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Hydrochlorothiazide–aniline (1/1)

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Key indicators

Single-crystal X-ray study
T = 123 K
Mean σ(C–C) = 0.002 Å
R factor = 0.034
wR factor = 0.084
Data-to-parameter ratio = 23.1

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

Hydrochlorothiazide forms a 1:1 solvate with aniline, $C_7H_8ClN_3O_4S_2\cdot C_6H_7N$. The crystal structure contains a hydrogen-bonding network comprising two N—H···N and three N—H···O contacts.

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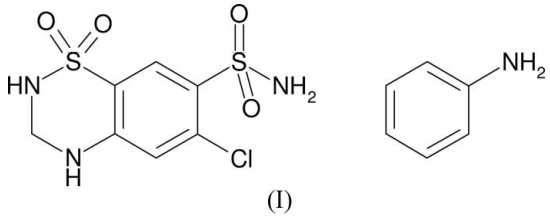
Comment

Hydrochlorothiazide (HCT) is a thiazide diuretic which is known to crystallize in at least one non-solvated form (Dupont & Dideberg, 1972). The aniline solvate, (I), was produced during an automated parallel crystallization polymorph screen on HCT. The sample was identified as a novel form using multisample X-ray powder diffraction analysis of all recrystallized samples (Florence *et al.*, 2003). Subsequent manual recrystallization from a saturated 1:1 acetone–aniline solution by slow evaporation at 278 K yielded samples of (I) suitable for single-crystal X-ray analysis (Fig. 1).



(I)

In (I), the N1—S1—C1—N2—C2—C7 six-membered ring in HCT adopts a non-planar conformation, with atoms S1 and N1 having deviations of 0.271 (1) and 0.843 (1) Å, respectively, from the least-squares plane through atoms C2–C7. The sulfonamide side chain adopts a torsion angle N3—S2—C5—C4 of 69.05 (12)°, such that atom O4 eclipses H6 and atoms O3

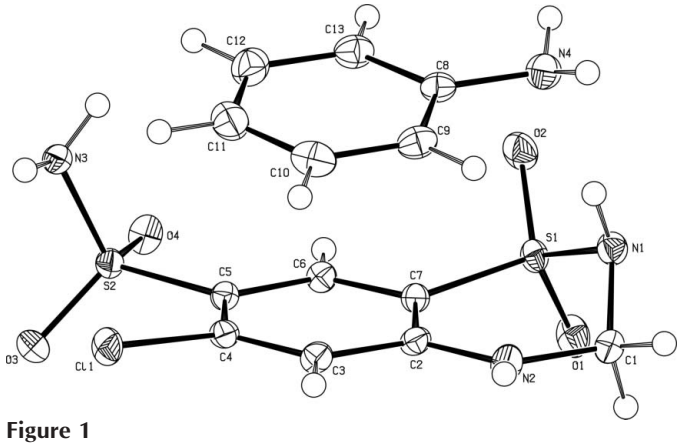


Figure 1
Plot of the asymmetric unit contents with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

