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(*E*)-9-(But-2-en-1-yl)-6-chloro-9*H*-purine

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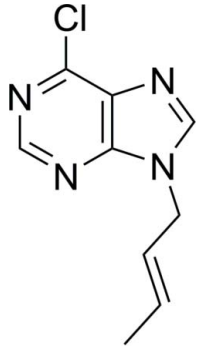
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; disorder in main residue; R factor = 0.027; wR factor = 0.070; data-to-parameter ratio = 15.6.

The asymmetric unit of the title compound, $C_9H_9ClN_4$, contains two molecules. In the crystal, the molecules are ordered in a chain-like fashion along the *a* axis, and form layers offset relative to the *C* plane by approximately 30°. This ordering does not, however, appear to be directed by classical hydrogen bonding.The allylic side chains of both independent molecules are disordered, with occupancies of 0.870 (4) and 0.934 (3) for the major components. The disorder components represent two possible spatial orientations of the atoms around the C=C double bond.

Related literature

For synthetic background and applications, see Kania & Gundersen (2013).



Experimental

Crystal data

$C_9H_9ClN_4$
M_r = 208.65
Triclinic, $P\overline{1}$
a = 8.1818 (11) Å
b = 9.7103 (13) Å
c = 13.9435 (19) Å
α = 69.642 (1)°
β = 75.448 (1)°
γ = 67.032 (1)°
V = 947.6 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.37 mm^{-1}
T = 100 K
1.13 × 0.35 × 0.22 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: numerical (*SADABS*; Bruker, 2005)
T_{min} = 0.683, T_{max} = 0.924
10801 measured reflections
4428 independent reflections
4103 reflections with $I > 2\sigma(I)$
R_{int} = 0.017

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.070
S = 1.04
4428 reflections
283 parameters
4 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2004) and *ChemBioDraw Ultra* (CambridgeSoft, 2009); software used to prepare material for publication: *publCIF* (Westrip, 2010).

We acknowledge the support from the Norwegian Research Council (KOSK II, project 184929 and RENERGI, project 190980) and from the Department of Chemistry, UiO. We also acknowledge Dr David S. Wragg for invaluable assistance with the refinement.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LR2100).

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Kania, J. & Gundersen, L.-L. (2013). *Eur. J. Org. Chem.* pp. 2008–2019.
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supplementary materials

Acta Cryst. (2013). E**69**, o849 [doi:10.1107/S1600536813010416]

(*E*)-9-(But-2-en-1-yl)-6-chloro-9*H*-purine

Fredrik Lundvall, Jindrich Kania and Lise-Lotte Gundersen

Comment

The title compound is an *N*-allyl substituted purine intended as a starting material for investigation of the double bond rearrangement in *N*-allylic systems. (*E*)-9-(but-2-en-1-yl)-6-chloro-9*H*-purine was recrystallized from a mixture of *E*/*Z*-isomers in order to obtain the pure *E*-isomer for the following rearrangement study. The structure and purity of this compound was originally determined by NMR. Further structure elucidation by SXRD supports the conclusions in our previous report (compound **14*a***, Kania *et al.*, 2013).

The structure of the title compound, $C_9H_9ClN_4$, has a triclinic P -1 symmetry. The asymmetric unit consists of two molecules of the title compound, with the full content of the unit cell generated by symmetry operations. The molecule has a planar bicyclic motif with an *N*-allylic chain oriented out of the plane described by the bicyclic main body. During the initial refinement of the structure it became apparent that the *N*-allylic chain is slightly disordered, with two possible spacial orientations. By using the PART instruction in *SHELXL*, the two possible orientations of the chain (A and B) were refined individually for the two molecules that form the asymmetric unit (residue 1 and 2). Orientation A is clearly preferred in the solid state, with the minor orientation B present in about 13% and 6.6% abundance for residue 1 and 2 respectively. The limited occupancy of the minor components prompted the use of restraints to achieve satisfactory refinement of the structure. We employed the SADI instruction in *SHELXL* and restrained the C10, C11B—C13B and C20, C21B—C23B bond distances in the minor components to be the same as the corresponding distances in the major components (C10, C11A—C13A and C20, C21A—C23A). This ensured realistic bond distances in the chains. Furthermore, C11B—C13B and C21B—C23B were refined isotropically since anisotropic refinement did not give realistic thermal parameters due to the low occupancy of the sites.

