

**REACTIONS OF 2,4-DISUBSTITUTED  
5-NITRO-1,2,3-TRIAZOLE 1-OXIDES.  
2.\* OXIDATION OF 2-SUBSTITUTED  
4-AMINO(ALKYLAMINO)-5-NITRO-  
1,2,3-TRIAZOLE 1-OXIDES**

**T. I. Godovikova, S. A. Vozchikova, E. L. Ignat'eva,  
L. I. Khmel'nitskii, and B. L. Korsunskii**

*The oxidation of substituted aminonitrotriazole oxides to the corresponding dinitro derivatives of triazole and azotriazole oxides has been studied.*

In previous work [2, 3], we have developed a method for the synthesis of 2-substituted 4-amino(alkylamino)-5-nitro-1,2,3-triazole 1-oxides. In the present communication, results are given for oxidation of a series of these compounds under various conditions.

For this study, we selected primary (Ia-d) and secondary amines (Ie,f) of this type, containing various substituents at N<sub>2</sub>, as well as 2-methyl-5-methylamino-4-nitro-1,2,3-triazole 1-oxide (II), which has a different arrangement of the alkylamino and nitro groups. The oxidation of these compounds by trifluoroacetic acid gave the following dinitro derivatives III in 30-67% yield (Scheme 1).

The oxidation of secondary amines proceeds approximately twice faster than oxidation of the primary amines. However, this does not affect the yield of the product IIIa (from 50 to 70%). Prior to our work, oxidation of secondary amino groups to nitro groups in nitrogen heterocycles had not been described.

We should note that 2-methyl-4,5-dinitro-1,2,3-triazole (IV) was also isolated by column chromatography from the reaction mixture in all the cases of formation of product IIIa. The yield of the compound IV and the IIIa:IV product ratio depend on the structure of the starting compound. Thus, the yield of triazole IV is 6% for primary amine Ia and the IIIa:IV ratio is approximately 9:1. The yield of IV increases from 13 to 33% for secondary amines Ie,f and II, while the IIIa:IV ratio ranges from 4.7:1 to 2:1.

The corresponding dinitrotriazole could not be detected in the trifluoroacetic acid oxidation of aminotriazole oxides Ib-d even using thin-layer chromatography.

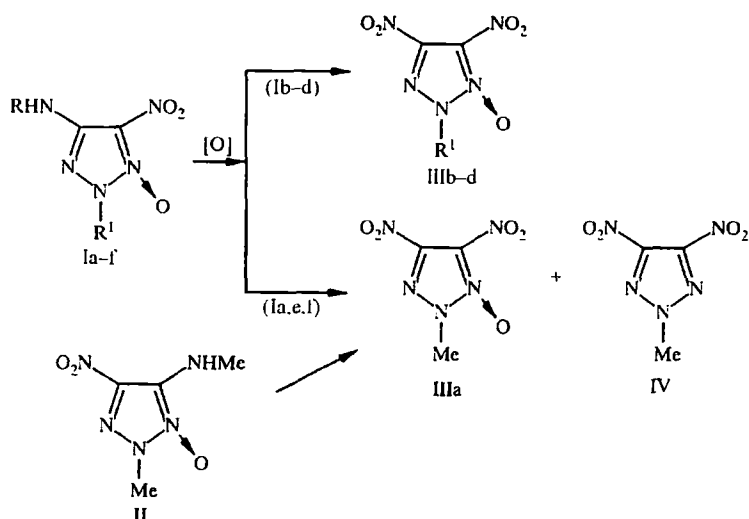
The yields of products IIIa and IV were 50 and 40%, respectively, in the Caro acid oxidation of compound Ia. Triazole IV is presumably obtained under the reaction conditions from triazole oxide IIIa. Indeed, triazole IV was obtained in 56% yield in the Caro acid oxidation of IIIa at 30-35°C.

The formation of 1,2,3-triazoles from the corresponding N-oxides by the action of peracids has not been described earlier.

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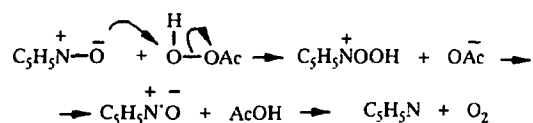
\* Communication 1, see ref. [1].

