

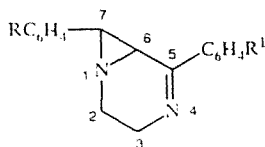
AZIRIDINYLKETONES AND THEIR CYCLIC ANILS. 12*

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We have measured the PMR spectra of 5,7-diaryl-1,4-diazabicyclo[4.1.0]hept-4-enes. We have studied the structure of the 5-(4-nitrophenyl)-7-phenyl derivative by x-ray diffraction. We show that the six-membered heterocycle has the half-chair form. The 6-H and 7-H protons of the aziridine ring are trans, while the 7-aryl is exo relative to the six-membered ring. Introduction of substituents into the aromatic ring has practically no effect on the geometry of the bicycle.

Cyclic anils of aziridinyllketones form a group of effective photochromic compounds, changing color under UV light from colorless to blue-violet [1, 2]. Their chemical conversions, in which they participate in ylide form, are also interesting [2-4].

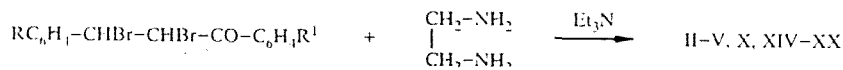
The goal of our work was to establish the structure of 5,7-diaryl-1,4-diazabicyclo[4.4.0]het-4-ene molecules (I-XXII) by PMR and x-ray diffraction analysis. Several papers have been devoted to these compounds: their synthesis has been described in [2, 5]; their acidolysis in [5]; their spectral and photochromic properties in [6]; the structure of four isomers of 2,3-diphenyl-substituted 1,4-diazabicyclo-[4.1.0]hept-4-ene has been discussed in [7].



I-XXII

I-III R = H, IV R = 4-Br, V R = 2-NO₂, VI R = 3-NO₂, VII-XIV, XIX, XXI, XXII R = 4-NO₂,
XV R = 4-C₆H₅, XVI-XVIII R = 2-Br-4-NO₂, XX R = 5-(2-NO₂-thienyl); I, IV-VII, XVI, XX
R¹ = H, II, III, XI, XV, XVIII R¹ = 4-NO₂, VIII R¹ = 4-Cl, IX R¹ = 4-Br, X R¹ = 2-NO₂,
XII R¹ = 4-CH₃, XIII R¹ = 4-OCH₃, XIV R¹ = 2,4-(OCH₃)₂, XVII R¹ = 4-Br, XIX R¹ = 2-thienyl
instead of C₆H₅R), XXI R¹ = 4-SO₂CHF₂, XXII R¹ = 4-SO₂C₃F₇

The compounds II-V, X, XIV-XX not described previously (Table 1) were obtained according to the technique in [5] by reaction of α,β -dibromides of the corresponding chalcones with ethylenediamine in methanol in the presence of triethylamine.



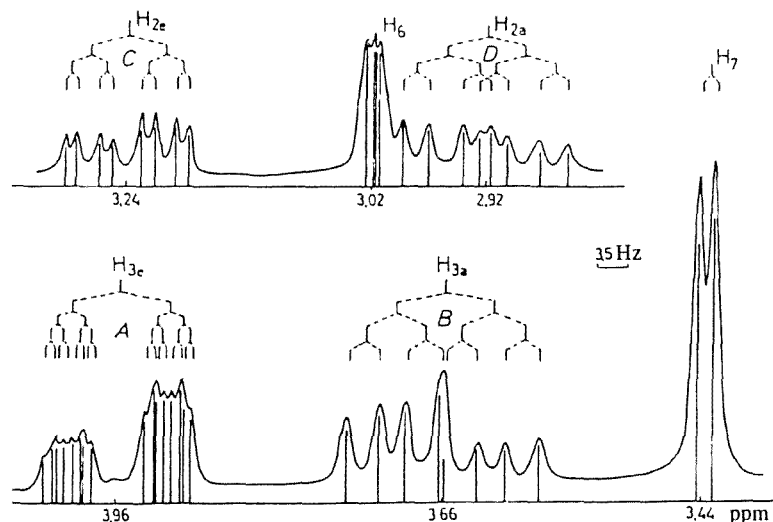
*For Communication 11, see [1].

