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Dedicated to Full Member of the Russian Academy of Sciences I. P. Beletskaya on occasion of her jubilee

VI.* Synthesis of Heterocyclic Analogs of γ-Aminobutyric Acid from 3,5-Dinitrobenzoic Acid

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Abstract—A series of 7-carboxy-3-R-1,5-dinitro-3-azabicyclo[3.3.1]non-6-enes was synthesized by reduction of 3,5-dinitrobenzoic acid with sodium borohydride followed by Mannich reaction with formaldehyde and primary amines. The mechanism of decomposition under electron impact of the 3-azabicyclo[3.3.1]nonane was established. Enthalpies of formation of compounds synthesized were calculated by semiempirical PM3 method.

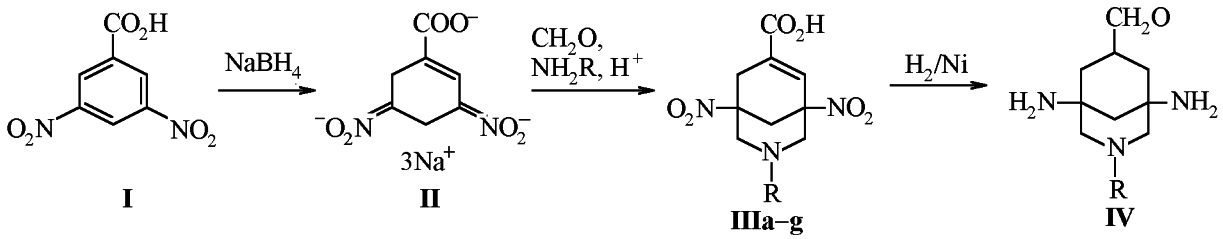
γ-Aminobutyric acid and its derivatives play a significant part in metabolism of brain [2]. Analogs of the acid containing a 3-azabicyclo[3.3.1]nonane fragment can be synthesized from 3,5-dinitrobenzoic acid (**I**) along Scheme 1.

In preceding communications we reported on the synthesis of compounds **IIIf, g** [3, 4], and also on development of catalytic hydrogenation procedure for 3-R-1,5-dinitro-3-azabicyclo[3.3.1]non-6-enes into the respective 1,5-diamino-3-azabicyclo[3.3.1]-nonanes [5]. Therefore bicyclic nitrocarboxylic acids **III** may be regarded as intermediate products in preparation of heterocyclic analogs of γ-aminobutyric acid of type **IV**.

The present study concerns the synthesis of nitrocarboxylic acids **IIIa–e** and investigation of physicochemical characteristics of this class compounds. In keeping with the above scheme we carried out selective reduction of the aromatic ring of acid **I** with sodium borohydride in 50% ethanol using the reagent in a 4-fold excess. The salt of 3,5-bis(*aci*-nitro)-1-cyclohexene-1-carboxylic acid (**II**) formed in the first stage was subjected to aminomethylation without isolation from the reaction mixture. The target products were obtained by adding 3-fold excess of formaldehyde and 2-fold excess of primary amine. As a result we obtained 7-carboxy-3-R-1,5-dinitro-3-azabicyclo[3.3.1]non-6-enes (**IIIa–e**) whose composition and structure were proved by elemental analysis (Table 1) and IR, ^{1}H and ^{13}C NMR spectroscopy (Tables 1, 2).

In the IR spectra of compounds **III** obtained (Table 1) appear strong bands of nitro groups stretch-

Scheme 1.



I **II** **IIIa–g** **IV**

R = CH_3 (**a**), C_2H_5 (**b**), $(CH_2)_2CO_2H$ (**c**), C_4H_9 (**d**), $(CH_2)_2Br$ (**e**), CH_2CO_2H (**f**), $(CH_2)_2OH$ (**g**).

* For communication V see [1].

