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Hydrochlorothiazide *N*,*N*-dimethylacetamide disolvate

Andrea Johnston,[a] Alastair J. Florence[a]* and Alan R. Kennedy[b]

[a]Department of Pharmaceutical Sciences, University of Strathclyde, 27 Taylor Street, Glasgow G4 0NR, Scotland, and [b]WestCHEM, Department of Pure & Applied Chemistry, University of Strathclyde, 295 Cathedral Street, Glasgow G1 1XL, Scotland

Correspondence e-mail: alastair.florence@strath.ac.uk

Key indicators

Single-crystal X-ray study
T = 123 K
Mean σ(C–C) = 0.004 Å
Disorder in solvent or counterion
R factor = 0.058
wR factor = 0.121
Data-to-parameter ratio = 16.5

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

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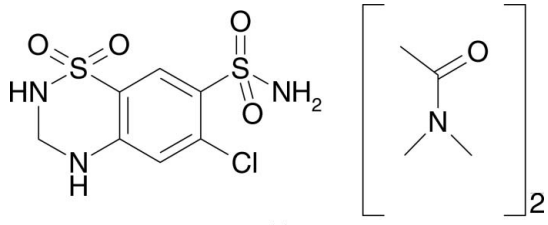
Hydrochlorothiazide forms a 1:2 solvate with *N*,*N*-dimethylacetamide (systematic name: 6-chloro-3,4-dihydro-2*H*-1,2,4-benzothiadiazine-7-sulfonamide 1,1-dioxide dimethylacetamide disolvate), $C_7H_8ClN_3O_4S_2\cdot 2C_4H_9NO$. The compound crystallizes with one hydrochorothiazide and two disordered solvent molecules in the asymmetric unit, with a hydrogen-bonding network comprising four N—H···O contacts.

Comment

Hydrochlorothiazide (HCT) is a thiazide diuretic which is known to crystallize in at least two non-solvated forms; form I (Dupont & Dideberg, 1972) and form II (Florence *et al.*, 2005). Compound (I) was produced during an automated parallel crystallization study on HCT. The sample was identified as a new form using multi-sample X-ray powder diffraction analysis of all recrystallized samples (Florence *et al.*, 2003). Subsequent manual recrystallization from a saturated *N*,*N*-dimethylacetamide (DMA) solution by slow evaporation at 298 K yielded samples of the HCT DMA disolvate suitable for single-crystal diffraction (Fig. 1).



(I)

The compound crystallizes with one HCT and two DMA molecules in the asymmetric unit. Both solvent molecules are disordered over two sites though the positions of the acetyl O atoms (O1*S* and O2*S*) and one methyl group from each solvent (C2*S* and C10*S*) are modelled as coincident. The S1/N1/C1/N2/C2/C7 six-membered ring in HCT adopts a non-planar conformation with atoms S1 and N1 having deviations of 0.105 (1) and 0.684 (3) Å, respectively, from the least-squares plane through atoms C2/C3/C4/C5/C6/C7. The sulfonamide side chain adopts a torsion angle N3—S2—C5—C4 of 60.7 (3)°, such that O3 eclipses H6, and O4 and N3 are staggered with respect to Cl1.

The structure contains four N—H···O hydrogen bonds, with N1, N2 and N3 of HCT donating contacts to adjacent acetyl O atoms of DMA (Fig. 2). In addition, there are two C—H···O contacts between HCT and solvent, with a third, *viz*. C3—H3···O1(x, 1 + y, z), forming an infinite chain of HCT molecules in the *b*-axis direction. Adjacent HCT chains

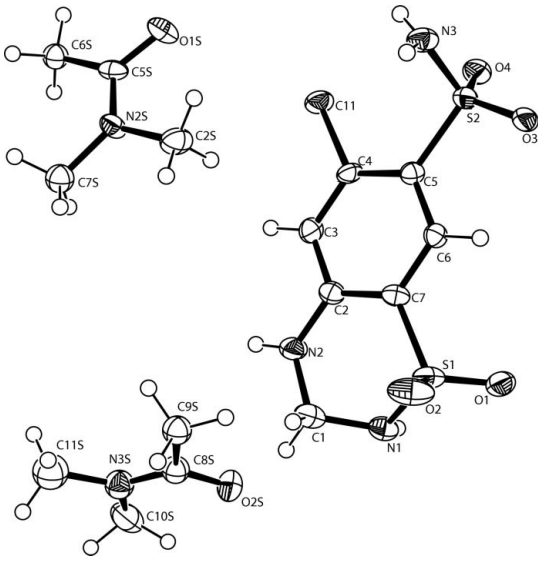


Figure 1
Drawing of the asymmetric unit, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. The minor occupancy disordered atom sites have been omitted for clarity.