[†]Deceased.

TABLE 1. Characteristics of Synthesized Compounds

Compound*	mp, °C	UV spectrum, λ_{\max} , nm ($\epsilon \cdot 10^{-3}$), molecular form	IR spectrum					k_f	Yield, %
			ν_{C-N}	$\nu_{NO_2^{ac}}$	$\nu_{NO_2^c}$	$\nu_{N^{ac}}$	ν_{N^c}		
II, III	124	275 (14,7)	1622	1523	1348	893	1236	0,26	60
IV	113	245 (20,4)	1619	—	—	881	1228	0,50	41
V	128	241 (21,5)	1626	1520	1350	892	1239	0,45	64
X	124	241 (15,4), 280 sh	1627	1519	1347	875	1222	0,47	65
XIV	167	269(13,2), 299 (13,0)	1615	1517	1343	889	1249	0,13	60
XV	156	265 (21,3)	1616	1527	1342	889	1225	0,57	78
XVI	126	254 (13,8)	1623	1522	1349	891	1235	0,36	81
XVII	161	263 (16,3)	1621	1521	1344	888	1229	0,43	87
XVIII	169	278 (15,1)	1622	1522	1349	890	1234	0,32	84
XIX	129	265 sh, 286 (26,3)	1611	1524	1351	883	1224	0,29	61
XX	126	241 (13,8), 336 (10,7)	1627	1495	1357	894	1230	0,40	55

*Melting points of the previously described compounds: I) 77 (71-73 [2], 69-71 [15]); VI) 82 (81-83 [2]); VII) 138 (137-140 [2, 5], 148-149 [15]); VIII) 144 (143-144 [5]); IX) 138 (138 [5]); XI) 158 (158 [5]); XII) 134 (134-135 [5]); XIII) 138 (138 [5]); XXI) 174 [1]; XXII) 156 [1].

Fig. 1. PMR spectrum of compound I ($CDCl_3$, 200 MHz).

The deuterio derivatives of III (7-D-II) were obtained from deuterobenzaldehyde and 4-nitroacetophenone through the corresponding 3-D-chalcone dibromide [8].

The IR and UV spectra confirm the structure of the compounds obtained and follow the regularities discussed previously in [5].

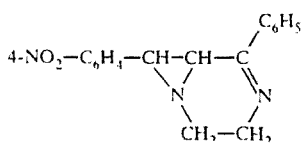
The PMR spectra of compounds I-III, V-IX, XI-XIII, XXI, XXII were measured at a frequency of 200 MHz; full analysis of the multiplets of the bicyclic protons was done using the program Laokoon-3. The data obtained are presented in Table 2 and in Fig. 1.

Comparison of the PMR spectra of compounds II and III shows that upon deuteration, the signal with δ 3.42 ppm disappears while the signal with δ 2.94 ppm becomes an asymmetric singlet. From this it follows that the proton in the 6 position resonates upfield from the 7-H proton. It is interesting to note that in the PMR spectra of the related compounds, aryl-substituted 1,3-diazabicyclo[3.1.0]hex-3-enes [9, 10], the region in which the signals from the aziridine ring protons is found, is the same (although the difference $\Delta\delta = \delta_{7-H} - \delta_{6-H}$ increases to ~ 1 ppm), but their relative location is directly opposite.

