6-(4-Bromobenzylamino)purine

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Key indicators

Single-crystal X-ray study
T = 110 K
Mean σ(C–C) = 0.004 Å
R factor = 0.029
wR factor = 0.071
Data-to-parameter ratio = 12.6

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

Molecules of the the title compound, $C_{12}H_{10}BrN_5$, are connected by N—H···N hydrogen bonds into linear chains. In contrast to similar electroneutral 6-benzylaminopurine derivatives, which are usually protonated at the N9 atom, the purine skeleton is protonated at the N7 position.

Comment

As part of a systematic crystallographic study of cytokinins and cyclin-dependent kinase (CDK) inhibitors, we have prepared the title compound, (I). As detailed in our earlier papers (Maloň *et al.*, 2001; Trávníček *et al.*, 2004; Trávníček & Zatloukal, 2004; Trávníček & Kryštof, 2004), cytokinins and CDK inhibitors are employed in many branches of agriculture, chemistry and medicine.

(I)
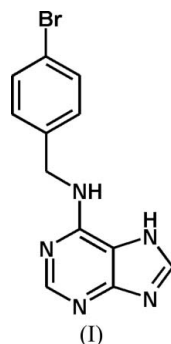
The molecular structure of (I) (Fig. 1) contains three different aromatic rings, benzene (*A*), pyrimidine (*B*) and imidazole (*C*). Each of these rings deviates slightly from planarity, with the maximum deviations from the mean planes being 0.007 (3) Å for C11 (ring *A*), 0.010 (3) Å for C5 (ring *B*) and 0.004 (3) Å for C8 (ring *C*). The dihedral angle between ring *A* and the purine skeleton (rings *B* and *C*) is 61.29 (7)°, whilst the *B* and *C* rings are nearly coplanar [dihedral angle 0.88 (9)°]. Bond lengths and angles in (I) are comparable with those in the structures of 6-(2-bromobenzylamino)purine monohydrate, (II) (Trávníček & Rosenker, 2006), 6-(2-chloro-4-fluorobenzylamino)purine (Trávníček *et al.*, 2006), 6-(2-chlorobenzylamino)purine dihydrate (Maloň *et al.*, 2001), 6-(2-hydroxybenzylamino)purine acetic acid solvate (Trávníček *et al.*, 1997) and 6-benzylaminopurine (Raghunathan *et al.*, 1983). Despite this, however, compound (I) is significantly different from these structures, on account of its protonation of atom N7, in contrast with N9 in compound (II) and all of the other above-mentioned 6-benzylaminopurine derivatives. Protonation at the N7 position is very rare for electroneutral forms.
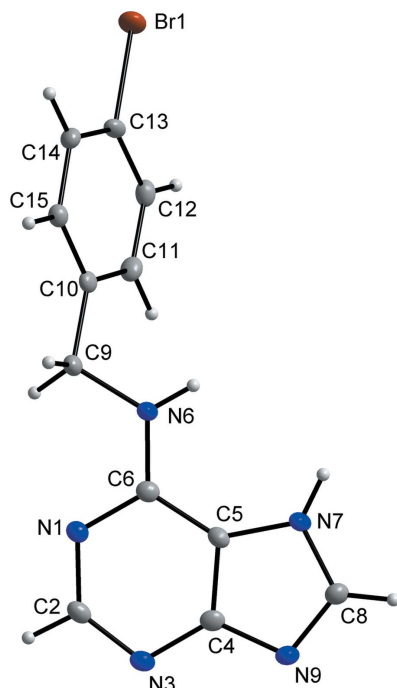
Figure 1
The molecular structure of (I), with displacement ellipsoids shown at the 50% probability level.
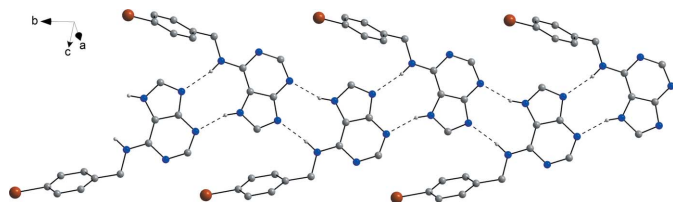

Figure 2
Part of the crystal structure of (I), showing a chain of hydrogen-bonded (dashed lines) molecules extending along [010]. H atoms not involved in hydrogen bonding have been omitted.