Experimental

The title compound was synthesized by the method described in Kania *et al.* (2013) (compound **14*a***).

Refinement

H-atoms were positioned geometrically at distances of 0.95 Å (CH), 0.99 Å (CH_2) and 0.98 Å (CH_3), and refined using a riding model with U_{iso} (H)=1.2 U_{eq} (CH and CH_2) and U_{iso} (H)=1.5 U_{eq} (CH_3)

Computing details

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2004) and *ChemBioDraw Ultra* (CambridgeSoft, 2009); software used to prepare material for publication: *publCIF* (Westrip, 2010).

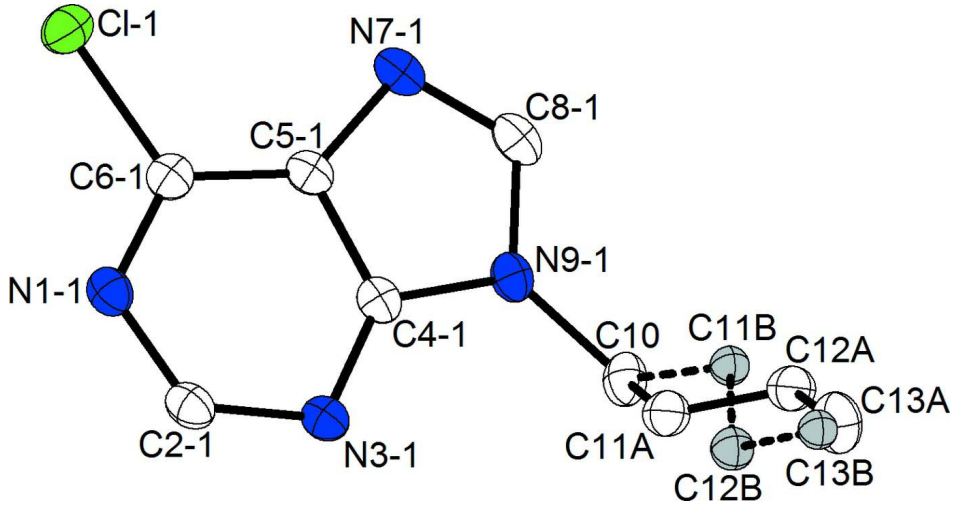

Figure 1

[One molecular unit of the title compound (residue 1, both A and B orientation) with atom labels and 50% probability displacement ellipsoids. Hydrogen atoms are omitted for clarity.]