TABLE 2. PMR Spectra of 1,4-Diazabicyclo[4.1.0]hept-4-enes

Com- pound	Chemical shift, δ , ppm							Spin-spin coupling constant, J, Hz							K	ψ
	3c	3d	2c	2d	6	7	3c3d	3c3e	3d3e	3c2d	2d2e	3d6	6,7			
I	3.96	3.66	3.24	2.92	3.02	3.44	-16.8	2.1	5.9	4.5	10.5	-12.9	1.4	2.6	48	
II	4.03	3.70	3.29	2.91	2.98	3.48	-16.8	1.8	6.3	4.3	10.2	-13.0	2.4	2.4	47	
III	3.98	3.65	3.24	2.85	2.94	—	-17.4	1.7	6.2	4.7	10.7	-13.4	1.4	—	47	
V	3.97	3.67	3.28	2.88	3.02	4.06	-16.8	1.4	6.1	4.4	10.5	-12.9	1.4	2.5	47	
VI	3.99	3.67	3.25	2.95	3.03	3.54	-16.0	2.0	5.6	4.5	11.2	-13.3	1.5	2.3	50	
VII	3.94	3.67	3.26	3.07	3.02	3.55	-16.9	2.3	5.1	5.9	10.2	-13.5	1.5	2.3	41	
VIII	3.98	3.67	3.27	2.96	2.96	3.52	-17.1	1.8	6.3	4.8	10.7	-11.9	1.5	2.2	47	
IX	3.96	3.64	3.25	2.97	2.95	3.52	-17.0	2.2	4.9	5.6	10.0	-13.3	1.8	2.4	48	
XI	4.07	3.71	3.33	2.95	2.99	3.57	-17.2	1.8	6.7	4.9	10.8	-14.9	1.8	2.3	47	
XII	3.95	3.64	3.25	2.97	3.01	3.55	-16.7	2.4	5.0	6.0	10.0	-13.4	2.3	2.3	47	
XIII	3.94	3.66	3.22	2.98	2.99	3.48	-16.5	2.2	5.9	4.6	10.3	-13.0	2.0	2.0	48	
XXI	4.09	3.72	3.33	2.96	3.00	3.58	-16.5	1.5	6.0	4.4	10.4	-12.7	1.2	1.9	47	
XXII	4.07	3.71	3.33	2.97	3.01	3.58	-16.8	1.4	6.2	4.8	10.6	-13.2	1.9	1.9	47	

TABLE 3. Bond Lengths (d) in Structure VII

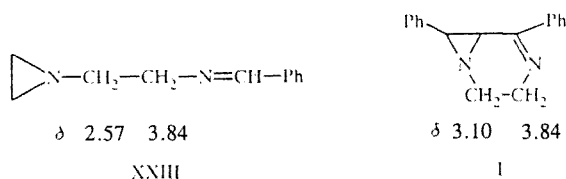


Bond	d, Å	Bond	d, Å
N(1)—C(2)	1,501 (7)	C(12)—C(13)	1,385 (6)
N(1)—C(6)	1,471 (6)	C(8)—C(13)	1,393 (6)
N(1)—C(7)	1,435 (6)	C(5)—C(14)	1,472 (6)
C(2)—C(3)	1,482 (8)	C(14)—C(15)	1,405 (6)
C(3)—N(4)	1,448 (7)	C(15)—C(16)	1,358 (7)
N(4)—C(5)	1,279 (6)	C(16)—C(17)	1,370 (7)
C(5)—C(6)	1,491 (6)	C(17)—C(18)	1,387 (8)
C(6)—C(7)	1,468 (7)	C(18)—C(19)	1,372 (7)
C(7)—C(8)	1,505 (6)	C(14)—C(19)	1,402 (6)
C(8)—C(9)	1,347 (6)	C(11)—N(20)	1,482 (6)
C(9)—C(10)	1,384 (7)	N(20)—O(21)	1,214 (5)
C(10)—C(11)	1,363 (7)	N(20)—O(22)	1,209 (5)
C(11)—C(12)	1,362 (6)		

The electronic effect of the substituents introduced into the aromatic moieties has little effect on the chemical shifts of the aziridine protons within each group of compared compounds (see [3, 9, 10] and Table 2). An exception is the spectrum of compound V, in which with the appearance of an *o*-NO₂ group in the 7-aryl substituent of this molecule, the chemical shift of the 7-H proton increases regularly by 0.6 ppm. Therefore we should probably look for an explanation of the observed "inversion" of the δ values in the conformations of the annelated five- and six-membered heterocycles, which are responsible for the different anisotropic effect of the azomethine group and the aromatic rings on the aziridine protons.

