

Crystal structure of 2-amino-5-methylsulfanyl-1,3,4-thiadiazol-3-ium chloride monohydrate

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The title salt, $C_3H_6N_3S_2^+ \cdot Cl^- \cdot H_2O$, crystallized with two organic cations, two chloride anions and two water molecules in the asymmetric unit. The methyl C atoms deviate from their respective bound ring planes by 0.081 and 0.002 Å. In the crystal, the components are connected *via* N–H...O, N–H...Cl and O–H...Cl hydrogen bonds, forming sheets lying parallel to (100). The sheets are linked into bilayers by O–H...Cl hydrogen bonds involving the chloride ions and water molecules. Within the bilayers there are π – π interactions [inter-centroid distances = 3.4654 (4) and 3.4789 (4) Å] involving inversion-related cations.

Keywords: crystal structure; 1,3,4-thiadiazole; biological activity; organic-inorganic hybrid.

CCDC reference: 1012703

1. Related literature

For the medicinal importance and biological activity of thiadiazol isomers, see: Demirbas *et al.* (2009). For applications of 1,3,4 thiadiazoles in agriculture, see: Wei *et al.* (2006). For C–N bond lengths in the 2-amino-5-methylsulfanyl-1,3,4-thiadiazol-3-ium cation, see: Mrad *et al.* (2012).

2. Experimental

2.1. Crystal data

$C_3H_6N_3S_2^+ \cdot Cl^- \cdot H_2O$
 $M_r = 201.69$
 Monoclinic, $P2_1/n$
 $a = 13.3826$ (2) Å
 $b = 9.4258$ (1) Å
 $c = 13.5762$ (2) Å
 $\beta = 99.453$ (1)°

$V = 1689.27$ (4) Å³
 $Z = 8$
 Mo $K\alpha$ radiation
 $\mu = 0.89$ mm⁻¹
 $T = 110$ K
 $0.42 \times 0.31 \times 0.15$ mm

2.2. Data collection

Agilent SuperNova (Dual, Cu at zero, Atlas) diffractometer
 Absorption correction: analytical [CrysAlis PRO (Agilent, 2012), using a multi-faceted crystal model based on expressions

derived by Clark & Reid (1995)
 $T_{min} = 0.769$, $T_{max} = 0.894$
 65950 measured reflections
 8394 independent reflections
 7745 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.021$

2.3. Refinement

$R[F^2 > 2\sigma(F^2)] = 0.020$
 $wR(F^2) = 0.054$
 $S = 1.12$
 8394 reflections
 224 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{max} = 0.47$ e Å⁻³
 $\Delta\rho_{min} = -0.28$ e Å⁻³

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

D–H...A	D–H	H...A	D...A	D–H...A
N3–H3...O17	0.861 (13)	1.818 (13)	2.6778 (7)	176.4 (12)
N6–H6A...Cl1	0.846 (12)	2.296 (12)	3.1178 (6)	164.2 (11)
N6–H6B...Cl2 ⁱ	0.829 (13)	2.392 (13)	3.2139 (7)	171.4 (13)
N11–H11...O18	0.914 (14)	1.751 (14)	2.6632 (7)	176.7 (13)
N14–H14A...Cl2	0.877 (12)	2.289 (12)	3.1287 (6)	160.3 (11)
N14–H14B...Cl1 ⁱⁱ	0.807 (13)	2.461 (13)	3.2648 (6)	173.6 (13)
O17–H17A...Cl2 ⁱ	0.796 (14)	2.373 (14)	3.1593 (5)	169.8 (14)
O17–H17B...Cl2 ⁱⁱⁱ	0.826 (15)	2.340 (15)	3.1649 (6)	175.5 (14)
O18–H18A...Cl1 ⁱⁱ	0.780 (13)	2.414 (13)	3.1708 (5)	163.8 (13)
O18–H18B...Cl1 ^{iv}	0.837 (15)	2.318 (15)	3.1517 (6)	174.1 (14)

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

Data collection: CrysAlis PRO (Agilent, 2012); cell refinement: CrysAlis PRO; data reduction: CrysAlis PRO; program(s) used to solve structure: SIR92 (Altomare *et al.*, 1994); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 for Windows (Farrugia, 2012); software used to prepare material for publication: SHELXL97 and PLATON (Spek, 2009).

