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2-Amino-6-(2,6-difluorobenzamido)-pyridinium chloride

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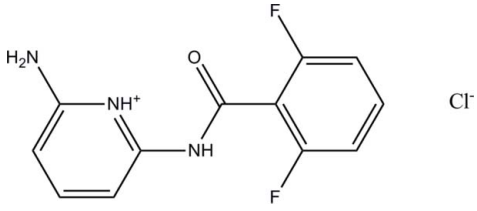
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.047; wR factor = 0.113; data-to-parameter ratio = 18.7.

In the cation of the title compound, $C_{12}H_{10}F_2N_3O^+\cdot Cl^-$, the dihedral angle between the pyridine and benzene rings is 16.1 (1)°. In the crystal structure, molecules linked into two-dimensional sheets parallel to the *bc* plane by intermolecular N—H···Cl, C—H···Cl and C—H···F hydrogen bonds.

Related literature

For general background to 2,6-diflorobenzylchloride derivatives, see: Beavo (1995); Beavo & Reifsnyder (1990); Hidaka & Asano (1976); Nicholson *et al.* (1991). For the stability of the temperature controller used in the data collection, see: Cosier & Glazer (1986).



Experimental

Crystal data

$C_{12}H_{10}F_2N_3O^+\cdot Cl^-$
M_r = 285.68
Monoclinic, $P2_1/c$
a = 7.3196 (2) Å
b = 13.6314 (3) Å
c = 12.2892 (3) Å
β = 99.755 (1)°
V = 1208.44 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.34 mm^{-1}
T = 100 K
0.34 × 0.12 × 0.08 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.895, T_{max} = 0.972
11996 measured reflections
3524 independent reflections
2628 reflections with $I > 2\sigma(I)$
R_{int} = 0.041

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.113
S = 1.07
3524 reflections
188 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*1···Cl1^{i}	0.84 (3)	2.35 (2)	3.1622 (18)	163 (2)
N2—H1*N*2···Cl1	0.87 (2)	2.41 (2)	3.1678 (17)	146 (2)
N3—H1*N*3···Cl1^{ii}	0.84 (2)	2.39 (2)	3.2140 (17)	166 (2)
N3—H2*N*3···Cl1	0.84 (2)	2.51 (2)	3.2346 (18)	145 (2)
C3—H3*A*···F2^{iii}	0.93	2.52	3.414 (3)	162
C10—H10*A*···Cl1^{iv}	0.93	2.74	3.581 (2)	151

Symmetry codes: (i) $-x, -y+1, -z+1$; (ii) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (iii) $x, -y+\frac{3}{2}, z+\frac{1}{2}$; (iv) $-x, y+\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; 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* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5090).

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§ Thomson Reuters ResearcherID: A-3561-2009.

supplementary materials

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2-Amino-6-(2,6-difluorobenzamido)pyridinium chloride

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Comment

Thr derivatives of 2,6-diflorobenzylchloride involved in the inhibition of phosphodiesterases (PDEs) are enzymes which catalyze PDEs. These derivatives are classified into seven families, five of which, PDE1–PDE5, have been characterized (Beavo, 1995). The hydrolysis of cyclic nucleotides was evaluated according to the methods of Beavo & Reifsnyder (1990); Hidaka & Asano, (1976); Nicholson *et al.* (1991).

The asymmetric unit of the title compound contains one protonated 2-amino-6-(2,6-difluorobenzamido)pyridin-1-ium cation and one chloride anion (Fig. 1). The cation molecule is twisted with the dihedral angle between the pyridine ring and the benzene ring being 16.1 (1)°. In the crystal structure, molecules are linked into infinite chains along *c* axis by intermolecular C3—H3A···F2 hydrogen bonds. The chloride anions link these chains into two-dimensional sheets parallel to the *bc* plane by intermolecular N—H···Cl and C—H···Cl hydrogen bonds (Fig. 2, Table 1).