Intermolecular N—H···N hydrogen bonds (Table 2) link the molecules of (I) into one-dimensional chains extending along [010] (Fig. 2). The chains lie in layers parallel to (001), with π–π stacking interactions between them (Fig. 3). The shortest C···C contacts between chains are C8···C8^{ii} = 3.260 (4) Å and C2···C4^{iii} = 3.340 (4) Å [symmetry codes: (ii) $1 - x$, y, $\frac{1}{2} - z$; (iii) $-x$, y, $\frac{1}{2} - z$]. Between these layers, the bromobenzyl moietiess also involve π–π stacking arrangements, with an interplanar separation of 3.54 (1) Å. In this region, short Br···Br contacts [3.577 (4) Å] are observed. By comparison, the shortest such contacts in (II) are 4.673 (5) Å. This difference may be related to the presence of solvent water molecules in (II).
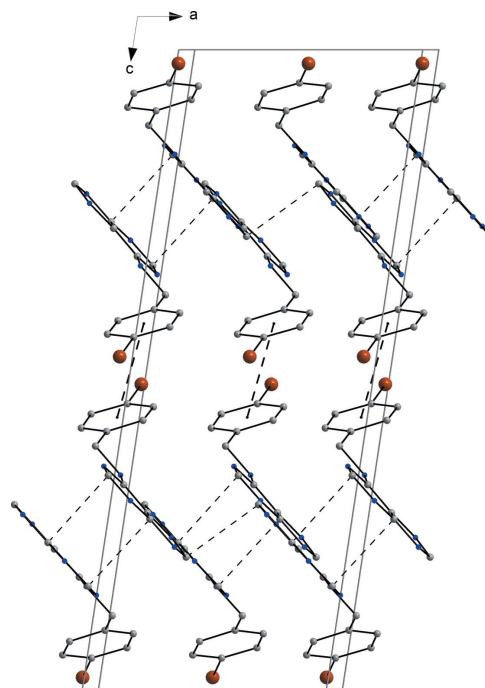
Experimental

Figure 3
A projection of (I), along the *b* direction, showing π–π stacking interactions as dashed lines. H atoms have been omitted.

Crystal data

$C_{12}H_{10}BrN_5$	$Z = 8$
$M_r = 304.16$	$D_x = 1.734$ Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation
$a = 8.8944$ (4) Å	$\mu = 3.52$ mm^{-1}
$b = 11.2015$ (5) Å	$T = 110$ (2) K
$c = 23.6518$ (9) Å	Prism, colourless
$\beta = 98.564$ (3)°	$0.30 \times 0.30 \times 0.25$ mm
$V = 2330.17$ (17) Å^3	

Data collection

Oxford Xcalibur CCD area-detector diffractometer	6766 measured reflections
ω scans	2061 independent reflections
Absorption correction: multi-scan (Blessing, 1995)	1976 reflections with $I > 2\sigma(I)$
$T_{min} = 0.369$, $T_{max} = 0.417$	$R_{int} = 0.024$
	$\theta_{max} = 25.0°$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0233P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.029$	$\quad + 9.0814P]$
$wR(F^2) = 0.071$	$\quad$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.19$	$(\Delta/\sigma)_{max} < 0.001$
2061 reflections	$\Delta\rho_{max} = 0.39$ e Å^{-3}
163 parameters	$\Delta\rho_{min} = -0.27$ e Å^{-3}
H-atom parameters constrained	

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

D—H···A	D—H	H···A	D···A	D—H···A
N6—H6A···N9^i	0.88	2.07	2.935 (3)	166
N7—H7A···N3^i	0.88	2.01	2.856 (3)	161

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

All H atoms were located in a difference map and refined using a riding model, with C—H distances of 0.95 and 0.99 Å and N—H distances of 0.88 Å, and with $U_{iso}(H) = 1.2U_{eq}(C,N)$.

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: *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *SHELXL97* and *DIAMOND*.

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