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: SU2749).

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supporting information

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Crystal structure of 2-amino-5-methylsulfanyl-1,3,4-thiadiazol-3-ium chloride monohydrate

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S1. Chemical context

The amine under investigation, 2-amino-5-(methylthio)-1,3,4-thiadiazole, belongs to the larger family of heterocyclic compounds with potential medicinal importance and broad spectra of biological activities. Among these types of organic molecules are several isomers of thiadiazole (Demirbas *et al.*, 2009). 1,3,4-thiadiazoles represent one of the most biologically active classes of compounds with a wide spectrum of activities. A large number of 1,3,4-thiadiazoles have been patented in the agricultural field as herbicides and bactericides (Wei *et al.*, 2006). In the present investigation, we report the synthesis and crystal structure of a new organic-inorganic hybrid compound prepared from the reaction of the title amine with the hydrochloric acid in aqueous medium.

S2. Structural commentary

As shown in Fig. 1, the asymmetric unit of the title compound contains two 2-amino-5-(methylthio)-1,3,4-thiadiazol-3-ium cations, two chloride anions and two water molecules. These entities are connected by N—H \cdots Cl, N—H \cdots O and O—H \cdots Cl hydrogen bonds, forming sheets parallel to (100) (Table 1 and Fig. 2). The layers are connect through O—H \cdots Cl contacts, forming bilayers (Figs. 2 and 3).

Examination of the geometrical features of the organic cations shows that the exocyclic C—N bond lengths are similar in length to those of the thiadiazole rings, probably due to delocalization of the ring π density with the *p*-orbital electrons of the amino group. These C—N distances, *ca.* 1.33 Å, are compatible to that of [C₃H₆N₃S₂]H₂PO₄ which contains the same organic entity (Mrad *et al.*, 2012).

S3. Supramolecular features

The crystal packing is also influenced by intermolecular π – π stacking interactions between inversion-related anti-parallel organic cations within the bilayers (Fig. 3); Cg1 \cdots Cg1ⁱ = 3.4654 (4) Å [Cg1 is the centroid of the S1/N3/N4/C2/C5 ring; symmetry code: (i) -x, -y+2, -z+1] and Cg2 \cdots Cg2ⁱⁱ = 3.4789 (4) Å [Cg2 is the centroid of the S9/N11/N12/C10/C13 ring; symmetry code: -x+1, -y+1, -z+1].

S4. Synthesis and crystallization

An aqueous 1 M HCl solution and 2-amino-5-(methylthio)-1,3,4-thiadiazole in a 1:1 molar ratio were mixed and dissolved in sufficient ethanol. Crystals of the title salt grew as the ethanol evaporated at room temperature over the course of a few days.

S5. Refinement

The N- and O-bound H atoms were located in difference Fourier maps and freely refined. The C-bound H atoms were included in calculated positions and treated as riding atoms: C—H = 0.98 Å with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. Experimental

details are given in Table 2.