Experimental

2,6-Difluorobenzylchloride (0.01 mol, 1.7 g) was added drop-wise into a round bottom flask containing 25 ml mixture of tetrahydrofuran (THF) and 2,6-diamino pyridine (0.01 mol, 1.1 g) with stirring. The mixture was then refluxed for two and a half hours. The oily precipitate formed was filtrated and dissolved in water and then filtrated and evaporated. The green precipitate formed was dissolved in methanol. Green needle-shaped crystals which were formed at room temperature overnight and were filtrated and dried at 333 K.

Refinement

The N-bound hydrogen atoms were located from a difference Fourier map and refined freely. The remaining H atoms were positioned geometrically [C–H = 0.93 Å and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C)].

Figures

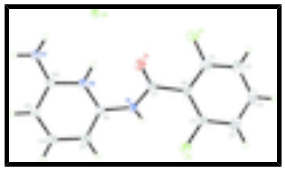

Fig. 1. The molecular structure of the title compound with atom labels and 50% probability ellipsoids for non-H atoms.

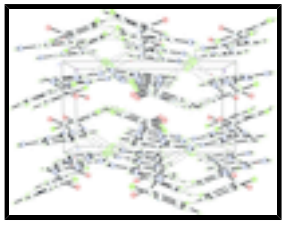

Fig. 2. The crystal packing of title compound, viewed down the *c* axis, showing two 2-D planes parallel to *bc* plane.

2-Amino-6-(2,6-difluorobenzamido)pyridinium chloride

Crystal data

$C_{12}H_{10}F_2N_3O^+\cdot Cl^-$	$F(000) = 584$
$M_r = 285.68$	$D_x = 1.570$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 3017 reflections
$a = 7.3196$ (2) Å	$\theta = 3.4$–$30.0°$
$b = 13.6314$ (3) Å	$\mu = 0.34$ mm^{-1}
$c = 12.2892$ (3) Å	$T = 100$ K
$\beta = 99.755$ (1)°	Needle, green
$V = 1208.44$ (5) Å^3	0.34 × 0.12 × 0.08 mm
$Z = 4$	

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	3524 independent reflections
Radiation source: fine-focus sealed tube	2628 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.041$
φ and ω scans	$\theta_{max} = 30.0°$, $\theta_{min} = 2.3°$
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)	$h = -7\rightarrow10$
$T_{min} = 0.895$, $T_{max} = 0.972$	$k = -15\rightarrow19$
11996 measured reflections	$l = -17\rightarrow17$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.047$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.113$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.07$	$w = 1/[\sigma^2(F_o^2) + (0.0421P)^2 + 0.7252P]$ where $P = (F_o^2 + 2F_c^2)/3$
3524 reflections	$(\Delta/\sigma)_{max} < 0.001$
188 parameters	$\Delta\rho_{max} = 0.37$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.35$ e Å^{-3}