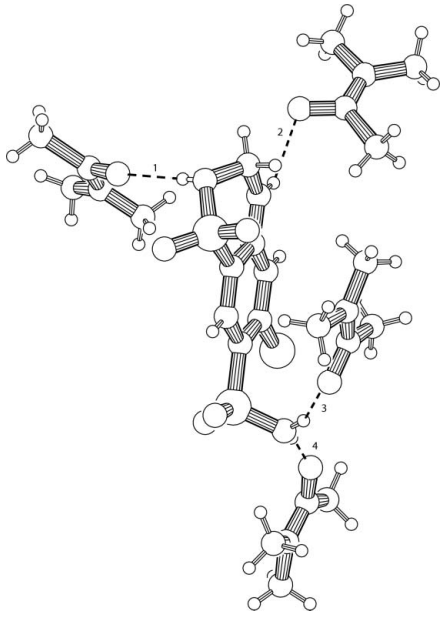


Figure 2
A partial packing diagram illustrating unique hydrogen bonds (dashed lines). Contacts are labelled as follow: 1 = N1···O2*S*($1-x, -\frac{1}{2}+y, \frac{1}{2}-z$) of 2.834 (4) Å; 2 = N2···O2*S* of 2.912 (4) Å; 3 = N3···O1*S*($x, -1+y, z$) of 2.873 (4) Å; 4 = N3···O1*S*($-x, -\frac{1}{2}+y, \frac{1}{2}-z$) of 2.881 (4) Å. Contacts illustrated using *PLATON* (Spek, 2003; Version 280604).

pack as layers in the *ab* plane and form an alternating stacked arrangement with layers of solvent molecules in the direction of the *c* axis (Fig. 3).

Experimental

A single-crystal sample of the title compound was recrystallized from a saturated dimethylacetamide solution by isothermal solvent evaporation at 298 K.

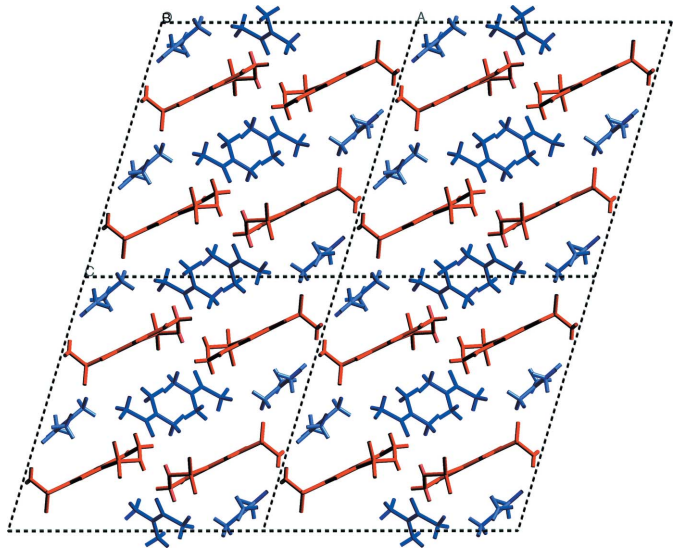


Figure 3
The crystal packing in the structure of (I), viewed down the *b* axis, showing the alternating layers of HCT and DMA molecules.

Crystal data

$C_7H_8ClN_3O_4S_2\cdot 2C_4H_9NO$
$M_r = 471.98$
Monoclinic, $P2_1/c$
$a = 17.0841\ (6)$ Å
$b = 7.3905\ (3)$ Å
$c = 17.7937\ (7)$ Å
$\beta = 106.875\ (2)^\circ$
$V = 2149.89\ (14)$ Å^3
$Z = 4$
$D_x = 1.458$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.41$ mm^{-1}
$T = 123\ (2)$ K
Prism, colourless
0.20 × 0.14 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer
ω and φ scans
Absorption correction: none
7559 measured reflections
4744 independent reflections
2544 reflections with $I > 2\sigma(I)$
$R_{int} = 0.067$
$\theta_{max} = 27.1^\circ$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.121$
$S = 1.01$
4744 reflections
288 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0486P)^2 + 0.4808P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.38$ e Å^{-3}
$\Delta\rho_{min} = -0.38$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*···O2*S*^{i}	0.75 (3)	2.08 (3)	2.834 (4)	175 (3)
N2—H2*N*···O2*S*	0.84 (4)	2.26 (4)	2.912 (4)	135 (3)
N3—H3*N*···O1*S*^{ii}	0.84 (4)	2.05 (4)	2.881 (4)	170 (3)
N3—H4*N*···O1*S*^{iii}	0.81 (3)	2.11 (4)	2.873 (4)	156 (3)
C1—H1*A*···O2*S*	0.99	2.56	3.100 (4)	114
C3—H3···O1^{iv}	0.95	2.42	3.275 (4)	149