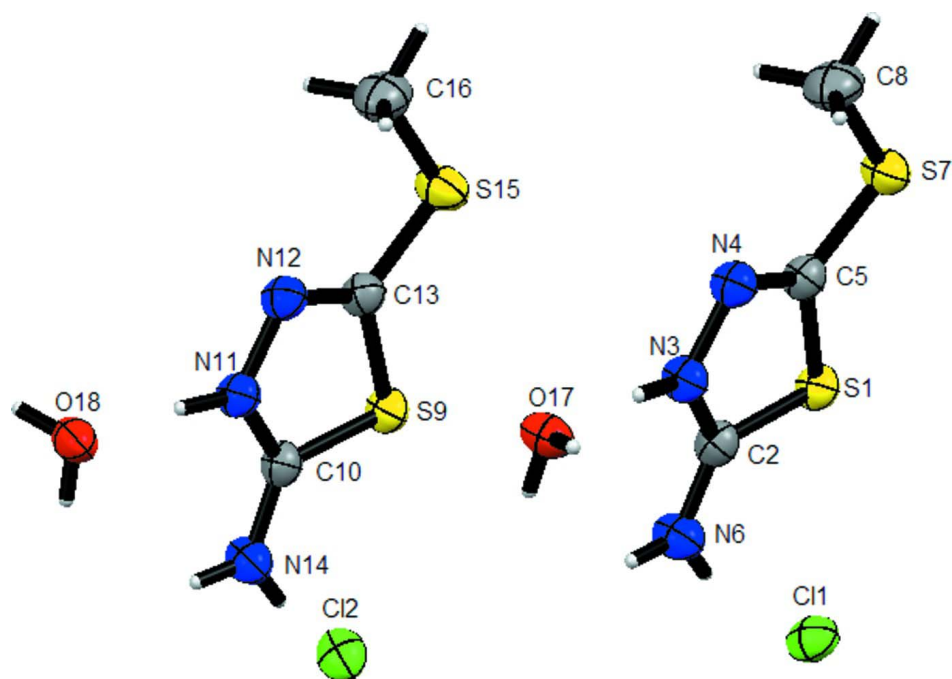


Figure 1

A view of molecular structure of the title salt, with atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

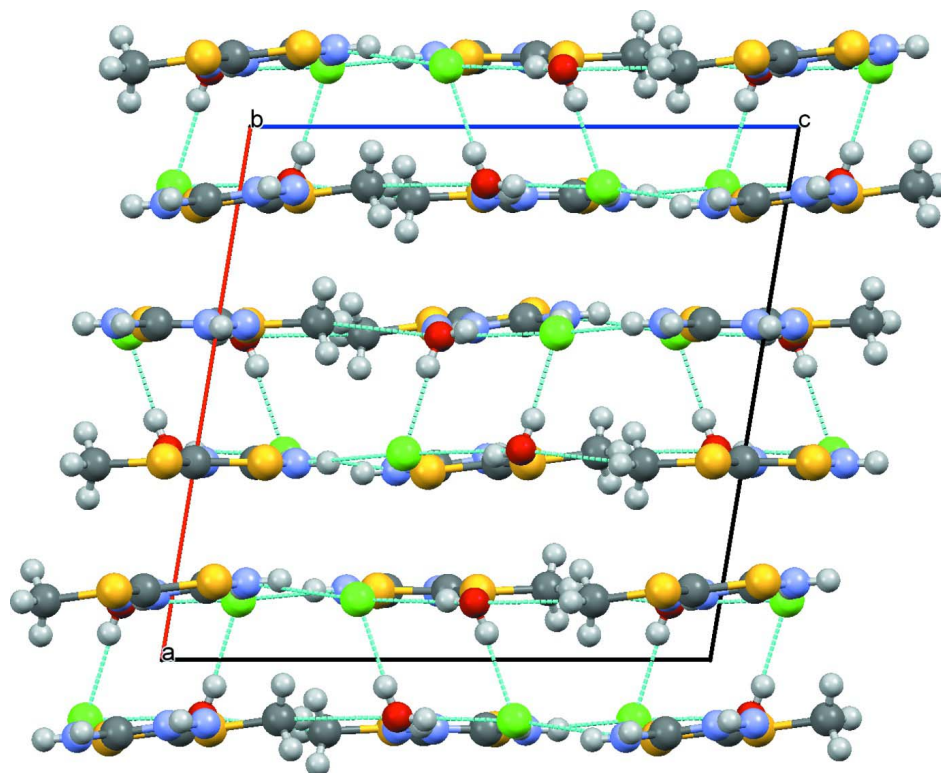


Figure 2

A view along the *b* axis of the crystal packing of the title compound. The hydrogen bonds are shown as dashed lines (see Table 1 for details).

