

## 3-Azabicyclo[3.3.1]nonane Derivatives: V.\* Synthesis of 7-Polyfluoroalkoxy-1,5-dinitro-3-azabicyclo[3.3.1]non-6-enes

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Received February 14, 2000

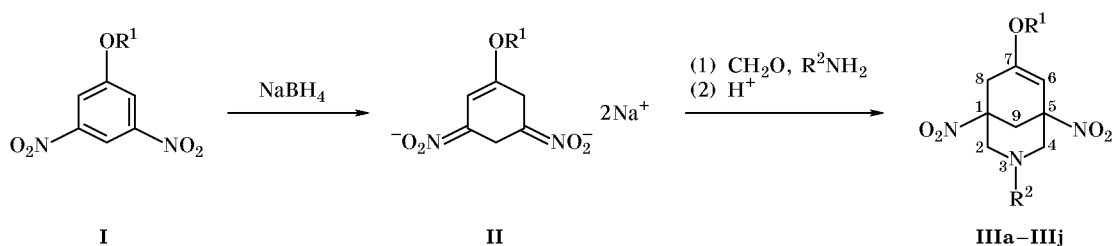
**Abstract**—A number of 7-polyfluoroalkoxy-1,5-dinitro-3-azabicyclo[3.3.1]non-6-enes were synthesized by reduction of 3,5-dinitrophenyl polyfluoroalkyl ethers with sodium tetrahydridoborate, followed by the Mannich reaction with formaldehyde and primary amines.

Heteroanalogs of bicyclo[3.3.1]nonane are valuable intermediate products in organic synthesis and convenient models for conformational analysis; they constitute structural fragments of natural terpenoid alkaloids and exhibit biological activity [2, 3]. It is known that in some cases introduction of fluoroalkyl groups into heterocyclic compounds strongly enhances their biological activity [4]. Direct fluorination of heterocycles is characterized by low efficiency because of the lack of regio- and stereoselectivity and poor yields of the target products. Therefore, fluorine-containing synthons are used for this purpose [5]. The goal of the present work was to synthesize 3-azabicyclo[3.3.1]nonane derivatives having a fluorinated fragment. The procedure for preparation of bicycnonanes from 1,3-dinitrobenzene derivatives was

reported in [6, 8], but it was not applied to synthesis of fluorinated compounds of this series.

Shevelev *et al.* [9] recently developed a procedure for preparation of initial 3,5-dinitrophenyl polyfluoroalkyl ethers **I** by replacement of the nitro group in 1,3,5-trinitrobenzene by the action of polyfluorinated alcohols [9]. The reduction of ethers **I** with sodium tetrahydridoborate gave disodium salts **II** which were brought (without isolation) into the Mannich reaction with formaldehyde and primary amines. As a result, the corresponding 3-substituted 1,5-dinitro-7-polyfluoroalkoxy-3-azabicyclo[3.3.1]non-6-enes **III** were obtained (Scheme 1). The structure of products **III** was proved by the data of IR and <sup>1</sup>H and <sup>19</sup>F NMR spectroscopy (Tables 1, 2) and elemental analysis (Table 3). Their IR spectra (Table 1) contained bands

Scheme 1.



R<sup>1</sup> = CH<sub>2</sub>CF<sub>3</sub>, R<sup>2</sup> = Me (a), Et (b), PhCH<sub>2</sub> (c), CH<sub>2</sub>CH<sub>2</sub>Br (d), CH<sub>2</sub>CH<sub>2</sub>OH (e), CH<sub>2</sub>COOH (f), CH<sub>2</sub>CH<sub>2</sub>COOH (g);  
R<sup>1</sup> = CH<sub>2</sub>CF<sub>2</sub>CF<sub>2</sub>H, R<sup>2</sup> = Me (h), PhCH<sub>2</sub> (i); R<sup>1</sup> = CH<sub>2</sub>(CF<sub>2</sub>)<sub>3</sub>CF<sub>2</sub>H, R<sup>2</sup> = Me (j).

