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AZIRIDINYL KETONES AND THEIR CYCLIC ANILS.

7.* SOLVOLYSIS OF 1,2-DIARYL-1,1a-DIHYDROAZIRINO[1,2-a]-QUINOXALINES

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Refluxing of 1,2-diaryl-1,1a-dihydroazirino[1,2-a]quinoxaline in 1-propanol leads to 3-aryl-1-arylmethyl-2-propoxy-1,2-dihydroquinoxalines or to aryl(3-aryl-1-arylmethyl-1,2-dihydroquinoxalin-2-yl)(3-aryl-2-hydroxy-1,2-dihydroquinoxalin-1-yl)methanes. Both processes are due to opening of the C—C bond of the aziridine ring; however, in the first process the ylids formed react with solvent molecules, whereas in the second process dimerization of the ylids with the participation of the water present in the solvent is observed.

1,1a-Dihydroazirino[1,2-a]quinoxaline derivatives have an interesting chemical peculiarity — their three-membered ring is capable of opening at either of its external bonds. Thus in acidic media these compounds readily undergo rearrangement to quinoxaline derivatives as a result of intermediate opening of the aziridine ring at the C—N bond [2, 3]. The capacity of 1,2-diaryl-1,1a-dihydroazirino[1,2-a]quinoxalines for photochromism [2] is due to opening of the C—C bond of the three-membered ring and the formation of deeply colored 1,1a-ylids [4, 5]. The thermochromism of these compounds noted in [6] is probably due to a similar process (evidence for which is provided by the identical character of the coloration that develops); however, this problem has not been subjected to a detailed study.

It is also known [7] that cleavage of the common bond to give a seven-membered azepine ring is possible to polycyclic systems that contain a 1,2-fused aziridine ring.

In the present research we studied the chemical behavior of 1,2-diaryl-1,1a-dihydroazirino[1,2-a]quinoxalines (I) when they are refluxed in methanol, ethanol, and 1-propanol.

*See [1] for communication 6.

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TABLE 1. Products of the Transformations of Dihydroquinoxalines I

Compound	mp, °C	R_f	λ_{max}^{abs}, nm ($\varepsilon \cdot 10^{-3}$)	λ_{max}^{em}, nm	Found, %			Empirical formula	Calc., %			Yield, %
					C	H	N		C	H	N	
IIa	94	0,13	372 (6,9), 296 (12,4), 262 sh., 240 (21,9)	510	80,95	6,80	7,91	$C_{24}H_{24}N_2O$	80,89	6,74	7,86	46
IIb	119	0,11	369 (4,7), 291 sh., 260 (25,3), 236 (21,3)	480	71,89	5,79	10,48	$C_{24}H_{23}N_3O_3$	71,82	5,74	10,47	58
IIc	129	0,12	413 (8,7), 331 sh., 272 (24,1), 232 (28,1)	—[a]	51,25	5,04	12,49	$C_{24}H_{22}N_4O_5$	51,12	4,93	12,56	63
IId	128	0,13	376 (7,7), 291 sh., 266 (27,7), 237 (21,9)	506	60,09	4,45	8,82	$C_{24}H_{22}BrN_3O_3$	60,00	4,58	8,75	72
IIe	124	0,08	412 (3,9), 333 (16,5), 276 (20,2), 236 (23,7)	524[a]	71,97	5,79	10,52	$C_{24}H_{23}N_3O_3$	71,82	5,74	10,47	63
IIIa	216	0,36	397 (1,0), 314 (8,7), 255 (32,8)	510	82,73	5,61	9,11	$C_{42}H_{34}N_4O$	82,62	5,57	9,18	71
IIId	228	0,17	434 sh., 364 (22,5), 306 sh., 270 sh.	446	58,82	3,64	9,92	$C_{42}H_{30}Br_2N_6O_5$	58,74	3,50	9,79	49
IIIe	183	0,12	413 (2,1), 333 (26,2), 279 (35,5), 242 (36,9)	—[a]	72,12	4,51	12,15	$C_{42}H_{32}N_6O_5$	72,01	4,57	12,00	27
IIIf	232	0,34	397 (3,0), 314 (10,7), 256 sh.	506	82,81	6,09	9,05	$C_{44}H_{38}N_4O$	82,75	6,00	8,77	61
IV	189	0,09	392 (2,7), 305 sh., 262 (46,7)[b]	—[a]	73,83	4,45	11,90	$C_{42}H_{30}N_6O_4$	73,90	4,40	12,30	10

[a]Very weak fluorescence.
[b]In $CHCl_3$.

