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2-Chloro-6-[(4-hydroxy-3,5-dimethoxybenzyl)amino]-9-isopropylpurine

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Key indicators

Single-crystal X-ray study
T = 105 K
Mean σ(C–C) = 0.003 Å
R factor = 0.041
wR factor = 0.091
Data-to-parameter ratio = 12.7

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

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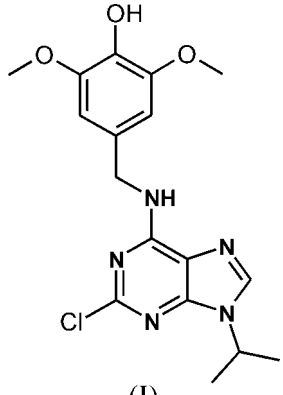
The structure of the title compound, $C_{17}H_{20}ClN_5O_3$, consists of discrete molecules of a 6-(benzylamino)purine derivative. The secondary structure is stabilized by N—H···O hydrogen bonds connecting molecules into centrosymmetric dimers and by weak interatomic contacts of the types C—H···Cl and C—H···O, and by π–π stacking interactions.

Comment

In continuation of our systematic crystallographic study of aromatic cytokinins and cyclin-dependent kinase (CDK) inhibitors derived from 6-benzylaminopurine (Trávníček & Zatloukal, 2004; Trávníček & Kryštof, 2004; Trávníček *et al.*, 2006; Trávníček & Rosenker, 2006; Trávníček & Maľarová-Matiková, 2006; Trávníček & Matiková-Maľarová, 2006; Trávníček & Popa, 2007), we now report the structure of the title compound, (I). The synthesis and study of these compounds are motivated by the fact that small changes in the substitution of the 6-benzylaminopurine system, and consequently in the structure, may have a significant impact on their biological activity.



(I)

The structure of compound (I) is composed of discrete molecules (Fig. 1). There are three different aromatic rings in the molecule, benzene (*A*), pyrimidine (*B*) and imidazole (*C*). Each ring is essentially planar, the maximum deviations from the mean planes being 0.014 (2) Å for atom C12 (ring *A*), 0.035 (2) Å for atom C5 (ring *B*) and 0.007 (2) Å for atom C4 (ring *C*) (*DIAMOND*; Brandenburg, 2006). The dihedral angle between ring *A* and the purine skeleton (consisting of fused rings *B* and *C*) is 62.18 (6)°, and that between rings *B* and *C* is 4.29 (6)°. However, the latter value differs significantly from those previously reported for C2/N9/phenyl-substituted 6-benzylaminopurines, *e.g.* *N*-[2-(azepan-1-yl)-9-isopropyl-9*H*-purin-6-yl]-4-methoxybenzylamine [2.99 (4)°;

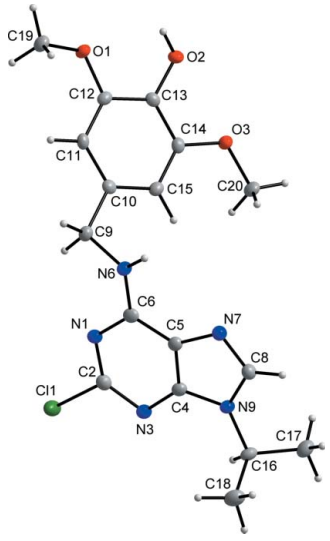


Figure 1
The molecular structure of (I), showing the atomic labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

Trávníček & Zatloukal, 2004], 6-benzylamino-2-(2-hydroxyethylamino)-9-methylpurine-1,7-diium bis(perchlorate) monohydrate [0.79 (8)°; Trávníček & Kryštof, 2004] and 2-chloro-6-[(2,6-dimethoxybenzyl)amino]-9-isopropylpurine 1/8 hydrate [1.77 (6)°; Trávníček & Popa, 2007].

Intermolecular N—H···O and O—H···N hydrogen bonds link the molecules into centrosymmetric dimers (Fig. 2, Table 1). There are also some additional intermolecular non-bonding contacts of the types C—H···Cl and C—H···O (Figs. 2 and 3, Table 1), and π–π stacking interactions of the type C4···C2 [C4···C2^{xiii} = 3.391 (3) Å; symmetry code: (xiii) $1-x$, $1-y$, $-1-z$]. The stacking interactions also correspond to a $Cg\cdots Cg^{xiii}$ distance of 3.7056 (11) Å, where Cg is the centroid of the ring N1/N3/C2–C6.

Experimental

The title compound, (I), was prepared as an intermediate during the preparation of a hydroxydimethoxy-Roscovitine derivative by the same procedure as described previously for Roscovitine, *i.e.* 2-{[1-(hydroxymethyl)propyl]amino}-6-benzylamino-9-isopropylpurine

