TMS), δ: 9.54 (s, 1H, 1-H), 8.94 (s, 1H, 10-H), 8.68 (s, 1H, 5-H), 6.34 (s, 2H, NCH_2), and 2.62 ppm (s, 3H, 8-CH_3). Found %: N 4.7; M^+ 311. $C_{22}H_{17}NO$. Calculated %: N 4.5; M 311.

Benzo[g]isoquinolinium Dibenzoylmethylid (IX). A 1.2-ml (8 mmole) sample of benzoyl chloride and 3 ml (20 mmole) of triethylamine were added successively to a solution of 2.5 g (6.6 mmole) of quaternary salt IV in 30 ml of chloroform, and the mixture was stirred for 1 h. The solvent was removed, and the residue was dissolved in ethanol and precipitated by the addition of absolute ether to give 2.05 g (70%) of dibenzoylmethylid IX as a red-brown powder with mp 172-174°C. IR spectrum: 1503 cm^{-1} (CO). UV spectrum, λ_{max}, nm (log ε): 256 (4.68), 340 (3.88), 396 (3.48), 436 sh (3.42), and 500 nm (3.20). PMR spectrum ($CDCl_3$, TMS), δ: 9.50 (s, 1H, 1-H), 8.75 (s, 1H, 10-H), and 8.37 ppm (s, 1H, 5-H). Found %: N 3.3; M^+ 401. $C_{28}H_{19}NO_2$. Calculated %: N 3.5; M 401.

3-Benzoyl-1,2-dicarbomethoxynaphth[2,3-g]indolizine (X). A 1.64-g (14 mmole) sample of dimethylacetylenedicarboxylate and 1.7 g (17 mmole) of triethylamine were added to a solution of 2.2 g (6 mmole) of salt IV in 35 ml of chloroform, and the mixture was refluxed for 8 h. It was then cooled and washed with water. The chloroform solution was dried with magnesium sulfate, and the chloroform was removed by distillation. The residue was chromatographed with a column (H = 50 cm, d = 2.5 cm, activity II aluminum oxide, elution with ether) to give 0.4 g (15%) of X in the form of golden crystals with mp 198-199°C (from ethanol). UV spectrum, λ_{max} (log ε): 260 (4.90), 300 (5.00), 330 (4.60), 385 (4.48), and 405 nm (4.44). PMR spectrum ($CDCl_3$, TMS), δ: 9.30 (s, 1H, 11-H), 8.48 (d, 1H, 4-H), 8.07 (s, 1H, 6-H), 7.10 (d, 1H, 5-H), and 4.00 and 3.23 ppm (s, 3H each, $COOCH_3$). Found %: C 74.3; H 4.6; N 3.2; M^+ 437. $C_{27}H_{19}NO_5$. Calculated %: C 74.1; H 4.3; N 3.2; M 437.

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PREPARATION AND SOME PROPERTIES OF 2H-IMIDAZOLE 1,3-DIOXIDES, DERIVATIVES OF ALICYCLIC 1,2-DIOXIMES

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UDC 547.785.5

The corresponding 2H-imidazole 1,3-dioxides were obtained by the reaction of cyclohexanedione and cycloheptadione 1,2-dioximes with acetone, cyclopentanone, and methyl ethyl ketone. The reactions of these compounds with hydroxylamine hydrochloride, $NaBH_4$, a Grignard reagent, and acetic anhydride in the presence of H_2SO_4 were studied in the case of 2,2-dimethyl-4,5,6,7-tetrahydro-2H-benzimidazole 1,3-dioxide. Bromination of the latter and 2,2-dimethylcyclohepta-2H-imidazole 1,3-dioxide with N-bromosuccinimide gave the corresponding dibromo derivatives, the bromine atoms in which are replaced by acetoxy and hydroxy groups. 4,7-Dihydroxy-2,2-dimethyl-4,5,6,7-tetrahydro-2H-benzimidazole 1,3-dioxide, which was obtained by oxidation with MnO_2, was converted to a quinone, viz., 2,2-dimethyl-4,7-dioxo-4,7-dihydro-2H-benzimidazole 1,3-dioxide.

