Interaction of perfluoropent-2-ene and its 2-amino-4-imino derivative with ethylenediamine and with diethylenetriamine

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(Received June 8, 1993; accepted November 5, 1993)

Abstract

5,7-Trifluoromethyl-6-fluoro-2,3-dihydro-1*H*-1,4-diazepine has been prepared by the reaction of perfluoropent-2-ene (or 2-amino-4-iminoperfluoropent-2-ene) with ethylenediamine. Both perfluoropent-2-ene and 2-amino-4-iminoperfluoropent-2-ene were found to react with diethylenetriamine to form 1,9-trifluoromethyl-3,4,6,7-tetrahydro-2*H*-pyrazino[1,2-*a*]pyrazine (established by X-ray study). Some salts and the complex with BF_3 of this bicyclic compound are described.

Introduction

It is well known that perfluoropent-2-ene reacts with ammonia to give 2-amino-4-iminoperfluoropent-2-ene [1]. This 1,3-amino vinylimine is an azo analogue of heptafluoroacetylacetone although it could not be synthesized by the direct interaction of fluoroalkyl-containing 1,3-diketones with amines. It seems to us that the reactivity of 2-amino-4-iminoperfluoropent-2-ene may be comparable with that of fluoroalkyl-containing 1,3-amino vinyl ketones, particularly in transamination reactions. These reactions are well known for fluorinated 1,3-amino vinyl ketones [2, 3]. In our view both perfluoropent-2-ene and its 2-amino-4-imino derivative may serve as key precursors to a variety of novel heterocycles.

This paper describes the interaction of perfluoropent-2-ene and 2-amino-4-iminoperfluoropent-2-ene with ethylenediamine and diethylenetriamine.

Experimental

Melting points were measured in open capillaries and are reported uncorrected. Infrared spectra were measured on a Specord 75 IR spectrometer. ^{1}H NMR spectra were recorded on a Tesla BS-567A instrument (^{1}H: 100 MHz) in CD_3COCD_3 using TMS as an internal standard. ^{19}F NMR spectra were recorded on a Tesla BS-587A instrument (^{19}F: 75 MHz) in CD_3COCD_3 using $CFCl_3$ as an internal standard. All chemical shifts are reported in ppm and wavenumbers in cm^{-1}. Mass spectral data were obtained using a MAT-311a mass spectrometer. Column chromatography was performed on silica gel L 100/250. Thin-layer chromatography was performed on 'Silufol-UV-254' plates in methanol.

X-Ray crystallographic study of compound 6

Crystal data: M = 275.20, monoclinic, $a = 14.116(4)$, $b = 10.128(3)$, $c = 7.699(2)$, $\beta = 103.54(3)°$, $V = 1070.1(9)$ Å^3, $P2_1/a$, $D_x = 1.708$ g cm^{-3}, $Z = 4$, $F(000) = 560.0$, λ (Mo Kα) = 0.71069 Å, μ (Mo Kα) = 1.96 cm^{-1}.

A prismatic crystal (0.1 × 0.1 × 0.2 mm) was selected and mounted on a Philips PW-1100 diffractometer. Unit cell parameters were determined from the automatic centring of 25 reflections ($8 \leqslant \theta \leqslant 16°$) and refined by the least-squares method. Intensities were collected using graphite monochromatized Mo Kα radiation employing the $\omega/2\theta$ scan technique. 2748 reflections were measured in the range $2 \leqslant \theta \leqslant 30°$, of which 1608 were considered as observed according to the condition $I \geqslant 2.5\sigma(I)$. Three reflections were measured every 2 h as a means of orientation and intensity control, but no significant intensity decay was observed. Lorentz polarization, but no absorption, corrections were made.