\* For communication IV, see [1].

**Table 1.** IR spectra of 3-substituted 7-polyfluoroalkoxy-1,5-dinitro-3-azabicyclo[3.3.1]non-6-enes **IIIa–IIIj**

Comp. no.	IR spectrum, $\nu$ , $\text{cm}^{-1}$					
	$\text{NO}_2$ (s)	$\text{NO}_2$ (as)	C–F	C–O	C–H (aliph.)	other bands
<b>IIIa</b>	1363	1553	1080	1137, 1177	2821, 2953	
<b>IIIb</b>	1353, 1380	1555	1075	1176, 1233, 1293	2832, 2975	
<b>IIIc</b>	1349, 1383	1563	1087	1176, 1231, 1289	2816, 2920, 2948	3027, 3060, 3080 (C–H <sub>arom</sub> )
<b>III d</b>	1339, 1357	1532	1072	1153, 1213, 1271	2814, 2877, 2945	
<b>IIIe</b>	1385	1559	1091	1177, 1241, 1296	2840, 2892, 2959	3433 (O–H)
<b>III f</b>	1355, 1373	1551	1085	1167, 1223, 1288	2847, 2887, 2947	1731 (C–O), 2500–3400 (O–H)
<b>III g</b>	1349, 1370	1550	1099	1155, 1185, 1220	2837, 2880, 2933	1713 (C–O), 2500–3400 (O–H)
<b>III h</b>	1361	1527	1087	1137, 1200, 1257	2847, 2876, 2943	
<b>III i</b>	1357, 1387	1560	1124	1164, 1225	2820, 2889, 2917	3028, 3064, 3084 (C–H <sub>arom</sub> )
<b>III j</b>	1345, 1369	1540	1073	1172, 1250, 1288	2809, 2853, 2957	

at 2830–2990 [ $\nu(\text{C–H}_{\text{aliph}})$ ], 1340–1380 and 1540–1560 [ $\nu_{\text{s,as}}(\text{NO}_2)$ ], and 1070–1100  $\text{cm}^{-1}$  [ $\nu(\text{C–F})$ ]. The carboxy group in compounds **III f** and **III g** gives rise to absorption in the regions 2500–3100 (OH) and 1710–1755  $\text{cm}^{-1}$  (C=O).

When analyzing the  $^1\text{H}$  NMR spectra of compounds **III** (Table 2) we assumed that the piperidine ring adopts a *chair* conformation with no eclipsed groups and that the cyclohexene ring is nearly planar. Such a structure was determined for related 3-azabicyclo[3.3.1]nonanes by the X-ray diffraction data [10]. The methylene protons  $8\text{-H}_{\text{eq}}$  and  $8\text{-H}_{\text{ax}}$  are nonequivalent due to their different arrangement with respect to the equatorial plane of the piperidine ring and  $\text{C}^1\text{NO}_2$  group; they are coupled through a constant  $^2J$  of 17 to 18 Hz. The presence of a double bond in molecules **III** follows from the position of the 6-H signal,  $\delta$  5.20–5.28 ppm. The coupling constants  $^4J_{6,8}$  are small, and the 6-H signal appears as a slightly broadened singlet. The methylene protons in positions 2, 4, and 9 of the piperidine ring give rise to three pairs of broadened doublets from equatorial and axial protons ( $^2J = 10\text{--}12$  Hz). These signals are poorly resolved, for the coupling constants  $^4J$  approach the performance limit of the NMR spectrometer. The doublets at  $\delta$  3.30–3.40 ppm were assigned to  $4\text{-H}_{\text{eq}}$  which suffers the strongest anisotropic effect of the double bond. Compounds **III** are also characterized by nonequivalence of methylene protons in the fluoroalkoxy group. The corresponding signals are doublets each component of which is split into a triplet or

quartet due to coupling with difluoromethyl or trifluoromethyl group. The 3-carboxymethyl group in compound **III d** cannot adopt *endo* configuration, for its two carbon and two oxygen atoms should occur in one plane. As a result, the methylene protons therein become nonequivalent due to asymmetric arrangement relative to the  $\text{C}^2\text{N}^3\text{C}^4$  plane; they are coupled through a constant  $^2J$  of 17.2 Hz. The methylene protons of the *N*-benzyl group in compounds **III c** and **III i** are also nonequivalent. The number of signals observed in the  $^{19}\text{F}$  NMR spectra is consistent with the number of nonequivalent fluorine atoms.

