Bis(trifluoromethyl)-containing 1-azatricycloheptanes

2.* Conjugated addition of O-nucleophiles to 3-halo-7,7-bis(trifluoromethyl)-1-azatricyclo[2.2.1.$0^{2,6}$]heptanes

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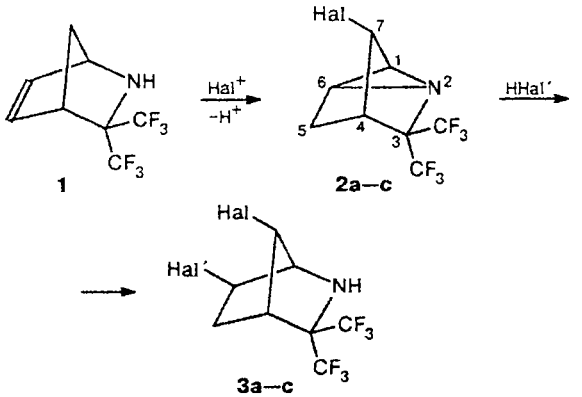
6-Substituted 7-halo-3,3-bis(trifluoromethyl)-2-azabicyclo[2.2.1]heptanes were synthesized by the addition of water, alcohols, and acetic acid to 3-halo-7,7-bis(trifluoromethyl)-1-azatricyclo[2.2.1.$0^{2,6}$]heptanes in the presence of H_2SO_4. 5,6-Disubstituted 3,3-bis(trifluoromethyl)-2-azabicyclo[2.2.1]heptanes were prepared by oxymercuration of 3,3-bis(trifluoromethyl)-2-azabicyclo[2.2.1]hept-5-ene.

Key words: 1-azatricycloheptanes, conjugated addition, rearrangements; "nonclassical cation"; 2-azabicycloheptanes; 2-azabicycloheptenes, oxymercuration.

It has been shown in the previous communication[1] that halogenation of 3,3-bis(trifluoromethyl)-2-azabicyclo[2.2.1]hept-5-ene (**1**) under conditions of conjugated reactions affords 3-*anti*-halo-containing 1-azanortricyclanes **2** (halogen is Cl, Br, or I); hydrohalogenation of the latter yields the corresponding 6,7-dihalo-substituted 2-azanorbornanes **3** (Scheme 1) including those containing different halogen atoms.

Scheme 1



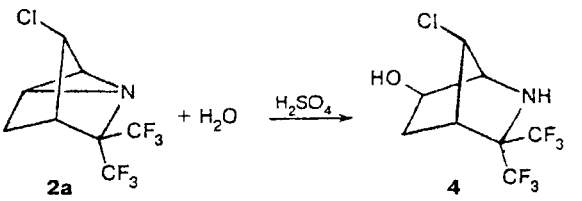
1 **2a—c** **3a—c**

Hal = Cl (**a**), Br (**b**), I (**c**)

The occurrence of the **2** → **3** transformation has been explained by the tendency of the aziridine moiety in 1-azanortricyclanes to undergo decyclization upon protonation by hydrogen halides to give the corresponding azabicyclic carbonium cation, which is stabilized by adding the counterion (F^-, Cl^-, Br^-, I^-). In the present communication, we consider the legitimacy of the hypothesis concerning the crucial role of the proton attack in relation to reactions of substituted 1-azanortricyclanes **2** with H_2O, ROH, and AcOH in the presence of H_2SO_4. It should be noted that in the absence of H_2SO_4, 1-azatricyclanes are inert with respect to the above-listed reagents and weak acids like acetic acid.[1]

When 1-azanortricyclane **2a** is treated with dilute aqueous H_2SO_4 (Scheme 2), 7-chloro-6-hydroxy-3,3-bis(trifluoromethyl)-2-azabicyclo[2.2.1]heptane (**4**) is formed (yield >70%).

Scheme 2



2a **4**

Treatment of solutions of each of 1-azanortricyclanes **2a—c** in MeOH with catalytic amounts of H_2SO_4 ($d \geq 1.8$) gave 7-chloro-, 7-bromo-, and 7-iodo-6-methoxy-3,3-bis(trifluoromethyl)-2-azabicyclo[2.2.1]heptanes **5—7**, respectively, in high yields (Scheme 3).

