



Received 10 September 2018

Accepted 1 April 2019

Edited by P. Bombicz, Hungarian Academy of
Sciences, Hungary

‡ Contributed equally to this work.

Keywords: crystal structure; oxetanacin; cyclobutanone; cyclobutanol; HIV; hydrogen bonding; offset π - π interactions; supramolecular framework.**CCDC references:** 1907200; 1907199;
1907198**Supporting information:** this article has
supporting information at journals.iucr.org/e

Crystal structures of the synthetic intermediate 3-[(6-chloro-7*H*-purin-7-yl)methyl]cyclobutan-1-one, and of two oxetanocin derivatives: 3-[(6-chloro-8,9-dihydro-7*H*-purin-7-yl)methyl]cyclobutan-1-ol and 3-[(6-chloro-9*H*-purin-9-yl)methyl]cyclobutan-1-ol

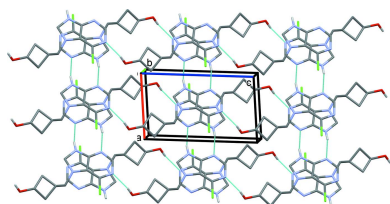
Ayat Yaseen,‡ Muhammad Murtaza Hassan,† Edward Lee-Ruff and Gerald F. Audette*

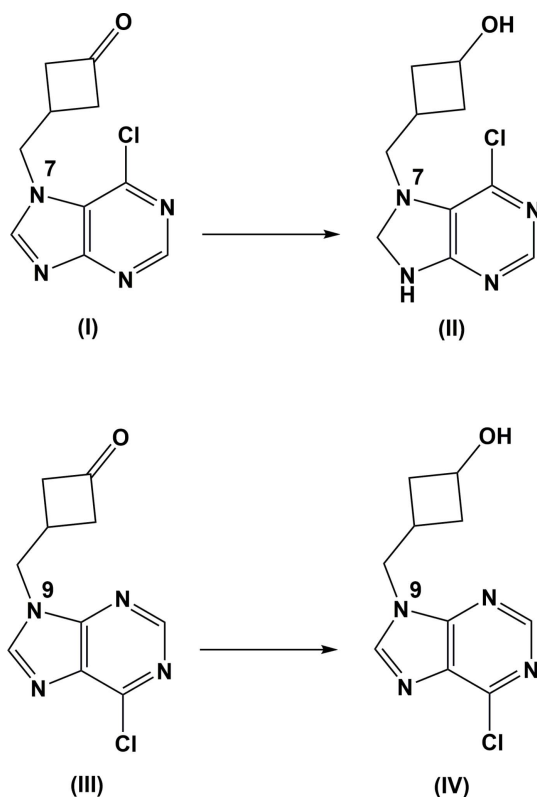
Department of Chemistry, York University, 4700 Keele St., Toronto, ON, M3J 1P3, Canada. *Correspondence e-mail: audette@yorku.ca

The crystal structures of an intermediate, C₁₀H₉ClN₄O, 3-[(6-chloro-7*H*-purin-7-yl)methyl]cyclobutan-1-one (I), and two N-7 and N-9 regioisomeric oxetanocin nucleoside analogs, C₁₀H₁₃ClN₄O, 3-[(6-chloro-8,9-dihydro-7*H*-purin-7-yl)methyl]cyclobutan-1-ol (II) and C₁₀H₁₁ClN₄O, 3-[(6-chloro-9*H*-purin-9-yl)methyl]cyclobutan-1-ol (IV), are reported. The crystal structures of the nucleoside analogs confirmed the reduction of the N-7- and N-9-substituted cyclobutanones with LiAl(OtBu)₃ to occur with facial selectivity, yielding *cis*-nucleosides analogs similar to those found in nature. Reduction of the purine ring of the N-7 cyclobutanone to a dihydropurine was observed for compound (II) but not for the purine ring of the N-9 cyclobutanone on formation of compound (IV). In the crystal of (I), molecules are linked by a weak Cl···O interaction, forming a 2₁ helix along [010]. The helices are linked by offset π - π interactions [intercentroid distance = 3.498 (1) Å], forming layers parallel to (101). In the crystal of (II), molecules are linked by pairs of O—H···N hydrogen bonds, forming inversion dimers with an R₂²(8) ring motif. The dimers are linked by O—H···N hydrogen bonds, forming chains along [001], which in turn are linked by C—H··· π and offset π - π interactions [intercentroid distance = 3.509 (1) Å], forming slabs parallel to the *ac* plane. In the crystal of (IV), molecules are linked by O—H···N hydrogen bonds, forming chains along [101]. The chains are linked by C—H···N and C—H···O hydrogen bonds and C—H··· π and offset π - π interactions [intercentroid distance = 3.364 (1) Å], forming a supramolecular framework.

1. Chemical context

Derivatives of naturally occurring nucleotides are an emerging class of antiviral therapeutics that are used to target tumors, herpes virus and the human immunodeficiency virus (HIV) (De Clercq, 2005). The development of new and different nucleoside analogs is important in combating drug-resistant mutants and increasing therapeutic effectivity. The naturally occurring oxetanocin A, a nucleoside analog, demonstrated efficacy against herpes and HIV (Hoshino *et al.*, 1987). Further exploration of oxetanocin A derivatives such as cyclobut-A and cyclobut-B (Lobucavir) represented an increase in potency and metabolic stability (Hoshino *et al.*, 1987; Bisacchi *et al.*, 1991). The current study focuses on the structural characterization of two nucleoside analogs, (II) and (IV), as well as the purinyl-cyclobutanone intermediate (I), prior to reduction.





2. Structural commentary

The molecular structures of compounds (I), (II) and (IV) are illustrated in Figs. 1, 2 and 3, respectively. In compounds (I) and (II) there is a short intramolecular C—H...Cl interaction present (Tables 1 and 2, respectively).

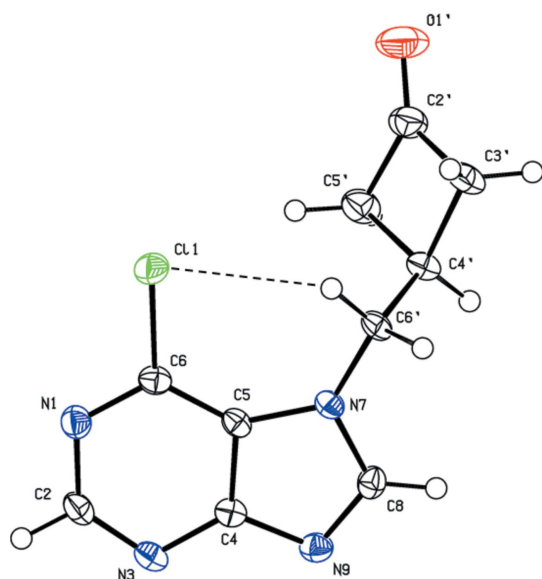


Figure 1
The molecular structure of compound (I), with the atom labelling. Displacement ellipsoids are drawn at the 50% probability level. The intramolecular C—H...Cl interaction (Table 1) is shown as a thin dashed line.

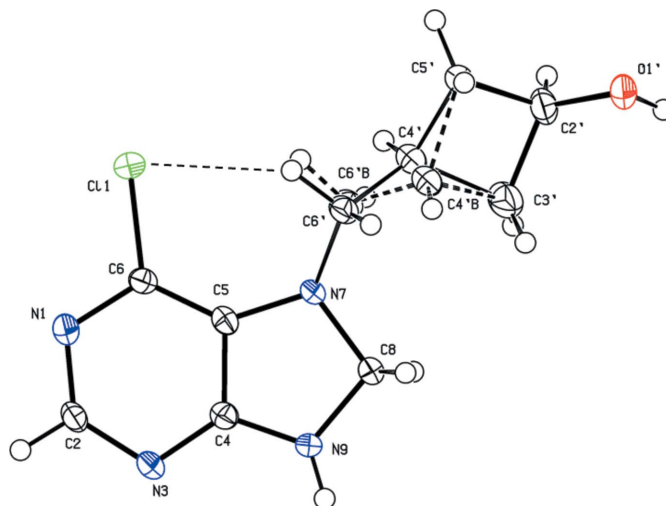


Figure 2
The molecular structure of compound (II), with the atom labelling. Displacement ellipsoids are drawn at the 50% probability level. The intramolecular C—H...Cl interaction (Table 2) is shown as a thin dashed line. The minor fraction of the disordered atoms C4' and C6', i.e. C4'B and 6'B, are shown with dashed bonds.