The structure was solved by direct methods using a SHELXS computer program [4] and refined by the full-matrix least-squares method using the SHELX-76 computer program [5]. The function minimized was

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SSDI 0022-1139(93)03055-Q

$\Sigma w[|F_o| - |F_c|]^2$, where $w = \sigma^{-2}(F_o)$. The quantities f, f' and f'' were taken from *International Tables for X-ray Crystallography* [6]. Two fluorine atoms were located in disordered positions and an occupancy factor of 0.5 was assigned according to the peak heights arising from difference synthesis. The position of all the H atoms was computed and refined with an overall isotropic temperature factor using a riding model. The final R factor was 0.061 ($R_w = 0.061$) for all observed reflections. The number of refined parameters was 182. Maximum shift/e.s.d = 0.08, with the maximum and minimum peaks in the final difference synthesis being 0.3 and −0.2 e $Å^{-3}$, respectively.

TABLE 1. Bond lengths (Å) and angles (°) for compound **6**

C(8)–F(1)	1.346(5)	C(5)–N(3)	1.368(5)
C(8)–F(2)	1.317(6)	C(6)–N(3)	1.434(6)
C(8)–F(3)	1.420(23)	C(2)–C(1)	1.536(7)
C(9)–F(4)	1.307(6)	C(4)–C(3)	1.462(5)
C(9)–F(5)	1.353(12)	C(8)–C(3)	1.504(5)
C(9)–F(6)	1.375(5)	C(5)–C(4)	1.384(5)
C(1)–N(1)	1.469(5)	C(9)–C(5)	1.472(6)
C(4)–N(1)	1.403(5)	C(7)–C(6)	1.542(6)
C(7)–N(1)	1.459(5)	F(3)′–C(8)	1.297(16)
C(2)–N(2)	1.499(5)	F(5)′–C(9)	1.355(17)
C(3)–N(2)	1.269(5)		
C(4)–N(1)–C(1)	116.4(3)	F(2)–C(8)–F(1)	104.8(4)
C(7)–N(1)–C(1)	113.3(3)	F(3)–C(8)–F(1)	101.2(11)
C(7)–N(1)–C(4)	115.8(3)	F(3)–C(8)–F(2)	119.1(10)
C(3)–N(2)–C(2)	113.5(3)	C(3)–C(8)–F(1)	114.6(3)
C(6)–N(3)–C(5)	118.7(3)	C(3)–C(8)–F(2)	114.4(3)
C(2)–C(1)–N(1)	110.4(4)	C(3)–C(8)–F(3)	102.4(9)
C(1)–C(2)–N(2)	107.4(3)	F(3)′–C(8)–F(1)	106.2(8)
C(4)–C(3)–N(2)	123.7(4)	F(3)′–C(8)–F(2)	102.4(10)
C(8)–C(3)–N(2)	111.7(3)	F(3)′–C(8)–C(3)	113.3(7)
C(8)–C(3)–C(4)	123.8(3)	F(5)–C(9)–F(4)	108.2(7)
C(3)–C(4)–N(1)	113.8(3)	F(6)–C(9)–F(4)	106.8(4)
C(5)–C(4)–N(1)	118.4(3)	F(6)–C(9)–F(5)	103.1(8)
C(5)–C(4)–C(3)	127.8(4)	C(5)–C(9)–F(4)	116.4(3)
C(4)–C(5)–N(3)	122.3(4)	C(5)–C(9)–F(5)	109.5(7)
C(9)–C(5)–N(3)	113.9(3)	C(5)–C(9)–F(6)	111.9(4)
C(9)–C(5)–C(4)	123.3(4)	F(5)′–C(9)–F(4)	104.9(9)
C(7)–C(6)–N(3)	109.9(4)	F(5)′–C(9)–F(6)	103.8(9)
C(6)–C(7)–N(1)	106.6(3)	F(5)′–C(9)–C(5)	112.1(9)

