazoline series have been obtained from the silver salts of dinitromethane and 1,1-dinitroethane when they are reacted with primary alkyl halides in the presence of alkenes [102]:

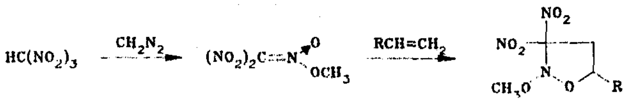
The O ester formed from dinitromethane and trimethylchlorosilane readily undergo a 1,3-dipolar cycloaddition reaction to form substituted isoxazolines [37].

The thermal decomposition of the potassium salt of phenyldinitromethane in the presence of alkenes gives 3-phenyl-5-alkylisoxazolines [103, 104].

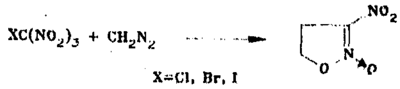
The synthesis of 2,4,6-trinitropyridine from 2,2-dinitroethanol was described in [105].

1,1,1-TRINITRO COMPOUNDS

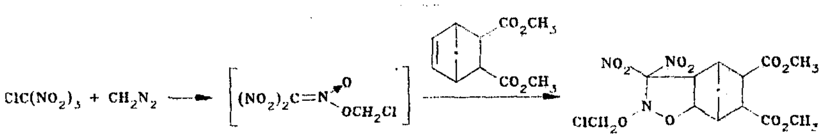
Trinitromethane, like mono- and gem-dinitro compounds, reacts with diazomethane to form an O-methyl ester, which is a reactive 1,3-dipole [27, 106, 107]:



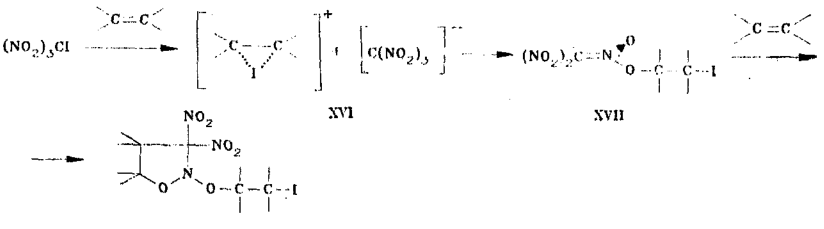
Halotrinitromethanes react vigorously and sometimes explosively in an ethereal solution with diazomethane at temperatures from −10 to −15°C with the formation of 3-nitroisoxazoline N-oxide as the main product [108]:



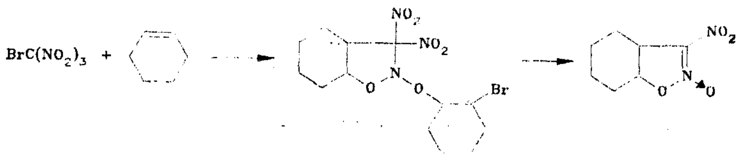
The reactions of halotrinitromethanes with other diazo compounds of the aliphatic series have a similar course [109]. In [110] it was postulated that the intermediates of the reaction of halotrinitromethanes with diazomethane include halomethyl esters of nitronic acid. Such a conclusion is supported by the isolation of the corresponding adduct when the reaction is carried out in the presence of an active dipolarophile:



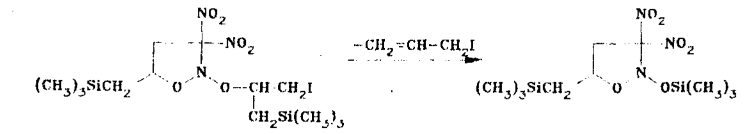
Iodotrinitromethane reacts with alkenes to form isoxazolidines. Some investigators consider the first step of the reaction to be the alkylation of the anion of trinitromethane by cations of the π-complex type (XVI). The formation of the heterocycle occurs as a result of the reaction of iodoalkyl nitronate XVII with another molecule of the alkene [111, 112]:



