Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Hydrochlorothiazide *N*,*N*-dimethylformamide solvate

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Key indicators

Single-crystal synchrotron study
T = 150 K
Mean σ(C–C) = 0.003 Å
R factor = 0.040
wR factor = 0.107
Data-to-parameter ratio = 10.4

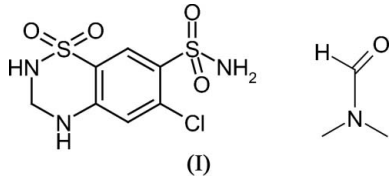
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 23 March 2006
Accepted 28 March 2006
Online 7 April 2006

Hydrochlorothiazide forms a 1:1 solvate with *N*,*N*-dimethylformamide (systematic name: 6-chloro-3,4-dihydro-2*H*-1,2,4-benzothiadiazine-7-sulfonamide-1,1-dioxide dimethylformamide solvate), $C_7H_8ClN_3O_4S_2\cdot C_3H_7NO$. The compound crystallizes with two molecules of hydrochlorothiazide and two solvent molecules in the asymmetric unit and displays an extensive hydrogen-bonding network.

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). Form (I) was produced during an automated parallel crystallization polymorph screen on HCT. The sample was identified as a novel 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*-dimethylformamide (DMF):acetone solution by slow evaporation at 278 K yielded samples of the HCT DMF solvate suitable for a synchrotron microcrystal study (Cernik *et al.*, 1997, 2000).



(I)

The compound crystallizes as a 1:1 solvate with $Z' = 2$ (Fig. 1). The benzothiadiazine ring of HCT adopts a non-planar conformation in both residues, with the largest deviations from the least-squares plane through atoms C2–C7 observed for atoms S1 and N1 in residue *A* [0.278 (1) and 0.770 (2) Å respectively] and atom N1*A* in residue *B* [0.6802 (2) Å]. In residue *A*, the sulphonamide side chain adopts a torsion angle N3—S2—C5—C6 of −57.7 (2)° such that the NH_2 group is located on the same side of the molecule as the H atom (H1N) bonded to N1, a similar arrangement to that in both of the non-solvated forms of HCT. The corresponding torsion angle in residue *B* is 60.76 (2)°, such that the NH_2 group lies on the opposite side of the molecule to the H atom (H5N) bonded to N1*A*.

The crystal structure is stabilized by a network of seven N—H···O and one N—H···N intermolecular hydrogen bonds (Table 1). These contacts interconnect (*a*) HCT molecules (Fig. 2, contacts 1, 2, 4 and 6) and (*b*) HCT and solvent molecules (Fig. 2 contacts 3, 4, 5, 7 and 8). Residues *A* and *B* form parallel *C*(8) (Etter, 1990) hydrogen-bonded chains in the direction of the *b* axis *via* contacts 2 and 6, respectively (Fig. 3),

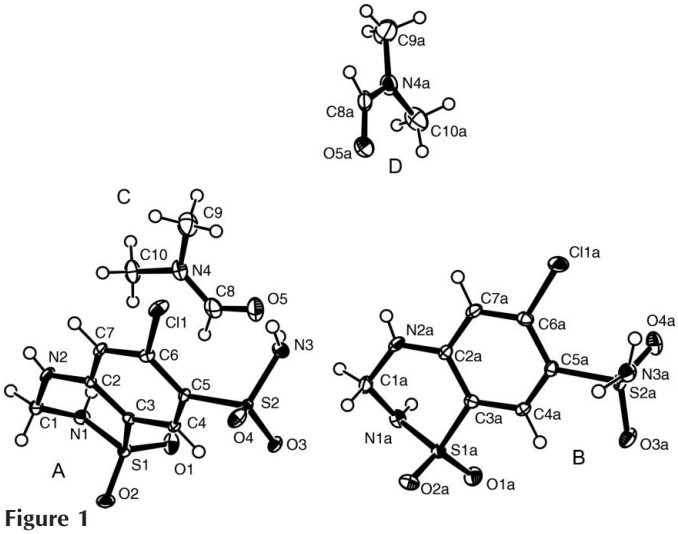


Figure 1
The asymmetric unit, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

with the chains interconnected *via* C—H···O contacts to form layers in the *ab* plane. The layers stack along the *c* axis with solvent residue *C* lying between layers of HCT molecules, interconnected *via* contacts 3 and 6 to HCT residues *A* and *B*, respectively. The remaining solvent molecule (residue *D*) lies approximately perpendicular to the *ab* plane and forms hydrogen bonds with HCT residue *B* (Fig. 2, contacts 5 and 8). The structure is further stabilized by seven C—H···O contacts (Table 1).

Experimental

The sample of HCT used to prepare the solvate was used as received from Sigma–Aldrich. This was recrystallized from a 50:50 DMF/acetone solution by isothermal solvent evaporation at 278 K.