In compound (I) the purine ring is attached to the cyclobutanone unit through atom N7, rendering the attachment *cis* to the chlorine atom bound to the aromatic ring at the C6 position. The mean plane of the cyclobutane ring ($A = C2'–C5'$) is inclined to the mean plane of the purine ring system ($B = N1/N3N7/N9/C2/C4/C5/C6/C8$) by $52.62(11)^\circ$, while the torsion angle $N7–C5'–C4' \cdots C2'$ is *ca* 125.4° .

Reduction of compound (I) with lithium tri-*tert*-butoxy-aluminum hydride lead to the formation of the oxetanocin derivative compound (II). Here the the mean plane of the cyclobutanol ring (A) is inclined to the mean plane of the

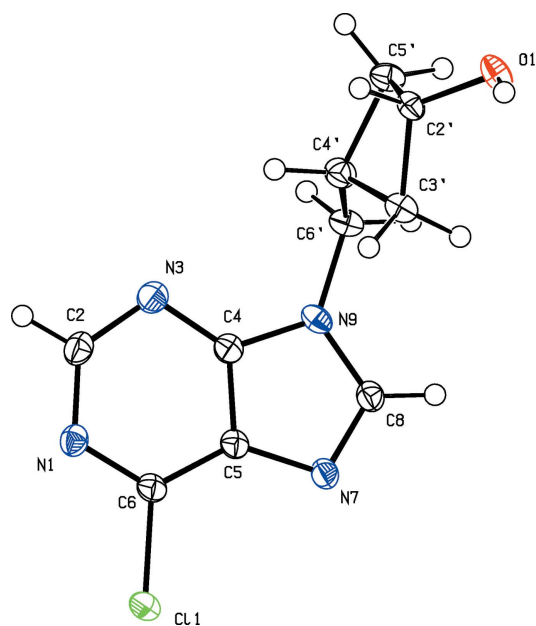


Figure 3
The molecular structure of compound (IV), with the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C6'-H6'B\cdots Cl1$	0.99	2.66	3.407 (2)	132

Table 2
Hydrogen-bond geometry (Å, °) for (II).

$Cg1$ is the centroid of the N1/C2/N3/C4/C5/C6 ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C6'-H6'2\cdots Cl1$	0.99	2.64	3.390 (3)	132
$N9-H9\cdots N3^i$	0.83 (2)	2.14 (2)	2.952 (2)	166 (2)
$O1'-H1'\cdots N1^{ii}$	0.84 (3)	2.09 (3)	2.909 (2)	164 (3)
$C4'-H4'\cdots Cg1^{iii}$	0.99	2.87	3.857 (3)	170

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

purine ring system (B) by $26.37 (15)^\circ$, while the torsion angle $N7-C5'-C4'\cdots C2'$ is *ca* 120.0° . Atoms $C6'$ and $C4'$ are positionally disordered and were split giving a refined occupancy ratio for $C6':C6'B$ and $C4':C4'B$ of $0.858 (4):0.142 (4)$ (Fig. 2).

In compound (IV), the cyclobutanol ring is attached to atom N9 of the purine ring (Fig. 3). As a result of the *trans* positioning of the cyclobutanol unit, there are no intramolecular hydrogen bonds between the chlorine atom and the cyclobutanol or methylene connector as observed in compounds (I) and (II). Here, the mean plane of the cyclobutanol ring (A) is inclined to the mean plane of the purine ring system (B) by $71.20 (13)^\circ$, and the torsion angle $N7-C5'-C4'\cdots C2'$ is *ca* 144.8° .

Reduction of the purine ring of the N-7 cyclobutanone to a dihydropurine was observed for compound (II) but not for the purine ring of the N-9 cyclobutanone on formation of compound (IV). This is confirmed by the values of the bond lengths and bond angles involving atom C8; see Table 3. Similar over-reductions of purine derivatives can be found in the literature, where electron-deficient purines are dearomatized by $NaBH_4$ to a dihydropurine (Aarhus *et al.*, 2014). We speculate the reason for over-reduction of the N-7 ketone may be due to the strain associated with the system. N-7

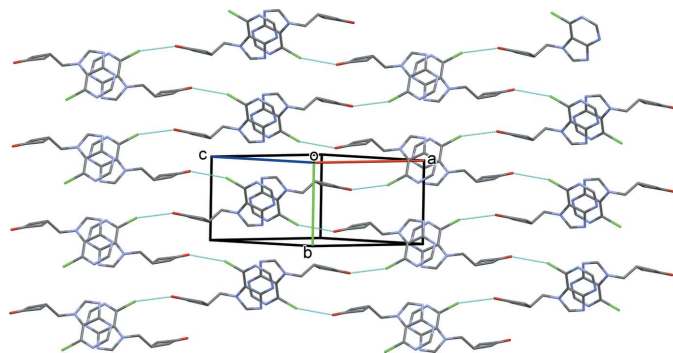


Figure 4
Crystal packing of compound (I), viewed normal to (101). The weak intermolecular $Cl\cdots O$ interactions are shown as dashed lines. For clarity, the C-bound H atoms have been omitted.

Table 3
Hydrogen-bond geometry (Å, °) for (IV).

$Cg1$ is the centroid of the N1/C2/N3/C4/C5/C6 ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$O1'-H1'\cdots N7^i$	0.84	2.03	2.853 (3)	168
$C8-H8\cdots O1^{ii}$	0.95	2.27	3.148 (2)	153
$C2-H2\cdots N3^{iii}$	0.95	2.48	3.311 (3)	146
$C2'-H2'\cdots Cg1^{iv}$	0.99	2.84	3.628 (2)	136

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

alkylation forces the chlorine of the purine ring to be oriented towards the cyclobutanone ring, which increases the strain energy of the system. This strain energy is released when the rigid aromatic structure of the purine is reduced to a more flexible dihydropurine (sp^2 C8 to sp^3 C8). This strained orientation is not observed for the N-9 ketone, hence the integrity of its purine ring is preserved.

3. Supramolecular features

In the crystal of (I), molecules are linked by a weak $Cl\cdots O$ interaction [$Cl1\cdots O1'(-x+1, y-\frac{1}{2}, -z+\frac{3}{2}) = 3.180 (2) \text{ \AA}$], forming a 2_1 helix along [010], see Fig. 4. The helices are linked by offset $\pi-\pi$ interactions, forming layers parallel to (101): $CgB\cdots CgB^i = 3.498 (1) \text{ \AA}$, CgB is the centroid of the purine ring system, $\alpha = 0.00 (5) \text{ \AA}$, $\beta = 21.6^\circ$, interplanar distance = $3.252 (1) \text{ \AA}$, offset = 1.289 \AA , symmetry code (i) $-x+2, -y+1, -z+1$.

In the crystal of (II), molecules are linked by pairs of $N-H\cdots N$ and $O-H\cdots N$ hydrogen bonds, forming inversion dimers with an $R_2^2(8)$ ring motif (Table 2 and Fig. 5). The dimers are linked by $O-H\cdots N$ hydrogen bonds, forming ribbons along [001], which in turn are linked by $C-H\cdots \pi$ (Table 2) and offset $\pi-\pi$ interactions, forming slabs parallel to the *ac* plane. [Details of the offset $\pi-\pi$ interactions: $CgB\cdots CgB^v = 3.498 (1) \text{ \AA}$, CgB is the centroid of the purine ring system, $\alpha = 0.00 (5) \text{ \AA}$, $\beta =$

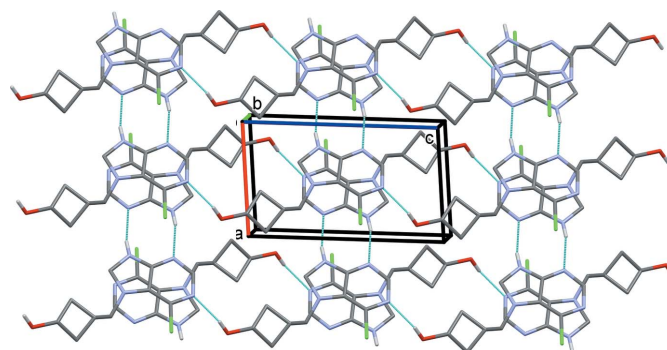


Figure 5
Crystal packing of compound (II), viewed along the b axis. The $N-H\cdots N$ and $O-H\cdots N$ hydrogen bonds (Table 2) are shown as dashed lines. For clarity, the C-bound H atoms have been omitted. The minor components of the disordered atoms $C4'$ and $C6'$ (*i.e.* $C4'B$ and $6'B$) have been omitted.

