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(1*S*,5*R*)-1-(4-Fluorophenyl)-3-azoniabicyclo[3.1.0]hexane chloride

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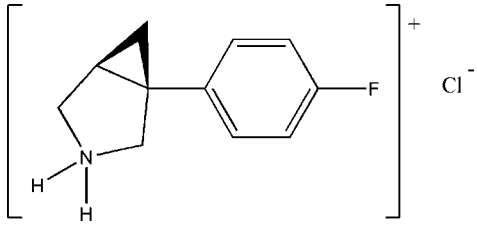
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; R factor = 0.025; wR factor = 0.066; data-to-parameter ratio = 17.2.

The absolute structure of the title compound, $C_{11}H_{13}FN^{+}\cdot Cl^{-}$, has been determined. The five-membered ring has an envelope conformation with the N atom at the flap position. In the crystal structure, the Cl^{-} anion links with the organic cation *via* N—H···Cl hydrogen bonding.

Related literature

For related structures, see: McArdle *et al.* (2004).



Experimental

Crystal data

$C_{11}H_{13}FN^{+}\cdot Cl^{-}$
M_r = 213.67
Orthorhombic, $P2_12_12_1$
a = 6.9146 (10) Å
b = 7.8048 (11) Å
c = 19.448 (3) Å
V = 1049.6 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.34 mm^{-1}
T = 296 K
0.50 × 0.36 × 0.25 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.766, T_{max} = 0.919
6726 measured reflections
2292 independent reflections
2244 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.066
S = 1.05
2292 reflections
133 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Absolute structure: Flack (1983), 934 Friedel pairs
Flack parameter: −0.03 (5)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···Cl1	0.939 (18)	2.146 (18)	3.0837 (13)	176.1 (15)
N1—H2···Cl1^{i}	0.899 (17)	2.275 (17)	3.0907 (13)	150.8 (14)

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT-Plus* (Bruker, 2007); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2479).

References

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supporting information

Acta Cryst. (2009). E**65**, o677 [doi:10.1107/S1600536809006953]

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S1. Comment

The title compound was prepared as a potential triple neurotransmittor reuptake inhibitor. Details will be published elsewhere. The molecular structure is shown in Fig. 1. The five-membered ring has an envelope conformation with N1 located 0.454 (2) Å above the plane constituted by C1, C2, C4 and C5, on the same side as C3, giving the six-membered ring N1—C1—C2—C3—C4—C5 a distinct boat conformation.

Two different $P2_1/c$ polymorphs, with $Z' = 1$ and 4, respectively, were obtained for the racemate of bicifadine hydrochloride (McArdle *et al.*, 2004), which has a methyl group rather than a F atom in the phenyl ortho position. Ring puckering remains unchanged, but phenyl rotations vary; C5—C4—C6—C11 is thus 59.81 (19)° for the title compound, but 83.9° for polymorph 1 of bicifadine and between -10.1 and -30.8° for polymorph 2 (1*S*,5*R*-enatiomers).

S2. Experimental

Block-shaped crystals were prepared from an acetonitrile solution by slow evaporation at room temperature.

S3. Refinement

Positional parameters were refined for the two H atoms bonded to N. Other H atoms were positioned with idealized geometry and fixed C—H = 0.93 (aromatic), 0.97 (methylene) or 0.98 Å (methine). U_{iso}(H) values were 1.2U_{eq}(C,N).

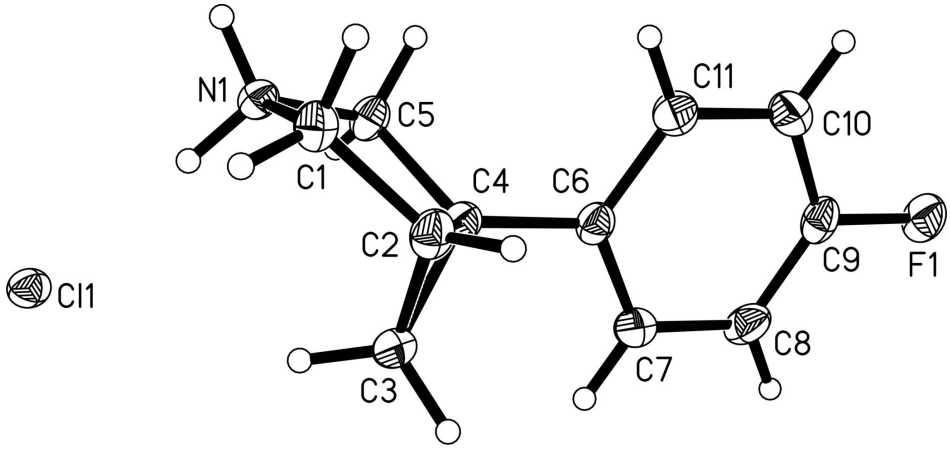

Figure 1

The asymmetric unit of (I). Displacement ellipsoids are shown at the 50% probability level and H atoms are shown as spheres of arbitrary size.