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Table 1. Yields, melting points, retention factors, elemental analyses, and IR spectra of 7-carboxy-3-R-1,5-dinitro-3-azabicyclo[3.3.1]non-6-enes (**IIIa**–**e**) and 1,*n*-bis(7-carboxy-1,5-dinitro-3-azabicyclo[3.3.1]non-6-en-3-yl)alkanes (**Va, b**)

Compd. no.	Yield %	mp, °C[a]	R_f	IR spectra, ν, cm^{-1} [b]				Found, %			Formula	Calculated, %		
				OH	C=O	C=C	NO_2	C	H	N		C	H	N
IIIa	35	179–180	0.67	2635–3216	1689	1662	1513, 1365, 1325	43.97, 44.23	4.58, 4.79	17.20	$C_{10}H_{13}N_3O_6$	44.28	4.80	15.50
IIIb	45	169–170	0.69	2540–3100	1693	1642	1522, 1345	45.83, 46.08	4.92, 5.15	14.64, 14.41	$C_{11}H_{15}N_3O_6$	46.32	5.26	14.76
IIIc	38	196–197	0.42	2885–3171	1685	1643	1542, 1342	41.62	4.39	12.64	$C_{12}H_{15}N_3O_8$	43.76	4.56	12.77
IIId	19	131–132	0.80	2540–3080	1693	1664	1564, 1385, 1357	52.72, 52.96	6.17, 6.10	13.50, 13.54	$C_{13}H_{19}N_3O_6$	49.84	6.07	13.42
IIIe[c,d]	52	194–195 (decomp.)	0.67		1685	1657	1528, 1340	36.63, 36.74	4.19, 3.54	11.50, 11.52	$C_{11}H_{14}N_3O_6Br$	36.26	3.85	11.54
Va[d]	43	201–202 (decomp.)	0.31		1686	1643	1529, 1371, 1343	44.45, 44.72	4.85, 4.88	15.23, 15.16	$C_{20}H_{24}N_6O_{12}$	44.44	4.44	15.56
Vb[d]	37	192–193 (decomp.)	0.37		1671	1645	1514, 1341	47.53, 47.61	5.38, 5.47	15.24, 15.39	$C_{22}H_{28}N_6O_{12}$	47.47	4.91	14.78

[a] Melting points measured on derivatograph.

[b] δ(CH) 1390–1475 cm^{-1}.

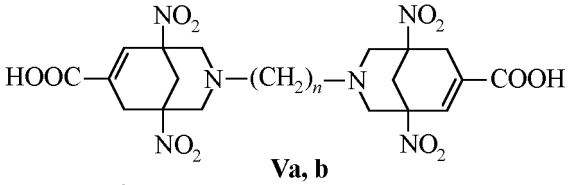
[c] Found, %: Br 21.87, 21.86. Calculated, %: Br 21.39.

[d] IR spectrum from mull in mineral oil.

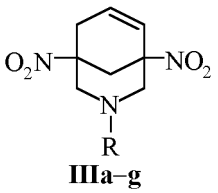
ing vibrations in the range 1515–1570 and 1325–1385 cm^{-1}, broad bands in the region 2400–3400 cm^{-1}, and absorption bands of COOH group vibrations at 1685–1715 cm^{-1}.

In the ^{1}H NMR spectra of the 3-azabicyclo[3.3.1]-nonane derivatives (Table 2) the most downfield broadened singlet (δ ~13 ppm) belongs to the proton of carboxy group. The H^6 proton attached to the double bond appears as a singlet at δ 6.95–7.15 ppm. Axial and equatorial protons H^2, H^4, and H^9 of the piperidine ring are observed as a group of six doublets in the region δ 2.55–3.30 ppm with geminal coupling constants ^2J 10.4–11.8 Hz. Doublets of H^8 protons from the cyclohexene moiety appear at δ 2.25–3.00 ppm, and they possess larger coupling constants (^2J 18.4–19.0 Hz). The protons of substituents attached to nitrogen atom give the expected pattern of signals. Further confirmation of the structure of compounds prepared was obtained from their ^{13}C NMR spectra (Table 2) which conform to the assumed structures. The carbon atoms of the carboxy groups appear as signals in the region δ_C 168–175 ppm, and those of the C=C bond at δ_C 135–136 ppm. The signals of carbon atoms directly bonded to electron-withdrawing nitro groups (C^1, C^5) or to a heteroatom (C^2, C^4, C^{10}) are shifted downfield and are observed at δ_C 87–90 and 52–64 respectively.