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

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. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.00538 (7)	0.25071 (3)	0.48845 (3)	0.02010 (13)
F1	0.25530 (18)	0.41301 (9)	0.78112 (10)	0.0280 (3)
F2	0.45736 (17)	0.69690 (9)	0.61851 (10)	0.0250 (3)
O1	0.3723 (2)	0.41722 (10)	0.57487 (11)	0.0235 (3)
N1	0.2196 (2)	0.55280 (13)	0.49862 (13)	0.0179 (3)
N2	0.1692 (2)	0.42610 (12)	0.36587 (13)	0.0174 (3)
N3	0.1129 (3)	0.29372 (13)	0.24827 (14)	0.0212 (4)
C1	0.3195 (3)	0.50617 (15)	0.78901 (16)	0.0209 (4)
C2	0.3485 (3)	0.55016 (18)	0.89145 (16)	0.0267 (5)
H2A	0.3219	0.5170	0.9530	0.032*
C3	0.4181 (3)	0.64466 (18)	0.90068 (17)	0.0284 (5)
H3A	0.4396	0.6750	0.9695	0.034*
C4	0.4564 (3)	0.69494 (16)	0.80912 (17)	0.0245 (4)
H4A	0.5063	0.7578	0.8156	0.029*
C5	0.4181 (3)	0.64856 (15)	0.70798 (16)	0.0193 (4)
C6	0.3492 (3)	0.55372 (14)	0.69319 (15)	0.0175 (4)
C7	0.3170 (3)	0.50038 (14)	0.58476 (15)	0.0176 (4)
C8	0.1933 (3)	0.52380 (14)	0.38828 (15)	0.0164 (4)
C9	0.1878 (3)	0.58888 (15)	0.30421 (16)	0.0195 (4)
H9A	0.2019	0.6557	0.3187	0.023*
C10	0.1605 (3)	0.55403 (15)	0.19510 (16)	0.0208 (4)
H10A	0.1580	0.5981	0.1371	0.025*
C11	0.1375 (3)	0.45590 (15)	0.17297 (15)	0.0188 (4)
H11A	0.1215	0.4333	0.1006	0.023*
C12	0.1384 (3)	0.38928 (14)	0.26127 (14)	0.0164 (4)
H1N1	0.181 (3)	0.6086 (19)	0.512 (2)	0.031 (7)*
H1N2	0.166 (3)	0.3855 (18)	0.420 (2)	0.027 (6)*
H1N3	0.095 (3)	0.2722 (18)	0.183 (2)	0.030 (7)*
H2N3	0.105 (3)	0.2579 (18)	0.303 (2)	0.025 (6)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0327 (3)	0.0136 (2)	0.01412 (19)	0.00100 (19)	0.00408 (17)	0.00081 (17)
F1	0.0345 (7)	0.0215 (7)	0.0282 (6)	−0.0066 (5)	0.0053 (5)	0.0033 (5)

F2	0.0321 (7)	0.0177 (6)	0.0235 (6)	−0.0045 (5)	−0.0004 (5)	0.0025 (5)
O1	0.0313 (8)	0.0155 (7)	0.0210 (7)	0.0069 (6)	−0.0038 (6)	−0.0023 (6)
N1	0.0250 (9)	0.0120 (8)	0.0155 (7)	0.0029 (7)	0.0005 (6)	−0.0011 (6)
N2	0.0245 (9)	0.0145 (8)	0.0130 (7)	−0.0002 (7)	0.0027 (6)	0.0010 (6)
N3	0.0359 (10)	0.0154 (9)	0.0124 (7)	−0.0024 (7)	0.0048 (7)	−0.0008 (7)
C1	0.0200 (10)	0.0200 (11)	0.0222 (9)	0.0002 (8)	0.0020 (8)	0.0003 (8)
C2	0.0256 (11)	0.0372 (13)	0.0173 (9)	0.0028 (10)	0.0037 (8)	−0.0016 (9)
C3	0.0273 (12)	0.0364 (13)	0.0205 (9)	0.0056 (10)	0.0009 (8)	−0.0120 (9)
C4	0.0244 (11)	0.0202 (11)	0.0271 (10)	0.0047 (8)	−0.0011 (8)	−0.0085 (8)
C5	0.0214 (10)	0.0160 (10)	0.0193 (9)	0.0018 (8)	0.0006 (7)	−0.0013 (7)
C6	0.0189 (9)	0.0160 (10)	0.0166 (8)	0.0025 (7)	−0.0002 (7)	−0.0019 (7)
C7	0.0181 (9)	0.0167 (10)	0.0170 (8)	−0.0012 (8)	0.0003 (7)	−0.0007 (7)
C8	0.0175 (9)	0.0148 (10)	0.0159 (8)	0.0007 (7)	0.0001 (7)	−0.0025 (7)
C9	0.0244 (10)	0.0127 (10)	0.0208 (9)	−0.0005 (8)	0.0019 (8)	0.0008 (7)
C10	0.0255 (10)	0.0183 (10)	0.0180 (8)	−0.0001 (8)	0.0025 (8)	0.0046 (8)
C11	0.0233 (10)	0.0199 (10)	0.0130 (8)	−0.0012 (8)	0.0024 (7)	−0.0005 (7)
C12	0.0186 (9)	0.0152 (9)	0.0150 (8)	0.0007 (7)	0.0017 (7)	−0.0007 (7)

