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## Photochemical isomerization of 2*H*-imidazole *N*-oxides\*

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1,3-Diaza-6-oxabicyclo[3.1.0]hex-3-enes and their 3-oxides were obtained by the photolysis of 2*H*-imidazole 1-oxides and 2*H*-imidazole 1,3-dioxides. 1,3-Diaza-6-oxabicyclo[3.1.0]hex-3-ene 3-oxides are thermally unstable and are converted to the starting 2*H*imidazole 1,3-dioxides; their further photolysis affords a mixture of stereoisomeric 1,3-diaza-4,7-dioxatricyclo[4.1.0.0<sup>3,5</sup>]heptanes.

Key words: 2*H*-imidazole *N*-oxides, photolysis; photochemical isomerization; nitrones; oxaziridines.

Photochemical isomerization of nitrones to oxaziridines has been extensively studied for various heterocyclic and acyclic nitrones.<sup>2-4</sup> When this reaction is reversible, its investigation is of interest in view of accumulating luminous energy; the use of cyclic nitrones is the most promising in this respect.<sup>5,6</sup> The synthesis of 2*H*-imidazole 1-oxides and 1,3-dioxides, which we have recently accomplished,<sup>7,8</sup> made it possible to study the photochemical isomerization of these compounds. In analogy with  $\alpha$ -dinitrones of the pyrazine series,<sup>9</sup> we could expect that the photolysis of 2*H*-imidazole 1,3-dioxides would afford highly strained tricyclic compounds containing two oxaziridine cycles.

The UV spectra of symmetric 2*H*-imidazole 1,3-dioxides (**1a,b**) exhibit a long-wave absorption band with  $\lambda_{max} \approx 360$  nm (Fig. 1).<sup>8</sup> Photolysis of solutions of these compounds with the light (an SS-1 filter) of a highpressure mercury lamp results in a decrease in the intensity of the long-wave absorption. Simultaneously a band appears with a shorter wavelength, characteristic of the mononitrone group, with maxima at 300 nm in the



Fig. 1. Changes in the UV spectrum during the photolysis of a 0.1 mM solution of compound 1b in hexane (l = 1 cm): the original spectrum (1); after photolysis with an SS-1 filter for 10 s (2) and for 45 s (3); after continued photolysis with unfiltered light for 30 s (4) and for 180 s (5).

<sup>\*</sup> For the previous communication, see Ref. 1.

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 $R^1 = Ph (a,c), Me (b); R^2 = Ph (a), Me (b), H (c)$ 

case of **1a** and 253 nm for **1b**. The fact that an isosbestic point exists indicates that the photochemical process is unidirectional. The quantum yield of the conversion in absolute ethanol at  $\lambda = 365$  nm is 0.38 for **1a** and 0.20 for **1b**.

After preparative-scale photolysis of solutions of 2*H*-imidazole 1,3-dioxides **1a,b** in nonpolar solvents the products of the isomerization of one of the nitrone groups (**2a,b**) were isolated. Compounds **2a,b** are readily and quantitatively isomerized into the starting 2*H*-imidazole 1,3-dioxides on a silica gel column, by boiling in hexane or ethyl acetate, or by storing their alcoholic solutions (Scheme 1). The time of the half-conversion of these oxaziridines to 2*H*-imidazole 1,3-dioxides in methanol at 50 °C is 45 min for **2a** and 60 min for **2b**. Due to the fast re-isomerization in MeOH, the preparative photolysis of compounds **1a,b** cannot be brought to completion.

When asymmetrical dinitrone **1c** is photolyzed in the long-wave absorption region (an SS-1 filter), the absorption band at 370 nm disappears, and two bands,  $\lambda_{\text{max}} = 302$  and 255 nm, appear; these bands are associated with the products of the isomerization of one of the nitrone groups, 2c and 3; the overall quantum yield ( $\phi$ ) of the conversion at  $\lambda_{max} = 365$  nm is 0.50. Oxaziridine 2c is thermally unstable and isomerizes at ~20 °C into the starting dinitrone 1c after 1 h. The intensity of the band at 302 nm associated with the absorption of oxaziridine 3 does not change under the experimental conditions. This made it possible to estimate the ratio between isomers 2c and 3, which was equal to 1: 3.3. Under the conditions of the preparative-scale photolysis of compound 1c in MeOH or in benzene, only one isomer, 3, can be isolated; it is stable and is not converted to the starting compound upon boiling in MeOH and sublimation.



