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Benzyl 2-amino-6-chloro-9*H*-purine-9-carboxylate

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.034
wR factor = 0.095
Data-to-parameter ratio = 14.8

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

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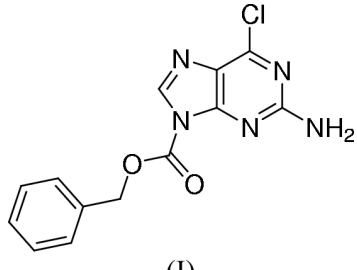
The title compound, $C_{13}H_{10}ClN_5O_2$, crystallizes with two molecules in the asymmetric unit. These are connected by five hydrogen bonds, *viz.* three N—H···N interactions, two longer C=O···H—N interactions, bifurcated at the O atom, and a C—H···N contact.

Comment

The chemistry of purines has been largely driven in recent years by the desire to synthesize oligonucleotides and their analogues as well as novel purine-containing nucleosides for a wide range of medicinal applications (Vyle & Howarth, 2001). We have previously reported the synthesis and polymerization of lipophilic polyamide nucleic acids (PNA) as potential colorimetric diagnostics (Howarth, Lindsell *et al.*, 2003), and the design and synthesis of true peptide mimics of DNA for possible use as antigene agents (Howarth & Wakelin, 1997; Howarth, Wakelin & Walker, 2003). During these studies, we have encountered numerous difficulties in preparing the required *N*-2-benzyloxycarbonyl-protected guanine monomers from 2-amino-6-chloropurine (Howarth & Wakelin, 1997). Inspired by the work reported by Dey & Garner (2000) on the synthesis of tris-*tert*-butoxycarbonyl 2-amino-6-chloropurine, we decided to employ a similar strategy for preparing these monomers. As had been found by Dey & Garner (2000), this reaction afforded a single product. However, analysis of the product by ^{1}H NMR spectroscopy showed the presence of only one benzyloxycarbonyl group rather than three, which had been the case when 2-amino-6-chloropurine was treated with di-*tert*-butyl dicarbonate under analogous conditions (Dey & Garner, 2000). The exact identity of the monobenzyloxycarbonyl-protected product was revealed to be that of the title compound, (I), by a single-crystal X-ray study.



(I)

Compound (I) crystallizes as two crystallographically independent molecules (*A* and *B*) (Fig. 1). These differ in the relative ring orientations about the C10—N9 bonds [C4*A*—N9*A*—C10*A*—O10*A* = −5.0 (2)° and C4*B*—N9*B*—C10*B*—

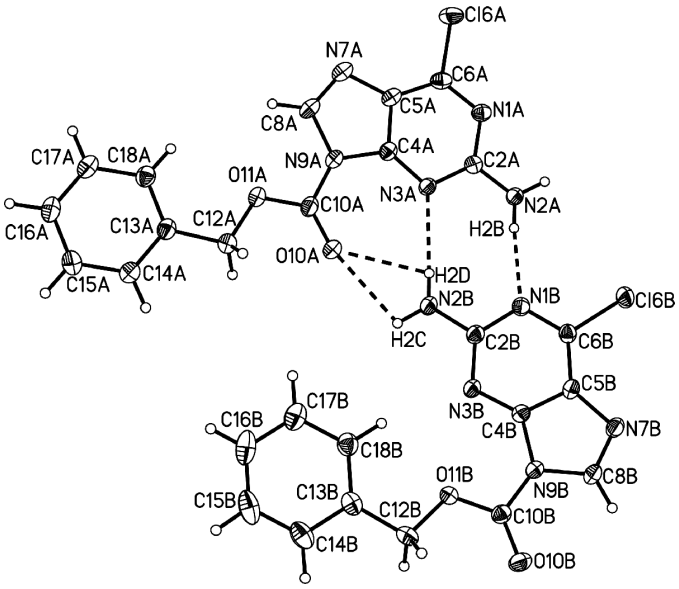


Figure 1
Perspective view of the asymmetric unit in (I), with hydrogen bonds shown as dashed lines. Displacement ellipsoids are shown at the 50% probability level and H atoms have arbitrary radii of 0.1 Å for clarity.