It is interesting that, unlike reduction of 2,4,6-trinitroanisole with  $\text{NaBH}_4$  [11], in our case the C–O bond in the substrates is retained. The presence of various functional groups in molecules of products **III** provides wide possibilities for their chemical modification with a view to obtain compounds possessing desired properties. Assessment of a probable range of biological activity of 3-azabicyclo[3.3.1]nonane derivatives **III** showed\* that they are potential neurotropic and antibacterial substances.

## EXPERIMENTAL

The IR spectra (film, from acetone) were recorded on a Specord 75IR spectrometer. The  $^1\text{H}$  and  $^{19}\text{F}$  NMR spectra were obtained on a Bruker AC-300

\* PASS-C 4.0 program created by V.V. Poroikov and D.A. Filimonov (IBMKh RAMN, Moscow).

**Table 2.**  $^1\text{H}$  and  $^{19}\text{F}$  NMR spectra of 7-polyfluoroalkoxy-1,5-dinitro-3-azabicyclo[3.3.1]non-6-enes **IIIa–IIIj**

Comp. no.	Chemical shifts $\delta$ and $\delta_{\text{F}}$ , ppm ( $J$ , Hz)							
	R <sup>1</sup>	R <sup>2</sup>	R <sup>1</sup>	R <sup>2</sup>	2-H <sub>eq</sub>	2-H <sub>ax</sub>	4-H <sub>eq</sub>	4-H <sub>ax</sub>
<b>IIIa</b>	CH <sub>2</sub> <sup><math>\alpha,\beta</math></sup> CF <sub>3</sub>	CH <sub>3</sub>	4.32 d.q (H <sup><math>\alpha</math></sup> ), 4.24 d.q (H <sup><math>\beta</math></sup> ), ( $J_{\alpha,\beta}$ 11.1; $^3J_{\text{HF}}$ 8.3)	2.38 s	3.05 d (10.4)	2.48 d (10.4)	3.30 d (10.6)	2.49 d (10.6)
<b>IIIb</b>	CH <sub>2</sub> <sup><math>\alpha,\beta</math></sup> CF <sub>3</sub>	CH <sub>2</sub> CH <sub>3</sub>	4.34 d.q (H <sup><math>\alpha</math></sup> ), 4.24 d.q (H <sup><math>\beta</math></sup> ), ( $J_{\alpha,\beta}$ 12; $^3J_{\text{HF}}$ 8.5)	1.04 t (CH <sub>3</sub> ), 2.64 q (CH <sub>2</sub> , 7.1)	3.10 d (10.4)	2.57 d (10.4)	3.33 d (10.5)	2.60 d (10.5)
<b>IIIc</b>	CH <sub>2</sub> <sup><math>\alpha,\beta</math></sup> CF <sub>3</sub>	CH <sub>2</sub> <sup><math>\gamma,\delta</math></sup> Ph	4.39 d.q (H <sup><math>\alpha</math></sup> ), 4.33 d.q (H <sup><math>\beta</math></sup> ), ( $J_{\alpha,\beta}$ 9.7; $^3J_{\text{HF}}$ 9.5)	3.77 d (H <sup><math>\gamma</math></sup> ) 3.70 d (H <sup><math>\delta</math></sup> , 13.8) 7.31 m (Ph)	3.03 d (10.2)	2.60 d (10.2)	3.30 d (10.2)	2.66 d (10.2)
<b>III d</b>	CH <sub>2</sub> <sup><math>\alpha,\beta</math></sup> CF <sub>3</sub>	CH <sub>2</sub> <sup><math>\gamma</math></sup> CH <sub>2</sub> <sup><math>\delta</math></sup> Br	4.32 d.q (H <sup><math>\alpha</math></sup> ), 4.27 d.q (H <sup><math>\beta</math></sup> ), ( $J_{\alpha,\beta}$ 14; $^3J_{\text{HF}}$ 8.5)	3.00 t (H <sup><math>\gamma</math></sup> ) 3.47 t (H <sup><math>\delta</math></sup> , 6.3)	3.11 d (10.4)	2.87 d (10.4)	3.38 d (10.4)	2.81 d (10.4)
<b>III e</b>	CH <sub>2</sub> <sup><math>\alpha,\beta</math></sup> CF <sub>3</sub>	CH <sub>2</sub> <sup><math>\gamma</math></sup> CH <sub>2</sub> <sup><math>\delta</math></sup> OH	4.35 d.q (H <sup><math>\alpha</math></sup> ), 4.25 d.q (H <sup><math>\beta</math></sup> ), ( $J_{\alpha,\beta}$ 11; $^3J_{\text{HF}}$ 8.5)	2.75 t (H <sup><math>\gamma</math></sup> ) 3.58 q (H <sup><math>\delta</math></sup> , 7) 2.55 t (OH, 7)	3.14 d (10.3)	2.75 d (10.3)	3.39 d (10.3)	2.77 d (10.3)