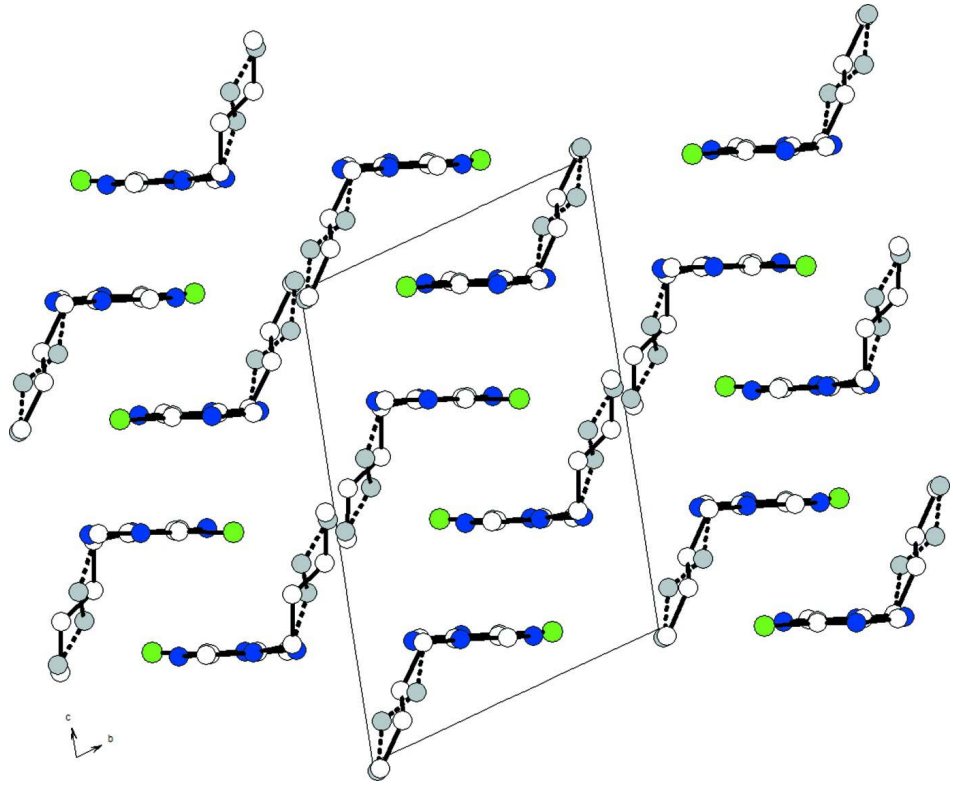

Figure 2

[Packing diagram of the title compound viewed along the *a* axis.]

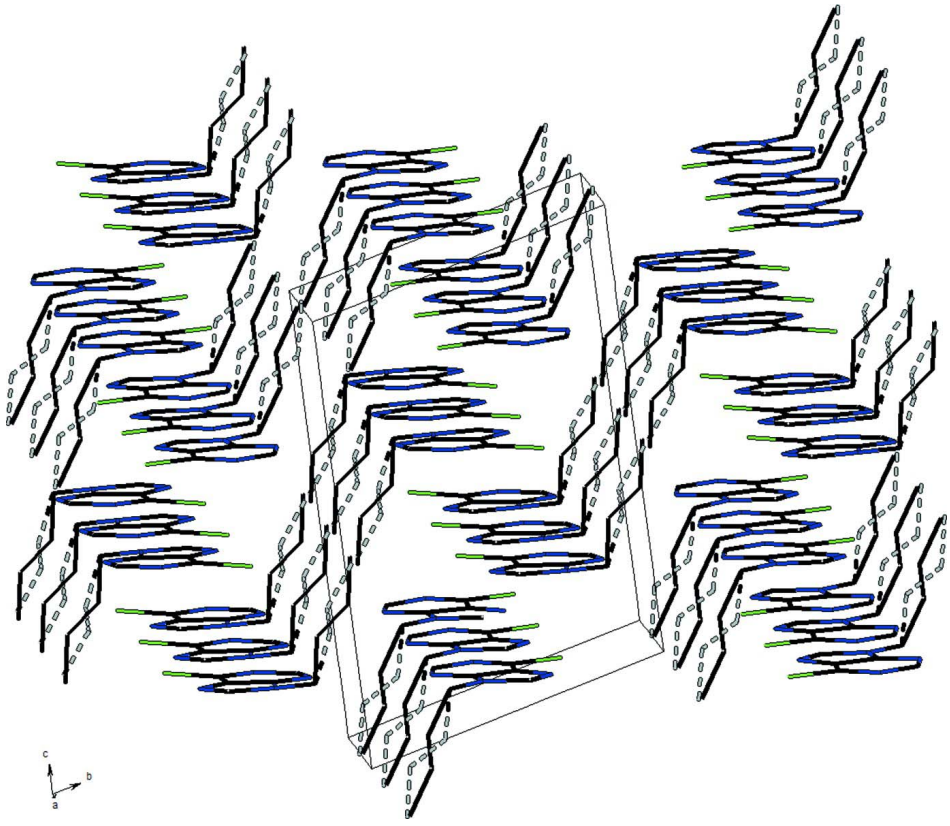

Figure 3

[Graphical projection of the packing of (*E*)-9-(but-2-en-1-yl)-6-chloro-9*H*-purine, $C_9H_9ClN_4$.]

