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Key indicators

Single-crystal X-ray study

T = 105 K

Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$

Disorder in main residue

R factor = 0.043

wR factor = 0.108

Data-to-parameter ratio = 11.9

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

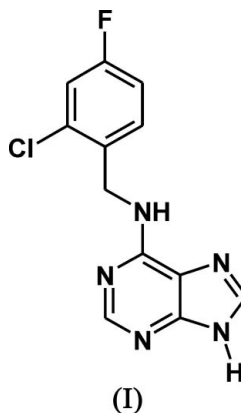
6-(2-Chloro-4-fluorobenzylamino)purine

In the title molecule, $\text{C}_{12}\text{H}_9\text{ClFN}_5$, all bond lengths and angles show normal values. The mean planes of the benzene ring and the purine ring system make a dihedral angle of $77.79(5)^\circ$. Intermolecular $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds link the molecules into ribbons extending along the $[1\bar{1}0]$ direction. The crystal packing is further stabilized by weak $\text{C}-\text{H}\cdots\text{F}$ and $\text{C}-\text{H}\cdots\text{Cl}$ interactions.

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Comment

As part of our studies of the correlation between the structure and the biological activity of cytokinins and cyclin-dependent kinase (CDK) inhibitors, we have determined the structure of the title compound, (I). A broad spectrum of possible applications, as well as some crystal structures of the above-mentioned compounds, have previously been described briefly (Trávníček, Popa & Doležal, 2004; Trávníček & Zatloukal, 2004; Trávníček & Kryštof, 2004)



To date, 36 structures of both organic and organometallic compounds with the 6-benzylaminopurine (Bap) unit have been included in the Cambridge Structural Database (Version 5.27; Allen, 2002). However, the title compound, (I), represents the first structure of a Bap derivative substituted on the benzene ring by two different halogens, in this case F and Cl.

The bond lengths and angles in (I) (Fig. 1 and Table 1) show normal values, but one should mention that the values of interatomic parameters in an adenine system are significantly influenced by the extent of protonation and substitution, as discussed in greater detail by Trávníček, Popa & Doležal (2004). The mean planes of the nearly planar benzene ring and the purine ring system make a dihedral angle of $77.79(5)^\circ$.

Intermolecular $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds (Table 2) link the molecules into ribbons (Fig. 2) extending along the $[1\bar{1}0]$

direction. The crystal packing is further stabilized by weak C—H···F and C—H···Cl interactions (Table 2) and π – π stacking interactions [$C4 \cdots C6^v = 3.347$ (3) Å; symmetry code: (v) $1 - x, 1 - y, 2 - z$].

Experimental

6-(2-Chloro-4-fluorobenzylamino)purine, (I), was prepared according to the literature method of Doležal *et al.* (2006). Crystals suitable for single-crystal X-ray analysis were obtained by evaporation of a deuterated dimethyl sulfoxide solution of (I) in an NMR tube.

Crystal data

$C_{12}H_9ClFN_5$	$Z = 2$
$M_r = 277.69$	$D_x = 1.573 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 7.2544$ (6) Å	Cell parameters from 3235 reflections
$b = 7.3755$ (7) Å	$\theta = 1.8$ – 29.7°
$c = 11.8649$ (11) Å	$\mu = 0.33 \text{ mm}^{-1}$
$\alpha = 98.108$ (8) $^\circ$	$T = 105$ (2) K
$\beta = 101.603$ (7) $^\circ$	Prism, colourless
$\gamma = 105.518$ (8) $^\circ$	$0.30 \times 0.30 \times 0.25 \text{ mm}$
$V = 586.28$ (10) Å ³	

Data collection

Oxford Xcalibur diffractometer	$R_{\text{int}} = 0.019$
ω scans	$\theta_{\text{max}} = 25.0^\circ$
Absorption correction: none	$h = -8 \rightarrow 8$
5335 measured reflections	$k = -8 \rightarrow 7$
2063 independent reflections	$l = -14 \rightarrow 14$
1908 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.05P)^2 + 0.65P]$
$R[F^2 > 2\sigma(F^2)] = 0.043$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.108$	$(\Delta/\sigma)_{\text{max}} = 0.002$
$S = 1.04$	$\Delta\rho_{\text{max}} = 0.48 \text{ e \AA}^{-3}$
2063 reflections	$\Delta\rho_{\text{min}} = -0.25 \text{ e \AA}^{-3}$
173 parameters	
H-atom parameters constrained	

Table 1

Selected geometric parameters (Å, $^\circ$).