Table 4
Geometric parameters (Å, °) about atom C8 for compounds (I), (II) and (IV).

Bond/angle	(I)	(II)	(IV)
C8—N7	1.381 (2)	1.471 (3)	1.362 (3)
C8—N9	1.301 (2)	1.455 (3)	1.321 (3)
N7—C8—N9	114.95 (15)	103.41 (15)	114.28 (18)

21.6°, interplanar distance = 3.252 (1) Å, offset = 1.289 Å, symmetry code (v) $-x + 2, -y + 1, -z + 1$.]

In the crystal of (IV), molecules are linked by O—H...N hydrogen bonds (Table 3), forming chains along direction [101]. The chains are linked by C—H...O and C—H...N hydrogen bonds, and C—H... π (Table 4) and offset π - π interactions, forming a supramolecular framework (see Fig. 6). [Details of the offset π - π interactions: $CgB \cdots CgB^{vi} = 3.534$ (1) Å, CgB is the centroid of the purine ring system, $\alpha = 0.02$ (10) Å, $\beta = 17.8^\circ$, interplanar distance = 3.364 (1) Å, offset = 1.08 Å, symmetry code (vi) $-x + 1, -y + 1, -z$.]

4. Database survey

A search of the Cambridge Structural Database (CSD, Version 5.40, update February 2019; Groom *et al.*, 2016) found two related structure, *viz.* 6-chloro-9-(3-hydroxymethyl-3-hydroxycyclobutyl)purine (CSD refcode SOGROV; Boumchita *et al.*, 1991) and *cis*-1-bromomethyl-3-(6-chloro-9H-purin-

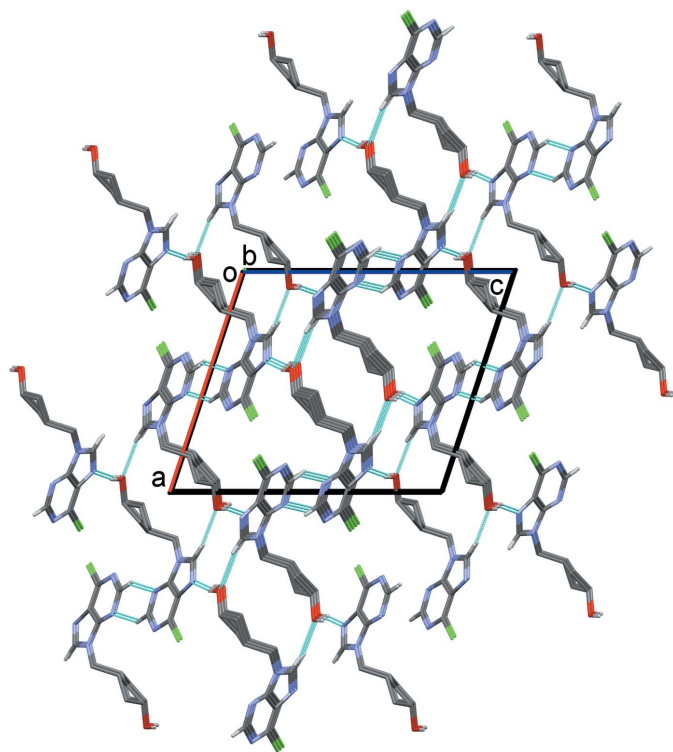


Figure 6
Crystal packing of compound (IV), viewed along the *b* axis. The various hydrogen bonds (Table 3) are shown as dashed lines. For clarity, only the H atoms involved in these interactions have been included.

yl)cyclobutanol (ZUMHAQ; Gharbaoui *et al.*, 1995). The coordinates are not available for either structure.

5. Synthesis and crystallization

Synthesis of compounds (I) and (III): Potassium carbonate (12.0 mmol) was added to a solution of (3-oxocyclobutyl)methyl benzoate (10.0 mmol) in methanol (20 ml) and stirred for 1 h at room temperature. Saturated sodium bicarbonate (10.0 ml) was added and stirring continued for an additional 15 min. The solvent was evaporated under vacuum, followed by purification by flash column chromatography with ethyl acetate, resulting in 3-(hydroxymethyl)cyclobutan-1-one in 70% yield.

3-(Hydroxymethyl)cyclobutan-1-one (1 mmol) was dissolved in 10 ml of dry dichloromethane and cooled to 195 K. Hunig's base (3.2 mmol) was added, followed by trifluoromethanesulfonic anhydride (1 mmol) and the mixture was stirred for 10 min, cooled to 273 K and stirred to obtain the qualitative conversion to (3-oxocyclobutyl)methyl trifluoromethanesulfonate.

The (3-oxocyclobutyl)methyl trifluoromethanesulfonate (5.61 mmol) was added to a mixture containing 6-chloro-7H-purine (5.61 mmol), potassium hydroxide (5.61 mmol), tris[2-(2-methoxyethoxy)ethyl]amine (0.28 mmol), magnesium sulfate (2 g) and anhydrous acetonitrile (100 ml), which was then heated to 333 K for 5 h and cooled to room temperature. The product was purified using 5% methanol and 5% trimethylamine in chloroform, which yielded two UV-active compounds.

The two UV-active compounds were separated using flash column chromatography with ethyl acetate, giving 51% of the N-9 alkylated derivative; 3-[(6-chloro-9H-purin-9-yl)methyl]cyclobutan-1-one (III) and 37% of the N-7 alkylated derivative 3-[(6-chloro-7H-purin-7-yl)methyl]cyclobutan-1-one (I).

Synthesis of 3-[(6-chloro-8,9-dihydro-7H-purin-7-yl)methyl]cyclobutan-1-ol (II): 3-[(6-Chloro-7H-purin-7-yl)methyl]cyclobutan-1-one (I) (0.21 mmol) in dichloromethane (10 ml) was cooled to 195 K and lithium tri-*tert*-butoxyaluminum hydride was added. The mixture was cooled to room temperature and sodium borohydride (0.32 mmol) was added and the resulting mixture allowed to stir overnight. Methanol (2 ml) was added and the mixture allowed to stir overnight to convert the over-reduced 3-[(6-chloro-7H-purin-7-yl)methyl]cyclobutanone(I) to 3-[(6-chloro-8,9-dihydro-7H-purin-7-yl)methyl]cyclobutan-1-ol (II).

Synthesis of *cis*-3-[(6-chloro-9H-purin-9-yl)methyl]cyclobutan-1-ol (IV): 3-[(6-Chloro-9H-purin-9-yl)methyl]cyclobutan-1-one (III) (0.21 mmol) was added to diethyl ether and cooled to 195 K and lithium tri-*tert*-butoxyaluminum hydride (0.32 mmol) was added. The reaction was allowed to warm to room temperature and left to stir overnight, which provided quantitative conversion to *cis*-3-[(6-chloro-9H-purin-9-yl)methyl]cyclobutan-1-ol (IV). Crystallization was achieved through evaporation over three days with tetrahydrofuran as the solvent.

Table 5
Experimental details.

	(I)	(II)	(IV)
Crystal data			
Chemical formula	C ₁₀ H ₉ ClN ₄ O	C ₁₀ H ₁₃ ClN ₄ O	C ₁₀ H ₁₁ ClN ₄ O
<i>M_r</i>	236.66	240.69	238.68
Crystal system, space group	Monoclinic, <i>P</i> 2 ₁ / <i>c</i>	Triclinic, <i>P</i> $\bar{1}$	Monoclinic, <i>P</i> 2 ₁ / <i>n</i>
Temperature (K)	110	110	110
<i>a</i> , <i>b</i> , <i>c</i> (Å)	11.9736 (5), 6.8854 (4), 12.2746 (5)	6.1101 (4), 8.6075 (5), 11.0083 (7)	12.7276 (8), 5.9725 (4), 14.819 (1)
α , β , γ (°)	90, 92.938 (4), 90	68.957 (6), 83.799 (5), 87.189 (5)	90, 108.250 (3), 90
<i>V</i> (Å ³)	1010.63 (8)	537.15 (6)	1069.81 (12)
<i>Z</i>	4	2	4
Radiation type	Mo <i>K</i> α	Cu <i>K</i> α	Cu <i>K</i> α
μ (mm ⁻¹)	0.36	3.03	3.04
Crystal size (mm)	0.43 × 0.21 × 0.04	0.44 × 0.30 × 0.12	0.50 × 0.21 × 0.07
Data collection			
Diffractometer	Bruker APEXII CCD	Bruker–Nonius Kappa CCD	Bruker APEXII CCD
Absorption correction	Multi-scan (<i>CrysAlis PRO</i> ; Rigaku OD, 2018)	Multi-scan (<i>CrysAlis PRO</i> ; Rigaku OD, 2018)	Numerical (<i>CrysAlis PRO</i> ; Rigaku OD, 2018)
<i>T</i> _{min} , <i>T</i> _{max}	0.661, 1.000	0.771, 1.000	0.043, 0.741
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	35539, 2508, 2217	9261, 1793, 1681	7078, 1732, 1569
<i>R</i> _{int}	0.046	0.028	0.045
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.667	0.592	0.587
Refinement			
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.043, 0.111, 1.07	0.037, 0.098, 1.07	0.038, 0.103, 0.94
No. of reflections	2508	1793	1732
No. of parameters	145	160	146
No. of restraints	0	1	0
H-atom treatment	H-atom parameters constrained	H atoms treated by a mixture of independent and constrained refinement	H-atom parameters constrained
$\Delta\rho_{\text{max}}$, $\Delta\rho_{\text{min}}$ (e Å ⁻³)	0.60, -0.28	0.71, -0.27	0.35, -0.31

