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(*E*)-2-[(2-Butyl-4-chloro-1*H*-imidazol-5-yl)methylidene]-*N*-methylhydrazine-1-carbothioamide monohydrate

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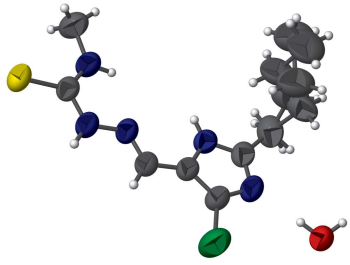
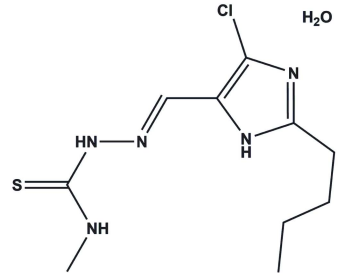
Structural data: full structural data are available from iucrdata.iucr.org

The title thiosemicarbazide derivative, $C_{10}H_{16}ClN_5S\cdot H_2O$, crystallized as a monohydrate. The molecule has an *E* conformation about the azomethine C=N bond that links the methylhydrazine-1-carbothioamide moiety to the imidazole ring. The butyl chain substituent on the imdazole ring is disordered over two sets of sites, with a refined occupancy ratio of 0.509 (9):0.491 (9). In the crystal, molecules are linked by O—H···N and N—H···O hydrogen bonds involving the solvent water molecule, forming chains along the *c*-axis direction. The chains are linked by O—H···S and N—H···S hydrogen bonds, forming a three-dimensional framework.

3D view



Chemical scheme



Structure description

Thiosemicarbazones belongs to a large group of thiourea derivatives which are derived from aldehyde and ketones. The biological activities of these compounds depends on the parent aldehyde or ketone (Beraldo & Gambinob, 2004). The co-ordination chemistry of thiosemicarbazones has been described (Sreekanth *et al.*, 2004; Beraldo *et al.*, 2001; Mazlan *et al.*, 2014).

The title thiosemicarbazide derivative, Fig. 1, crystallized as a monohydrate. The molecule has an *E* conformation about azomethine C3=N3 bond that links the methylhydrazine-1-carbothioamide moiety to the imidazole ring.

In the crystal, molecules are linked by O—H···N and N—H···O hydrogen bonds involving the solvent water molecule, forming chains along the *c*-axis direction (Table 1

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1E···N2	0.85	1.94	2.790 (3)	174
N1—H1···O1^{i}	0.86	1.97	2.832 (3)	177
N5—H5···O1^{i}	0.86	2.30	3.114 (3)	159
O1—H1D···S1^{ii}	0.85	2.51	3.345 (2)	169
N4—H4···S1^{iii}	0.86	2.52	3.374 (2)	169

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

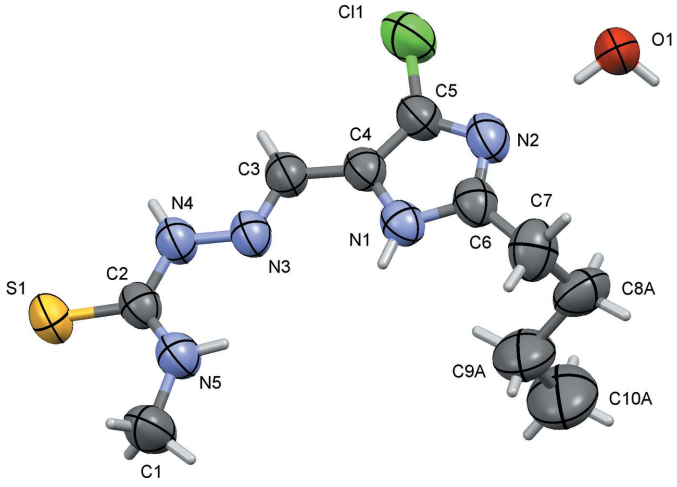


Figure 1
A view of the molecular structure of the title compound, showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level. For clarity, the minor component (atoms C8–C10) of the disordered butyl side chain has been omitted.