The photolysis of 2*H*-imidazole 1-oxide **7a** (Scheme 2) in a way similar to that described for compounds **1a** and **1b** results in a decrease in the intensity of the band with  $\lambda_{max} = 293$  nm and in the appearance of the characteristic absorption band of phenylimine with a maximum at 252 nm. When 2*H*-imidazole 1-oxides **7a,b** were subjected to preparative-scale photolysis in the region of the long-wave absorption maximum, 1,3-diaza-6-oxabicyclo[3.1.0]hexanes<sup>10</sup> **8a,b** were isolated in both a protic (MeOH) and an aprotic (benzene) solvent (see Scheme 2). They are stable crystalline compounds and are not converted into the starting *N*-oxides either on silica gel or upon heating in ethanol or hexane. The characteristics of the compounds synthesized are given in Table 1.

The signals for the protons of the *gem*-methyl groups in the <sup>1</sup>H NMR spectra and the signals for the carbon atoms in the <sup>13</sup>C NMR spectra of 1,3-diaza-6-oxabicyclo[3.1.0]hexane 3-oxides **2a,b** and **3** differ noticeably from those of 1,3-diaza-6-oxabicyclo[3.1.0]hexanes **8a,b**. The signals for the protons at the C atom of the oxaziridine ring in the <sup>1</sup>H NMR spectra of compounds **3** and **8a** are found in the region of 5.2–5.52 ppm typical of oxaziridines (Tables 2, 3).

The continued photolysis of compounds 2a,b and 3 in benzene, THF, or ether (see Scheme 1) under conditions similar to those described for dihydropyrazine 1,4-dioxides,<sup>9</sup> gives pairs of diastereomeric dioxaziridines 4a-c and 5a-c (in the ratio 9 : 1 for 2a,b and 1 : 1 for 3), which were isolated. The resulting compounds do not exhibit absorption bands at  $\lambda > 210$  nm. The data from the <sup>1</sup>H NMR spectra of these compounds (see Table 2) do not allow the unambiguous determination of the spatial structure of the isomers. The assignment can be made on the basis of the <sup>13</sup>C NMR spectra (see Table 3): the spectra of *cis*-isomers 5, unlike those of trans-isomers 4 exhibit two signals for the gem-dimethyl carbon atoms. In addition, the signals for the C-4, C-5, and C-2 atoms of the imidazolidine ring are located in a higher field for cis-isomers, which may be due to an increased s-character of the hybridized orbitals of the atoms of the ring caused by high steric strain in the cisisomers. Thus, the photolysis of compounds 2a,b affords predominantly cis-isomers.

In contrast to compounds **2a-c** and 1,4-diaza-5,8dioxatricyclo[5.1.0.0<sup>4,6</sup>]octanes,<sup>9</sup> 1,3-diaza-4,7-dioxatri-