Computer programs: *CrysAlis PRO* (Rigaku OD, 2018), *SHELXS* (Sheldrick, 2008), *SHELXL* (Sheldrick, 2015), *PLATON* (Spek, 2009) and *Mercury* (Macrae *et al.*, 2008), *OLEX2* (Dolomanov *et al.*, 2009), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

Pale-yellow plate-like crystals of (I), suitable for X-ray diffraction analysis, were obtained by slow evaporation of a solution in dichloromethane and heptane. Colourless plate-like crystals of (II), were obtained by slow evaporation of a solution in methanol, dichloromethane and diethyl ether (1:1:1, 9 ml). Colorless plate-like crystals of (IV), were obtained by slow evaporation of a solution in methanol (3 ml).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 5. For all three compounds, C-bound H atoms were placed in calculated positions and refined as riding: C–H = 0.95–0.99 Å with *U*_{iso}(H) = 1.2*U*_{eq}(C). For compound (II), the OH and NH H atoms were located in a difference-Fourier map. While the OH H atom was freely refined the NH H atom was refined with a distance restraint: N–H = 0.86 (2) Å. For compound (IV), the OH H atom was located in a difference-Fourier map and freely refined. In compound (II), atoms C6' and C4' are positionally disordered and were split giving a refined occupancy ratio for C6':C6'B and C4':C4'B of 0.858 (4):0.142 (4). For the final refinement of compound (II) three most disagreeable reflections (371, 372, 370) were omitted, and for the final refinement of compound

(IV) four most disagreeable reflections ($\bar{8}58$, $\bar{1}66$, $\bar{7}57$, $\bar{2}67$) were omitted.

Acknowledgements

The authors are grateful to Dr Paul Doyle of the Department of Chemistry X-ray Facility at University of Western Ontario for assistance with the data collection.

Funding information

Funding for this research was provided by: Natural Sciences and Engineering Research Council of Canada (grant No. RGPIN-06218-2014 to Gerald F. Audette).

References

- Aarhus, T. I., Fritze, U. F., Hennem, M. & Gundersen, L. L. (2014). *Tetrahedron Lett.* **55**, 5748–5750.
- Bisacchi, G. S., Braitman, A., Cianci, C. W., Clark, J. M., Field, A. K., Hagen, M. E., Hockstein, D. R., Malley, M. F., Mitt, T., Slusarchyk, W. A., *et al.* (1991). *J. Med. Chem.* **34**, 1415–1421.
- De Clercq, E. (2005). *Expert Opin. Emerg. Drugs*, **10**, 241–273.
- Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
- Gharbaoui, T., Legraverend, M., Ludwig, O., Bisagni, E., Aubertin, A.-M. & Chertanova, L. (1995). *Tetrahedron*, **51**, 1641–1652.
- Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* **B72**, 171–179.

- Hoshino, H., Shimizu, N., Shimada, N., Takita, T. & Takeuchi, T. (1987). *J. Antibiot.* **40**, 1077–1078.
- Legraverend, M., Boumchita, H., Guilhem, J. & Bisagni, E. (1991). *Heterocycles*, **32**, 867–871.
- Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
- Rigaku OD (2018). *CrysAlis PRO*. Rigaku Oxford Diffraction, Yarnton, England.
- Sheldrick, G. M. (2008). *Acta Cryst. A* **64**, 112–122.
- Sheldrick, G. M. (2015). *Acta Cryst. C* **71**, 3–8.
- Spek, A. L. (2009). *Acta Cryst. D* **65**, 148–155.
- Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supporting information

Acta Cryst. (2019). E75, 732-737 [https://doi.org/10.1107/S2056989019004432]

Crystal structures of the synthetic intermediate 3-[(6-chloro-7*H*-purin-7-yl)methyl]cyclobutan-1-one, and of two oxetanocin derivatives: 3-[(6-chloro-8,9-dihydro-7*H*-purin-7-yl)methyl]cyclobutan-1-ol and 3-[(6-chloro-9*H*-purin-9-yl)methyl]cyclobutan-1-ol

Ayat Yaseen, Muhammad Murtaza Hassan, Edward Lee-Ruff and Gerald F. Audette

Computing details

For all structures, data collection: *CrysAlis PRO* (Rigaku OD, 2018); cell refinement: *CrysAlis PRO* (Rigaku OD, 2018); data reduction: *CrysAlis PRO* (Rigaku OD, 2018); program(s) used to solve structure: *SHELXS* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015); molecular graphics: *PLATON* (Spek, 2009) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

3-[(6-Chloro-7*H*-purin-7-yl)methyl]cyclobutan-1-one (I)

Crystal data

$C_{10}H_9ClN_4O$	$F(000) = 488$
$M_r = 236.66$	$D_x = 1.555 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$a = 11.9736 (5) \text{ \AA}$	Cell parameters from 9903 reflections
$b = 6.8854 (4) \text{ \AA}$	$\theta = 3.3\text{--}32.6^\circ$
$c = 12.2746 (5) \text{ \AA}$	$\mu = 0.36 \text{ mm}^{-1}$
$\beta = 92.938 (4)^\circ$	$T = 110 \text{ K}$
$V = 1010.63 (8) \text{ \AA}^3$	Plate, pale_yellow
$Z = 4$	$0.43 \times 0.21 \times 0.04 \text{ mm}$

Data collection

Bruker APEXII CCD diffractometer	35539 measured reflections
Radiation source: sealed X-ray tube	2508 independent reflections
Graphite monochromator	2217 reflections with $I > 2\sigma(I)$
ω scans	$R_{\text{int}} = 0.046$
Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2018)	$\theta_{\text{max}} = 28.3^\circ$, $\theta_{\text{min}} = 3.3^\circ$
$T_{\text{min}} = 0.661$, $T_{\text{max}} = 1.000$	$h = -15 \rightarrow 15$
	$k = -9 \rightarrow 9$
	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	$S = 1.07$
Least-squares matrix: full	2508 reflections
$R[F^2 > 2\sigma(F^2)] = 0.043$	145 parameters
$wR(F^2) = 0.111$	0 restraints

Primary atom site location: structure-invariant direct methods
 Secondary atom site location: difference Fourier map
 Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0508P)^2 + 0.9477P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.60 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.28 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.70136 (4)	0.21385 (7)	0.52904 (4)	0.02685 (14)
N1	0.81306 (13)	0.2530 (2)	0.35301 (12)	0.0230 (3)
N3	0.93051 (12)	0.5196 (2)	0.30500 (11)	0.0219 (3)
N7	0.84068 (11)	0.6388 (2)	0.56456 (11)	0.0183 (3)
N9	0.95030 (12)	0.7744 (2)	0.44062 (12)	0.0216 (3)
O1'	0.43878 (13)	0.6449 (3)	0.76126 (16)	0.0503 (5)
C2'	0.53242 (16)	0.6753 (3)	0.73657 (17)	0.0303 (4)
C3'	0.64220 (16)	0.7042 (3)	0.80129 (15)	0.0297 (4)
H3'A	0.670598	0.585740	0.839075	0.036*
H3'B	0.642461	0.816481	0.851695	0.036*
C4'	0.69801 (14)	0.7464 (3)	0.69174 (14)	0.0215 (4)
H4'	0.720328	0.885805	0.685533	0.026*
C5'	0.58520 (15)	0.7032 (3)	0.62724 (16)	0.0297 (4)
H5'A	0.554763	0.814870	0.584483	0.036*
H5'B	0.586295	0.584132	0.582080	0.036*
C6'	0.79428 (14)	0.6106 (3)	0.67259 (12)	0.0198 (3)
H6'A	0.854160	0.632156	0.729957	0.024*
H6'B	0.768262	0.474799	0.678746	0.024*
C2	0.88240 (14)	0.3491 (3)	0.28787 (13)	0.0230 (4)
H2	0.898377	0.285913	0.221649	0.028*
C4	0.90857 (13)	0.6006 (3)	0.40103 (13)	0.0183 (3)
C5	0.83895 (13)	0.5123 (2)	0.47653 (12)	0.0176 (3)
C6	0.79181 (14)	0.3358 (3)	0.44645 (13)	0.0198 (3)
C8	0.90917 (14)	0.7900 (3)	0.53629 (14)	0.0205 (3)
H8	0.925073	0.897356	0.583167	0.025*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0305 (2)	0.0264 (2)	0.0241 (2)	-0.00833 (17)	0.00535 (16)	0.00170 (17)
N1	0.0231 (7)	0.0246 (8)	0.0213 (7)	0.0002 (6)	0.0002 (6)	-0.0029 (6)
N3	0.0215 (7)	0.0272 (8)	0.0173 (7)	0.0026 (6)	0.0046 (5)	0.0014 (6)
N7	0.0202 (7)	0.0202 (7)	0.0146 (6)	0.0007 (5)	0.0030 (5)	0.0009 (5)

