HETEROADAMANTANES AND THEIR DERIVATIVES.

6.* SYNTHESIS AND MASS-SPECTROMETRIC INVESTIGATION OF 5-MONO- AND 5,6-DISUBTITUTED 6-OXO-1,3-DIAZAADAMANTANES

A. I. Kuznetsov, E. B. Basargin,
A. S. Moskovkin, M. Kh. Ba, I. V. Miroshnichenko,
M. Ya. Botnikov, and B. V. Unkovskii

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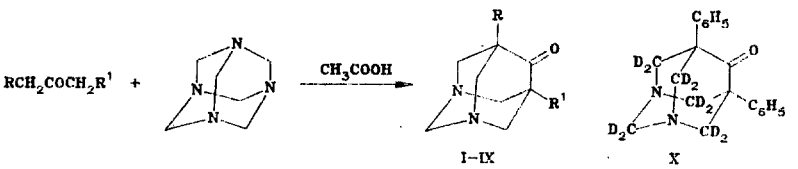
The corresponding 5-mono- and 5,7-disubstituted 6-oxo-1,3-diazaadamantanes were obtained with high yields by the condensation of mono- and α,α'-disubstituted acetones with hexamethylenetetramine in the presence of glacial acetic acid, and their structures were confirmed by IR and PMR spectra. The behavior of the compounds under electron impact was studied, and the main fragmentation paths of their molecules were determined.

Various 5,7-disubstituted 6-oxo-1,3-diazaadamantanes are obtained by the aminomethylation of the corresponding α,α'-disubstituted acetones with a mixture of paraform and ammonium acetate [2, 3]. Until recently only ketones containing substituents which activate the α-methylene protons such as aryl [4, 5], arylthio [6-8], or alkoxycarbonyl groups [8-10], had been used in this reaction. The use of dialkyl ketones leads to a marked reduction in the yield of the desired adamantanones, which in the case of diethyl ketone amounts to less than 20% [11]. Attempts to use methyl ethyl ketone and acetone in the reaction were unsuccessful [11].

For the production of substituted 6-oxo-1,3-diazaadamantanes we proposed a modification of the Mannich reaction, in which a mixture of hexamethylenetetramine and glacial acetic acid in an anhydrous lower alcohol was used instead of a mixture of paraform and ammonium acetate as aminomethylating agent. This made it possible not only to achieve high yields of the desired adamantanones but also, in addition to our previously described [1] 5-methyl- and 5-phenyl-6-oxo-1,3-diazaadamantanes, to synthesize a series of difficultly obtainable or undescribed 5,7-dialkyl-6-oxo-1,3-diazaadamantanes from dialkyl ketones not containing actived α-methylene groups.



I R = CH_3, R^1 = H; II R = R^1 = CH_3; III R = CH_3; R^1 = n-C_3H_7; IV R = R^1 = C_2H_5; V R = R^1 = n-C_3H_7; VI R = R^1 = iso-C_3H_7; VII R = R^1 = n-C_5H_{11}; VIII R = C_6H_5, R^1 = H; IX R = R^1 = C_6H_5

Thus, in the case of 5,7-dimethyl-6-oxo-1,3-diazaadamantane (II), obtained by the method in [11] with a yield of 19.5%, the use of the proposed modification made it possible to increase its yield to 73.6%. In the case of 5,7-diphenyl-6-oxo-1,3-diazadamantane (IX), synthesized from dibenzyl ketone activated by phenyl groups, it increased the yield from 59.0% by the method in [4] to 90.4%.

The structure of the synthesized compounds was confirmed by data from the IR, PMR, and mass spectra. The characteristics of the compounds are given in Tables 1 and 2.

*For Communication 5, see [1].

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TABLE 1. 5,7-Disubstituted 6-Oxo-1,3-diazaadamantanes (III-VII, X)