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

Both DMA molecules were modelled as disordered over two sites. Occupancy factors refined to 0.61 (1):0.39 (1) for the molecule including atom O1*S* and to 0.56 (1):0.44 (1) for that including O2*S*. The DMA atoms N1*S*, N3*S*, N4*S*, C1*S*, C3*S*, C4*S*, C7*S*, C8*S*, C9*S*, C11*S*, C12*S*, C13*S* and C14*S* were treated isotropically. The amine H atoms were found through difference syntheses and refined [isotropically for those on N2 and N3, and with $U_{iso}(H) = 1.2U_{eq}(N1)$ for H1*N*]. All other H atoms were constrained to idealized positions using a riding model; $U_{iso}(H) = 1.2U_{eq}$ for CH and CH_2, $U_{iso}(H) = 1.5U_{eq}$ for CH_3, and C—H = 0.95, 0.99 and 0.98 Å for CH, CH_2 and CH_3, respectively.

Data collection: *COLLECT* (Hooft, 1988) and *DENZO* (Otwinowski & Minor, 1997); cell refinement: *DENZO* and *COLLECT*; data reduction: *DENZO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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supporting information

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Hall symbol: -P 2ybc
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$c = 17.7937$ (7) Å
$\beta = 106.875$ (2)°
$V = 2149.89$ (14) Å^3
$Z = 4$
$F(000) = 992$
$D_x = 1.458$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4979 reflections
$\theta = 1.0$–$27.1°$
$\mu = 0.41$ mm^{-1}
$T = 123$ K
Prism, colourless
0.20 × 0.14 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
7559 measured reflections
4744 independent reflections
2544 reflections with $I > 2\sigma(I)$
$R_{int} = 0.067$
$\theta_{max} = 27.1°$, $\theta_{min} = 1.3°$
$h = -21 \rightarrow 21$
$k = -9 \rightarrow 6$
$l = -22 \rightarrow 22$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.121$
$S = 1.01$
4744 reflections
288 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
$w = 1/[\sigma^2(F_o^2) + (0.0486P)^2 + 0.4808P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.38$ e Å^{-3}
$\Delta\rho_{min} = -0.38$ 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}	Occ. (<1)
Cl1	0.16219 (5)	0.42882 (11)	0.32597 (5)	0.0324 (2)	
S1	0.35182 (5)	−0.20503 (12)	0.22599 (5)	0.0282 (2)	
S2	0.11163 (5)	0.00683 (11)	0.35166 (5)	0.0275 (2)	
O1	0.37516 (12)	−0.3338 (3)	0.28875 (13)	0.0336 (6)	
O2	0.31010 (13)	−0.2697 (3)	0.14910 (13)	0.0420 (7)	
O3	0.10866 (13)	−0.1865 (3)	0.34756 (14)	0.0366 (6)	
O4	0.12786 (13)	0.0924 (3)	0.42702 (12)	0.0323 (6)	
N1	0.43442 (16)	−0.1002 (4)	0.22180 (16)	0.0277 (7)	
N2	0.37250 (16)	0.1930 (4)	0.20213 (18)	0.0327 (8)	
N3	0.02765 (17)	0.0846 (5)	0.2979 (2)	0.0296 (7)	
C1	0.4161 (2)	0.0582 (5)	0.1713 (2)	0.0343 (9)	
H1A	0.4677	0.1109	0.1666	0.041*	
H1B	0.3826	0.0217	0.1182	0.041*	
C2	0.31340 (18)	0.1506 (4)	0.23653 (18)	0.0241 (8)	
C3	0.27045 (17)	0.2885 (4)	0.26157 (18)	0.0265 (8)	
H3	0.2826	0.4114	0.2540	0.032*	
C4	0.21091 (18)	0.2481 (4)	0.29705 (18)	0.0235 (8)	
C5	0.19042 (17)	0.0684 (4)	0.30895 (17)	0.0218 (7)	
C6	0.23353 (17)	−0.0684 (4)	0.28549 (17)	0.0219 (7)	
H6	0.2211	−0.1911	0.2932	0.026*	
C7	0.29477 (17)	−0.0286 (4)	0.25075 (17)	0.0220 (8)	
O1S	0.02987 (13)	0.9177 (3)	0.15273 (13)	0.0345 (6)	
C2S	0.1018 (2)	0.6895 (4)	0.0857 (2)	0.0353 (9)	
H2S1	0.1452	0.6625	0.1334	0.053*	0.608 (12)
H2S2	0.1116	0.6214	0.0413	0.053*	0.608 (12)
H2S3	0.0488	0.6563	0.0913	0.053*	0.608 (12)
H2S4	0.1564	0.6609	0.1202	0.053*	0.392 (12)
H2S5	0.1030	0.6907	0.0310	0.053*	0.392 (12)
H2S6	0.0628	0.5977	0.0921	0.053*	0.392 (12)
N2S	0.1023 (3)	0.8849 (7)	0.0686 (3)	0.0252 (19)	0.608 (12)
C6S	0.0727 (5)	1.1960 (13)	0.1004 (5)	0.027 (3)*	0.608 (12)
H6S1	0.0425	1.2554	0.1326	0.041*	0.608 (12)
H6S2	0.0492	1.2310	0.0454	0.041*	0.608 (12)
H6S3	0.1302	1.2330	0.1184	0.041*	0.608 (12)
C5S	0.0668 (3)	0.9902 (10)	0.1081 (3)	0.0244 (19)	0.608 (12)
C7S	0.1485 (4)	0.9454 (12)	0.0156 (4)	0.0322 (19)*	0.608 (12)
H7S1	0.1233	1.0552	−0.0118	0.048*	0.608 (12)
H7S2	0.1480	0.8502	−0.0228	0.048*	0.608 (12)
H7S3	0.2051	0.9713	0.0461	0.048*	0.608 (12)
C1S	0.0755 (6)	0.8753 (16)	0.1077 (7)	0.029 (3)*	0.392 (12)

