

7-Benzyloxymethyl-9-bromo-6-chloro-9-deazapurine

Graeme J. Gainsford,* Jennifer M. Mason and Shivali A. Gulab

Industrial Research Limited, PO Box 31-310, Lower Hutt, New Zealand

Correspondence e-mail: g.gainsford@irl.cri.nz

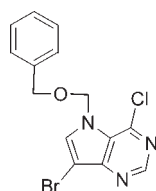
Received 18 November 2009; accepted 25 November 2009

Key indicators: single-crystal X-ray study; $T = 118$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.039; wR factor = 0.081; data-to-parameter ratio = 26.0.

The title compound, $\text{C}_{14}\text{H}_{11}\text{BrClN}_3\text{O}$, crystallizes with two independent molecules in the asymmetric unit. In the crystal, the molecules are linked by $\text{C}-\text{N}\cdots\text{Br}$ halogen bonds, as well as weak methylene $\text{C}-\text{H}\cdots\pi$, phenyl $\text{C}-\text{H}\cdots\pi$, $\text{C}-\text{H}\cdots\text{Br}$ and phenyl $\text{C}-\text{H}\cdots\text{O}$ (ether) interactions.

Related literature

For synthetic details, see Clinch *et al.* (2010). For $\text{Br}\cdots\text{N}$ halogen bonding, see: Kubicki (2004); Metrangolo *et al.* (2008). For a related structure, see: Sakore & Sobell (1969).



Experimental

Crystal data

$\text{C}_{14}\text{H}_{11}\text{BrClN}_3\text{O}$	$\gamma = 89.037$ (2)°
$M_r = 352.62$	$V = 1403.95$ (8) Å ³
Triclinic, $P\bar{1}$	$Z = 4$
$a = 7.8999$ (2) Å	Mo $K\alpha$ radiation
$b = 11.5023$ (4) Å	$\mu = 3.12$ mm ⁻¹
$c = 15.5571$ (5) Å	$T = 118$ K
$\alpha = 86.111$ (2)°	$0.41 \times 0.21 \times 0.02$ mm
$\beta = 84.564$ (2)°	

Data collection

Bruker APEXII CCD diffractometer	40101 measured reflections
Absorption correction: multi-scan (Blessing, 1995)	9403 independent reflections
$T_{\min} = 0.606$, $T_{\max} = 0.747$	6435 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.060$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$	$S = 1.01$
$wR(F^2) = 0.081$	9403 reflections

361 parameters
H-atom parameters constrained

$\Delta\rho_{\text{max}} = 0.54$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.45$ e Å⁻³

Table 1

Selected torsion angles (°).

$\text{C5}-\text{N7}-\text{C8}-\text{C9}$	0.3 (3)	$\text{C5}'-\text{N7}'-\text{C8}'-\text{C9}'$	0.1 (3)
$\text{N7}-\text{C10}-\text{O11}-\text{C12}$	-68.2 (3)	$\text{N7}'-\text{C10}'-\text{O11}'-\text{C12}'$	-77.3 (2)
$\text{C10}-\text{O11}-\text{C12}-\text{C13}$	-172.6 (2)	$\text{C10}'-\text{O11}'-\text{C12}'-\text{C13}'$	-161.5 (2)

Table 2

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C2}-\text{H2}\cdots\text{Br9}^{\text{i}}$	0.95	2.90	3.805 (3)	159
$\text{C8}-\text{H8}\cdots\text{N1}^{\text{ii}}$	0.95	2.46	3.394 (3)	166
$\text{C8}'-\text{H8}'\cdots\text{N1}'^{\text{ii}}$	0.95	2.48	3.410 (3)	166
$\text{C10}'-\text{H10C}\cdots\text{Br9}^{\text{iii}}$	0.99	2.78	3.700 (2)	156
$\text{C15}-\text{H15}\cdots\text{O11}^{\text{iv}}$	0.95	2.60	3.497 (4)	158
$\text{C10}-\text{H10A}\cdots\text{Cg1}^{\text{v}}$	0.99	2.60	3.466 (3)	146
$\text{C14}-\text{H14}\cdots\text{Cg2}^{\text{iv}}$	0.95	2.87	3.794 (3)	165

Symmetry codes: (i) $x+1, y, z$; (ii) $x-1, y, z$; (iii) $-x+1, -y, -z+1$; (iv) $x-1, y, z+1$; (v) $-x+1, -y+1, -z+1$. Cg1 and Cg2 are the centroids of the $\text{N7}, \text{C4}', \text{C5}', \text{C8}', \text{C9}'$ and $\text{C13}'-\text{C18}'$ rings, respectively.

Table 3

$\text{C}-\text{N}\cdots\text{Br}$ interactions (Å, °).

$\text{C}-\text{N}\cdots\text{Br}$	$\text{C}-\text{N}$	$\text{N}\cdots\text{Br}$	$\text{C}\cdots\text{Br}$	$\text{C}-\text{N}\cdots\text{Br}$
$\text{C2}-\text{N3}\cdots\text{Br9}^{\text{vi}}$	1.320 (3)	2.964 (2)	3.457 (3)	100.45 (17)
$\text{C2}'-\text{N3}'\cdots\text{Br}^{\text{vii}}$	1.324 (3)	3.043 (2)	3.565 (3)	102.17 (14)

Symmetry codes: (vi) $x, y-1, z$; (vii) $1+x, 1+y, z$.