Scheme 1



Ia R = H, R<sup>1</sup> = Me; b R = H, R<sup>1</sup> = CH<sub>2</sub>Ph; c R = H, R<sup>1</sup> = CH<sub>2</sub>CH<sub>2</sub>OH; d R = H, R<sup>1</sup> = CH<sub>2</sub>CH(OH)Me;  
 e R = R<sup>1</sup> = Me; f R = Et, R<sup>1</sup> = Me; IIIa,e,f R<sup>1</sup> = Me; b R<sup>1</sup> = CH<sub>2</sub>Ph; c R<sup>1</sup> = CH<sub>2</sub>CH<sub>2</sub>OH; d R<sup>1</sup> = CH<sub>2</sub>CH(OH)Me

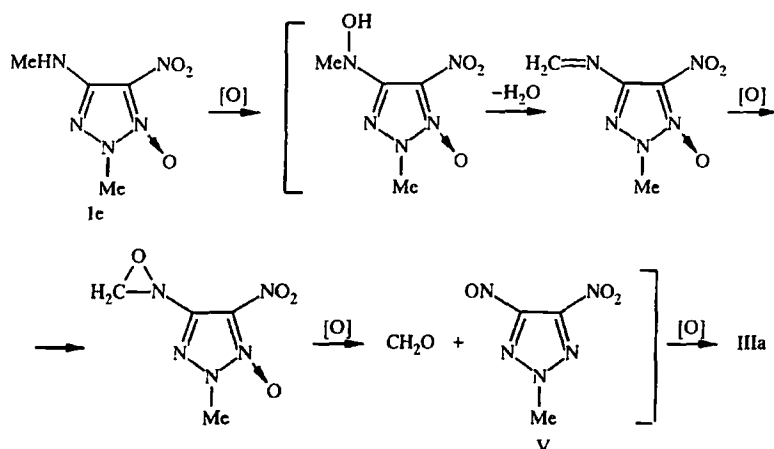
The loss of the N-oxide oxygen atom in the presence of oxidizing agents has been examined for N-oxides of some other nitrogen heterocycles [4]. In the paper [5] a scheme was presented for such transformation for pyridine N-oxide:



This reaction is not observed in acidic media without peracid.

The oxidation of secondary amines by peracids is also known to lead to the corresponding N,N-disubstituted hydroxylamines [6]. If an alkyl substituent may be removed as carbonyl compound in the products obtained, the reaction proceeds to give a nitroso derivative [7], which is readily oxidized to the nitro compound [8].

By analogy with literature data [7], the oxidation of triazole oxide Ie, for example, probably proceeds as follows:



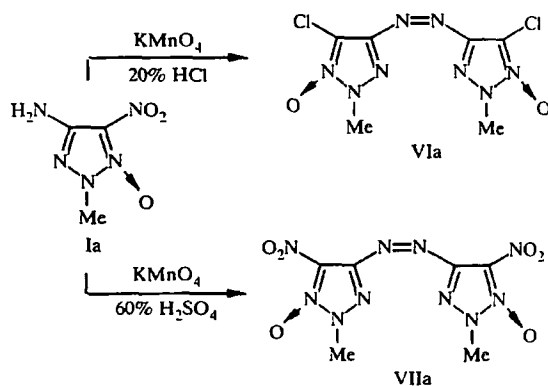
The trifluoroperacetic acid oxidation of triazole oxide Ia at 10°C gives 1:1 mixture of the compound IIIa and an intermediate, 2-methyl-5-nitro-4-nitroso-1,2,3-triazole 1-oxide (V). This mixture could not be resolved by column chromatography since the  $R_f$  values of the two compounds were identical using various solvents as eluents. The similar solubility prevented their resolution by recrystallization. The mixture of compounds IIIa and V has an intense green color, indicating that the nitroso product exists largely as the monomer. Maintenance of this mixture at room temperature for two or three hours did not lead to significant decomposition. The existence of the product V was confirmed by IR and  $^{14}\text{N}$  NMR spectroscopy and mass spectrometry.

The described method permits to synthesize the dinitro derivatives of 1,2,3-triazole 1-oxides containing a functional substituent such as hydroxyl group at  $\text{N}_{(2)}$  as in the case of compounds IIIc and IIIe.

Triazole oxide IIIa has been obtained in the nitration of 2-methyl-substituted triazole oxide using a mixture of concentrated nitric and sulfuric acids at 100°C [9]. The vigorous conditions of this method limit its use in synthesis.