TABLE 2. Final hydrogen coordinates (×10⁴) for compound **6**

	x/a	*y/b*	*z/c*
H(1)	8019(3)	5557(5)	7771(6)
H(1)A	8011(3)	4075(5)	6555(6)
H(2)	6863(4)	5674(4)	4778(6)
H(2)A	6248(4)	5811(4)	6518(6)
H(4)	5656(3)	3225(3)	8430(5)
H(6)	7719(3)	4042(5)	13148(6)
H(6)A	7960(3)	2742(5)	11789(6)
H(7)	8414(3)	4878(4)	10648(6)
H(7)A	7224(3)	5482(4)	10482(6)

TABLE 3. Final atomic coordinates (×10⁴) for compound **6**

	x/a	*y/b*	*z/c*
F(1)	5396(2)	1015(2)	6942(4)
F(2)	4658(3)	2003(3)	4602(5)
F(3)	6288(18)	1278(22)	5073(33)
F(4)	4379(2)	2965(3)	7983(4)
F(5)	4590(9)	3480(17)	10764(16)
F(6)	4838(3)	1498(3)	10031(5)
N(1)	7332(2)	3966(3)	8688(4)
N(2)	6084(3)	3952(3)	5198(4)
N(3)	6508(3)	3203(4)	11490(4)
C(1)	7591(3)	4704(5)	7223(6)
C(2)	6667(4)	5161(4)	5874(6)
C(3)	5977(3)	3176(4)	6432(5)
C(4)	6394(3)	3415(3)	8330(5)
C(5)	5986(3)	3130(4)	9755(5)
C(6)	7511(3)	3598(5)	11838(6)
C(7)	7655(3)	4608(4)	10424(6)
C(8)	5523(4)	1888(4)	5696(6)
C(9)	4954(3)	2787(4)	9564(6)
F(5)′	4540(14)	3492(21)	10689(26)
F(3)′	6016(15)	1276(15)	4718(24)

Preparation of compounds 1 and 2

Perfluoropent-2-ene (**1**) was prepared by the method described previously [7] and was found to contain *trans* and *cis* isomers in a ratio of 81:19.

2-Amino-4-iminoperfluoropent-2-ene (**2**) was prepared according to the literature method [1].

Synthesis of 2,3-dihydro-6-fluoro-5,7-trifluoromethyl-1H-1,4-diazepine (3) (nc)

Method A

To the solution of ethylenediamine (15.0 g, 250 mmol) in 250 ml of CH_2Cl_2 was added compound **1** (25.0 g, 100 mmol) over a 30 min period under stirring and cooling at 0 °C. The mixture was stirred for 24 h at 20 °C when the precipitated en·2HF was filtered off. Removal of the solvent gave compound **3** as a crude product which was dissolved in 20 ml of methanol. Pure **3** (18.5 g, 74%) (m.p. 98–99 °C) was obtained by precipitation through the addition of water.

^{1}H NMR ($CDCl_3$) δ: 2.26 (2H, m, CH_2); 3.06 (2H, m, CH_2); 4.34 (1H, w s, NH) ppm. ^{19}F NMR ($CDCl_3$) δ: −66.03 (3F, d, CF_3, J(F−F) = 19.5 Hz); −69.33 (3F, d, CF_3, J(F−F) = 19.5 Hz); −159.61 (1F, m, CF, J(F−F) = 19.5 Hz) ppm. IR (cm^{-1}): 3200, 1580, 1530 (NH); 1710, 1640 (C=N, C=C). MS *m/e* (relative intensity, ion): 250 (100.0%, M); 231 (19.2%, M−F); 222 (99.2%, M−C_2H_4); 202 (47.8%, M−HF, C_2H_4); 153 (16.0, M−CF_3, C_2H_2); 69 (42.3%, CF_3). Analysis: Found: C, 33.70; H, 2.48; F, 53.16; N, 10.78%. Calc. for $C_7H_5F_7N_2$: C, 33.61; H, 2.07; F, 53.17; N, 11.20%.