We should also note the small values of the vicinal spin-spin coupling constants (2.0-2.6 Hz) of the protons of the aziridine ring, which suggests their *trans* geometry, and consequently an *exo* orientation of the aryl in the 7 position relative to the six-membered heterocycle.

For analysis of the spectrum of the dimethylene bridge protons, which was considered as an ABCD multiplet system, the following factors are important. In the A and B subspectra, we observe the same constant $J = 17$ Hz, which is assigned to geminal protons. The analogous spin-spin coupling constant in the C and D subspectra has the value 13 Hz. The chemical shifts of the geminal protons on C₍₃₎ should be greater due to the more electronegative character of the C=N group compared with the aziridine ring, which is supported by data for the model compound XXIII [11].



Evidence in favor of such an assignment also comes from the effect of an electron-acceptor substituent in the 5-aryl on the chemical shifts of the methylene protons: the appearance of a nitro- or a sulfo group leads to an increase in the δ values of the protons of the neighboring 3-methylene group (compare compound I with II; VII with XI; XXI with XXII, Table 2).

Introduction of an electron-acceptor substituent also leads to an increase in ${}^2J_{3-H}$, which also can be illustrated in the example of the mentioned compounds. For the entire series of diazabicycloheptenes, we observe the following regularity: ${}^2J_{3-H}/>{}^2J_{2-H}/$, which probably is connected with the effect of the neighboring C=N [Ref. 12, p. 117; ref. 13].

In the spectra of most of the compounds of the diazabicycloheptane series, the signal from the aziridine 6-H proton in addition is split as a result of spin coupling with one of the protons of the CH₂ group in the 3 position, the signal from which undergoes the greatest downfield shift. Deuteration at the 7 position of the bicycle (compound III) is reflected only in the signal from the 6-H proton, which was already noted above, from which it follows that the additional splitting of the signals is due to the homoallylic interaction of the protons with spin-spin coupling constant 5J 1.2-1.8 Hz.

TABLE 4. Bond Angles (β) and Torsional Angles (τ) in Bicyclic Part of Molecule VII

Angle	β	Angle	τ
C(6)N(1)C(2)	113.6(4)	N(1)C(2)C(3),N(4)	-50.9(5)
N(1)C(2)C(3)	114.0(4)	C(2)C(3)N(4)C(5)	40.0(6)
C(2)C(3)N(4)	113.7(4)	C(3)N(4)C(5)C(6)	-5.1(6)
C(3)N(4)C(5)	118.0(4)	N(4)C(5)C(6)N(1)	-19.0(5)
N(4)C(5)C(6)	122.6(4)	C(5)C(6)N(1)C(2)	6.1(5)
C(5)C(6)C(7)	124.6(4)	C(6)N(1)C(2)C(3)	27.0(6)
C(5)C(6)N(1)	118.4(4)	C(6)C(7)C(8)C(9)	-124.8(7)
C(6)C(7)N(1)	60.9(3)		
C(7)N(1)C(2)	117.2(4)		
C(7)N(1)C(6)	60.7(3)		
N(1)C(6)C(7)	58.4(3)		
C(6)C(7)C(8)	120.4(4)		
N(1)C(7)C(8)	116.5(4)		
N(4)C(5)C(14)	119.0(4)		
C(6)C(5)C(14)	118.3(4)		