Crystal data

$C_7H_8ClN_3O_4S_2\cdot C_3H_7NO$	V = 1509.50 (7) Å^3
M_r = 370.83	Z = 4
Triclinic, $P\overline{1}$	D_x = 1.632 Mg m^{-3}
a = 7.3028 (2) Å	Synchrotron radiation
b = 9.1492 (2) Å	λ = 0.8466 Å
c = 23.6989 (6) Å	μ = 0.56 mm^{-1}
α = 86.194 (1)°	T = 150 (2) K
β = 89.841 (1)°	Plate, colourless
γ = 72.855 (1)°	0.18 × 0.10 × 0.03 mm

Data collection

Bruker SMART APEX2 CCD diffractometer	4574 independent reflections
	4311 reflections with $I > 2\sigma(I)$
ω scans	R_{int} = 0.018
Absorption correction: none	θ_{max} = 29.0°
10824 measured reflections	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0721P)^2 + 1.2599P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.040	
$wR(F^2)$ = 0.107	
S = 1.05	$(\Delta/\sigma)_{max}$ = 0.001
4574 reflections	$\Delta\rho_{max}$ = 0.52 e Å^{-3}
441 parameters	$\Delta\rho_{min}$ = −0.52 e Å^{-3}
H atoms treated by a mixture of independent and constrained refinement	

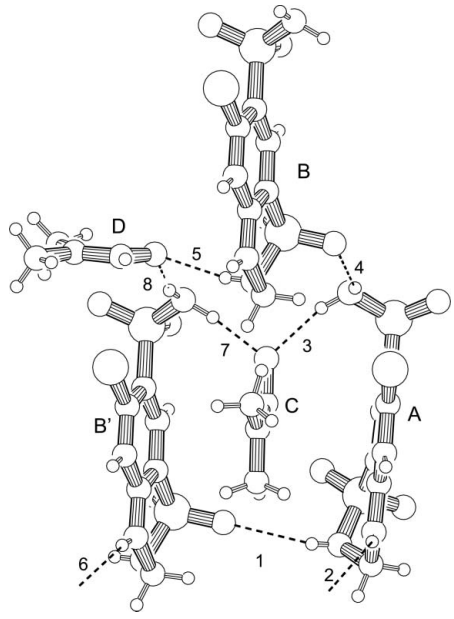


Figure 2
A packing diagram illustrating hydrogen bonds in (I). Unique contacts are labelled as follow: 1 = N1···O2A^{i}; 2 = N2···O4^{i}; 3 = N3···O5; 4 = N3···O2A^{ii}; 5 = N1A···O5A^{iii}; 6 = N2A^{i}···N3A(x, −2 + y, z); 7 = N3A—H7N···O5^{iv}; 8 = N3A—H8N···O5A^{iv} (see Table 1 for symmetry codes and geometry). Contacts calculated and illustrated using *PLATON* (Spek, 2003; program version 280604). Contact 6 is shown outwith the asymmetric unit for clarity.