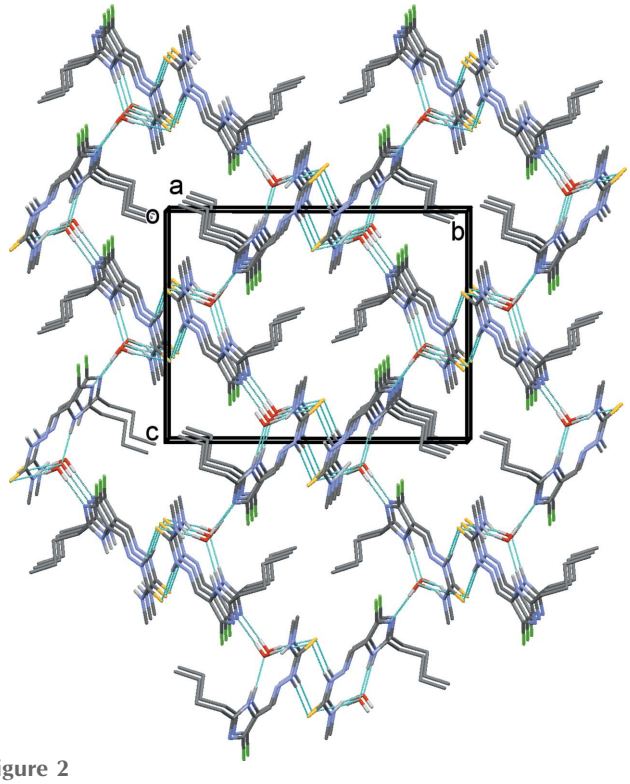


Figure 2
A view along the *a* axis of the crystal packing of the title compound. Hydrogen bonds are shown as dashed lines (see Table 1) and H atoms not involved in the hydrogen bonding have been omitted for clarity.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{10}H_{16}ClN_5S·H_2O$
M_r	291.80
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	300
a, b, c (Å)	6.494 (4), 17.665 (10), 13.508 (8)
β (°)	92.402 (9)
V (Å^3)	1548.2 (16)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.38
Crystal size (mm)	0.3 × 0.2 × 0.14
Data collection	
Diffractometer	Bruker *SMART* CCD area detector
Absorption correction	Multi-scan (*SADABS*; Bruker, 2004)
T_{min}, T_{max}	0.895, 0.949
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	15205, 3151, 2347
R_{int}	0.039
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.627
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.049, 0.150, 1.03
No. of reflections	3151
No. of parameters	192
No. of restraints	90
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.23, −0.31

Computer programs: *SMART* and *SAINT* (Bruker, 2004), *SHELXS* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *OLEX2* (Dolomanov *et al.*, 2009) and *Mercury* (Macrae *et al.*, 2008).

and Fig. 2). The chains are linked by O—H···S and N—H···S hydrogen bonds, forming a three-dimensional framework (Table 1 and Fig. 2).

Synthesis and crystallization

An ethanol solution (10 ml) of 2-butyl-4-chloro-5-formylimidazole (0.02 moles, 3.74 g) was added slowly to a hot ethanol solution (10 ml) of 4-methyl-3-thiosemicarbazide (0.02 moles, 2.12 g) under constant stirring. The mixture was refluxed for *ca* 3 h and the precipitate formed was collected by filtration, washed with dry ethanol and dried *in vacuo*. Yellow block-like crystals were obtained by slow evaporation of a solution in ethanol after 15 days.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. Elongated displacement ellipsoids on atoms C8, C9, and C10 suggested disorder. It was modelled successfully between two positions with a refined occupancy ratio of 0.509 (9):0.491 (9) for atoms C8*A*:C8, C9*A*:C9 and C10*A*:C10. Several restraints and/or constraints were necessary to keep bond distances, angles, and displacement ellipsoids meaningful.