2H-Imidazole N,N'-dioxides are presently difficult-to-obtain compounds to which very little study has been devoted; only a few examples of their synthesis are known [2, 3]. At the same time, 2H-benzimidazole 1,3-dioxides are rather well known and have unusual reactivities [4]. The aim of the present research was to synthesize and study the properties of

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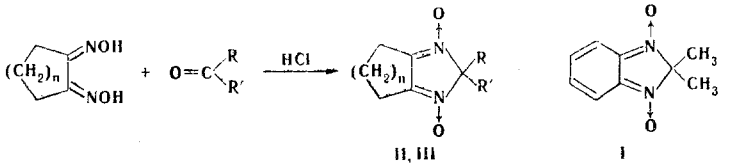
2H-imidazole 1,3-dioxides. It was recently shown [5] that the reaction of o-benzoquinone dioxime with acetone in the presence of $HAuCl_4$ leads to 2,2-dimethyl-2H-benzimidazole 1,3-dioxide. These results, as well as data on the condensation of dioximes of 1,2-diketones with aldehydes [6, 7], enabled us to assume that 2H-imidazole N,N'-dioxides could be obtained by condensation of dioximes with ketones in the presence of acids.

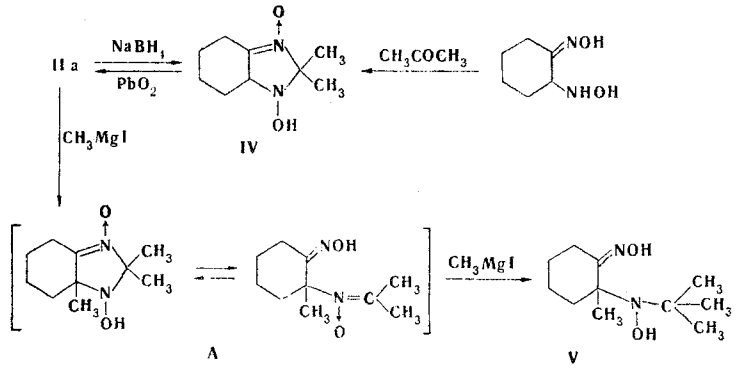
In fact, we have observed [1] that 2,2-dimethyl-2H-benzimidazole 1,3-dioxide (I) is formed in 76% yield by the reaction of o-benzoquinone dioxime with acetone in the presence of hydrochloric acid. Cyclohexanedione and cycloheptadione 1,2-oximes also react readily with acetone in the presence of HCl to give 2H-imidazole 1,3-dioxides IIa and IIIa in 50-60% yields and with cyclopentanone and methyl ethyl ketone to give dioxides IIb, c and IIIb.



II, III I

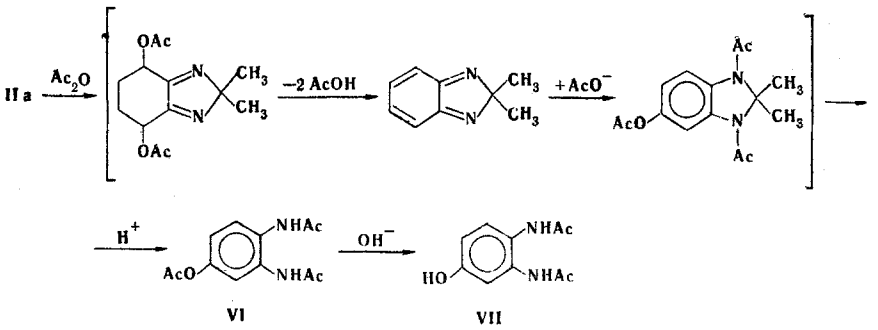
II n=2; III n=3; a R=R'=CH_3; b R+R'=$(CH_2)_4$; c R=CH_3, R'=C_2H_5

Signals of two equivalent methyl groups at 1.67 ppm and of methylene groups of the cyclohexane ring at 1.7-2.9 ppm are observed in the PMR spectrum of IIa. The IR spectra of IIa-c and IIIa, b contain a band of stretching vibrations of the C=N bond at 1560-1590 cm^{-1}. The characteristic absorption in the UV spectra of these compounds at λ_{max} 350 nm (logε 3.90) indicates the presence of two conjugated nitrone groups [3]. A monohydrochloride is formed when dry HCl is bubbled into an ether solution of IIa. Cyclohexanedione 1,2-dioxime is regenerated when dioxide IIa is refluxed in an alcohol solution of hydroxylamine hydrochloride. The reduction of dioxide IIa with $NaBH_4$ leads to the previously described 1-hydroxy-3-imidazoline-3-oxide (IV) [8], the oxidation of which again gives starting dioxide IIa. The previously described N-(1-oximino-2-methyl-2-cyclohexyl)-tert-butylhydroxylamine (V) [9] is formed when IIa is treated with excess methylmagnesium iodide. The reaction can be explained by the addition of CH_3MgI to IIa with the formation of intermediate A, which is capable of existing as a tautomeric mixture of cyclic and open forms [9], with the subsequent addition of another molecule of the Grignard reagent to the nitrone group of the open form.