(*E*)-9-(But-2-en-1-yl)-6-chloro-9*H*-purine

Crystal data

$C_9H_9ClN_4$
$M_r = 208.65$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 8.1818\ (11)$ Å
$b = 9.7103\ (13)$ Å
$c = 13.9435\ (19)$ Å
$\alpha = 69.642\ (1)°$
$\beta = 75.448\ (1)°$
$\gamma = 67.032\ (1)°$
$V = 947.6\ (2)$ Å^3
$Z = 4$
$F(000) = 432$
$D_x = 1.462$ Mg m^{-3}
Melting point: 339 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 8169 reflections
$\theta = 2.5–28.8°$
$\mu = 0.37$ mm^{-1}
$T = 100$ K
Rod, colourless
1.13 × 0.35 × 0.22 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: numerical (*SADABS*; Bruker, 2005)
$T_{min} = 0.683$, $T_{max} = 0.924$
10801 measured reflections
4428 independent reflections
4103 reflections with $I > 2\sigma(I)$
$R_{int} = 0.017$
$\theta_{max} = 28.8°$, $\theta_{min} = 1.6°$
$h = -11\rightarrow10$
$k = -12\rightarrow12$
$l = -18\rightarrow18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.070$
$S = 1.04$
4428 reflections
283 parameters
4 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
$w = 1/[\sigma^2(F_o^2) + (0.0292P)^2 + 0.3823P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.34$ e Å^{-3}
$\Delta\rho_{min} = -0.30$ e Å^{-3}

Special details

Experimental. Due to the weak scattering nature of the compound, a large crystal (1.13x0.35x0.22 mm) was used to get satisfactory data for refinement.

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}	Occ. (<1)
Cl-1	0.80714 (4)	0.34683 (3)	0.90852 (2)	0.02519 (8)	
Cl-2	0.56350 (4)	0.66115 (3)	0.58169 (2)	0.02513 (8)	
N1-1	0.68069 (12)	0.64840 (11)	0.83023 (7)	0.02118 (19)	
N3-1	0.37832 (12)	0.80607 (11)	0.79779 (7)	0.01992 (18)	
N9-1	0.18663 (12)	0.65085 (11)	0.83852 (7)	0.01988 (18)	
N7-1	0.38398 (13)	0.41103 (11)	0.89887 (7)	0.02212 (19)	
N9-2	0.18425 (12)	0.33227 (11)	0.67452 (7)	0.01990 (18)	
N3-2	0.49116 (12)	0.19050 (10)	0.70937 (7)	0.02115 (19)	
N1-2	0.66555 (13)	0.36050 (11)	0.66514 (7)	0.02249 (19)	
N7-2	0.19524 (13)	0.57777 (11)	0.61328 (7)	0.0240 (2)	
C2-1	0.54905 (15)	0.78443 (13)	0.79925 (8)	0.0217 (2)	
H2-1	0.5818	0.8756	0.7757	0.026*	
C4-1	0.34503 (14)	0.67189 (12)	0.83172 (7)	0.0180 (2)	
C10	0.01759 (15)	0.77488 (14)	0.81086 (8)	0.0229 (2)	
H10A	−0.0707	0.7281	0.8113	0.028*	
H10B	0.0382	0.8425	0.7403	0.028*	
C11A	−0.05515 (18)	0.87065 (15)	0.88579 (10)	0.0221 (3)	0.870 (4)
H11A	0.0139	0.9269	0.8901	0.027*	0.870 (4)
C12A	−0.21002 (18)	0.88140 (15)	0.94633 (10)	0.0218 (3)	0.870 (4)
H12A	−0.2772	0.8236	0.9419	0.026*	0.870 (4)
C13A	−0.2880 (4)	0.9771 (4)	1.0211 (2)	0.0282 (7)	0.870 (4)
H13A	−0.3312	0.9147	1.0874	0.042*	0.870 (4)
H13B	−0.3880	1.0691	0.9940	0.042*	0.870 (4)
H13C	−0.1958	1.0101	1.0308	0.042*	0.870 (4)