Com- pound	Yield (%)	M.p./°C	IR spectrum, v/cm <sup>-1</sup>	UV spectrum, $\lambda_{max}/nm \ (\log \epsilon)$	<u>I</u> (	Found Calculated	ī (%)	Molecular formula		
					С	Н	N			
2a	70	118-120	1540 (C=N)	300 (4.15)	<u>73.0</u> 72.8	<u>5.8</u> 5.8	<u>10.0</u> 10.0	C <sub>17</sub> H <sub>16</sub> N <sub>2</sub> O <sub>2</sub>		
2b	80	6870	1590 (C=N)	253 (4.08)	<u>53.5</u> 53.8	<u>8.1</u> 7.8	<u>17.9</u> 17.9	$C_7H_{12}N_2O_2$		
3	70	133-137	1550 (C=N)	302 (4.00)	<u>64.3</u> 64.7	<u>6.0</u> 5.9	<u>13.6</u> 13.2	$C_{11}H_{12}N_2O_2$		
<b>4</b> a	70	117-119		_	<u>72.7</u> 72.8	<u>5.8</u> 5.8	<u>9.8</u> 10.0	$C_{17}H_{16}N_2O_2$		
4b	45	35-38			<u>53.5</u> 53.8	<u>7.4</u> 7.8	<u>17.5</u> 17.9	$C_7H_{12}N_2O_2$		
4c	40	67—69			<u>64.2</u> 64.7	<u>5.9</u> 5.9	<u>13.2</u> 13.2	$C_{11}H_{12}N_2O_2$		
5a	8	7475			<u>73.0</u> 72.8	<u>5.8</u> 5.8	<u>9.5</u> 10.0	$C_{17}H_{16}N_2O_2$		
5b	5	Oil			<u>53.3</u> 53.8	<u>7.8</u> 7.8	<u>17.7</u> 17.9	$C_7H_{12}N_2O_2$		
5c	40	9799		-	<u>64.8</u> 64.7	<u>6.0</u> 5.9	<u>13.6</u> 13.2	$C_{11}H_{12}N_2O_2$		
7b	30	95—97	1600 (C=N); 1520, 1350 (NO <sub>2</sub> )	337 (4.00) 265 (4.15)	<u>58.9</u> 58.4	<u>5.5</u> 5.3	<u>17.1</u> 17.0	C <sub>12</sub> H <sub>13</sub> N <sub>3</sub> O <sub>3</sub>		
7c	60	122-124	1600 (C=N); 1530, 1370 (NO <sub>2</sub> )	290 (3.91)	<u>58.2</u> 58.4	<u>5.3</u> 5.3	<u>17.1</u> 17.0	$C_{12}H_{13}N_3O_3$		
8a	95	89—91	1610 (C=N)	252 (4.25)	<u>70.6</u> 70.2	<u>6.6</u> 6.4	<u>15.0</u> 14.9	$C_{11}H_{12}N_2O$		
8b	95	101-102	1630 (C=N); 1515, 1350 (NO <sub>2</sub> )	264 (4.13)	<u>58.8</u> 58.4	<u>5.4</u> 5.3	<u>17.1</u> 17.0	$C_{12}H_{13}N_3O_3$		
10	90	53-55	1695 (C=O); 1650, 1590 (C=N)	300 (3.80)	<u>54.6</u> 54.5	<u>7.1</u> 7.1	<u>14.2</u> 14.1	$C_9H_{14}N_2O_3$		
11	30	123—125	1740, 1700 (C=O)	372 (3.48), 285 (3.85), 247 (4.15)	<u>52.4</u> 52.5	<u>6.6</u> 6.6	<u>13.2</u> 13.1	C <sub>3</sub> H <sub>14</sub> N <sub>2</sub> O <sub>4</sub>		

Table 1. Characteristics of the compounds synthesized

cyclo[4.1.0.0<sup>3,5</sup>]heptanes 4 and 5 proved to be stable towards contact with silica gel and in polar solvents. The transformation of compound 4a to the starting  $\alpha$ -dinitrone 1a only occurs when it is heated in acetic acid at 80 °C for 3 h. The corresponding *trans*-isomer, 5a, is still more stable: it does not give compound 1a in acetic acid at 80 °C. Prolonged heating of compound 5a at a higher temperature affords a mixture of compounds that does not contain 1a, judging from the TLC data.

The melting point and spectral characteristics of isomer **5a** are close to those of a compound that has been obtained previously by the oxidation of 2,2-dimethyl-4,5-diphenyl-2*H*-imidazole (6) with  $H_2O_2$  in acetic acid.<sup>11</sup> The latter was mistaken for 2*H*-imidazole 1,3-dioxide. As we have already reported,<sup>8</sup> we did not manage to prepare this compound under the conditions described in Ref. 11. However, when 2*H*-imidazole **6** was kept in a mixture of AcOH and 30 %  $H_2O_2$  with the addition of few drops of  $H_2SO_4$  for 200 h at 25 °C,

compound 5a was obtained in 70 % yield, and isomer 4a was not formed at all. Isomer 5a is also obtained upon the oxidation of compound 6 with *m*-chloroperbenzoic acid.