Geometric parameters (Å, °)

F1—C1	1.352 (2)	C2—H2A	0.9300
F2—C5	1.354 (2)	C3—C4	1.386 (3)
O1—C7	1.217 (2)	C3—H3A	0.9300
N1—C7	1.373 (2)	C4—C5	1.380 (3)
N1—C8	1.394 (2)	C4—H4A	0.9300
N1—H1N1	0.84 (3)	C5—C6	1.388 (3)
N2—C12	1.363 (2)	C6—C7	1.501 (3)
N2—C8	1.365 (2)	C8—C9	1.357 (3)
N2—H1N2	0.87 (2)	C9—C10	1.405 (3)
N3—C12	1.322 (3)	C9—H9A	0.9300
N3—H1N3	0.84 (3)	C10—C11	1.370 (3)
N3—H2N3	0.84 (2)	C10—H10A	0.9300
C1—C2	1.378 (3)	C11—C12	1.414 (3)
C1—C6	1.393 (3)	C11—H11A	0.9300
C2—C3	1.383 (3)		
C7—N1—C8	124.78 (17)	C4—C5—C6	123.94 (19)
C7—N1—H1N1	117.9 (17)	C5—C6—C1	115.33 (17)
C8—N1—H1N1	117.0 (17)	C5—C6—C7	124.48 (17)
C12—N2—C8	123.00 (16)	C1—C6—C7	120.10 (18)
C12—N2—H1N2	117.7 (16)	O1—C7—N1	123.10 (18)
C8—N2—H1N2	119.2 (16)	O1—C7—C6	122.36 (17)
C12—N3—H1N3	117.0 (17)	N1—C7—C6	114.54 (17)
C12—N3—H2N3	120.1 (16)	C9—C8—N2	119.86 (17)
H1N3—N3—H2N3	123 (2)	C9—C8—N1	122.45 (18)
F1—C1—C2	118.17 (18)	N2—C8—N1	117.68 (16)
F1—C1—C6	118.59 (17)	C8—C9—C10	119.13 (18)
C2—C1—C6	123.2 (2)	C8—C9—H9A	120.4
C1—C2—C3	118.5 (2)	C10—C9—H9A	120.4
C1—C2—H2A	120.7	C11—C10—C9	120.84 (18)