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT (Bruker, 2005); data reduction: SAINT and SADABS (Bruker, 2005); program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 (Farrugia, 1997) and PLATON (Spek, 2009); software used to prepare material for publication: SHELXL97 and PLATON.

This work was supported by a New Zealand Foundation for Research Science and Technology contract [number C08X0701]. We thank Dr C. Fitchett of the University of Canterbury, New Zealand, for his assistance.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WN2368).

References

- Blessing, R. H. (1995). *Acta Cryst.* **A51**, 33–38.
 Bruker (2005). APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.
 Clinch, K., Evans, G. B., Fröhlich, R. F. G., Gutierrez, J. A., Mason, J. M., Schramm, V. L., Tyler, P. C. & Woolhouse, A. D. (2010). *Bioorg. Med. Chem.* Submitted.
 Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
 Kubicki, M. (2004). *Acta Cryst.* **B60**, 333–342.
 Metrangolo, P., Meyer, F., Pilati, T., Resnati, G. & Terraneo, G. (2008). *Angew. Chem. Int. Ed.* **47**, 6114–6127.
 Sakore, T. D. & Sobell, H. M. (1969). *J. Mol. Biol.* **43**, 77–87.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.

supplementary materials

Acta Cryst. (2010). E66, o138 [doi:10.1107/S1600536809050879]

7-Benzyloxymethyl-9-bromo-6-chloro-9-deazapurine

G. J. Gainsford, J. M. Mason and S. A. Gulab

Comment

The title compound was prepared for incorporation into potential transition state analogue inhibitors of human methylthioadenosine phosphorylase and bacterial methylthioadenosine/S-adenosylhomocysteine nucleosidase (Clinch *et al.*, 2010).

The asymmetric unit of the title compound contains two independent 7-benzyloxymethyl-9-bromo-6-chloro-9-deazapurine molecules (Fig. 1, Table 1) which are nearly identical. The molecular labels are related by the addition of a prime (*e.g.* molecule 1, Br9 and molecule 2, Br9'). The overlay figure (Fig. 2) illustrates the rmsd fit of 0.077 Å, with maximum deviation at the Cl6 (C16') atom sites of 0.274 Å. The five-membered rings (*i.e.* C4,C5,C8,C9,N7) are planar (maximum deviations 0.004 (2), 0.001 (2) Å), as are the fused six-membered rings (C4,C5,C6,N1,C2,N3) with maximum deviations 0.017 (2), 0.010 (2) Å; the angles between these two planes are 2.62 (13) and 1.00 (12)°, respectively. The dihedral angles between the mean planes through the deazapurine ring systems and the phenyl rings are 52.76 (12) and 58.02 (11)°.

Torsion angles (Table 1) are similar to those observed earlier in 9-ethyl-8-bromohypoxanthine (Sakore & Sobell, 1969).

Lattice binding is provided principally by Br⋯N halogen bonding (Metrangolo *et al.*, 2008; Kubicki, 2004) between the two independent molecules, forming layers parallel to the (0,1,2) plane (Table 3; Fig. 3). Inter-layer links are provided by weak methylene C–H⋯π, phenyl C–H⋯π, C–H⋯Br, and phenyl C–H⋯O(ether) interactions (Table 2). In Table 2, Cg1 and Cg2 are the centroids of N7',C4',C5',C8',C9' and C13'–C18', respectively.

Experimental

Synthetic details are given in Clinch *et al.* (2010).

Refinement

Data was restricted to $2\theta = 64^\circ$. All carbon-bound H atoms were constrained to their expected geometries [C–H 0.95, 0.99 Å] and refined with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{parent atom})$. All non-hydrogen atoms were refined with anisotropic thermal parameters.

Figures

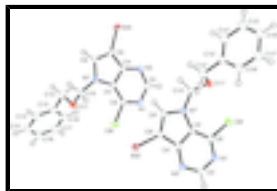


Fig. 1. Asymmetric unit of the title compound (Farrugia, 1997).

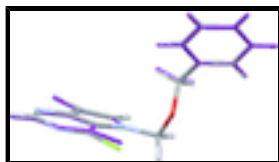


Fig. 2. Overlap view of the two molecules (Mercury 2.3, Macrae *et al.* (2008)). Single coloured (purple) molecule is primed molecule 2.

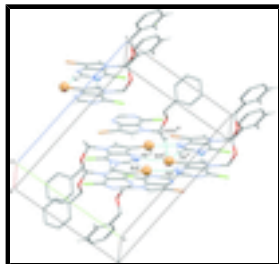


Fig. 3. A packing view (Mercury 2.3, Macrae *et al.* (2008)) of the unit cell, highlighting the layers formed by Br...N bonding in the crystal structure (Table 3). Some of the other weak interactions (Table 2) are shown, with contact atoms in ball mode; only H atoms involved in short contacts are included. Symmetry codes: (i) $x, 1 + y, z$ (ii) $x - 1, y, z$ (iii) $1 - x, 1 - y, 1 - z$ (iv) $x - 1, 1 + y, z$.

