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2-Chloro-6-[(2,6-dimethoxybenzyl)amino]-9-isopropylpurine 0.125-hydrate

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Key indicators

Single-crystal X-ray study
T = 109 K
Mean σ(C–C) = 0.003 Å
Disorder in solvent or counterion
R factor = 0.036
wR factor = 0.087
Data-to-parameter ratio = 13.0

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

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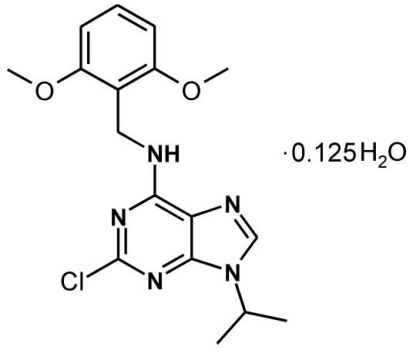
The secondary structure in the title compound, $C_{17}H_{20}ClN_5O_2\cdot0.125H_2O$, is stabilized by N—H···N hydrogen bonds that connect molecules into centrosymmetric dimers; the dimers are linked *via* a variety of intermolecular interactions.

Comment

We have recently reported the molecular structures of a number 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*a*,*b*). As a continuation of this study, we now report the structure of the title compound, (I), which represents an intermediate formed during the preparation of a roscovitine derivative. Roscovitine, also named as Seliciclib or CYC202, *i.e.* 2-{[1-(hydroxymethyl)propyl]amino}-6-benzylamino-9-isopropylpurine, is one of the more potent CDK inhibitors.



(I)

The asymmetric unit of (I) (Fig. 1) comprises a molecule of 2-chloro-6-[(2,6-dimethoxybenzyl)amino]-9-isopropylpurine and 1/8 water molecule. Aromatic cytokinins derived from 6-benzylaminopurine are generally all very similar from the chemical point of view, but various substitution patterns influence the extent of hydrogen bonds and non-bonding interatomic contacts in their crystal structures. There are three different aromatic rings in (I), *viz.* benzene (*A*), pyrimidine (*B*) and imidazole (*C*). Each of these is almost planar, the maximum deviations from the mean planes being 0.008 (2) for C11 (ring *A*), 0.007 (2) for C6 (ring *B*) and 0.009 (2)Å for C4 (ring *C*) (Brandenburg, 2006). The dihedral angle between ring *A* and the purine skeleton (rings *B* and *C*) is 61.76 (4)°, whilst rings *B* and *C* are nearly coplanar [dihedral angle 1.77 (6)°]. The spatial arrangement between ring *A* and the

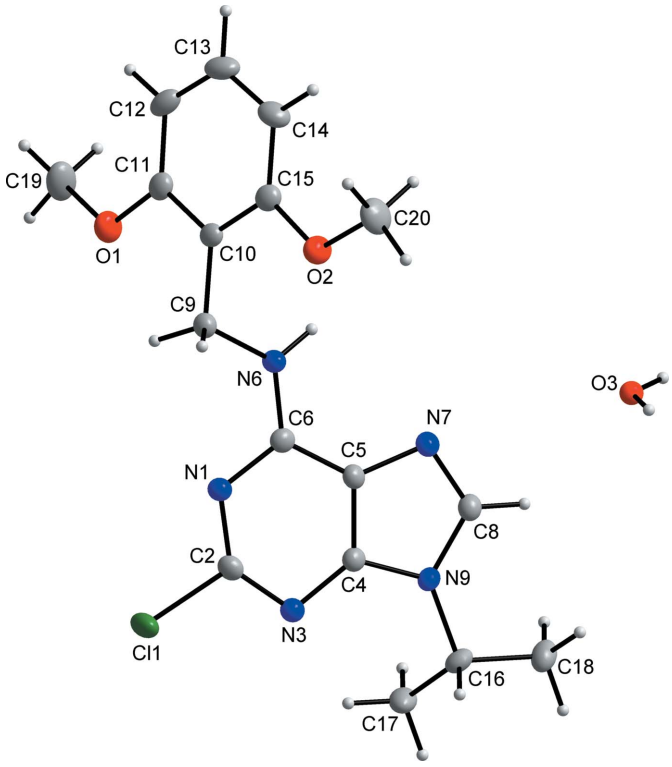


Figure 1
The molecular structure of (I), showing the atomic labelling scheme and 50% probability displacement ellipsoids. Atom O3 of the disordered water molecule is drawn as a sphere.

