SYNTHESIS AND PROPERTIES OF 1,2,3-TRIAZOLE 1-OXIDES (REVIEW)

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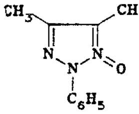
Data on the preparation, properties, and use of uncondensed 1,2,3-triazole 1-oxides are correlated for the first time.

1,2,3-Triazole 1-oxides and their derivatives, which have several reaction centers, are starting substances for a number of compounds. They are also of practical interest from the point of view of their use in industry and agriculture.

Up until now there have been no reviews devoted to the N-oxides of vicinal triazoles, although the first reports regarding these compounds were published in 1898 [1, 2]. Systematized data on 1,2,3-triazoles are presented in [3-7], but only very little space is devoted to their N-oxides.

Initially, for the first representative of compounds of this type, viz., 4,5-dimethyl-2-phenyl-1,2,3-triazole 1-oxide, Ponzio [1, 2] proposed structural formulas I and II, while in subsequent publications [8-12] preference was given to bicyclic structure II. Formula III, with an exocyclic oxygen atom bonded by means of a semipolar bond, is currently generally accepted for substituted triazole N-oxides.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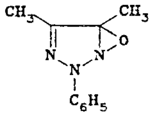
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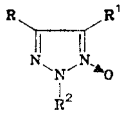
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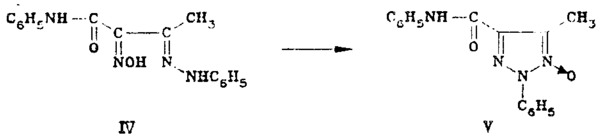
1. SYNTHESIS OF 1,2,3-TRIAZOLE 1-OXIDES

1.1. Oxidative Cyclization of Hydrazonoximes of 1,2-Dicarbonyl Compounds

This method has been studied most extensively and is utilized widely. Precisely by means of this method 4,5-dimethyl-2-phenyl-1,2,3-triazole 1-oxide was synthesized in 25% yield for the first time by oxidation of diacetyl phenylhydrazonoxime with nitrogen tetroxide [1, 2]. The use of yellow mercuric oxide as the oxidizing agent made it possible to increase the yield to 75% [1]. The structure of the compound isolated was confirmed by its conversion to the previously known 4,5-dimethyl-2-phenyl-triazole via reduction.

The oxidation of hydrazonoximes is general in character and makes it possible to obtain, using yellow mercuric oxide [13-15] or copper(II) sulfate [15-32], 1,2,3-triazole 1-oxides with various substituents of aliphatic and aromatic character attached to both the carbon atoms and to the $N(_2)$ atom of the triazole ring.

The reactivities of a number of oxidizing agents were investigated in the case of the conversion of hydrazonoxime IV to the corresponding triazole N-oxide [20]:

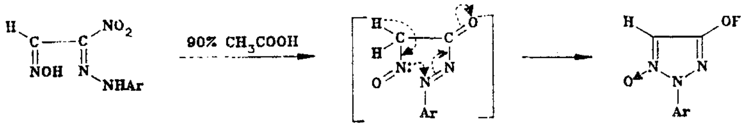

IV V

N. D. Zelinskii Institute of Organic Chemistry, Academy of Sciences of the USSR, Moscow 117913. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 2, pp 147-156, February, 1989. Original article submitted October 12, 1987; revision submitted March 22, 1988.

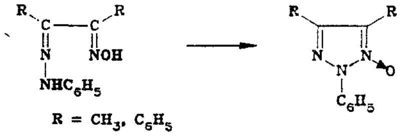
As a result of a comparison of the data obtained, the oxidizing agents were arranged in the following order with respect to activity: $CuSO_4 < PbO_2 \sim MnO_2 < FeCl_3 < K_3[Fe(CN)_6]$. It was found that the use of one-electron oxidizing agents such as $FeCl_3$ and $K_3[Fe(CN)_6]$ makes it possible to obtian the desired product in 45-60% yield, while the triazole N-oxide is formed in 15-35% yield with two-electron oxidizing agents ($CuSO_4$, PbO_2, and MnO_2).

Oxidizing agents such as hydrogen peroxide [20, 25], pyrosulfuric acid [20], nickel oxide [33], and iodosuccinimidae [34] are also used to form an N-oxidotriazole ring from hydrazonoximes of 1,2-dicarbonyl compounds.