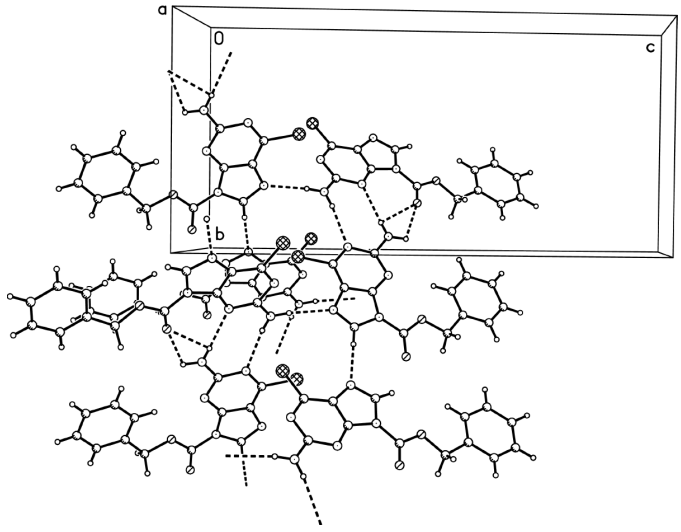


Figure 2
View of the packing arrangement for (I). Dashed lines indicate hydrogen bonds.

O10*B* = −173.60 (13)°]. The independent molecules *A* and *B* have different hydrogen-bonding arrangements. There is extensive hydrogen bonding between the two crystallographically independent molecules. They are connected by five intermolecular hydrogen bonds [N2*A*—H2*B*···N1*B*, N2*B*—H2*D*···N3*A*, N2*B*—H2*C*···O10*A*, N2*B*—H2*D*···O10*A* and N2*A*—H2*B*···N7*B*^{i} [symmetry code: (i) $2-x, y-\frac{1}{2}, \frac{1}{2}-z$; Table 1], where the N—H···N contacts are the shortest. The first four hydrogen bonds are shown in Fig. 1. The hydrogen-bonding links between molecules *A* and *B* result in the formation of two eight-membered rings. The N—H···N contacts have a symmetrical carboxylic acid dimer motif, $R_2^2(8)$ (Bernstein *et al.*, 1995). The geometry of the N—H···O contact is very different, the angles at H2*C* and H2*D* being 101.6 (13) and 101.1 (13)°, respectively. The fifth intermolecular contact is another N—H···N contact, N2*A*—H2*B*···N7*B*^{i}, which is almost parallel to the *c* axis and gives rise to an infinite chain that runs parallel to the *b* axis, shown in Fig. 2. However, N7*A* does not take part in such a close intermolecular contact. The closest contact for N7*A* is C8*B*—H8*B*···N7*A*^{ii} [symmetry code (ii) $1+x, 1+y, z$].

Experimental

Dibenzyl dicarbonate (2.40 ml, 9.42 mmol, 4 equivalents) was added to a stirred solution of 2-amino-6-chloropurine (0.40 g, 2.36 mmol, 1 equivalent) and DMAP (dimethylaminopyridine, 0.03 g, 0.1 equivalent) in anhydrous dimethylformamide (50 ml) at room temperature under argon, and the resulting mixture was left to stir for 18 h. Subsequently, the solvent was removed *in vacuo* and the residue was purified by column chromatography using ethyl acetate/petroleum ether (2:1) as the eluting solvent. The product-containing fractions were combined to afford a brown oily solid, which was further purified by trituration with diethyl ether to give (I) as a colourless solid (yield 0.80 g, 26%). Compound (I) was crystallized from deuterochloroform. M.p. 417–418 K; R_f 0.35 (ethyl acetate/petroleum ether, 2:1). Analysis found: C 51.18, H 3.32, N 22.95%; $C_{13}H_{10}O_2N_5Cl$ requires: C 51.41, H 3.32, N 23.06%. ν max (KBr, cm^{-1}): 3497, 3313, 3198, 1775, 1742, 1626, 1561, 1512, 1485, 1395, 1368, 1301, 1192, 1175 and 1107; ^{1}H NMR (200 MHz, $CDCl_3$): δ 5.49 (*s*, 2H), 5.64 (*br s*, 2H), 7.34–7.53 (*m*, 5H), 8.23 (*s*, 1H); ^{13}C NMR (50 MHz, $CDCl_3$): δ 70.2, 128.8, 129.2, 133.5, 139.6, 147.2, 152.4, 153.0, 160.4. NMR spectra were recorded on Bruker DPX400 and AC200 spectrometers, from $CDCl_3$ solutions at 293 K.

Crystal data

$C_{13}H_{10}ClN_5O_2$	D_x = 1.529 Mg m^{-3}
M_r = 303.71	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 7840 reflections
a = 9.2724 (5) Å	θ = 2.2–27.4°
b = 11.7943 (6) Å	μ = 0.30 mm^{-1}
c = 24.4404 (11) Å	T = 100 (2) K
β = 99.180 (2)°	Block, colourless
V = 2638.6 (2) Å^3	0.20 × 0.16 × 0.14 mm
Z = 8	