TABLE 2. Chemical Shifts (δ, ppm) in the PMR Spectra of 2-Propoxy-1,2-dihydroquinoxalines II in $CDCl_3$

Compound	*n*-C_3H_7[a]			CH_2 (s)	2-CH (s)
	CH_3 (t)	CH_2 (m)	CH_2 (t)		
IIa	0,75	1,46	3,05	4,90	6,08
IIb	1,00	1,52	3,20	4,98	5,98
IIc	0,92	1,47	3,06	5,05	6,12
IId	0,91	1,53	3,05	5,00	6,05
IIe	0,85	1,46	3,05	5,12	5,97

[a]The spin–spin coupling constants range from 6.0 to 6.4 Hz.

The starting compounds do not undergo appreciable changes in refluxing methanol and ethanol; in fact, they are recrystallized (one should also bear in mind that the synthesis of azirinoquinoxalines is also realized in refluxing methanol [6]). On the other hand, the formation of new compounds that can be arbitrarily separated into low-melting (II, mp <130°C) and high-melting (III and IV, mp >180°C) compounds is observed even in the case of brief refluxing of I in 1-propanol.

The yields of products of the II type are higher when derivatives that contain an NO_2 group (Ib-e) are subjected to the reaction. The presence of water in the 1-propanol favors the formation of substances of the III series. For example, IIId,e can be obtained only when the alcohol is saturated with water, whereas IIIa,f are the only products of solvolysis of azirinoquinoxalines Ia,f simply in the ordinary undried solvent. Correspondingly, we were able to obtain IIa only when we rendered the propanol absolute with sodium metal and heated the reaction mixture in sealed ampuls, whereas If did not give a product of the II series even under these conditions.

Compound IV was obtained in low yield in those experiments on the solvolysis of azirinoquinoxaline Ib in which the filtrate remaining after separation of product IIb was evaporated and subjected to additional refluxing for 2-3 h. We were unable to detect the formation of compounds similar to IV in the remaining cases.

TABLE 3. Chemical Shifts (δ, ppm) in the PMR Spectra of III at 30°C

Compound	Solvent	CH_AH_B[a]		CH_A-CH_B[b]		CHOH	
		H_A	H_B	H_A	H_B	OH[c]	CH
IIIa	$CDCl_3$	3,74	4,28	4,29	4,45	6,04	4,43
IIIa	$(CD_3)_2CO$	3,72	4,32	4,36	4,42	5,68	4,37
IIIa	$(CD_3)_2SO$	3,66	4,26	4,27	4,40	6,02	4,36
IIIf[d]	$(CD_3)_2SO$	3,81	4,37	4,33	4,48	5,96	4,47

[a] $^2J_{AB}$ = −15.5 Hz.
[b] $^3J_{AB}$ = 8.5 Hz.
[c] The δ_{OH} values for IIIa are 5.45 ppm in $(CD_3)_2CO$ (at 60°C), 5.81 ppm in $(CD_3)_2SO$ (at 60°C), and 5.67 ppm in $(CD_3)_2SO$ (at 80°C); the δ_{OH} value for IIIf is 5.87 ppm in $(CD_3)_2SO$ (at 60°C).
[d] The methyl groups give two singlet signals at 2.22 and 2.26 ppm.

TABLE 4. Mass Spectra of II-IV[a]