Hydrazonoximes that contain a nitro group undergo transformations similar to the Neff reaction at 20-30°C in the presence of an aliphatic acid (90% acetic acid is best) to form 4-hydroxy-1,2,3 triazole 1-oxides [35-40].

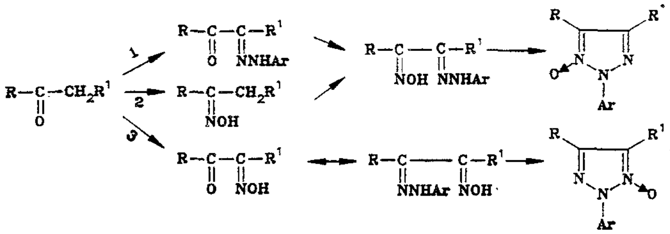


An electrochemical method is also used for the oxidation of phenylhydrazonoximes. Thus the corresponding 4,5-dimethyl- [41] and 4,5-diphenyltriazole 1-oxides [41, 42] were obtained in good yields by anode oxidation.

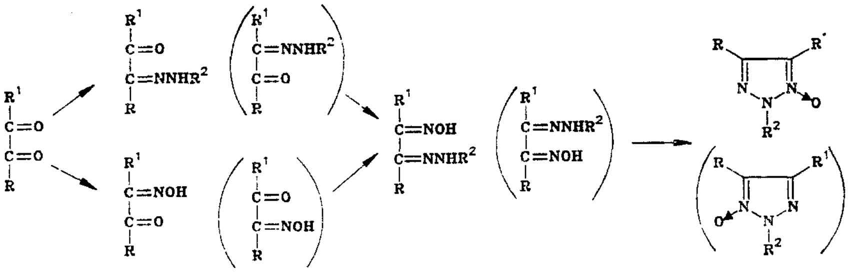


In [16, 17], which are devoted to 1,2,3-triazole 1-oxides, general schemes for their preparation starting from mono- and dicarbonyl compounds, are presented.

The following pathways for the synthesis of triazole N-oxides are possible when ketones are used as the starting substances:



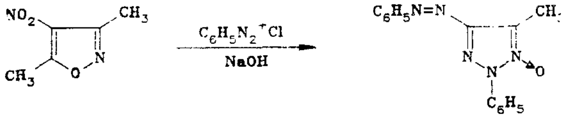
If one proceeds from 1,2-dicarbonyl compounds, two pathways that lead to N-alkyl- [16] and N-aryltriazole N-oxides [17] are obvious.



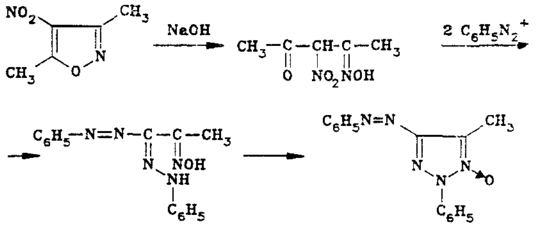
The reactions that are presented in the schemes can also proceed without isolation of the intermediately formed products. The formation of isomeric 1,2,3-triazole 1-oxides is possible when unsymmetrically substituted dicarbonyl compounds are used.

1.2. Other Methods for Obtaining Vicinal Triazole N-Oxides

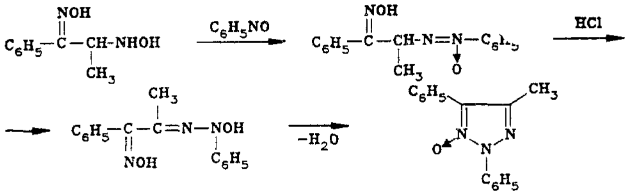
The reaction of 3,5-dimethyl-4-nitroisoxazole with benzene-diazonium chloride in the presence of alkali leads to the formation of 5-methyl-2-phenyl-4-phenylazo-1,2,3-triazole 1-oxide [43].



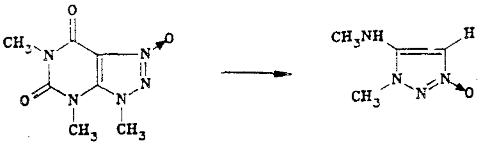
The proposed mechanism suggests opening of the isoxazole ring with the subsequent addition of two molecules of the diazo compound, which is accompanied by splitting out of the acetyl and nitro groups [43].