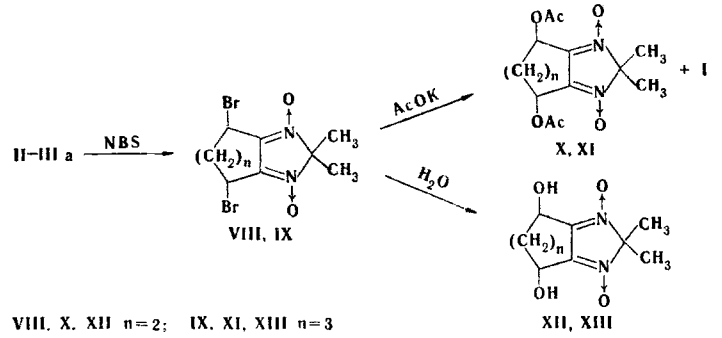


IV A V

In an attempt to remove the N-oxide oxygen atoms in IIa by the action of acetic anhydride in the presence of an acid as described for the N-oxides of dihydropyrazines [10, 11], we isolated the previously described 1,2-diacetamido-4-acetoxybenzene (VI), treatment of which with alkali led to 1-hydroxy-3,4-diacetamidobenzene (VII) [12]. The formation of VI evidently proceeds through a step involving the initial conversion of 2H-imidazole 1,3-dioxide IIa to 2H-benzimidazole (see [11]), which then undergoes nucleophilic attack in the 5 position as in [13, 14].



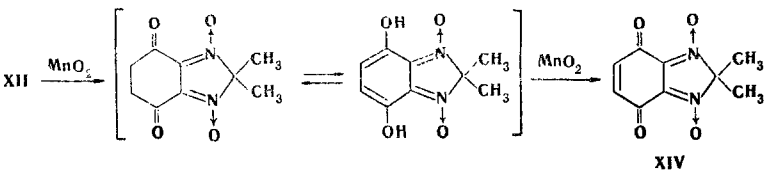
The bromination of 2H-imidazole 1,3-dioxides IIa and IIIa with N-bromosuccinimide leads to, respectively, 4,7-dibromo-2,2-dimethyl-4,5,6,7-tetrahydro-2H-benzimidazole 1,3-dioxide (VIII) and to 4,8-dibromo-2,2-dimethylcyclohepta-2H-imidazole 1,3-dioxide (IX). The PMR



spectra of VIII and IX contain signals of protons of two methylidyne groups in the form of a complex multiplet at 5.3-5.4 ppm.

4,7-Diacetoxy-2,2-dimethyl-4,5,6,7-tetrahydro-2H-benzimidazole 1,3-dioxide (X), 4,8-diacetoxy-2,2-dimethylcyclohepta-2H-imidazole 1,3-dioxide (XI), 4,7-dihydroxy-2,2-dimethyl-4,5,6,7-tetrahydro-2H-benzimidazole 1,3-dioxide (XII), and 4,8-dihydroxy-2,2-dimethylcyclohepta-2H-imidazole 1,3-dioxide (XIII), respectively, are formed in the reaction of dibromo derivatives VIII and IX with potassium acetate in acetonitrile and also by heating these compounds with water as a result of nucleophilic substitution of the bromine atoms by acetoxy and hydroxy groups. In the case of dibromo derivative VIII, in addition to substitution, one observes elimination to give 2H-benzimidazole 1,3-dioxide (I) in the reaction with potassium acetate.

The oxidation of XII with active MnO_2 did not lead to the diketone but rather to a quinone, viz., 2,2-dimethyl-4,7-dihydro-2H-benzimidazole 1,3-dioxide (XIV), the formation of which is probably due to oxidation of the enol form of the initially formed 4,7-diketone.



Thus, the results open up new possibilities for the synthesis and study of derivatives of 2H-imidazole 1,3-dioxides.