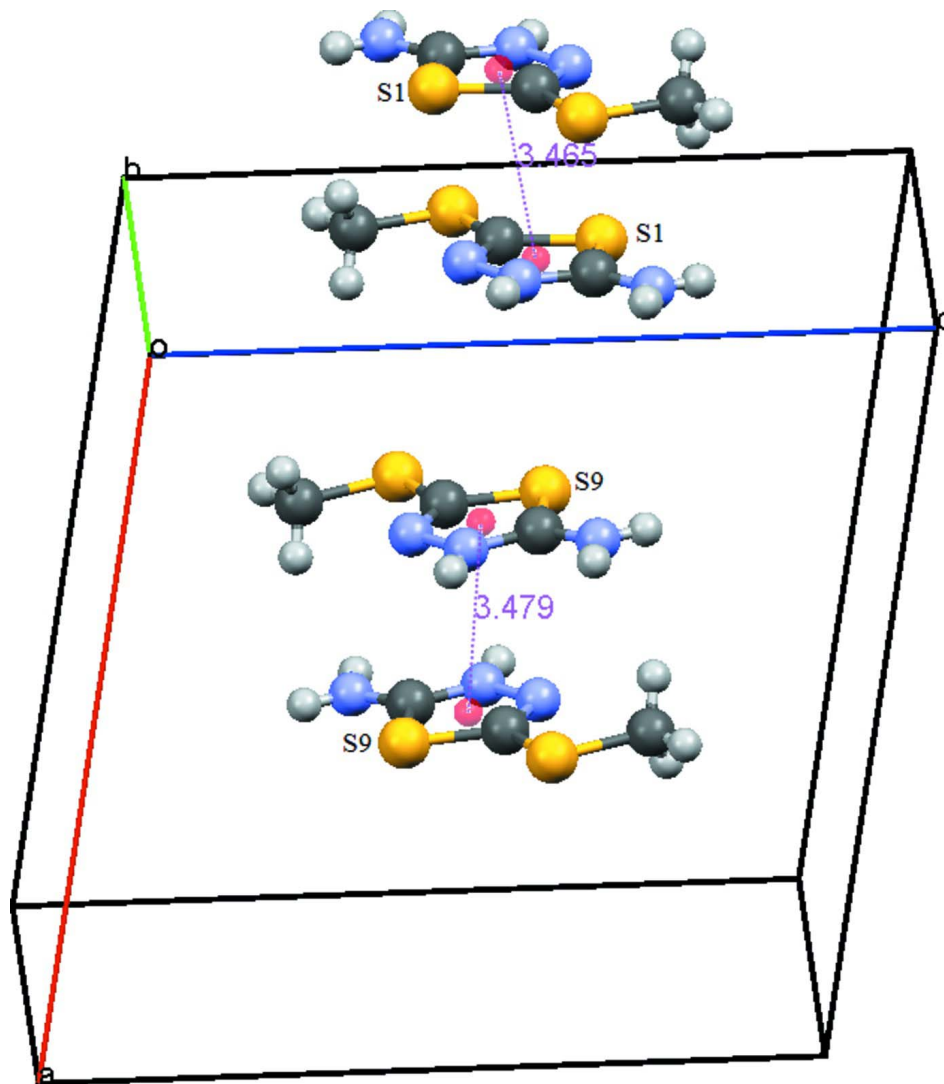


Figure 3

A partial view along the *b* axis of the crystal packing of the title compound, showing the intermolecular π - π stacking interactions between inversion-related organic cations.

2-Amino-5-methylsulfanyl-1,3,4-thiadiazol-3-ium chloride monohydrate

Crystal data

$C_3H_6N_3S_2^+ \cdot Cl^- \cdot H_2O$

$M_r = 201.69$

Monoclinic, $P2_1/n$

Hall symbol: $-P\ 2_1\ yac$

$a = 13.3826\ (2)\ \text{\AA}$

$b = 9.4258\ (1)\ \text{\AA}$

$c = 13.5762\ (2)\ \text{\AA}$

$\beta = 99.453\ (1)^\circ$

$V = 1689.27\ (4)\ \text{\AA}^3$

$Z = 8$

$F(000) = 832$

$D_x = 1.586\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.7107\ \text{\AA}$

Cell parameters from 30742 reflections

$\theta = 2.9\text{--}37.8^\circ$

$\mu = 0.89\ \text{mm}^{-1}$

$T = 110\ \text{K}$

Prism, colorless

$0.42 \times 0.31 \times 0.15\ \text{mm}$

Data collection

Agilent SuperNova (Dual, Cu at zero, Atlas) diffractometer
 Radiation source: SuperNova (Mo) X-ray Source
 Mirror monochromator
 Detector resolution: 10.4508 pixels mm⁻¹
 ω scans