N9	0.0213 (7)	0.0210 (7)	0.0229 (7)	-0.0006 (6)	0.0036 (5)	0.0027 (6)
O1'	0.0302 (8)	0.0585 (12)	0.0639 (11)	-0.0075 (8)	0.0186 (8)	0.0010 (9)
C2'	0.0257 (9)	0.0282 (10)	0.0377 (10)	0.0011 (7)	0.0102 (8)	-0.0006 (8)
C3'	0.0284 (9)	0.0409 (12)	0.0207 (8)	0.0025 (8)	0.0091 (7)	0.0002 (8)
C4'	0.0198 (8)	0.0263 (9)	0.0187 (8)	0.0019 (6)	0.0046 (6)	0.0022 (6)
C5'	0.0215 (8)	0.0425 (12)	0.0251 (9)	0.0037 (8)	0.0000 (7)	-0.0010 (8)
C6'	0.0228 (8)	0.0253 (9)	0.0116 (7)	0.0016 (7)	0.0033 (6)	0.0011 (6)
C2	0.0229 (8)	0.0301 (9)	0.0162 (7)	0.0041 (7)	0.0021 (6)	-0.0038 (7)
C4	0.0177 (7)	0.0204 (8)	0.0168 (7)	0.0022 (6)	0.0003 (6)	0.0037 (6)
C5	0.0179 (7)	0.0213 (8)	0.0135 (7)	0.0035 (6)	0.0004 (6)	0.0011 (6)
C6	0.0195 (7)	0.0223 (8)	0.0175 (7)	0.0007 (6)	0.0015 (6)	0.0039 (6)
C8	0.0205 (7)	0.0176 (8)	0.0232 (8)	0.0009 (6)	-0.0014 (6)	-0.0022 (6)

Geometric parameters (Å, °)

C11—C6	1.7372 (17)	C3'—H3'A	0.9900
N1—C6	1.317 (2)	C3'—H3'B	0.9900
N1—C2	1.354 (2)	C4'—C6'	1.512 (2)
N3—C2	1.320 (2)	C4'—C5'	1.559 (2)
N3—C4	1.342 (2)	C4'—H4'	1.0000
N7—C8	1.381 (2)	C5'—H5'A	0.9900
N7—C5	1.387 (2)	C5'—H5'B	0.9900
N7—C6'	1.4765 (19)	C6'—H6'A	0.9900
N9—C8	1.301 (2)	C6'—H6'B	0.9900
N9—C4	1.376 (2)	C2—H2	0.9500
O1'—C2'	1.195 (2)	C4—C5	1.415 (2)
C2'—C3'	1.514 (3)	C5—C6	1.382 (2)
C2'—C5'	1.524 (3)	C8—H8	0.9500
C3'—C4'	1.559 (2)		
C6—N1—C2	117.04 (16)	C2'—C5'—H5'B	114.0
C2—N3—C4	113.96 (15)	C4'—C5'—H5'B	114.0
C8—N7—C5	105.24 (13)	H5'A—C5'—H5'B	111.2
C8—N7—C6'	125.49 (14)	N7—C6'—C4'	112.54 (14)
C5—N7—C6'	128.74 (15)	N7—C6'—H6'A	109.1
C8—N9—C4	104.09 (14)	C4'—C6'—H6'A	109.1
O1'—C2'—C3'	133.7 (2)	N7—C6'—H6'B	109.1
O1'—C2'—C5'	133.0 (2)	C4'—C6'—H6'B	109.1
C3'—C2'—C5'	93.29 (14)	H6'A—C6'—H6'B	107.8
C2'—C3'—C4'	88.33 (14)	N3—C2—N1	128.03 (16)
C2'—C3'—H3'A	113.9	N3—C2—H2	116.0
C4'—C3'—H3'A	113.9	N1—C2—H2	116.0
C2'—C3'—H3'B	113.9	N3—C4—N9	126.01 (15)
C4'—C3'—H3'B	113.9	N3—C4—C5	123.02 (16)
H3'A—C3'—H3'B	111.1	N9—C4—C5	110.96 (14)
C6'—C4'—C5'	116.77 (16)	C6—C5—N7	138.56 (15)
C6'—C4'—C3'	112.49 (15)	C6—C5—C4	116.68 (15)
C5'—C4'—C3'	90.22 (14)	N7—C5—C4	104.75 (14)

C6'—C4'—H4'	111.9	N1—C6—C5	121.22 (16)
C5'—C4'—H4'	111.9	N1—C6—C11	116.95 (14)
C3'—C4'—H4'	111.9	C5—C6—C11	121.83 (13)
C2'—C5'—C4'	87.96 (14)	N9—C8—N7	114.95 (15)
C2'—C5'—H5'A	114.0	N9—C8—H8	122.5
C4'—C5'—H5'A	114.0	N7—C8—H8	122.5
O1'—C2'—C3'—C4'	-175.7 (3)	C8—N7—C5—C6	179.2 (2)
C5'—C2'—C3'—C4'	3.32 (16)	C6'—N7—C5—C6	7.2 (3)
C2'—C3'—C4'—C6'	-122.53 (16)	C8—N7—C5—C4	0.10 (17)
C2'—C3'—C4'—C5'	-3.24 (16)	C6'—N7—C5—C4	-171.83 (15)
O1'—C2'—C5'—C4'	175.7 (3)	N3—C4—C5—C6	-0.8 (2)
C3'—C2'—C5'—C4'	-3.32 (16)	N9—C4—C5—C6	-179.83 (14)
C6'—C4'—C5'—C2'	118.72 (16)	N3—C4—C5—N7	178.47 (15)
C3'—C4'—C5'—C2'	3.22 (15)	N9—C4—C5—N7	-0.52 (18)
C8—N7—C6'—C4'	76.0 (2)	C2—N1—C6—C5	-0.4 (2)
C5—N7—C6'—C4'	-113.61 (19)	C2—N1—C6—C11	179.16 (13)
C5'—C4'—C6'—N7	72.5 (2)	N7—C5—C6—N1	-177.58 (18)
C3'—C4'—C6'—N7	174.86 (15)	C4—C5—C6—N1	1.4 (2)
C4—N3—C2—N1	2.1 (3)	N7—C5—C6—C11	2.9 (3)
C6—N1—C2—N3	-1.5 (3)	C4—C5—C6—C11	-178.08 (12)
C2—N3—C4—N9	178.09 (16)	C4—N9—C8—N7	-0.70 (19)
C2—N3—C4—C5	-0.8 (2)	C5—N7—C8—N9	0.4 (2)
C8—N9—C4—N3	-178.22 (16)	C6'—N7—C8—N9	172.66 (15)
C8—N9—C4—C5	0.74 (18)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C6'—H6' <i>B</i> '...C11	0.99	2.66	3.407 (2)	132
C4'—H4'...C11 ⁱ	1.00	2.97	3.7889 (19)	140
C6'—H6' <i>B</i> '...N1 ⁱⁱ	0.99	2.68	3.343 (2)	124

Symmetry codes: (i) *x*, *y*+1, *z*; (ii) *x*, -*y*+1/2, *z*+1/2.3-[(6-Chloro-8,9-dihydro-7*H*-purin-7-yl)methyl]cyclobutan-1-ol (II)