<b>III f</b>	CH <sub>2</sub> <sup><math>\alpha,\beta</math></sup> CF <sub>3</sub>	CH <sub>2</sub> <sup><math>\gamma,\delta</math></sup> COOH	4.33 d.q (H <sup><math>\alpha</math></sup> ), 4.25 d.q (H <sup><math>\beta</math></sup> ), ( $J_{\alpha,\beta}$ 9.6; $^3J_{\text{HF}}$ 9.5)	3.52 d (H <sup><math>\gamma</math></sup> ) 3.44 d (H <sup><math>\delta</math></sup> , 17.2) 9.50 s (COOH)	3.10 d (10.5)	3.06 d (10.5)	3.37 d (10.5)	3.05 d (10.5)
<b>III g</b>	CH <sub>2</sub> CF <sub>3</sub>	CH <sub>2</sub> <sup><math>\alpha</math></sup> CH <sub>2</sub> <sup><math>\beta</math></sup> CO <sub>2</sub> H	4.27 q (8.3),	2.87 t (H <sup><math>\alpha</math></sup> ) 2.46 t (H <sup><math>\beta</math></sup> , 6.1) 10.70 s (COOH)	3.17 d (10.6)	2.71 d (10.6)	3.42 d (10.5)	2.79 d (10.5)
<b>III h</b>	CH <sub>2</sub> <sup><math>\alpha,\beta</math></sup> CF <sub>2</sub> CF <sub>2</sub> H <sup><math>\gamma</math></sup>	CH <sub>3</sub>	4.27 d.t (H <sup><math>\alpha</math></sup> ), 4.14 d.t (H <sup><math>\beta</math></sup> ), ( $J_{\alpha,\beta} = J_{\text{HF}} = 12$ ), 6.20 t.t (H <sup><math>\gamma</math></sup> ) ( $^2J_{\text{HF}}$ 52; $^3J_{\text{HF}}$ 5.3)	2.39 s	3.06 d (10.0)	2.48 d (10.0)	3.29 d (10.0)	2.49 d (10.0)
<b>III i</b>	CH <sub>2</sub> <sup><math>\alpha,\beta</math></sup> CF <sub>2</sub> CF <sub>2</sub> H <sup><math>\epsilon</math></sup>	CH <sub>2</sub> <sup><math>\gamma,\delta</math></sup> Ph	4.37 d.t (H <sup><math>\alpha</math></sup> ), 4.31 d.t (H <sup><math>\beta</math></sup> ), ( $J_{\alpha,\beta} = J_{\text{HF}} = 11.2$ ), 6.20 t.t (H <sup><math>\gamma</math></sup> ) ( $^2J_{\text{HF}}$ 52; $^3J_{\text{HF}}$ 5.5)	3.76 d (H <sup><math>\gamma</math></sup> ) 3.70 d (H <sup><math>\delta</math></sup> , 13.5) 7.31 m (Ph)	3.04 d (10.6)	2.60 d (10.6)	3.29 d (10.8)	2.66 d (10.8)
<b>III j</b>	CH <sub>2</sub> <sup><math>\alpha,\beta</math></sup> (CF <sub>2</sub> ) <sub>3</sub> CF <sub>2</sub> H <sup><math>\gamma</math></sup>	CH <sub>3</sub>	4.43 d.t (H <sup><math>\alpha</math></sup> ), 4.34 d.t (H <sup><math>\beta</math></sup> ), ( $J_{\alpha,\beta} = J_{\text{HF}} = 13.4$ ), 6.43 t.t (H <sup><math>\gamma</math></sup> ) ( $^2J_{\text{HF}}$ 50.8; $^3J_{\text{HF}}$ 5.5)	2.39 s	3.06 d (10.4)	2.48 d (10.4)	3.30 d (10.4)	2.50 d (10.4)
Comp. no.	6-H	8-H <sub>eq</sub>	8-H <sub>ax</sub>	9-H <sub>eq</sub>	9-H <sub>ax</sub>	$^{19}\text{F}$		
<b>IIIa</b>	5.23 s	2.92 d (17.9)	2.82 d (17.9)	2.81 d (11.2)	2.68 d (11.2)	-73.27, -73.43		
<b>IIIb</b>	5.23 s	2.93 d (17.4)	2.78 d (17.4)	2.81 d.t (11.1; 2.0)	2.69 d (11.1)	-73.12, -73.25		
<b>IIIc</b>	5.20 s	2.92 d (18.0)	2.76 d (18.0)	2.82 d (11.4)	2.71 d (11.4)	-73.19		
<b>III d</b>	5.21 s	2.94 d (17.4)	2.82 d (17.4)	2.80 d (11.0)	2.71 d (11.0)	-73.19, -73.27		
<b>III e</b>	5.24 s	2.94 d (17.8)	2.80 d (17.8)	2.82 d (11.0)	2.71 d (11.0)	-73.13, -73.22		