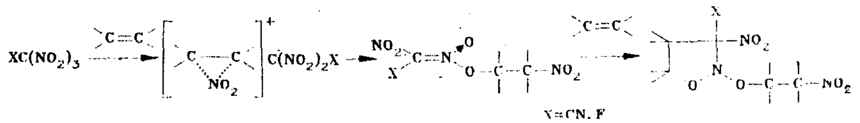
When bromotrinitromethane is reacted with cyclohexene, it forms 4,5-tetramethylene-3-nitroisoxazoline N-oxide with a small yield, presumably according to the scheme [113]



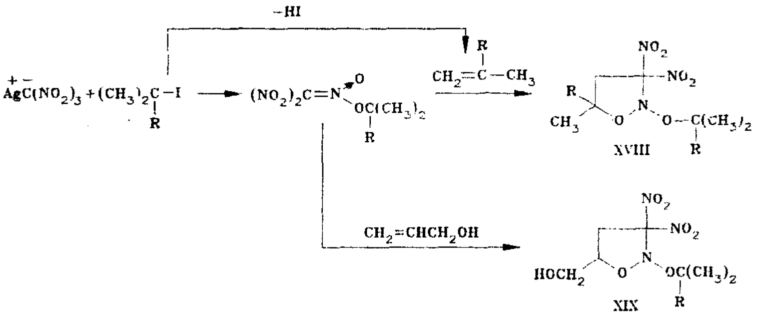
Vinyl- and allyltrimethylsilane can serve as alkenes in the reaction [114, 115]. It is interesting that in the case of allyltrimethylsilane, the N-alkoxyisoxazolidine formed is unstable and undergoes β-decomposition under the conditions of the reaction:



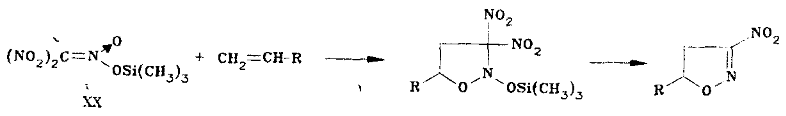
Trinitroacetonitrile [116] and fluorotrinitromethane [117] react with alkenes according to a similar scheme; however, under these conditions the nitro group participates in the formation of the ion pair in these cases, and the reaction product is a cyano- or fluoroisoxazolidine:



Dinitroisoxalidines of type XVIII have been obtained by reacting the silver salt of trinitromethane with sec- and tert-alkyl halides. The heterocycles are adducts of the initially formed nitronic esters with alkenes, which are obtained as a result of the dehydrohalogenatior of the original alkyl halides. When a foreign dipolarophile is introduced into the reaction sphere, the corresponding heterocycle (XIX) is recovered [118, 119]:



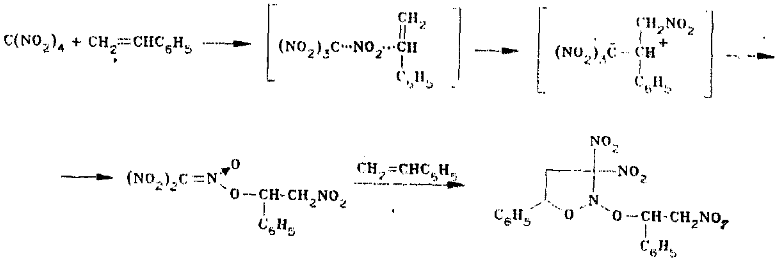
Trinitromethane, its silver salt, and its mercury salt form trimethylsilyl esters XX, which readily undergo 1,3-dipolar cycloaddition; the 5-substituted 2-trimethylsiloxy-3,3-dinitroisoxazolidines formed can be converted into 3-nitroisoxazolines [120-122]:



The reaction of dilute mineral acids with 1,1,1,3-tetranitroalkanes gives 3,5-dinitroisoxazoles [123].