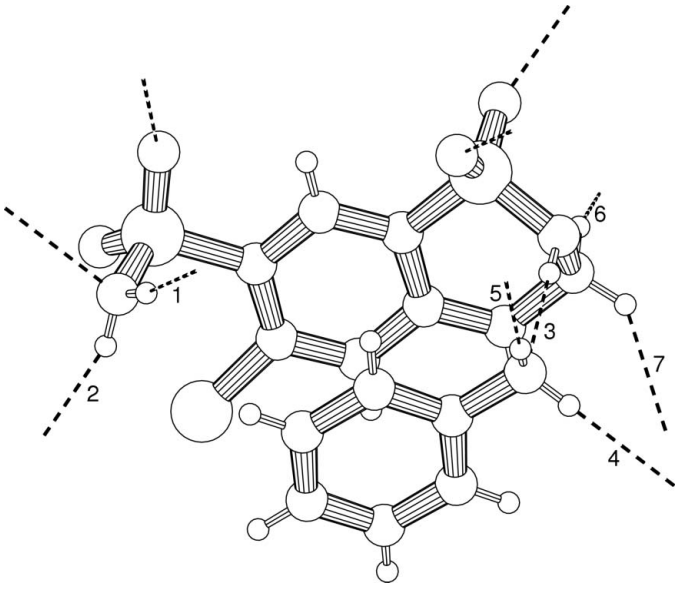


Figure 2
A packing diagram of (I). Dashed lines indicate hydrogen bonds and unique contacts are labelled as follows: 1 = N3···O2 [2.9725 (16) Å, O2 in the molecule at $(1-x, 1-y, 2-z)$]; 2 = N3···O1 [2.9390 (15) Å, O1 in the molecule at $(\frac{1}{2}+x, \frac{1}{2}-y, -\frac{1}{2}+z)$]; 3 = N1···N4 [2.9652 (17) Å]; 4 = N4···N3 [3.3944 (18) Å, in the molecule at $(-1+x, y, z)$]; 5 = N4···O4 [3.1000 (17) Å, O4 in the molecule at $(1-x, 1-y, 2-z)$]; 6 = C1···O3 [3.2535 (18) Å, O3 in the molecule at $(1-x, -y, 2-z)$]; 7 = C1···O3 [3.2852 (17) Å, O3 in the molecule at $(-1+x, y, z)$]. Contacts calculated and illustrated using *PLATON* (Spek, 2003; program version 280604)

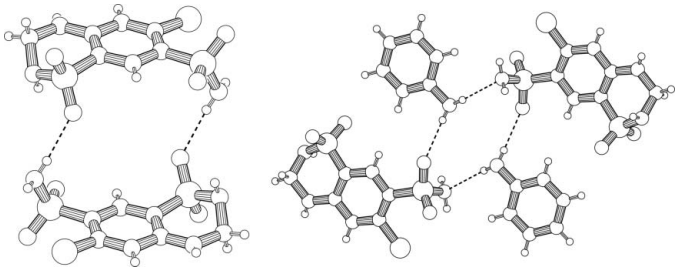


Figure 3
The $R_2^2(16)$ (left) and $R_4^4(12)$ hydrogen-bond motifs in the crystal structure of (I).

and N3 are staggered with respect to Cl1. In the non-solvated structure (Dupont & Dideberg, 1972), this group is rotated by approximately 130° compared to (I) such that the amine group lies on the opposite side of the benzothiadiazine moiety. The aniline molecule is planar, the greatest deviation of any non-H atom from the least-squares plane through C8–C13/N4 being 0.013 (1) Å for C8.

The crystal structure is stabilized by a network of hydrogen bonds interconnecting (*a*) HCT molecules (Fig. 2, contacts 1 and 2) and (*b*) HCT and solvent molecules (contacts 3, 4 and 5). Two C—H···O contacts also exist between HCT molecules (contacts 6 and 7). Contact 1 (N3—H3*N*···O2) forms a centrosymmetric $R_2^2(16)$ motif between molecules of HCT, whilst contacts 4 and 5 (N4—H5N···N3 and N4—H6N···O4) combine to form an $R_4^4(12)$ motif between aniline and HCT (Fig. 3). Hydrophobic interactions between HCT and aniline include a C—H···π contact and an offset face-to-face (off) π–π approach (Fig. 4).

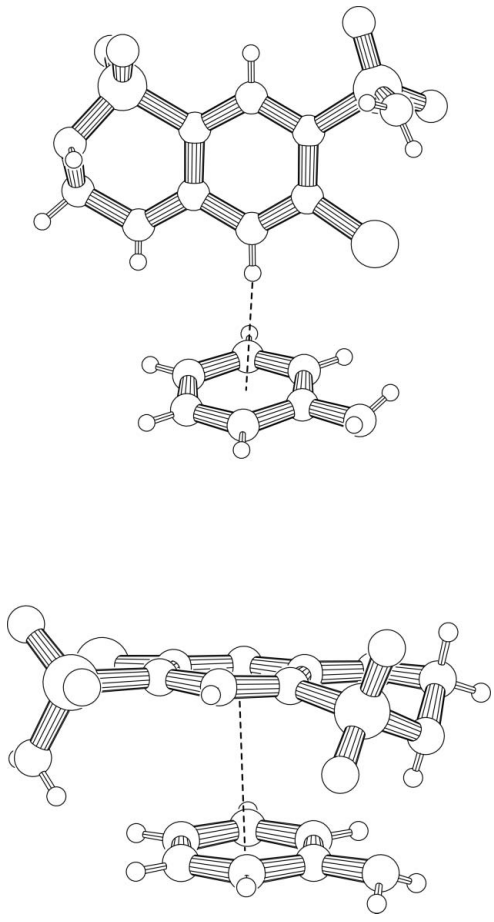


Figure 4
Hydrophobic interactions in (I), showing a C3—H3···centroid contact to the centroid of the benzene ring of aniline [C3···centroid = 3.618 (2) Å] (top) and a π–π off-stacking interaction between HCT and aniline with a centroid–centroid distance of 3.6955 (8) Å (bottom). Contacts are illustrated using dashed lines.