Data collection

Bruker–Nonius APEX2 CCD area-detector diffractometer	6497 independent reflections
φ and ω scans	5110 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)	R_{int} = 0.050
T_{min} = 0.942, T_{max} = 0.959	θ_{max} = 28.2°
90 190 measured reflections	$h = -12 \rightarrow 12$
	$k = -15 \rightarrow 15$
	$l = -32 \rightarrow 32$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0493P)^2 + 0.5642P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.034	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.095	$\Delta\rho_{max}$ = 0.31 e Å^{-3}
S = 1.08	$\Delta\rho_{min}$ = −0.26 e Å^{-3}
6497 reflections	
440 parameters	
Only H-atom coordinates refined	

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

D—H···A	D—H	H···A	D···A	D—H···A
N2A—H2A···N7B^{i}	0.890 (17)	2.197 (17)	3.0771 (17)	169.9 (14)
N2A—H2B···N1B	0.861 (17)	2.212 (18)	3.0634 (16)	169.9 (15)
N2B—H2D···N3A	0.836 (18)	2.302 (19)	3.1378 (17)	177.8 (17)
N2B—H2D···O10A	0.836 (18)	2.464 (17)	2.7501 (15)	101.1 (13)
N2B—H2C···O10A	0.886 (18)	2.432 (17)	2.7501 (15)	101.6 (13)
C8B—H8B···N7A^{ii}	0.915 (17)	2.375 (17)	3.2779 (18)	169.1 (14)

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

The coordinates of all H atoms were refined freely, whilst the isotropic displacement parameters were treated as riding on the bound atom such that U_{iso}(H) = 1.2U_{eq}(C,N).

Data collection: *APEX2* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7840 reflections
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Block, colourless
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Data collection

Bruker–Nonius Apex2 CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
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$T_{min} = 0.942$, $T_{max} = 0.959$
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$\theta_{max} = 28.2°$, $\theta_{min} = 2.4°$
$h = -12 \rightarrow 12$
$k = -15 \rightarrow 15$
$l = -32 \rightarrow 32$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.095$
$S = 1.08$
6497 reflections
440 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
Only H-atom coordinates refined
$w = 1/[\sigma^2(F_o^2) + (0.0493P)^2 + 0.5642P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1A	0.61778 (12)	0.60392 (9)	0.27356 (5)	0.0221 (2)
C2A	0.68744 (14)	0.68392 (11)	0.30916 (5)	0.0198 (3)
N2A	0.79239 (13)	0.74276 (10)	0.29036 (5)	0.0216 (2)
H2A	0.8060 (17)	0.7303 (13)	0.2556 (7)	0.026*
H2B	0.8279 (17)	0.8019 (15)	0.3083 (7)	0.026*
N3A	0.66031 (12)	0.70736 (9)	0.36113 (5)	0.0200 (2)
C4A	0.55724 (14)	0.64099 (11)	0.37592 (5)	0.0205 (3)
C5A	0.47945 (14)	0.55588 (11)	0.34388 (6)	0.0220 (3)
C6A	0.51639 (15)	0.54252 (11)	0.29143 (6)	0.0235 (3)
Cl6A	0.42563 (4)	0.44372 (3)	0.246400 (16)	0.03247 (10)
N7A	0.38063 (13)	0.50166 (10)	0.37267 (5)	0.0257 (3)
C8A	0.39612 (15)	0.55187 (12)	0.42032 (6)	0.0251 (3)
H8A	0.3424 (18)	0.5360 (14)	0.4494 (7)	0.030*
N9A	0.50257 (12)	0.63862 (10)	0.42625 (5)	0.0226 (2)
C10A	0.53597 (15)	0.71210 (12)	0.47149 (6)	0.0232 (3)
O10A	0.62625 (11)	0.78565 (9)	0.47466 (4)	0.0296 (2)
O11A	0.45297 (10)	0.68712 (8)	0.50966 (4)	0.0253 (2)
C12A	0.46867 (17)	0.76553 (13)	0.55606 (6)	0.0279 (3)
H12A	0.5735 (19)	0.7687 (14)	0.5731 (7)	0.033*
H12B	0.4332 (18)	0.8442 (15)	0.5419 (7)	0.033*
C13A	0.37689 (15)	0.72644 (12)	0.59783 (6)	0.0238 (3)
C14A	0.39219 (17)	0.78581 (14)	0.64765 (6)	0.0298 (3)
H14A	0.4569 (19)	0.8464 (15)	0.6517 (7)	0.036*
C15A	0.31117 (18)	0.75506 (14)	0.68822 (7)	0.0342 (4)
H15A	0.3227 (19)	0.7941 (16)	0.7231 (8)	0.041*
C16A	0.21629 (17)	0.66388 (14)	0.68009 (6)	0.0313 (3)
H16A	0.1622 (19)	0.6419 (14)	0.7096 (7)	0.038*
C17A	0.20041 (16)	0.60416 (13)	0.63074 (6)	0.0278 (3)
H17A	0.1355 (19)	0.5397 (15)	0.6259 (7)	0.033*
C18A	0.28058 (15)	0.63531 (12)	0.58904 (6)	0.0247 (3)
H18A	0.2728 (17)	0.5952 (14)	0.5553 (7)	0.030*
N1B	0.89220 (12)	0.97073 (9)	0.34342 (4)	0.0214 (2)
C2B	0.87924 (15)	0.99619 (11)	0.39669 (5)	0.0214 (3)
N2B	0.79408 (15)	0.92729 (11)	0.42127 (5)	0.0281 (3)
H2C	0.7907 (19)	0.9371 (14)	0.4570 (8)	0.034*
H2D	0.7575 (19)	0.8698 (16)	0.4045 (7)	0.034*
N3B	0.94051 (12)	1.08495 (9)	0.42677 (5)	0.0218 (2)
C4B	1.01863 (14)	1.15002 (11)	0.39844 (5)	0.0199 (3)
C5B	1.04168 (14)	1.13338 (11)	0.34371 (5)	0.0208 (3)
C6B	0.97127 (15)	1.03962 (11)	0.31767 (5)	0.0207 (3)