The reaction of the syn isomer of an α-hydroxyamino oxime with nitrosobenzene gives the α-azoxy oxime, which is converted to the corresponding vicinal triazole N-oxide under the influence of gaseous hydrogen chloride [13]. It is assumed that the formation of the triazole N-oxide commences with inversion of the configuration of the oxime group with subsequent splitting out of a molecule of water. This reaction does not take place with the anti-isomer of the starting compound.



The synthesis of 3-methyl-4-methylamino-1,2,3-triazole 1-oxide (VII) from condensed triazole N-oxide VI has been described [44].



VI VII

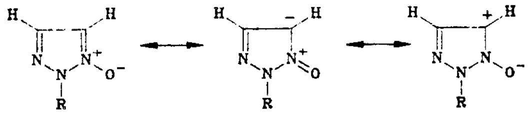
Attempts to oxidize 2-phenyl-1,2,3-triazole with peracetic, trifluoroperacetic, dichloropermaleic, m-chloroperbenzoic, and pyrosulfuric acids or tert-phenyl hydroperoxide in the presence of molybdenum pentachloride to the corresponding triazole N-oxide did not lead to positive results [17].

2. CHEMICAL PROPERTIES

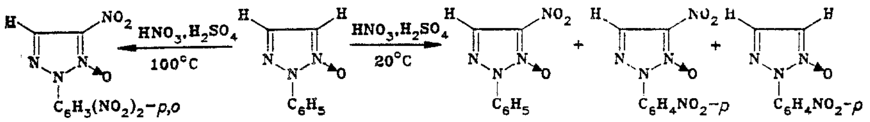
The reactions of triazole N-oxides can be arbitrarily divided into four groups: 1) reactions with retention of the 1,2,3-triazole 1-oxide ring; 2) reactions at the N-oxide oxygen atom without its detachment from the ring; 3) reactions with detachment of the oxygen atom of the N-oxide fragment – reduction to 1,2,3-triazoles; 4) recyclization to other nitrogen-containing heterocycles.

2.1. Reactions of 1,2,3-Triazole 1-Oxides with Retention of the Ring and the N-Oxide Fragment

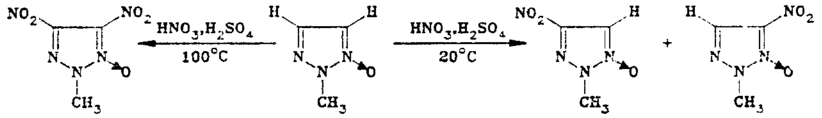
2.1.1. Electrophilic and Nucleophilic Substitution. These reactions were examined in detail in [16, 17]. It is assumed that in 2-substituted vicinal triazole N-oxides the oxide fragment activates the adjacent carbon atom to both electrophilic and nucleophilic attack. This is explained by the fact that both negatively and positively charged atoms in the 5 position are contained in the resonance structures of such compounds [17].



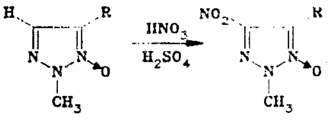
Electrophilic substitution for 1,2,3-triazole 1-oxides has been studied in the case of nitration and halogenation. Depending on the reaction conditions, the reaction of unsubstituted 2-phenyltriazole 1-oxide with a mixture of concentrated nitric and sulfuric acids leads to products of nitration of the N-oxidotriazole and benzene rings [17].



Whereas in 2-phenyltriazole oxide the triazole ring is nitrated only in the 5 position, the analogous reaction with 2-methyl-1,2,3-triazole 1-oxide under mild conditions (20°C) proceeds with the formation of isomeric 4- and 5-mononitro derivatives in a ratio of 1:3; under severe conditions (100°C) 4,5-dinitro-2-methyl-1,2,3-triazole 1-oxide is obtained in 77% yield [16].

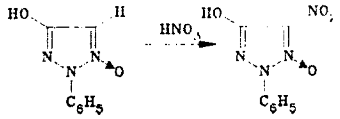


Under the influence of a mixture of concentrated nitric and sulfuric acids 5-substituted 2-methyl-1,2,3-triazole 1-oxides are converted to 4-nitro derivatives [16].