Similarly, chloro-containing 1-azanortricyclane **2a** reacts with EtOH, 2-chloroethanol, or ethylene glycol in the presence of H_2SO_4 to give the corresponding 6-alkoxy-7-chloro-3,3-bis(trifluoromethyl)-2-azabicyclo[2.2.1]heptanes **8—10** (Scheme 4).

* For Part 1, see Ref. 1

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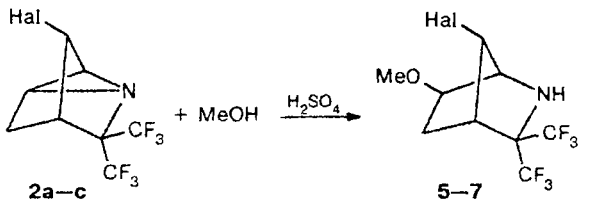
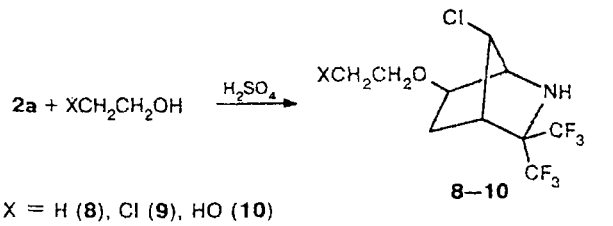
Scheme 3



Hal = Cl (**5**), Br (**6**), I (**7**)

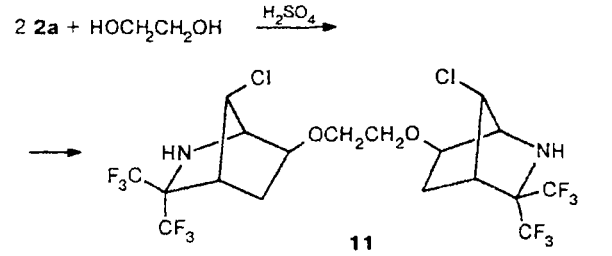
Scheme 4



X = H (**8**), Cl (**9**), HO (**10**)

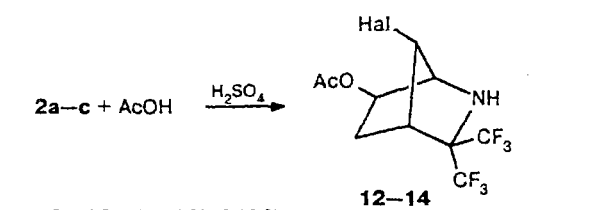
In the latter case, variation of the ratio of the reactants made it possible to obtain bis(2-azanorborn-6-yl) derivative **11** (Scheme 5).

Scheme 5



6-Acetoxy-7-halo-substituted 2-azabicyclo[2.2.1]heptanes **12—14** were synthesized by treatment of solutions of 1-azanortricyclanes **2a—c**, respectively, in AcOH with concentrated H_2SO_4 (Scheme 6).

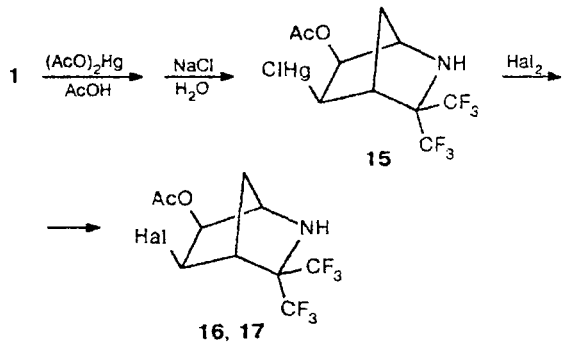
Scheme 6



Hal = Cl (**12**), Br (**13**), I (**14**)

It is noteworthy that regioisomers of compounds **13** and **14** can be obtained. The method is based on the ability of 2-azanorbornene **1** to undergo oxymercuration according to the Hofmann—Sand[2] reaction pattern to give 6-acetoxy-5-chloromercuro-3,3-bis(trifluoromethyl)-2-azabicyclo[2.2.1]heptane (**15**), which undergoes halodemercuration to yield the corresponding 6-acetoxy-5-bromo- and 6-acetoxy-5-iodo-3,3-bis(trifluoromethyl)-2-azabicyclo[2.2.1]heptanes **16** and **17** (Scheme 7).