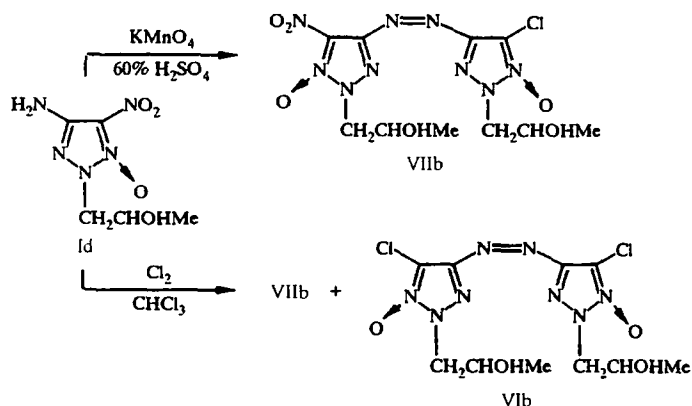
Potassium permanganate in acidic medium was also used in our laboratory as an oxidizing agent.

In the case of triazole oxide Ia, we showed that azo compounds of various structure are obtained depending on the acid being used. Thus, 4,4'-azo-2-methyl-5-chloro-1,2,3-triazole 1-oxide (VI) is obtained as the main product in 30% yield in hydrochloric acid medium along with traces of 4,4'-azo-2-methyl-5-nitro-1,2,3-triazole 1-oxide (VIIa). The oxidation of triazole oxide Ia in sulfuric acid leads only to formation of dinitro derivative VIIa in 40% yield.



We propose that formation of the compound VIa proceeds through nucleophilic substitution of the nitro groups by chlorine atom in one of the intermediate steps of oxidation of the aminonitro derivative and not in the final product since we have found that azotriazole oxide VIIa under conditions analogous to this oxidation is not converted to the compound VIa.

Potassium permanganate in 60% sulfuric acid was also used to oxidize aminotriazole oxide Id in order to obtain the corresponding azo product, VIIb. However, the final product contained a significant amount of impurities, which were difficult to eliminate.



Thus, gaseous chlorine as a freshly prepared solution in chloroform was used as the oxidizing agent for the synthesis of the compound VIIb. Let us note that the content of the side-product, namely, the corresponding dichloro derivative, VIb, increases with increasing temperature of the reaction and increasing amount of oxidizing agent.

## EXPERIMENTAL

The IR spectra were recorded on Specord IR-75 and UR-20 spectrometers for KBr pellets. The UV spectra were taken on a Specord UV spectrometer for solutions in absolute ethanol. The mass spectra were taken on a Varian mass spectrometer with direct sample inlet into the ion source. The ionizing voltage was 70 eV, the accelerating voltage was 1.75 kV, and the emission current was 100 mA. The  $^{13}\text{C}$  and  $^{14/15}\text{N}$  NMR spectra were obtained on a Bruker AM-200 spectrometer at 21.67, 30.42, and 75.47 MHz. The PMR spectra were recorded on a Bruker WP-250 spectrometer. The thin-layer chromatography was carried out on Silufol UV-254 plates using methylene chloride as the eluent and detection with UV light. Column chromatography was carried out on  $70 \times 1.5$ -cm column packed with silica gel L40/100 $\mu$  using 4:1 methylene chloride-carbon tetrachloride as the eluent.

The syntheses of compounds Ia and Ie were described in our previous work [3]. The syntheses of compounds Ib-d were also described in previous work [10], while the syntheses of If and II were described in work [1].

**Oxidation of 2-R'-4-R-Amino-5-nitro- (Ia-f) and 2-Methyl-5-methylamino-4-nitro-1,2,3-triazole 1-oxides (II) using Trifluoroacetic Acid (General Method).** Sample of compound Ia-f or II (2 mmol) was added to mixture of 80% hydrogen peroxide (1.1 g, 40 mmol) and trifluoroacetic acid (13 ml, 171 mmol) over 15 min at 18-20°C. The reaction mixture was maintained at this temperature for 2 h until the complete disappearance of the starting triazole oxide as indicated by thin-layer chromatography using methylene chloride as the eluent. Then, 10-15 ml of water were added and the mixture was extracted with three 30-ml methylene chloride portions. The combined extracts were dried over magnesium sulfate. The solvent was removed in vacuum. Products III and IV were separated from the residue by column chromatography. The identical nature of samples of IIIa and IV obtained by different methods was established by IR and PMR spectroscopy and mass spectrometry.