EXPERIMENTAL

The IR spectra of KBr pellets of the compounds (0.25% concentrations) were recorded with a UR-20 spectrometer. The UV spectra of solutions of the compounds in alcohol were recorded with a Specord UV-vis spectrophotometer. The PMR spectra were obtained with a Varian A-56-60A spectrometer with hexamethyldisiloxane as the internal standard. The identical character of the previously described compounds was established from the melting points and a comparison of the IR and UV spectra. The yields, melting points, results of elementary analysis, and spectral characteristics of the synthesized compounds are presented in Tables 1 and 2.

TABLE 1. Characteristics of the Synthesized Compounds

Compound	mp, °C	UV spectrum, λ_{max}, nm (log ε)	Found, %			Empirical formula	Calc., %			Yield %
			C	H	N		C	H	N	
IIa	160—162[a]	212 (3,90), 350 (3,90)	58,9	7,7	15,9	$C_9H_{14}N_2O_2$	59,3	7,7	15,4	60
IIb	172—174[a]	212 (3,90), 350 (3,90)	63,1	7,6	13,7	$C_{11}H_{16}N_2O_2$	63,4	7,7	13,5	50
IIc	77—79[b]	212 (3,90), 350 (3,90)	61,0	8,2	14,2	$C_{10}H_{16}N_2O_2$	61,2	8,2	14,3	32
IIIc	146—148[a]	220 (4,10), 350 (4,00)	61,0	8,2	14,3	$C_{10}H_{16}N_2O_2$	61,2	8,2	14,3	50
IIIb	159—161[a]	220 (3,90), 350 (3,90)	64,7	8,2	12,5	$C_{12}H_{18}N_2O_2$	64,8	8,2	12,6	50
VIII	133—135[c]	270 (4,00), 365 (3,80)	32,1	3,4	8,6	$C_9H_{12}Br_2N_2O_2$[d]	31,7	3,5	8,2	80
IX	145—147[a]	260 (3,10), 365 (2,80)	33,9	4,0	8,0	$C_{16}H_{14}Br_2N_2O_2$[e]	33,9	4,0	7,9	76
X	160—162[c]	228 (3,90), 255 (3,30), 360 (3,70),	52,5	6,1	9,0	$C_{13}H_{18}N_2O_6$	52,3	6,1	9,4	23
XI	171—173[c]	228 (4,02), 355 (3,90)	53,5	6,4	8,9	$C_{14}H_{20}N_2O_6$	53,9	6,4	9,0	28
XII	160 dec. [c]	222 (4,02), 355 (3,90)	51,1	6,4	12,7	$C_9H_{14}N_2O_4$	50,9	5,7	13,2	44
XIII	187—189[c]	228 (3,78), 355 (3,90)	52,7	7,0	12,2	$C_{10}H_{16}N_2O_4$	52,7	7,0	12,2	35
XIV	213 dec.[c]	230 (4,06), 298 (3,73), 390 (3,55), 435 (3,30)	51,5	4,1	12,8	$C_9H_8N_2O_4$	51,9	3,9	13,5	25
IIa · HCl	150 dec.	212 (3,90), 350 (3,90)	49,0	6,8	12,9	$C_9H_{14}N_2O_2 \cdot HCl$[f]	48,4	6,8	12,8	70

[a]From ethyl acetate. [b]From petroleum ether (40-70°C). [c]From alcohol. [d]Found %: Br 46.8. Calculated %: Br 47.0. [e]Found %: Br 45.2. Calculated %: Br 44.8. [f]Found %: Cl 16.2. Calculated %: Cl 16.2.

2,2-Dimethyl-4,5,6,7-tetrahydro-2H-benzimidazole 1,3-Dioxide (IIa). A 1-ml smaple of 5% HCl was added to a solution of 1 g (7.05 mmole) of cyclohexanedione 1,2-dioxime in 50 ml of acetone, and the mixture was refluxed for 2.5 h. The acetone was removed by evaporation, and the residual oil was chromatographed on Al_2O_3 (activity II, elution with chloroform). The chloroform was removed by distillation, and the residue was triturated with ether, and the solid IIa (0.77 g) was removed by filtration. 2-Spirocyclopentane-4,5,6,7-tetrahydro-2H-benzimidazole 1,3-dioxide (IIb), 2-methyl-2-ethyl-4,5,6,7-tetrahydro-2H-benzimidazole 1,3-dioxide (IIc), 2,2-dimethylcyclohepta-2H-imidazole 1,3-dioxide (IIIa), 2-spirocyclopentane-cyclohepta-2H-imidazole 1,3-dioxide (IIIb), and 2,2-dimethyl-2H-benzimidazole 1,3-dioxide (I) were similarly obtained. 2H-Imidazole 1,3-dioxides IIa-c and IIIa, b were obtained as light-yellow crystalline substances that were readily soluble in water and ordinary organic solvents.