Compound	m/z values (relative intensities, %)[b]
IIb	342 (2), 207 (17), 206 (100), 205 (8), 180 (8), 179 (43), 178 (16), 177 (5), 153 (5), 152 (9), 151 (9), 104 (8), 103 (21), 77 (18), 76 (47), 75 (12), 60 (56), 59 (58), 57 (13)
IIc	387 (2), 270 (19), 252 (20), 251 (100), 224 (9), 221 (22), 205 (35), 193 (13), 178 (28), 177 (13), 152 (9), 151 (25), 150 (16), 103 (11), 102 (22), 77 (36), 76 (45), 75 (17), 60 (48), 59 (50), 57 (14)
IIe	342 (1), 252 (8), 251 (53), 221 (13), 205 (21), 193 (8), 180 (18), 179 (21), 178 (7), 151 (8), 150 (13), 149 (84), 108 (21), 107 (100), 106 (21), 105 (47), 103 (8), 102 (16), 79 (40), 78 (13), 77 (76), 76 (30), 75 (13), 60 (47), 59 (42), 57 (23)
IIIa	610 (4), 387 (2), 299 (5), 298 (30), 297 (100), 295 (5), 222 (1), 208 (10), 207 (59), 206 (24), 194 (1), 179 (10), 129 (6), 103 (5), 92 (7), 91 (50), 77 (5), 76 (6)
IIId	302 (5), 300 (5), 286 (14), 284 (14), 274 (5), 272 (5), 205(6), 184 (5), 183 (5), 178 (8), 152 (5), 151 (14), 150 (10), 102 (6), 86 (32), 77 (15), 76 (15), 75 (8), 71 (12), 65 (7), 58 (8), 57 (100), 56 (82)
IIIf	638 (1), 401 (1), 312 (19), 311 (100), 222 (6), 221 (48), 220 (22), 208 (1), 193 (5), 129 (3), 92 (8), 91 (21), 77 (2), 76 (3)
IV	342 (8), 341 (15), 340 (8), 294 (5), 207 (18), 206 (100), 180 (9), 179 (48), 178 (18), 153 (6), 152 (11), 151 (11), 150 (6), 137 (22), 107 (9), 106 (6), 104 (9), 103 (23), 102 (9), 91 (28), 77 (38), 76 (54), 75 (14), 74 (9), 65 (28), 63 (19), 60 (16), 59 (14), 57 (13)

[a] The temperatures at which the samples were heated were 80°C for IIb, 60°C for IIc, 60°C for IIe, 40°C for IIIa, 60°C for IIId, 30-50°C for IIIf, and 120°C for IV.
[b] The peaks of ions with m/z 50 and with intensities ≥5% of the maximum intensity, as well as the molecular-ion peaks, are presented.

The PMR, UV (absorption and emission), and mass spectra of the compounds obtained, as well as the results of elementary analysis, are presented in Tables 1-4.

Intense νC=N bands (1600-1616 cm^{-1}) are observed in the IR spectra of all II-IV, but bands of vibrations of a three-membered ring at 1010-1075 cm^{-1} [6] are absent; in the case of the compounds obtained from azirinoquinoxalines Ib-e the $\nu_{as}NO_2$ (1516-1522 cm^{-1}) and ν_sNO_2 (1343-1356 cm^{-1}) bands are retained in the spectra. It may be assumed that the aziridine ring of I is opened in the process of solvolysis. The formation of primarily derivatives of quinoxaline, dihydroquinoxaline, or diazepine derivatives is possible in this case. However, in acidic media diazepines give the intense dark-blue coloration that is so characteristic for the diazatropylium ion [8], whereas II-IV do not display this reaction, and this makes it possible to exclude the assumption of a diazepine structure.

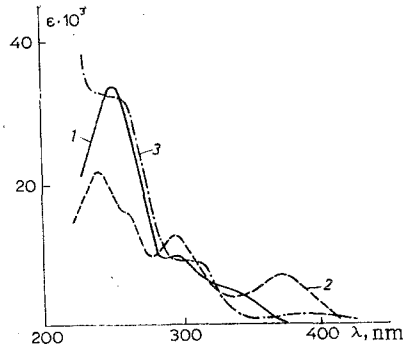


Fig. 1. Electronic absorption spectra in methanol: 1) Ia; 2) IIa; 3) IIIa.