Absorption correction: analytical [CrysAlis PRO (Agilent, 2012), using a multi-faceted crystal model based on expressions derived by Clark & Reid (1995)]
 $T_{\min} = 0.769$, $T_{\max} = 0.894$
 65950 measured reflections
 8394 independent reflections
 7745 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.021$
 $\theta_{\max} = 36.7^\circ$, $\theta_{\min} = 2.9^\circ$
 $h = -22 \rightarrow 22$
 $k = -15 \rightarrow 15$
 $l = -22 \rightarrow 22$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.020$
 $wR(F^2) = 0.054$
 $S = 1.12$
 8394 reflections
 224 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 atoms treated by a mixture of independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0211P)^2 + 0.370P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.003$
 $\Delta\rho_{\max} = 0.47 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.28 \text{ e } \text{\AA}^{-3}$
 Extinction correction: SHELXL97 (Sheldrick, 2008), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.00111 (13)

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 $F^2 > 2\sigma(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
S7	0.127439 (13)	1.268596 (18)	0.444671 (12)	0.01782 (3)
Cl2	0.386288 (13)	0.472130 (17)	0.838806 (12)	0.01897 (3)
S1	0.129775 (12)	1.086700 (17)	0.628112 (12)	0.01498 (3)
S9	0.353350 (12)	0.596116 (17)	0.572019 (12)	0.01549 (3)
S15	0.370685 (15)	0.777122 (18)	0.392656 (13)	0.02012 (3)
Cl1	0.112933 (13)	0.978631 (17)	0.878768 (12)	0.01911 (3)
O18	0.39361 (4)	0.11409 (5)	0.40860 (4)	0.01921 (9)
O17	0.10727 (4)	0.60603 (5)	0.44691 (4)	0.01852 (9)
N12	0.38085 (4)	0.49199 (6)	0.40204 (4)	0.01616 (9)
N3	0.12906 (4)	0.87190 (6)	0.51663 (4)	0.01608 (9)
N6	0.13116 (5)	0.81093 (7)	0.68457 (4)	0.01909 (10)