Scheme 7

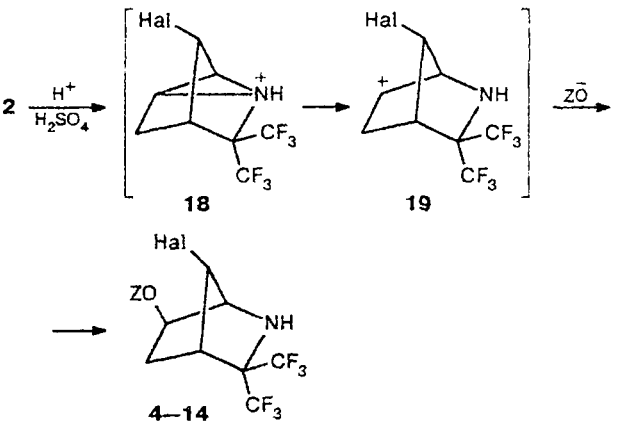


Hal = Br (**16**), I (**17**)

The ^{19}F NMR spectra of *C*-substituted 2-azabicyclo[2.2.1]heptanes **4—17** exhibit characteristic signals for the $C(CF_3)_2$ group as two quartets at 66.0—67.2 and 72.0—73.3 ppm with the spin-spin coupling constant ^4J = 11 to 13 Hz. The parameters of the ^1H NMR spectra of 6,7-disubstituted 2-azanorbornanes **4—10** and **12—14** are listed in Table 1.

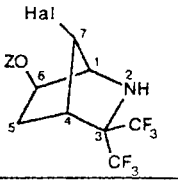
Analysis of the ability of 3-halo-7,7-bis(trifluoromethyl)-1-azanortricyclanes of type **2** to add O-nucleophiles in the presence of H_2SO_4 made it possible to

Scheme 8



Z = H, Alk, Ac

Table 1. ^{1}H NMR spectra of 6,7-disubstituted 3,3-bis(trifluoromethyl)-2-azabicyclo[2.2.1]heptanes



Compound	Z	Hal	δ (*J*/Hz)						
			HC(1)	HN	HC(4)	2 HC(5)	HC(6)	HC(7)	Z
4	H	Cl	3.53 (s)	2.40 (s)	3.03 (m)	3.03 (m)	4.10 (s)	4.67 (s)	2.40 (m)
5	Me	Cl	3.50 (s)	2.32 (s)	2.83 (s)	2.27 (m)	3.60 (t, J = 6)	4.50 (s)	3.30 (s)
6	Me	Br	3.56 (s)	2.33 (s)	2.83 (s)	2.33 (m)	3.46 (m)	4.56 (s)	3.36 (s)
7	Me	I	3.50 (s)	2.30 (s)	2.82 (s)	2.27 (m)	3.58 (s)	4.45 (s)	3.30 (s)
8	$MeCH_2$	Cl	3.57 (s)	2.27 (s)	2.97 (s)	2.33 (d, J = 6)	3.77 (t, J = 6)	4.55 (s)	1.27 (t); 3.53 (q, J = 7)
9	$ClCH_2CH_2$	Cl	3.43—3.76 (m)	2.40 (s)	2.90 (s)	2.38 (m)	3.83 (t, J = 6)	4.53 (s)	3.43—3.76 (m)
10	$HOCH_2CH_2$	Cl	2.93—3.20 (m)		2.43 (s)	1.76 (m)	3.37 (t, J = 6)	4.03 (s)	2.93—3.20 (m)
12	Ac	Cl	3.43 (s)	2.63 (s)	2.80 (s)	2.30 (m)	4.63 (m)	4.40 (s)	1.97 (s)
13	Ac	Br	3.20 (s)	3.47 (s)	2.60 (s)	1.97 (m)	4.37 (t, J = 6)	4.10 (s)	1.53 (s)
14	Ac	I	3.62 (s)	2.67 (s)	2.83 (s)	2.43 (m)	4.73 (m)	4.45 (s)	2.00 (s)

conclude that these reactions follow a common pathway. It can be claimed that protonation to give the ammonium ion of type **18** is the primary act in all these reactions (Scheme 8); cleavage of the three-membered ring in ion **18** accompanied by transfer of the reaction site affords carbonium ion **19**. The latter is stabilized due to the addition of the counterion to yield the corresponding *C*-substituted 2-azanorbornane **4—14**.