5-Benzyloxymethyl-7-bromo-4-chloro-5H-pyrrolo[3,2-d]pyrimidine

Crystal data

$C_{14}H_{11}BrClN_3O$	$Z = 4$
$M_r = 352.62$	$F(000) = 704$
Triclinic, $P\bar{1}$	$D_x = 1.668 \text{ Mg m}^{-3}$
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$a = 7.8999 (2) \text{ \AA}$	Cell parameters from 7237 reflections
$b = 11.5023 (4) \text{ \AA}$	$\theta = 2.3\text{--}28.2^\circ$
$c = 15.5571 (5) \text{ \AA}$	$\mu = 3.12 \text{ mm}^{-1}$
$\alpha = 86.111 (2)^\circ$	$T = 118 \text{ K}$
$\beta = 84.564 (2)^\circ$	Plate, colourless
$\gamma = 89.037 (2)^\circ$	$0.41 \times 0.21 \times 0.02 \text{ mm}$
$V = 1403.95 (8) \text{ \AA}^3$	

Data collection

Bruker APEXII CCD diffractometer	9403 independent reflections
Radiation source: fine-focus sealed tube graphite	6435 reflections with $I > 2\sigma(I)$
Detector resolution: $8.333 \text{ pixels mm}^{-1}$	$R_{\text{int}} = 0.060$
φ and ω scans	$\theta_{\text{max}} = 32.0^\circ, \theta_{\text{min}} = 2.8^\circ$
Absorption correction: multi-scan (Blessing, 1995)	$h = -11 \rightarrow 11$
$T_{\text{min}} = 0.606, T_{\text{max}} = 0.747$	$k = -17 \rightarrow 17$
40101 measured reflections	$l = -23 \rightarrow 23$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map

$$R[F^2 > 2\sigma(F^2)] = 0.039$$

$$wR(F^2) = 0.081$$

$$S = 1.01$$

9403 reflections

361 parameters

0 restraints

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0269P)^2 + 0.5143P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} = 0.002$$

$$\Delta\rho_{\max} = 0.54 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.44 \text{ e } \text{\AA}^{-3}$$

Special details

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Br9	0.07829 (3)	-0.13291 (2)	0.558649 (16)	0.02240 (6)
Cl6	0.55993 (8)	0.24034 (6)	0.75030 (4)	0.03239 (15)
N1	0.6523 (2)	0.0585 (2)	0.66667 (15)	0.0294 (5)
C2	0.6243 (3)	-0.0336 (3)	0.62176 (19)	0.0337 (7)
H2	0.7222	-0.0760	0.6010	0.040*
N3	0.4768 (2)	-0.07252 (19)	0.60296 (14)	0.0271 (5)
C4	0.3419 (3)	-0.0102 (2)	0.63424 (15)	0.0204 (5)
C5	0.3551 (3)	0.0859 (2)	0.68449 (15)	0.0194 (5)
C6	0.5190 (3)	0.1184 (2)	0.69711 (16)	0.0226 (5)
N7	0.1938 (2)	0.12957 (17)	0.70536 (13)	0.0200 (4)
C8	0.0815 (3)	0.0629 (2)	0.66795 (15)	0.0207 (5)
H8	-0.0383	0.0745	0.6720	0.025*
C9	0.1670 (3)	-0.0223 (2)	0.62432 (15)	0.0187 (5)
C10	0.1459 (3)	0.2263 (2)	0.75875 (16)	0.0244 (5)
H10A	0.2034	0.2975	0.7321	0.029*
H10B	0.0218	0.2400	0.7592	0.029*