Experimental

A single-crystal sample of the title compound was recrystallized from a 1:1 acetone–aniline solution by slow evaporation at 278 K.

Crystal data

$C_7H_8ClN_3O_4S_2\cdot C_6H_7N$	D_x = 1.621 Mg m^{-3}
M_r = 390.86	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 5824 reflections
a = 9.7757 (3) Å	θ = 1.0–32.0°
b = 10.5004 (3) Å	μ = 0.53 mm^{-1}
c = 15.6093 (4) Å	T = 123 (2) K
β = 91.692 (2)°	Prism, colourless
V = 1601.58 (8) Å^3	0.38 × 0.30 × 0.18 mm
Z = 4	

Data collection

Nonius KappaCCD diffractometer	R_{int} = 0.027
ω and φ scans	θ_{max} = 32.0°
Absorption correction: none	$h = -14 \rightarrow 14$
10766 measured reflections	$k = -15 \rightarrow 15$
5573 independent reflections	$l = -23 \rightarrow 23$
4379 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.084$
$S = 1.02$
5573 reflections
241 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.036P)^2 + 0.781P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.48$ e Å^{-3}
$\Delta\rho_{min} = -0.53$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*···N4	0.87 (2)	2.10 (2)	2.9652 (17)	174 (2)
N3—H3*N*···O2^{i}	0.85 (2)	2.13 (2)	2.9725 (16)	169 (2)
N3—H4*N*···O1^{ii}	0.90 (2)	2.09 (2)	2.9390 (15)	157 (2)
N4—H5*N*···N3^{iii}	0.89 (2)	2.52 (2)	3.3944 (18)	169 (2)
N4—H6*N*···O4^{i}	0.83 (2)	2.28 (2)	3.1000 (17)	173 (2)
C1—H1*A*···O3^{iv}	0.99	2.37	3.2535 (18)	148
C1—H1*B*···O3^{iii}	0.99	2.47	3.2852 (17)	139
C6—H6···O4	0.95	2.38	2.8101 (16)	107

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

The N-bound H atoms were found in difference maps and refined freely [N—H = 0.77 (2)–0.90 (2) Å]. The remaining H atoms were positioned geometrically at distances of 0.95 (CH) and 0.99 Å (CH_2) from the parent C atoms; a riding model was used [U_{iso}(H) = 1.2U_{eq}(C)] during the refinement process.