The use in the Mannich reaction of aliphatic diamines provided 1,*n*-bis(7-carboxy-1,5-dinitro-3-



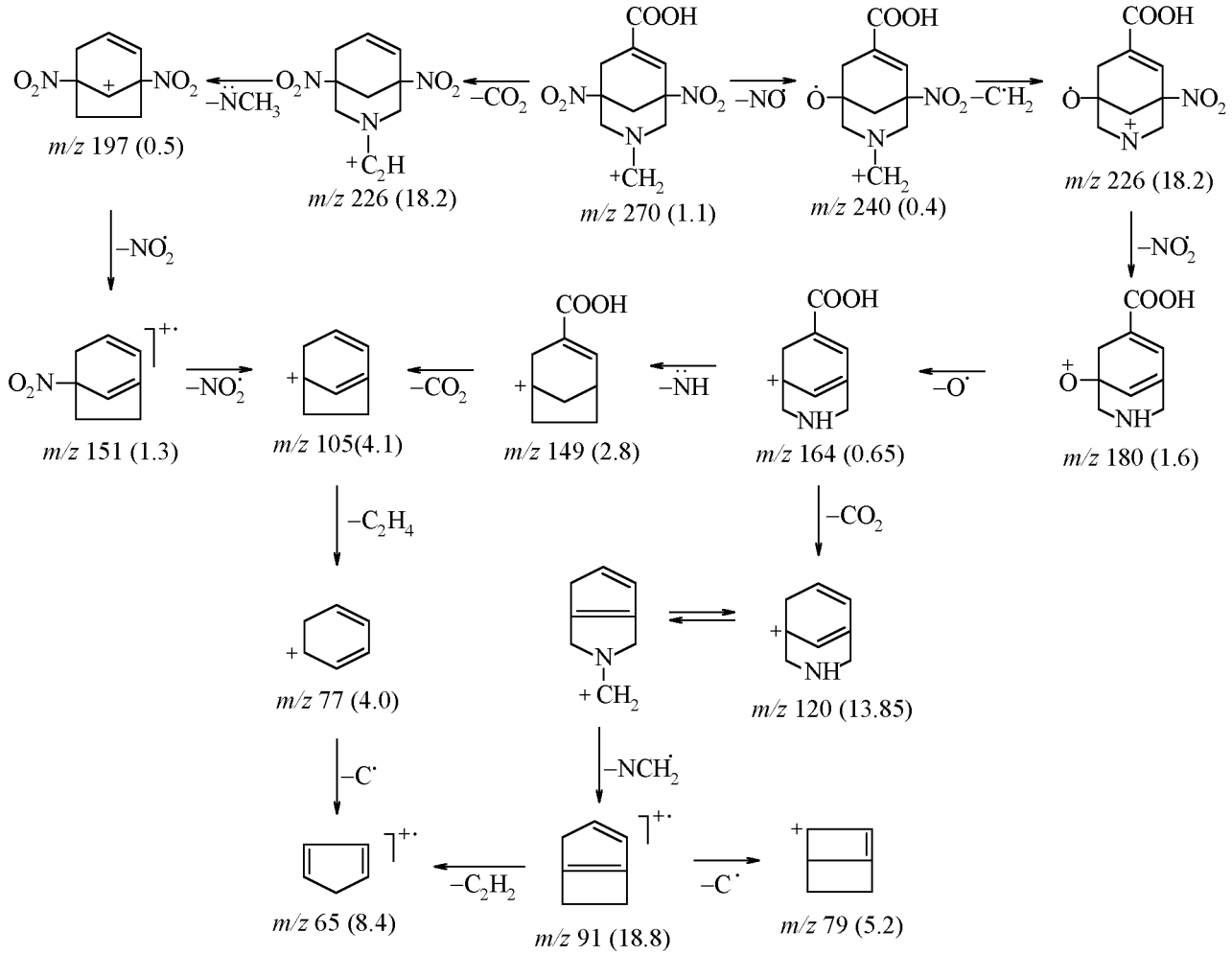
Va, b



IIIa–g

V, *n* = 2 (**a**), 4 (**b**); **VI**, R = CH_3 (**a**), $CH_2C_6H_5$ (**b**), $(CH_2)_2Br$ (**c**), $(CH_2)_2OH$ (**d**).