supplementary materials

O11	0.1867 (2)	0.20846 (15)	0.84366 (11)	0.0265 (4)
C12	0.0892 (4)	0.1195 (3)	0.89288 (19)	0.0465 (8)
H12A	0.1187	0.0426	0.8702	0.056*
H12B	-0.0336	0.1344	0.8888	0.056*
C13	0.1286 (4)	0.1199 (3)	0.98518 (18)	0.0343 (6)
C14	0.0310 (4)	0.1865 (2)	1.0417 (2)	0.0362 (7)
H14	-0.0592	0.2333	1.0215	0.043*
C15	0.0629 (4)	0.1861 (3)	1.12723 (19)	0.0377 (7)
H15	-0.0061	0.2318	1.1656	0.045*
C16	0.1938 (4)	0.1200 (3)	1.1573 (2)	0.0395 (7)
H16	0.2158	0.1200	1.2163	0.047*
C17	0.2929 (4)	0.0539 (3)	1.1013 (2)	0.0446 (8)
H17	0.3841	0.0082	1.1215	0.054*
C18	0.2602 (4)	0.0536 (3)	1.0156 (2)	0.0410 (7)
H18	0.3289	0.0073	0.9773	0.049*
Br9'	0.59248 (3)	0.72770 (2)	0.500913 (16)	0.02152 (6)
Cl6'	1.15241 (7)	0.36355 (6)	0.28870 (4)	0.02845 (14)
N1'	1.2108 (2)	0.54047 (18)	0.37585 (13)	0.0228 (4)
C2'	1.1644 (3)	0.6305 (2)	0.42386 (17)	0.0257 (5)
H2'	1.2547	0.6744	0.4411	0.031*
N3'	1.0089 (2)	0.66584 (18)	0.45013 (13)	0.0223 (4)
C4'	0.8857 (3)	0.6018 (2)	0.42261 (15)	0.0182 (5)
C5'	0.9182 (3)	0.5069 (2)	0.37055 (14)	0.0175 (4)
C6'	1.0881 (3)	0.4791 (2)	0.34985 (15)	0.0197 (5)
N7'	0.7642 (2)	0.46355 (17)	0.35311 (12)	0.0190 (4)
C8'	0.6378 (3)	0.5300 (2)	0.39368 (15)	0.0199 (5)
H8'	0.5194	0.5186	0.3921	0.024*
C9'	0.7066 (3)	0.6140 (2)	0.43611 (15)	0.0190 (5)
C10'	0.7317 (3)	0.3645 (2)	0.30284 (16)	0.0225 (5)
H10C	0.8014	0.2971	0.3222	0.027*
H10D	0.6107	0.3427	0.3150	0.027*
O11'	0.76778 (19)	0.38677 (15)	0.21388 (11)	0.0250 (4)
C12'	0.6421 (3)	0.4559 (3)	0.17201 (17)	0.0320 (6)
H12C	0.6546	0.5392	0.1820	0.038*
H12D	0.5264	0.4312	0.1953	0.038*
C13'	0.6698 (3)	0.4377 (2)	0.07720 (17)	0.0271 (6)
C14'	0.5969 (3)	0.3438 (3)	0.04404 (19)	0.0361 (7)
H14'	0.5247	0.2928	0.0810	0.043*
C15'	0.6289 (4)	0.3240 (3)	-0.04277 (19)	0.0408 (7)
H15'	0.5790	0.2592	-0.0652	0.049*
C16'	0.7331 (3)	0.3979 (3)	-0.09698 (19)	0.0371 (7)
H16'	0.7555	0.3842	-0.1565	0.045*
C17'	0.8045 (4)	0.4920 (3)	-0.06387 (19)	0.0397 (7)
H17'	0.8758	0.5434	-0.1008	0.048*
C18'	0.7726 (3)	0.5115 (3)	0.02262 (18)	0.0357 (7)
H18'	0.8221	0.5766	0.0448	0.043*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br9	0.01902 (11)	0.02210 (13)	0.02763 (14)	-0.00162 (8)	-0.00850 (9)	-0.00370 (10)
Cl6	0.0302 (3)	0.0355 (4)	0.0336 (4)	-0.0109 (3)	-0.0062 (3)	-0.0109 (3)
N1	0.0160 (9)	0.0408 (14)	0.0332 (13)	-0.0029 (8)	-0.0051 (8)	-0.0111 (11)
C2	0.0144 (11)	0.0463 (18)	0.0423 (18)	0.0040 (10)	-0.0035 (10)	-0.0164 (14)
N3	0.0163 (9)	0.0343 (13)	0.0323 (13)	0.0021 (8)	-0.0042 (8)	-0.0107 (10)
C4	0.0173 (10)	0.0242 (13)	0.0198 (13)	-0.0013 (8)	-0.0034 (8)	0.0004 (10)