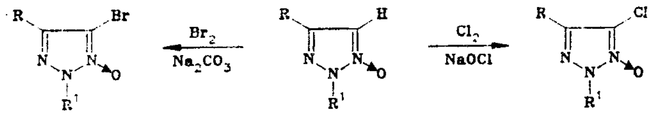


R = CH_3, Br

4-Hydroxy-2-phenyl-1,2,3-triazole 1-oxide is nitrated by nitric acid only in the triazole ring [40].



The bromination and chlorination of the 4-methyl derivatives of 2-methyl- [16], 2-phenyl- [17], and 2-benzyltriazole 1-oxide [16], as well as 2-substituted 4-hydroxy-1,2,3-triazole 1-oxides [36], proceeds in the 5 position. 4,5-Unsubstituted triazole oxides are halogenated selectively in the same position; the reaction does not take place in the 4-position even at 60°C [16, 17].



R=H, CH_3, OH; R^1=CH_3, C_6H_5, $CH_2C_6H_5$

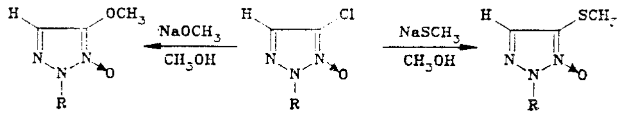
N-Bromo- and N-chlorosuccinimide [36, 37], sulfuryl chloride [29], and thionyl chloride [22] have also been used as halogenating agents.

Thus the halogenation of 1,2,3-triazole 1-oxides leads to 5-halo derivatives, while the formation of both the 4-nitro and 5-nitro isomers is possible in the case of nitration.

A comparison of the reactivities of triazoles and their N-oxides showed that the introduction of an N-oxide fragment activates the molecule to electrophilic substitution reactions. Thus 2-methyl-1,2,3-triazole is nitrated to the corresponding dinitro derivatives by a mixture of concentrated nitric and sulfuric acids at 100°C after 10 h, while under these conditions

2-methyl-1,2,3-triazole 1-oxide is converted to 4,5-dinitro-2-methyl-1,2,3-triazole 1-oxide after 30 min [16]. The bromination of 2-methyl-1,2,3-triazole proceeds at 90-100°C and takes 15 h [45], while the reaction of its N-oxide with bromine at 0°C takes 3 h [16].

Nucleophilic substitution in the 1,2,3-triazole 1-oxide series has been studied in the case of the reaction of 5-chloro derivatives of 2-phenyl- and 2-benzyl-1,2,3-triazole 1-oxides with sodium methoxide and lithium and sodium methylthiolates [16,17].



R = C_6H_5, $CH_2C_6H_5$

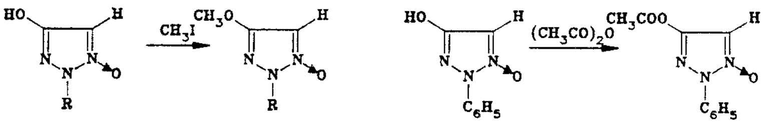
Sodium methoxide reacts more slowly than sodium methylthiolate, while a weak nucleophile such as thioacetate does not react at all with the starting compounds.

Comparing these results with the known data that 4-bromo-2-methyl-1,2,3-triazole does not react with a 5% alcohol solution of ammonia [46], Begtrup and Nytoft [16] assumed that 2-substituted 5-halo-1,2,3-triazole 1-oxides are more active with respect to nucleophilic reagents than halogen-containing 1,2,3-triazoles.

In our opinion, this comparison is incorrect, since it was made with respect to compounds with different structures and with different nucleophiles.

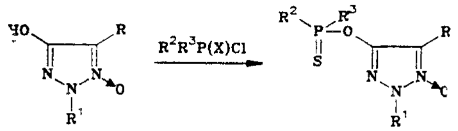
2.1.2. Reactions with the Participation of Substituents in the Triazole N-Oxide Ring. As we have already noted (see subsection 2.1.1), nitration of both the N-oxidotriazole ring and the benzene ring is observed in the action of a mixture of concentrated nitric and sulfuric acids on 2-phenyl-1,2,3-triazole 1-oxide [17]. In the reaction of 4-mono- and 4,5-disubstituted 2-phenyltriazole 1-oxides with this nitrating agent or with concentrated nitric acid the nitro group goes into the para position of the benzene ring [1, 2, 9-11].

Methyl iodide methylates the hydroxy group in 4-hydroxy derivatives of 2-(p-tolyl)- [19] and 2-phenyl-1,2,3-triazole 1-oxide [40], whereas the reaction of the latter with acetic anhydride leads to the formation of the corresponding acetate [40].