Scheme 2.



azabicyclo[3.3.1]non-6-en-3-yl)alkanes (**Va, b**) containing two bicyclononane fragments. Polynitropolycarboxylic acids are interesting in particular as initial compounds for synthesis of bifunctional complexones that are extensively used in biology and in medicine [6]. The spectral characteristics of compounds **Va, b** supporting their structure are given in Tables 1 and 2.

Mass spectra additionally prove the structure of compounds obtained. We studied as an example the decomposition under the electron impact of 1,2-bis-(7-carboxy-1,5-dinitro-3-azabicyclo[3.3.1]non-6-en-3-yl)ethane (**Va**) (Scheme 2; the relative abundance of ions with respect to the overall ionic current is given in parentheses). Note that no published data exist on mass-spectrometric studies of bicyclic carboxylic acids with nitro groups in the molecule [7]. We previously demonstrated [4] that the main path of decomposition of the molecular ions arising from 1,5-dinitro-3-azabicyclo[3.3.1]non-6-enes was the rupture of C–N bond with elimination of radicals $NO_2^{\cdot}$ which was sometimes accompanied by nitro-nitrite rearrangement common for nitro compounds [8]. The peak with maximum mass of *m/z* 270 in the mass spectrum of compound **Va** is most probably provided by the thermal decomposition of the molecular ion (*m/z* 540) in two symmetrical fragments already at admission of the sample into the ion source. Low abundance of this fragment ion (I_{rel} 1.1%) is due to high probability of its further decomposition along two paths: with the loss of nitro and carboxy groups respectively (Scheme 2).

The thermal stability of compounds **III, V** synthesized was estimated by derivatography and compared (Table 3) with bicyclononanes **VIa–d** that we had obtained before from unsubstituted *m*-dinitrobenzene [9]. The search in literature revealed that the only study on thermogravimetry of 3-azabicyclo[3.3.1]nonane derivatives [10] which was found did not contain any data on nitro compounds.

Table 2. ^{1}H and ^{13}C NMR spectra of heterocyclic compounds (**IIIa–g**) and (**Va, b**) DMSO-d_6