Cl6B	0.97980 (4)	1.01065 (3)	0.248990 (14)	0.02927 (10)
N7B	1.12714 (13)	1.21991 (10)	0.32651 (5)	0.0235 (2)
C8B	1.15482 (15)	1.28696 (12)	0.36905 (6)	0.0231 (3)
H8B	1.2067 (17)	1.3530 (14)	0.3698 (6)	0.028*
N9B	1.09377 (12)	1.25019 (9)	0.41508 (5)	0.0212 (2)
C10B	1.09411 (14)	1.31502 (11)	0.46355 (5)	0.0213 (3)
O10B	1.15479 (11)	1.40544 (8)	0.47069 (4)	0.0289 (2)
O11B	1.01832 (10)	1.26210 (8)	0.49711 (4)	0.0226 (2)
C12B	1.00086 (16)	1.31964 (13)	0.54817 (6)	0.0251 (3)
H12C	1.0960 (18)	1.3284 (14)	0.5704 (7)	0.030*
H12D	0.9605 (17)	1.3948 (15)	0.5387 (6)	0.028 (4)*
C13B	0.89917 (15)	1.24971 (12)	0.57672 (6)	0.0245 (3)
C14B	0.87699 (18)	1.28219 (14)	0.62963 (6)	0.0322 (3)
H14B	0.9291 (19)	1.3492 (16)	0.6463 (7)	0.039*
C15B	0.7849 (2)	1.22082 (16)	0.65775 (7)	0.0394 (4)
H15B	0.773 (2)	1.2449 (16)	0.6929 (8)	0.047*
C16B	0.71261 (19)	1.12573 (17)	0.63370 (7)	0.0411 (4)
H16B	0.651 (2)	1.0837 (17)	0.6524 (8)	0.049*
C17B	0.73360 (18)	1.09264 (15)	0.58100 (7)	0.0354 (4)
H17B	0.682 (2)	1.0251 (16)	0.5640 (8)	0.043*
C18B	0.82698 (16)	1.15414 (13)	0.55256 (6)	0.0278 (3)
H18B	0.8412 (18)	1.1321 (14)	0.5157 (7)	0.033*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1A	0.0207 (6)	0.0198 (5)	0.0251 (6)	0.0002 (4)	0.0013 (4)	−0.0006 (4)
C2A	0.0191 (6)	0.0184 (6)	0.0215 (6)	0.0023 (5)	0.0023 (5)	0.0011 (5)
N2A	0.0245 (6)	0.0206 (6)	0.0208 (6)	−0.0034 (5)	0.0067 (5)	−0.0023 (5)
N3A	0.0195 (5)	0.0194 (5)	0.0213 (5)	−0.0020 (4)	0.0037 (4)	0.0012 (4)
C4A	0.0192 (6)	0.0194 (6)	0.0229 (6)	0.0008 (5)	0.0037 (5)	0.0027 (5)
C5A	0.0177 (6)	0.0190 (6)	0.0293 (7)	−0.0014 (5)	0.0037 (5)	0.0017 (5)
C6A	0.0203 (7)	0.0196 (6)	0.0289 (7)	0.0006 (5)	−0.0010 (5)	−0.0017 (5)
Cl6A	0.03002 (19)	0.02945 (19)	0.0366 (2)	−0.00807 (15)	0.00110 (15)	−0.00987 (15)
N7A	0.0202 (6)	0.0228 (6)	0.0347 (7)	−0.0033 (5)	0.0065 (5)	0.0016 (5)
C8A	0.0212 (7)	0.0208 (7)	0.0340 (8)	−0.0038 (5)	0.0069 (6)	0.0033 (6)
N9A	0.0210 (6)	0.0230 (6)	0.0244 (6)	−0.0050 (5)	0.0059 (4)	0.0016 (4)
C10A	0.0198 (7)	0.0272 (7)	0.0232 (7)	−0.0014 (5)	0.0049 (5)	0.0049 (5)
O10A	0.0298 (5)	0.0343 (6)	0.0256 (5)	−0.0129 (5)	0.0074 (4)	−0.0011 (4)
O11A	0.0252 (5)	0.0288 (5)	0.0233 (5)	−0.0062 (4)	0.0080 (4)	0.0009 (4)
C12A	0.0286 (8)	0.0328 (8)	0.0230 (7)	−0.0079 (6)	0.0058 (6)	−0.0008 (6)
C13A	0.0204 (7)	0.0273 (7)	0.0240 (7)	0.0005 (5)	0.0042 (5)	0.0045 (5)
C14A	0.0289 (8)	0.0333 (8)	0.0276 (7)	−0.0075 (6)	0.0057 (6)	0.0002 (6)
C15A	0.0362 (9)	0.0422 (9)	0.0259 (8)	−0.0039 (7)	0.0108 (7)	−0.0025 (7)
C16A	0.0288 (8)	0.0370 (8)	0.0302 (8)	−0.0001 (6)	0.0115 (6)	0.0060 (6)
C17A	0.0221 (7)	0.0278 (7)	0.0345 (8)	−0.0009 (6)	0.0077 (6)	0.0053 (6)
C18A	0.0210 (7)	0.0273 (7)	0.0260 (7)	0.0011 (5)	0.0045 (5)	0.0026 (6)
N1B	0.0248 (6)	0.0194 (5)	0.0206 (5)	−0.0009 (4)	0.0055 (4)	−0.0006 (4)