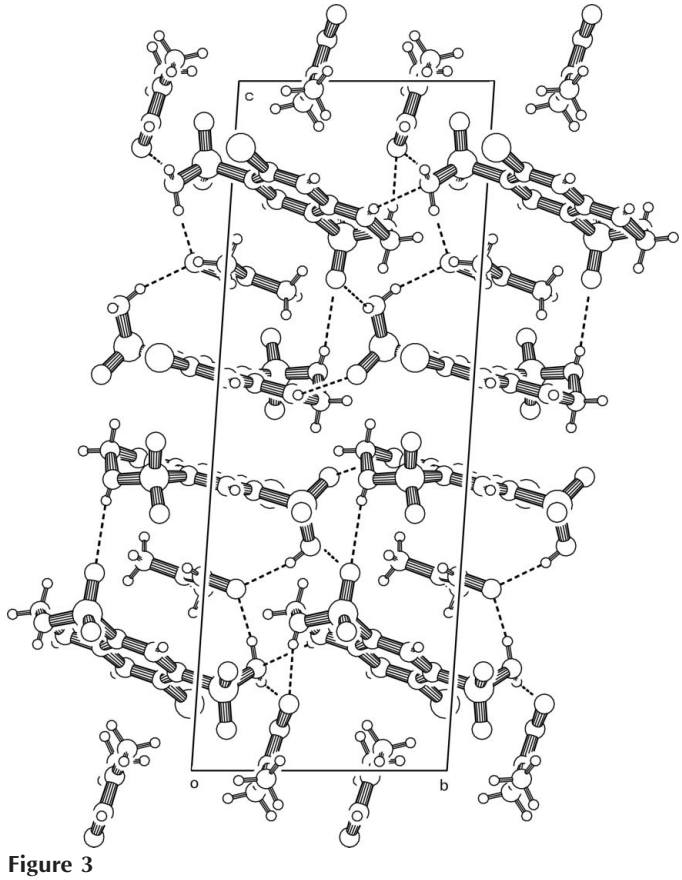


Figure 3
The crystal packing in (I), viewed down the *a* axis, showing the alternating layers of HCT and DMF molecules stacked along *c*. Hydrogen bonds are shown as dashed lines.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O2A^{i}	0.80 (3)	2.60 (3)	3.290 (3)	146 (3)
N2—H2N···O4^{i}	0.74 (3)	2.47 (3)	3.023 (3)	134 (3)
N3—H3N···O5	0.90 (3)	2.07 (3)	2.954 (3)	165 (3)
N3—H4N···O2A^{ii}	0.85 (4)	2.35 (4)	3.092 (3)	146 (3)
N1A—H5N···O5A^{iii}	0.78 (4)	2.12 (4)	2.882 (3)	167 (3)
N2A—H6N···N3A^{i}	0.77 (4)	2.48 (4)	3.177 (3)	150 (3)
N3A—H7N···O5^{iv}	0.95 (4)	1.89 (4)	2.820 (3)	164 (3)
N3A—H8N···O5A^{iv}	0.86 (3)	2.12 (3)	2.942 (3)	163 (2)
C1A—H1A1···O3A^{i}	0.99	2.42	3.157 (3)	131
C1—H1A···O3^{i}	0.99	2.56	3.235 (3)	125
C1A—H1A2···O3	0.99	2.51	3.467 (3)	162
C7—H7···O2^{ii}	0.95	2.56	3.466 (3)	159
C7A—H7A···O1A^{ii}	0.95	2.45	3.163 (3)	132
C9—H9B···O1^{ii}	0.98	2.54	3.442 (3)	152
C10—H10C···O1^{ii}	0.98	2.51	3.323 (3)	141

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

The amine and aldehyde H atoms were located by difference synthesis and refined isotropically. The remaining H atoms were positioned geometrically and a riding model with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(C_{methyl})$ was used during the refinement process (C—H distances 0.95, 0.99 and 0.98 Å for CH, CH_2 and CH_3 groups, respectively).

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; 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*.

The authors thank the Basic Technology programme of the UK Research Councils for funding this work under the project Control and Prediction of the Organic Solid State (URL: http://www.cposs.org.uk). Thanks are also due to the CCLRC for provision of a beamtime grant at Daresbury SRS.

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supporting information

Acta Cryst. (2006). E**62**, o1730–o1732 [https://doi.org/10.1107/S1600536806011391]

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6-chloro-3,4-dihydro-2*H*-1,2,4-benzothiadiazine-7-sulfonamide-1,1-dioxide dimethylformamide solvate

Crystal data

$C_7H_8ClN_3O_4S_2\cdot C_3H_7NO$
$M_r = 370.83$
Triclinic, $P\overline{1}$
$a = 7.3028$ (2) Å
$b = 9.1492$ (2) Å
$c = 23.6989$ (6) Å
$\alpha = 86.194$ (1)°
$\beta = 89.841$ (1)°
$\gamma = 72.855$ (1)°
$V = 1509.50$ (7) Å^3
$Z = 4$
$F(000) = 768$
$D_x = 1.632$ Mg m^{-3}
Synchrotron radiation, $\lambda = 0.8466$ Å
Cell parameters from 7222 reflections
$\theta = 4.1$–$32.9°$
$\mu = 0.56$ mm^{-1}
$T = 150$ K
Plate, colourless
0.18 × 0.10 × 0.03 mm

Data collection

Bruker SMART APEX2 CCD diffractometer
Radiation source: Station 16.2SMX, Daresbury SRS
Si111 monochromator
fine–slice ω scans
10824 measured reflections
4574 independent reflections
4311 reflections with $I > 2\sigma(I)$
$R_{int} = 0.018$
$\theta_{max} = 29.0°$, $\theta_{min} = 4.0°$
$h = -8\rightarrow8$
$k = -10\rightarrow10$
$l = -26\rightarrow27$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.107$
$S = 1.05$
4574 reflections
441 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
$w = 1/[\sigma^2(F_o^2) + (0.0721P)^2 + 1.2599P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.52$ e Å^{-3}
$\Delta\rho_{min} = -0.52$ e Å^{-3}

Special details

Experimental. Collected at Daresbury SRS Station 16.2SMX, *SADABS* used to correct for beam decay (entered as _diffrn_standards_decay_% below).