Compound	mp, °C	IR spectrum ν_{CO}, cm^{-1}	PMR spectrum, ppm (in CCl_4)*		Found, %			Molecular formula	Calculated, %			Yield, %
			N–CH_2–N	R, R¹	C	H	N		C	H	N	
III	66–67	1710	2,98 dd (2,88 4H$_\alpha$, 3,08 4H$_e$, J = 13 Hz)	1,16, m (4H, CH_2 in R¹); 0,85, m (3H, CH_3inR¹); 0,74 s (3H, CH_3inR)	68,9	9,7	13,5	$C_{12}H_{20}N_2O$	69,2	9,7	13,4	68,4
IV	46–47	1710	2,98 dd (2,88 4H$_\alpha$, 3,08 4H$_e$, J = 13Hz)	1,29 q (4H, J = 7Hz, CH_2inR, R¹); 0,79 t (6H, J = 7Hz, CH_3inR, R¹)	69,4	9,7	13,4	$C_{12}H_{20}N_2O$	69,2	9,7	13,4	72,7
V	83–84	1710	2,98 dd (2,88 4H$_\alpha$, 3,08 4H$_e$, J = 13Hz)	1,16m (8H, CH_2inR, R¹); 0,85m (6H, CH_3inR, R¹)	71,0	10,3	11,8	$C_{14}H_{24}N_2O$	71,1	10,2	11,8	72,3
VI	116–117†	1710	3,11 dd (2,94 4H$_\alpha$, 3,28 4H$_e$, J = 13Hz)	1,93m (2H, J = 7 Hz, CHinR, R¹); 0,87 d (12H, J = 7Hz, CH_3inR, R¹)	70,8	10,2	11,6	$C_{14}H_{24}N_2O$	71,1	10,2	11,8	75,5
VII	39–40	1710	2,98 dd (2,88 4H$_\alpha$, 3,08 4H$_e$, J = 13Hz)	1,17m (16H, CH_2inR, R¹); 0,84m (6H, CH_3inR, R¹)	74,2	11,1	9,5	$C_{18}H_{32}N_2O$	73,9	11,0	9,6	70,1
X	255–256	1710, 1595 (C_6H_5)	—	—	72,6	—	8,6	$C_{20}H_{10}D_{10}N_2O$	72,7	—	8,5	61,8

*For compounds (III-VII), obtained for N–CH_2–N: 2H, 3.88, s; there are not data for X.
†bp at 0.25 mm Hg.

TABLE 2. The Mass Spectra of Compounds (I-X)*

Compound	m/z values (relative intensity, %)
I	166 (73), 124 (100), 96 (11), 84, (11), 70 (12), 69 (31), 68 (19), 55 (29), 43 (13), 42 (47), 41 (48)
II	180 (84), 138 (100), 137 (19), 110 (10), 84 (11), 82 (15), 69 (35), 55 (18), 43 (11), 42 (50), 41 (41)
III	208 (60), 179 (19), 166 (100), 165 (22), 136 (18), 82 (15), 69 (22), 58 (25), 55 (18), 42 (55), 41 (38)
IV	208 (88), 179 (15), 166 (100), 165 (27), 150 (33), 83 (24), 69 (13), 58 (19), 55 (41), 42 (45), 41 (28)
V	236 (67), 207 (33), 194 (98), 193 (43), 164 (46), 69 (33), 58 (68), 55 (38), 43 (27), 42 (100), 41 (52)
VI	236 (100), 221 (32), 194 (88), 193 (96), 165 (34), 164 (79), 69 (45), 58 (53), 55 (33), 42 (77), 41 (75)
VII	292 (55), 250 (100), 236 (22), 235 (36), 221 (26), 192 (37), 69 (22), 5 (44), 55 (51), 42 (58), 41 (39)
VIII	228 (74), 186 (100), 103 (41), 91 (16), 77 (19), 70 (13), 68 (22), 58 (23), 43 (13), 42 (62), 41 (18)
IX	304 (40), 262 (63), 261 (90), 233 (19), 144 (22), 131 (47), 103 (100), 91 (56), 77 (38), 42 (39), 41 (24)
X	314 (74), 266 (47), 265 (15), 240 (19), 238 (27), 163 (18), 133 (41), 123 (15), 105 (100), 78 (15), 46 (57)

*The M^+ peaks and the 10 strongest ion peaks in the mass spectra apart from the isotopic peaks are given.