**2-Methyl-4,5-dinitro-1,2,3-triazole 1-Oxide (IIIa);** mp 130-132°C (132°C [1]), was obtained in 55% yield (0.2 g) and **2-methyl-4,5-dinitro-1,2,3-triazole 1-oxide (IV);** mp 77-78°C (from  $\text{CCl}_4$ , literature data 81-82°C from ethyl acetate-hexane [9]), was obtained in 6% yield (0.02 g) from 4-amino-2-methyl-5-nitro-1,2,3-triazole 1-oxide (Ia). (The elemental analysis data and spectral data are not given in this work). IR spectrum: 2960, 2930, 2860, 1580, 1570, 1510, 1440, 1370, 1350, 1320, 1145, 865, 815, 755  $\text{cm}^{-1}$ . PMR spectrum in  $(\text{CD}_3)_2\text{CO}$ : 4.30 ppm (3H, s,  $\text{CH}_3$ ).  $^{13}\text{C}$  NMR spectrum ( $(\text{CD}_3)_2\text{CO}$ ): 143.57 ( $\text{C}_{(4)}$ ,  $\text{C}_{(5)}$ ); 44.53 ppm ( $\text{CH}_3$ ).  $^{14}\text{N}$  NMR spectrum ( $(\text{CD}_3)_2\text{CO}$ ): -32.96 ppm ( $\text{NO}_2$ ).  $^{15}\text{N}$  NMR spectrum ( $(\text{CD}_3)_2\text{CO}$ ): -48.84 ( $\text{N}_{(1)}$ ,  $\text{N}_{(3)}$ ); -139.04 ppm ( $\text{N}_{(2)}$ ). Mass spectrum,  $m/z$ : 173  $[\text{M}]^+$ , 81  $[\text{M} - 2\text{NO}_2]^+$ , 43  $[\text{CH}_3\text{N}_2]^+$ , 42  $[\text{CH}_2\text{N}_2]^+$ . Found, %: C 21.17; H 1.90; N 40.55.  $\text{C}_3\text{H}_5\text{N}_5\text{O}_4$ . Calculated, %: C 20.80; H 1.73; N 40.46.

**2-Benzyl-4,5-dinitro-1,2,3-triazole 1-Oxide (IIIb)** was obtained from 4-amino-2-benzyl-5-nitro-1,2,3-triazole 1-oxide (Ib). Yield 0.1 g (30%); mp 121-122°C ( $\text{CCl}_4$ ). IR spectrum: 1580, 1560, 1510, 1470, 1450, 1430, 1385, 1360, 1340, 1305, 1255, 1210, 1080, 915, 835, 810, 775, 745, 715  $\text{cm}^{-1}$ . PMR spectrum ( $(\text{CD}_3)_2\text{CO}$ ): 5.75 (2H, s,  $\text{CH}_2$ ); 7.41 ppm (5H, s,  $\text{C}_6\text{H}_5$ ).  $^{14}\text{N}$  NMR spectrum ( $(\text{CD}_3)_2\text{CO}$ ): -34.39 ( $\text{NO}_2$ ); -40.35 ( $\text{NO}_2$ ); -82.00 ppm ( $\text{N} \rightarrow \text{O}$ ). Mass spectrum,  $m/z$ : 265  $[\text{M}]^+$ , 249  $[\text{M} - \text{O}]^+$ , 248  $[\text{M} - \text{OH}]^+$ , 91  $[\text{C}_6\text{H}_5\text{CH}_2]^+$ , 77  $[\text{C}_6\text{H}_5]^+$ . Found, %: C 40.84; H 2.71; N 26.56.  $\text{C}_9\text{H}_7\text{N}_5\text{O}_5$ . Calculated, %: C 40.76; H 2.66; N 26.41.

**2-(2-Hydroxyethyl)-4,5-dinitro-1,2,3-triazole 1-Oxide (IIIc)** was obtained in 67% yield (0.3 g), mp 106°C (dec.), from 4-amino-2-(2-hydroxyethyl)-5-nitro-1,2,3-triazole 1-oxide (Ic). IR spectrum: 3490, 2960, 2920, 1580, 1575, 1550, 1530, 1455, 1385, 1335, 1250, 1080, 1050, 965, 895, 875, 835, 795, 760, 740, 710  $\text{cm}^{-1}$ . Found, %: C 21.85; H 2.10; N 31.86.  $\text{C}_4\text{H}_5\text{N}_5\text{O}_6$ . Calculated, %: C 21.98; H 2.30; N 31.96.