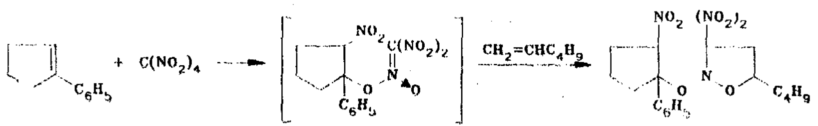
TETRANITROMETHANE

In 1967 Perekalin and Altukhov [124] described the reaction of tetranitromethane with ethylene, which produces 3,3-dinitro-2-(2-nitroethoxy)isoxazolidine. In [125] the mechanism of the reaction was discussed in the example of the interaction of tetranitromethane with styrene, and the similarity of this reaction to the reaction between halotrinitromethanes and alkenes was pointed out [111]:



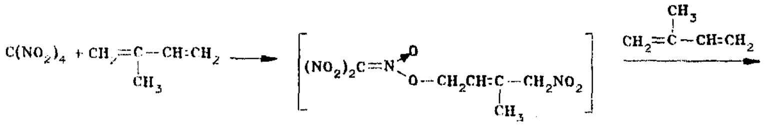
Cyclohexene also reacts with tetranitromethane to form the corresponding gem-dinitroisoxazolidine [113].

The intermediate formation of the nitronic ester of trinitromethane XXI during the reaction with cycloalkenes was demonstrated by isolating the corresponding cycloadduct when the reaction was carried out in the presence of hexene [126]:

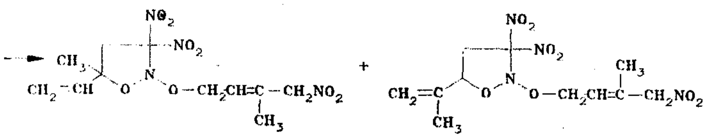


XXI

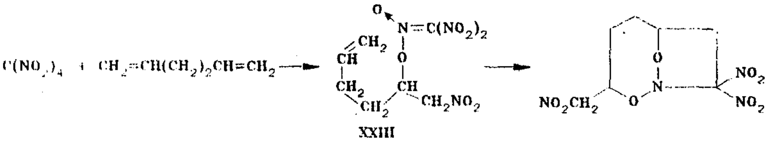
Adducts of tetranitromethane and cumulenes could not be isolated in [127]. When it is reacted with conjugated dienes, the reaction has a course similar to that of the reaction with alkenes [127, 128]. In this case, O esters are intermediately formed as a result of the addition of tetranitromethane to the diene in positions 1 and 4 (butadiene) and positions 1 and 2 (piperylene). 1,4-Addition takes place with isoprene, the nitro group being added in position 1. The nitronic ester formed (XXII) reacts nonselectively with a second molecule of isoprene, giving a mixture of two adducts:



XXII



The unconjugated diene 1,5-hexadiene reacts with tetranitromethane to form a bicyclic system as a result of the intramolecular cycloaddition of initially formed O ester XIII to the second double bond [128]:

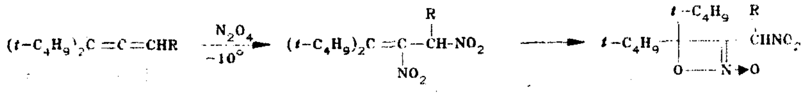


XXIII

In general, the reactions of tetranitromethane with alkenes can take place in two directions: addition at the double bond with the formation of tetranitroalkanes or O-alkylation with the subsequent formation of a heterocycle. The influence of the electronic effects of the substituents in the alkene molecule on the direction of the reaction was studied in [129].

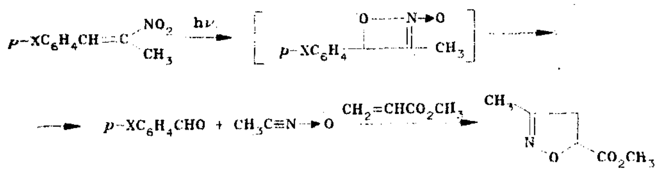
A number of examples of the readily occurring intramolecular heterocyclization of nitroalkenes as a result of the interaction of the nitro group with the double bond have been described. For example, the formation of 4,4-di-tert-butyl-3-methyl-4H-1,2-oxazete N-oxide from the corresponding nitroalkene takes place spontaneously [130]:

1,2-Oxazete N-oxides are also produced by the reactions of 1,1-di-tert-butylallenes with nitrogen tetroxide, which involve the intermediate formation of dinitroalkenes XXIV [131]:

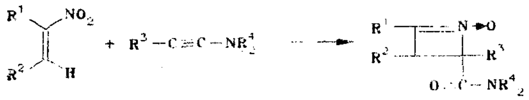


XXIV

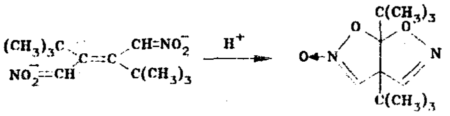
The formation of substituted 1,2-oxazete N-oxides XXV as intermediates was postulated in the photochemical reaction of β-nitrostyrenes [132]. The opening of the oxazete ring results in the formation of an aldehyde and acetonitrile N-oxide, which is isolated in the form of an adduct with methyl acrylate.