N11	0.37722 (4)	0.38126 (6)	0.46775 (4)	0.01566 (9)
N14	0.35554 (5)	0.32153 (6)	0.63065 (4)	0.01771 (9)
N4	0.12853 (4)	0.98357 (6)	0.45103 (4)	0.01622 (9)
C10	0.36248 (5)	0.41383 (6)	0.55932 (5)	0.01406 (9)
C5	0.12929 (5)	1.10218 (7)	0.49890 (5)	0.01482 (9)
C13	0.36960 (5)	0.61102 (7)	0.44630 (5)	0.01494 (10)
C2	0.13012 (5)	0.90405 (7)	0.61203 (5)	0.01490 (10)
C16	0.38878 (6)	0.72871 (9)	0.26821 (5)	0.02396 (13)
H16B	0.4528	0.6771	0.2715	0.036*
H16A	0.3906	0.8146	0.2279	0.036*
H16C	0.3327	0.6681	0.2376	0.036*
C8	0.13379 (6)	1.21799 (9)	0.31782 (5)	0.02475 (13)
H8B	0.1962	1.1643	0.3161	0.037*
H8A	0.1335	1.3033	0.2765	0.037*
H8C	0.0751	1.1589	0.2919	0.037*
H18B	0.4504 (11)	0.0902 (15)	0.3960 (11)	0.045 (4)*
H18A	0.3831 (10)	0.0703 (14)	0.4546 (10)	0.033 (3)*
H6A	0.1303 (9)	0.8410 (13)	0.7431 (9)	0.028 (3)*
H14B	0.3640 (10)	0.2376 (14)	0.6235 (10)	0.033 (3)*
H14A	0.3506 (9)	0.3519 (14)	0.6907 (9)	0.031 (3)*
H6B	0.1286 (10)	0.7247 (14)	0.6724 (10)	0.036 (3)*
H17B	0.0504 (11)	0.5876 (15)	0.4157 (11)	0.045 (4)*
H17A	0.1117 (10)	0.5630 (15)	0.4979 (10)	0.037 (3)*
H3	0.1242 (9)	0.7870 (14)	0.4929 (9)	0.033 (3)*
H11	0.3851 (10)	0.2905 (15)	0.4467 (10)	0.043 (4)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S7	0.02231 (7)	0.01564 (6)	0.01558 (6)	-0.00113 (5)	0.00333 (5)	-0.00038 (5)
Cl2	0.02398 (7)	0.01862 (7)	0.01352 (6)	0.00121 (5)	0.00077 (5)	-0.00314 (5)
S1	0.01744 (6)	0.01459 (6)	0.01294 (6)	-0.00041 (5)	0.00259 (5)	-0.00314 (5)
S9	0.01931 (7)	0.01389 (6)	0.01357 (6)	0.00047 (5)	0.00357 (5)	-0.00272 (5)
S15	0.02861 (8)	0.01529 (7)	0.01681 (7)	-0.00147 (6)	0.00476 (6)	0.00022 (5)
Cl1	0.02353 (7)	0.01790 (6)	0.01740 (6)	-0.00308 (5)	0.00778 (5)	-0.00374 (5)
O18	0.0268 (2)	0.01509 (19)	0.0167 (2)	0.00361 (17)	0.00633 (18)	0.00171 (16)
O17	0.0251 (2)	0.0155 (2)	0.01450 (19)	-0.00279 (17)	0.00197 (17)	0.00079 (15)
N12	0.0187 (2)	0.0157 (2)	0.0147 (2)	-0.00090 (17)	0.00432 (17)	-0.00181 (17)
N3	0.0205 (2)	0.0144 (2)	0.0132 (2)	-0.00115 (17)	0.00220 (17)	-0.00289 (16)
N6	0.0265 (3)	0.0163 (2)	0.0143 (2)	-0.0013 (2)	0.00311 (19)	-0.00122 (18)
N11	0.0194 (2)	0.0141 (2)	0.0139 (2)	-0.00025 (17)	0.00404 (17)	-0.00234 (16)
N14	0.0241 (2)	0.0152 (2)	0.0137 (2)	0.00121 (19)	0.00275 (18)	-0.00082 (17)
N4	0.0190 (2)	0.0158 (2)	0.0137 (2)	-0.00094 (17)	0.00235 (17)	-0.00223 (17)
C10	0.0144 (2)	0.0141 (2)	0.0135 (2)	0.00004 (17)	0.00170 (18)	-0.00237 (17)
C5	0.0149 (2)	0.0160 (2)	0.0135 (2)	-0.00038 (18)	0.00204 (18)	-0.00209 (18)
C13	0.0159 (2)	0.0150 (2)	0.0140 (2)	-0.00095 (18)	0.00286 (18)	-0.00155 (18)
C2	0.0153 (2)	0.0154 (2)	0.0139 (2)	-0.00111 (18)	0.00202 (18)	-0.00291 (18)
C16	0.0260 (3)	0.0305 (4)	0.0159 (3)	0.0014 (3)	0.0047 (2)	0.0019 (2)

C8	0.0297 (3)	0.0296 (4)	0.0161 (3)	-0.0003 (3)	0.0069 (2)	0.0003 (2)
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Geometric parameters (Å, °)