Table 2. (Contd.)

Comp. no.	6-H	8-H <sub>eq</sub>	8-H <sub>ax</sub>	9-H <sub>eq</sub>	9-H <sub>ax</sub>	<sup>19</sup> F
<b>III f</b>	5.22 s	2.92 d (17.7)	2.83 d (17.7)	2.82 d (11.3)	2.71 d (11.3)	-71.55
<b>III g</b>	5.20 s	2.96 d (17.8)	2.78 d (17.8)	2.85 d (11.2)	2.71 d (11.2)	-73.07, -73.13
<b>III h</b>	5.24 s	2.92 d (17.0)	2.81 d (17.0)	2.80 d (11.0)	2.68 d (11.0)	-125.02, -139.5
<b>III i</b>	5.20 s	2.93 d (17.0)	2.75 d (17.0)	2.83 d (12.0)	2.72 d (12.0)	-124.9, -139.35
<b>III j</b>	5.28 s	2.92 d (17.6)	2.82 d (17.6)	2.81 d (11.3)	2.69 d (11.3)	-119.27, -124.51 -129.64, -137.84

Table 3. Yields, melting points, R<sub>f</sub> values, and elemental analyses of 3-substituted 7-polyfluoroalkoxy-1,5-dinitro-3-azabicyclo[3.3.1]non-6-enes **IIIa–IIIj**