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Crystal data

$C_{11}H_{13}FN^{+}\cdot Cl^{-}$
$M_r = 213.67$
Orthorhombic, $P2_12_12_1$
$a = 6.9146$ (10) Å
$b = 7.8048$ (11) Å
$c = 19.448$ (3) Å
$V = 1049.6$ (3) Å^3
$Z = 4$
$F(000) = 448$
$D_x = 1.352$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4938 reflections
$\theta = 2.1$–$27.1°$
$\mu = 0.34$ mm^{-1}
$T = 296$ K
Block, colourless
0.50 × 0.36 × 0.25 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.3 pixels mm^{-1}
sets of exposures each taken over 0.5° ω rotation scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.766$, $T_{max} = 0.919$
6726 measured reflections
2292 independent reflections
2244 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 27.1°$, $\theta_{min} = 2.1°$
$h = -8\rightarrow4$
$k = -9\rightarrow10$
$l = -24\rightarrow24$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.025$
$wR(F^2) = 0.066$
$S = 1.05$
2292 reflections
133 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.036P)^2 + 0.1918P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.38$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}
Absolute structure: Flack (1983), 934 Friedel pairs
Absolute structure parameter: −0.03 (5)

Special details

Experimental. Crystallized from acetonitrile solution

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Data were collected by measuring three sets of exposures with the detector set at $2\theta = 29°$, crystal-to-detector distance 6.00 cm. Refinement of F^2 against ALL reflections.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.90409 (5)	0.57733 (4)	0.024884 (16)	0.01896 (9)
F1	−0.08580 (14)	0.45054 (11)	0.37784 (4)	0.0280 (2)
C6	0.2562 (2)	0.43164 (19)	0.20420 (7)	0.0184 (3)
C5	0.3941 (2)	0.60246 (17)	0.10328 (6)	0.0185 (3)

H51	0.2695	0.6595	0.1020	0.022*
H52	0.4871	0.6774	0.1254	0.022*
N1	0.45923 (17)	0.55635 (15)	0.03221 (6)	0.0168 (2)
H1	0.595 (3)	0.557 (2)	0.0300 (8)	0.020*
H2	0.422 (3)	0.640 (2)	0.0035 (8)	0.020*
C4	0.3797 (2)	0.43318 (18)	0.14105 (6)	0.0172 (3)
C7	0.3340 (2)	0.3976 (2)	0.26874 (7)	0.0225 (3)
H71	0.4644	0.3701	0.2726	0.027*
C11	0.0603 (2)	0.4731 (2)	0.19938 (7)	0.0235 (3)
H111	0.0067	0.4972	0.1566	0.028*
C9	0.0279 (2)	0.44386 (19)	0.32031 (7)	0.0217 (3)
C1	0.3826 (2)	0.38066 (16)	0.01663 (7)	0.0190 (3)
H11	0.4678	0.3197	−0.0145	0.023*
H12	0.2545	0.3868	−0.0036	0.023*
C8	0.2196 (2)	0.4041 (2)	0.32777 (7)	0.0248 (3)
H81	0.2720	0.3821	0.3709	0.030*
C2	0.3764 (2)	0.29489 (18)	0.08635 (7)	0.0206 (3)
H21	0.2921	0.1953	0.0929	0.025*
C3	0.5512 (2)	0.31501 (19)	0.13212 (7)	0.0224 (3)
H31	0.6677	0.3624	0.1119	0.027*
H32	0.5730	0.2285	0.1671	0.027*
C10	−0.0560 (2)	0.4790 (2)	0.25780 (8)	0.0255 (3)
H101	−0.1867	0.5059	0.2546	0.031*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.01835 (16)	0.01641 (15)	0.02212 (15)	−0.00080 (13)	−0.00166 (13)	0.00305 (12)
F1	0.0339 (5)	0.0273 (5)	0.0227 (4)	0.0066 (4)	0.0116 (4)	0.0024 (3)
C6	0.0212 (7)	0.0151 (6)	0.0189 (6)	−0.0006 (6)	0.0024 (5)	0.0012 (5)
C5	0.0235 (7)	0.0150 (6)	0.0170 (6)	−0.0025 (6)	0.0013 (6)	0.0003 (5)
N1	0.0187 (6)	0.0147 (5)	0.0169 (5)	0.0003 (5)	−0.0001 (4)	0.0017 (4)
C4	0.0182 (6)	0.0161 (6)	0.0172 (6)	0.0000 (6)	0.0002 (5)	0.0013 (5)
C7	0.0215 (7)	0.0241 (7)	0.0217 (6)	0.0035 (6)	0.0007 (5)	0.0037 (6)
C11	0.0230 (8)	0.0292 (7)	0.0184 (6)	−0.0007 (6)	−0.0016 (5)	0.0019 (5)
C9	0.0292 (7)	0.0160 (7)	0.0198 (6)	0.0007 (6)	0.0086 (6)	0.0005 (5)
C1	0.0209 (7)	0.0162 (6)	0.0198 (6)	−0.0026 (5)	0.0019 (6)	−0.0017 (5)
C8	0.0318 (8)	0.0253 (7)	0.0174 (6)	0.0046 (7)	0.0003 (6)	0.0049 (6)
C2	0.0250 (7)	0.0137 (6)	0.0232 (6)	−0.0006 (6)	0.0044 (6)	0.0004 (5)
C3	0.0227 (7)	0.0230 (7)	0.0216 (7)	0.0055 (6)	0.0024 (5)	0.0066 (5)
C10	0.0204 (7)	0.0302 (8)	0.0259 (7)	0.0024 (6)	0.0025 (6)	0.0016 (6)