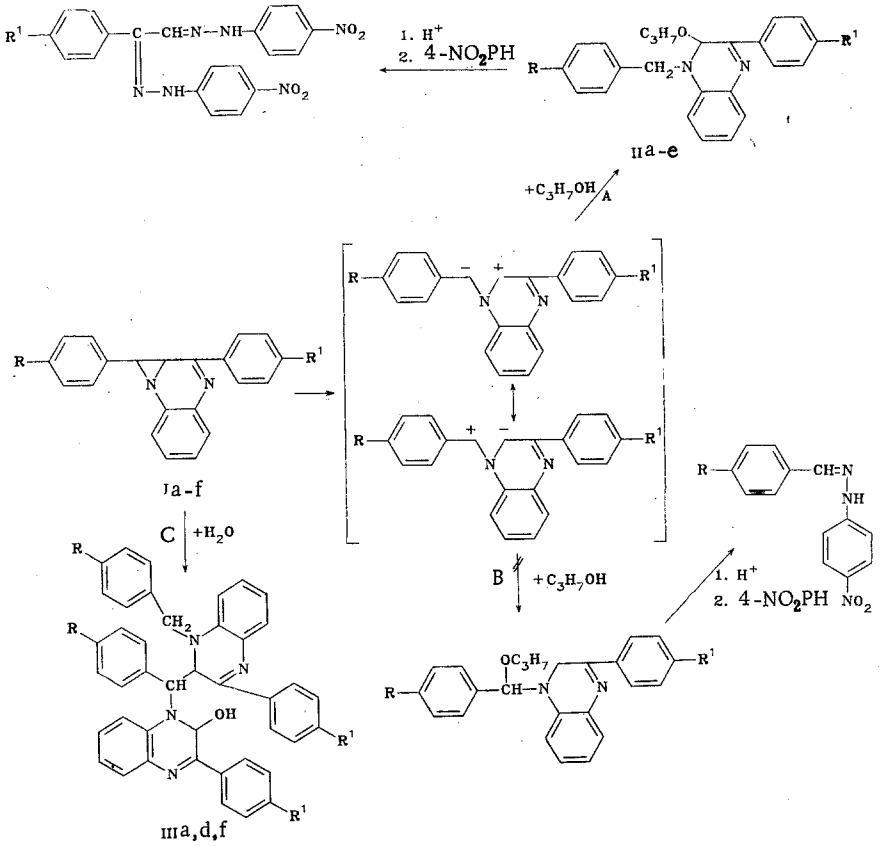
A common feature of II-IV is the presence of green or blue fluorescence (although it is weak in a number of the compounds), which is absent in the case of the starting compounds. Considering the fact that fluorescence is not characteristic for quinoxalines, whereas, on the other hand, it is characteristic for their dihydro derivatives [9], it may be assumed that the molecules of the compounds that are formed when they are heated in propanol contain a dihydroquinoxaline fragment. Moreover, in contrast to NH-dihydroquinoxalines [10], II-IV are stable during storage, which indicates the tertiary character of the nitrogen atom in the 1 position of the heteroring.

The UV spectra of several groups of compounds that have the same $-\underset{|}{N}-C_6H_4-N=\underset{|}{C}-Ar$ chromophore system were analyzed thoroughly in [11]. Their common properties include a monotonic decrease in the intensities of the bands in the near-UV and visible regions with an increase in the starting and a bathochromic shift of the long-wave band with a decrease in the strain of the chromophore system or when electron-acceptor substituents are introduced into the aryl ring. The absorption spectra of the starting compound (Ia) and the final compounds (IIa and IIIa) presented in Fig. 1 are also characterized by the above-noted sequence in the change in the intensities of the bands and, in addition, by the deeper absorption of products IIa and IIIa as compared with Ia. These regularities are also retained in the spectra of the other transformation products (IIb-e and IIId-f, as well as IV). The inclusion of an NO_2 group as substituent R^1 in the molecule gives rise to a bathochromic shift of the long-wave absorption of both the starting aziridinyl ketones (see the λ_{max} values of Ia,c,e, which are equal to 345, 385 [11], and 387 nm) and final products II and IV (see Table 1). These facts constitute evidence for the retention of the chromophore system under discussion in the course of the chemical transformations of azirinoquinoxalines I and are in agreement with the proposed dihydroquinoxaline structure of II-IV.

The PMR spectra gave valuable information regarding the structures of the compounds obtained, although the measurements were hindered because of the low solubilities of most of the products. We found that, in addition to signals of aromatic protons, signals of isolated $n-C_3H_7$, CH_2, and CH groups are observed in the spectra of II (Table 2). Their positions constitute evidence that the indicated groups are bonded to heteroatoms, and the presence of a propyl group indicates reaction of the starting azirinoquinoxaline with the solvent (1-propanol). One can conceive of this process if one takes into account our previously obtained data [6], according to which azirinoquinoxalines at temperatures above 95°C form 1,1a-ylids, which in turn could react with 1-propanol via, in principle, two pathways (pathways A and B in the reaction scheme).

The available spectral characteristics of the products are in equally good agreement with each of the two possible structures, and II were therefore subjected to hydrolysis in an acidic medium with treatment of the resulting complex mixtures of products with p-nitrophenylhydrazine in order to establish the specificity of the solvolysis of II. In all cases we identified osazones of the corresponding arylglyoxals, which indicates that the process takes place via pathway A. In fact, if solvolysis had proceeded via pathway B, hydrolysis of the products with subsequent treatment with p-nitrophenylhydrazine would have led to hydrazones of substituted benzaldehydes, and this was not observed experimentally. These results, together with the similarity in the PMR spectra (Table 2), make it possible to assign II to