This scheme is in agreement with that considered previously[1] for hydrohalogenation of substituted 1-azatricyclo[2.2.1.$0^{2,6}$]heptanes.

Experimental

^{1}H and ^{19}F NMR spectra were recorded on a Varian EM-890 spectrometer (in CCl_4).* The chemical shifts were referred to $SiMe_4$ and $CFCl_3$, respectively. IR spectra were measured on a Perkin—Elmer PE-286 spectrophotometer.

3-Chloro-, 3-bromo-, and 3-iodo-7,7-bis(trifluoromethyl)-1-azatricyclo[2.2.1.$0^{2,6}$]heptanes (2a—c) were synthesized by a procedure described previously.[1]

7-Chloro-6-hydroxy-3,3-bis(trifluoromethyl)-2-azabicyclo[2.2.1]heptane (4). Water (10 mL) and H_2SO_4 (d = 1.81, 0.5 mL) were added to a solution of compound **2a** (5.0 g) in 40 mL of MeCN. The solution was stirred for 5 h at 60—80 °C and diluted with 200 mL of H_2O. The product was extracted with CH_2Cl_2 (2×10 mL), and the extract was washed with 50 mL of a 10% aqueous solution of Na_2CO_3 and dried with $CaCl_2$. The solvent was evaporated. Repeated recrystallization of the residue from hexane gave 3.9 g (73%) of compound **1**, m.p. 47 °C. Found (%): C, 34.18; H, 2.54; Cl, 11.71; N, 4.91. $C_8H_8ClF_6NO$. Calculated (%): C, 33.86; H, 2.82; Cl, 12.52; N, 4.94. ^{19}F NMR, δ: 66.5 (q, 3 F, J = 11.3 Hz); 72.0 (q, 3 F, J = 11.3 Hz). IR (CCl_4), ν/cm^{-1}: 3595 (O—H); 3391 (N—H).

7-Chloro-6-methoxy-3,3-bis(trifluoromethyl)-2-azabicyclo[2.2.1]heptane (5). Sulfuric acid (d = 1.81, 0.2 mL) was added to a solution of compound **2a** (10 g) in 15 mL of MeOH. The solution was stirred for 6 h at 20 °C, neutralized with Na_2CO_3, and filtered. The filtrate was concentrated, and the residue was twice recrystallized from hexane to give 8.5 g (75.9%) of compound **5**, m.p. 79 °C. Found (%): C, 35.94; H, 3.08; Cl, 11.07; N, 4.67. $C_9H_{10}ClF_6NO$. Calculated (%): C, 36.30; H, 3.36; Cl, 11.90; N, 4.71. ^{19}F NMR, δ: 66.7 (q, 3 F, J = 11.3 Hz); 72.0 (q, 3 F, J = 11.3 Hz). IR (CCl_4), ν/cm^{-1}: 3390 (N—H).

7-Bromo-6-methoxy-3,3-bis(trifluoromethyl)-2-azabicyclo[2.2.1]heptane (6). Compound **2b** (2.6 g) was converted in a similar way into product **6** (2.2 g, 76.7%), m.p. 54 °C (from hexane). Found (%): C, 31.67; H, 2.28; Br, 22.97; N, 4.10. $C_9H_{10}BrF_6NO$. Calculated (%): C, 31.58; H, 2.93; Br, 23.52; N, 4.09. ^{19}F NMR, δ: 66.7 (q, 3 F, J = 11.3 Hz); 72.0 (q, 3 F, J = 11.3 Hz). IR (pellets with KBr), ν/cm^{-1}: 3392 (N—H).

7-Iodo-6-methoxy-3,3-bis(trifluoromethyl)-2-azabicyclo[2.2.1]heptane (7). Compound **2c** (7.8 g) was converted in a

* In some cases, in acetone-d_6 (specially noted).

similar way into product **7** (4.7 g, 55.3%), m.p. 60 °C (from hexane). Found (%): C, 28.04; H, 1.93; I, 33.11; N, 3.90. $C_9H_{10}F_6INO$. Calculated (%): C, 27.76; H, 2.57; I, 32.65; N, 3.60. ^{19}F NMR, δ: 67.4 (q, 3 F, J = 11.3 Hz); 72.7 (q, 3 F, J = 11.3 Hz). IR (CCl_4), ν/cm^{-1}: 3383 (N—H).