Crystal data

C₁₀H₁₃ClN₄O*M_r* = 240.69Triclinic, *P* $\bar{1}$ *a* = 6.1101 (4) Å*b* = 8.6075 (5) Å*c* = 11.0083 (7) Å α = 68.957 (6)° β = 83.799 (5)° γ = 87.189 (5)°*V* = 537.15 (6) Å³*Z* = 2*F*(000) = 252*D_x* = 1.488 Mg m⁻³Cu *K* α radiation, λ = 1.54184 Å

Cell parameters from 8605 reflections

 θ = 4.3–65.4° μ = 3.03 mm⁻¹*T* = 110 K

Plate, colorless

0.44 × 0.30 × 0.12 mm

Data collection

Bruker–Nonius Kappa CCD diffractometer	9261 measured reflections
Radiation source: sealed X-ray tube, Enhance (Cu) X-ray Source	1793 independent reflections
Detector resolution: 7.9 pixels mm ⁻¹	1681 reflections with $I > 2\sigma(I)$
ω scans	$R_{\text{int}} = 0.028$
Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2018)	$\theta_{\text{max}} = 66.0^\circ$, $\theta_{\text{min}} = 4.3^\circ$
$T_{\text{min}} = 0.771$, $T_{\text{max}} = 1.000$	$h = -7 \rightarrow 5$
	$k = -10 \rightarrow 10$
	$l = -13 \rightarrow 12$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: mixed
$R[F^2 > 2\sigma(F^2)] = 0.037$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.098$	$w = 1/[\sigma^2(F_o^2) + (0.0479P)^2 + 0.5464P]$
$S = 1.07$	where $P = (F_o^2 + 2F_c^2)/3$
1793 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
160 parameters	$\Delta\rho_{\text{max}} = 0.71 \text{ e } \text{\AA}^{-3}$
1 restraint	$\Delta\rho_{\text{min}} = -0.27 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
C11	0.16834 (8)	0.41808 (6)	0.40624 (5)	0.02598 (19)	
N1	0.4817 (3)	0.2850 (2)	0.28999 (16)	0.0208 (4)	
C2	0.6669 (3)	0.2012 (3)	0.28642 (19)	0.0208 (4)	
H2	0.719070	0.193795	0.204161	0.025*	
N3	0.7920 (3)	0.1241 (2)	0.38609 (16)	0.0201 (4)	
C4	0.7122 (3)	0.1387 (2)	0.49898 (19)	0.0181 (4)	
C5	0.5139 (3)	0.2254 (2)	0.51675 (19)	0.0175 (4)	
C6	0.4064 (3)	0.2984 (2)	0.40720 (19)	0.0185 (4)	
N7	0.4843 (3)	0.2163 (2)	0.64573 (16)	0.0195 (4)	
C8	0.6671 (3)	0.1184 (3)	0.71453 (19)	0.0217 (4)	
H8A	0.750275	0.184768	0.750830	0.026*	
H8B	0.612778	0.016417	0.786389	0.026*	
N9	0.8027 (3)	0.0776 (2)	0.61286 (16)	0.0212 (4)	
H9	0.916 (3)	0.018 (3)	0.627 (2)	0.021 (6)*	
O1'	0.1576 (3)	0.2578 (2)	1.12373 (14)	0.0278 (4)	
H1'	0.256 (5)	0.284 (4)	1.161 (3)	0.045 (8)*	
C2'	0.2169 (4)	0.3455 (3)	0.9900 (2)	0.0273 (5)	
H2'	0.230429	0.466833	0.973632	0.033*	
C3'	0.4091 (4)	0.2873 (3)	0.9160 (2)	0.0346 (6)	

H3'1	0.446403	0.167641	0.956402	0.041*	
H3'2	0.541933	0.356932	0.895749	0.041*	
C5'	0.0629 (4)	0.3203 (3)	0.8985 (2)	0.0277 (5)	
H5'1	-0.044286	0.412524	0.866879	0.033*	
H5'2	-0.011259	0.211215	0.933326	0.033*	
C4'	0.2642 (4)	0.3281 (3)	0.7998 (2)	0.0240 (6)	0.858 (4)
H4'	0.288618	0.445876	0.739504	0.029*	0.858 (4)
C6'	0.2716 (4)	0.2157 (3)	0.7213 (2)	0.0229 (6)	0.858 (4)
H6'1	0.239112	0.100518	0.781565	0.027*	0.858 (4)
H6'2	0.155181	0.251106	0.660680	0.027*	0.858 (4)
C4'B	0.246 (3)	0.216 (2)	0.8417 (15)	0.0240 (6)	0.142 (4)
H4'B	0.228733	0.092243	0.870206	0.029*	0.142 (4)
C6'B	0.332 (3)	0.308 (2)	0.7043 (15)	0.0229 (6)	0.142 (4)
H6'3	0.206224	0.345077	0.650824	0.027*	0.142 (4)
H6'4	0.406521	0.409522	0.701360	0.027*	0.142 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0222 (3)	0.0321 (3)	0.0240 (3)	0.0101 (2)	-0.0049 (2)	-0.0110 (2)
N1	0.0227 (9)	0.0239 (9)	0.0162 (8)	0.0000 (7)	-0.0018 (7)	-0.0078 (7)
C2	0.0227 (11)	0.0255 (10)	0.0158 (10)	-0.0004 (8)	0.0012 (8)	-0.0104 (8)
N3	0.0198 (9)	0.0244 (9)	0.0173 (8)	0.0013 (7)	0.0010 (7)	-0.0100 (7)
C4	0.0171 (10)	0.0192 (10)	0.0186 (10)	-0.0008 (8)	-0.0003 (8)	-0.0080 (8)
C5	0.0181 (10)	0.0193 (9)	0.0166 (10)	-0.0025 (8)	0.0006 (8)	-0.0087 (8)
C6	0.0168 (10)	0.0204 (10)	0.0192 (10)	0.0014 (8)	-0.0004 (8)	-0.0087 (8)
N7	0.0173 (9)	0.0277 (9)	0.0155 (8)	0.0057 (7)	-0.0014 (7)	-0.0109 (7)
C8	0.0212 (11)	0.0293 (11)	0.0176 (10)	0.0059 (8)	-0.0036 (8)	-0.0121 (8)
N9	0.0181 (9)	0.0294 (10)	0.0181 (9)	0.0094 (7)	-0.0038 (7)	-0.0114 (7)
O1'	0.0275 (9)	0.0394 (9)	0.0177 (7)	0.0028 (7)	-0.0043 (6)	-0.0115 (7)
C2'	0.0286 (12)	0.0371 (12)	0.0179 (10)	-0.0017 (9)	-0.0008 (9)	-0.0119 (9)
C3'	0.0257 (12)	0.0522 (15)	0.0340 (13)	0.0000 (11)	-0.0030 (10)	-0.0253 (12)
C5'	0.0250 (11)	0.0400 (13)	0.0215 (11)	0.0119 (9)	-0.0052 (9)	-0.0157 (10)
C4'	0.0258 (13)	0.0263 (14)	0.0205 (12)	0.0035 (11)	0.0002 (10)	-0.0104 (11)
C6'	0.0164 (12)	0.0341 (16)	0.0216 (12)	0.0011 (11)	0.0000 (10)	-0.0147 (11)
C4'B	0.0258 (13)	0.0263 (14)	0.0205 (12)	0.0035 (11)	0.0002 (10)	-0.0104 (11)
C6'B	0.0164 (12)	0.0341 (16)	0.0216 (12)	0.0011 (11)	0.0000 (10)	-0.0147 (11)

Geometric parameters (Å, °)

Cl1—C6	1.739 (2)	C2'—C3'	1.524 (3)
N1—C2	1.317 (3)	C2'—C5'	1.527 (3)
N1—C6	1.365 (3)	C2'—H2'	1.0000
C2—N3	1.355 (3)	C3'—C4'	1.561 (3)
C2—H2	0.9500	C3'—C4'B	1.626 (16)
N3—C4	1.332 (3)	C3'—H3'1	0.9900
C4—N9	1.342 (3)	C3'—H3'2	0.9900
C4—C5	1.424 (3)	C5'—C4'	1.537 (3)