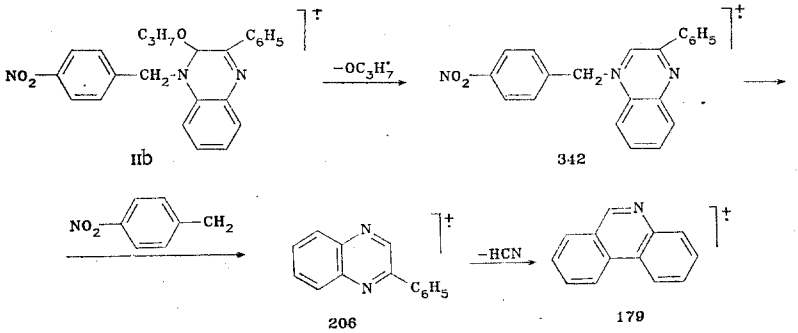
to the same series of isomers and thereby confirm the general character of the scheme of their formation.



a R=R¹=H; b R=NO₂, R¹=H; c R=R¹=NO₂;
d R=NO₂, R¹=Br; e R=H, R¹=NO₂; f R=H, R¹=CH₃

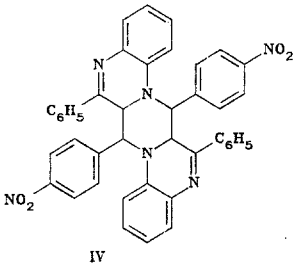
High-melting products III have extremely low solubilities. We were able to record sufficiently resolved PMR spectra only for IIIa and IIIf (Table 3). A singlet, the shift of which is sensitive to changes in the temperature and solvent, is present at medium field in the spectra. In addition, this signal vanishes when D_2O is added to the solutions. It follows from these data that the signal belongs to the OH group. A doublet, which is a part of an AB spin system with a constant of 15.5 Hz, the magnitude of which is typical for geminal spin-spin coupling constants (SSCC), is observed at 3.57-3.80 ppm. The doublet of the second proton of this spin system is found at 4.20-4.50 ppm, in which region, according to the results of measurements of the integral intensities, one finds signals of yet another three protons, two of which form a separate AB system with SSCC 8.5 Hz, whereas one gives a singlet peak. The signals of the aromatic protons form a complex multiplet at weak field (6.0-8.5 ppm) with an overall integral intensity corresponding to more than 25 protons. The data obtained can be interpreted only by assuming that III have dimeric structures and that water participates in their formation. The results of elementary analysis are also in good agreement with this. Only the possible structure presented above in the scheme can correspond to dimers III, which contain both dihydroquinoxaline fragments and the indicated proton systems.

The proposed structures of IIb,c,e are also in agreement with their mass spectra (Table 4). A common feature of these spectra is the fact that, in contrast to starting azirinoquinoxalines I, molecular ions are absent in this case. The instabilities of the molecular ions of adducts II are probably due to the presence of an N,O-acetal fragment [12]; the OC_3H_7 group is split out extremely readily and gives the $(M - 59)^+$ ion observed in the spectra. The fragmentation of II under the influence of electron impact can be represented as follows in the case of adduct IIb:



The molecules of dimers III undergo thermal decomposition during recording of the mass spectra, and the intensities of the individual ions therefore depend substantially on the input temperature. Molecular-ion peaks can be recorded in the spectra of IIIa,f measured at low input temperatures, and the ions of monomeric fragments become the most intense ions. A molecular-ion peak is not observed in the spectrum of IIId, and all of the largest fragment ions contain bromine atoms.

Compound IV occupies a special place in the group of high-melting products. We were unable to record its PMR spectrum because of its extremely low solubility. According to the results of elementary analysis, the composition of IV coincides with the composition of the



IV

starting compound, and its mass spectrum is virtually identical to the mass spectrum of IIb. One can only assume that this compound is the product of dimerization of the 1,1a-ylid with a saturated pyrazine ring, viz., 7,15-bis(p-nitrophenyl)-6,14-diphenyl-6a,7,14a,15-tetrahydroquinoxalino[1',2':1,2]pyrazino[4,5-a]quinoxaline (IV).

This assumption explains the stability of IV and the retention of its luminescence properties and is in agreement with its UV and IR spectra.