The formation of different stereomeric oxaziridines during the oxidation of imines or the photolysis of the corresponding nitrones has been previously reported for five-membered heterocyclic compounds.<sup>2</sup> The oxidation of imines with peracids proved to be a more stereoselective reaction than the photochemical isomerization of nitrones. Actually, the oxidation of compound **8a** with  $H_2O_2$  in AcOH afforded only *trans*-dioxaziridine **5c** (cf. Ref. 10).

The data obtained on the ability of oxaziridines 2, 4, 5, and 8 to isomerize to the corresponding nitrones indicates that the formation of 2*H*-imidazole 1,3-dioxides during the oxidation of 2*H*-imidazoles with peracids or hydrogen peroxide is unlikely. At the same time, the oxidation of 2*H*-imidazole 1-oxides should

Compound	$Me_2C$ (s, 6 H)	Protons in R <sup>1</sup>	Protons in R <sup>2</sup>
2a	1.76, 1.72	7.34 (m, 5 H)	7.90–7.84 (m, 2 H, <i>o</i> -H); 7.37–7.30 (m, 3 H, <i>m</i> , <i>p</i> -H)
2b	1.44, 1.42	1.60 (s, 3 H)	1.98 (s, 3 H)
3	1.66, 1.65	8.30-8.20 (m, 2 H, o-H); 7.52-7.40 (m, 3 H, m,p-H)	5.52 (s, 1 H)
4a 4b	1.76, 1.68 1.42	7.24 (m, 5 H) 1.66 (s, 3 H)	7.24 (m, 5 H) 1.66 (m, 3 H)
4c	1.57, 1.55	7.65–7.55 (m, 2 H, o-H); 7.55–7.38 (m, 3 H, <i>m</i> , <i>p</i> -H)	5.10 (m, 1 H)
5 <b>a</b>	1.58	7.59–7.50 (m, 2 H, <i>o</i> -H); 7.40–7.30 (m, 3 H, <i>m</i> , <i>p</i> -H)	7.59–7.50 (m, 2 H, <i>o</i> -H); 7.40–7.30 (m, 3 H, <i>m,p</i> -H)
5b	1.38	1.67 (s, 3 H)	1.67 (m, 3 H)
5c	1.54, 1.50	7.57–7.53 (m, 2 H, o-H); 7.45–7.42 (m, 3 H, <i>m</i> , <i>p</i> -H)	5.38 (m, 1 H)
7 <b>b</b>	1.53	2.53 (s, 3 H)	8.10 and 8.23 (o-H and $m$ -H, AA'BB', $J = 9$ Hz)
7c	1.50	2.17 (s, 3 H)	8.00-8.23 (m, 1 H); 7.30-7.77 (m, 3 H)
8a	1.51, 1.50	7.90–7.80 (m, 2 H, <i>o</i> -H); 7.50–7.40 (m, 3 H, <i>m,p</i> -H)	5.21 (s, 1 H)
8b	1.52, 1.48	2.13 (s, 3 H)	7.58 and 8.28 (o-H and $m$ -H, AA'BB', $J = 8$ Hz)
10	1.38	2.37 (s, 3 H)	1.27 (t, 3 H); 4.27 (q, 2 H, $J = 7$ Hz)
11	1.66	2.34	1.31 (t, 3 H); 4.32 (q, 2 H, $J = 7$ Hz)

**Table 2.** <sup>1</sup>H NMR spectral data (CDCl<sub>3</sub>,  $\delta$ ) for the compounds synthesized

Table 3. <sup>13</sup>C NMR spectral data (CDCl<sub>3</sub>,  $\delta$ ) for the compounds synthesized