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}
Cl1	0.16046 (7)	0.23586 (6)	0.40126 (3)	0.01755 (17)
Cl1A	0.30299 (9)	0.97445 (7)	0.09678 (3)	0.02518 (18)
S1	0.98182 (8)	−0.22413 (6)	0.42499 (2)	0.01265 (16)
S1A	1.02805 (8)	0.55067 (6)	0.22707 (2)	0.01389 (17)
S2	0.53889 (7)	0.36026 (6)	0.38526 (2)	0.01035 (16)
S2A	0.66913 (8)	1.10301 (6)	0.11975 (2)	0.01556 (17)
O1	1.0676 (2)	−0.19429 (19)	0.37273 (7)	0.0221 (4)
O1A	1.1984 (2)	0.5695 (2)	0.20171 (8)	0.0254 (4)
O2	1.0864 (2)	−0.22855 (19)	0.47642 (7)	0.0199 (4)
O2A	1.0034 (2)	0.5673 (2)	0.28664 (7)	0.0211 (4)
O3	0.7321 (2)	0.36121 (17)	0.37450 (7)	0.0142 (3)
O3A	0.8435 (2)	1.11607 (19)	0.14354 (8)	0.0223 (4)
O4	0.4310 (2)	0.45978 (17)	0.42562 (7)	0.0171 (4)
O4A	0.6407 (3)	1.1259 (2)	0.05959 (7)	0.0251 (4)
O5	0.5475 (3)	0.1246 (2)	0.26342 (7)	0.0291 (4)
O5A	0.1090 (3)	0.3633 (2)	0.09657 (7)	0.0239 (4)
N1	0.9323 (3)	−0.3863 (2)	0.42229 (9)	0.0156 (4)
N1A	1.0131 (3)	0.3819 (2)	0.21446 (9)	0.0176 (4)
N2	0.6067 (3)	−0.2923 (2)	0.45327 (9)	0.0159 (4)
N2A	0.6726 (3)	0.4691 (2)	0.19691 (9)	0.0182 (5)
N3	0.4220 (3)	0.4054 (2)	0.32568 (9)	0.0174 (4)
N3A	0.4927 (3)	1.2259 (2)	0.14799 (10)	0.0186 (5)
N4	0.4364 (3)	−0.0809 (2)	0.28300 (8)	0.0204 (5)
N4A	0.0586 (3)	0.2989 (3)	0.00804 (9)	0.0258 (5)
C1	0.7916 (3)	−0.4047 (3)	0.46429 (10)	0.0158 (5)
H1A	0.7776	−0.5090	0.4636	0.019*
H1B	0.8384	−0.3937	0.5025	0.019*
C1A	0.8273 (3)	0.3648 (3)	0.23111 (10)	0.0177 (5)
H1A1	0.8272	0.2580	0.2268	0.021*
H1A2	0.8061	0.3857	0.2714	0.021*
C2	0.5902 (3)	−0.1436 (2)	0.43836 (9)	0.0104 (5)
C2A	0.6700 (3)	0.6140 (2)	0.17986 (9)	0.0118 (5)
C3	0.7519 (3)	−0.0915 (2)	0.42761 (9)	0.0098 (4)

C3A	0.8251 (3)	0.6725 (3)	0.19079 (9)	0.0126 (5)
C4	0.7321 (3)	0.0619 (2)	0.41259 (9)	0.0101 (4)
H4	0.8432	0.0944	0.4067	0.012*
C4A	0.8206 (3)	0.8199 (3)	0.17185 (9)	0.0128 (5)
H4A	0.9278	0.8552	0.1790	0.015*
C5	0.5525 (3)	0.1677 (2)	0.40615 (9)	0.0100 (4)
C5A	0.6629 (3)	0.9165 (3)	0.14273 (9)	0.0130 (5)
C6	0.3912 (3)	0.1149 (2)	0.41410 (9)	0.0114 (5)
C6A	0.5078 (3)	0.8593 (3)	0.13203 (9)	0.0144 (5)
C7	0.4086 (3)	−0.0352 (3)	0.43099 (9)	0.0121 (5)
H7	0.2967	−0.0660	0.4378	0.015*
C7A	0.5110 (3)	0.7131 (3)	0.14953 (10)	0.0151 (5)
H7A	0.4047	0.6779	0.1411	0.018*
C8	0.5705 (4)	−0.0106 (3)	0.28142 (10)	0.0226 (6)
C8A	0.0065 (4)	0.3334 (3)	0.06038 (11)	0.0229 (6)
C9	0.2396 (4)	−0.0010 (3)	0.26562 (13)	0.0324 (7)
H9A	0.2216	0.1097	0.2621	0.049*
H9B	0.1522	−0.0238	0.2940	0.049*
H9C	0.2122	−0.0350	0.2291	0.049*
C9A	−0.0677 (5)	0.2566 (4)	−0.03098 (13)	0.0444 (8)
H9D	−0.1918	0.2667	−0.0133	0.067*
H9E	−0.0101	0.1502	−0.0403	0.067*
H9F	−0.0861	0.3244	−0.0656	0.067*
C10	0.4762 (4)	−0.2415 (3)	0.30413 (11)	0.0234 (6)
H10A	0.6137	−0.2854	0.3122	0.035*
H10B	0.4368	−0.2992	0.2755	0.035*
H10C	0.4046	−0.2474	0.3388	0.035*
C10A	0.2476 (4)	0.2942 (4)	−0.01197 (12)	0.0354 (7)
H10D	0.3094	0.3439	0.0144	0.053*
H10E	0.2363	0.3483	−0.0495	0.053*
H10F	0.3250	0.1873	−0.0143	0.053*
H1N	0.905 (4)	−0.407 (4)	0.3917 (14)	0.030 (9)*
H2N	0.519 (4)	−0.316 (3)	0.4553 (12)	0.020 (8)*
H3N	0.464 (5)	0.331 (4)	0.3015 (14)	0.037 (9)*
H4N	0.301 (5)	0.433 (4)	0.3292 (13)	0.035 (9)*
H5N	1.036 (5)	0.364 (4)	0.1832 (15)	0.033 (9)*
H6N	0.594 (5)	0.438 (4)	0.1852 (13)	0.030 (9)*
H7N	0.486 (5)	1.202 (4)	0.1875 (16)	0.046 (9)*
H8	0.692 (4)	−0.071 (3)	0.2937 (12)	0.023 (7)*
H8A	−0.126 (5)	0.339 (3)	0.0660 (13)	0.034 (8)*
H8N	0.388 (4)	1.249 (3)	0.1287 (12)	0.021 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0063 (3)	0.0127 (3)	0.0325 (4)	−0.0006 (2)	0.0005 (2)	−0.0033 (2)
Cl1A	0.0177 (3)	0.0213 (3)	0.0350 (4)	−0.0046 (3)	−0.0123 (3)	0.0041 (3)
S1	0.0092 (3)	0.0083 (3)	0.0189 (3)	−0.0007 (2)	−0.0010 (2)	0.0013 (2)