Four-membered cyclic nitrones form when nitroalkenes are reacted with ynamines [133, 134]:

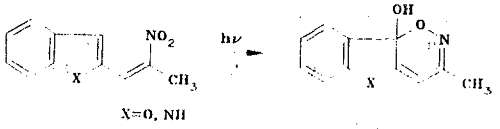


Acidification of a salt of 2,3-di-tert-butyl-1,4-dinitro-2-butene produces 3a,6a-di-tert-butyl-7,8-dihydroisoxazolo[5,4-d]isoxazole N-oxide with a good yield [135]

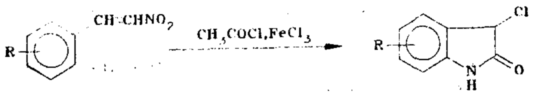


There is some indication that 3-nitroisoxazoline N-oxide forms from 4,4-dinitrobutenoic acid [136].

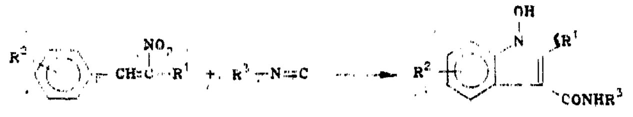
The UV irradiation of nitrovinyl derivatives of indole and benzoruran produces structures containing a 1,2-oxazine ring, generally with high yields [137]:



β-Nitrostyrenes form indolones under the action of acetyl chloride in the presence of ferric chloride [138-140]:

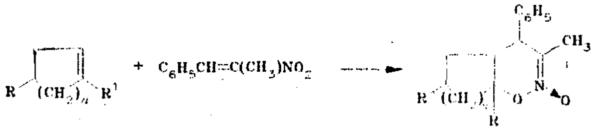


An unusual path for the synthesis of indole derivatives from nitrostyrenes and isonitriles has been described [141]:

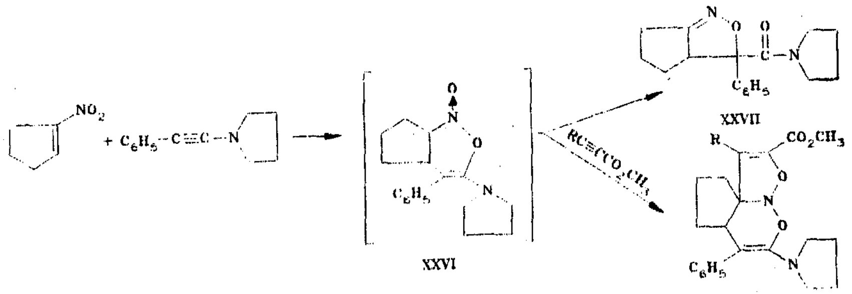


1,3-Cyclohexanedione reacts with nitrostyrene to form a heterocycle, for which various structures have been proposed: either a cyclic nitronic ester (n-hydroxy-4-phenyl-5,6,7,8-tetrahydro-4H-1,2-benzoxazine-5-one) [142], or 4-phenyl-3,4,5,6,7,8-hexahydro-2H-1,2-benzoxazine-3,5-dione [143], or an N-hydroxylactam (1-hydroxy-3-phenyl,2,3,4,5,6,7-hexahydroindole-2,4-dione) [144].