Method B

A mixture of anhydrous ethylenediamine (0.3 g, 5 mmol) and **2** (1.1 g, 5 mmol) in 50 ml of anhydrous alcohol was heated under reflux for 16 h. The solvent was removed under reduced pressure and the residue recrystallized from hexane to give 0.8 g (65%) of compound **3** (m.p. 98–99 °C). Analysis: Found: C, 33.65; H, 2.31; F, 53.10; N, 10.95%. Calc. for $C_7H_5F_7N_2$: C, 33.61; H, 2.07; F, 53.17; N, 11.20%. The physicochemical data were identical to those listed above.

Reaction of 2,3-dihydro-6-fluoro-5,7-trifluoromethyl-1H-1,4-diazepine (3) with diethylenetriamine

A mixture of anhydrous diethylenetriamine (2.5 g, 25 mmol) and **3** (1.25 g, 5 mmol) in 30 ml of chloroform was refluxed for 0.5 h when the precipitated dien·3HF was filtered off. After removal of the solvent from the residue using column chromatography (chloroform as the eluant), followed by recrystallization from hexane, 1.0 g (86%) of compound **4** (m.p. 109–110 °C) was obtained. Analysis: Found: C, 36.11; H, 2.73; F, 49.03; N, 12.53%. Calc. for $C_7H_6F_6N_2$: C, 36.22; H, 2.60; F, 49.11; N, 12.07%. The ^{1}H NMR and IR spectra were identical to those reported previously [3, 8].

Synthesis of 1,9-trifluoromethyl-3,4,6,7-tetrahydro-2H-pyrazino[1,2-a]pyrazine (6) (nc)

Method A

To a solution of diethylenetriamine (24.0 g, 230 mmol) in 250 ml of CH_2Cl_2 was added compound **1** (25.0 g, 100 mmol) over 0.5 h with cooling at 0 °C. The mixture was stirred for 24 h at 20 °C when the precipitated dien·3HF was filtered off. After removal of solvent from the residue using column chromatography (methanol as the eluant), recrystallization from CCl_4 gave 6.9 g (25%) of compound **6** as orange coloured crystals (m.p. 129–130 °C). ^{1}H NMR ($CDCl_3$) δ: 3.10 (4H, m, CH_2-N-CH_2); 3.42 (2H, m, CH_2-NH); 3.76 (2H, m, $CH_2=N$); 1.64 (1 H, w s, NH) ppm. ^{19}F NMR δ: −63.1 (3F, q, CF_3, J(F−F) = 11 Hz); −64.4 (3F, q, CF_3, J(F−F) = 11 Hz) ppm. IR (cm^{-1}): 3280, 1580 (NH); 1600 (C=N, C=C). MS *m/e* (relative intensity, ion): 273 (100%, M); 254 (11.5%, M−F); 245 (6.9%, M−C_2H_4); 244 (8.0%, M−CH_2NH); 204 (19.8%, M−CF_3); 69 (6.4%, CF_3). Analysis: Found: C, 39.03; H, 4.03; F, 41.79; N, 14.81%. Calc. for $C_9H_9F_6N_3$: C, 39.28; H, 4.03; F, 41.42; N, 15.27%.

Method B

A mixture of anhydrous diethylenetriamine (0.5 g, 5 mmol) and compound **2** (1.1 g, 5 mmol) in 50 ml of anhydrous alcohol was heated under reflux for 24 h. The solvent was then removed under reduced pressure. From the residue using column chromatography (CH_2Cl_2 as the eluant) with recrystallization from CCl_4, 0.6 g (44%) of compound **6** (m.p. 129–130 °C) was obtained. Analysis: Found: C, 39.14; H, 3.65; F, 41.83; N, 14.97%. Calc. for $C_9H_9F_6N_3$: C, 39.28; H, 4.03; F, 41.42; N, 15.27%. The physicochemical data were identical to those listed above.