TABLE 3. The Intensity of the Peaks for the Characteristic Fragment Ions in the Mass Spectra of Compounds (I-X) as Percentages of the Total Ion Current

Compound	Indices of ions																
	W_M	$[M-(R-CH_2)]^+$	Φ_1	Φ_2	Φ_3	Φ_4	Φ_5	Φ_6	Φ_7	Φ_8	Φ_9	Φ_{10}	Φ_{11}	Φ_{12}	Φ_{13}	Φ_{14}	Φ_{15}	$[C_4H_7]^+$
I	14,5	0,4	0,2	—	8,4	17,9	1,9	1,4	1,0	4,9*	4,0*	3,6	8,7	—	0,8	0,4	0,7	5,2
II	16,9	0,6	0,7	1,2	8,8	17,7	1,8	3,3	1,1	2,7	2,0	4,2	7,3	2,1	1,0	0,3	1,0	3,2
III	10,7	2,8	0,6	2,7	8,4	15,4	1,6	3,4	0,6	2,6	1,9*	3,6*	7,5*	—	1,0	1,6	3,8	2,8
IV	14,4	1,3	2,3	5,1†	7,2	15,8	—	4,3	0,9	1,3	1,4	2,6	6,4	1,3	1,8	5,1**	3,0	5,4
V	7,3	3,1	1,3	4,3	6,1	9,2	1,7	2,6	0,5	1,1	0,8	1,3	3,1	0,7	0,9	1,7	6,4	3,6
VI	10,6	—	3,5	7,0	6,8	7,8	—	5,2	0,6	1,1	0,6	2,4	3,2	—	1,0	1,9	4,2	2,5
VII	6,1	1,1	2,3	3,4	5,3	9,0	0,8	—	0,5	0,5	0,4	0,3	0,3	—	2,2	—	4,0	4,6
VIII	12,2	—	—	—	8,7	13,9	0,6	0,6	0,6	3,8*	2,2*	0,8	5,7	0,4	0,6	0,3	3,3	1,6
IX	4,0	—	0,1	—	4,6	5,3	1,0	7,5	0,5	1,8	1,1	1,7	8,4	0,9	1,1	0,1	0,8	1,0
X	10,7	—	—	—	6,6	1,5	2,1	5,4	0,5	1,5	1,0	3,1	11,5	1,6	1,4	—	0,2	0,2

*The overall intensity of the peaks for ions with substituents R and R^1.
†The $[C_8H_{10}NO]^+$.

The IR spectra of the adamantanones (III-VII, X) contain characteristic absorption bands for the stretching vibrations of the carbonyl in the ketone group at 1710 cm^{-1}. In the spectrum of (X) there is also an absorption band for the aromatic ring at 1595 cm^{-1}.

The PMR spectra of the synthesized compounds (III-VI) contain signals for the resonance absorption of eight methylene protons of the N–CH_2 fragments characteristic of 6-oxo-1,3-diazaadamantane structures; they form an AB system, which appears at 2.98 ppm (δ_{Ha} 2.88, δ_{He} 3.08 ppm, J_{ae} = 13 Hz) in the case of (III-V, VII) and at 3.11 ppm (δ_{Ha} 2.94, δ_{He} 3.28 ppm, J_{ae} = 13 Hz) in the case of (VI). There is a singlet for the two methylene protons of the animal fragment N–CH_2–N at 3.88 ppm. The signals for the protons of the alkyl substituents at the $C_{(5)}$ and $C_{(7)}$ atoms appear in the spectra in the form of groups of signals of appropriate multiplicity in the region of 0.68-1.93 ppm.

In addition to the dialkyladamantanones (III-VII), as model compound for the mass-spectrometric investigation we synthesized 2,4,8,9,10-decadeutero-5,7-diphenyl-6-oxo-1,3-diazaadamantane (X) by a method similar to that described in [4]. The mass spectra of the monoalkyl and monoaryl [12-15] and dialkyl derivatives of adamantane [16] with substituents at the bridgehead positions and also of 2-oxoadamantane and its derivatives [17, 18] have been described in the literature. Earlier [19] we showed that the main relationships governing the dissociation of 1-substituted adamantanes under electron impact, determined by the nature of the functional group, also remain for 7-substituted 1,3,5-triazaadamantanes. In this connection it was interesting to undertake a mass-spectrometric investigation of the 5-mono- and 5,7-disubstituted 6-oxo-1,3-diazaadamantanes (I-IX), especially as data on this aspect are practically absent in the literature except for [20, 21], where mass spectrometry was used to identify 6-oxo-1,3-diazaadamantane [20] and its 5,7-diphenyl and 5,7-dimethoxycarbonyl derivatives [21].