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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$F(000) = 808$
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$\theta = 1.0$–$32.0°$
$\mu = 0.53$ mm^{-1}
$T = 123$ K
Prism, colourless
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Radiation source: fine-focus sealed tube
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ω and φ scans
10766 measured reflections
5573 independent reflections
4379 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 32.0°$, $\theta_{min} = 2.3°$
$h = -14 \rightarrow 14$
$k = -15 \rightarrow 15$
$l = -23 \rightarrow 23$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.084$
$S = 1.02$
5573 reflections
241 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
$w = 1/[\sigma^2(F_o^2) + (0.036P)^2 + 0.781P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.48$ e Å^{-3}
$\Delta\rho_{min} = -0.53$ 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}
Cl1	0.60656 (4)	0.10198 (3)	0.76024 (2)	0.02108 (8)
S1	0.31003 (3)	0.26967 (3)	1.08865 (2)	0.01544 (7)
S2	0.77750 (3)	0.24797 (3)	0.91533 (2)	0.01472 (7)
O1	0.32719 (11)	0.18921 (11)	1.16255 (6)	0.0239 (2)
O2	0.34691 (11)	0.40187 (10)	1.09743 (7)	0.0236 (2)
O3	0.85848 (10)	0.14419 (10)	0.88491 (6)	0.0208 (2)
O4	0.81363 (10)	0.30354 (10)	0.99740 (6)	0.0201 (2)
N1	0.15186 (12)	0.26228 (12)	1.05407 (7)	0.0174 (2)
N2	0.19235 (13)	0.10009 (12)	0.94740 (8)	0.0197 (2)
N3	0.78744 (12)	0.36009 (12)	0.84548 (7)	0.0169 (2)
N4	0.10950 (14)	0.44391 (13)	0.91027 (8)	0.0213 (2)
C1	0.11769 (14)	0.13361 (14)	1.02355 (9)	0.0186 (3)
H1A	0.1397	0.0714	1.0696	0.022*
H1B	0.0182	0.1286	1.0103	0.022*
C2	0.32626 (13)	0.13305 (13)	0.93952 (8)	0.0154 (2)
C3	0.39693 (14)	0.10032 (13)	0.86510 (8)	0.0168 (2)
H3	0.3510	0.0541	0.8205	0.020*
C4	0.53177 (14)	0.13466 (12)	0.85650 (8)	0.0153 (2)
C5	0.60499 (13)	0.19994 (12)	0.92215 (8)	0.0144 (2)
C6	0.53557 (13)	0.23532 (13)	0.99458 (8)	0.0145 (2)
H6	0.5823	0.2815	1.0389	0.017*
C7	0.39858 (13)	0.20412 (12)	1.00318 (8)	0.0143 (2)
C8	0.19792 (14)	0.41456 (13)	0.84290 (8)	0.0176 (3)
C9	0.15625 (15)	0.33026 (14)	0.77775 (9)	0.0210 (3)
H9	0.0674	0.2936	0.7780	0.025*
C10	0.24467 (17)	0.30030 (15)	0.71291 (9)	0.0253 (3)
H10	0.2160	0.2423	0.6693	0.030*
C11	0.37402 (17)	0.35352 (16)	0.71078 (10)	0.0266 (3)
H11	0.4332	0.3336	0.6655	0.032*
C12	0.41639 (16)	0.43643 (16)	0.77571 (10)	0.0264 (3)
H12	0.5049	0.4736	0.7748	0.032*
C13	0.32954 (15)	0.46523 (14)	0.84205 (10)	0.0219 (3)
H13	0.3603	0.5199	0.8871	0.026*
H1N	0.1400 (19)	0.3198 (19)	1.0149 (12)	0.028 (5)*
H2N	0.152 (2)	0.076 (2)	0.9075 (13)	0.034 (5)*
H3N	0.740 (2)	0.426 (2)	0.8565 (13)	0.038 (6)*
H4N	0.777 (2)	0.334 (2)	0.7909 (13)	0.036 (5)*
H5N	0.023 (2)	0.434 (2)	0.8934 (13)	0.039 (6)*
H6N	0.126 (2)	0.515 (2)	0.9315 (12)	0.030 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.02599 (17)	0.02251 (16)	0.01513 (14)	−0.00209 (13)	0.00727 (11)	−0.00585 (12)
S1	0.01651 (15)	0.01855 (15)	0.01136 (13)	0.00149 (12)	0.00224 (10)	−0.00156 (11)
S2	0.01443 (14)	0.01536 (15)	0.01445 (14)	0.00048 (12)	0.00169 (10)	0.00067 (11)
O1	0.0255 (5)	0.0336 (6)	0.0129 (4)	0.0078 (5)	0.0032 (4)	0.0041 (4)
O2	0.0244 (5)	0.0211 (5)	0.0255 (5)	−0.0013 (4)	0.0048 (4)	−0.0089 (4)
O3	0.0187 (5)	0.0184 (5)	0.0254 (5)	0.0046 (4)	0.0034 (4)	0.0008 (4)
O4	0.0185 (5)	0.0260 (5)	0.0157 (4)	−0.0035 (4)	−0.0012 (4)	−0.0012 (4)
N1	0.0160 (5)	0.0192 (6)	0.0169 (5)	0.0017 (4)	0.0012 (4)	0.0015 (4)
N2	0.0194 (6)	0.0228 (6)	0.0168 (5)	−0.0055 (5)	0.0021 (4)	−0.0043 (5)
N3	0.0201 (6)	0.0151 (5)	0.0157 (5)	0.0008 (4)	0.0045 (4)	0.0015 (4)
N4	0.0229 (6)	0.0213 (6)	0.0196 (6)	0.0003 (5)	0.0012 (5)	0.0001 (5)
C1	0.0182 (6)	0.0193 (6)	0.0186 (6)	−0.0012 (5)	0.0041 (5)	0.0011 (5)
C2	0.0180 (6)	0.0133 (6)	0.0149 (5)	−0.0024 (5)	0.0016 (4)	0.0001 (4)
C3	0.0206 (6)	0.0154 (6)	0.0143 (5)	−0.0034 (5)	0.0017 (5)	−0.0026 (5)
C4	0.0207 (6)	0.0129 (6)	0.0125 (5)	−0.0001 (5)	0.0039 (4)	−0.0014 (4)
C5	0.0155 (6)	0.0141 (6)	0.0136 (5)	−0.0002 (5)	0.0019 (4)	0.0002 (4)
C6	0.0162 (6)	0.0162 (6)	0.0111 (5)	−0.0002 (5)	−0.0002 (4)	−0.0004 (4)
C7	0.0173 (6)	0.0143 (6)	0.0112 (5)	0.0005 (5)	0.0023 (4)	−0.0002 (4)
C8	0.0203 (6)	0.0159 (6)	0.0167 (6)	0.0015 (5)	−0.0016 (5)	0.0034 (5)
C9	0.0232 (7)	0.0188 (7)	0.0208 (6)	−0.0025 (5)	−0.0042 (5)	0.0024 (5)
C10	0.0357 (8)	0.0218 (7)	0.0183 (6)	0.0040 (6)	−0.0042 (6)	−0.0014 (5)
C11	0.0306 (8)	0.0283 (8)	0.0213 (7)	0.0092 (7)	0.0045 (6)	0.0064 (6)
C12	0.0205 (7)	0.0266 (8)	0.0320 (8)	0.0001 (6)	0.0010 (6)	0.0064 (6)
C13	0.0221 (7)	0.0184 (6)	0.0250 (7)	−0.0020 (5)	−0.0037 (5)	0.0001 (5)