C3—C2—H2A	120.7	C11—C10—H10A	119.6
C2—C3—C4	121.09 (19)	C9—C10—H10A	119.6
C2—C3—H3A	119.5	C10—C11—C12	119.36 (17)
C4—C3—H3A	119.5	C10—C11—H11A	120.3
C5—C4—C3	117.8 (2)	C12—C11—H11A	120.3
C5—C4—H4A	121.1	N3—C12—N2	118.32 (17)
C3—C4—H4A	121.1	N3—C12—C11	123.91 (17)
F2—C5—C4	118.02 (18)	N2—C12—C11	117.77 (17)
F2—C5—C6	117.99 (16)		
F1—C1—C2—C3	178.58 (19)	C5—C6—C7—O1	131.1 (2)
C6—C1—C2—C3	−2.9 (3)	C1—C6—C7—O1	−45.2 (3)
C1—C2—C3—C4	0.7 (3)	C5—C6—C7—N1	−49.3 (3)
C2—C3—C4—C5	1.7 (3)	C1—C6—C7—N1	134.4 (2)
C3—C4—C5—F2	−179.22 (18)	C12—N2—C8—C9	−0.2 (3)
C3—C4—C5—C6	−2.1 (3)	C12—N2—C8—N1	178.56 (18)
F2—C5—C6—C1	177.18 (17)	C7—N1—C8—C9	−144.7 (2)
C4—C5—C6—C1	0.1 (3)	C7—N1—C8—N2	36.5 (3)
F2—C5—C6—C7	0.7 (3)	N2—C8—C9—C10	−1.2 (3)
C4—C5—C6—C7	−176.40 (19)	N1—C8—C9—C10	−179.89 (18)
F1—C1—C6—C5	−178.98 (17)	C8—C9—C10—C11	0.8 (3)
C2—C1—C6—C5	2.5 (3)	C9—C10—C11—C12	1.0 (3)
F1—C1—C6—C7	−2.4 (3)	C8—N2—C12—N3	−178.62 (18)
C2—C1—C6—C7	179.1 (2)	C8—N2—C12—C11	2.0 (3)
C8—N1—C7—O1	−9.0 (3)	C10—C11—C12—N3	178.30 (19)
C8—N1—C7—C6	171.45 (18)	C10—C11—C12—N2	−2.3 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N1···Cl1^{i}	0.84 (3)	2.35 (2)	3.1622 (18)	163 (2)
N2—H1N2···Cl1	0.87 (2)	2.41 (2)	3.1678 (17)	146 (2)
N3—H1N3···Cl1^{ii}	0.84 (2)	2.39 (2)	3.2140 (17)	166 (2)
N3—H2N3···Cl1	0.84 (2)	2.51 (2)	3.2346 (18)	145 (2)
C3—H3A···F2^{iii}	0.93	2.52	3.414 (3)	162.
C10—H10A···Cl1^{iv}	0.93	2.74	3.581 (2)	151.

Symmetry codes: (i) −*x*, −*y*+1, −*z*+1; (ii) *x*, −*y*+1/2, *z*−1/2; (iii) *x*, −*y*+3/2, *z*+1/2; (iv) −*x*, *y*+1/2, −*z*+1/2.

Fig. 1

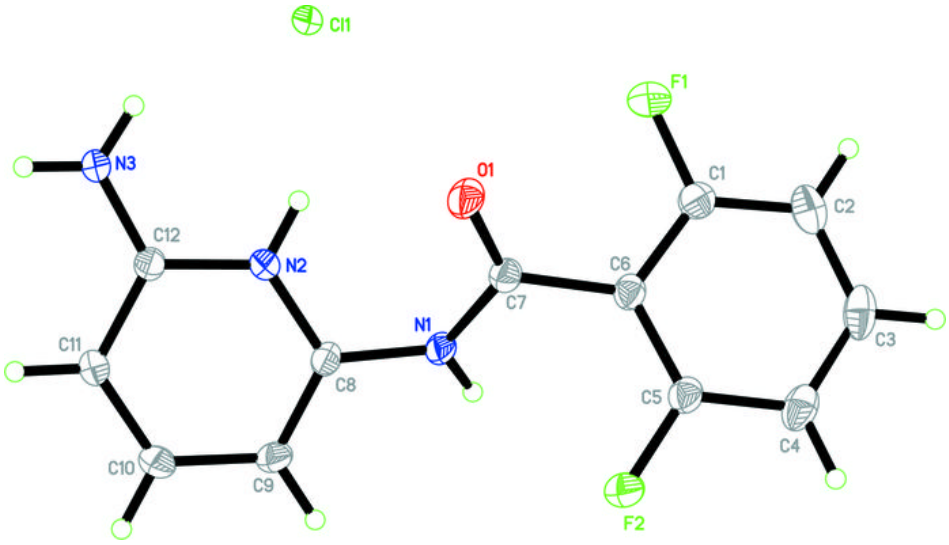

Fig. 2