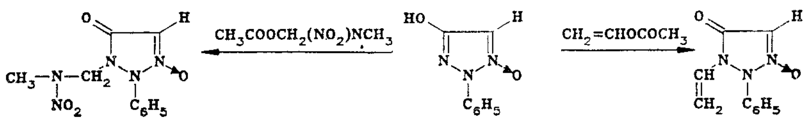
R = C_6H_5, p-$CH_3C_6H_4$

The reactions of other hydroxytriazole N-oxides with phosphorus and thiophosphorus compounds proceed similarly [47].



R=H, Cl, Br; R^1=C_6H_5, aryl R^2=CH_3NH, $(CH_3)_2N$, CH_3O, C_2H_5O; R^3=CH_3O, C_2H_5O, $(CH_3)_2N$; X=O, S

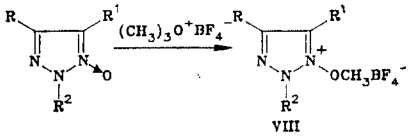
4-Hydroxy-2-phenyl-1,2,3-triazole 1-oxide reacts with vinyl acetate and acetoxynitrazapropane in the keto form [40].



2.2. Reactions of 1,2,3-Triazole 1-Oxides at the N-Oxide Oxygen Atom without Its Detachment from the Ring

Vicinal triazole N-oxides are weak bases, and some of them, depending on the nature of the substituents, give salts with hydrochloric acid [1, 2, 10-12].

Methylation of the N-oxide oxygen atom with the formation of 1-methoxytriazolium tetrafluoroborates VIII occurs in the reaction of substituted 1,2,3-triazole 1-oxides with trimethyloxonium tetrafluoroborate [16-18].



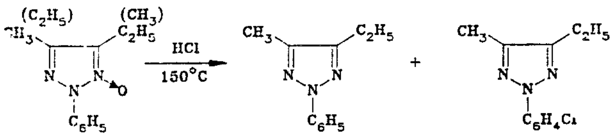
$R = H, CH_3$; $R^1 = H, CH_3, SCH_3, Cl$; $R^2 = C_6H_5, CH_2C_6H_5$

High reactivities of salts VIII in both nucleophilic and electrophilic substitution reactions have been demonstrated; this makes it possible to synthesize difficult-to-obtain 1,2,3-triazole 1-oxides and triazoles [16-18].

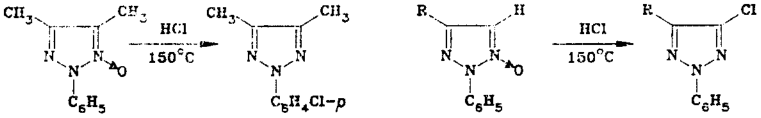
2.3. Reduction of 1,2,3-Triazole 1-Oxides to 1,2,3-Triazoles

1,2,3-Triazole 1-oxides are reduced to triazoles on reaction with hydrohalic acids. This reaction proceeds ambiguously; depending on the structure of the starting compound, halogenation of the substituent or the ring may also occur in addition to reduction.

Thus the reactions of isomeric methyl(ethyl)phenyltriazole oxides with concentrated hydrochloric acid by heating in sealed ampuls lead to the formation of 4-methyl-2-phenyl-5-ethyl-1,2,3-triazole and the product of its chlorination in the benzene ring [10].



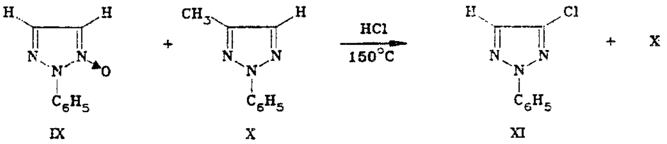
Under these conditions 4,5-dimethyl-2-phenyl-1,2,3-triazole 1-oxide is converted only to a triazole that contains a chorine atom in the para position of the benzene ring [10], whereas unsubstituted 2-phenyl-1,2,3-triazole 1-oxide and its 4-methyl derivative are chlorinated in the 5 position of the ring with subsequent reduction to a triazole [17].