C5	0.0167 (10)	0.0251 (13)	0.0166 (12)	-0.0010 (8)	-0.0030 (8)	-0.0005 (10)
C6	0.0216 (11)	0.0289 (14)	0.0182 (12)	-0.0054 (9)	-0.0058 (9)	-0.0025 (10)
N7	0.0170 (9)	0.0223 (11)	0.0213 (11)	0.0000 (7)	-0.0034 (7)	-0.0049 (8)
C8	0.0149 (10)	0.0263 (13)	0.0214 (13)	-0.0008 (8)	-0.0044 (8)	-0.0019 (10)
C9	0.0174 (10)	0.0204 (12)	0.0191 (12)	-0.0024 (8)	-0.0052 (8)	-0.0022 (9)
C10	0.0272 (12)	0.0232 (13)	0.0230 (14)	0.0031 (9)	-0.0011 (9)	-0.0045 (11)
O11	0.0326 (9)	0.0286 (10)	0.0185 (9)	-0.0086 (7)	-0.0009 (7)	-0.0031 (8)
C12	0.062 (2)	0.051 (2)	0.0268 (17)	-0.0310 (16)	-0.0012 (14)	0.0029 (14)
C13	0.0421 (15)	0.0349 (16)	0.0258 (15)	-0.0169 (12)	-0.0013 (11)	0.0004 (12)
C14	0.0421 (16)	0.0290 (16)	0.0390 (18)	-0.0022 (12)	-0.0110 (13)	-0.0023 (13)
C15	0.0475 (17)	0.0343 (17)	0.0324 (17)	-0.0003 (13)	-0.0042 (13)	-0.0100 (13)
C16	0.0494 (17)	0.0420 (18)	0.0281 (16)	-0.0092 (14)	-0.0097 (13)	0.0007 (14)
C17	0.0379 (16)	0.049 (2)	0.047 (2)	-0.0003 (14)	-0.0113 (14)	0.0032 (16)
C18	0.0364 (15)	0.0460 (19)	0.0393 (19)	-0.0044 (13)	0.0072 (13)	-0.0086 (15)
Br9'	0.01786 (10)	0.02498 (14)	0.02185 (13)	0.00062 (8)	-0.00097 (8)	-0.00401 (10)
Cl6'	0.0234 (3)	0.0316 (3)	0.0305 (4)	0.0022 (2)	0.0014 (2)	-0.0094 (3)
N1'	0.0177 (9)	0.0271 (12)	0.0235 (12)	-0.0024 (8)	-0.0023 (7)	-0.0005 (9)
C2'	0.0148 (10)	0.0315 (14)	0.0315 (15)	-0.0057 (9)	-0.0052 (9)	-0.0012 (11)
N3'	0.0187 (9)	0.0254 (11)	0.0235 (11)	-0.0034 (8)	-0.0044 (8)	-0.0029 (9)
C4'	0.0177 (10)	0.0192 (12)	0.0178 (12)	-0.0013 (8)	-0.0034 (8)	0.0009 (9)
C5'	0.0166 (9)	0.0222 (12)	0.0142 (11)	-0.0025 (8)	-0.0035 (8)	-0.0017 (9)
C6'	0.0199 (10)	0.0236 (12)	0.0151 (12)	0.0011 (9)	0.0007 (8)	0.0001 (10)
N7'	0.0151 (8)	0.0243 (11)	0.0187 (11)	-0.0031 (7)	-0.0040 (7)	-0.0045 (8)
C8'	0.0122 (9)	0.0282 (13)	0.0189 (12)	-0.0001 (8)	-0.0013 (8)	0.0021 (10)
C9'	0.0188 (10)	0.0224 (12)	0.0158 (12)	-0.0008 (8)	-0.0014 (8)	-0.0007 (9)
C10'	0.0236 (11)	0.0229 (13)	0.0219 (13)	-0.0037 (9)	-0.0056 (9)	-0.0037 (10)
O11'	0.0226 (8)	0.0349 (10)	0.0187 (9)	0.0060 (7)	-0.0058 (6)	-0.0064 (8)
C12'	0.0312 (13)	0.0412 (17)	0.0249 (15)	0.0107 (11)	-0.0078 (10)	-0.0060 (12)
C13'	0.0260 (12)	0.0348 (15)	0.0219 (14)	0.0063 (10)	-0.0079 (10)	-0.0055 (11)
C14'	0.0411 (15)	0.0394 (17)	0.0282 (16)	-0.0068 (12)	-0.0063 (12)	0.0005 (13)
C15'	0.0510 (17)	0.0438 (19)	0.0299 (17)	-0.0083 (14)	-0.0098 (13)	-0.0089 (14)
C16'	0.0405 (15)	0.0497 (19)	0.0226 (15)	0.0026 (13)	-0.0079 (11)	-0.0064 (13)
C17'	0.0404 (16)	0.052 (2)	0.0272 (16)	-0.0108 (13)	-0.0059 (12)	0.0008 (14)
C18'	0.0412 (15)	0.0386 (17)	0.0294 (16)	-0.0075 (12)	-0.0114 (12)	-0.0052 (13)