Compd. no.	^{1}H NMR spectrum δ, ppm (*J*, Hz)											^{13}H NMR, δ, ppm
	CO_2H	H^6	H_e^4	H_a^4	H_e^2	H_a^4	H_e^9	H_a^9	H_e^8	H_a^8	R	
IIIa	12.86 br.s	6.96 s	3.27 d (10.38)	2.55 d (10.38)	3.12 d (10.38)	2.59 d (10.38)	2.80 d (10.99)	2.74 d (10.99)	2.91 br.s		2.34 s (3H, CH_3)	168.22, 136.19, 135.65, 89.51, 87.05, 63.19, 59.11, 52.48, 38.83, 37.63
IIIb	13.00 br.s	6.97 s	3.29 d (10.45)	2.60 d (10.45)	3.17 d (10.45)	2.67 d (10.45)	2.85 d (11.60)	2.83 d (11.60)	2.93 d (18.16)	2.86 d (18.16)	2.57 q (2H, CH_2CH_3, 7.15), 0.957 t (2H, CH_2CH_3, 7.15)	168.22, 136.07, 135.67, 89.44, 86.99, 63.12, 58.97, 52.40, 38.72, 37.51
IIIc	13.00 br.s	7.00 s	3.35 d (10.38)	2.73 d (10.38)	3.21 d (10.38)	2.85–2.76 m			2.95 d (18.31)	2.90 d (18.31)	2.81 m (2H, CH_2CH_2COOH), 2.39 t (2H, CH_2CH_2COOH, 7.02)	175.61,168.09,136.18,135.41, 89.36, 86.94, 63.15, 59.17, 55.10, 38.54, 37.38, 34.07
IIId	13.02 br.s	6.96 s	3.27 d (10.38)	2.61 d (10.38)	3.15 d (10.38)	2.67 d (10.38)	2.82 d (10.99)	2.78 d (10.99)	2.92 d (18.92)	2.85 d (18.92)	2.48 t (2H, $CH_2CH_2CH_2CH_3$, 7.32), 1.35 m (2H, $CH_2CH_2CH_2CH_3$), 1.24 m (2H, $CH_2CH_2CH_2CH_3$), 0.83 t (2H, $CH_2CH_2CH_2CH_3$, 7.32)	–
IIIe	13.00 br.s	7.02 s	3.42 d (10.38)	2.83 d (10.38)	3.28 d (10.38)	2.96 d (10.38)	2.86 d (10.99)	2.84 d (10.99)	2.97 br.s		3.57 m (2H, CH_2CH_2Br), 2.99 m (2H, CH_2CH_2Br)	168.17, 136.37, 135.18, 89.34, 86.89, 62.77, 58.98, 58.87, 38.52, 37.35, 33.36
IIIf	12.48 br.s	6.96 s	3.37 d (10.5)	3.07 d (10.5)	3.20 d (10.5)	3.14 d (10.5)	2.81 d (11.8)	2.73 d (11.8)	2.98 d (18.4)	2.86 d (18.4)	3.47 d and 3.36 d (2H, CH_2COOH, 17.7)	174.01,168.19,136.25,135.39, 89.33, 86.80, 62.43, 58.43, 58.33, 38.37, 37.26
IIIg	5.05[a] br.s	7.15 s	3.55 d (10.50)	2.95 d (10.50)	3.35 d (10.50)	3.05 d (10.50)	2.80 d (10.99)	2.70 d (10.99)	2.90 d (18.90)	2.85 d (18.90)	3.65 t (2H, CH_2CH_2OH, 7.02), 2.85 t (2H, CH_2CH_2OH, 7.02)	168.21, 136.15, 135.51, 89.46, 86.99, 63.98, 61.18, 60.58, 59.83, 38.55, 37.38
Vd	12.89 br.s	6.90 s	3.20 d (10.69)	2.67 d (10.69)	3.15 d (10.83)	2.71 d (10.83)	2.76 d (11.29)	2.72 d (11.29)	2.87 br.s		2.63 m [4H, $(CH_2)2$]	–
Vb	13.04 br.s	6.94 s	3.14 d (10.38)	2.59 d (10.38)	3.11 d (9.77)	2.63 d (9.77)	2.79 d (10.98)	2.76 d (10.98)	2.89 d (20.45)	2.85 d (20.45)	2.47 m (4H, $CH_2CH_2CH_2CH_2$), 1.33 m (4H, $CH_2CH_2CH_2CH_2$)	– –

[a] δ(OH + COOH).

Table 3. Heat resistance of 1,5-dinitro-3-azabicyclo[3.3.1]nonane derivatives **IIIa**–**g, Va, b** and **VIa**–**d**

Compd. no.	Stage of process	DTG			TG (mass loss, %)	DTA		Thermal effect, kJ mol^{-1} °C
		start, °C	maximum, °C	end, °C		start, °C	maximum, °C	
IIIa	1. Melting	–	–	–	–	175	180	–9.9
	2. Decomposition	185	193	200	39.5	183	191	16.4
	3. Decomposition	258	282	335	51.4	252	280	–
IIIb	1. Melting	–	–	–	–	158	170	–14.6
	2. Decomposition	180	192	220	36.4	179	190	132
	3. Decomposition	225	275	385	50.1	223	277	–
IIIc	1. Melting	–	–	–	–	195	197	–6.4
	2. Decomposition	198	208	228	28.1	196	206	184
	3. Decomposition	250	320	350	47.9	250	317	–
IIId	1. Elimination of solvent	80	105	120	5.4	95	105	–
	2. Melting	–	–	–	–	120	132	–24.2
	3. Decomposition	153	178	204	17.6	155	180	48.5
	4. Decomposition	205	243	305	42.2	207	245	–
IIIe	1. Decomposition	185	195	212	16.9	187	196	267
	2. Decomposition	212	240	270	29.5	225	243	–
IIIf	1. Elimination of solvent	80	91	95	9.7	78	88	–68.1
	2. Decomposition	191	200	220	36.5	190	198	169
	3. Decomposition	225	255	310	51.2	225	310	–
IIIg	1. Melting	–	–	–	–	155	165	–
	2. Decomposition	165	177	208	25.6	164	176	196
	3. Decomposition	207	280	350	49.8	208	295	–
Va	1. Elimination of solvent	68	88	100	4.7	68	88	–
	2. Decomposition	180	202	211	21.4	182	203	269
Vb	1. Decomposition	187	193	206	56.5	186	192	363
VIa	1. Melting	–	–	–	–	76	80	–
	2. Decomposition	233	255	305	38.4	235	257	455
VIb	1. Melting	–	–	–	–	40	44	–
	2. Decomposition	255	292	323	37.6	257	294	317
VIc	1. Melting	–	–	–	–	76	78	–
	2. Decomposition	210	218	238	12.5	211	220	749
VId	1. Melting	–	–	–	–	105	108	–
	2. Decomposition	190	205	220	15.0	192	200	273