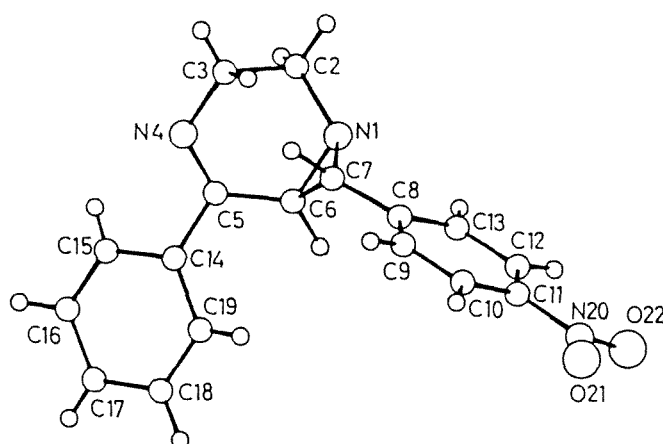


Fig. 2. Projection of a spatial model of molecule VII.

In the A and C subspectra, we observe a small vicinal spin-spin coupling constant (~ 2 Hz), easily assigned to $^3J_{ee}$, while in the B and D subspectra one of the vicinal J constants has a significant magnitude (10.0-10.8 Hz) and reflects the axial-pseudoaxial arrangement (150°) of the interacting protons. The *cis*-vicinal spin-spin coupling constants are approximately equal (5-6 Hz). From this it follows that the A, B, C, and D subspectra belong to 3e-H, 3a-H, 2e-H, and 2a-H respectively.

The set of four vicinal spin-spin coupling constants makes it possible to determine the angle of rotation (ψ) of one CH_2 group relative to the other about the C-C bond (the R-factor method) [13]. The values of the R-factor and ψ are presented in Table 2. As we see from these data, the values of ψ lie within a narrow range (47 - 50°) and consequently the electronic nature of the substituents of the aromatic rings in the 5 and 7 positions has practically no effect on the conformation of the six-membered ring.

Analysis of Dreiding models shows that such values for the angle between the $\text{N}_{(1)}-\text{C}_{(2)}$ and $\text{C}_{(3)}-\text{N}_{(4)}$ bonds can correspond to two forms of the bicycle, *syn* and *anti*. Based on the assignments made above for the protons of the CH_2CH_2 moiety ($\delta_{3e} > \delta_{3a} > \delta_{2e} > \delta_{2a}$), it is impossible to give preference to one of them. The use of the nuclear Overhauser effect (on 3a-, 3e-, and 7-H) also did not give an unambiguous answer. Thus the only available experimental criterion for choosing one of the forms remains the homollylic spin-spin coupling constant between 6-H and 3e-H.

From the stereochemical dependence for constants of this type [14], it follows that the appearance of such an interaction is possible in the case when the torsional angles ϕ_1 and ϕ_2 are not equal to 0° (the maximum value of the constant is observed at 90°). In the considered forms, these angles are: *syn* ϕ_1 and $\phi_2 = 30^\circ$; *anti* ϕ_1 60° , ϕ_2 40° . Obviously in the *anti* form, the protons are oriented in such a way that long-range spin coupling is allowed with higher probability. Therefore the

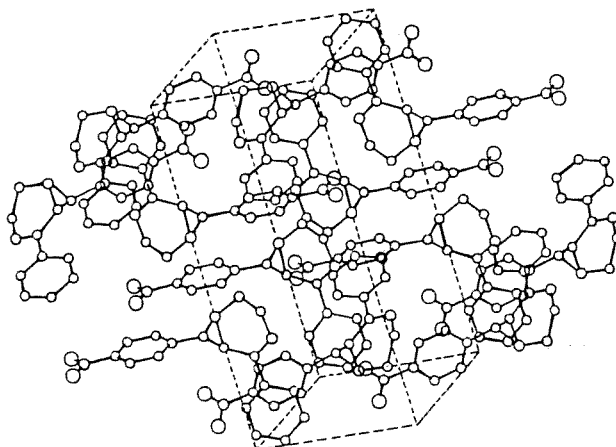


Fig. 3. Packing of molecules in crystals of VII.

relatively high values of the spin-spin coupling constants between 6-H and 3e-H observed in the experiment (1.4-1.8 Hz) allow us to give preference to the *anti* form. It is interesting to note that for 2,3,5,7-tetraphenyl-1,4-diazabicyclo[4.1.0]hept-4-ene [7], the *anti* form also predominates in solutions.