C2B	0.0244 (7)	0.0184 (6)	0.0222 (6)	−0.0012 (5)	0.0055 (5)	0.0006 (5)
N2B	0.0392 (7)	0.0243 (6)	0.0232 (6)	−0.0124 (5)	0.0124 (5)	−0.0036 (5)
N3B	0.0264 (6)	0.0185 (5)	0.0216 (5)	−0.0040 (4)	0.0069 (5)	0.0004 (4)
C4B	0.0205 (6)	0.0179 (6)	0.0213 (6)	−0.0009 (5)	0.0035 (5)	−0.0001 (5)
C5B	0.0226 (7)	0.0190 (6)	0.0219 (6)	−0.0001 (5)	0.0068 (5)	0.0026 (5)
C6B	0.0237 (7)	0.0197 (6)	0.0194 (6)	0.0011 (5)	0.0057 (5)	0.0001 (5)
Cl6B	0.0416 (2)	0.02730 (18)	0.02115 (16)	−0.00514 (15)	0.01167 (14)	−0.00339 (13)
N7B	0.0244 (6)	0.0220 (6)	0.0253 (6)	−0.0037 (5)	0.0075 (5)	0.0017 (5)
C8B	0.0234 (7)	0.0212 (7)	0.0258 (7)	−0.0036 (5)	0.0074 (5)	0.0029 (5)
N9B	0.0232 (6)	0.0192 (5)	0.0218 (6)	−0.0044 (4)	0.0055 (5)	−0.0003 (4)
C10B	0.0195 (6)	0.0206 (6)	0.0232 (6)	−0.0007 (5)	0.0021 (5)	0.0013 (5)
O10B	0.0322 (6)	0.0224 (5)	0.0330 (6)	−0.0078 (4)	0.0077 (4)	−0.0042 (4)
O11B	0.0265 (5)	0.0227 (5)	0.0192 (5)	−0.0049 (4)	0.0053 (4)	−0.0028 (4)
C12B	0.0278 (7)	0.0258 (7)	0.0212 (7)	0.0000 (6)	0.0027 (6)	−0.0065 (5)
C13B	0.0230 (7)	0.0285 (7)	0.0214 (7)	0.0069 (6)	0.0018 (5)	0.0012 (5)
C14B	0.0357 (9)	0.0368 (9)	0.0238 (7)	0.0123 (7)	0.0034 (6)	−0.0014 (6)
C15B	0.0439 (10)	0.0508 (10)	0.0262 (8)	0.0200 (8)	0.0140 (7)	0.0065 (7)
C16B	0.0370 (9)	0.0504 (11)	0.0405 (9)	0.0116 (8)	0.0201 (7)	0.0167 (8)
C17B	0.0304 (8)	0.0390 (9)	0.0389 (9)	−0.0006 (7)	0.0116 (7)	0.0066 (7)
C18B	0.0256 (7)	0.0324 (8)	0.0262 (7)	0.0007 (6)	0.0062 (6)	0.0007 (6)