S1A	0.0098 (3)	0.0154 (3)	0.0163 (3)	−0.0040 (2)	−0.0005 (2)	0.0013 (2)
S2	0.0088 (3)	0.0065 (3)	0.0156 (3)	−0.0023 (2)	0.0000 (2)	0.0002 (2)
S2A	0.0169 (3)	0.0123 (3)	0.0182 (3)	−0.0059 (2)	0.0027 (2)	0.0010 (2)
O1	0.0177 (9)	0.0180 (9)	0.0269 (10)	−0.0005 (7)	0.0089 (7)	0.0022 (7)
O1A	0.0110 (9)	0.0306 (10)	0.0340 (10)	−0.0071 (7)	−0.0001 (7)	0.0074 (8)
O2	0.0156 (8)	0.0176 (8)	0.0261 (9)	−0.0051 (7)	−0.0096 (7)	0.0023 (7)
O2A	0.0218 (9)	0.0253 (9)	0.0161 (9)	−0.0063 (7)	−0.0037 (7)	−0.0036 (7)
O3	0.0095 (8)	0.0105 (8)	0.0225 (9)	−0.0032 (6)	−0.0001 (6)	0.0021 (6)
O3A	0.0165 (9)	0.0176 (9)	0.0347 (10)	−0.0087 (7)	0.0014 (7)	0.0014 (7)
O4	0.0171 (9)	0.0101 (8)	0.0256 (9)	−0.0051 (7)	0.0042 (7)	−0.0070 (7)
O4A	0.0329 (10)	0.0239 (10)	0.0178 (9)	−0.0088 (8)	0.0045 (7)	0.0049 (7)
O5	0.0504 (12)	0.0222 (10)	0.0186 (9)	−0.0170 (9)	0.0040 (8)	−0.0018 (8)
O5A	0.0266 (10)	0.0251 (10)	0.0185 (9)	−0.0051 (8)	0.0023 (8)	−0.0032 (7)
N1	0.0173 (11)	0.0098 (10)	0.0191 (11)	−0.0029 (8)	−0.0030 (8)	−0.0021 (8)
N1A	0.0201 (11)	0.0156 (11)	0.0157 (11)	−0.0025 (8)	0.0027 (9)	−0.0026 (9)
N2	0.0123 (11)	0.0128 (10)	0.0247 (11)	−0.0081 (9)	−0.0021 (8)	0.0028 (8)
N2A	0.0175 (11)	0.0171 (11)	0.0237 (11)	−0.0111 (9)	−0.0023 (9)	−0.0008 (8)
N3	0.0156 (11)	0.0147 (11)	0.0196 (11)	−0.0020 (9)	−0.0054 (9)	0.0035 (9)
N3A	0.0181 (12)	0.0135 (10)	0.0232 (12)	−0.0031 (9)	−0.0004 (9)	−0.0010 (9)
N4	0.0252 (11)	0.0162 (10)	0.0175 (11)	−0.0030 (9)	0.0038 (9)	0.0007 (8)
N4A	0.0355 (13)	0.0252 (12)	0.0186 (11)	−0.0121 (10)	0.0005 (9)	0.0001 (9)
C1	0.0188 (12)	0.0098 (11)	0.0194 (12)	−0.0062 (9)	−0.0046 (10)	0.0036 (9)
C1A	0.0222 (13)	0.0135 (12)	0.0191 (12)	−0.0083 (10)	0.0007 (10)	0.0010 (9)
C2	0.0136 (11)	0.0103 (11)	0.0089 (10)	−0.0059 (9)	−0.0007 (8)	0.0000 (8)
C2A	0.0137 (11)	0.0136 (11)	0.0102 (11)	−0.0067 (9)	0.0041 (9)	−0.0037 (9)
C3	0.0092 (11)	0.0091 (11)	0.0099 (10)	−0.0010 (9)	−0.0007 (8)	−0.0006 (8)
C3A	0.0108 (11)	0.0156 (12)	0.0111 (11)	−0.0036 (9)	0.0010 (9)	−0.0012 (9)
C4	0.0092 (11)	0.0110 (11)	0.0111 (10)	−0.0044 (9)	0.0007 (8)	−0.0010 (8)
C4A	0.0111 (11)	0.0149 (12)	0.0142 (11)	−0.0064 (9)	0.0020 (9)	−0.0030 (9)
C5	0.0107 (11)	0.0079 (10)	0.0119 (11)	−0.0036 (9)	0.0009 (8)	−0.0006 (8)
C5A	0.0159 (12)	0.0116 (11)	0.0125 (11)	−0.0054 (9)	0.0022 (9)	−0.0020 (9)
C6	0.0086 (11)	0.0111 (11)	0.0135 (11)	−0.0012 (9)	0.0013 (8)	−0.0035 (9)
C6A	0.0111 (11)	0.0184 (12)	0.0131 (11)	−0.0036 (9)	0.0000 (9)	−0.0010 (9)
C7	0.0099 (11)	0.0139 (11)	0.0148 (11)	−0.0070 (9)	0.0018 (9)	−0.0013 (9)
C7A	0.0130 (11)	0.0184 (12)	0.0169 (12)	−0.0089 (10)	0.0009 (9)	−0.0026 (9)
C8	0.0322 (15)	0.0249 (15)	0.0110 (12)	−0.0083 (12)	0.0036 (10)	−0.0046 (10)
C8A	0.0290 (15)	0.0186 (13)	0.0218 (14)	−0.0094 (11)	0.0008 (11)	0.0052 (10)
C9	0.0257 (15)	0.0276 (15)	0.0387 (17)	−0.0019 (12)	0.0025 (12)	0.0070 (12)
C9A	0.062 (2)	0.061 (2)	0.0252 (15)	−0.0406 (18)	0.0013 (14)	−0.0061 (14)
C10	0.0296 (14)	0.0159 (12)	0.0213 (13)	−0.0022 (11)	0.0081 (11)	0.0003 (10)
C10A	0.0318 (16)	0.0441 (18)	0.0236 (14)	−0.0013 (13)	0.0058 (12)	−0.0008 (13)