EXPERIMENTAL

The IR spectra of KBr pellets of the compounds were recorded with a Specord IR-75 spectrometer. The electronic absorption spectra of solutions in methanol [$(2\text{-}4)\cdot10^{-5}$ mole/liter] were recorded with a Specord UV-vis spectrophotometer. The fluorescence spectra were obtained in methanol with an apparatus created on the basis of the monochromator from an SF-4 spectrophotometer and an FÉU-38 energy adapter. Monochromatic light (365 nm) was isolated from the spectrum of a DRSh-500 mercury lamp with UFS-I-2 glass filters. The spectra were corrected for the spectral sensitivity of the apparatus. The PMR spectra of solutions in $CDCl_3$ and $(CD_3)_2SO$ were recorded at 30-80°C with Varian XL-100 (100 MHz) (IIa,b and IIIa,f) and Tesla BS 487-B (80 MHz) (IIa-d) spectrometers, as well as with a Brucker WH-200 spectrometer (200 MHz) [for IIIa in $(CD_3)_2CO$)], with tetramethylsilane as the internal standard. The mass spectra were obtained with a Varian MAT CH-6 mass spectrometer with direct introduction of the samples into the ion source; the temperature of the ionization chamber was 180°C, the ionizing voltage was 70 eV, and the temperature at which the samples was heated was varied as a function of their volatilities (see Table 4). The individuality of the compound obtained was monitored by TLC on Silufol UV-254 plates with elution by chloroform.

The synthesis of Ia-f was described in [2, 6].

1-Benzyl-2-propoxy-3-phenyl-1,2-dihydroquinoxaline (IIa). A 5-ml sample of 1-propanol that had been dried in a Fischer pistol over P_2O_5, and the resulting solution was heated for 20 min in a sealed ampul on a boiling-water bath. It was then cooled and chromatographed with a column packed with silica gel by elution with hexane. Evaporation gave 0.44 g (46%) of light-yellow crystals of IIa with mp 94°C (from hexane).

1-(4-Nitrophenylmethyl)-2-propoxy-3-phenyl-1,2-dihydroquinoxaline (IIb). A 3.0-g (8.8 mmole) sample of Ib was dissolved in 30 ml of dry 1-propanol, and the solution was refluxed for 1 h, during which the formation of a precipitate was observed. The mixture was cooled to give 2.04 g (58%) of IIb with mp 119°C (from methanol). Compounds IIc,d,e were similarly obtained.

(2-Hydroxy-3-phenyl-1,2-dihydroquinoxalin-1-yl)phenyl(3-phenyl-1-phenylmethyl-1,2-dihydroquinoxalin-2-yl)methane (IIIa). A 3.0-g (10.1 mmole) sample of Ia was heated in 30 ml of 1-propanol (chemically pure) for 1 h, after which the mixture was cooled to precipitate 2.2 g (71%) of light-yellow crystals of IIIa with mp 216°C (from chloroform).

Compounds IIId,f were similarly obtained. The conditions used to synthesize IIIe were identical, except that 0.5 ml of water was added to 30 ml of 1-propanol.

7,15-Bis(p-nitrophenyl)-6,14-diphenyl-6a,7,14a,15-tetrahydroquinoxalino[1',2':1,2]-pyrazino[4,5-a]quinoxaline (IV). The filtrate obtained after separation of IIb was evaporated to a volume of 10 ml, and the concentrate was refluxed for 2-3 h to precipitate 0.3 g (10%) of IV with mp 189°C.

Hydrolysis of 1-(4-Nitrophenylmethyl)-2-propoxy-3-phenyl-1,2-dihydroquinoxaline (IIb). Hydrolysis was realized by the method proposed in [13] for oxazolidine derivatives. A mixture of 1 g of IIb, 2 ml of concentrated hydrochloric acid, and 15 ml of water was heated for 30 min on a boiling-water bath, after which it was cooled and treated with ether. The ether extract was dried, the solvent was evaporated, and the residue was dissolved in 10 ml of methanol. The resulting solution was mixed with 1 ml of a 5% alcohol solution of p-nitrophenylhydrazine hydrochloride to precipitate phenylglyoxal osazone with mp 310°C (dec.) (mp 310-311°C [14]) and R_f 0.06. No melting-point depression was observed for a mixture of this product with a sample obtained from phenylglyoxal. A similar procedure was used for the hydrolysis of IIa (the same osazone), IIc,e [p-nitrophenylglyoxal p-nitrophenylosazone, with mp 320°C (dec.) and R_f 0.13, was isolated. Found: N 21.52%. $C_{20}H_{15}N_7O_6$. Calculated: N 21.82%], and IId [p-bromophenylglyoxal p-nitrophenylosazone, with mp 330°C (dec.) and R_f 0.10, was obtained. Found: N 16.85%. $C_{20}H_{15}BrN_6O_4$. Calculated: N 17.39%. The product did not give a Beilstein reaction for halogens].

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