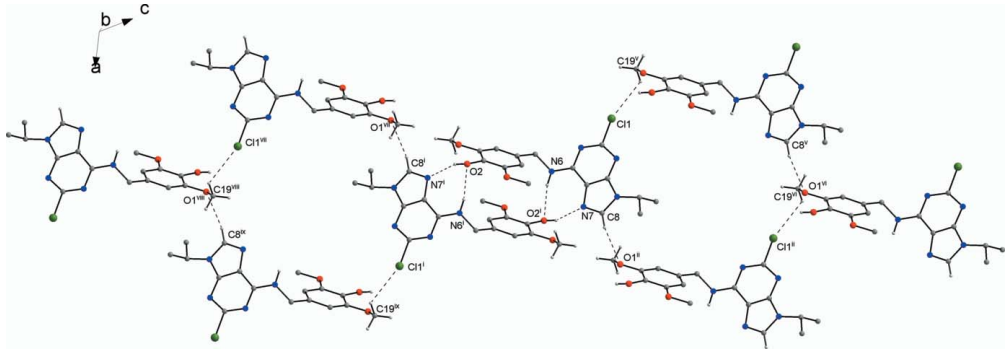


Figure 2
Part of the crystal structure of (I), showing the formation of centrosymmetric dimers connected *via* N6—H6*A*···O2^{i} and O2—H2···N7^{i} hydrogen bonds, and the C8—H8*A*···O1^{ii} and C19—H19*B*···Cl1^{iv} non-bonding interactions. These interactions are represented by dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity. [Symmetry codes: (i) $1-x$, $1-y$, $-z$; (ii) $1+x$, y, $1+z$; (iv) x, y, $z-1$; (v) x, y, $1+z$; (vi) $1+x$, y, $2+z$; (vii) $-x$, $1-y$, $-1-z$; (viii) $-x$, $1-y$, $-2-z$; (ix) $1-x$, $1-y$, $-1-z$.]

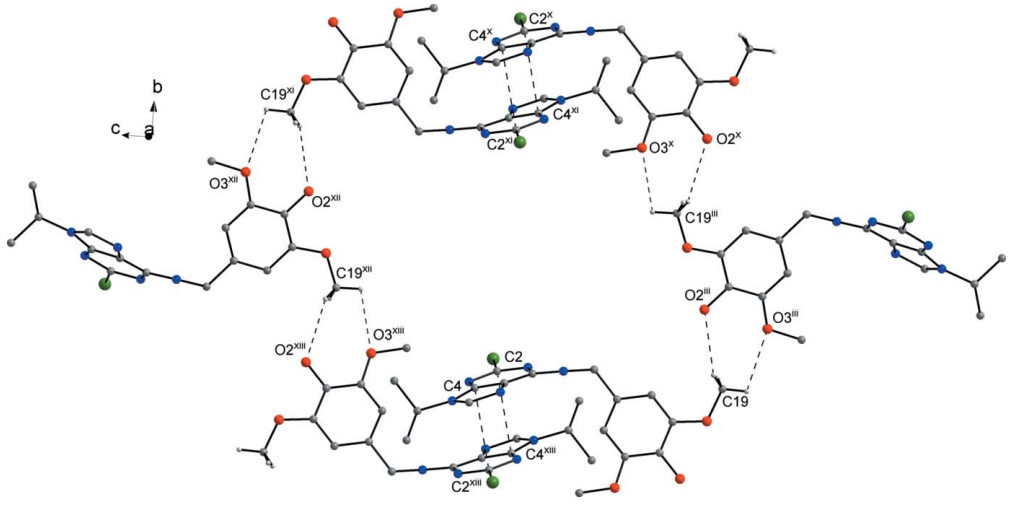


Figure 3
Part of the crystal structure of (I), showing non-bonding contacts of types C19—H19*B*···O2^{iii} and C19—H19*A*···O3^{iii}, and C4···C2^{xiii} π–π stacking interactions. These interactions are represented by dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity. [Symmetry codes: (iii) $\frac{1}{2}-x$, $\frac{1}{2}+y$, $-\frac{1}{2}-z$; (*x*) x, $1+y$, z; (xi) $1-x$, $2-y$, $1-z$; (xii) $\frac{1}{2}+x$, $\frac{3}{2}-y$, $\frac{3}{2}+z$; (xiii) $1-x$, $1-y$, $1-z$.]

(Havlíček *et al.*, 1996). The product obtained was recrystallized from hot ethanol and well developed single crystals suitable for X-ray analysis were chosen directly from the mother liquor after several days.

Crystal data

$C_{17}H_{20}ClN_5O_3$
M_r = 377.83
Monoclinic, $P2_1/n$
a = 11.8410 (8) Å
b = 13.5939 (9) Å
c = 12.1693 (8) Å
β = 118.318 (7)°
V = 1724.4 (2) Å^3
Z = 4
D_x = 1.455 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 105 (2) K
Prism, colourless
0.25 × 0.20 × 0.20 mm

Data collection

Oxford Diffraction Xcalibur2 diffractometer
ω scans
Absorption correction: none
9956 measured reflections
3045 independent reflections
2608 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.091
S = 1.07
3045 reflections
240 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0402P)^2 + 0.896P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···N7^{i}	0.84	1.94	2.717 (2)	153
N6—H6A···O2^{i}	0.88	2.19	3.042 (2)	163
C8—H8A···O1^{ii}	0.95	2.50	3.336 (2)	147
C19—H19B···O2^{iii}	0.98	2.65	3.383 (2)	132
C19—H19A···O3^{iii}	0.98	2.72	3.101 (2)	104
C19—H19B···Cl1^{iv}	0.98	2.90	3.507 (2)	121

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

All H atoms were located in a difference map and refined using a riding model, with C—H = 0.95–0.99 Å, N—H = 0.88 Å and O—H = 0.84 Å, and with U_{iso}(H) = 1.2U_{eq}(C,N) or 1.5U_{eq}(C_{methyl},O).

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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