Geometric parameters (Å, º)

Cl1—C4	1.7248 (13)	C1—H1B	0.9900
S1—O1	1.4357 (10)	C2—C3	1.4114 (18)
S1—O2	1.4398 (11)	C2—C7	1.4151 (18)
S1—N1	1.6244 (12)	C3—C4	1.3769 (19)
S1—C7	1.7524 (13)	C3—H3	0.9500
S2—O3	1.4359 (10)	C4—C5	1.4103 (18)
S2—O4	1.4421 (10)	C5—C6	1.3864 (17)
S2—N3	1.6095 (12)	C6—C7	1.3890 (18)
S2—C5	1.7665 (13)	C6—H6	0.9500
N1—C1	1.4678 (18)	C8—C13	1.393 (2)
N1—H1N	0.86 (2)	C8—C9	1.3997 (19)
N2—C2	1.3631 (18)	C9—C10	1.386 (2)
N2—C1	1.4564 (17)	C9—H9	0.9500
N2—H2N	0.77 (2)	C10—C11	1.384 (2)
N3—H3N	0.85 (2)	C10—H10	0.9500
N3—H4N	0.90 (2)	C11—C12	1.390 (2)
N4—C8	1.4148 (18)	C11—H11	0.9500
N4—H5N	0.89 (2)	C12—C13	1.392 (2)

N4—H6N	0.83 (2)	C12—H12	0.9500
C1—H1A	0.9900	C13—H13	0.9500

O1—S1—O2	117.90 (7)	C4—C3—C2	120.64 (12)
O1—S1—N1	109.05 (6)	C4—C3—H3	119.7
O2—S1—N1	108.12 (6)	C2—C3—H3	119.7
O1—S1—C7	109.38 (6)	C3—C4—C5	121.55 (12)
O2—S1—C7	108.86 (6)	C3—C4—Cl1	117.67 (10)
N1—S1—C7	102.42 (6)	C5—C4—Cl1	120.70 (10)
O3—S2—O4	118.55 (6)	C6—C5—C4	118.21 (12)
O3—S2—N3	106.63 (6)	C6—C5—S2	117.63 (10)
O4—S2—N3	106.73 (6)	C4—C5—S2	124.05 (10)
O3—S2—C5	109.87 (6)	C5—C6—C7	120.81 (12)
O4—S2—C5	105.74 (6)	C5—C6—H6	119.6
N3—S2—C5	109.06 (6)	C7—C6—H6	119.6
C1—N1—S1	110.92 (9)	C6—C7—C2	121.30 (11)
C1—N1—H1N	112.9 (13)	C6—C7—S1	118.70 (10)
S1—N1—H1N	107.9 (13)	C2—C7—S1	119.60 (10)
C2—N2—C1	121.20 (12)	C13—C8—C9	118.93 (13)
C2—N2—H2N	118.6 (15)	C13—C8—N4	120.58 (13)
C1—N2—H2N	119.0 (15)	C9—C8—N4	120.44 (13)
S2—N3—H3N	114.2 (14)	C10—C9—C8	119.97 (14)
S2—N3—H4N	114.1 (13)	C10—C9—H9	120.0
H3N—N3—H4N	113.3 (19)	C8—C9—H9	120.0
C8—N4—H5N	110.7 (13)	C11—C10—C9	121.05 (14)
C8—N4—H6N	112.1 (14)	C11—C10—H10	119.5
H5N—N4—H6N	113.4 (19)	C9—C10—H10	119.5
N2—C1—N1	111.88 (11)	C10—C11—C12	119.23 (14)
N2—C1—H1A	109.2	C10—C11—H11	120.4
N1—C1—H1A	109.2	C12—C11—H11	120.4
N2—C1—H1B	109.2	C11—C12—C13	120.23 (14)
N1—C1—H1B	109.2	C11—C12—H12	119.9
H1A—C1—H1B	107.9	C13—C12—H12	119.9
N2—C2—C3	120.47 (12)	C12—C13—C8	120.54 (14)
N2—C2—C7	122.12 (12)	C12—C13—H13	119.7
C3—C2—C7	117.38 (12)	C8—C13—H13	119.7