Geometric parameters (\AA , $^\circ$)

Br9—C9	1.870 (2)	Br9'—C9'	1.873 (2)
Cl6—C6	1.726 (2)	Cl6'—C6'	1.728 (2)

supplementary materials

N1—C6	1.316 (3)	N1'—C6'	1.318 (3)
N1—C2	1.341 (3)	N1'—C2'	1.344 (3)
C2—N3	1.320 (3)	C2'—N3'	1.324 (3)
C2—H2	0.9500	C2'—H2'	0.9500
N3—C4	1.345 (3)	N3'—C4'	1.348 (3)
C4—C5	1.407 (3)	C4'—C5'	1.409 (3)
C4—C9	1.415 (3)	C4'—C9'	1.417 (3)
C5—N7	1.380 (3)	C5'—N7'	1.379 (3)
C5—C6	1.390 (3)	C5'—C6'	1.388 (3)
N7—C8	1.377 (3)	N7'—C8'	1.379 (3)
N7—C10	1.457 (3)	N7'—C10'	1.465 (3)
C8—C9	1.363 (3)	C8'—C9'	1.355 (3)
C8—H8	0.9500	C8'—H8'	0.9500
C10—O11	1.390 (3)	C10'—O11'	1.394 (3)
C10—H10A	0.9900	C10'—H10C	0.9900
C10—H10B	0.9900	C10'—H10D	0.9900
O11—C12	1.427 (3)	O11'—C12'	1.438 (3)
C12—C13	1.498 (4)	C12'—C13'	1.499 (4)
C12—H12A	0.9900	C12'—H12C	0.9900
C12—H12B	0.9900	C12'—H12D	0.9900
C13—C14	1.378 (4)	C13'—C18'	1.377 (4)
C13—C18	1.380 (4)	C13'—C14'	1.385 (4)
C14—C15	1.377 (4)	C14'—C15'	1.384 (4)
C14—H14	0.9500	C14'—H14'	0.9500
C15—C16	1.372 (4)	C15'—C16'	1.381 (4)
C15—H15	0.9500	C15'—H15'	0.9500
C16—C17	1.376 (4)	C16'—C17'	1.380 (4)
C16—H16	0.9500	C16'—H16'	0.9500
C17—C18	1.382 (4)	C17'—C18'	1.378 (4)
C17—H17	0.9500	C17'—H17'	0.9500
C18—H18	0.9500	C18'—H18'	0.9500
C6—N1—C2	117.6 (2)	C6'—N1'—C2'	117.19 (19)
N3—C2—N1	127.8 (2)	N3'—C2'—N1'	128.2 (2)
N3—C2—H2	116.1	N3'—C2'—H2'	115.9
N1—C2—H2	116.1	N1'—C2'—H2'	115.9
C2—N3—C4	113.9 (2)	C2'—N3'—C4'	113.4 (2)
N3—C4—C5	123.5 (2)	N3'—C4'—C5'	123.7 (2)
N3—C4—C9	130.0 (2)	N3'—C4'—C9'	129.7 (2)
C5—C4—C9	106.56 (19)	C5'—C4'—C9'	106.58 (19)
N7—C5—C6	135.4 (2)	N7'—C5'—C6'	135.8 (2)
N7—C5—C4	108.42 (18)	N7'—C5'—C4'	108.19 (18)
C6—C5—C4	116.1 (2)	C6'—C5'—C4'	116.05 (19)
N1—C6—C5	121.1 (2)	N1'—C6'—C5'	121.4 (2)
N1—C6—C16	116.47 (17)	N1'—C6'—C16'	115.92 (17)
C5—C6—C16	122.42 (19)	C5'—C6'—C16'	122.65 (18)
C8—N7—C5	107.50 (19)	C8'—N7'—C5'	107.52 (19)
C8—N7—C10	124.96 (19)	C8'—N7'—C10'	123.80 (18)
C5—N7—C10	127.53 (19)	C5'—N7'—C10'	128.67 (19)
C9—C8—N7	110.16 (19)	C9'—C8'—N7'	110.33 (19)

C9—C8—H8	124.9	C9'—C8'—H8'	124.8
N7—C8—H8	124.9	N7'—C8'—H8'	124.8
C8—C9—C4	107.4 (2)	C8'—C9'—C4'	107.4 (2)
C8—C9—Br9	128.03 (17)	C8'—C9'—Br9'	127.83 (17)
C4—C9—Br9	124.56 (18)	C4'—C9'—Br9'	124.78 (17)
O11—C10—N7	113.9 (2)	O11'—C10'—N7'	113.58 (19)
O11—C10—H10A	108.8	O11'—C10'—H10C	108.9
N7—C10—H10A	108.8	N7'—C10'—H10C	108.9
O11—C10—H10B	108.8	O11'—C10'—H10D	108.9
N7—C10—H10B	108.8	N7'—C10'—H10D	108.9
H10A—C10—H10B	107.7	H10C—C10'—H10D	107.7
C10—O11—C12	113.6 (2)	C10'—O11'—C12'	115.05 (18)
O11—C12—C13	108.2 (2)	O11'—C12'—C13'	107.2 (2)
O11—C12—H12A	110.1	O11'—C12'—H12C	110.3
C13—C12—H12A	110.1	C13'—C12'—H12C	110.3
O11—C12—H12B	110.1	O11'—C12'—H12D	110.3
C13—C12—H12B	110.1	C13'—C12'—H12D	110.3
H12A—C12—H12B	108.4	H12C—C12'—H12D	108.5
C14—C13—C18	118.8 (3)	C18'—C13'—C14'	119.2 (3)
C14—C13—C12	119.8 (3)	C18'—C13'—C12'	120.5 (2)
C18—C13—C12	121.4 (3)	C14'—C13'—C12'	120.2 (3)
C15—C14—C13	120.7 (3)	C15'—C14'—C13'	120.2 (3)
C15—C14—H14	119.7	C15'—C14'—H14'	119.9
C13—C14—H14	119.7	C13'—C14'—H14'	119.9
C16—C15—C14	120.4 (3)	C16'—C15'—C14'	120.3 (3)
C16—C15—H15	119.8	C16'—C15'—H15'	119.9
C14—C15—H15	119.8	C14'—C15'—H15'	119.9
C15—C16—C17	119.3 (3)	C17'—C16'—C15'	119.4 (3)
C15—C16—H16	120.3	C17'—C16'—H16'	120.3
C17—C16—H16	120.3	C15'—C16'—H16'	120.3
C16—C17—C18	120.3 (3)	C18'—C17'—C16'	120.2 (3)
C16—C17—H17	119.9	C18'—C17'—H17'	119.9
C18—C17—H17	119.9	C16'—C17'—H17'	119.9
C13—C18—C17	120.5 (3)	C13'—C18'—C17'	120.7 (3)
C13—C18—H18	119.8	C13'—C18'—H18'	119.7
C17—C18—H18	119.8	C17'—C18'—H18'	119.7
C6—N1—C2—N3	0.7 (5)	C6'—N1'—C2'—N3'	1.0 (4)
N1—C2—N3—C4	-0.4 (4)	N1'—C2'—N3'—C4'	-1.0 (4)
C2—N3—C4—C5	-1.7 (4)	C2'—N3'—C4'—C5'	-0.4 (3)
C2—N3—C4—C9	177.1 (3)	C2'—N3'—C4'—C9'	-178.8 (2)
N3—C4—C5—N7	179.8 (2)	N3'—C4'—C5'—N7'	-178.6 (2)
C9—C4—C5—N7	0.8 (3)	C9'—C4'—C5'—N7'	0.1 (3)
N3—C4—C5—C6	3.3 (4)	N3'—C4'—C5'—C6'	1.7 (3)
C9—C4—C5—C6	-175.7 (2)	C9'—C4'—C5'—C6'	-179.6 (2)
C2—N1—C6—C5	1.2 (4)	C2'—N1'—C6'—C5'	0.5 (3)
C2—N1—C6—Cl6	-177.1 (2)	C2'—N1'—C6'—Cl6'	-179.70 (19)
N7—C5—C6—N1	-178.2 (3)	N7'—C5'—C6'—N1'	178.7 (2)
C4—C5—C6—N1	-3.0 (4)	C4'—C5'—C6'—N1'	-1.7 (3)
N7—C5—C6—Cl6	0.0 (4)	N7'—C5'—C6'—Cl6'	-1.0 (4)