7-Chloro-6-ethoxy-3,3-bis(trifluoromethyl)-2-azabicyclo[2.2.1]heptane (8). The reaction of compound **2a** (2.6 g) with 10 mL of EtOH carried out in a similar way gave 1.8 g (59%) of product **8**, m.p. 44 °C (from hexane). Found (%): C, 38.34; H, 3.19; Cl, 11.68; N, 4.42. $C_{10}H_{12}ClF_6NO$. Calculated (%): C, 38.52; H, 3.85; Cl, 11.40; N, 4.49. ^{19}F NMR, δ: 66.7 (q, 3 F, J = 11.3 Hz); 72.0 (q, 3 F, J = 11.3 Hz). IR (CCl_4), ν/cm^{-1}: 3391 (N—H).

7-Chloro-6-(2-chloroethoxy)-3,3-bis(trifluoromethyl)-2-azabicyclo[2.2.1]heptane (9). Sulfuric acid (d = 1.81, 0.5 mL) was added to a solution of compound **2a** (6.0 g, 0.023 mol) and 2-chloroethanol (7.5 g, 0.113 mol) in 25 mL of dioxane. The mixture was refluxed for 3 h and then diluted with 200 mL of H_2O. The product was extracted with CH_2Cl_2 (2×20 mL). The extract was washed with 100 mL of a 10% solution of Na_2CO_3 and dried with $CaCl_2$. The subsequent fractional distillation gave 5.2 g of compound **9** (69%), m.p. 86 °C (0.5 Torr); d_4^{20} 1.558; n_D^{25} 1.4371. Found (%): C, 34.90; H, 3.01; Cl, 21.03; N, 4.19. $C_{10}H_{11}Cl_2F_6NO$. Calculated (%): C, 34.68; H, 3.18; Cl, 20.52; N, 4.04. ^{19}F NMR, δ: 67,0 (q, 3 F, J = 11.3 Hz); 72.3 (q, 3 F, J = 11.3 Hz). IR (CCl_4), ν/cm^{-1}: 3391 (N—H).

7-Chloro-6-(2-hydroxyethoxy)-3,3-bis(trifluoromethyl)-2-azabicyclo[2.2.1]heptane (10). Sulfuric acid (d = 1.81, 0.2 mL) was added to a solution of compound **2a** (3.5 g, 0.013 mol) and ethylene glycol (2.5 g, 0.039 mol) in 10 mL of dioxane. The mixture was kept for 16 h at 20 °C and diluted with 150 mL of H_2O. The product was extracted with CH_2Cl_2 (2×10 mL). The extract was washed with 150 mL of H_2O and with 50 mL of a 10% solution of Na_2CO_3 and dried with $CaCl_2$. During the subsequent fractional distillation, the fraction boiling at 112 °C (0.5 Torr) was collected. On standing, this fraction crystallized. Recrystallization from a hexane—CCl_4 mixture (1 : 1) gave 2.3 g (53.5%) of compound **10**, m.p. 77 °C. Found (%): C, 37.59; H, 4.33; Cl, 21.26; N, 4.39. $C_{10}H_{12}ClF_6NO_2$. Calculated (%): C, 36.64; H, 3.66; Cl, 10.86; N, 4.27. ^{19}F NMR, δ: 67.5 (q, 3 F, J = 11.3 Hz); 74.0 (q, 3 F, J = 11.3 Hz). IR (pellets with KBr), ν/cm^{-1}: 3497 (O—H); 3395 (N—H).

