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## (-)-(S)-2-(Fluorodiphenylmethyl)-pyrrolidinium chloride

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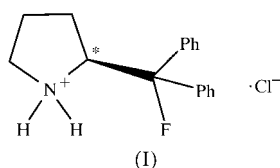
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The title compound,  $C_{17}H_{19}FN^+ \cdot Cl^-$ , has an ionic structure, and cations and anions are linked into infinite chains by  $Cl \cdots H-N-H \cdots Cl$  hydrogen bonds. The absolute configuration (S) was confirmed.

### Comment

Homochiral amines are valuable as auxiliaries in enantioselective C–C bond-forming reactions (Morrison, 1983) and as chiral resolving agents (Parker, 1991). (-)-(S)-2-(Fluorodiphenylmethyl)pyrrolidine, (II), was prepared and studied (Bailey *et al.*, 1997; Tavasli, 1999; O'Hagan *et al.*, 2000) as a potential auxiliary in asymmetric alkylation reactions and as a chiral solvating agent for  $^1H$  NMR analysis of the enantiomeric purity of chiral carboxylic acids and alcohols. In the course of this work, we studied the structure of the title compound, (I), the hydrochloride salt of (II).



The five-membered heterocycle adopts an envelope conformation; the N, C2, C4 and C5 atoms are coplanar within experimental error, with C3 deviating from their mean plane by 0.60 Å. The fluorodiphenylmethyl substituent is in an equatorial orientation. The conformation around the C1–C2 bond is staggered, with the F atom directed between N and C3. Cations and anions are linked by  $N-H \cdots Cl$  hydrogen bonds into an infinite chain, spiralling around the  $2_1$  axis ( $x, \frac{3}{4} - y, \frac{1}{2} - z$ ).

Although the F–C1–C2–N torsion angle of  $-63.9$  (1)° brings the H2N atom close to the F atom, the  $F \cdots H2N$  distance of 2.53 (2) Å and a narrow N–H $\cdots$ F angle of 104 (2)° are indicative of a slightly shortened van der Waals

contact (Howard *et al.*, 1996) rather than an intramolecular hydrogen bond. Genuine intermolecular N–H $\cdots$ F–C bonds (which are extremely rare) display H $\cdots$ F distances from 2.10 to 2.33 Å (Dunitz & Taylor, 1997) and N–H $\cdots$ F arrangements much closer to linear.

### Experimental

Enantiomerically pure (S)-2-(fluorodiphenylmethyl)pyrrolidine, (II), was prepared (from the corresponding oxazolidinone) and characterized spectroscopically by O'Hagan *et al.* (2000). Optical rotation was measured on an Optical Activity Ltd AA-10 automatic polarimeter;  $[\alpha]_D^{25} = -0.81$  cm<sup>2</sup> g<sup>-1</sup> (MeOH, *c* 7.4). Salt (I) formed readily as a white precipitate on bubbling gaseous HCl through a solution of (II) in diethyl ether (Tavasli, 1999). Crystals of X-ray quality were obtained by recrystallization from 2-propanol.

#### Crystal data

$C_{17}H_{19}FN^+ \cdot Cl^-$

$M_r = 291.78$

Orthorhombic,  $P2_12_12_1$

$a = 7.415$  (3) Å

$b = 8.353$  (4) Å

$c = 24.406$  (10) Å

$V = 1511.6$  (11) Å<sup>3</sup>

$Z = 4$

$D_x = 1.282$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation

Cell parameters from 840

reflections

$\theta = 12.0$ – $26.4^\circ$

$\mu = 0.253$  mm<sup>-1</sup>

$T = 120$  (2) K

Plate, colourless

$0.58 \times 0.45 \times 0.14$  mm

#### Data collection

SMART 1 K CCD area-detector diffractometer

$\omega$  scans

Absorption correction: multi-scan

(SADABS; Sheldrick, 1998);

$R_{int} = 0.030$  before correction;

$T_{min} = 0.847$ ,  $T_{max} = 0.962$

14 327 measured reflections

4007 independent reflections

3844 reflections with  $I > 2\sigma(I)$

$R_{int} = 0.020$

$\theta_{max} = 29.14^\circ$

$h = -9 \rightarrow 10$

$k = -11 \rightarrow 10$

$l = -33 \rightarrow 33$

Intensity decay: none

#### Refinement

Refinement on  $F^2$

$R[F^2 > 2\sigma(F^2)] = 0.027$

$wR(F^2) = 0.067$

$S = 1.079$

4007 reflections

257 parameters

All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0417P)^2 + 0.1226P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.003$

$\Delta\rho_{max} = 0.29$  e Å<sup>-3</sup>

$\Delta\rho_{min} = -0.19$  e Å<sup>-3</sup>

Absolute structure: Flack (1983)

Flack parameter =  $-0.01$  (4)

**Table 1**

Selected geometric parameters (Å, °).

F–C1	1.4226 (13)	C1–C2	1.5349 (15)
N–C2	1.5093 (15)	C2–C3	1.5267 (17)
N–C5	1.5159 (15)	C3–C4	1.5398 (18)
C1–C11	1.5310 (16)	C4–C5	1.5274 (18)
C1–C21	1.5335 (17)		
C2–N–C5	107.87 (9)	N–C2–C3	103.45 (9)
F–C1–C11	106.90 (9)	N–C2–C1	110.78 (9)
F–C1–C21	107.91 (9)	C3–C2–C1	116.89 (10)
C11–C1–C21	111.50 (9)	C2–C3–C4	102.77 (9)
F–C1–C2	106.43 (8)	C5–C4–C3	104.17 (9)
C11–C1–C2	112.40 (9)	N–C5–C4	105.80 (9)
C21–C1–C2	111.36 (9)		
F–C1–C2–N	$-63.89$ (11)	F–C1–C11–C16	38.68 (14)
F–C1–C2–C3	54.23 (12)	F–C1–C21–C26	$-5.33$ (14)

**Table 2**

Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N-H1N $\cdots$ Cl	0.897 (18)	2.196 (17)	3.0664 (17)	163.4 (14)
N-H2N $\cdots$ Cl <sup>i</sup>	0.886 (17)	2.229 (17)	3.0712 (16)	158.9 (14)

Symmetry code: (i)  $x - \frac{1}{2}, \frac{3}{2} - y, 1 - z$ .

2337 symmetrically independent reflections and 1670 Friedel equivalents were measured [respectively, 2252 and 1592 with  $I > 2\sigma(I)$ ]. All H atoms were refined in isotropic approximation; bond distances Csp<sup>2</sup>-H = 0.93 (2)-1.01 (2) Å and Csp<sup>3</sup>-H = 0.94 (2)-0.98 (2) Å.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SMART*; data reduction: *SAINTE* (Bruker, 1999); program(s) used to solve structure: *SHELXTL* (Bruker, 1997); program(s) used to refine structure: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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