**2-(2-Hydroxypropyl)-4,5-dinitro-1,2,3-triazole 1-Oxide (IIIId)** was obtained from 4-amino-2-(2-hydroxypropyl)-5-nitro-1,2,3-triazole 1-oxide (Id). Yield 0.2 g (43%); mp 110-111°C (ether). IR spectrum: 3450, 2980, 1580, 1560, 1510, 1455, 1430, 1385, 1340, 1270, 1250, 1135, 1085, 945, 850, 840, 805, 740  $\text{cm}^{-1}$ . PMR spectrum ( $\text{DMSO}-d_6$ ): 4.3 (3H, m,  $\text{CH}_2\text{CH}$ ); 1.1 ppm (3H, d,  $\text{CH}_3$ ).  $^{14}\text{N}$  NMR spectrum ( $\text{DMSO}-d_6$ ): -33.18 ( $\text{NO}_2$ );

-39.17 ppm (NO<sub>2</sub>). <sup>15</sup>N NMR spectrum (DMSO-d<sub>6</sub>): -78.10 (N<sub>(1)</sub>); -98.54 (N<sub>(3)</sub>); -137.77 (N<sub>(2)</sub>). <sup>13</sup>C NMR spectrum (DMSO-d<sub>6</sub>): 139.50 (C<sub>(4)</sub>); 129.84 (C<sub>(5)</sub>); 63.15 (CH); 56.32 (CH<sub>2</sub>); 20.18 ppm (CH<sub>3</sub>). Found, %: C 25.71; H 3.12; N 29.89. C<sub>5</sub>H<sub>7</sub>N<sub>5</sub>O<sub>6</sub>. Calculated, %: C 25.76; H 3.03; N 30.04.

Triazole oxide IIIa was obtained in 50% yield and triazole IV was obtained in 13% yield from 2-methyl-4-methylamino-5-nitro-1,2,3-triazole 1-oxide (Ie).

Triazole oxide IIIa was obtained in 70% yield and triazole IV was obtained in 22% yield from 4-ethylamino-2-methyl-5-nitro-1,2,3-triazole 1-oxide (If).

Triazole oxide IIIa was obtained in 66% yield and triazole IV was obtained in 33% yield from 2-methyl-5-methylamino-4-nitro-1,2,3-triazole 1-oxide (II).

**2-Methyl-5-nitro-4-nitroso-1,2,3-triazole 1-Oxide (V).** Sample of triazole oxide Ia (0.4 g, 2.5 mmol) was added to a mixture of 80% hydrogen peroxide (1.1 g, 40 mmol) in trifluoroacetic acid (13 ml, 171 mmol) over 10 min at 10°C. The reaction mixture was left for 1 h at the same temperature and turned bright green. Starting amine Ia completely disappeared as indicated by thin-layer chromatography using methylene chloride as the eluent (*R<sub>f</sub>* 0.2 for Ia). The reaction mixture was treated as described above. Column chromatography using methylene chloride as the eluent gave 0.3 g of bright green crystals (1:1 mixture of compounds IIIa and V as indicated by <sup>14</sup>N NMR spectroscopy); mp 97-104°C, *R<sub>f</sub>* 0.8 (methylene chloride). IR spectrum: 1570, 1530, 1480, 1430, 1385, 1370, 1250, 1110, 1085, 1035, 920, 830, 805, 790, 765, 750 cm<sup>-1</sup>. UV spectrum, λ: 230 and 960 nm. <sup>14</sup>N NMR spectrum ((CD<sub>3</sub>)<sub>2</sub>CO): -34.33 (4-NO<sub>2</sub>, IIIa); -37.42 (NO<sub>2</sub>, V); -40.18 (5-NO<sub>2</sub>, IIIa); -78.79 ppm (N→O). The nitroso group signal at 500-400 ppm could not be detected due to its great width (Δ*v*<sub>1/2</sub> > 1000 Hz).

PMR spectrum (CDCl<sub>3</sub>): 4.10 (3H, s, CH<sub>3</sub>) and 4.09 ppm (3H, s, CH<sub>3</sub>). Mass spectrum, *m/z*: 189 [M<sub>IIIa</sub>]<sup>+</sup>, 173 [M<sub>IIIa</sub> - O]<sup>+</sup> and [M<sub>IV</sub>]<sup>+</sup>, 143 [M<sub>IIIa</sub> - NO<sub>2</sub>]<sup>+</sup> and [M<sub>IV</sub> - NO]<sup>+</sup>, 97 [M<sub>IIIa</sub> - 2NO<sub>2</sub>]<sup>+</sup> and [M<sub>V</sub> - NO - NO<sub>2</sub>]<sup>+</sup>.