Synthesis of 1,9-trifluoromethyl-3,4,6,7-tetrahydro-2H-pyrazino[1,2-a]pyrazine monohydrochloride (7) (nc)

Hydrogen chloride was bubbled into a solution of compound **6** (1.37 g, 5 mmol) in 50 ml of ether. The resulting precipitate was collected by filtration and washed with ether and dried under reduced pressure to give 1.3 g (84%) of compound **7** (m.p. 120 °C decomp.) as dark red crystals. ^{1}H NMR δ: 3.50 (4H, t, CH_2-N-CH_2, J(H−H) = 4.4 Hz); 3.96 (4H, t, $CH_2=N^+$, J(H−H) = 4.4 Hz); 1.3 (1H, m, NH or CH) ppm. ^{19}F NMR δ: −63.6 (6F, s, $2CF_3$) ppm. IR (cm^{-1}): 3300, 1585 (NH); 2700 ($C=N^+H$); 1610 (C=C); 720 (Cl). Analysis: Found: C, 34.70; H, 3.61; F, 36.52; Cl, 11.64; N, 13.48%. Calc. for $C_9H_9F_6N_3 \cdot HCl$: C, 34.91; H, 3.26; F, 36.81; Cl, 11.45; N, 13.57%.

Synthesis of 1,9-trifluoromethyl-3,4,6,7-tetrahydro-2H-pyrazino[1,2-a]pyrazine hexafluorophosphate (8) (nc)

To a solution of compound **6** (1.37 g, 5 mmol) in 30 ml of methanol was added a solution of $NaPF_6$ (1.68 g, 10 mmol) saturated with gaseous HCl in 20 ml of methanol. The resulting precipitate was collected and washed with chloroform to give 1.7 g (81%) of compound **8** (m.p. 198–199 °C) as red crystals. ^{1}H NMR δ: 3.02 (4H, t, CH_2-N-CH_2, J(H−H) = 4.6 Hz); 3.48 (4H, t, $CH_2=N^+$, J(H−H) = 4.6 Hz); 1.20 (1H, m, NH or CH) ppm. ^{19}F NMR δ: −63.60 (6F, s, $2CF_3$); −74.70 (6F, s, PF_6) ppm. IR (cm^{-1}): 3630, 3550, 1520 (NH); 2650 ($C=N^+H$); 1640, 1540 (C=C); 530, 550 (PF_6). Analysis: Found: C, 26.04; H, 2.61; F, 53.96; N, 10.24%. Calc. for $C_9H_9F_6N_3 \cdot HPF_6$: C, 25.79; H, 2.41; F, 54.39; N, 10.02%.

Synthesis of 1,9-trifluoromethyl-3,4,6,7-tetrahydro-2H-pyrazino[1,2-a]pyrazine trifluoroborane (9) (nc)

To a solution of compound **6** (1.37 g, 5 mmol) in 50 ml of ether was added $BF_3 \cdot O(Et)_2$ until violet crystals precipitated. The resulting precipitate was collected and washed with ether and then dried under reduced pressure to give 1.2 g (88%) of compound **9** (m.p. 178–180 °C). ^{1}H NMR δ: 3.10 (4H, t, CH_2-N-CH_2, J(H−H) = 4.6 Hz); 3.60 (4H, t, $CH_2=N^+$, J(H−H) = 4.6 Hz) ppm. ^{19}F NMR δ: −60.30 (6F, s, $2CF_3$); −158.20 (3F, s, BF_3) ppm. IR (cm^{-1}): 3280; 1520 (NH); 1540 (sh), 3050 (C=C); 1000–1060 (BF_3). MS *m/e* (relative intensity, ion): 273 (100%, M); 258 (25%, M−F); 254 (19%, M−BF_3, F); 245 (62%, M−C_2H_4, BF_3); 244 (24%, M−BF_3, CH_2NH); 233 (53%, M−BF_3, $CH_2-C\equiv N$); 204 (92%, M−BF_3, CF_3); 202 (78%, M−BF_3, $H_2C=NCH_2CH_2-NH$); 184 (18%, M−BF_3, CF_3, HF); 175 (38%, M−BF_3, CF_3, CH_2-NH); 136