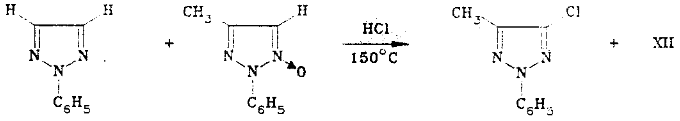


$R = H, CH_3$

Begtrup and Holm [17] assume that the introduction of a chlorine atom into the molecule proceeds through a step involving the formation of 1-hydroxytriazolium ions. Evidence in favor of the existence of these ions is provided by the ^{13}C NMR spectra of the starting triazole N-oxides in dilute sulfuric acid, in which the chemical shifts and the spin-spin coupling constants (SSCC) are close to the analogous values for 1-methoxytriazolium ions, which have high reactivities.

The correctness of these observations was confirmed by the following experiments. In the reaction of a mixture (1:1) of 2-phenyltriazole 1-oxide (IX) and 4-methyl-2-phenyltriazole (X) with concentrated hydrochloric acid the principal product is 2-phenyl-4-chlorotriazole (XI), whereas 4-methyl-2-phenyl-5-chloro-1,2,3-triazole (XIV) was isolated in the case of treatment of a mixture (1:1) of 2-phenyltriazole (XII) and 4-methyl-2-phenyl-1,2,3-triazole 1-oxide (XIII).



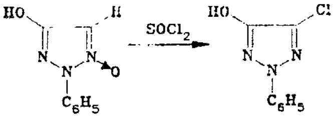


XII XIII XIV

The absence of products of chlorination of the triazoles that are present in the reaction mixture constitutes evidence that the chlorination of triazole N-oxides does not proceed through a step involving the formation of a triazole.

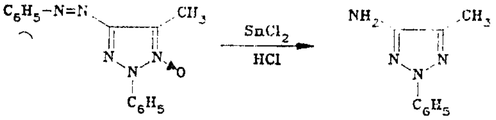
The reaction of 1,2,3-triazole 1-oxides with hydrogen halides has been studied in many cases and it has been demonstrated that the corresponding triazoles are formed in all cases [1, 2, 9, 11, 19, 21, 30, 31, 35, 39, 48].

The reaction of 4-hydroxy-2-phenyl-1,2,3-triazole 1-oxide with thionyl chloride also proceeds with chlorination and reduction of the ring [40].



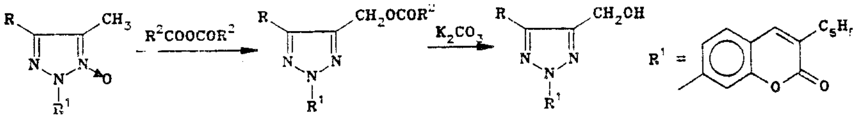
Other reducing agents are also used for the conversion of vicinal triazole N-oxides to triazoles. Zinc in an acidic medium is widely used for this purpose [1, 2, 9-11, 19, 22-24, 26, 27, 29, 30, 35, 38].

The possibility of the use of stannous chloride in concentrated hydrochloric acid for the reduction of triazole N-oxides has been demonstrated [43]. For example, 4-methyl-2-phenyl-1,2,3-triazole 1-oxide is converted by the action of this reagent to the corresponding triazole, whereas the reduction of 5-methyl-2-phenyl-4-phenylazo-1,2,3-triazole 1-oxide gives 4-amino-5-methyl-2-phenyl-1,2,3-triazole.



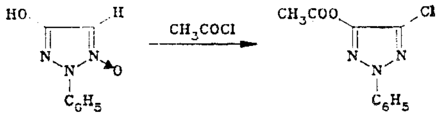
Phosphorus trichloride is also used as a reducing agent for vicinal triazole N-oxides [16, 17, 21].

Also included in the reactions of triazole N-oxides with splitting out of an oxygen atom from the N-oxide fragment is their reaction with acid anhydrides [21, 25] and acid chlorides [16, 17, 28, 40, 49]. It has been established that triazole N-oxides that contain a methyl group in the 5 position of the ring are converted, under the influence of an acid anhydride, to vicinal triazoles with an ester grouping, which is readily saponified [21, 25].