**Oxidation of Triazole Oxide Ia by Caro Acid.** Sample of 80% hydrogen peroxide (4 ml) and compound Ia (0.5 g, 3 mmol) was added to 30% oleum (10 ml) at 0-10°C under intense stirring. The temperature was gradually raised to 20°C and the reaction mixture was maintained under these conditions for 14-15 h. The reaction mixture was poured onto ice. Potassium bicarbonate was added to bring the solution to pH 1 and the solution was extracted with three 30-ml methylene chloride portions. The combined extract was dried over calcined magnesium sulfate. The solvent was removed in vacuum. Column chromatography of the residue gave 0.3 g (50%) of triazole oxide IIIa and 0.1 g (40%) of triazole IV.

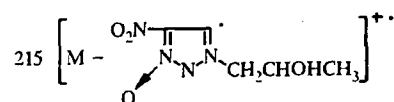
**Reaction of Triazole Oxide IIIa with Caro Acid.** Sample of 80% hydrogen peroxide (10 ml) and triazole oxide IIIa (1.1 g, 6 mmol) was added to 30% oleum (35 ml) at 0-10°C under intense stirring. The temperature was raised to 30-35°C and the reaction mixture was maintained under these conditions for 4 h. The reaction mixture was treated as in the case of Ia. Column chromatography gave 0.55 g (56%) of triazole IV and 0.4 g (36%) of the starting compound IIIa.

**Oxidation of 4-Amino-2-methyl-5-nitro-1,2,3-triazole 1-Oxide (Ia) by Potassium Permanganate in an Acidic Medium. A. Oxidation in Hydrochloric Acid.** Solution of potassium permanganate (0.2 g, 1.2 mmol) in water (10 ml) was added dropwise to mixture of amine Ia (0.1 g, 0.6 mmol) and 20% hydrochloric acid (10 ml) at 20°C under intense stirring. After 18-20 h, oxalic acid was added until the brown color disappeared. The precipitate was filtered off, washed with water, ether, and methylene chloride, and dried in the air to give 0.05 g (30%) of **4,4'-azo-2-methyl-5-chloro-1,2,3-triazole 1-oxide (VIa)**; mp 328°C (dec.). IR spectrum: 3050, 1500, 1430, 1340, 1260, 1220, 1100, 1050, 900, 840, 730, 710 cm<sup>-1</sup>. Mass spectrum, *m/z*: 292, 294, 296. Found, %: C 24.64; H 1.98; N 38.43. C<sub>6</sub>H<sub>6</sub>Cl<sub>2</sub>N<sub>8</sub>O<sub>2</sub>. Calculated, %: C 24.57; H 2.05; N 38.22; Cl 24.22. (Thin-layer chromatography using 1:1 ether-methylene chloride as eluent indicated traces of dinitroazo derivative VIIa, *R<sub>f</sub>* 0.6 in the filtrate).

**B. Oxidation in Sulfuric Acid.** Solution of potassium permanganate (0.3 g, 1.5 mmol) in water (15 ml) was added dropwise to a rapidly stirred mixture of triazole oxide Ia (0.5 g, 3 mmol) and 62% sulfuric acid (30 ml) at -10° to 0°C. After 2.5-3 h, oxalic acid was added to the reaction mixture until the brown color disappeared. The precipitate was filtered off, washed with water, and dried in the air to give 0.2 g (40%) of **4,4'-azo-2-methyl-5-nitro-1,2,3-triazole 1-oxide (VIIa)**; mp 281°C (dec.) (acetone). IR spectrum: 1570, 1505, 1430, 1370, 1345, 1250, 1135, 1035, 940, 835, 750, 690 cm<sup>-1</sup>. <sup>14</sup>N NMR spectrum ((CD<sub>3</sub>)<sub>2</sub>CO): -35.61 ppm (NO<sub>2</sub>). The mass spectrum contains a molecular ion peak [M]<sup>+</sup> (*m/z* 314). Found, %: C 23.11; H 2.10; N 44.52. C<sub>6</sub>H<sub>6</sub>N<sub>10</sub>O<sub>6</sub>. Calculated, %: C 22.92; H 1.91; N 44.58.