C5—C6	1.366 (3)	C5'—C4'B	1.616 (15)
C5—N7	1.386 (2)	C5'—H5'1	0.9900
N7—C6'B	1.440 (15)	C5'—H5'2	0.9900
N7—C6'	1.465 (3)	C4'—C6'	1.508 (3)
N7—C8	1.471 (3)	C4'—H4'	1.0000
C8—N9	1.455 (3)	C6'—H6'1	0.9900
C8—H8A	0.9900	C6'—H6'2	0.9900
C8—H8B	0.9900	C4'B—C6'B	1.48 (2)
N9—H9	0.833 (17)	C4'B—H4'B	1.0000
O1'—C2'	1.407 (3)	C6'B—H6'3	0.9900
O1'—H1'	0.84 (3)	C6'B—H6'4	0.9900
C2—N1—C6	116.73 (17)	C2'—C3'—H3'1	114.0
N1—C2—N3	127.76 (18)	C4'—C3'—H3'1	114.0
N1—C2—H2	116.1	C2'—C3'—H3'2	114.0
N3—C2—H2	116.1	C4'—C3'—H3'2	114.0
C4—N3—C2	113.74 (17)	H3'1—C3'—H3'2	111.2
N3—C4—N9	126.70 (19)	C2'—C5'—C4'	88.64 (17)
N3—C4—C5	124.39 (18)	C2'—C5'—C4'B	92.8 (6)
N9—C4—C5	108.91 (17)	C2'—C5'—H5'1	113.9
C6—C5—N7	136.29 (19)	C4'—C5'—H5'1	113.9
C6—C5—C4	115.33 (18)	C2'—C5'—H5'2	113.9
N7—C5—C4	108.35 (17)	C4'—C5'—H5'2	113.9
N1—C6—C5	122.04 (18)	H5'1—C5'—H5'2	111.1
N1—C6—C11	115.38 (15)	C6'—C4'—C5'	118.5 (2)
C5—C6—C11	122.56 (15)	C6'—C4'—C3'	120.1 (2)
C5—N7—C6'B	128.7 (6)	C5'—C4'—C3'	87.40 (17)
C5—N7—C6'	125.55 (17)	C6'—C4'—H4'	109.7
C5—N7—C8	108.55 (16)	C5'—C4'—H4'	109.7
C6'B—N7—C8	121.7 (6)	C3'—C4'—H4'	109.7
C6'—N7—C8	118.33 (17)	N7—C6'—C4'	113.3 (2)
N9—C8—N7	103.41 (15)	N7—C6'—H6'1	108.9
N9—C8—H8A	111.1	C4'—C6'—H6'1	108.9
N7—C8—H8A	111.1	N7—C6'—H6'2	108.9
N9—C8—H8B	111.1	C4'—C6'—H6'2	108.9
N7—C8—H8B	111.1	H6'1—C6'—H6'2	107.7
H8A—C8—H8B	109.0	C6'B—C4'B—C5'	112.7 (13)
C4—N9—C8	110.76 (17)	C6'B—C4'B—C3'	99.0 (12)
C4—N9—H9	125.9 (17)	C5'—C4'B—C3'	82.6 (7)
C8—N9—H9	123.2 (17)	C6'B—C4'B—H4'B	118.5
C2'—O1'—H1'	103 (2)	C5'—C4'B—H4'B	118.5
O1'—C2'—C3'	121.1 (2)	C3'—C4'B—H4'B	118.5
O1'—C2'—C5'	114.52 (18)	N7—C6'B—C4'B	115.0 (14)
C3'—C2'—C5'	89.12 (16)	N7—C6'B—H6'3	108.5
O1'—C2'—H2'	110.1	C4'B—C6'B—H6'3	108.5
C3'—C2'—H2'	110.1	N7—C6'B—H6'4	108.5
C5'—C2'—H2'	110.1	C4'B—C6'B—H6'4	108.5
C2'—C3'—C4'	87.87 (18)	H6'3—C6'B—H6'4	107.5

C2'—C3'—C4'B	92.5 (5)		
C6—N1—C2—N3	0.8 (3)	N7—C8—N9—C4	-1.3 (2)
N1—C2—N3—C4	-0.3 (3)	O1'—C2'—C3'—C4'	138.2 (2)
C2—N3—C4—N9	-178.94 (19)	C5'—C2'—C3'—C4'	19.78 (18)
C2—N3—C4—C5	0.4 (3)	O1'—C2'—C3'—C4'B	105.1 (6)
N3—C4—C5—C6	-1.1 (3)	C5'—C2'—C3'—C4'B	-13.3 (6)
N9—C4—C5—C6	178.36 (17)	O1'—C2'—C5'—C4'	-144.2 (2)
N3—C4—C5—N7	-179.35 (17)	C3'—C2'—C5'—C4'	-20.09 (19)
N9—C4—C5—N7	0.1 (2)	O1'—C2'—C5'—C4'B	-110.7 (6)
C2—N1—C6—C5	-1.6 (3)	C3'—C2'—C5'—C4'B	13.4 (6)
C2—N1—C6—Cl1	177.14 (14)	C2'—C5'—C4'—C6'	142.7 (2)
N7—C5—C6—N1	179.3 (2)	C2'—C5'—C4'—C3'	19.62 (18)
C4—C5—C6—N1	1.6 (3)	C2'—C3'—C4'—C6'	-141.4 (2)
N7—C5—C6—Cl1	0.7 (3)	C2'—C3'—C4'—C5'	-19.66 (18)
C4—C5—C6—Cl1	-176.96 (14)	C5—N7—C6'—C4'	-135.6 (2)
C6—C5—N7—C6'B	-10.1 (10)	C8—N7—C6'—C4'	78.4 (3)
C4—C5—N7—C6'B	167.6 (10)	C5'—C4'—C6'—N7	-171.4 (2)
C6—C5—N7—C6'	32.6 (4)	C3'—C4'—C6'—N7	-66.6 (3)
C4—C5—N7—C6'	-149.7 (2)	C2'—C5'—C4'B—C6'B	-109.4 (12)
C6—C5—N7—C8	-178.7 (2)	C2'—C5'—C4'B—C3'	-12.7 (6)
C4—C5—N7—C8	-0.9 (2)	C2'—C3'—C4'B—C6'B	124.6 (10)
C5—N7—C8—N9	1.3 (2)	C2'—C3'—C4'B—C5'	12.7 (6)
C6'B—N7—C8—N9	-168.2 (9)	C5—N7—C6'B—C4'B	146.2 (9)
C6'—N7—C8—N9	152.69 (18)	C8—N7—C6'B—C4'B	-46.6 (16)
N3—C4—N9—C8	-179.79 (18)	C5'—C4'B—C6'B—N7	-175.7 (10)
C5—C4—N9—C8	0.8 (2)	C3'—C4'B—C6'B—N7	98.5 (13)

Hydrogen-bond geometry (Å, °)

Cg1 is the centroid of ring N1/C2/N3/C4/C5/C6.

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C6'—H6'2...C11	0.99	2.64	3.390 (3)	132
N9—H9...N3 ⁱ	0.83 (2)	2.14 (2)	2.952 (2)	166 (2)
O1'—H1'...N1 ⁱⁱ	0.84 (3)	2.09 (3)	2.909 (2)	164 (3)
C4'—H4'...Cg1 ⁱⁱⁱ	0.99	2.87	3.857 (3)	170

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

3-[(6-Chloro-9*H*-purin-9-yl)methyl]cyclobutan-1-ol (IV)

Crystal data

C₁₀H₁₁ClN₄O

M_r = 238.68

Monoclinic, *P*2₁/*n*

a = 12.7276 (8) Å

b = 5.9725 (4) Å

c = 14.819 (1) Å

β = 108.250 (3)°

V = 1069.81 (12) Å³

Z = 4

F(000) = 496

D_x = 1.482 Mg m⁻³

Cu *K*α radiation, λ = 1.54178 Å

Cell parameters from 4964 reflections

θ = 4.0–64.9°

$\mu = 3.04 \text{ mm}^{-1}$
 $T = 110 \text{ K}$

Plate, colourless
 $0.50 \times 0.21 \times 0.07 \text{ mm}$

Data collection

Bruker APEXII CCD
 diffractometer
 Radiation source: sealed X-ray tube
 Graphite monochromator
 Detector resolution: 7.9 pixels mm^{-1}
 ω scans
 Absorption correction: numerical
 (CrysAlisPro; Rigaku OD, 2018)
 $T_{\min} = 0.043$, $T_{\max} = 0.741$