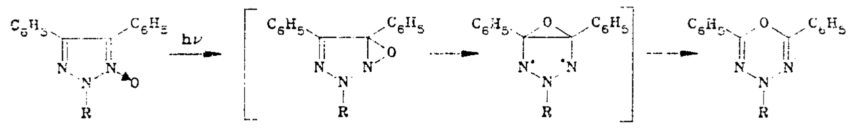
R = CH_3, p-$CH_3C_6H_4$, o,p-$(CH_3)_2C_6H_3$, p-ClC_6H_4; R^2 = alkyl (C_1—C_4), C_6H_5

If there is a hydrogen atom in the 5 position of the starting triazole N-oxides, the corresponding 4-acetoxytriazoles are formed [21]. Triazoles with an ester group are also obtained by the action of acid chlorides on triazole N-oxides [16, 17, 28, 49]. In the case of 2-phenyl-1,2,3-triazole 1-oxide a small amount of 2-phenyl-4-chloro-1,2,3-triazole is also formed in this reaction [17], whereas 4-hydroxy-2-phenyl-1,2,3-triazole 1-oxide reacts with acetyl chloride via the scheme [40]



2.4. Recyclization of 1,2,3-Triazole 1-Oxides

One's attention is directed to the photochemical recyclization of 1,2,3-triazole 1-oxides in high yields to 1,3,4,5-oxatriazines, which were first obtained by this method [14]. Gainsford and Woolhouse [14] propose the following reaction scheme:



R = C_6H_5, p-$CH_3C_6H_4$

3. PHYSICOCHEMICAL PROPERTIES

The UV spectra of 1,2,3-triazole 1-oxides, together with other heterocyclic compounds, have been thoroughly analyzed [50]. The absorption curves of 4-methyl- and 4,5-dimethyl-2-phenyl-1,2,3-triazole 1-oxides have maxima at 265-280 nm. These maxima are shifted 5-10 nm to the lower-wavelength region and are less intense than in the spectra of the corresponding triazoles. Changes in the concentrations of the solutions of triazole N-oxides (from 0.01 N to 3 N) have virtually no effect on the spectra, while replacement of an aprotic solvent (hexane, chloroform) by a protic solvent (alcohol, alcohol—water) shifts the absorption curve only slightly.

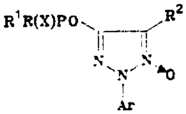
The IR spectra of some vicinal triazoles and their N-oxides were compared and the following differences were found [51]: 1) two absorption bands at 1100-1200 and 795-850 cm^{-1} appear in the spectra of the N-oxides; 2) the spectra of the N-oxides have higher absorption frequencies at 945-965 cm^{-1}; 3) a band of medium intensity at 480-502 cm^{-1}, which is not present in the spectra of vicinal triazoles, appears; 4) the spectra of triazole N-oxides do not contain the absorption band at 1050-1110 cm^{-1} that is characteristic for 1,2,3-triazoles.

Data on the ^{13}C NMR spectra of triazole N-oxides have been presented [17]. It was noted that the presence of an N-oxide fragment in the molecule gives rise to a shift of the chemical shifts of the $C_{(5)}$ atom (by 22.5 ppm) and, in part, the $C_{(4)}$ atom (by 6.7 ppm) to strong field and an increase in the direct C—H spin-spin coupling constants (SSCC).

A study of the mass spectra of the vicinal triazole N-oxides [16-18] showed that these compounds, under the influence of electron impact, give a molecular-ion peak and a peak of an $[M^{+\cdot} - 16]$ ion, which indicates detachment of an oxygen atom from the N-oxide fragment, whereas triazole N-oxides that contain a hydrogen atom in the 5 position contain a peak of the $[M^{+\cdot} - OH]$ ion in their spectra.

4. APPLICATIONS OF 1,2,3-TRIAZOLE 1-OXIDES

2-Aryl-4-hydroxy-1,2,3-triazole 1-oxides and substituted 2-aryl-1,2,3-triazole 1-oxides with the general formula XV have been recommended as insecticides, fungicides, bactericides, nematocides and acaricides [37, 47].



XV

R=OMe, OEt, NMe_2, SPr, NHMe; R^1=OMe, OEt, NMe_2; R^2=H, Cl, Br; X=O, S

2-(3-Phenyl-7-coumarinyl)-1,2,3-triazole 1-oxides that contain alkyl or aryl substituents in the 4 and 5 positions may be of interest as wetting [24] and fluorescent bleaching agents [24].

Substituted 1,2,3-triazole 1-oxides with various structures are widely used as starting compounds for the synthesis of triazoles that have fluorescent bleaching properties and are difficult to obtain by other methods [19, 22, 27, 29. 38, 39, 48].

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