With the goal of independent determination of the spatial structure of diazabicycloheptenes, we carried out an x-ray diffraction investigation of the compounds VII. The values of the bond lengths, bond angles, and torsional angles found are presented in Tables 3 and 4 in Figs. 2 and 3.

The geometric characteristics of the aziridine ring in the studied molecule are somewhat different from those in other aziridine derivatives, in particular the previously discussed diazabicyclohexenes [9, 10]. This difference involves the $C_{(6)}-C_{(7)}$ bond, which is equal to 1.468 Å (for the reference systems, it is 1.502-1.493 Å). We should turn our attention to this fact, since it is specifically the C-C bond of the aziridine ring which is ruptured in photochemical processes.

In the tetrahydropyrazine ring, essentially only the $C_{(6)}-N_{(1)}$ bond angles are deformed as a result of the linkage with the aziridine ring. The $C_{(2)}-C_{(3)}$ bond proved to be somewhat shortened (1.482 Å). The conformation of this ring is close to the half-chair form, and the $C_{(3)}N_{(4)}C_{(5)}C_{(6)}N_{(1)}$ moiety is only slightly twisted along a spiral, and the $C_{(3)}$ atom deviates from the mean plane of the moiety under discussion to the same side as the three-membered ring. In other words, the molecules of the compound VII in the crystal are found in the *syn* half-chair form. From this it follows that the formation of crystals is accompanied by a change in the conformation of the bicycle of the VII molecule. Moreover, the dihedral angle ϕ formed by the $N_{(1)}-C_{(2)}$ and $N_{(4)}-C_{(3)}$ bonds is -50.9° for crystals of compound VII, i.e., it is very close to the angle calculated from the PMR spectral data.

The aromatic ring in the 5 position of the bicycle is conjugated with the π -electrons C=N bond, which is indicated by the shortening of the $C_{(5)}-C_{(Ar)}$ bond. Moreover, the $C_{(3)}N_{(4)}C_{(5)}C_{(6)}$ moiety makes a dihedral angle of -5.1° , which suggests insignificant rehybridization of the $N_{(4)}$ nitrogen atom. The $N_{(1)}$ nitrogen atom has pyramidal coordination.

From x-ray diffraction analysis data for compound VII, we can draw the conclusion that interaction occurs between the aziridine ring and the nitrophenyl radical. Evidence for this comes from the shortening of the bond between these moieties in the VII molecule (1.505 Å). The plane in which we find the aromatic ring at $C_{(7)}$ cuts the angle $C_{(6)}-C_{(7)}-N_{(1)}$, making a 12° angle with the $C_{(7)}-N_{(1)}$ bond. For such an orientation, favorable conditions are achieved for orbital interaction.

Thus the information obtained from PMR and x-ray diffraction data on the one hand is quite internally consistent, and on the other hand confirms the absence of any appreciable strains in the six-membered heterocycle.

EXPERIMENTAL

X-ray Diffraction Investigation. Crystals of 5-phenyl-7-(4-nitrophenyl)-1,4-diazabicyclo[4.1.0]hept-4-ene (VII, $C_{17}H_{15}N_3O_2$) are monoclinic: at $20^\circ C$, $a = 9.057(2)$, $b = 16.698(4)$, $c = 9.952(2)$ Å, $\beta = 102.18(2)^\circ$, $Z = 4$, $d_{calc} = 1.33$ g/cm³, space group $P2_1/n$. The cell parameters and intensities of 1335 independent reflections with $F > 6\sigma(F)$ were measured on the Syntex $P2_1$ four-circle automatic diffractometer ($\lambda_{CuK\alpha}$, graphite monochromator, $\theta/2\theta$ scanning, $2\theta_{max} = 115^\circ$).