Cyclohexanedione 1,2-Dioxime. A mixture of 2 g (11 mmole) of IIa, 80 ml of alcohol, and 2 g (28.8 mmole) of hydroxylamine hydrochloride was refluxed for 2 h, after which the alcohol was removed by distillation, and the residue was chromatographed on silica gel [chloroform—ethanol (9:1)] to give 1 g (50%) of cyclohexanedione 1,2-dioxime.

N-(1-Oximino-2-methyl-2-cyclohexyl)-tert-butylhydroxylamine (V). A 1-g (5.5 mmole) sample of IIa was added to a solution of 55 mmole of methylmagnesium iodide (prepared from 1.35 g of magnesium and 10 g of methyl iodide) in 100 ml of ether, and the mixture was refluxed for 2 h, after which it was decomposed with 100 ml of water. The organic layer was separated, the aqueous layer was extracted with ether, and the combined organic layer and ether extract was evaporated. The residue was treated with hexane, and the precipitated V was removed by filtration. The yield was 0.67 g (57%).

1-Hydroxy-2,2-dimethyl-4,5,6,7-tetrahydrobenzimidazoline 3-Oxide (IV). A 0.11-g (2.9 mmole) sample of $NaBH_4$ was added in portions at 5°C to a solution of 1 g (5.5 mmole) of dioxide IIa in 20 ml of water, and the mixture was stirred at this temperature for 30 min and at room temperature for 1 h. The solution was extracted with chloroform, and the chloroform

TABLE 2. Spectral Characteristics of the Synthesized 2H-Imidazole 1,3-Dioxides

Compound	PMR spectra, ppm					IR spectra,[b] cm^{-1}
	Solvent	2,2-CH_3	R	—CHR	—$(CH_2)_n$—[a]	
IIa	$CDCl_3$	1,67	H	2,5—2,9	1,7—2,2	1100, 1380, 1470, 1580
IIIa	$CDCl_3$	1,63	H	2,6—3,0	1,7—1,9	1070, 1300, 1380, 1560
VIII	$CDCl_3$	1,63	Br	5,3—5,4	1,9—2,8	1395, 1550
IX	$CDCl_3$	1,58; 1,67	Br	5,5—5,8	1,7—2,9	1160, 1405, 1530
X	$(CD_3)_2SO$	1,54	OH 3,33	4,5—4,9	1,6—2,0	1060, 1350, 1580
XI	CD_3OD	1,63	OH —	5,0—5,3	1,4—2,5	1060, 1380, 1470, 1530
XII	$(CD_3)_2SO$	1,67	$OCOCH_3$ 2,18	5,8—6,2	2,8—3,8	1060, 1230, 1410, 1570, 1750
XIII	$(CD_3)_2SO$	1,63	$OCOCH_3$ 2,04	5,8—6,2	1,3—2,3	1060, 1210, 1360, 1530, 1750

[a]For IIa, VIII, X, and XII, n = 2, while n = 3 for IIIa, IX, and XIII. [b]The most intense absorption bands at 1000-1800 cm^{-1} are presented.

extract was dried with $MgSO_4$. The solvent was removed by distillation, the residue was dissolved in ether, and the solution was allowed to stand in a refrigerator for 24 h. The resulting precipitate was removed by filtration to give 0.48 g (45%) of IV.

2,2-Dimethyl-4,5,6,7-tetrahydro-2H-benzimidazole 1,3-Dioxide (IIa). A 6.7-g (28 mmole) sample of PbO_2 was added to a suspension of 0.5 g (2.8 mmole) of IV in 100 ml of dry ether, and the mixture was stirred at 20°C for 40 h. The precipitate was removed by filtration, the solvent was removed from the filtrate by distillation, and the residual IIa was recrystallized. The yield was 0.15 g (30%).

4,7-Dibromo-2,2-dimethyl-4,5,6,7-tetrahydro-2H-benzimidazole 1,3-Dioxide (VIII). A 2.65-g (15 mmole) sample of N-bromosuccinimide and 0.001 g of benzoyl peroxide were added to a solution of 1 g (5.5 mmole) of dioxide IIa in 40 ml of CCl_4, and the mixture was refluxed for 1 h. The precipitate was removed by filtration, and the CCl_4 was removed from the filtrate by evaporation. The residue was triturated with ether, and the VIII was removed by filtration. The yield was 1.15 g. 4,8-Dibromo-2,2-dimethylcyclohepta-2H-imidazole 1,3-dioxide (IX) was similarly obtained.