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full crystallographic data

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(*E*)-2-[(2-Butyl-4-chloro-1*H*-imidazol-5-yl)methylidene]-*N*-methylhydrazine-1-carbothioamide monohydrate

Crystal data

$C_{10}H_{16}ClN_5S·H_2O$
M_r = 291.80
Monoclinic, $P2_1/c$
a = 6.494 (4) Å
b = 17.665 (10) Å
c = 13.508 (8) Å
β = 92.402 (9)°
V = 1548.2 (16) Å^3
Z = 4
$F(000)$ = 616
D_x = 1.252 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5524 reflections
θ = 2.3–24.9°
μ = 0.38 mm^{-1}
T = 300 K
Block, yellow
0.3 × 0.2 × 0.14 mm

Data collection

Bruker SMART CCD area detector diffractometer
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2004)
T_{min} = 0.895, T_{max} = 0.949
15205 measured reflections
3151 independent reflections
2347 reflections with $I > 2\sigma(I)$
R_{int} = 0.039
θ_{max} = 26.5°, θ_{min} = 1.9°
h = −8→8
k = −22→21
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.150
S = 1.03
3151 reflections
192 parameters
90 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0896P)^2 + 0.1249P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-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.

Refinement. 1. Fixed Uiso At 1.2 times of: All C(H) groups, All C(H,H) groups, All C(H,H,H,H) groups, All N(H) groups At 1.5 times of: All C(H,H,H) groups, All O(H,H) groups 2. Restrained distances C9A-C10A = C9-C10 = C9-C8 = C9A-C8A = C8-C7 = C8A-C7 1.54 with sigma of 0.02 3. Uiso/Uaniso restraints and constraints C8 ~C8A ~C9 ~C9A ~C10 ~C10A: within 1.7A with sigma of 0.02 and sigma for terminal atoms of 0.02 4. Rigid body (RIGU) restrains C8, C8A, C9, C9A, C10, C10A with sigma for 1-2 distances of 0.004 and sigma for 1-3 distances of 0.004 5. Others Sof(H7BC)=Sof(H7BD)=Sof(C8A)=Sof(H8AA)=Sof(H8AB)=Sof(C9A)=Sof(H9AA)=Sof(H9AB)= Sof(C10A)=Sof(H10D)=Sof(H10E)=Sof(H10F)=1-FVAR(1) Sof(H7AA)=Sof(H7AB)=Sof(C8)=Sof(H8A)=Sof(H8B)=Sof(C9)=Sof(H9A)=Sof(H9B)= Sof(C10)=Sof(H10A)=Sof(H10B)=Sof(H10C)=FVAR(1) 6.a Riding coordinates: O1(H1D,H1E) 6.b Secondary CH2 refined with riding coordinates: C7(H7AA,H7AB), C7(H7BC,H7BD), C8(H8A,H8B), C8A(H8AA,H8AB), C9(H9A,H9B), C9A(H9AA,H9AB) 6.c Me refined with riding coordinates: C10(H10A,H10B,H10C), C10A(H10D,H10E,H10F) 6.d Aromatic/amide H refined with riding coordinates: N1(H1), N4(H4), N5(H5), C3(H3) 6.e Idealised Me refined as rotating group: C1(H1A,H1B,H1C)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Cl1	1.09917 (13)	0.28647 (5)	1.33664 (5)	0.1203 (3)	
S1	1.40870 (8)	0.50705 (3)	0.84042 (4)	0.0775 (2)	
N1	0.7716 (2)	0.30301 (9)	1.09668 (13)	0.0680 (4)	
H1	0.7390	0.3177	1.0374	0.082*	
N2	0.7516 (3)	0.24365 (10)	1.23866 (15)	0.0803 (5)	
N3	1.0786 (2)	0.39480 (9)	1.02455 (12)	0.0676 (4)	
N4	1.2297 (2)	0.43919 (9)	0.98710 (13)	0.0713 (4)	
H4	1.3325	0.4541	1.0243	0.086*	
N5	1.0461 (3)	0.43806 (11)	0.84178 (13)	0.0777 (5)	
H5	0.9546	0.4141	0.8737	0.093*	
C1	1.0031 (4)	0.45223 (17)	0.73736 (19)	0.1025 (8)	
H1A	0.9799	0.5054	0.7270	0.154*	
H1B	0.8824	0.4245	0.7155	0.154*	
H1C	1.1184	0.4362	0.7004	0.154*	
C2	1.2143 (3)	0.45908 (10)	0.89093 (15)	0.0650 (5)	
C3	1.0989 (3)	0.37222 (11)	1.11367 (16)	0.0689 (5)	
H3	1.2117	0.3870	1.1538	0.083*	
C4	0.9444 (3)	0.32364 (11)	1.15086 (14)	0.0668 (5)	
C5	0.9271 (3)	0.28602 (12)	1.23801 (17)	0.0776 (6)	
C6	0.6607 (3)	0.25575 (11)	1.15151 (18)	0.0726 (5)	
C7	0.4665 (4)	0.21935 (15)	1.1134 (2)	0.0981 (8)	
H7AA	0.3930	0.2024	1.1704	0.118*	0.491 (9)
H7AB	0.3830	0.2585	1.0812	0.118*	0.491 (9)
H7BC	0.4113	0.2473	1.0565	0.118*	0.509 (9)
H7BD	0.3654	0.2206	1.1642	0.118*	0.509 (9)
C8	0.4739 (19)	0.1576 (8)	1.0468 (9)	0.138 (4)	0.491 (9)
H8A	0.4192	0.1769	0.9838	0.166*	0.491 (9)
H8B	0.3732	0.1216	1.0692	0.166*	0.491 (9)