N1S	0.0990 (5)	1.0187 (13)	0.0733 (5)	0.028 (3)*	0.392 (12)
C4S	0.1538 (6)	1.0100 (17)	0.0235 (6)	0.029 (3)*	0.392 (12)
H4S1	0.2031	0.9418	0.0506	0.044*	0.392 (12)
H4S2	0.1691	1.1329	0.0125	0.044*	0.392 (12)
H4S3	0.1258	0.9494	−0.0261	0.044*	0.392 (12)
C3S	0.0738 (8)	1.201 (2)	0.0833 (8)	0.035 (4)*	0.392 (12)
H3S1	0.0215	1.1984	0.0956	0.053*	0.392 (12)
H3S2	0.0676	1.2694	0.0348	0.053*	0.392 (12)
H3S3	0.1153	1.2592	0.1265	0.053*	0.392 (12)
C10S	0.5241 (2)	0.7173 (5)	0.0822 (2)	0.0492 (11)	
H10A	0.5221	0.8096	0.1210	0.074*	0.559 (10)
H10B	0.5516	0.7690	0.0451	0.074*	0.559 (10)
H10C	0.5527	0.6112	0.1070	0.074*	0.559 (10)
H10D	0.5168	0.8230	0.1127	0.074*	0.441 (10)
H10E	0.5208	0.7548	0.0286	0.074*	0.441 (10)
H10F	0.5777	0.6626	0.1068	0.074*	0.441 (10)
O2S	0.45606 (14)	0.4448 (3)	0.12565 (13)	0.0381 (6)	
C13S	0.3276 (6)	0.4946 (15)	0.0121 (6)	0.032 (3)*	0.441 (10)
H13A	0.3461	0.3763	0.0355	0.048*	0.441 (10)
H13B	0.3022	0.4803	−0.0446	0.048*	0.441 (10)
H13C	0.2876	0.5452	0.0361	0.048*	0.441 (10)
N4S	0.3859 (5)	0.5960 (10)	0.0233 (5)	0.035 (3)*	0.441 (10)
C12S	0.4554 (6)	0.5744 (13)	0.0803 (5)	0.031 (2)*	0.441 (10)
C14S	0.3631 (7)	0.7524 (13)	−0.0302 (6)	0.048 (3)*	0.441 (10)
H14A	0.3999	0.8539	−0.0091	0.072*	0.441 (10)
H14B	0.3066	0.7879	−0.0350	0.072*	0.441 (10)
H14C	0.3678	0.7192	−0.0820	0.072*	0.441 (10)
N3S	0.4420 (3)	0.6676 (8)	0.0396 (3)	0.036 (2)*	0.559 (10)
C8S	0.4111 (4)	0.5235 (10)	0.0650 (4)	0.031 (2)*	0.559 (10)
C9S	0.3193 (4)	0.4448 (12)	0.0216 (5)	0.032 (2)*	0.559 (10)
H9S1	0.3060	0.3488	0.0539	0.048*	0.559 (10)
H9S2	0.3178	0.3956	−0.0299	0.048*	0.559 (10)
H9S3	0.2792	0.5428	0.0150	0.048*	0.559 (10)
C11S	0.4025 (6)	0.7766 (11)	−0.0286 (4)	0.052 (2)*	0.559 (10)
H11A	0.3549	0.7120	−0.0618	0.078*	0.559 (10)
H11B	0.4413	0.8004	−0.0586	0.078*	0.559 (10)
H11C	0.3849	0.8915	−0.0114	0.078*	0.559 (10)
H1N	0.461 (2)	−0.085 (6)	0.263 (2)	0.062*	
H3N	0.016 (2)	0.188 (5)	0.311 (2)	0.041 (12)*	
H4N	0.0164 (19)	0.058 (5)	0.252 (2)	0.031 (11)*	
H2N	0.3790 (19)	0.302 (5)	0.1912 (19)	0.038 (11)*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0320 (5)	0.0210 (4)	0.0495 (6)	0.0033 (4)	0.0199 (4)	−0.0037 (4)
S1	0.0227 (4)	0.0283 (5)	0.0371 (5)	−0.0016 (4)	0.0141 (4)	−0.0104 (5)
S2	0.0283 (5)	0.0218 (5)	0.0390 (6)	0.0032 (4)	0.0201 (4)	0.0029 (4)