C11B	−0.1202 (11)	0.8205 (9)	0.9029 (6)	0.015 (2)*	0.130 (4)
H11B	−0.1846	0.7520	0.9435	0.018*	0.130 (4)
C12B	−0.1540 (11)	0.9477 (9)	0.9284 (6)	0.018 (2)*	0.130 (4)
H12B	−0.1012	1.0218	0.8819	0.022*	0.130 (4)
C13B	−0.267 (3)	0.987 (3)	1.0233 (14)	0.015 (4)*	0.130 (4)
H13D	−0.3374	0.9172	1.0560	0.022*	0.130 (4)
H13E	−0.3476	1.0951	1.0050	0.022*	0.130 (4)
H13F	−0.1897	0.9748	1.0714	0.022*	0.130 (4)
C6-1	0.63857 (14)	0.51980 (12)	0.86514 (8)	0.0193 (2)	
C8-1	0.21905 (16)	0.49369 (13)	0.87965 (8)	0.0230 (2)	
H8-1	0.1289	0.4480	0.8932	0.028*	
C5-1	0.46671 (14)	0.52249 (12)	0.86842 (8)	0.0183 (2)	
C20	0.11732 (15)	0.20286 (13)	0.69402 (8)	0.0215 (2)	
H20A	−0.0036	0.2258	0.7343	0.026*	
H20B	0.1973	0.1061	0.7357	0.026*	
C21A	0.10844 (17)	0.17794 (14)	0.59490 (9)	0.0218 (3)	0.934 (3)
H21A	0.0198	0.2537	0.5533	0.026*	0.934 (3)
C22A	0.21845 (17)	0.05548 (15)	0.56322 (9)	0.0238 (3)	0.934 (3)
H22A	0.3056	−0.0205	0.6058	0.029*	0.934 (3)
C23A	0.2142 (4)	0.0289 (3)	0.4634 (2)	0.0325 (6)	0.934 (3)
H23A	0.1207	0.1167	0.4264	0.049*	0.934 (3)
H23B	0.1884	−0.0676	0.4786	0.049*	0.934 (3)
H23C	0.3307	0.0203	0.4204	0.049*	0.934 (3)
C21B	0.209 (2)	0.1272 (18)	0.6064 (11)	0.015 (4)*	0.066 (3)
H21B	0.3352	0.0771	0.5981	0.018*	0.066 (3)
C22B	0.112 (2)	0.1321 (17)	0.5430 (11)	0.015 (4)*	0.066 (3)
H22B	−0.0106	0.1928	0.5380	0.018*	0.066 (3)
C23B	0.229 (5)	0.026 (4)	0.481 (3)	0.021 (8)*	0.066 (3)
H23D	0.1854	0.0602	0.4137	0.031*	0.066 (3)
H23E	0.2261	−0.0801	0.5169	0.031*	0.066 (3)
H23F	0.3515	0.0260	0.4695	0.031*	0.066 (3)
C4-2	0.35605 (14)	0.31944 (12)	0.67688 (8)	0.0183 (2)	
C2-2	0.63962 (15)	0.22142 (13)	0.70213 (9)	0.0240 (2)	
H2-2	0.7401	0.1342	0.7261	0.029*	
C6-2	0.52708 (15)	0.48383 (12)	0.63305 (8)	0.0194 (2)	
C5-2	0.36118 (14)	0.47249 (12)	0.63838 (8)	0.0188 (2)	
C8-2	0.09700 (15)	0.48857 (13)	0.63580 (9)	0.0244 (2)	
H8-2	−0.0256	0.5292	0.6260	0.029*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl-1	0.02417 (14)	0.02408 (14)	0.02272 (13)	−0.00338 (10)	−0.00490 (10)	−0.00524 (10)
Cl-2	0.03597 (16)	0.02178 (14)	0.02075 (13)	−0.01493 (11)	−0.00189 (10)	−0.00491 (10)
N1-1	0.0223 (5)	0.0252 (5)	0.0193 (4)	−0.0112 (4)	−0.0031 (3)	−0.0061 (4)
N3-1	0.0234 (5)	0.0209 (4)	0.0174 (4)	−0.0099 (4)	−0.0032 (3)	−0.0045 (3)
N9-1	0.0194 (4)	0.0244 (5)	0.0193 (4)	−0.0103 (4)	−0.0013 (3)	−0.0077 (3)
N7-1	0.0272 (5)	0.0224 (5)	0.0196 (4)	−0.0133 (4)	0.0011 (4)	−0.0066 (4)
N9-2	0.0189 (4)	0.0207 (4)	0.0199 (4)	−0.0057 (3)	−0.0028 (3)	−0.0063 (3)
N3-2	0.0211 (4)	0.0181 (4)	0.0223 (4)	−0.0046 (4)	−0.0039 (3)	−0.0048 (3)