(50%, M−BF_3, CF_3−CH=CHN−CH_2−CH=N); 96 (42%, HC=N−CH_2CH_2−N=CH−CH_2); 69 (90%, CF_3). Analysis: Found: C, 31.39; H, 2.91; F, 50.21; N, 12.52%. Calc. for $C_9H_9F_6N_3 \cdot BF_3$: C, 31.70; H, 2.66; F, 50.14; N, 12.32%.

Results and discussion

Reaction of perfluoropent-2-ene and 2-amino-3-imino-perfluoropent-2-ene with ethylenediamine

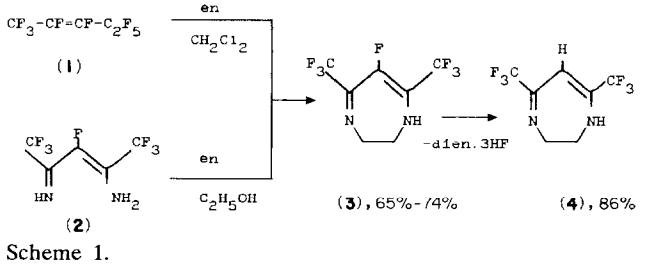
In the present work, it has been found that perfluoropent-2-ene (**1**) forms 2,3-dihydro-6-fluoro-5,7-trifluoromethyl-1*H*-1,4-diazepine (3) by reaction with ethylenediamine (Scheme 1). A transamination reaction of 2-amino-4-iminoperfluoropent-2-ene (**2**) with ethylenediamine also gives the same compound (**3**) (Scheme 1).

The ^{1}H NMR spectrum of **3** exhibited only methylenic protons corresponding to ethylenediamine and one proton of a secondary amine. In the ^{19}F NMR spectrum of compound **3**, two doublets at δ −66.03 and −69.33 ppm with a coupling of 19.5 Hz were attributed to the fluorine atoms of the non-equivalent CF_3 groups. A multiplet at δ −159.64 ppm in the expected ratio was found for the C-6 fluorine atom of **3**.

Heating compound **3** with diethylenetriamine (which is a stronger base than ethylenediamine [9, 10]) in $CHCl_3$ did not lead to a further transamination reaction, but gave the known 1,4-diazepine (**4**) (Scheme 1). Compound **4** may result from the nucleophilic substitution of the vinylic fluorine of **3** (by analogy with previously reported reactions of perfluoro-1-methylcyclopentene with amines [11]), followed by transformation of the unstable intermediate. 2-Chloro- and 2,2-dibromo-polyfluoroacylacetic esters [12, 13] were similarly transformed into 2-unsubstituted polyfluoroacylacetic esters.

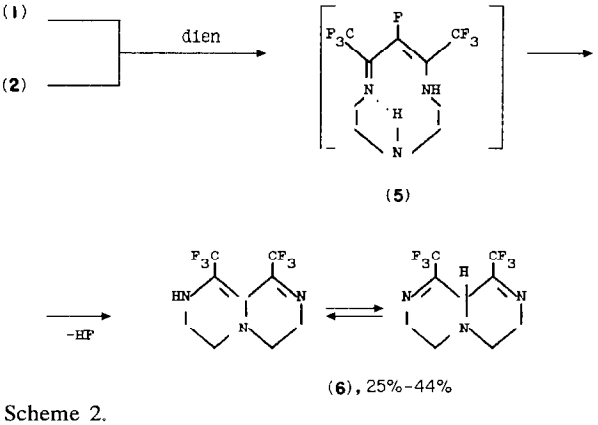
Reaction of perfluoropent-2-ene and its 2-amino-4-imino derivative with diethylenetriamine

Interaction of perfluoropent-2-ene (**1**) with diethylenetriamine leads to 1,9-trifluoromethyl-3,4,6,7-tetrahydro-2*H*-pyrazino[1,2-*a*]pyrazine (**6**), similar to that produced by the transamination reaction of 2-amino-4-iminoperfluoropent-2-ene (**2**) with diethylenetriamine. This obviously occurs through the formation of intermediate **5** (Scheme 2). Structure **6** may result from the intramolecular nucleophilic substitution of the labile fluorine atom of the macrocyclic compound **5**.