O1	0.0307 (13)	0.0238 (13)	0.0510 (16)	0.0030 (10)	0.0191 (12)	−0.0005 (12)
O2	0.0286 (13)	0.0564 (17)	0.0419 (15)	−0.0058 (12)	0.0115 (11)	−0.0280 (13)
O3	0.0413 (14)	0.0198 (13)	0.0620 (17)	0.0033 (11)	0.0358 (12)	0.0054 (12)
O4	0.0352 (13)	0.0386 (14)	0.0274 (13)	0.0058 (11)	0.0159 (11)	0.0018 (12)
N1	0.0217 (16)	0.0312 (17)	0.0326 (18)	−0.0019 (13)	0.0116 (13)	−0.0030 (16)
N2	0.0296 (17)	0.0246 (18)	0.051 (2)	0.0003 (15)	0.0238 (15)	0.0020 (16)
N3	0.0270 (16)	0.0279 (19)	0.037 (2)	0.0005 (14)	0.0137 (15)	−0.0080 (18)
C1	0.0305 (19)	0.041 (2)	0.037 (2)	0.0010 (17)	0.0182 (17)	−0.0030 (19)
C2	0.0182 (17)	0.0271 (19)	0.0272 (19)	−0.0020 (15)	0.0070 (15)	−0.0006 (16)
C3	0.0227 (17)	0.0206 (18)	0.036 (2)	−0.0029 (15)	0.0087 (16)	0.0019 (17)
C4	0.0231 (17)	0.0194 (19)	0.030 (2)	0.0053 (14)	0.0103 (15)	−0.0035 (15)
C5	0.0196 (16)	0.0221 (18)	0.0233 (18)	0.0004 (15)	0.0059 (14)	−0.0009 (15)
C6	0.0242 (17)	0.0188 (17)	0.0236 (18)	−0.0021 (15)	0.0082 (14)	−0.0015 (15)
C7	0.0181 (16)	0.027 (2)	0.0217 (18)	−0.0017 (14)	0.0069 (14)	−0.0065 (15)
O1S	0.0334 (13)	0.0397 (15)	0.0380 (14)	−0.0056 (12)	0.0224 (12)	−0.0023 (12)
C2S	0.035 (2)	0.024 (2)	0.051 (2)	0.0001 (17)	0.0188 (18)	−0.0026 (18)
N2S	0.030 (3)	0.024 (4)	0.027 (3)	−0.003 (2)	0.016 (2)	−0.003 (2)
C5S	0.016 (3)	0.035 (4)	0.022 (4)	0.000 (3)	0.005 (3)	−0.006 (3)
C10S	0.060 (3)	0.046 (3)	0.053 (3)	−0.004 (2)	0.035 (2)	−0.003 (2)
O2S	0.0426 (15)	0.0359 (15)	0.0347 (14)	0.0057 (12)	0.0097 (12)	0.0093 (12)

Geometric parameters (Å, º)