The comparative evaluation of the heat resistance of bicyclononanes was performed basing on the temperature of the start of decomposition, on the character of the decomposition process, and on the value of mass loss (Table 3). The analysis of the thermogravimetric data (DTG and DTA) revealed that the behavior pattern at heating is considerably different for unsubstituted compounds **VI** and for 7-carboxy bicyclononane derivatives **III**. For instance, all dinitro compounds **VI** have low melting point distinctly appearing on the DTA curve as a pronounced *endo*-effect in the temperature range below 110°C; it is not accompanied by mass loss. In the temperature range from 200 to 320°C the bicyclononanes under study start to decompose. The decomposition is accompanied by mass loss that depends on the character of the R substituent linked to the heterocycle, by a significant heat evolution, and a characteristic exo-effect on the DTA curve. Further heating occurs with mass loss but with no considerable

Table 4. Enthalpy of formation (ΔH_f) of 1,5-dinitro-3-azabicyclo[3.3.1]nonane derivatives **IIIa–g, Va, b,** and **VIa–d** calculated by PM3 method

Compd. no.	ΔH_f, kJ mol^{-1}			
	A	B	C	D
IIIa	–326.50	–361.62	–357.33	–355.09
IIIb	–387.39	–383.37	–380.36	–376.18
IIIc	–749.06	–749.48	–741.28	–741.32
IIId	–432.5	–427.27	–422.58	–422.08
IIIe	–349.65	–347.38	–342.46	–330.11
IIIf	–727.43	–723.09	–716.97	–713.74
IIIg	–545.84	–544.08	–535.80	–535.80
Va	–681.91[a]	–677.56[b]	–671.28[c]	–543.29[d]
Vb	–733.66[e]	–725.71[b]	–719.31[c]	–718.47[f]
VIa	118.57	117.53	121.84	121.88
VIb	2.51	0.84	5.19	5.82
VIc	15.27	11.00	20.59	21.38
VId	–180.54	–182.80	–171.92	–172.13

[a] alculation done for AC conformations.
[b] BC conformation.
[c] CC conformation.
[d] CD conformation.
[e] AD conformation.
[f] DD conformation.

thermal effects which corresponds mainly to elimination of volatile decomposition products.

On the DTA curves belonging to the majority of bicyclononanes **III** three thermal effects were observed (Table 3). The first one is a small endo-effect in the temperature range from 120 to 200°C corresponding to their melting; it smoothly transforms into an exo-effect due to the start of decomposition of the compounds. No melting effects were observed with compounds **IIIe, f**. The first decomposition stage is accompanied by mass loss from 17 to 39% caused apparently by rupture of one or two nitro groups in keeping with the data on the thermal degradation of aliphatic nitro compounds [11]. The thermal effect of the first stage of dinitrobicyclononanes **III** decomposition varies in a wide range: from 16.4 kJ mol^{-1} for compound **IIIa** to 267 kJ mol^{-1} for compound **IIIe**. A special behavior was observed at heating compounds **Va, b** containing two bicyclononane moieties: A single thermal effect appeared on the DTA curves of these compounds in the range of 200°C corresponding to considerable heat evolution (Table 3).