7078 measured reflections
 1732 independent reflections
 1569 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.045$
 $\theta_{\max} = 64.9^\circ$, $\theta_{\min} = 4.0^\circ$
 $h = -14 \rightarrow 14$
 $k = -4 \rightarrow 7$
 $l = -17 \rightarrow 17$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.038$
 $wR(F^2) = 0.103$
 $S = 0.94$
 1732 reflections
 146 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0599P)^2 + 1.4189P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.35 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.31 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.65978 (4)	0.22748 (10)	0.21719 (4)	0.0258 (2)
O1'	-0.06577 (11)	0.5696 (3)	-0.19248 (10)	0.0233 (4)
H1'	-0.067187	0.456986	-0.226798	0.035*
N3	0.44729 (14)	0.7995 (3)	0.06539 (12)	0.0218 (4)
N1	0.61729 (14)	0.5895 (3)	0.11346 (12)	0.0196 (4)
N7	0.39848 (13)	0.3289 (3)	0.19580 (12)	0.0199 (4)
N9	0.30402 (13)	0.6280 (3)	0.11873 (11)	0.0186 (4)
C4	0.40817 (16)	0.6422 (4)	0.11001 (13)	0.0178 (5)
C5	0.46564 (16)	0.4569 (4)	0.15808 (13)	0.0168 (5)
C3'	0.12604 (16)	0.5194 (4)	-0.07173 (14)	0.0201 (5)
H3'A	0.090406	0.436047	-0.031431	0.024*
H3'B	0.179230	0.424010	-0.090614	0.024*
C6	0.57424 (16)	0.4399 (4)	0.15774 (13)	0.0180 (5)
C8	0.30351 (16)	0.4385 (4)	0.16970 (14)	0.0192 (5)
H8	0.240264	0.389301	0.185165	0.023*
C2	0.55210 (18)	0.7598 (4)	0.07007 (15)	0.0223 (5)
H2	0.584609	0.865250	0.038801	0.027*

C2'	0.04431 (16)	0.6477 (4)	-0.15487 (14)	0.0177 (5)
H2'	0.077386	0.673588	-0.206946	0.021*
C4'	0.17237 (18)	0.7495 (4)	-0.03173 (15)	0.0216 (5)
H4'	0.231950	0.796287	-0.058547	0.026*
C6'	0.21115 (17)	0.7800 (4)	0.07552 (15)	0.0208 (5)
H6'A	0.234806	0.936928	0.091463	0.025*
H6'B	0.149539	0.747512	0.100968	0.025*
C5'	0.05979 (17)	0.8537 (4)	-0.09087 (15)	0.0217 (5)
H5'A	0.005170	0.866225	-0.056036	0.026*
H5'B	0.066593	0.996160	-0.122717	0.026*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0169 (3)	0.0313 (4)	0.0291 (3)	0.0052 (2)	0.0069 (2)	0.0101 (2)
O1'	0.0144 (7)	0.0311 (10)	0.0231 (8)	-0.0006 (6)	0.0037 (6)	-0.0095 (7)
N3	0.0217 (9)	0.0229 (11)	0.0184 (9)	-0.0037 (8)	0.0027 (7)	0.0013 (8)
N1	0.0194 (9)	0.0206 (10)	0.0183 (8)	-0.0042 (8)	0.0051 (7)	-0.0005 (8)
N7	0.0155 (9)	0.0260 (11)	0.0177 (8)	-0.0011 (8)	0.0045 (7)	0.0024 (8)
N9	0.0142 (8)	0.0232 (11)	0.0160 (8)	0.0021 (8)	0.0013 (7)	-0.0017 (8)
C4	0.0169 (10)	0.0206 (12)	0.0129 (9)	-0.0030 (9)	0.0004 (8)	-0.0016 (8)
C5	0.0156 (9)	0.0207 (12)	0.0121 (9)	-0.0022 (8)	0.0014 (7)	-0.0014 (8)
C3'	0.0188 (10)	0.0231 (12)	0.0177 (10)	0.0034 (9)	0.0045 (8)	-0.0013 (9)
C6	0.0160 (10)	0.0219 (12)	0.0134 (9)	-0.0010 (9)	0.0006 (8)	-0.0002 (9)
C8	0.0159 (10)	0.0234 (13)	0.0177 (9)	-0.0013 (9)	0.0045 (8)	0.0003 (9)
C2	0.0230 (11)	0.0232 (13)	0.0193 (10)	-0.0064 (9)	0.0047 (9)	0.0015 (9)
C2'	0.0134 (9)	0.0220 (12)	0.0165 (10)	0.0005 (9)	0.0031 (8)	-0.0007 (9)
C4'	0.0186 (11)	0.0249 (13)	0.0194 (11)	0.0010 (9)	0.0035 (9)	0.0009 (9)
C6'	0.0193 (11)	0.0209 (12)	0.0194 (10)	0.0043 (9)	0.0021 (8)	0.0000 (9)
C5'	0.0206 (10)	0.0223 (13)	0.0185 (10)	0.0032 (9)	0.0011 (8)	0.0001 (9)

Geometric parameters (Å, °)

C11—C6	1.723 (2)	C3'—C2'	1.544 (3)
O1'—C2'	1.415 (2)	C3'—H3'A	0.9900
O1'—H1'	0.8400	C3'—H3'B	0.9900
N3—C4	1.332 (3)	C8—H8	0.9500
N3—C2	1.335 (3)	C2—H2	0.9500
N1—C6	1.325 (3)	C2'—C5'	1.528 (3)
N1—C2	1.342 (3)	C2'—H2'	1.0000
N7—C8	1.321 (3)	C4'—C6'	1.520 (3)
N7—C5	1.388 (3)	C4'—C5'	1.556 (3)
N9—C8	1.362 (3)	C4'—H4'	1.0000
N9—C4	1.374 (3)	C6'—H6'A	0.9900
N9—C6'	1.469 (3)	C6'—H6'B	0.9900
C4—C5	1.393 (3)	C5'—H5'A	0.9900
C5—C6	1.388 (3)	C5'—H5'B	0.9900
C3'—C4'	1.539 (3)		

C2'—O1'—H1'	109.5	N3—C2—H2	116.0
C4—N3—C2	111.81 (19)	N1—C2—H2	116.0
C6—N1—C2	117.32 (17)	O1'—C2'—C5'	115.42 (17)
C8—N7—C5	103.39 (18)	O1'—C2'—C3'	119.16 (18)
C8—N9—C4	106.00 (17)	C5'—C2'—C3'	88.88 (16)
C8—N9—C6'	127.78 (17)	O1'—C2'—H2'	110.6
C4—N9—C6'	126.10 (18)	C5'—C2'—H2'	110.6
N3—C4—N9	127.7 (2)	C3'—C2'—H2'	110.6
N3—C4—C5	126.60 (19)	C6'—C4'—C3'	118.02 (19)
N9—C4—C5	105.69 (18)	C6'—C4'—C5'	118.75 (18)
C6—C5—N7	134.5 (2)	C3'—C4'—C5'	88.05 (16)
C6—C5—C4	114.87 (19)	C6'—C4'—H4'	110.1
N7—C5—C4	110.64 (17)	C3'—C4'—H4'	110.1
C4'—C3'—C2'	86.87 (17)	C5'—C4'—H4'	110.1
C4'—C3'—H3'A	114.2	N9—C6'—C4'	109.58 (17)
C2'—C3'—H3'A	114.2	N9—C6'—H6'A	109.8
C4'—C3'—H3'B	114.2	C4'—C6'—H6'A	109.8
C2'—C3'—H3'B	114.2	N9—C6'—H6'B	109.8
H3'A—C3'—H3'B	111.3	C4'—C6'—H6'B	109.8
N1—C6—C5	121.3 (2)	H6'A—C6'—H6'B	108.2
N1—C6—C11	117.18 (15)	C2'—C5'—C4'	86.82 (16)
C5—C6—C11	121.52 (16)	C2'—C5'—H5'A	114.2
N7—C8—N9	114.28 (18)	C4'—C5'—H5'A	114.2
N7—C8—H8	122.9	C2'—C5'—H5'B	114.2
N9—C8—H8	122.9	C4'—C5'—H5'B	114.2
N3—C2—N1	128.1 (2)	H5'A—C5'—H5'B	111.3

Hydrogen-bond geometry (Å, °)

Cg1 is the centroid of ring N1/C2/N3/C4/C5/C6.

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O1'—H1'...N7 ⁱ	0.84	2.03	2.853 (3)	168
C8—H8...O1' ⁱⁱ	0.95	2.27	3.148 (2)	153
C2—H2...N3 ⁱⁱⁱ	0.95	2.48	3.311 (3)	146
C2'—H2'...Cg1 ^{iv}	0.99	2.84	3.628 (2)	136

Symmetry codes: (i) $x-1/2, -y+1/2, z-1/2$; (ii) $-x, -y+1, -z$; (iii) $-x+1, -y+2, -z$; (iv) $x-3/2, -y+1/2, z-3/2$.