TABLE 5. Coordinates of Nonhydrogen Atoms ($\times 10^4$) and Hydrogen Atoms ($\times 10^3$) in Molecule VII

Atom	x	y	z	Atom	x	y	z
N(1)	5387(5)	1004(2)	4239(4)	C(15)	8609(5)	3441(3)	4385(5)
N(4)	7531(4)	2145(3)	5783(4)	C(16)	9022(6)	4106(3)	3799(5)
C(2)	6534(6)	774(3)	5501(6)	C(17)	8037(6)	4452(3)	2719(5)
C(3)	7045(6)	1449(3)	6451(5)	C(18)	6638(6)	4114(3)	2212(5)
C(5)	6769(5)	2340(3)	4596(4)	C(19)	6241(5)	3426(3)	2794(5)
C(6)	5492(5)	1847(3)	3832(5)	N(20)	-1636(4)	1207(2)	942(4)
C(7)	4228(5)	1556(3)	4412(5)	O(21)	-1766(4)	825(2)	-115(3)
C(8)	2693(5)	1433(3)	3503(4)	O(22)	-2677(4)	1538(2)	1299(4)
C(9)	1493(5)	1803(3)	3823(5)	H(6)	531(6)	189(4)	260(6)
C(10)	60(5)	1726(3)	3008(5)	H(7)	457(2)	173(1)	548(2)
C(11)	-115(5)	1269(3)	1850(4)	H(2.1)	609(5)	30(3)	600(5)
C(12)	1062(5)	876(3)	1493(4)	H(2.2)	765(6)	64(3)	516(5)
C(13)	2483(5)	959(3)	2330(4)	H(3.1)	774(5)	129(3)	717(4)
C(14)	7196(5)	3065(3)	3928(4)	H(3.2)	593(4)	148(2)	671(4)

The structure was deciphered by the direct method using the program MULTAN. The positions of the hydrogen atoms were determined from an electron density difference synthesis. Refinement of the structure in the anisotropic approximation (isotropic for hydrogen atoms) led to $R = 7.3\%$, $R_w = 8.0\%$. The coordinates of the atoms are presented in Table 5.

The IR spectra of compounds II-V, XIV-XX were measured in KBr pellets on the Specord IR-75 spectrometer; the UV spectra were measured on the Specord M-40 in methanol [$c = (2-4) \cdot 10^{-5}$ moles/liter]. The PMR spectra of compounds I-III, V-IX, XI-XIII, XXI, XXII were taken on the Bruker WH-200 (200 MHz) in $CDCl_3$, internal standard HMDS. The course of the reactions and the purity of the compounds formed were monitored using TLC on Silufol UV-254 plates, chloroform as the eluent.

The nitrogen content in the compounds obtained corresponds to the calculated value.

5-(4-Nitrophenyl)-7-phenyl-1,4-diazabicyclo[4.1.0]hept-4-ene (II). 0.5 ml ethylenediamine (dried and distilled over KOH) and 1.0 ml triethylamine were added to a suspension of 1.03 g (2.5 moles) 1-(4-nitrophenyl)-3-phenyl-2,3-dibromopropan-1-one in 50 ml methanol. The mixture obtained was heated until the product II began to precipitate. Then the reaction mixture was held in the dark at $10^\circ C$ for two days. The product was filtered and washed on the filter with copious amounts of 50% aqueous methanol. By evaporation of the filtrate, an additional amount of more massive product was isolated. Overall yield of II, 0.44 g (60%). mp $124^\circ C$ [$CHCl_3-CH_3OH$, 1:3].

The compounds III, IV, V, XIV, XV-XX, not described previously, were synthesized analogously.

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