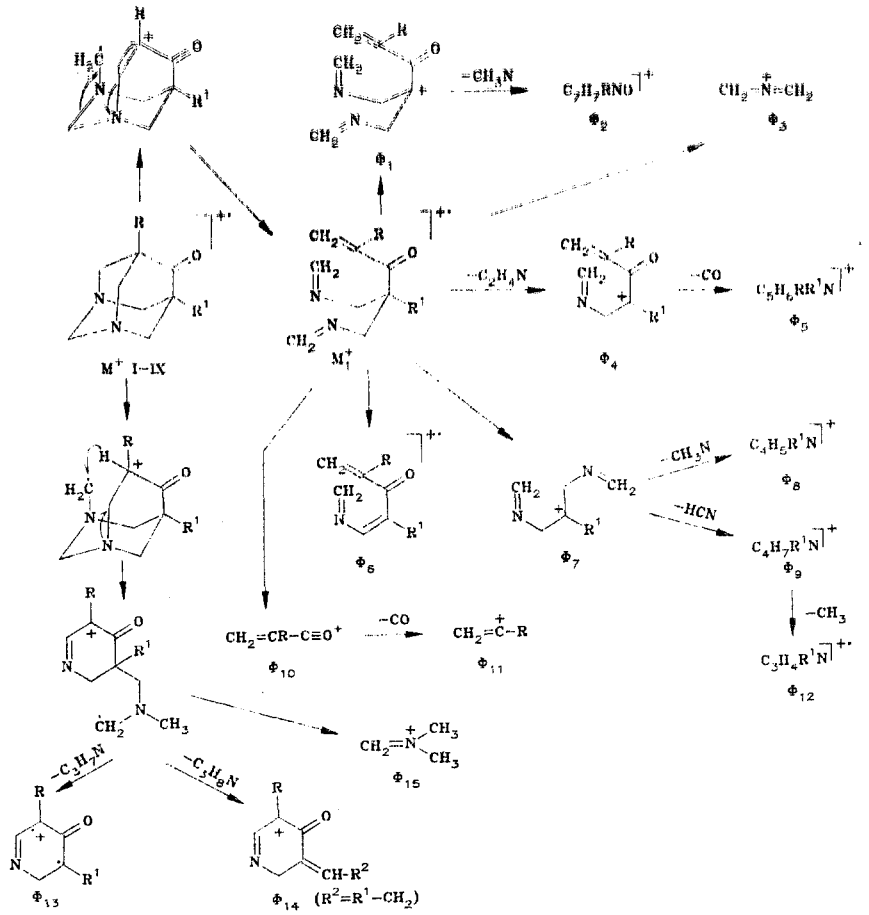
The mass spectra of compounds (I-IX) (Table 2) contain strong peaks for the M^+ ions. The stability of the molecular ions to dissociation W_M (Table 3) decreases with increase in the length of the hydrocarbon chain of the alkyl substituent. The largest W_M value is given by the M^+ ion of (II), which contains two methyl substituents at the bridgehead positions of the 1,3-diazaadamantane ring. Its analog (IX) with two phenyl substituents has a significantly smaller W_M value, and this is evidently due to the higher probability of dissociation of the adamantane ring with the formation of phenyl-containing ions.

Analysis of the mass spectra of (I-IX) (Table 3) shows that in contrast to 1-alkyl- and 1-aryladamantanes, whose mass spectra are determined to a significant degree by the nature of the substituent in the adamantane ring [12-15], the behavior of 6-oxo-1,3-diazaadamantanes under electron impact obeys the general relationships and does not depend on the structure of the substituting group.

The mass-spectral dissociation of compounds (I-IX) is accompanied primarily by destruction of the 1,3-diazaadamantane skeleton and the formation of the characteristic ions $[C_2H_4N]^+(\Phi_3)$, $[M-C_2H_4N]^+$ (Φ_4), $[M-C_2H_5N]^+$ (Φ_6), and $[C_3H_2RO]^+(\Phi_{10})$(see the scheme). The formation of these ions probably takes place from the open form of the M_1^+ ion, proposed for the dissociation of 1-aryladamantanes [15], with cleavage of the bonds around the quaternary carbon atom.

While studying the mass spectra of methyladamantanones, Mitera and coworkers [22] suggested that the elimination of the methyl group from the M^+ ion is prevented by the localization of charge at the double bond of the carbonyl group. This suggestion evidently explains the lower intensity of the peaks for the Φ_1 ions, due to the loss of an alkyl substituent by the M^+ ion, in the spectra of compounds (I-VII) compared with the intensity of the peaks for the analogous ions in the spectra of 1-alkyladamantanes [12-14, 16]. The removal of the phenyl substitutent from the M^+ ion of compounds (VIII-X), as for 1-aryladamantanes [15], is uncharacteristic, and the weak peak for the Φ_1 ion is only observed in the spectra of the diphenyl-substituted compounds (IX, X).