Geometric parameters (Å, º)

Cl1—C6	1.736 (2)	N4A—C8A	1.327 (3)
Cl1A—C6A	1.734 (2)	N4A—C10A	1.448 (4)
S1—O2	1.4321 (17)	N4A—C9A	1.455 (4)
S1—O1	1.4328 (18)	C1—H1A	0.9900

S1—N1	1.633 (2)	C1—H1B	0.9900
S1—C3	1.762 (2)	C1A—H1A1	0.9900
S1A—O1A	1.4305 (18)	C1A—H1A2	0.9900
S1A—O2A	1.4354 (17)	C2—C7	1.406 (3)
S1A—N1A	1.626 (2)	C2—C3	1.414 (3)
S1A—C3A	1.756 (2)	C2A—C7A	1.409 (3)
S2—O4	1.4330 (16)	C2A—C3A	1.419 (3)
S2—O3	1.4355 (16)	C3—C4	1.389 (3)
S2—N3	1.617 (2)	C3A—C4A	1.384 (3)
S2—C5	1.772 (2)	C4—C5	1.384 (3)
S2A—O3A	1.4337 (18)	C4—H4	0.9500
S2A—O4A	1.4339 (18)	C4A—C5A	1.381 (3)
S2A—N3A	1.617 (2)	C4A—H4A	0.9500
S2A—C5A	1.770 (2)	C5—C6	1.407 (3)
O5—C8	1.244 (3)	C5A—C6A	1.410 (3)
O5A—C8A	1.236 (3)	C6—C7	1.373 (3)
N1—C1	1.466 (3)	C6A—C7A	1.368 (3)
N1—H1N	0.80 (3)	C7—H7	0.9500
N1A—C1A	1.461 (3)	C7A—H7A	0.9500
N1A—H5N	0.78 (3)	C8—H8	0.93 (3)
N2—C2	1.353 (3)	C8A—H8A	0.96 (3)
N2—C1	1.448 (3)	C9—H9A	0.9800
N2—H2N	0.74 (3)	C9—H9B	0.9800
N2A—C2A	1.354 (3)	C9—H9C	0.9800
N2A—C1A	1.453 (3)	C9A—H9D	0.9800
N2A—H6N	0.77 (3)	C9A—H9E	0.9800
N3—H3N	0.90 (3)	C9A—H9F	0.9800
N3—H4N	0.85 (3)	C10—H10A	0.9800
N3A—H7N	0.95 (4)	C10—H10B	0.9800
N3A—H8N	0.85 (3)	C10—H10C	0.9800
N4—C8	1.320 (3)	C10A—H10D	0.9800
N4—C9	1.454 (3)	C10A—H10E	0.9800
N4—C10	1.465 (3)	C10A—H10F	0.9800
O2—S1—O1	118.00 (11)	N2A—C2A—C7A	120.5 (2)
O2—S1—N1	108.58 (10)	N2A—C2A—C3A	122.4 (2)
O1—S1—N1	108.28 (11)	C7A—C2A—C3A	117.1 (2)
O2—S1—C3	110.17 (10)	C4—C3—C2	121.31 (19)
O1—S1—C3	108.51 (10)	C4—C3—S1	118.17 (16)
N1—S1—C3	102.12 (10)	C2—C3—S1	120.11 (16)
O1A—S1A—O2A	118.65 (11)	C4A—C3A—C2A	121.3 (2)
O1A—S1A—N1A	108.46 (11)	C4A—C3A—S1A	120.39 (17)
O2A—S1A—N1A	107.67 (11)	C2A—C3A—S1A	118.29 (17)
O1A—S1A—C3A	109.92 (10)	C5—C4—C3	120.7 (2)
O2A—S1A—C3A	108.80 (10)	C5—C4—H4	119.6
N1A—S1A—C3A	102.04 (11)	C3—C4—H4	119.6
O4—S2—O3	118.49 (9)	C5A—C4A—C3A	121.1 (2)
O4—S2—N3	107.21 (11)	C5A—C4A—H4A	119.5