supplementary materials

C4—C5—C6—C16	175.20 (18)	C4'—C5'—C6'—C16'	178.51 (17)
C6—C5—N7—C8	174.8 (3)	C6'—C5'—N7'—C8'	179.5 (3)
C4—C5—N7—C8	-0.7 (3)	C4'—C5'—N7'—C8'	-0.1 (3)
C6—C5—N7—C10	-6.7 (4)	C6'—C5'—N7'—C10'	0.6 (4)
C4—C5—N7—C10	177.9 (2)	C4'—C5'—N7'—C10'	-179.0 (2)
C5—N7—C8—C9	0.3 (3)	C5'—N7'—C8'—C9'	0.1 (3)
C10—N7—C8—C9	-178.3 (2)	C10'—N7'—C8'—C9'	179.0 (2)
N7—C8—C9—C4	0.2 (3)	N7'—C8'—C9'—C4'	0.0 (3)
N7—C8—C9—Br9	-177.46 (17)	N7'—C8'—C9'—Br9'	179.70 (17)
N3—C4—C9—C8	-179.5 (3)	N3'—C4'—C9'—C8'	178.6 (2)
C5—C4—C9—C8	-0.6 (3)	C5'—C4'—C9'—C8'	-0.1 (3)
N3—C4—C9—Br9	-1.8 (4)	N3'—C4'—C9'—Br9'	-1.1 (4)
C5—C4—C9—Br9	177.16 (17)	C5'—C4'—C9'—Br9'	-179.77 (17)
C8—N7—C10—O11	116.7 (2)	C8'—N7'—C10'—O11'	107.8 (2)
C5—N7—C10—O11	-61.7 (3)	C5'—N7'—C10'—O11'	-73.4 (3)
N7—C10—O11—C12	-68.2 (3)	N7'—C10'—O11'—C12'	-77.3 (2)
C10—O11—C12—C13	-172.6 (2)	C10'—O11'—C12'—C13'	-161.5 (2)
O11—C12—C13—C14	91.4 (3)	O11'—C12'—C13'—C18'	-92.3 (3)
O11—C12—C13—C18	-89.4 (3)	O11'—C12'—C13'—C14'	85.3 (3)
C18—C13—C14—C15	-0.7 (4)	C18'—C13'—C14'—C15'	0.7 (4)
C12—C13—C14—C15	178.6 (3)	C12'—C13'—C14'—C15'	-177.0 (3)
C13—C14—C15—C16	0.7 (4)	C13'—C14'—C15'—C16'	-0.3 (5)
C14—C15—C16—C17	-0.2 (5)	C14'—C15'—C16'—C17'	-0.2 (5)
C15—C16—C17—C18	-0.3 (5)	C15'—C16'—C17'—C18'	0.3 (4)
C14—C13—C18—C17	0.2 (4)	C14'—C13'—C18'—C17'	-0.6 (4)
C12—C13—C18—C17	-179.0 (3)	C12'—C13'—C18'—C17'	177.0 (3)
C16—C17—C18—C13	0.3 (5)	C16'—C17'—C18'—C13'	0.1 (5)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C2—H2 \cdots Br9 ⁱ	0.95	2.90	3.805 (3)	159
C8—H8 \cdots N1 ⁱⁱ	0.95	2.46	3.394 (3)	166
C8'—H8' \cdots N1 ⁱⁱⁱ	0.95	2.48	3.410 (3)	166
C10'—H10C \cdots Br9 ⁱⁱⁱ	0.99	2.78	3.700 (2)	156
C15—H15 \cdots O11 ^{iv}	0.95	2.60	3.497 (4)	158
C10—H10A \cdots Cg1 ^v	0.99	2.60	3.466 (3)	146
C14—H14 \cdots Cg2 ^{iv}	0.95	2.87	3.794 (3)	165