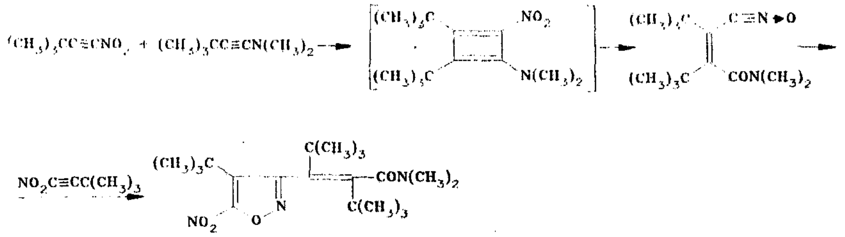
A (4 + 2)-cycloaddition reaction is characteristic of conjugated nitroalkenes. For example, nitrostyrenes react with alkenes [145, 146] or enamines [147-149] according to such schemes to form cyclic nitronic esters.



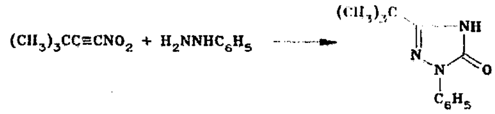
When 1-nitrocyclopentene is reacted with ynamines, it is converted into unstable cyclic nitronic ester XXVI; the latter readily undergoes isomerization to isoxazolines XXVII and may undergo 1,3-dipolar cycloaddition [150, 151]:



The reaction of nitro-tert-butylacetylene with ynamines, which involves the intermediate formation of a stable nitrile N-oxide, has been described [152].



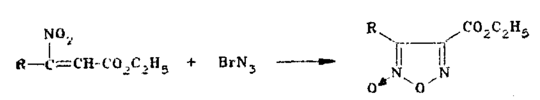
The reaction of nitro-tert-butylacetylene with phenylhydrazine gives triazoline [153]:



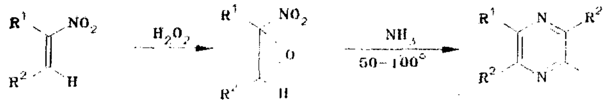
Derivatives of 3-nitroisoxazoline N-oxide have been obtained from some derivatives of diazomethane and gem-dinitroalkenes [154, 155]:

1,2-Dinitroalkenes react according to a similar scheme [156].

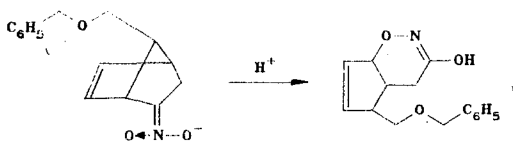
Unsaturated nitro esters react with bromine azide to form furoxans [157]:



A method for the synthesis of pyrazines from nitroalkenes according to the following scheme was proposed in [158]:

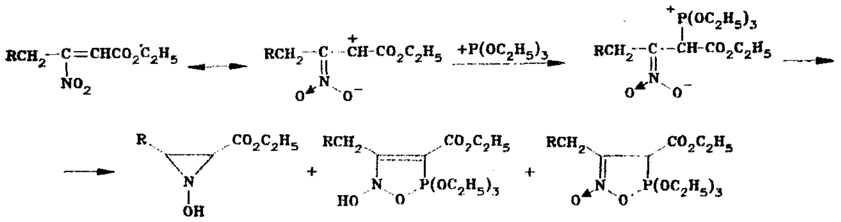


An unusual reaction takes place when a salt of bicyclic nitroalkene XXVIII is acidified [159]:

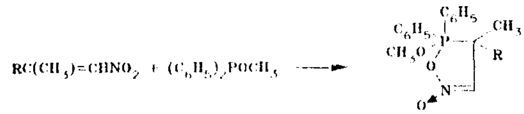


XXVIII

The formation of five-membered O,N,P-containing heterocycles is a characteristic reaction of various nitroalkenes with derivatives of trivalent phosphorus. For example, when esters of unsaturated β-nitro acids are reacted with triethyl phosphite, a mixture of three- and five-membered heterocycles forms [160, 161]:



The reaction of nitroalkenes with diphenylmethoxyphosphine, which gives the corresponding heterocycles, has been studied in detail [162-165]:



Conjugated nitroalkenes react at positions 1 and 4 with $C_6H_5P(OCH_3)_2$ [166, 167], $RP(OR)_2$ [168], $P(OCH_2CH_2Cl)_3$ [169], and $P(OCH_3)_3$ [170] according to a similar scheme. The mechanism of the reaction was considered in the example of the reaction of nitrostyrene with $P(OCH_3)_3$ in [171].