Com-	C-2	C-4	C-5	gem-CH <sub>3</sub>	R <sup>1</sup>							
pound					ipso-C	<i>o</i> -C	m-C	p-C	ipso-C	<i>o</i> -C	m-C	p-C
2a	94.26	86.68	135.72	24.75, 20.95	131.57	128.12	128.16	130.19	126.56	127.08	128.68	130.07
2b	95.01	86.96	138.65	26.14, 22.30	17.72				11.19			
3	95.37	133.95	77.66	24.29, 21.11	126.66	126.05	128.72	130.77				
<b>4</b> a	83.19	88.33	88.33	23.53, 20.11	131.78	127.55	128.03	129.26	131.78	127.55	128.03	129.26
4b	82.32	84.96	84.96	23.66, 19.97	16.61				16.61			
<b>4</b> c	84.72	85.36	85.36	23.28, 20.05	131.41	127.08	128.86	130.28				
5a	87.33	90.35	90.35	22.34	130.54	127.36	128.35	129.99	130.54	127.36	128.35	129.99
5b	86.10	90.46	90.46	22.30	14.06				14.06			
5c	88.36	89.81	89.81	22.20	130.12	127.04	128.61	130.51				
7b	100.48	164.35	133.80	24.37	19.50				132.30	123.75	128.57	147.75
7c	100.44	164.41	132.43	23.67	17.41				127.71	125.32, 148.06	130.99, 133.79	130.99
8a	94.90	166.22	80.05	26.72, 22.37	130.88	127.61	128.66	131.50				

afford unstable 1,3-diaza-6-oxabicyclo[3.1.0]hex-3-ene 3-oxides, which readily isomerize to 2*H*-imidazole 1,3-di-oxides (see Scheme 1).

In fact, the action of *m*-chloroperbenzoic acid in  $CHCl_3$  on 5-ethoxycarbonyl-2,2,4-trimethyl-2*H*-imidazole 1-oxide (10) prepared from ethyl isonitrosoacetoacetate (9) similarly to the procedure described in Ref. 7 resulted in the corresponding 2*H*-imidazole 1,3-dioxide (11) in 30 % yield (Scheme 3).



Scheme 3

## Experimental

IR spectra were recorded on a UR-20 spectrometer in KBr pellets (the concentration was 0.25 %, l = 1 mm). UV spectra were obtained with a Specord UV-VIS spectrometer in EtOH. <sup>1</sup>H and <sup>13</sup>C NMR spectra were run on a Bruker AC 200 spectrometer for 1-5 % solutions in CDCl<sub>3</sub>, the signal of the solvent was used as the standard. The assignment of the signals in the <sup>13</sup>C NMR spectra was based on the analysis of the intensities and on the spectra with incomplete <sup>1</sup>H-<sup>13</sup>C decoupling. The photolysis was performed with the light of a DRSh-500 high-pressure mercury lamp equipped with a condenser. Standard light filters were used for the isolation of the intensities of spectral bands. The rate of photochemical isomerization was determined on the basis of the absorption of the starting compounds at 345-370 nm, the light intensity was measured with the aid of a ferrioxalate actinometer.<sup>12</sup> The quantum yield of the isomerization was determined at 365 nm (the combination of BS-6 and UFS-6 filters) at a concentration of 10 mol  $L^{-1}$ . The course of the process was monitored with a Specord UV-VIS spectrophotometer. The dissolved oxygen was not removed from the solutions. The reaction mixtures were analyzed by TLC on Silufol UV 254 plates using chloroform or a 1 : 1 ether-hexane mixture as the eluent and a 10 % aqueous solution of NaI as the spraying reagent for oxaziridines.