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

Table 3

$C-N\cdots Br$ interactions (\AA , $^\circ$)

$C-N\cdots Br$	$C-N$	$N\cdots Br$	$C\cdots Br$	$C-N\cdots Br$
C2—N3 \cdots Br9 ^{vi}	1.320 (3)	2.964 (2)	3.457 (3)	100.45 (17)
C2'—N3' \cdots Br ^{vii}	1.324 (3)	3.043 (2)	3.565 (3)	102.17 (14)

Symmetry codes: (vi) $x, y-1, z$; (vii) $1+x, 1+y, z$.

Fig. 1

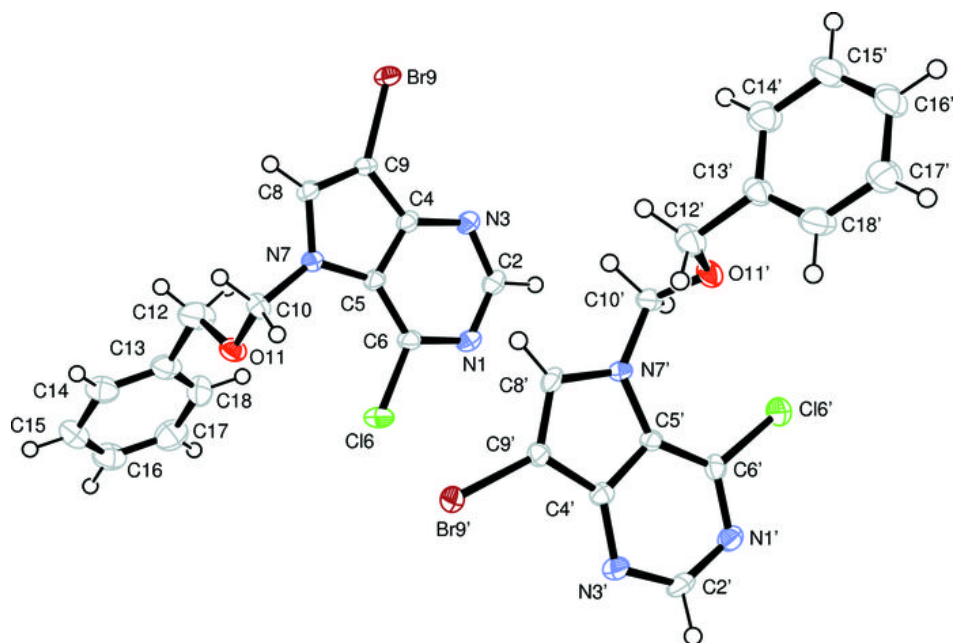


Fig. 2

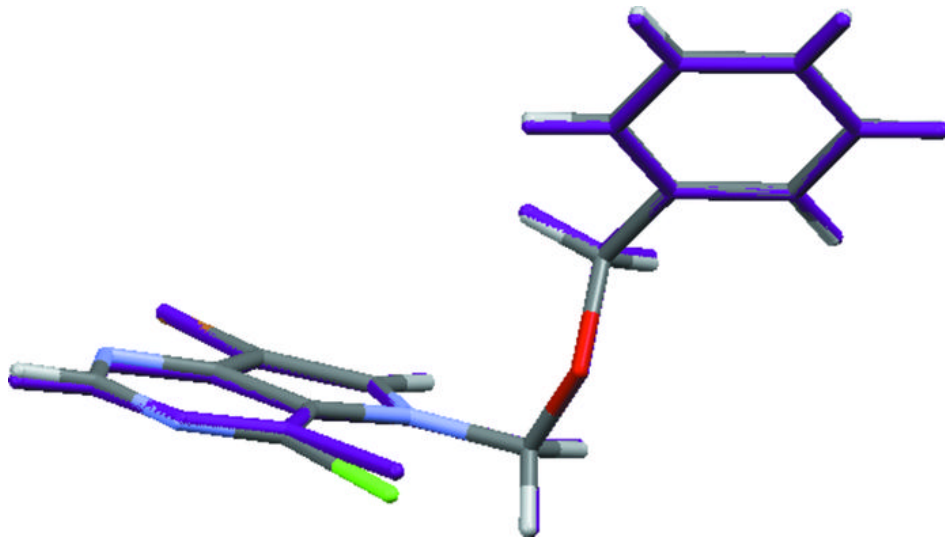


Fig. 3