N1-2	0.0213 (4)	0.0238 (5)	0.0229 (5)	−0.0082 (4)	−0.0017 (4)	−0.0074 (4)
N7-2	0.0250 (5)	0.0199 (5)	0.0249 (5)	−0.0029 (4)	−0.0077 (4)	−0.0059 (4)
C2-1	0.0259 (5)	0.0226 (5)	0.0202 (5)	−0.0128 (4)	−0.0037 (4)	−0.0045 (4)
C4-1	0.0206 (5)	0.0229 (5)	0.0134 (4)	−0.0098 (4)	−0.0012 (4)	−0.0063 (4)
C10	0.0191 (5)	0.0302 (6)	0.0210 (5)	−0.0089 (4)	−0.0039 (4)	−0.0075 (4)
C11A	0.0226 (7)	0.0210 (6)	0.0243 (6)	−0.0085 (5)	−0.0078 (5)	−0.0036 (5)
C12A	0.0236 (7)	0.0205 (6)	0.0226 (6)	−0.0086 (5)	−0.0066 (5)	−0.0037 (5)
C13A	0.0264 (11)	0.0339 (12)	0.0311 (11)	−0.0128 (9)	−0.0020 (7)	−0.0152 (7)
C6-1	0.0216 (5)	0.0220 (5)	0.0143 (4)	−0.0068 (4)	−0.0027 (4)	−0.0056 (4)
C8-1	0.0270 (6)	0.0263 (6)	0.0213 (5)	−0.0158 (5)	0.0021 (4)	−0.0091 (4)
C5-1	0.0229 (5)	0.0198 (5)	0.0138 (4)	−0.0094 (4)	−0.0004 (4)	−0.0056 (4)
C20	0.0218 (5)	0.0242 (5)	0.0203 (5)	−0.0106 (4)	−0.0005 (4)	−0.0066 (4)
C21A	0.0210 (6)	0.0247 (6)	0.0205 (6)	−0.0097 (5)	−0.0039 (4)	−0.0039 (4)
C22A	0.0253 (6)	0.0246 (6)	0.0212 (6)	−0.0098 (5)	−0.0033 (5)	−0.0043 (5)
C23A	0.0420 (11)	0.0391 (10)	0.0225 (10)	−0.0165 (7)	−0.0021 (8)	−0.0141 (8)
C4-2	0.0199 (5)	0.0204 (5)	0.0145 (4)	−0.0065 (4)	−0.0012 (4)	−0.0059 (4)
C2-2	0.0215 (5)	0.0204 (5)	0.0277 (6)	−0.0046 (4)	−0.0050 (4)	−0.0056 (4)
C6-2	0.0265 (5)	0.0194 (5)	0.0140 (4)	−0.0097 (4)	−0.0004 (4)	−0.0059 (4)
C5-2	0.0227 (5)	0.0177 (5)	0.0149 (4)	−0.0048 (4)	−0.0026 (4)	−0.0054 (4)
C8-2	0.0218 (5)	0.0233 (5)	0.0262 (5)	−0.0021 (4)	−0.0072 (4)	−0.0079 (4)