C8A	0.5058 (15)	0.1367 (5)	1.0833 (7)	0.101 (2)	0.509 (9)
H8AA	0.3772	0.1097	1.0707	0.122*	0.509 (9)
H8AB	0.5871	0.1102	1.1343	0.122*	0.509 (9)
C9	0.6576 (17)	0.1117 (7)	1.0227 (7)	0.141 (3)	0.491 (9)
H9A	0.6684	0.0647	1.0598	0.169*	0.491 (9)
H9B	0.7858	0.1398	1.0288	0.169*	0.491 (9)
C9A	0.6316 (17)	0.1452 (5)	0.9831 (7)	0.130 (3)	0.509 (9)
H9AA	0.5431	0.1671	0.9310	0.157*	0.509 (9)
H9AB	0.7488	0.1785	0.9949	0.157*	0.509 (9)
C10	0.5775 (17)	0.1002 (7)	0.9120 (6)	0.157 (3)	0.491 (9)
H10A	0.6748	0.0702	0.8776	0.235*	0.491 (9)
H10B	0.4467	0.0747	0.9108	0.235*	0.491 (9)
H10C	0.5621	0.1486	0.8803	0.235*	0.491 (9)
C10A	0.708 (2)	0.0645 (5)	0.9501 (9)	0.173 (4)	0.509 (9)
H10D	0.7830	0.0693	0.8906	0.259*	0.509 (9)
H10E	0.7966	0.0433	1.0016	0.259*	0.509 (9)
H10F	0.5915	0.0319	0.9379	0.259*	0.509 (9)
O1	0.6591 (2)	0.15312 (9)	1.40012 (11)	0.0909 (5)	
H1D	0.6078	0.1117	1.3785	0.136*	
H1E	0.6784	0.1808	1.3499	0.136*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.1252 (6)	0.1591 (8)	0.0757 (5)	−0.0300 (5)	−0.0068 (4)	0.0263 (4)
S1	0.0773 (4)	0.0824 (4)	0.0740 (4)	−0.0057 (2)	0.0155 (3)	0.0169 (3)
N1	0.0741 (9)	0.0658 (9)	0.0648 (10)	0.0046 (7)	0.0131 (8)	0.0015 (8)
N2	0.0886 (11)	0.0791 (11)	0.0750 (13)	−0.0082 (9)	0.0248 (10)	0.0076 (9)
N3	0.0701 (9)	0.0660 (9)	0.0678 (11)	−0.0028 (7)	0.0160 (7)	0.0061 (8)
N4	0.0708 (9)	0.0766 (10)	0.0671 (11)	−0.0081 (8)	0.0091 (8)	0.0110 (8)
N5	0.0788 (11)	0.0886 (12)	0.0664 (11)	−0.0083 (9)	0.0103 (9)	0.0075 (9)
C1	0.1038 (17)	0.132 (2)	0.0707 (16)	−0.0195 (16)	−0.0034 (13)	0.0140 (15)
C2	0.0692 (11)	0.0610 (11)	0.0656 (12)	0.0053 (8)	0.0138 (9)	0.0047 (9)
C3	0.0759 (11)	0.0653 (11)	0.0661 (13)	−0.0026 (9)	0.0101 (9)	0.0034 (9)
C4	0.0760 (11)	0.0633 (11)	0.0619 (12)	−0.0017 (9)	0.0146 (9)	0.0001 (9)
C5	0.0891 (14)	0.0808 (13)	0.0636 (13)	−0.0051 (11)	0.0133 (10)	0.0068 (10)
C6	0.0743 (11)	0.0682 (12)	0.0771 (15)	0.0021 (9)	0.0231 (10)	−0.0008 (10)
C7	0.0727 (13)	0.0956 (17)	0.127 (2)	−0.0029 (12)	0.0189 (13)	0.0024 (16)
C8	0.123 (6)	0.180 (8)	0.110 (7)	−0.019 (5)	−0.016 (5)	−0.040 (6)
C8A	0.086 (4)	0.115 (5)	0.101 (5)	−0.017 (3)	−0.013 (4)	−0.027 (4)
C9	0.173 (5)	0.138 (7)	0.111 (6)	0.018 (5)	−0.001 (5)	−0.024 (5)
C9A	0.165 (6)	0.119 (5)	0.109 (6)	−0.019 (4)	0.027 (5)	−0.041 (4)
C10	0.182 (7)	0.172 (7)	0.115 (5)	0.007 (6)	0.003 (5)	−0.018 (5)
C10A	0.227 (8)	0.136 (6)	0.159 (7)	−0.014 (5)	0.057 (6)	−0.057 (5)
O1	0.1094 (11)	0.0934 (10)	0.0699 (10)	−0.0319 (9)	0.0023 (8)	0.0073 (8)