O3—S2—N3	107.14 (11)	C3A—C4A—H4A	119.5
O4—S2—C5	109.69 (10)	C4—C5—C6	118.10 (19)
O3—S2—C5	106.03 (9)	C4—C5—S2	118.06 (16)
N3—S2—C5	107.87 (10)	C6—C5—S2	123.78 (16)
O3A—S2A—O4A	118.50 (11)	C4A—C5A—C6A	117.9 (2)
O3A—S2A—N3A	107.69 (11)	C4A—C5A—S2A	118.19 (17)
O4A—S2A—N3A	107.36 (12)	C6A—C5A—S2A	123.85 (17)
O3A—S2A—C5A	104.96 (10)	C7—C6—C5	121.7 (2)
O4A—S2A—C5A	109.65 (10)	C7—C6—Cl1	116.86 (17)
N3A—S2A—C5A	108.34 (11)	C5—C6—Cl1	121.38 (17)
C1—N1—S1	112.79 (16)	C7A—C6A—C5A	121.9 (2)
C1—N1—H1N	111 (2)	C7A—C6A—Cl1A	117.40 (17)
S1—N1—H1N	116 (2)	C5A—C6A—Cl1A	120.73 (18)
C1A—N1A—S1A	111.16 (16)	C6—C7—C2	120.7 (2)
C1A—N1A—H5N	112 (2)	C6—C7—H7	119.7
S1A—N1A—H5N	111 (2)	C2—C7—H7	119.7
C2—N2—C1	121.7 (2)	C6A—C7A—C2A	120.8 (2)
C2—N2—H2N	119 (2)	C6A—C7A—H7A	119.6
C1—N2—H2N	120 (2)	C2A—C7A—H7A	119.6
C2A—N2A—C1A	123.0 (2)	O5—C8—N4	125.3 (3)
C2A—N2A—H6N	118 (2)	O5—C8—H8	119.4 (17)
C1A—N2A—H6N	118 (2)	N4—C8—H8	115.2 (17)
S2—N3—H3N	111 (2)	O5A—C8A—N4A	125.3 (3)
S2—N3—H4N	114 (2)	O5A—C8A—H8A	123.6 (18)
H3N—N3—H4N	113 (3)	N4A—C8A—H8A	111.0 (18)
S2A—N3A—H7N	111 (2)	N4—C9—H9A	109.5
S2A—N3A—H8N	113.8 (19)	N4—C9—H9B	109.5
H7N—N3A—H8N	117 (3)	H9A—C9—H9B	109.5
C8—N4—C9	121.6 (2)	N4—C9—H9C	109.5
C8—N4—C10	121.9 (2)	H9A—C9—H9C	109.5
C9—N4—C10	116.5 (2)	H9B—C9—H9C	109.5
C8A—N4A—C10A	121.4 (2)	N4A—C9A—H9D	109.5
C8A—N4A—C9A	121.7 (2)	N4A—C9A—H9E	109.5
C10A—N4A—C9A	116.8 (2)	H9D—C9A—H9E	109.5
N2—C1—N1	111.50 (18)	N4A—C9A—H9F	109.5
N2—C1—H1A	109.3	H9D—C9A—H9F	109.5
N1—C1—H1A	109.3	H9E—C9A—H9F	109.5
N2—C1—H1B	109.3	N4—C10—H10A	109.5
N1—C1—H1B	109.3	N4—C10—H10B	109.5
H1A—C1—H1B	108.0	H10A—C10—H10B	109.5
N2A—C1A—N1A	111.24 (19)	N4—C10—H10C	109.5
N2A—C1A—H1A1	109.4	H10A—C10—H10C	109.5
N1A—C1A—H1A1	109.4	H10B—C10—H10C	109.5
N2A—C1A—H1A2	109.4	N4A—C10A—H10D	109.5
N1A—C1A—H1A2	109.4	N4A—C10A—H10E	109.5
H1A1—C1A—H1A2	108.0	H10D—C10A—H10E	109.5
N2—C2—C7	120.5 (2)	N4A—C10A—H10F	109.5
N2—C2—C3	122.1 (2)	H10D—C10A—H10F	109.5