Geometric parameters (Å, º)

Cl-1—C6-1	1.7302 (11)	C13A—H13C	0.9800
Cl-2—C6-2	1.7325 (11)	C11B—C12B	1.311 (11)
N1-1—C6-1	1.3205 (14)	C11B—H11B	0.9500
N1-1—C2-1	1.3483 (15)	C12B—C13B	1.487 (15)
N3-1—C2-1	1.3334 (14)	C12B—H12B	0.9500
N3-1—C4-1	1.3338 (13)	C13B—H13D	0.9800
N9-1—C4-1	1.3644 (13)	C13B—H13E	0.9800
N9-1—C8-1	1.3711 (14)	C13B—H13F	0.9800
N9-1—C10	1.4687 (14)	C6-1—C5-1	1.3856 (15)
N7-1—C8-1	1.3130 (15)	C8-1—H8-1	0.9500
N7-1—C5-1	1.3847 (13)	C20—C21A	1.5069 (15)
N9-2—C4-2	1.3697 (14)	C20—C21B	1.526 (15)
N9-2—C8-2	1.3721 (14)	C20—H20A	0.9900
N9-2—C20	1.4756 (14)	C20—H20B	0.9900
N3-2—C4-2	1.3311 (14)	C21A—C22A	1.3227 (18)
N3-2—C2-2	1.3333 (15)	C21A—H21A	0.9500
N1-2—C6-2	1.3195 (15)	C22A—C23A	1.513 (3)
N1-2—C2-2	1.3486 (15)	C22A—H22A	0.9500
N7-2—C8-2	1.3123 (15)	C23A—H23A	0.9800
N7-2—C5-2	1.3848 (14)	C23A—H23B	0.9800
C2-1—H2-1	0.9500	C23A—H23C	0.9800
C4-1—C5-1	1.4035 (15)	C21B—C22B	1.308 (15)
C10—C11A	1.4975 (17)	C21B—H21B	0.9500
C10—C11B	1.547 (8)	C22B—C23B	1.475 (18)
C10—H10A	0.9900	C22B—H22B	0.9500
C10—H10B	0.9900	C23B—H23D	0.9800
C11A—C12A	1.323 (2)	C23B—H23E	0.9800

C11A—H11A	0.9500	C23B—H23F	0.9800
C12A—C13A	1.494 (3)	C4-2—C5-2	1.4075 (15)
C12A—H12A	0.9500	C2-2—H2-2	0.9500
C13A—H13A	0.9800	C6-2—C5-2	1.3857 (15)
C13A—H13B	0.9800	C8-2—H8-2	0.9500