**Oxidation of 4-Amino-2-(2-hydroxypropyl)-5-nitro-1,2,3-triazole 1-Oxide (Id) by Potassium Permanganate and Chlorine. A. Oxidation by Potassium Permanganate.** Solution of potassium permanganate (0.1 g, 0.5 mmol) in water (10 ml) was added to solution of triazole oxide Id (0.2 g, 0.9 mmol) in 60% sulfuric acid (30 ml) at -10°C over 30 min. The mixture was maintained at this temperature for 4.5 h and then for 30 min at 18-20°C. The reaction mixture was poured onto ice and extracted with two 50-ml ether portions and two 50-ml ethyl acetate portions. The combined extract was dried over calcined magnesium sulfate. After removal of the solvent, the precipitate was subjected to column chromatography using ether as the eluent, collecting the fraction with  $R_f$  0.07 (ether as eluent) to give 0.09 g (50%) of 4,4'-azo-2-(2-hydroxypropyl)-5-nitro-1,2,3-triazole 1-oxide (VIIb); mp 142-145°C, containing a large amount of impurities as indicated by thin-layer chromatography. Repurification by column chromatography and recrystallization from 1:1 ethyl acetate-ether did not provide complete purification of the product.

**B. Oxidation by Chlorine.** Solution of sodium bicarbonate (0.3 g, 3.6 mmol) in water (5 ml) was added to stirred solution of triazole oxide Id (0.2 g, 0.9 mmol) in chloroform (5 ml) at room temperature and, then, 5 ml of chloroform saturated with chlorine were added dropwise at 0-5°C. The reaction mixture was maintained at this temperature for 1 h. The precipitate formed was filtered off, washed with chloroform and ether, and dried in the air to give a mixture of the products VIIb and VIb, which was treated with three 10-ml acetone portions. After removal of the organic solvent 0.11 g (59%) of 4,4'-azo-2-(hydroxypropyl)-5-nitro-1,2,3-triazole 1-oxide (VIIb) were obtained,  $R_f$  0.55 (ethyl acetate); mp 151°C (dec.). IR spectrum: 3600, 3400, 2980, 2920, 1620, 1550, 1500, 1490, 1440, 1410, 1350, 1240, 1110, 1040, 940, 910, 820, 740, 720  $\text{cm}^{-1}$ . UV spectrum,  $\lambda$ : 233.0, 386.4 nm. Mass spectrum,  $m/z$ : 402  $[\text{M}]^+$ ; 356  $[\text{M} - \text{NO}_2]^+$ ; 340  $[\text{M} - \text{NO}_2 - \text{O}]^+$ ; 298  $[\text{M} - \text{NO}_2 - \text{CH}_2\text{CHOHCH}_3 + \text{H}]^+$ ;



Found, %: C 29.80; H 3.50; N 35.01.  $\text{C}_{10}\text{H}_{14}\text{N}_4\text{O}_8$ . Calculated, %: C 29.85; H 3.48; N 34.83.

The precipitate insoluble in acetone was recrystallized from ethanol to give 0.05 g (28%) of 4,4'-azo-5-chloro-2-(2-hydroxypropyl)-1,2,3-triazole 1-oxide (VIb);  $R_f$  0.25 (ethyl acetate); mp 198-199°C. IR spectrum: 3480, 3450, 2960, 2930, 1620, 1550, 1490, 1460, 1420, 1370, 1350, 1320, 1290, 1270, 1220, 1130, 1100, 1050, 940, 910, 890, 830, 820, 740, 650  $\text{cm}^{-1}$ . Mass spectrum,  $m/z$ : 380, 382, 384  $[\text{M}]^+$ , 364, 366, 368  $[\text{M} - \text{O}]^+$ , 345, 347  $[\text{M} - \text{Cl}]^+$ , 322, 324, 326  $[\text{M} - \text{CH}_2\text{CHOHCH}_3 + \text{H}]^+$ , 306, 308, 310  $[\text{M} - \text{CH}_2\text{CHOHCH}_3 + \text{H} - \text{O}]^+$ . Found, %: C 31.34; H 4.00; N 29.50; Cl 18.55.  $\text{C}_{10}\text{H}_{14}\text{Cl}_2\text{N}_4\text{O}_4$ . Calculated, %: C 31.58; H 3.68; N 29.47; Cl 18.62.

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