The material presented in this review demonstrates the great variety of the chemical conversions of nitro compounds which result in the formation of heterocyclic systems. It should be noted that in a number of cases, the conversions take place according to the same pattern, apparently according to similar mechanisms with the formation of heterocycles of similar structure. For example, the reactions involving the cycloaddition of 1,3-dipoles formed from nitro compounds and the intramolecular cyclizations occurring as a result of the attack of an oxygen atom of a nitro group on an electron-deficient carbon atom may be included among such reactions which have become preparative methods for the synthesis of heterocycles. In the latter case, various fragments of the molecule can act as the "leaving group" when the ring is closed. It should be noted that a similar attack and the formation of a heterocycle are not always accompanied by an elimination reaction. It is more commonly clearly observed in the formation of heterocycles as a result of the interaction of tri- and tetranitro derivatives with alkenes. At the same time, in a whole series of cases, the type of conversion occurring upon heterocyclization does not fit any general scheme, and in some cases, the reactions presented are still the only examples of the formation of the structure indicated. Nevertheless, we felt it was necessary to present some experimental data of this kind without further discussion, assuming that such material would be interesting and useful to chemists becoming familiar with this area in organic chemistry.

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SOME REACTIONS OF 3,7-DIMETHYL-2,3-EPOXYOCTANAL AND ITS DERIVATIVES

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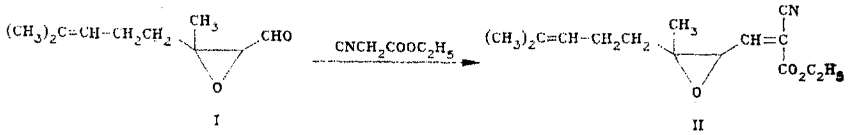
The oxide of citral (3,7-dimethyl-2,3-epoxioctanal) readily enters into the Knoevenagel condensation with the formation of the corresponding epoxydienecarboxylic acid. The epoxidation and addition of dichlorocarbene to similar epoxydienes proceeds at the double bond which does not adjoin the epoxide ring. The condensation of the acetal of citral oxide with ketones with the formation of 1,3-dioxolanes proceeds with the participation of the epoxide ring.

We have previously studied several reactions of citral oxide: the Darzan reaction, the Wittig reaction, acetalization, etc. [1]. Continuing the work in this field, we accomplished the Knoevenagel condensation of citral oxide (I) with ethyl cyanoacetate in the presence of catalytic amounts of piperidine; the condensation only affects the carbonyl group. An attempt to perform this reaction under the conditions of interphase catalysis (50% aqueous solution of NaOH, TEBA) proved to be unsuccessful.



Absorption bands in the region of 1610 ($C=C$), 860, 1240 (epoxide ring), 2180 (CN), and 1730 cm^{-1} (COOEt) were present in the IR spectrum of the resulting epoxydienic compound II. In the PMR spectrum, the signal of the aldehyde proton disappears, and the signals of the ethyl group appear; all the remaining signals are retained (see the Experimental section).

We performed the epoxidation of 6,10-dimethyl-5,6-epoxy-3-ethoxycarbonylundeca-3,9-dien-2-one (III), an analog of the diene II, using monoperphthalic acid according to the method of [2]. We performed the addition of dichlorocarbene to III using dichlorocarbene generated under the conditions of interphase catalysis by the method of [2]. We previously synthesized III by the condensation of citral oxide with acetoacetic ester. In both cases, the attack only proceeds at the isolated double bond which does not adjoin the epoxide ring and the acceptor groupings, and possesses higher nucleophilicity. The formation of the respective compounds IV and V is indicated by the disappearance of the signals of the vinyl group in the PMR spectrum and the appearance of the signals of the new epoxide ring [2.16 ppm for IV] or the dichlorocyclopropane ring [2.3 ppm for V], while the signals of all the remaining protons are retained. (Formula, below table, following page.)

On the reaction of the acetal of citral oxide VI, which was obtained by the acetalization of citral oxide with orthoformic ester according to [1], with ketones in the presence of an acid catalyst, the opening of the epoxide ring occurs with the formation of the correspond-

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