C6-1—N1-1—C2-1	117.58 (9)	N1-1—C6-1—C5-1	121.81 (10)
C2-1—N3-1—C4-1	111.92 (9)	N1-1—C6-1—Cl-1	117.06 (8)
C4-1—N9-1—C8-1	105.55 (9)	C5-1—C6-1—Cl-1	121.12 (8)
C4-1—N9-1—C10	125.75 (9)	N7-1—C8-1—N9-1	114.96 (10)
C8-1—N9-1—C10	128.65 (9)	N7-1—C8-1—H8-1	122.5
C8-1—N7-1—C5-1	103.03 (9)	N9-1—C8-1—H8-1	122.5
C4-2—N9-2—C8-2	105.39 (9)	N7-1—C5-1—C6-1	135.09 (10)
C4-2—N9-2—C20	126.48 (9)	N7-1—C5-1—C4-1	110.75 (9)
C8-2—N9-2—C20	127.49 (9)	C6-1—C5-1—C4-1	114.16 (9)
C4-2—N3-2—C2-2	111.78 (9)	N9-2—C20—C21A	111.69 (9)
C6-2—N1-2—C2-2	116.99 (10)	N9-2—C20—C21B	106.9 (6)
C8-2—N7-2—C5-2	103.20 (9)	N9-2—C20—H20A	109.3
N3-1—C2-1—N1-1	127.67 (10)	C21A—C20—H20A	109.3
N3-1—C2-1—H2-1	116.2	C21B—C20—H20A	135.2
N1-1—C2-1—H2-1	116.2	N9-2—C20—H20B	109.3
N3-1—C4-1—N9-1	127.49 (10)	C21A—C20—H20B	109.3
N3-1—C4-1—C5-1	126.81 (10)	C21B—C20—H20B	83.6
N9-1—C4-1—C5-1	105.70 (9)	H20A—C20—H20B	107.9
N9-1—C10—C11A	110.39 (9)	C22A—C21A—C20	123.02 (11)
N9-1—C10—C11B	115.3 (3)	C22A—C21A—H21A	118.5
N9-1—C10—H10A	109.6	C20—C21A—H21A	118.5
C11A—C10—H10A	109.6	C21A—C22A—C23A	124.07 (14)
C11B—C10—H10A	80.2	C21A—C22A—H22A	118.0
N9-1—C10—H10B	109.6	C23A—C22A—H22A	118.0
C11A—C10—H10B	109.6	C22B—C21B—C20	119.2 (14)
C11B—C10—H10B	128.2	C22B—C21B—H21B	120.4
H10A—C10—H10B	108.1	C20—C21B—H21B	120.4
C12A—C11A—C10	123.78 (12)	C21B—C22B—C23B	107.1 (19)
C12A—C11A—H11A	118.1	C21B—C22B—H22B	126.5
C10—C11A—H11A	118.1	C23B—C22B—H22B	126.5
C11A—C12A—C13A	125.33 (17)	N3-2—C4-2—N9-2	127.88 (10)
C11A—C12A—H12A	117.3	N3-2—C4-2—C5-2	126.46 (10)
C13A—C12A—H12A	117.3	N9-2—C4-2—C5-2	105.66 (9)
C12B—C11B—C10	124.2 (8)	N3-2—C2-2—N1-2	128.37 (10)
C12B—C11B—H11B	117.9	N3-2—C2-2—H2-2	115.8
C10—C11B—H11B	117.9	N1-2—C2-2—H2-2	115.8
C11B—C12B—C13B	126.3 (11)	N1-2—C6-2—C5-2	121.91 (10)
C11B—C12B—H12B	116.9	N1-2—C6-2—Cl-2	116.70 (8)
C13B—C12B—H12B	116.9	C5-2—C6-2—Cl-2	121.39 (8)
C12B—C13B—H13D	109.5	N7-2—C5-2—C6-2	134.92 (10)
C12B—C13B—H13E	109.5	N7-2—C5-2—C4-2	110.64 (9)
H13D—C13B—H13E	109.5	C6-2—C5-2—C4-2	114.43 (9)
C12B—C13B—H13F	109.5	N7-2—C8-2—N9-2	115.10 (10)

H13D—C13B—H13F	109.5	N7-2—C8-2—H8-2	122.4
H13E—C13B—H13F	109.5	N9-2—C8-2—H8-2	122.4