Geometric parameters (Å, º)

F1—C9	1.3683 (15)	C7—H71	0.9300
C6—C7	1.3911 (19)	C11—C10	1.393 (2)
C6—C11	1.396 (2)	C11—H111	0.9300
C6—C4	1.4959 (18)	C9—C8	1.369 (2)

C5—N1	1.4977 (16)	C9—C10	1.375 (2)
C5—C4	1.5149 (18)	C1—C2	1.5128 (18)
C5—H51	0.9700	C1—H11	0.9700
C5—H52	0.9700	C1—H12	0.9700
N1—C1	1.5011 (16)	C8—H81	0.9300
N1—H1	0.939 (18)	C2—C3	1.509 (2)
N1—H2	0.899 (17)	C2—H21	0.9800
C4—C3	1.512 (2)	C3—H31	0.9700
C4—C2	1.5156 (19)	C3—H32	0.9700
C7—C8	1.395 (2)	C10—H101	0.9300

C7—C6—C11	118.70 (12)	F1—C9—C8	118.57 (13)
C7—C6—C4	121.43 (13)	F1—C9—C10	118.25 (13)
C11—C6—C4	119.80 (12)	C8—C9—C10	123.18 (13)
N1—C5—C4	104.93 (10)	N1—C1—C2	103.48 (11)
N1—C5—H51	110.8	N1—C1—H11	111.1
C4—C5—H51	110.8	C2—C1—H11	111.1
N1—C5—H52	110.8	N1—C1—H12	111.1
C4—C5—H52	110.8	C2—C1—H12	111.1
H51—C5—H52	108.8	H11—C1—H12	109.0
C5—N1—C1	107.42 (10)	C9—C8—C7	118.03 (13)
C5—N1—H1	109.9 (9)	C9—C8—H81	121.0
C1—N1—H1	110.4 (10)	C7—C8—H81	121.0
C5—N1—H2	108.2 (11)	C3—C2—C1	117.40 (13)
C1—N1—H2	116.1 (11)	C3—C2—C4	59.99 (9)
H1—N1—H2	104.7 (16)	C1—C2—C4	108.28 (11)
C3—C4—C2	59.78 (10)	C3—C2—H21	118.9
C3—C4—C5	115.15 (12)	C1—C2—H21	118.9
C3—C4—C6	122.49 (12)	C4—C2—H21	118.9
C6—C4—C5	116.26 (12)	C2—C3—C4	60.23 (9)
C6—C4—C2	124.19 (12)	C2—C3—H31	117.7
C5—C4—C2	106.36 (10)	C4—C3—H31	117.7
C6—C7—C8	121.09 (14)	C2—C3—H32	117.7
C6—C7—H71	119.5	C4—C3—H32	117.7
C8—C7—H71	119.5	H31—C3—H32	114.9
C10—C11—C6	120.85 (13)	C9—C10—C11	118.15 (14)
C10—C11—H111	119.6	C9—C10—H101	120.9
C6—C11—H111	119.6	C11—C10—H101	120.9

C1—C2—C4—C5	−1.68 (16)	C5—N1—C1—C2	−30.53 (14)
N1—C1—C2—C4	19.46 (15)	F1—C9—C8—C7	179.97 (13)
N1—C5—C4—C2	−16.93 (15)	C10—C9—C8—C7	−0.6 (3)
C5—C4—C6—C11	59.81 (19)	C6—C7—C8—C9	0.4 (2)
C4—C5—N1—C1	29.91 (15)	N1—C1—C2—C3	−45.58 (15)
C7—C6—C4—C3	34.1 (2)	C6—C4—C2—C3	−110.84 (15)
C11—C6—C4—C3	−149.04 (14)	C5—C4—C2—C3	109.97 (13)
C7—C6—C4—C2	107.32 (17)	C6—C4—C2—C1	137.51 (14)
C11—C6—C4—C2	−75.82 (19)	C3—C4—C2—C1	−111.65 (14)

C7—C6—C4—C5	−117.04 (15)	C1—C2—C3—C4	96.23 (13)
N1—C5—C4—C6	−159.86 (12)	C6—C4—C3—C2	113.59 (15)
N1—C5—C4—C3	46.86 (15)	C5—C4—C3—C2	−94.97 (12)
C11—C6—C7—C8	0.2 (2)	F1—C9—C10—C11	179.61 (13)
C4—C6—C7—C8	177.04 (14)	C8—C9—C10—C11	0.2 (2)
C7—C6—C11—C10	−0.6 (2)	C6—C11—C10—C9	0.4 (2)
C4—C6—C11—C10	−177.53 (14)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···Cl1	0.939 (18)	2.146 (18)	3.0837 (13)	176.1 (15)
N1—H2···Cl1^{i}	0.899 (17)	2.275 (17)	3.0907 (13)	150.8 (14)

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