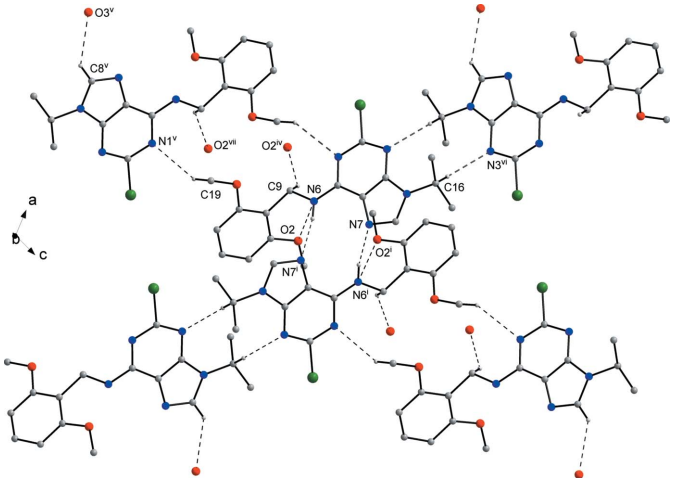


Figure 2
Part of the crystal structure of (I), showing the formation of the centrosymmetric dimers connected *via* N6—H···N7^{i} hydrogen bonds [symmetry code: (i) $-x+1, y, -z+\frac{3}{2}$] and some additional intra- and intermolecular non-bonding contacts of the types N···H—O, C—H···N and C—H···O. The view is along the [010] direction. [Symmetry codes: (iv) $1-x, -y, 1-z$; (v) $1-x, y, \frac{1}{2}-z$; (vi) $\frac{3}{2}-x, \frac{1}{2}-y, 2-z$; (vii) $x, -y, -\frac{1}{2}+z$]. These interactions are represented by dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity.

purine skeleton can be also seen from the torsion angles C6—N6—C9—C10, C9—N6—C6—N1 and N6—C9—C10—C11 [178.97 (14), 9.6 (2), and 109.15 (17)°, respectively].

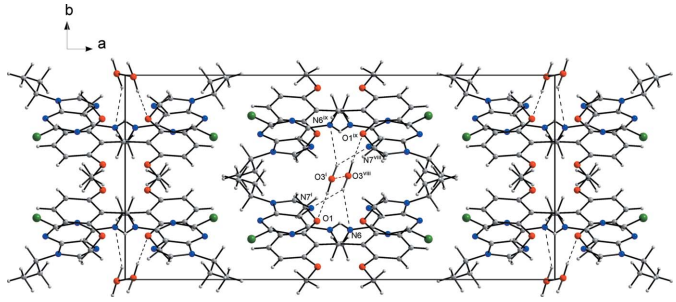


Figure 3
Part of the crystal structure of (I), viewed along the [001] direction, showing the cavities occupied by uncoordinated water molecules, and the O3^{i}—H···O1, O3^{viii}—H···N6 and O3^{viii}—H···N7^{i} hydrogen bonds (dashed lines), together with the O3^{i}···O3^{viii} contact of the disordered water molecule. [Symmetry codes: (i) $-x+1, y, -z+\frac{3}{2}$; (viii) $x, 1-y, -\frac{1}{2}+z$; (ix) $1-x, 1-y, 1-z$.]

Intermolecular N—H···N hydrogen bonds link the molecules into centrosymmetric dimers (Fig. 2 and Table 1). There are also some additional intra- and intermolecular non-bonding contacts of types N···O, C—H···N and C—H···O (Fig. 2 and Table 1), as well as O—H···O and O—H···N hydrogen bonds (Fig. 3) involving the disordered and partially occupied uncoordinated water molecules with O1···O3^{i} = 2.968 (16) Å [symmetry code: (i) $-x+1, y, -z+\frac{3}{2}$].

Experimental

Compound (I) was prepared as an intermediate during the preparation of a dimethoxy-roscovitine derivative by the same procedure as described previously for roscovitine, *i.e.*, 2-{[1-(hydroxymethyl)propyl]amino}-6-benzylamino-9-isopropylpurine (Havlíček *et al.*, 1996). The resulting product was recrystallized from hot ethanol and well developed single crystals suitable for X-ray analysis were formed after several days. They were filtered off, washed with ethanol and dried in air.

Crystal data

$C_{17}H_{20}ClN_5O_2\cdot 0.125H_2O$
M_r = 364.08
Monoclinic, $C2/c$
a = 27.159 (5) Å
b = 11.931 (2) Å
c = 11.785 (2) Å
β = 112.32 (3)°
V = 3532.6 (12) Å^3
Z = 8
D_x = 1.369 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 109 (2) K
Prism, colourless
0.35 × 0.25 × 0.25 mm

Data collection

Oxford Diffraction Xcalibur2 diffractometer
ω scans
Absorption correction: none
13528 measured reflections
3099 independent reflections
2808 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.087
S = 1.10
3099 reflections
239 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.035P)^2 + 5P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 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
N6—H6A···N7^{i}	0.88	2.13	3.0016 (19)	170
O3—H3W···O1^{i}	0.958 (15)	2.0136 (13)	2.968 (16)	173.9 (12)
O3—H3V···N6^{ii}	0.945 (14)	2.4006 (15)	3.167 (14)	138.0 (12)
O3—H3V···N7^{iii}	0.945 (14)	2.5335 (14)	3.358 (16)	145.8 (13)
C8—H8A···O3	0.95	2.65	3.429 (17)	140
C9—H9A···O2^{iv}	0.99	2.63	3.421 (2)	138
C19—H19C···N1^{v}	0.98	2.72	3.581 (3)	147
C16—H16A···N3^{vi}	1.00	2.71	3.663 (2)	160

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

All H atoms of the organic part of the molecule were included in the riding model approximation, with C—H = 0.95–1.00 and N—H = 0.88 Å, and with U_{iso}(H) = 1.2U_{eq}(C,N). Atoms of the partially occupied uncoordinated water molecule have occupancy 0.125 and are disordered over symmetry-related positions with an O3···O3^{iii} distance of 1.68 (4) Å [symmetry code: (iii) $-x+1, -y+1, -z+2$]. H atoms of the water molecule were positioned theoretically into the positions of expected O—H···O hydrogen bonds with restrained O—H bond lengths [0.84 (1) Å], and with U_{iso}(H) = 1.5U_{eq}(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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