Geometric parameters (Å, º)

N1A—C6A	1.3153 (18)	N1B—C6B	1.3195 (17)
N1A—C2A	1.3730 (17)	N1B—C2B	1.3600 (17)
C2A—N2A	1.3350 (17)	C2B—N2B	1.3403 (17)
C2A—N3A	1.3620 (17)	C2B—N3B	1.3513 (17)
N2A—H2A	0.890 (17)	N2B—H2C	0.886 (18)
N2A—H2B	0.861 (17)	N2B—H2D	0.836 (18)
N3A—C4A	1.3292 (17)	N3B—C4B	1.3240 (17)
C4A—C5A	1.4003 (18)	C4B—N9B	1.3986 (17)
C4A—N9A	1.4036 (17)	C4B—C5B	1.4017 (18)
C5A—C6A	1.388 (2)	C5B—C6B	1.3863 (19)
C5A—N7A	1.3962 (17)	C5B—N7B	1.3973 (17)
C6A—Cl6A	1.7267 (14)	C6B—Cl6B	1.7271 (13)
N7A—C8A	1.2940 (19)	N7B—C8B	1.2988 (18)
C8A—N9A	1.4130 (17)	C8B—N9B	1.4062 (17)
C8A—H8A	0.950 (17)	C8B—H8B	0.915 (17)
N9A—C10A	1.4000 (18)	N9B—C10B	1.4095 (17)
C10A—O10A	1.1994 (17)	C10B—O10B	1.2054 (16)
C10A—O11A	1.3336 (16)	C10B—O11B	1.3192 (16)
O11A—C12A	1.4525 (18)	O11B—C12B	1.4523 (16)
C12A—C13A	1.502 (2)	C12B—C13B	1.505 (2)
C12A—H12A	0.995 (18)	C12B—H12C	0.965 (17)
C12A—H12B	1.026 (18)	C12B—H12D	0.975 (17)
C13A—C18A	1.392 (2)	C13B—C18B	1.393 (2)
C13A—C14A	1.392 (2)	C13B—C14B	1.395 (2)
C14A—C15A	1.384 (2)	C14B—C15B	1.383 (2)

C14A—H14A	0.928 (18)	C14B—H14B	0.980 (18)
C15A—C16A	1.384 (2)	C15B—C16B	1.388 (3)
C15A—H15A	0.959 (19)	C15B—H15B	0.93 (2)
C16A—C17A	1.384 (2)	C16B—C17B	1.389 (2)
C16A—H16A	0.976 (18)	C16B—H16B	0.93 (2)
C17A—C18A	1.403 (2)	C17B—C18B	1.397 (2)
C17A—H17A	0.965 (18)	C17B—H17B	0.984 (19)
C18A—H18A	0.943 (17)	C18B—H18B	0.968 (17)