The most characteristic for our investigated compounds is the formation of Φ_4 ions, the peaks of which have maximum intensity in the spectra of compounds (I-V, VII, VIII) and are second in value in the spectrum of (VI). For compound (IX), which has two phenyl substituents, the formation of the Φ_6 ion is more likely. The dissociation of the Φ_1, Φ_4, Φ_7 and Φ_{10} ion takes place with the elimination of the neutral molecules CH_2=NH, HCN and CO, as shown in the scheme. By means of the high-resolution mass spectra it was established that the peaks for the ions with m/z 69 (II), 83 (III, IV) and 131 (VIII, IX) are composite peaks and correspond to the two ions Φ_{10} and Φ_{12} in a ratio of 2:1.



A different dissociation path for the M^+ ion, leading to the appearance of peaks for the $[M-C_3H_7N]^+$ (Φ_{13}), $[M-C_3H_8N]^+$ (Φ_{14}), and $[C_3H_8N]^+$ (Φ_{15}) ions in the spectra, is also possible for compounds (I-IX). In the spectra of compounds (I, II, VII-IX) the peak for the Φ_{13} ion, which is the analog of the $[M-C_4H_9]^+$ ion in the spectra of 1-aryladamantanes, has higher intensity than the peak for the Φ_{14} ion. Increase in the number of carbon atoms in the alkyl substituent [compounds (III-VI)] leads to the result that the intensity ratio of the peaks for these ions is reversed and the intensity of the peak for the Φ_{15} ion increases at the same time. This fact indicates that a hydrogen atom from the alkyl group mainly migrates to the radical center during the formation of the Φ_{15} ion.

Apart from those indicated in the scheme, the spectra of the investigated compounds also contain the peaks of $[M-(R-CH_2)]^+$ ions, due to loss of part of the alkyl substituent by the M^+ ion, and $[C_4H_7]+$ ions formed at later stages in the dissociation of the molecular ion. The elimination of a molecule of CO from the M^+ ion, as supposed in [17] during investigation of the mass spectrum of 2-oxoadamantane, does not take place in compounds (I-IX), and the low-intensity peak for the $[M-28]^+$ ion, observed in the spectra of compounds (I, II, VIII), corresponds to loss the CH_2N radical by the M^+ ion. A characteristic difference in the mass spectra of compounds (V, VI) is the absence of the peak for the ion with m/z 207 ($[M-C_2H_5]+$) in the latter and the presence of strong peaks for ions with m/z 193 ($\Phi_1 + \Phi_6$) and 221 ($[M-CH_3]^+$).

The composition of the base ions of compounds (I-IX) was confirmed by high-resolution mass spectra and also by the spectrum of deuterated compound (X). Thus, the following facts were established during mass-spectrometric investigation of compounds (I-IX). The mass spectra of the derivatives of 6-oxo-1,3-diazaadamantane are characterized by strong peaks for the molecular ions and by peaks for the Φ_3 and Φ_4 fragments; in addition to the above-mentioned ions, the diphenyl derivative (IX) is characterized by the presence of a strong peak for the Φ_{11} ion. Substitution of the alkyl substituents by phenyl does not introduce significant differences in the nature of the dissociation of the molecular ion. This makes

it possible to suppose that the positive charge in the molecular ion of 6-oxo-1,3-diazaadamantanes is localized preferentially at the double bond of the carbonyl group. The discovered characteristics of mass-spectral dissociation in the 6-oxo-1,3-diazaadamantane derivatives can be used for identification and structure determination in compounds of similar series.

EXPERIMENTAL

The IR spectra were obtained on a Specord 71-IR spectrometer in Vaseline oil. The PMR spectra were obtained in carbon tetrachloride on a Tesla-80 instrument with HMDS as internal standard on the δ scale. The mass spectra were obtained on an LKB-2091 chromato-mass spectrometer with direct injection into the ion source at 3.5 kV, a cathode emission current of 25 μA, 70-eV ionizing electrons, and an ionization chamber temperature of 200°C. The high-resolution mass spectra were obtained on a Kratos MS-80 instrument with direct injection at 3.0 kV, 100 μA, 70 eV, and 150°C with perfluorinated kerosene as standard. The resolution was $M/\Delta M = 7500$.