Bis[7-chloro-3,3-bis(trifluoromethyl)-2-azabicyclo[2.2.1]hept-6-yloxy]ethane (11). Sulfuric acid (d = 1.81, 0.2 mL) was added to a solution of compound **2a** (3.2 g, 0.12 mol) and ethylene glycol (0.3 g, 0.005 mol) in 5 mL of dioxane. The mixture was refluxed for 14 h and diluted with 100 mL of H_2O. The product was extracted with CH_2Cl_2 (2×20 mL). The extract was washed with 150 mL of a 10% solution of Na_2CO_3 and dried with $CaCl_2$. Then the extract was concentrated, and the residue was twice recrystallized from CCl_4 to give 1.6 g (44.6%) of compound **11**, m.p. 93 °C. Found (%): C, 37.70; H, 3.64; Cl, 12.15; N, 4.80. $C_{18}H_{18}Cl_2F_{12}N_2O_2$. Calculated (%): C, 36.42; H, 3.04; Cl, 11.97; N, 4.72. ^1H NMR (CD_3COCD_3), δ: 2.47 (s, 2 H, HC(4)); 1.83 (m, 4 H, HC(5)); 3.43 (m, 2 H, HC(6)); 4.10 (s, 2 H, HC(7)); 3.13—3.43 (group of signals, 8 H, CH_2CH_2, HC(1), HC′(1), NH, N′H). ^{19}F NMR (CD_3COCD_3), δ: 67.7 (q, 3 F, J = 11.3 Hz); 73.0 (q, 3 F, J = 11.3 Hz). IR (pellets with KBr), ν/cm^{-1}: 3384 (N—H).

7-Chloro-3,3-bis(trifluoromethyl)-2-azabicyclo[2.2.1]hept-6-yl acetate (12). Sulfuric acid (d = 1.81, 0.2 mL) was added to a solution of compound **2a** (5.4 g, 0.02 mol) in 10 mL of AcOH. The solution was stirred for 2 h and diluted with 100 mL of H_2O. Then the product was extracted with CH_2Cl_2 (2×20 mL), and the extract was washed with 50 mL of a 10% solution of Na_2CO_3, dried with $CaCl_2$, and concentrated. The residue was recrystallized from hexane to give 4.9 g (74%) of compound **12**, m.p. 95 °C. Found (%): C, 36.46; H, 2.49; Cl, 11.12; N, 4.22. $C_{10}H_{10}ClF_6NO_2$. Calculated (%): C, 36.87; H, 3.07; Cl, 10.91; N, 4.30. ^{19}F NMR, δ: 66.0 (q, 3 F, J = 11.3 Hz); 72.0 (q, 3 F, J = 11.3 Hz). IR (CCl_4), ν/cm^{-1}: 3393 (N—H); 1741 (C=O).

7-Bromo-3,3-bis(trifluoromethyl)-2-azabicyclo[2.2.1]hept-6-yl acetate (13). Compound **2b** (2.0 g, 0.0065 mol) was converted in a similar way into product **13** (1.8 g, 75.4%), m.p. 96 °C. Found (%): C, 32.63; H, 2.19; Br, 21.81; N, 4.25. $C_{10}H_{10}BrF_6NO_2$. Calculated (%): C, 32.43; H, 2.70; Br, 21.62; N, 3.78. ^{19}F NMR, δ: 68.3 (q, 3 F, J = 11.3 Hz); 74.0 (q, 3 F, J = 11.3 Hz). IR ($CHCl_3$), ν/cm^{-1}: 3392 (N—H); 1730 (C=O).

7-Iodo-3,3-bis(trifluoromethyl)-2-azabicyclo[2.2.1]hept-6-yl acetate (14). Compound **2c** (2.0 g, 0.0056 mol) was converted in a similar way into product **14** (1.6 g, 68.5%), m.p. 87 °C. Found (%): C, 28.78; H, 2.19; I, 27.39; N, 3.36. $C_{10}H_{10}F_6INO_2$. Calculated (%): C, 28.78; H, 2.40; I, 30.43; N, 3.36. ^{19}F NMR, δ: 66.3 (q, 3 F, J = 11.3 Hz); 72.0 (q, 3 F, J = 11.3 Hz). IR (CCl_4), ν/cm^{-1}: 3386 (N—H); 1741 (C=O).