Cl—C11	1.737 (2)	C4—C5	1.386 (3)
F—C13	1.364 (3)	C5—N7	1.385 (3)
N1—C2	1.332 (3)	C5—C6	1.407 (3)
N1—C6	1.366 (3)	C6—N6	1.344 (3)
C2—N3	1.336 (3)	N6—C9	1.449 (3)
N3—C4	1.353 (3)	C8—N9	1.364 (3)
C4—N9	1.365 (3)		
C2—N1—C6	118.16 (18)	C4—C5—C6	116.77 (19)
N1—C2—N3	129.6 (2)	N6—C6—N1	120.48 (19)
C2—N3—C4	110.65 (18)	N6—C6—C5	121.48 (19)
N3—C4—N9	128.00 (18)	N1—C6—C5	118.03 (18)
N3—C4—C5	126.74 (19)	C6—N6—C9	122.76 (18)
N9—C4—C5	105.26 (18)	C8—N7—C5	102.89 (17)
N7—C5—C4	111.41 (19)	N7—C8—N9	114.00 (19)
N7—C5—C6	131.81 (19)	C8—N9—C4	106.43 (17)
N1—C6—N6—C9	2.3 (3)	N6—C9—C10—C15	25.0 (3)
C6—N6—C9—C10	−92.3 (2)		

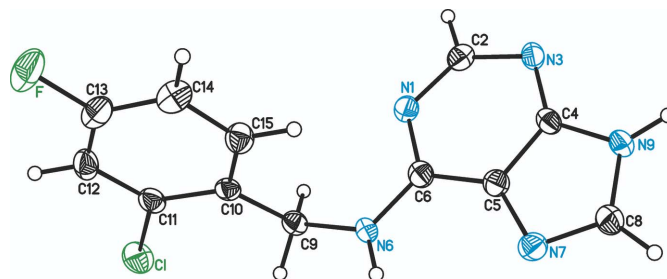


Figure 1

The molecular structure of (I), with 50% probability displacement ellipsoids. Only the major component (attached to N9) of the disordered atom H9 is shown.

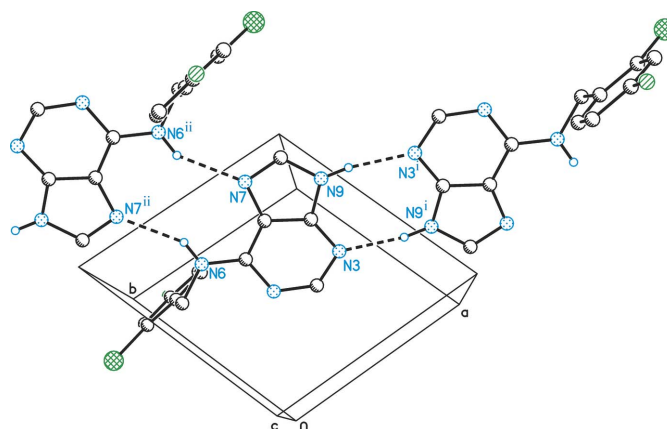


Figure 2

Part of the crystal packing of (I), showing the formation of an infinite ribbon of hydrogen-bonded (dashed lines) molecules [symmetry codes: (i) $2 - x, 1 - y, 2 - z$; (ii) $1 - x, 2 - y, 2 - z$]. C-bound H atoms have been omitted for clarity and only the major component of the disordered H atom (H9 attached to N9) is shown.

Table 2

Hydrogen-bond geometry (Å, $^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$N3-H3 \cdots N9^i$	0.88	2.02	2.870 (2)	163
$N6-H6 \cdots N7^{ii}$	0.88	2.16	2.953 (2)	149
$N9-H9 \cdots N3^i$	0.88	2.00	2.870 (2)	170
$C8-H8 \cdots F^{iii}$	0.95	2.55	3.029 (3)	112
$C15-H15 \cdots Cl^{iv}$	0.95	2.77	3.459 (2)	131

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

All H atoms were located in a difference map and refined using a riding model, with C—H = 0.95 and 0.99 Å, N—H = 0.88 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{parent atom})$. The H atom involved in the $N3-H \cdots N9^i$ intermolecular hydrogen bond (Table 2) was treated as disordered over two positions, H3 and H9, with refined occupancies of 0.18 (4) and 0.82 (4), respectively. We also tried to refine freely the H atoms involved in the hydrogen-bond system. However, the best result was achieved by using the riding-model approximation.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2002); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2002); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP III* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97* (Sheldrick, 1997) and *PARST* (Nardelli, 1995).

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