The characteristics of the adamantanones (III-VII, X) are given in Tables 1 and 2.

5,7-Dimethyl-6-oxo-1,3-diazaadamantane (II). A mixture of 1.46 g (17 mmole) of diethyl ketone, 2.45 g (17 mmole) of hexamethylenetetramine, and 2.40 g (40 mmole) of glacial acetic acid in 10 ml of n-butanol was heated with gentle boiling for 30 min. The red-brown reaction mass was concentrated to a fifth of the volume under vacuum. The viscous residue was extracted with hot n-heptane (4 × 20 ml), the hot extract was purified by passing through a layer of anhydrous aluminum oxide (4 g, II activity) placed on a Schott filter, the extractant was distilled under vacuum, and the solid residue was recrystallized fron n-heptane. We obtained 1.99 g (73.6%) of the adamantanone (II); mp 130-131°C. Published data [11]: mp 131-132°C.

5-Methyl-7-propyl-6-oxo-1,3-diazaadamantane (III). The compound was obtained similarly to (II) from 1.94 (17 mmole) of butyl ethyl ketone, 2.45 g (17 mmole) of hexamethylenetetramine, and 2.40 g (40 mmole) of glacial acetic acid in 10 ml of n-butanol. After recrystallization from n-heptane we obtained 2.14 g of the adamantanone (III).

5,7-Diethyl-6-oxo-1,3-diazaadamantane (IV). The compound was obtained similarly to (II) from 1.94 g (17 mmole) of di-n-propyl ketone, 2.45 g (17 mmole) of hexamethylenetetramine, and 2.40 g (40 mmole) of glacial acetic acid in 10 ml of n-butanol. After recrystallization from n-heptane we obtained 2.27 g of the adamantanone (IV).

5,7-Di-n-propyl-6-oxo-1,3-diazaadamantane (V). The compound was obtained similarly to (II) from 2.41 (17 mmole) of di-n-butyl ketone, 2.45 g (17 mmole) of hexamethylenetetramine, and 2.40 g (40 mmole) of glacial acetic acid in 10 ml of n-butanol. After recrystallization from n-heptane we obtained 2.56 g of the adamantanone (V).

5,7-Diisopropyl-6-oxo-1,3-diazaadamantane (VI). The compound was obtained similarly to (II) from 2.41 g (17 mmole) of diisobutyl ketone, 2.45 g (17 mmole) of hexamethylenetetramine, and 2.40 g (40 mmole) of glacial acetic acid in 10 ml of n-butanol. After vacuum distillation at 0.25 mm Hg we obtained 2.68 g of the adamantanone (VI).

5,7-Dipentyl-6-oxo-1,3-diazaadamantane (VII). The compound was obtained similarly to (II) from 3.37 g (17 mmole) of di-n-hexyl ketone, 2.45 g (17 mmole) of hexamethylenetetramine, and 2.40 g (40 mmole) of glacial acetic acid in 10 ml of n-butanol. After recrystallization from n-heptane we obtained 3.08 g of the adamantanone (VII).

5,7-Diphenyl-6-oxo-1,3-diazaadamantane (IX). A mixture of 6.00 g (29 mmole) of dibenzyl ketone, 4.01 g (29 mmole) of hexamethylenetetramine, and 2.98 g (52 mmole) of glacial acetic acid was heated in 20 ml of n-butanol with gentle boiling. After 10 min an abundant crystalline precipitate separated. After cooling the crystals were filtered off, washed with 20 ml of water and 15 ml of n-butanol, and dried in a desiccator over calcium chloride. After recrystallization from toluene we obtained 5.95 g (90.4%) of the adamantanone (IX); mp 254-255°C. Published data [4]: mp 257°C.

2,4,8,9,10-Decadeutero-5,7-diphenyl-6-oxo-1,3-diazaadamantane (X). A mixture of 2.10 g (10 mmole) of dibenzyl ketone, 2.0 ml of 25% aqueous ammonia solution, 1.60 g (50 mmole) of deuteroparaform, and 1.12 g (19 mmole) of glacial acetic acid in 7 ml of n-butanol was heated with gentle boiling for 1 h. The precipitate which separated from the reaction mass

on cooling was separated by filtration, washed with 5 ml of water and 5 ml of n-butanol, and dried in a desiccator over calcium chloride. After recrystallization from toluene we obtained 2.04 g of the adamantanone (X).

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