Preparation of oxaziridines 2a,b, 3, 8a,b, 4a-c, and 5a-c(general procedure). A solution of 1 mmol of the corresponding nitrone (compounds 1a-c, 7a,b, 3, and 2a) in 100 mL of benzene (or in THF in the case of compound 2b) was placed in a water-cooled quartz cell and irradiated through a light filter until the starting compound disappeared (according to TLC), and the solvent was evaporated. Mixtures of compounds 4 and 5 were separated by chromatography on a silica gel column using ether—hexane (1 : 4) as the eluent. Compounds 2a and 8a,b were recrystallized from a 3 : 1 hexane—ethyl acetate mixture, compounds 2b, 3, 4a-c, and 5a-c were recrystallized from hexane.

2,2,4-Trimethyl-5-(4-nitrophenyl)-2*H*-imidazole 1-oxide (7b) and 2,2,4-trimethyl-5-(2-nitrophenyl)-2*H*-imidazole 1-oxide (7c). 1.3 g (6 mmol) of 2,2,4-trimethyl-5-phenyl-2*H*imidazole 1-oxide (see Ref. 13) was dissolved in 35 mL of conc.  $H_2SO_4$ , and 10 mL of the nitrating mixture (prepared by mixing 100 mL of 56 % HNO<sub>3</sub> and 200 mL of 60 % oleum) was added dropwise with stirring. The reaction mixture was stirred for 2 h and poured into 300 mL of cold water.  $Na_2CO_3$ was added to the resulting mixture to pH 7, and the mixture was extracted with chloroform (3×50 mL). The extract was dried with MgSO<sub>4</sub>, the solvent was evaporated, and the residue was chromatographed on a silica gel column using chloroform as the eluent. The resulting compounds were recrystallized from a 2 : 1 hexane—ethyl acetate mixture.

2,2-Dimethyl-5,6-diphenyl-1,3-diaza-4,7-dioxatricyclo[4.1.0.0<sup>3,5</sup>]heptane (5a). 0.5 g (0.002 mmol) of 2*H*-imidazole 6 was dissolved in a mixture of 5 mL of AcOH and 1 mL of  $H_2O_2$ , and 1–2 drops of 20 %  $H_2SO_4$  was added. The mixture was allowed to stand for 200 h, diluted with 20 mL of water, and extracted with chloroform. The extract was dried with MgSO<sub>4</sub>, concentrated, and the residue was chromatographed on a column with silica gel using chloroform as the eluent.

In a similar way, 2,2-dimethyl-5-phenyl-1,3-diaza-4,7dioxatricyclo[ $(4.1.0.0^{3,5}]$ heptane (**5c**) was prepared by the oxidation of 2,2-dimethyl-4-phenyl-1,3-diaza-6-oxabicyclo[(3.1.0]hex-3-ene (**8a**). Isomerization of compound 4a to 2*H*-imidazole 1,3-dioxide (1a). A solution of oxaziridine 4a in AcOH was heated for 3 h at 80 °C, diluted with a tenfold amount of water, and extracted with chloroform. The extract was dried with MgSO<sub>4</sub>, concentrated, and chromatographed on a column with silica gel using chloroform as the eluent.

5-Ethoxycarbonyl-2,2,4-trimethyl-2*H*-imidazole 1-oxide (10). A mixture of 15 g (0.095 mol) of isonitrosoacetoacetic ester (9), 15 g of NH<sub>4</sub>OAc, 30 mL of acetone, and 30 mL of acetic acid was allowed to stand for 5 days at 20 °C, diluted with 150 mL of water, and extracted with chloroform. The extract was dried with MgSO<sub>4</sub> and concentrated, the residue was dissolved in 25 mL of a 4 : 1 hexane—ether mixture, and left to stand for 24 h at -5 °C. Compound 10 precipitated and was recrystallized from hexane.

5-Ethoxycarbonyl-2,2,4-trimethyl-2*H*-imidazole 1,3-dioxide (11). 2 g (0.0116 mol) of *m*-chloroperbenzoic acid was added to a solution of 1 g (0.0025 mol) of compound 10 in 5 mL of chloroform cooled to 10 °C, and the mixture was allowed to stand at this temperature for 48 h. The resulting solution was chromatographed on a column with silica gel (using ether as the eluent); the resulting compound was recrystallized from a 2 : 1 hexane—ethyl acetate mixture.

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