Geometric parameters (Å, º)

Cl1—C5	1.703 (3)	C7—H7BD	0.9700
S1—C2	1.688 (2)	C7—C8	1.415 (11)
N1—H1	0.8600	C7—C8A	1.540 (9)
N1—C4	1.363 (3)	C8—H8A	0.9700
N1—C6	1.345 (3)	C8—H8B	0.9700
N2—C5	1.364 (3)	C8—C9	1.490 (13)
N2—C6	1.312 (3)	C8A—H8AA	0.9700
N3—N4	1.370 (2)	C8A—H8AB	0.9700
N3—C3	1.270 (3)	C8A—C9A	1.617 (12)
N4—H4	0.8600	C9—H9A	0.9700
N4—C2	1.345 (3)	C9—H9B	0.9700
N5—H5	0.8600	C9—C10	1.575 (12)
N5—C1	1.448 (3)	C9A—H9AA	0.9700
N5—C2	1.308 (3)	C9A—H9AB	0.9700
C1—H1A	0.9600	C9A—C10A	1.580 (12)
C1—H1B	0.9600	C10—H10A	0.9600
C1—H1C	0.9600	C10—H10B	0.9600
C3—H3	0.9300	C10—H10C	0.9600
C3—C4	1.428 (3)	C10A—H10D	0.9600
C4—C5	1.361 (3)	C10A—H10E	0.9600
C6—C7	1.488 (3)	C10A—H10F	0.9600
C7—H7AA	0.9700	O1—H1D	0.8500
C7—H7AB	0.9700	O1—H1E	0.8500
C7—H7BC	0.9700		
C4—N1—H1	125.7	C8—C7—H7AB	107.3
C6—N1—H1	125.7	C8A—C7—H7BC	109.5
C6—N1—C4	108.54 (19)	C8A—C7—H7BD	109.5
C6—N2—C5	104.44 (17)	C7—C8—H8A	105.5
C3—N3—N4	118.86 (17)	C7—C8—H8B	105.5
N3—N4—H4	120.6	C7—C8—C9	127.4 (10)
C2—N4—N3	118.74 (17)	H8A—C8—H8B	106.0
C2—N4—H4	120.6	C9—C8—H8A	105.5
C1—N5—H5	117.7	C9—C8—H8B	105.5
C2—N5—H5	117.7	C7—C8A—H8AA	111.1
C2—N5—C1	124.62 (18)	C7—C8A—H8AB	111.1
N5—C1—H1A	109.5	C7—C8A—C9A	103.1 (7)
N5—C1—H1B	109.5	H8AA—C8A—H8AB	109.1
N5—C1—H1C	109.5	C9A—C8A—H8AA	111.1
H1A—C1—H1B	109.5	C9A—C8A—H8AB	111.1
H1A—C1—H1C	109.5	C8—C9—H9A	113.2
H1B—C1—H1C	109.5	C8—C9—H9B	113.2
N4—C2—S1	119.65 (16)	C8—C9—C10	92.4 (10)
N5—C2—S1	124.06 (17)	H9A—C9—H9B	110.6
N5—C2—N4	116.27 (17)	C10—C9—H9A	113.2
N3—C3—H3	120.8	C10—C9—H9B	113.2