O1—S1—N1—C1	−64.70 (10)	S2—C5—C6—C7	178.11 (10)
O2—S1—N1—C1	165.97 (9)	C5—C6—C7—C2	1.4 (2)
C7—S1—N1—C1	51.12 (10)	C5—C6—C7—S1	−171.34 (10)
C2—N2—C1—N1	40.07 (18)	N2—C2—C7—C6	179.10 (13)
S1—N1—C1—N2	−66.52 (13)	C3—C2—C7—C6	−2.93 (19)
C1—N2—C2—C3	−179.71 (13)	N2—C2—C7—S1	−8.21 (18)
C1—N2—C2—C7	−1.8 (2)	C3—C2—C7—S1	169.76 (10)
N2—C2—C3—C4	179.28 (13)	O1—S1—C7—C6	−87.71 (12)
C7—C2—C3—C4	1.3 (2)	O2—S1—C7—C6	42.40 (12)
C2—C3—C4—C5	1.9 (2)	N1—S1—C7—C6	156.72 (11)
C2—C3—C4—Cl1	−175.03 (10)	O1—S1—C7—C2	99.41 (11)

C3—C4—C5—C6	−3.4 (2)	O2—S1—C7—C2	−130.48 (11)
Cl1—C4—C5—C6	173.40 (10)	N1—S1—C7—C2	−16.17 (12)
C3—C4—C5—S2	−179.53 (11)	C13—C8—C9—C10	−1.2 (2)
Cl1—C4—C5—S2	−2.69 (17)	N4—C8—C9—C10	−178.78 (13)
O3—S2—C5—C6	136.39 (11)	C8—C9—C10—C11	−0.7 (2)
O4—S2—C5—C6	7.36 (12)	C9—C10—C11—C12	1.2 (2)
N3—S2—C5—C6	−107.07 (11)	C10—C11—C12—C13	0.1 (2)
O3—S2—C5—C4	−47.49 (13)	C11—C12—C13—C8	−2.0 (2)
O4—S2—C5—C4	−176.52 (11)	C9—C8—C13—C12	2.5 (2)
N3—S2—C5—C4	69.04 (13)	N4—C8—C13—C12	−179.93 (13)
C4—C5—C6—C7	1.8 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···N4	0.865 (19)	2.104 (19)	2.9652 (17)	174.0 (18)
N3—H3*N*···O2^{i}	0.85 (2)	2.13 (2)	2.9725 (16)	168.6 (18)
N3—H4*N*···O1^{ii}	0.90 (2)	2.09 (2)	2.9390 (15)	157.1 (17)
N4—H5*N*···N3^{iii}	0.89 (2)	2.52 (2)	3.3944 (18)	168.6 (18)
N4—H6*N*···O4^{i}	0.83 (2)	2.28 (2)	3.1000 (17)	173.1 (17)
C1—H1*A*···O3^{iv}	0.99	2.37	3.2535 (18)	148
C1—H1*B*···O3^{iii}	0.99	2.47	3.2852 (17)	139
C6—H6···O4	0.95	2.38	2.8101 (16)	107

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