4,7-Diacetoxy-2,2-dimethyl-4,5,6,7-tetrahydro-2H-benzimidazole 1,3-Dioxide (X). A 3-g (30.6 mmole) sample of potassium acetate was added to a solution of 1 g (2.94 mmole) of VIII in 20 ml of acetonitrile, and the mixture was stirred at room temperature with a magnetic stirrer for 24 h. The red solution was filtered, and the solvent was removed from the filtrate by distillation. The residue was treated with ether, and the ether-insoluble X was removed by filtration, washed with ether, and recrystallized to give 0.2 g of product. The ether solution was evaporated, and the residue was chromatographed on Al_2O_3 (elution with chloroform) to give 0.12 g (23%) of I. 4,8-Diacetoxy-2,2-dimethylcyclohepta-2H-imidazole 1,3-dioxide (XII) was similarly obtained.

4,7-Dihydroxy-2,2-dimethyl-4,5,6,7-tetrahydro-2H-benzimidazole 1,3-Dioxide (XII). A solution of 5 g (10 mmole) of dibromo derivative VIII in 150 ml of water was heated with stirring to 90°C. After the solid had dissolved completely, 2 g (20 mmole) of $CaCO_3$ was added in portions, and the mixture was stirred at 90°C for 30 min. The water was removed by evaporation in vacuo, and the residue was chromatographed on silica gel [elution with chloroform, chloroform—methanol (9:1), and methanol] to give 1.49 g of dihydroxy derivative XII. 4,8-Dihydroxy-2,2-dimethylcyclohepta-2H-imidazole 1,3-dioxide (XIII) was similarly obtained.

2,2-Dimethyl-4,7-dioxo-4,7-dihydro-2H-benzimidazole 1,3-Dioxide (XIV). A 3-g (34.5 mmole) sample of MnO_2 was added to a solution of 0.5 g (2.33 mmole) of dihydroxy derivative XII in 50 ml of acetone, and the mixture was stirred at room temperature for 24 h. The precipitate was removed by filtration, the filtrate was evaporated, and the residue was chromatographed on silica gel (elution with ether) to give 0.1 g of quinone XIV. IR spectrum: 1680 (C=O) and 1540 cm^{-1} (C=N). PMR spectrum (in $CDCl_3$): 1.86 ($2CH_3$) and 7.05 ppm (2CH).

1,2-Diacetamido-4-acetoxybenzene (VI). A 3-g (16.5 mmole) sample of dioxide IIa was added in portions with cooling to −5°C to a mixture of 30 ml of acetic anhydride and 2 ml of concentrated H_2SO_4, and the solution was stirred for 30 min. It was then neutralized with dry Na_2CO_3 and extracted with ethyl acetate. The extract was dried with $MgSO_4$ and evaporated, and the residual VI began to crystallize. The yield was 1.35 g (33%). An analytical sample was obtained by recrystallization from alcohol to give a product with mp 185-187°C (mp 187-188°C [12]). IR spectrum: 1680 (C=O), 1760 (C=O), and 1210 cm^{-1} (COOR). UV spectrum, λ_{max} (log ε): 220 (3.30) and 245 nm (3.15). PMR spectrum in $(CD_3)_2SO$: 2.0 ($2CH_3$), 6.8 (1H, q), 7.4 (1H, m), and 9.25 ppm (2NH, m).

1-Hydroxy-3,4-diacetamidobenzene (VII). A solution of 0.04 g (1 mmole) of NaOH in 5 ml of alcohol was added to a solution of 0.2 g (0.92 mmole) of VI in 10 ml of alcohol, and the mixture was heated to the boiling point and allowed to stand for 10 min. It was then neutralized with 5% HCl, and the solvent was evaporated. The residual VII began to crystallize. The yield of product with mp 212-215°C (from isopropyl alcohol) (mp 214-216°C [12]) was 0.12 g (70%). IR spectrum: 1680 cm^{-1} (C=O). UV spectrum, λ_{max} (log ε): 220 (3.30) and 245 nm (3.00). PMR spectrum in $(CD_3)_2SO$: 2.0 ($2CH_3$), 6.8 (1H, q), 7.4 (1H, m), and 9.25 ppm (2NH, m).

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