Cl1—C4	1.729 (3)	C7S—H7S1	0.9800
S1—O2	1.429 (2)	C7S—H7S2	0.9800
S1—O1	1.433 (2)	C7S—H7S3	0.9800
S1—N1	1.630 (3)	C1S—N1S	1.342 (19)
S1—C7	1.759 (3)	N1S—C3S	1.441 (18)
S2—O3	1.431 (2)	N1S—C4S	1.466 (14)
S2—O4	1.435 (2)	C4S—H4S1	0.9800
S2—N3	1.584 (3)	C4S—H4S2	0.9800
S2—C5	1.786 (3)	C4S—H4S3	0.9800
N1—C1	1.453 (4)	C3S—H3S1	0.9800
N1—H1N	0.75 (4)	C3S—H3S2	0.9800
N2—C2	1.360 (4)	C3S—H3S3	0.9800
N2—C1	1.444 (4)	C10S—N3S	1.435 (6)
N2—H2N	0.84 (3)	C10S—C12S	1.572 (10)
N3—H3N	0.84 (4)	C10S—H10A	0.9782
N3—H4N	0.81 (3)	C10S—H10B	0.9912
C1—H1A	0.9900	C10S—H10C	0.9606
C1—H1B	0.9900	C10S—H10D	0.9800
C2—C3	1.402 (4)	C10S—H10E	0.9800
C2—C7	1.403 (4)	C10S—H10F	0.9800
C3—C4	1.377 (4)	O2S—C12S	1.250 (10)
C3—H3	0.9500	O2S—C8S	1.269 (8)
C4—C5	1.405 (4)	C13S—N4S	1.215 (13)
C5—C6	1.384 (4)	C13S—H13A	0.9800
C6—C7	1.392 (4)	C13S—H13B	0.9800

C6—H6	0.9500	C13S—H13C	0.9800
O1S—C5S	1.267 (7)	N4S—C12S	1.329 (15)
O1S—C1S	1.308 (13)	N4S—C14S	1.476 (12)
C2S—N2S	1.476 (6)	C14S—H14A	0.9800
C2S—C1S	1.531 (12)	C14S—H14B	0.9800
C2S—H2S1	0.9729	C14S—H14C	0.9800
C2S—H2S2	0.9897	C14S—H11A	0.6155
C2S—H2S3	0.9710	C14S—H11C	1.1113
C2S—H2S4	0.9800	N3S—C8S	1.325 (12)
C2S—H2S5	0.9800	N3S—C11S	1.449 (9)
C2S—H2S6	0.9800	C8S—C9S	1.642 (10)
N2S—C5S	1.310 (10)	C9S—H9S1	0.9800
N2S—C7S	1.464 (9)	C9S—H9S2	0.9800
C6S—C5S	1.533 (12)	C9S—H9S3	0.9800
C6S—H6S1	0.9800	C11S—H11A	0.9800
C6S—H6S2	0.9800	C11S—H11B	0.9800
C6S—H6S3	0.9800	C11S—H11C	0.9800

O2—S1—O1	118.19 (14)	O1S—C5S—N2S	118.5 (6)
O2—S1—N1	108.25 (14)	O1S—C5S—C6S	122.2 (6)
O1—S1—N1	107.61 (14)	N2S—C5S—C6S	119.3 (7)
O2—S1—C7	109.22 (14)	O1S—C1S—N1S	113.6 (10)
O1—S1—C7	110.25 (14)	O1S—C1S—C2S	130.1 (10)
N1—S1—C7	102.07 (15)	N1S—C1S—C2S	116.3 (10)
O3—S2—O4	118.81 (14)	C1S—N1S—C3S	122.8 (11)
O3—S2—N3	108.62 (16)	C1S—N1S—C4S	124.7 (10)
O4—S2—N3	107.48 (17)	C3S—N1S—C4S	112.5 (9)
O3—S2—C5	104.63 (14)	N1S—C4S—H4S1	109.5
O4—S2—C5	108.69 (14)	N1S—C4S—H4S2	109.5
N3—S2—C5	108.22 (15)	H4S1—C4S—H4S2	109.5
C1—N1—S1	112.1 (2)	N1S—C4S—H4S3	109.5
C1—N1—H1N	116 (3)	H4S1—C4S—H4S3	109.5
S1—N1—H1N	109 (3)	H4S2—C4S—H4S3	109.5
C2—N2—C1	122.9 (3)	N1S—C3S—H3S1	109.5
C2—N2—H2N	119 (2)	N1S—C3S—H3S2	109.5
C1—N2—H2N	117 (2)	H3S1—C3S—H3S2	109.5
S2—N3—H3N	114 (2)	N1S—C3S—H3S3	109.5
S2—N3—H4N	115 (2)	H3S1—C3S—H3S3	109.5
H3N—N3—H4N	121 (4)	H3S2—C3S—H3S3	109.5
N2—C1—N1	111.3 (3)	N3S—C10S—C12S	37.6 (3)
N2—C1—H1A	109.4	N3S—C10S—H10A	108.9
N1—C1—H1A	109.4	C12S—C10S—H10A	107.8
N2—C1—H1B	109.4	N3S—C10S—H10B	109.0
N1—C1—H1B	109.4	C12S—C10S—H10B	137.8
H1A—C1—H1B	108.0	H10A—C10S—H10B	108.7
N2—C2—C3	120.1 (3)	N3S—C10S—H10C	108.9
N2—C2—C7	122.4 (3)	C12S—C10S—H10C	74.9
C3—C2—C7	117.5 (3)	H10A—C10S—H10C	111.2