N3—C3—C4	118.4 (2)	C8A—C9A—H9AA	109.8
C4—C3—H3	120.8	C8A—C9A—H9AB	109.8
N1—C4—C3	123.13 (19)	H9AA—C9A—H9AB	108.3
C5—C4—N1	103.76 (18)	C10A—C9A—C8A	109.2 (9)
C5—C4—C3	133.1 (2)	C10A—C9A—H9AA	109.8
N2—C5—Cl1	121.25 (17)	C10A—C9A—H9AB	109.8
C4—C5—Cl1	126.82 (18)	C9—C10—H10A	109.5
C4—C5—N2	111.9 (2)	C9—C10—H10B	109.5
N1—C6—C7	122.9 (2)	C9—C10—H10C	109.5
N2—C6—N1	111.4 (2)	H10A—C10—H10B	109.5
N2—C6—C7	125.6 (2)	H10A—C10—H10C	109.5
C6—C7—H7AA	107.3	H10B—C10—H10C	109.5
C6—C7—H7AB	107.3	C9A—C10A—H10D	109.5
C6—C7—H7BC	109.5	C9A—C10A—H10E	109.5
C6—C7—H7BD	109.5	C9A—C10A—H10F	109.5
C6—C7—C8A	110.7 (4)	H10D—C10A—H10E	109.5
H7AA—C7—H7AB	106.9	H10D—C10A—H10F	109.5
H7BC—C7—H7BD	108.1	H10E—C10A—H10F	109.5
C8—C7—C6	120.1 (5)	H1D—O1—H1E	106.8
C8—C7—H7AA	107.3		

N1—C4—C5—Cl1	178.29 (17)	C3—C4—C5—N2	−177.9 (2)
N1—C4—C5—N2	0.0 (2)	C4—N1—C6—N2	−0.4 (2)
N1—C6—C7—C8	75.8 (8)	C4—N1—C6—C7	−177.11 (19)
N1—C6—C7—C8A	101.2 (4)	C5—N2—C6—N1	0.4 (2)
N2—C6—C7—C8	−100.3 (8)	C5—N2—C6—C7	177.0 (2)
N2—C6—C7—C8A	−75.0 (5)	C6—N1—C4—C3	178.42 (17)
N3—N4—C2—S1	−174.50 (13)	C6—N1—C4—C5	0.2 (2)
N3—N4—C2—N5	4.4 (3)	C6—N2—C5—Cl1	−178.66 (17)
N3—C3—C4—N1	−3.0 (3)	C6—N2—C5—C4	−0.3 (2)
N3—C3—C4—C5	174.6 (2)	C6—C7—C8—C9	13 (2)
N4—N3—C3—C4	−177.68 (16)	C6—C7—C8A—C9A	−72.0 (8)
C1—N5—C2—S1	0.5 (3)	C7—C8—C9—C10	−146.0 (13)
C1—N5—C2—N4	−178.3 (2)	C7—C8A—C9A—C10A	172.9 (8)
C3—N3—N4—C2	175.86 (17)	C8—C7—C8A—C9A	45.5 (17)
C3—C4—C5—Cl1	0.4 (4)	C8A—C7—C8—C9	−60.1 (16)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*E*···N2	0.85	1.94	2.790 (3)	174
N1—H1···O1^{i}	0.86	1.97	2.832 (3)	177
N5—H5···O1^{i}	0.86	2.30	3.114 (3)	159
O1—H1*D*···S1^{ii}	0.85	2.51	3.345 (2)	169
N4—H4···S1^{iii}	0.86	2.52	3.374 (2)	169

Symmetry codes: (i) *x*, −*y*+1/2, *z*−1/2; (ii) *x*−1, −*y*+1/2, *z*+1/2; (iii) −*x*+3, −*y*+1, −*z*+2.