C6A—N1A—C2A	117.19 (12)	C6B—N1B—C2B	117.47 (11)
N2A—C2A—N3A	117.95 (12)	N2B—C2B—N3B	116.90 (12)
N2A—C2A—N1A	115.74 (12)	N2B—C2B—N1B	116.33 (12)
N3A—C2A—N1A	126.31 (12)	N3B—C2B—N1B	126.75 (12)
C2A—N2A—H2A	117.7 (10)	C2B—N2B—H2C	118.6 (11)
C2A—N2A—H2B	119.1 (11)	C2B—N2B—H2D	119.6 (12)
H2A—N2A—H2B	121.2 (15)	H2C—N2B—H2D	120.9 (16)
C4A—N3A—C2A	112.45 (11)	C4B—N3B—C2B	112.46 (11)
N3A—C4A—C5A	126.78 (12)	N3B—C4B—N9B	128.77 (12)
N3A—C4A—N9A	128.75 (12)	N3B—C4B—C5B	126.68 (12)
C5A—C4A—N9A	104.47 (11)	N9B—C4B—C5B	104.54 (11)
C6A—C5A—N7A	133.79 (13)	C6B—C5B—N7B	133.84 (12)
C6A—C5A—C4A	114.64 (12)	C6B—C5B—C4B	114.68 (11)
N7A—C5A—C4A	111.57 (12)	N7B—C5B—C4B	111.41 (11)
N1A—C6A—C5A	122.61 (12)	N1B—C6B—C5B	121.94 (12)
N1A—C6A—Cl6A	117.48 (11)	N1B—C6B—Cl6B	117.18 (10)
C5A—C6A—Cl6A	119.90 (11)	C5B—C6B—Cl6B	120.85 (10)
C8A—N7A—C5A	104.86 (12)	C8B—N7B—C5B	104.66 (11)
N7A—C8A—N9A	113.52 (12)	N7B—C8B—N9B	113.53 (12)
N7A—C8A—H8A	126.0 (10)	N7B—C8B—H8B	124.8 (10)
N9A—C8A—H8A	120.5 (10)	N9B—C8B—H8B	121.6 (10)
C10A—N9A—C4A	127.92 (11)	C4B—N9B—C8B	105.87 (11)
C10A—N9A—C8A	126.30 (11)	C4B—N9B—C10B	129.56 (11)
C4A—N9A—C8A	105.58 (11)	C8B—N9B—C10B	123.90 (11)
O10A—C10A—O11A	126.10 (13)	O10B—C10B—O11B	127.68 (13)
O10A—C10A—N9A	124.26 (12)	O10B—C10B—N9B	122.69 (12)
O11A—C10A—N9A	109.64 (11)	O11B—C10B—N9B	109.62 (11)
C10A—O11A—C12A	114.11 (11)	C10B—O11B—C12B	117.18 (11)
O11A—C12A—C13A	109.77 (12)	O11B—C12B—C13B	107.83 (11)
O11A—C12A—H12A	108.6 (10)	O11B—C12B—H12C	108.5 (10)
C13A—C12A—H12A	110.5 (10)	C13B—C12B—H12C	112.4 (10)
O11A—C12A—H12B	109.0 (9)	O11B—C12B—H12D	108.2 (9)
C13A—C12A—H12B	108.8 (9)	C13B—C12B—H12D	111.3 (10)
H12A—C12A—H12B	110.2 (13)	H12C—C12B—H12D	108.6 (14)
C18A—C13A—C14A	119.79 (13)	C18B—C13B—C14B	118.88 (14)
C18A—C13A—C12A	123.44 (13)	C18B—C13B—C12B	122.76 (13)
C14A—C13A—C12A	116.77 (13)	C14B—C13B—C12B	118.36 (13)
C15A—C14A—C13A	120.23 (15)	C15B—C14B—C13B	120.72 (16)
C15A—C14A—H14A	122.7 (11)	C15B—C14B—H14B	121.2 (10)