C4—C3—C2	120.9 (3)	H10B—C10S—H10C	110.1
C4—C3—H3	119.6	N3S—C10S—H10D	103.6
C2—C3—H3	119.6	C12S—C10S—H10D	109.5
C3—C4—C5	121.6 (3)	H10A—C10S—H10D	10.4
C3—C4—Cl1	116.9 (2)	H10B—C10S—H10D	102.9
C5—C4—Cl1	121.5 (2)	H10C—C10S—H10D	121.7
C6—C5—C4	117.8 (3)	N3S—C10S—H10E	77.7
C6—C5—S2	118.4 (2)	C12S—C10S—H10E	109.8
C4—C5—S2	123.8 (2)	H10A—C10S—H10E	119.0
C5—C6—C7	120.9 (3)	H10B—C10S—H10E	31.3
C5—C6—H6	119.5	H10C—C10S—H10E	123.8
C7—C6—H6	119.5	H10D—C10S—H10E	109.5
C6—C7—C2	121.2 (3)	N3S—C10S—H10F	140.7
C6—C7—S1	119.8 (2)	C12S—C10S—H10F	109.1
C2—C7—S1	118.9 (2)	H10A—C10S—H10F	101.0
C5S—O1S—C1S	39.1 (4)	H10B—C10S—H10F	83.9
N2S—C2S—C1S	36.4 (4)	H10C—C10S—H10F	34.3
N2S—C2S—H2S1	109.4	H10D—C10S—H10F	109.5
C1S—C2S—H2S1	99.6	H10E—C10S—H10F	109.5
N2S—C2S—H2S2	108.7	C12S—O2S—C8S	37.9 (4)
C1S—C2S—H2S2	142.2	C13S—N4S—C12S	124.0 (9)
H2S1—C2S—H2S2	109.3	C13S—N4S—C14S	109.1 (8)
N2S—C2S—H2S3	109.3	C12S—N4S—C14S	126.6 (8)
C1S—C2S—H2S3	81.3	O2S—C12S—N4S	115.0 (8)
H2S1—C2S—H2S3	110.8	O2S—C12S—C10S	129.4 (8)
H2S2—C2S—H2S3	109.4	N4S—C12S—C10S	115.6 (8)
N2S—C2S—H2S4	106.1	N4S—C14S—H11A	99.0
C1S—C2S—H2S4	109.2	H14A—C14S—H11A	132.3
H2S1—C2S—H2S4	20.3	H14B—C14S—H11A	95.3
H2S2—C2S—H2S4	92.3	H14C—C14S—H11A	22.9
H2S3—C2S—H2S4	128.9	N4S—C14S—H11C	121.9
N2S—C2S—H2S5	77.5	H14A—C14S—H11C	19.2
C1S—C2S—H2S5	109.8	H14B—C14S—H11C	90.7
H2S1—C2S—H2S5	129.7	H14C—C14S—H11C	113.8
H2S2—C2S—H2S5	32.4	H11A—C14S—H11C	133.8
H2S3—C2S—H2S5	113.3	C8S—N3S—C10S	116.7 (6)
H2S4—C2S—H2S5	109.5	C8S—N3S—C11S	126.8 (7)
N2S—C2S—H2S6	138.3	C10S—N3S—C11S	116.5 (5)
C1S—C2S—H2S6	109.4	O2S—C8S—N3S	117.2 (6)
H2S1—C2S—H2S6	97.3	O2S—C8S—C9S	119.8 (6)
H2S2—C2S—H2S6	91.1	N3S—C8S—C9S	123.0 (7)
H2S3—C2S—H2S6	29.3	N3S—C11S—H11A	109.7
H2S4—C2S—H2S6	109.5	N3S—C11S—H11B	109.4
H2S5—C2S—H2S6	109.5	H11A—C11S—H11B	109.5
C5S—N2S—C7S	125.7 (6)	N3S—C11S—H11C	109.3
C5S—N2S—C2S	116.1 (6)	H11A—C11S—H11C	109.5
C7S—N2S—C2S	117.9 (5)	H11B—C11S—H11C	109.5