Thus the comparison of the thermal stability of 1,5-dinitro-3-azabicyclo[3.3.1]non-6-enes showed that introduction of a carboxy group into the *7* position considerably increases the heat resistance of compounds.

The stability of organic compounds is often estimated by enthalpy of formation calculated by quantum-mechanical procedures. In this connection we calculated enthalpy of formation (ΔH_f) of heterocycles under study by semiempirical PM3 procedure with complete geometry optimization. Therewith we assume that for 3-azabicyclo[3.3.1]nonanes at least four most stable conformations should be taken into consideration: two conformations where the piperidine ring is in the *chair* form with equatorial (A) and axial (B) orientation of the substituent at the nitrogen atom, and two conformations where the saturated heterocycle is in the form of the *twisted boat* with equatorial (C) and axial (D) substituent position respectively [12]. In all compounds investigated the A and B conformations are more stable than C and D (Table 4). As seen from Table 4, compounds **IIIa, e, g** with a carboxy group in *7* position are significantly more stable than the corresponding derivatives **VIb–d** without the carboxy group. Among 7-carboxy-1,5-dinitro-3-azabicyclo[3.3.1]non-6-enes (**III**) the most stable are the molecules with a carboxyethyl (**IIIc**), carboxymethyl (**IIIf**), and hydroxyethyl (**IIIg**) substituents at the nitrogen atom. The comparison of thermal effects of destruction obtained by derivatography (Table 3) with the calculated values of enthalpy of formation (Table 4) indicates their qualitative agreement.

Thus the use of acid **I** as a substrate for Mannich condensation provided new bicyclic nitrocarboxylic acids. The computer estimation of the spectrum of biological activity (PASS 4.40 [13] program) showed that the compounds synthesized would have properties similar to the γ-aminobutyric acid.

EXPERIMENTAL

IR spectra were recorded on spectrophotometer Specord 75IR from film of acetone solution or mull in mineral oil. ^{1}H NMR spectra were registered on spectrometer Bruker DRX-500 (500 MHz) in DMSO-d_6, internal reference HMDS. Mass spectra of compounds were measured on Varian MAT-311 instrument at the energy of ionizing electrons 70 eV. Simultaneous recording of DTA, DTG, and TG curves was performed on derivatograph Q-1500 D (MOM, Hungary) in air in a crucible under dynamic conditions [14], heating rate 1.25 deg min^{-1} in the

temperature range 20–500°C. As inert substance was used alumina calcined at 1200°C. The weight of sample under study was 100 mg. The sensitivity to mass loss in TG was 0.2 mg, the sensitivity in recording DTA and DTG was 50 and 500 μV respectively. The thermal effect was calculated from DTA curves by procedure [15].

Quantum-chemical calculations were carried out by semiempirical PM3 procedure with the use of software HyperChem 6.0

The retention factors (R_f) were measured on Silufol UV-254 plates, eluent benzene–acetone (2 : 1), spots visualized by UV irradiation and iodine vapor.

7-Carboxy-3-R-1,5-dinitro-3-azabicyclo[3.3.1]-non-6-enes (IIIa–e). To a solution of 0.84 g (4 mmol) of 3,5-dinitrobenzoic acid (**I**) in 10 ml of 50% aqueous ethanol was added gradually at stirring 0.61 g (16 mmol) of sodium borohydride maintaining the reaction temperature in 10–15°C range. On completion of reduction the reaction mixture was cautiously neutralized with 20% H_3PO_4 till pH 7, and a mixture was added containing 2,3 ml of 30% formaldehyde solution (24 mmol) and 8 mmol of the required amine (or its hydrochloride), and the pH of the solution was adjusted with phosphoric acid at pH 5. The separated precipitate was filtered off and recrystallized from ethanol.

1,2-Bis(7-carboxy-3-R-1,5-dinitro-3-azabicyclo-[3.3.1]non-6-en-3-yl)alkanes (**Va, b**) were obtained in a similar way using as amines 1,2-diaminoethane and 1,4-diaminobutane.

The syntheses of the other compounds were described before [3, 4, 9].

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