Comp. no.	Yield, %	mp, °C	R <sub>f</sub>	Found, %			Formula	Calculated, %		
				C	H	N		C	H	N
<b>III a</b>	55	107–108	0.68	40.57, 40.51	4.35, 4.32	12.85, 12.89	C <sub>11</sub> H <sub>14</sub> F <sub>3</sub> N <sub>3</sub> O <sub>5</sub>	40.62	4.34	12.92
<b>III b</b>	48	73–74	0.79	42.76	4.70	12.41	C <sub>12</sub> H <sub>16</sub> F <sub>3</sub> N <sub>3</sub> O <sub>5</sub>	42.48	4.75	12.39
<b>III c</b>	37	111–112	0.87	50.72, 50.97	4.63, 4.62	10.91	C <sub>17</sub> H <sub>18</sub> F <sub>3</sub> N <sub>3</sub> O <sub>5</sub>	50.88	4.52	10.47
<b>III d</b>	27	98–99	0.78	34.61	3.56	9.98	C <sub>12</sub> H <sub>15</sub> BrF <sub>3</sub> N <sub>3</sub> O <sub>5</sub>	34.47	3.62	10.05
<b>III e</b>	23	77–78	0.33	40.52	4.51	11.78	C <sub>12</sub> H <sub>16</sub> F <sub>3</sub> N <sub>3</sub> O <sub>6</sub>	40.57	4.54	11.83
<b>III f</b>	25	138–139	0.36	39.06, 38.92	4.01, 3.93	11.40, 11.44	C <sub>12</sub> H <sub>14</sub> F <sub>3</sub> N <sub>3</sub> O <sub>7</sub>	39.03	3.82	11.38
<b>III g</b>	26	149–150	0.32	40.80	4.19	10.35	C <sub>13</sub> H <sub>16</sub> F <sub>3</sub> N <sub>3</sub> O <sub>7</sub>	40.74	4.21	10.96
<b>III h</b>	59	126–127	0.64	40.23, 40.20	4.32, 4.30	11.84	C <sub>12</sub> H <sub>15</sub> F <sub>4</sub> N <sub>3</sub> O <sub>5</sub>	40.34	4.23	11.76
<b>III i</b>	30	72–73	0.88	49.87, 50.01	4.34, 4.46	9.92	C <sub>18</sub> H <sub>19</sub> F <sub>4</sub> N <sub>3</sub> O <sub>5</sub>	49.89	4.49	9.70
<b>III j</b>	52	126–127	0.65	36.83, 36.81	3.32, 3.32	9.22	C <sub>14</sub> H <sub>15</sub> F <sub>8</sub> N <sub>3</sub> O <sub>5</sub>	36.77	3.31	9.19

instrument in CD<sub>3</sub>CN using HMDS as internal reference. Thin-layer chromatography was performed on Silufol UV-254 plates using toluene–acetone–hexane (4:1:1) as eluent; spots were visualized with UV light and iodine vapor. The melting points were determined on a Koeffler device (Boetius).

**3-Substituted 1,5-dinitro-7-polyfluoroalkoxy-3-azabicyclo[3.3.1]non-6-enes IIIa–IIIj.** A 4-mmol portion of 3,5-dinitrophenyl polyfluoroalkyl ether **I** was dissolved in 10 ml of tetrahydrofuran–water mixture (1:1, by volume). The mixture was cooled with ice water, 0.74 g (20 mmol) of NaBH<sub>4</sub> was added over a period of 10–15 min under continuous stirring, and the mixture was stirred for 15 min at 10–20°C.

Cold 30% aqueous formaldehyde, 2.3 ml (24 mmol), and 12 mmol of appropriate amine in 20 ml of water were added, and the mixture was acidified to pH 6 with glacial acetic acid and was extracted with toluene or dichloroethane. The extract was dried over CaCl<sub>2</sub> and evaporated under reduced pressure. The residue was subjected to column chromatography on ASKG silica gel, using the following solvent systems as eluent: **IIIa–III d**, **III h–III j**, toluene; **III e**, toluene–acetone (20:1); **III f**, **III g**, toluene–acetone (10:1). Compounds **IIIa–III d** and **III h–III j** were recrystallized from ethanol. Products **III e–III g** were dissolved in ethanol, the solution was treated with charcoal and filtered, and the product was precipitated with water.

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