6-Acetoxy-5-chloromercuro-3,3-bis(trifluoromethyl)-2-azabicyclo[2.2.1]heptane (15). A mixture of 3,3-bis(trifluoromethyl)-2-azabicyclo[2.2.1]hept-5-ene (10.9 g) prepared by a known procedure[3] and mercuric acetate (15.0 g) in 50 mL of AcOH was stirred for 2 h at 20—25 °C and for 1 h at 80—85 °C. The resulting mixture was diluted with 200 mL of brine and stirred for 2 h. The precipitate was filtered off, washed with 200 mL of H_2O, dried a little on the filter in air, washed with 100 mL of CCl_4, and kept *in vacuo* (1 Torr) to give compound **15** (22.9 g, 92.8%), m.p. 172—174 °C (decomp.). Found (%): C, 22.72; H, 1.55; N, 2.97. $C_{10}H_{10}F_6HgNO_2$. Calculated (%): C, 22.77; H, 1.90; N, 2.66. ^1H NMR (CD_3COCD_3), δ: 3.40 (s, 1 H, HC(1)); 3.20 (s, 1 H, NH); 2.93 (s, 1 H, HC(4)); 3.0 (d, 1 H, HC(5) or HC(6), J = 7 Hz); 4.80 (d, 1 H, HC(6) or HC(5), J = 7 Hz); 1.83 (s, 2 H, HC(7)); 1.83 (s, 3 H, CH_3). ^{19}F NMR (CD_3COCD_3), δ: 65.1 (q, 3 F, J = 11.3 Hz); 72.7 (q, 3 F, J = 11.3 Hz).

6-Acetoxy-5-bromo-3,3-bis(trifluoromethyl)-2-azabicyclo[2.2.1]heptane (16). Bromine (1.6 g) was added to a suspension of compound **15** (2.0 g) in 20 mL of CCl_4, and the resulting mixture was stirred for 7 h and filtered. The filtrate was distilled. The fraction boiling at 84—85 °C (0.5 Torr) was collected; it crystallized on standing. Recrystallization from hexane gave 0.8 g (58%) of compound **16**, m.p. 96 °C. Found (%): C, 32.49; H, 2.51; Br, 21.98; N, 4.05. $C_{10}H_{10}BrF_6NO_2$. Calculated (%): C, 32.43; H, 2.70; Br, 21.62; N, 3.78. ^1H NMR (CD_3COCD_3), δ: 3.13 (m, 2 H, NH, HC(1)); 2.67 (s, 1 H, HC(4)); 4.33 (m, 2 H, HC(5), HC(6)); 1.83 (s, 2 H, HC(7)); 1.83 (s, 3 H, CH_3). ^{19}F NMR (CD_3COCD_3), δ: 65.0 (q, 3 F, J = 11.3 Hz); 73.0 (q, 3 F, J = 11.3 Hz).

6-Acetoxy-5-iodo-3,3-bis(trifluoromethyl)-2-azabicyclo[2.2.1]heptane (17). Iodine (3.1 g, 12.2 mmol) was added to a solution of compound **15** (6.4 g, 12.2 mmol) in 50 mL of CH_2Cl_2. The solution was refluxed with stirring for 5 h. Then the precipitate of HgI_2 was filtered off, and the filtrate was washed with 100 mL of a 10% aqueous solution of KI and then with 100 mL of a 10% aqueous solution of $Na_2S_2O_3$; the solution in CH_2Cl_2 was dried with $CaCl_2$. After evaporation of

the solvent, the residue was recrystallized from hexane to give 4.1 g (80.9%) of compound **17**, m.p. 72 °C. Found (%): C, 28.97; H, 2.20; N, 3.48; I, 27.89. $C_{10}H_{10}F_6INO_2$. Calculated (%): C, 28.78; H, 2.40; N, 3.36; I, 30.43. ^1H NMR (CCl_4), δ: 2.20 (s, 4 H, CH_3, NH); 2.38 (s, 2 H, HC(7)); 3.18 (s, 1 H, HC(4)); 3.5 (s, 1 H, HC(1)); 4.53 (d, 1 H, HC(5), J = 6 Hz); 4.87 (d, 1 H, HC(5), J = 6 Hz); 4.87 (d, 1 H, HC(6), J = 6 Hz). ^{19}F NMR (CCl_4), δ: 65.0 (q, 3 F, J = 11.3 Hz); 72.3 (q, 3 F, J = 11.3 Hz). IR (CCl_4), ν/cm^{-1}: 3390 (N—H); 1750 (C=O).

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