O2—S1—N1—C1	63.1 (3)	N1—S1—C7—C2	20.2 (3)
O1—S1—N1—C1	−168.1 (2)	C1S—C2S—N2S—C5S	−1.0 (7)
C7—S1—N1—C1	−52.1 (2)	C1S—C2S—N2S—C7S	173.3 (9)
C2—N2—C1—N1	−39.1 (4)	C1S—O1S—C5S—N2S	5.0 (7)
S1—N1—C1—N2	64.2 (3)	C1S—O1S—C5S—C6S	−173.7 (11)
C1—N2—C2—C3	−176.7 (3)	C7S—N2S—C5S—O1S	−179.0 (5)
C1—N2—C2—C7	5.5 (5)	C2S—N2S—C5S—O1S	−5.2 (6)
N2—C2—C3—C4	−179.5 (3)	C7S—N2S—C5S—C6S	−0.2 (8)
C7—C2—C3—C4	−1.5 (4)	C2S—N2S—C5S—C6S	173.5 (5)
C2—C3—C4—C5	−0.5 (5)	C5S—O1S—C1S—N1S	0.6 (5)
C2—C3—C4—Cl1	179.7 (2)	C5S—O1S—C1S—C2S	−175.8 (15)
C3—C4—C5—C6	1.5 (4)	N2S—C2S—C1S—O1S	171.9 (14)
Cl1—C4—C5—C6	−178.7 (2)	N2S—C2S—C1S—N1S	−4.4 (5)
C3—C4—C5—S2	−177.9 (2)	O1S—C1S—N1S—C3S	−1.9 (13)
Cl1—C4—C5—S2	1.8 (4)	C2S—C1S—N1S—C3S	175.1 (8)
O3—S2—C5—C6	−3.1 (3)	O1S—C1S—N1S—C4S	176.6 (7)
O4—S2—C5—C6	124.8 (2)	C2S—C1S—N1S—C4S	−6.4 (12)
N3—S2—C5—C6	−118.8 (3)	C8S—O2S—C12S—N4S	1.3 (6)
O3—S2—C5—C4	176.4 (3)	C8S—O2S—C12S—C10S	179.9 (13)
O4—S2—C5—C4	−55.8 (3)	C13S—N4S—C12S—O2S	−3.6 (12)
N3—S2—C5—C4	60.7 (3)	C14S—N4S—C12S—O2S	169.6 (7)
C4—C5—C6—C7	−0.4 (4)	C13S—N4S—C12S—C10S	177.6 (8)
S2—C5—C6—C7	179.0 (2)	C14S—N4S—C12S—C10S	−9.2 (11)
C5—C6—C7—C2	−1.7 (4)	N3S—C10S—C12S—O2S	−178.8 (12)
C5—C6—C7—S1	176.9 (2)	N3S—C10S—C12S—N4S	−0.2 (5)
N2—C2—C7—C6	−179.5 (3)	C12S—C10S—N3S—C8S	−0.2 (6)
C3—C2—C7—C6	2.6 (4)	C12S—C10S—N3S—C11S	−178.1 (9)
N2—C2—C7—S1	2.0 (4)	C12S—O2S—C8S—N3S	−0.9 (7)
C3—C2—C7—S1	−175.9 (2)	C12S—O2S—C8S—C9S	178.8 (11)
O2—S1—C7—C6	87.2 (3)	C10S—N3S—C8S—O2S	1.5 (7)
O1—S1—C7—C6	−44.2 (3)	C11S—N3S—C8S—O2S	179.1 (6)
N1—S1—C7—C6	−158.3 (2)	C10S—N3S—C8S—C9S	−178.2 (5)
O2—S1—C7—C2	−94.2 (3)	C11S—N3S—C8S—C9S	−0.6 (10)
O1—S1—C7—C2	134.4 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O2*S*^{i}	0.75 (3)	2.08 (3)	2.834 (4)	175 (3)
N2—H2*N*···O2*S*	0.84 (4)	2.26 (4)	2.912 (4)	135 (3)
N3—H3*N*···O1*S*^{ii}	0.84 (4)	2.05 (4)	2.881 (4)	170 (3)
N3—H4*N*···O1*S*^{iii}	0.81 (3)	2.11 (4)	2.873 (4)	156 (3)
C1—H1*A*···O2*S*	0.99	2.56	3.100 (4)	114
C3—H3···O1^{iv}	0.95	2.42	3.275 (4)	149

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