S7—C5	1.7312 (7)	N3—H3	0.861 (13)
S7—C8	1.8024 (7)	N6—C2	1.3176 (9)
S1—C2	1.7355 (6)	N6—H6A	0.846 (12)
S1—C5	1.7593 (6)	N6—H6B	0.829 (13)
S9—C10	1.7331 (6)	N11—C10	1.3264 (8)
S9—C13	1.7613 (6)	N11—H11	0.913 (14)
S15—C13	1.7278 (7)	N14—C10	1.3164 (9)
S15—C16	1.8043 (8)	N14—H14B	0.808 (13)
O18—H18B	0.836 (15)	N14—H14A	0.876 (13)
O18—H18A	0.780 (14)	N4—C5	1.2923 (8)
O17—H17B	0.827 (15)	C16—H16B	0.9800
O17—H17A	0.796 (14)	C16—H16A	0.9800
N12—C13	1.2932 (8)	C16—H16C	0.9800
N12—N11	1.3791 (8)	C8—H8B	0.9800
N3—C2	1.3280 (8)	C8—H8A	0.9800
N3—N4	1.3781 (8)	C8—H8C	0.9800
C5—S7—C8	99.61 (3)	N11—C10—S9	110.27 (5)
C2—S1—C5	87.51 (3)	N4—C5—S7	124.86 (5)
C10—S9—C13	87.75 (3)	N4—C5—S1	115.35 (5)
C13—S15—C16	100.21 (3)	S7—C5—S1	119.78 (4)
H18B—O18—H18A	108.1 (13)	N12—C13—S15	125.50 (5)
H17B—O17—H17A	105.6 (13)	N12—C13—S9	115.05 (5)
C13—N12—N11	109.69 (5)	S15—C13—S9	119.45 (4)
C2—N3—N4	117.01 (5)	N6—C2—N3	125.04 (6)
C2—N3—H3	124.4 (8)	N6—C2—S1	124.52 (5)
N4—N3—H3	118.4 (8)	N3—C2—S1	110.44 (5)
C2—N6—H6A	118.6 (8)	S15—C16—H16B	109.5
C2—N6—H6B	120.6 (9)	S15—C16—H16A	109.5
H6A—N6—H6B	120.6 (12)	H16B—C16—H16A	109.5
C10—N11—N12	117.23 (5)	S15—C16—H16C	109.5
C10—N11—H11	123.5 (9)	H16B—C16—H16C	109.5
N12—N11—H11	119.3 (9)	H16A—C16—H16C	109.5
C10—N14—H14B	122.1 (9)	S7—C8—H8B	109.5
C10—N14—H14A	119.6 (8)	S7—C8—H8A	109.5
H14B—N14—H14A	117.7 (12)	H8B—C8—H8A	109.5
C5—N4—N3	109.70 (5)	S7—C8—H8C	109.5
N14—C10—N11	125.16 (6)	H8B—C8—H8C	109.5
N14—C10—S9	124.57 (5)	H8A—C8—H8C	109.5
C13—N12—N11—C10	0.74 (8)	C2—S1—C5—S7	179.35 (4)
C2—N3—N4—C5	0.04 (8)	N11—N12—C13—S15	179.19 (5)
N12—N11—C10—N14	178.94 (6)	N11—N12—C13—S9	-0.16 (7)
N12—N11—C10—S9	-0.95 (7)	C16—S15—C13—N12	0.81 (7)

C13—S9—C10—N14	-179.23 (6)	C16—S15—C13—S9	-179.87 (4)
C13—S9—C10—N11	0.67 (5)	C10—S9—C13—N12	-0.29 (5)
N3—N4—C5—S7	-179.17 (5)	C10—S9—C13—S15	-179.68 (4)
N3—N4—C5—S1	-0.44 (7)	N4—N3—C2—N6	-179.72 (6)
C8—S7—C5—N4	-3.94 (7)	N4—N3—C2—S1	0.37 (7)
C8—S7—C5—S1	177.38 (4)	C5—S1—C2—N6	179.60 (6)
C2—S1—C5—N4	0.55 (5)	C5—S1—C2—N3	-0.49 (5)

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>
N3—H3...O17	0.861 (13)	1.818 (13)	2.6778 (7)	176.4 (12)
N6—H6 <i>A</i> ...Cl1	0.846 (12)	2.296 (12)	3.1178 (6)	164.2 (11)
N6—H6 <i>B</i> ...Cl2 ⁱ	0.829 (13)	2.392 (13)	3.2139 (7)	171.4 (13)
N11—H11...O18	0.914 (14)	1.751 (14)	2.6632 (7)	176.7 (13)
N14—H14 <i>A</i> ...Cl2	0.877 (12)	2.289 (12)	3.1287 (6)	160.3 (11)
N14—H14 <i>B</i> ...Cl1 ⁱⁱ	0.807 (13)	2.461 (13)	3.2648 (6)	173.6 (13)
O17—H17 <i>A</i> ...Cl2 ⁱ	0.796 (14)	2.373 (14)	3.1593 (5)	169.8 (14)
O17—H17 <i>B</i> ...Cl2 ⁱⁱⁱ	0.826 (15)	2.340 (15)	3.1649 (6)	175.5 (14)
O18—H18 <i>A</i> ...Cl1 ⁱⁱ	0.780 (13)	2.414 (13)	3.1708 (5)	163.8 (13)
O18—H18 <i>B</i> ...Cl1 ^{iv}	0.837 (15)	2.318 (15)	3.1517 (6)	174.1 (14)

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