C13A—C14A—H14A	117.0 (11)	C13B—C14B—H14B	118.0 (11)
C16A—C15A—C14A	120.40 (15)	C14B—C15B—C16B	120.47 (15)
C16A—C15A—H15A	118.6 (11)	C14B—C15B—H15B	118.1 (13)
C14A—C15A—H15A	120.9 (11)	C16B—C15B—H15B	121.4 (12)
C15A—C16A—C17A	119.83 (14)	C15B—C16B—C17B	119.34 (16)
C15A—C16A—H16A	119.3 (10)	C15B—C16B—H16B	121.1 (12)
C17A—C16A—H16A	120.8 (10)	C17B—C16B—H16B	119.6 (12)
C16A—C17A—C18A	120.31 (14)	C16B—C17B—C18B	120.34 (17)
C16A—C17A—H17A	119.0 (10)	C16B—C17B—H17B	119.3 (11)
C18A—C17A—H17A	120.6 (10)	C18B—C17B—H17B	120.4 (11)
C13A—C18A—C17A	119.42 (14)	C13B—C18B—C17B	120.23 (14)
C13A—C18A—H18A	118.7 (10)	C13B—C18B—H18B	119.0 (10)
C17A—C18A—H18A	121.8 (10)	C17B—C18B—H18B	120.8 (10)
C6A—N1A—C2A—N2A	178.30 (12)	C6B—N1B—C2B—N2B	177.65 (12)
C6A—N1A—C2A—N3A	−1.07 (19)	C6B—N1B—C2B—N3B	−0.7 (2)
N2A—C2A—N3A—C4A	−177.89 (12)	N2B—C2B—N3B—C4B	−177.94 (13)
N1A—C2A—N3A—C4A	1.47 (18)	N1B—C2B—N3B—C4B	0.4 (2)
C2A—N3A—C4A—C5A	−0.68 (19)	C2B—N3B—C4B—N9B	177.97 (13)
C2A—N3A—C4A—N9A	178.64 (12)	C2B—N3B—C4B—C5B	−0.8 (2)
N3A—C4A—C5A—C6A	−0.4 (2)	N3B—C4B—C5B—C6B	1.4 (2)
N9A—C4A—C5A—C6A	−179.85 (11)	N9B—C4B—C5B—C6B	−177.62 (11)
N3A—C4A—C5A—N7A	179.15 (12)	N3B—C4B—C5B—N7B	178.68 (13)
N9A—C4A—C5A—N7A	−0.31 (15)	N9B—C4B—C5B—N7B	−0.33 (15)
C2A—N1A—C6A—C5A	−0.22 (19)	C2B—N1B—C6B—C5B	1.3 (2)
C2A—N1A—C6A—Cl6A	178.70 (9)	C2B—N1B—C6B—Cl6B	−176.89 (10)
N7A—C5A—C6A—N1A	−178.53 (14)	N7B—C5B—C6B—N1B	−178.12 (14)
C4A—C5A—C6A—N1A	0.87 (19)	C4B—C5B—C6B—N1B	−1.61 (19)
N7A—C5A—C6A—Cl6A	2.6 (2)	N7B—C5B—C6B—Cl6B	0.0 (2)
C4A—C5A—C6A—Cl6A	−178.02 (10)	C4B—C5B—C6B—Cl6B	176.55 (10)
C6A—C5A—N7A—C8A	179.87 (15)	C6B—C5B—N7B—C8B	176.41 (15)
C4A—C5A—N7A—C8A	0.45 (15)	C4B—C5B—N7B—C8B	−0.18 (15)
C5A—N7A—C8A—N9A	−0.41 (16)	C5B—N7B—C8B—N9B	0.65 (16)
N3A—C4A—N9A—C10A	5.5 (2)	N3B—C4B—N9B—C8B	−178.30 (14)
C5A—C4A—N9A—C10A	−175.06 (13)	C5B—C4B—N9B—C8B	0.67 (14)
N3A—C4A—N9A—C8A	−179.39 (13)	N3B—C4B—N9B—C10B	−7.6 (2)
C5A—C4A—N9A—C8A	0.06 (14)	C5B—C4B—N9B—C10B	171.37 (13)
N7A—C8A—N9A—C10A	175.45 (13)	N7B—C8B—N9B—C4B	−0.87 (16)
N7A—C8A—N9A—C4A	0.23 (16)	N7B—C8B—N9B—C10B	−172.23 (12)
C4A—N9A—C10A—O10A	−5.0 (2)	C4B—N9B—C10B—O10B	−173.60 (13)
C8A—N9A—C10A—O10A	−179.14 (14)	C8B—N9B—C10B—O10B	−4.4 (2)
C4A—N9A—C10A—O11A	175.20 (12)	C4B—N9B—C10B—O11B	5.50 (19)
C8A—N9A—C10A—O11A	1.04 (19)	C8B—N9B—C10B—O11B	174.71 (12)
O10A—C10A—O11A—C12A	5.9 (2)	O10B—C10B—O11B—C12B	2.0 (2)
N9A—C10A—O11A—C12A	−174.26 (11)	N9B—C10B—O11B—C12B	−177.00 (11)
C10A—O11A—C12A—C13A	−177.07 (12)	C10B—O11B—C12B—C13B	174.38 (11)
O11A—C12A—C13A—C18A	−6.4 (2)	O11B—C12B—C13B—C18B	−6.13 (18)
O11A—C12A—C13A—C14A	173.23 (12)	O11B—C12B—C13B—C14B	173.70 (12)

C18A—C13A—C14A—C15A	−0.5 (2)	C18B—C13B—C14B—C15B	0.0 (2)
C12A—C13A—C14A—C15A	179.89 (14)	C12B—C13B—C14B—C15B	−179.89 (14)
C13A—C14A—C15A—C16A	1.2 (2)	C13B—C14B—C15B—C16B	−0.1 (2)
C14A—C15A—C16A—C17A	−1.1 (2)	C14B—C15B—C16B—C17B	0.0 (2)
C15A—C16A—C17A—C18A	0.3 (2)	C15B—C16B—C17B—C18B	0.3 (2)
C14A—C13A—C18A—C17A	−0.3 (2)	C14B—C13B—C18B—C17B	0.3 (2)
C12A—C13A—C18A—C17A	179.33 (14)	C12B—C13B—C18B—C17B	−179.87 (14)
C16A—C17A—C18A—C13A	0.4 (2)	C16B—C17B—C18B—C13B	−0.4 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2*A*—H2*A*···N7*B*^{i}	0.890 (17)	2.197 (17)	3.0771 (17)	169.9 (14)
N2*A*—H2*B*···N1*B*	0.861 (17)	2.212 (18)	3.0634 (16)	169.9 (15)
N2*B*—H2*D*···N3*A*	0.836 (18)	2.302 (19)	3.1378 (17)	177.8 (17)
N2*B*—H2*D*···O10*A*	0.836 (18)	2.464 (17)	2.7501 (15)	101.1 (13)
N2*B*—H2*C*···O10*A*	0.886 (18)	2.432 (17)	2.7501 (15)	101.6 (13)
C8*B*—H8*B*···N7*A*^{ii}	0.915 (17)	2.375 (17)	3.2779 (18)	169.1 (14)

Symmetry codes: (i) −*x*+2, *y*−1/2, −*z*+1/2; (ii) *x*+1, *y*+1, *z*.