C7—C2—C3	117.30 (19)	H10E—C10A—H10F	109.5
O2—S1—N1—C1	69.22 (18)	S1A—C3A—C4A—C5A	179.31 (17)
O1—S1—N1—C1	−161.54 (15)	C3—C4—C5—C6	−1.3 (3)
C3—S1—N1—C1	−47.16 (18)	C3—C4—C5—S2	−178.50 (16)
O1A—S1A—N1A—C1A	−170.70 (16)	O4—S2—C5—C4	−124.24 (17)
O2A—S1A—N1A—C1A	59.74 (18)	O3—S2—C5—C4	4.8 (2)
C3A—S1A—N1A—C1A	−54.69 (18)	N3—S2—C5—C4	119.31 (19)
C2—N2—C1—N1	−42.9 (3)	O4—S2—C5—C6	58.7 (2)
S1—N1—C1—N2	65.1 (2)	O3—S2—C5—C6	−172.23 (18)
C2A—N2A—C1A—N1A	−37.9 (3)	N3—S2—C5—C6	−57.7 (2)
S1A—N1A—C1A—N2A	65.0 (2)	C3A—C4A—C5A—C6A	−1.1 (3)
C1—N2—C2—C7	−176.4 (2)	C3A—C4A—C5A—S2A	−179.77 (16)
C1—N2—C2—C3	5.9 (3)	O3A—S2A—C5A—C4A	−5.9 (2)
C1A—N2A—C2A—C7A	−176.3 (2)	O4A—S2A—C5A—C4A	122.37 (18)
C1A—N2A—C2A—C3A	4.5 (3)	N3A—S2A—C5A—C4A	−120.75 (19)
N2—C2—C3—C4	−179.7 (2)	O3A—S2A—C5A—C6A	175.47 (19)
C7—C2—C3—C4	2.5 (3)	O4A—S2A—C5A—C6A	−56.2 (2)
N2—C2—C3—S1	7.7 (3)	N3A—S2A—C5A—C6A	60.7 (2)
C7—C2—C3—S1	−170.05 (16)	C4—C5—C6—C7	3.8 (3)
O2—S1—C3—C4	84.64 (19)	S2—C5—C6—C7	−179.20 (17)
O1—S1—C3—C4	−45.9 (2)	C4—C5—C6—Cl1	−174.61 (16)
N1—S1—C3—C4	−160.14 (17)	S2—C5—C6—Cl1	2.4 (3)
O2—S1—C3—C2	−102.56 (18)	C4A—C5A—C6A—C7A	−0.1 (3)
O1—S1—C3—C2	126.87 (18)	S2A—C5A—C6A—C7A	178.50 (18)
N1—S1—C3—C2	12.7 (2)	C4A—C5A—C6A—Cl1A	178.92 (17)
N2A—C2A—C3A—C4A	178.6 (2)	S2A—C5A—C6A—Cl1A	−2.5 (3)
C7A—C2A—C3A—C4A	−0.7 (3)	C5—C6—C7—C2	−3.1 (3)
N2A—C2A—C3A—S1A	0.8 (3)	Cl1—C6—C7—C2	175.37 (17)
C7A—C2A—C3A—S1A	−178.55 (16)	N2—C2—C7—C6	−177.9 (2)
O1A—S1A—C3A—C4A	−39.7 (2)	C3—C2—C7—C6	−0.1 (3)
O2A—S1A—C3A—C4A	91.75 (19)	C5A—C6A—C7A—C2A	0.9 (3)
N1A—S1A—C3A—C4A	−154.64 (18)	Cl1A—C6A—C7A—C2A	−178.17 (17)
O1A—S1A—C3A—C2A	138.16 (17)	N2A—C2A—C7A—C6A	−179.8 (2)
O2A—S1A—C3A—C2A	−90.39 (19)	C3A—C2A—C7A—C6A	−0.5 (3)
N1A—S1A—C3A—C2A	23.2 (2)	C9—N4—C8—O5	−3.1 (4)
C2—C3—C4—C5	−1.8 (3)	C10—N4—C8—O5	179.1 (2)
S1—C3—C4—C5	170.89 (17)	C10A—N4A—C8A—O5A	−0.4 (4)
C2A—C3A—C4A—C5A	1.5 (3)	C9A—N4A—C8A—O5A	176.5 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O2*A*^{i}	0.80 (3)	2.60 (3)	3.290 (3)	146 (3)
N2—H2*N*···O4^{i}	0.74 (3)	2.47 (3)	3.023 (3)	134 (3)
N3—H3*N*···O5	0.90 (3)	2.07 (3)	2.954 (3)	165 (3)
N3—H4*N*···O2*A*^{ii}	0.85 (4)	2.35 (4)	3.092 (3)	146 (3)
N1*A*—H5*N*···O5*A*^{iii}	0.78 (4)	2.12 (4)	2.882 (3)	167 (3)

N2*A*—H6*N*···N3*A*^{i}	0.77 (4)	2.48 (4)	3.177 (3)	150 (3)
N3*A*—H7*N*···O5^{iv}	0.95 (4)	1.89 (4)	2.820 (3)	164 (3)
N3*A*—H8*N*···O5*A*^{iv}	0.86 (3)	2.12 (3)	2.942 (3)	163 (2)
C1*A*—H1*A*1···O3*A*^{i}	0.99	2.42	3.157 (3)	131
C1—H1*A*···O3^{i}	0.99	2.56	3.235 (3)	125
C1*A*—H1*A*2···O3	0.99	2.51	3.467 (3)	162
C7—H7···O2^{ii}	0.95	2.56	3.466 (3)	159
C7*A*—H7*A*···O1*A*^{ii}	0.95	2.45	3.163 (3)	132
C9—H9*B*···O1^{ii}	0.98	2.54	3.442 (3)	152
C10—H10*C*···O1^{ii}	0.98	2.51	3.323 (3)	141

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