Scheme 1.

To support the structure assigned to **6**, this compound was subjected to single-crystal X-ray analysis. The structure of compound **6**, as determined by this study, is shown in Fig. 1. Both amino-imine and imino-imine isomers are possible in structure **6**. X-Ray crystallographic analysis confirmed the existence of the imino-imine form in structure **6** in the solid state. However, the infrared spectrum of this compound displayed typical NH stretching absorption at 3280 cm^{-1} (s) attributable to the amino-imine form. Consequently, in the ^{1}H NMR spectrum of compound **6** three multiple resonance signals in the expected ratio were attributed to the methylene protons as follows: CH_2−N−CH_2 at δ 3.10 ppm; CH_2−NH at δ 3.42 ppm; and CH_2=N at δ 3.76 ppm. The last two signals were transformed into a singlet when CD_3COOD was added to the system. In this case, the ^{1}H NMR spectrum of compound **6** ex-



Scheme 2.

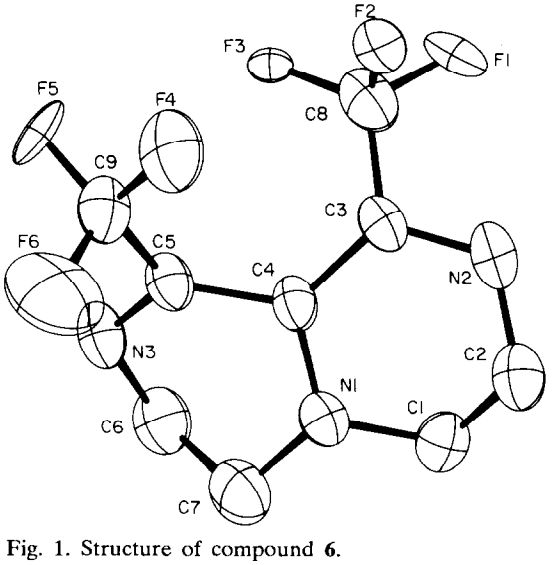
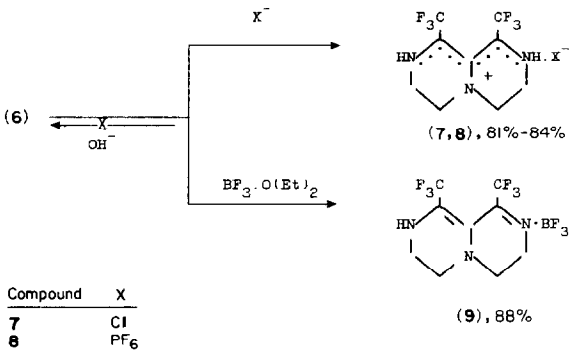


Fig. 1. Structure of compound **6**.



Compound	X
7	Cl
8	PF_6

Scheme 3.

hibited two equivalent signals as quartets at δ 3.10 and 3.60 ppm, respectively. The ^{19}F NMR spectrum of product **6** showed two equivalent quartets of fluorine atoms for the CF_3 groups, which also transformed to a singlet when CD_3COOD was added.

Compounds **7–9** were derived directly from heterocycle **6** under anhydrous conditions, since they are destroyed by water (Scheme 3). Of these, the most unstable is compound **7**; it decomposed when stored at room temperature for 3 weeks. The most stable was complex **9**.

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