organic papers

Powder study of hydrochlorothiazide–methyl acetate (1/1)

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A polycrystalline sample of the title compound, $C_7H_8ClN_3O_4S_2 \cdot C_3H_6O_2$, was produced during an automated parallel crystallization search on hydrochlorothiazide (HCT). The crystal structure was solved by simulated annealing from laboratory X-ray powder diffraction data collected at room temperature to 1.75 Å resolution. Subsequent Rietveld refinement yielded an R_{wp} value of 0.0182 to 1.54 Å resolution. The compound crystallizes with one molecule of HCT and one of methyl acetate 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). During an automated parallel crystallization search on HCT, multi-sample X-ray powder diffraction analysis (Florence *et al.*, 2003) of all recrystallized samples revealed a novel pattern which was identified as the methyl acetate solvate (I).

(I)
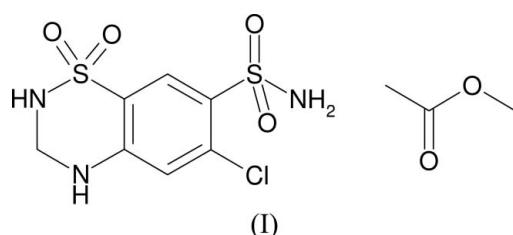
The crystal structure of (I) was solved by simulated annealing using laboratory X-ray powder diffraction data. The compound crystallizes in the space group $P2_1/c$ with one molecule of hydrochlorothiazide (HCT) and one of methyl acetate in the asymmetric unit (Fig. 1). In (I), the six-membered ring N2/S1/C1/C2/N1/C3 in HCT displays a non-planar conformation, atom N2 having the largest deviation [0.646 (2) Å] from the least-squares plane through the aromatic ring. The sulfonamide side chain displays a torsion angle N3—S2—C5—C6 of 65.1 (4)°, such that atom O1 eclipses H4, and atoms O4 and N3 are staggered with respect to Cl1.

The crystal packing (Fig. 2) is stabilized by intermolecular N—H···O hydrogen bonds (Table 2), which form a centrosymmetric $R_2^2(8)$ dimer motif between HCT molecules (Fig. 3, top), and an $R_4^4(24)$ motif interconnecting two molecules of HCT and two molecules of solvent (Fig. 3, bottom). In addition, adjacent HCT molecules are connected by a C—H···O interaction (Table 2).
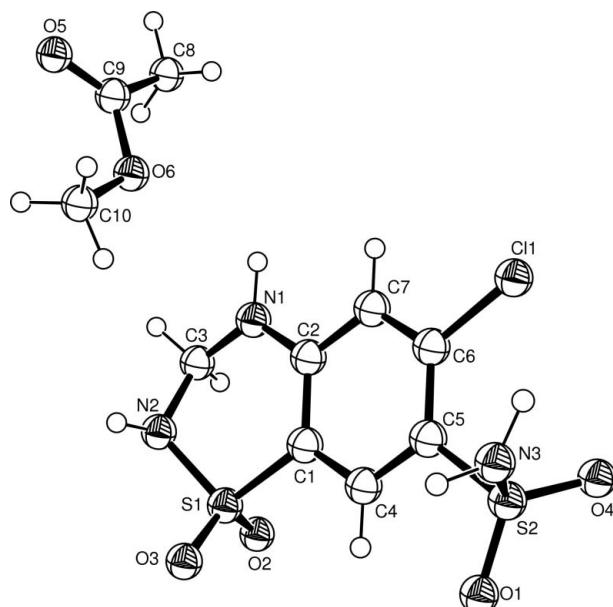
Figure 1
The atomic arrangement in (I), showing the contents of the asymmetric unit and the atom-numbering scheme. Isotropic displacement ellipsoids are shown at the 50% probability level.
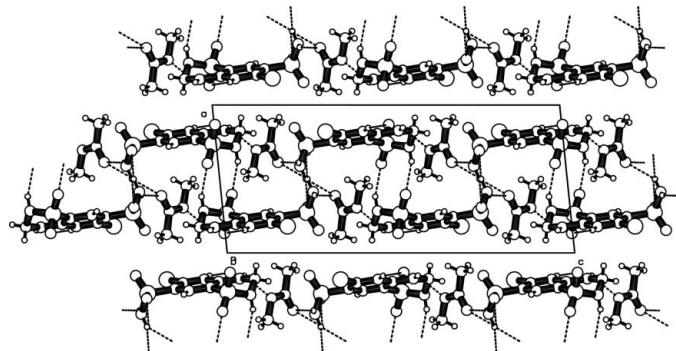

Figure 2
The crystal structure of (I), viewed along the *b* axis. Dashed lines indicate hydrogen bonds.
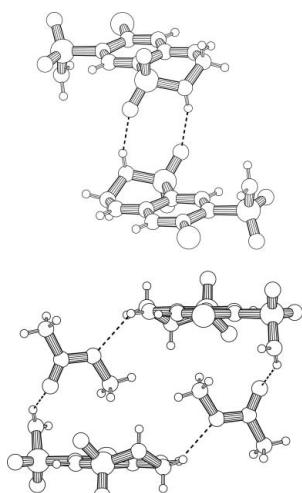

Figure 3
The $R_2^2(8)$ (top) and $R_4^4(24)$ (bottom) hydrogen-bonded motifs within the structure of (I).

Hydrophobic interactions within the structure of (I) include a C—H···π approach between C10—H10*A* and the centroid of the ring C1/C2/C4–C7 [C10···centroid distance of 3.364 (2) Å]. The structure also contains a short O···C intermolecular contact of 2.915 (4) Å between atom O1 of the HCT sulfonamide side chain and C9^{i} [symmetry code: (i) $x, \frac{3}{2} - y, -\frac{1}{2} + z$], the carbonyl C atom of methyl acetate. This type of contact is not unique, and a search of the Cambridge Structural Database (Version 5.26; Allen, 2002) for (O)S=O···C=O(ester) intermolecular contacts less than the sum of the van der Waals radii yielded 38 structures comprising 41 contacts within the range 2.83–3.21 Å. It is reasonable to consider this contact to be an attractive dipole–dipole interaction of the type S=O(δ^-)···C(δ^+)=O, similar to those described elsewhere for carbonyl–carbonyl interactions (Allen *et al.*, 1998).

Experimental

A polycrystalline sample of (I) was recrystallized by cooling a saturated methyl acetate–acetone (50:50) solution from 313 to 283 K. The sample was lightly ground in a mortar, loaded into a 0.7 mm borosilicate glass capillary and mounted on the diffractometer. Data were collected from a sample in a rotating 0.7 mm borosilicate glass capillary using a variable count time scheme (Shankland *et al.*, 1997; Hill & Madsen, 2002).

Crystal data
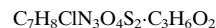
$C_7H_8ClN_3O_4S_2 \cdot C_3H_6O_2$	$D_x = 1.652$ Mg m^{-3}
$M_r = 371.83$	Cu $K\alpha_1$ radiation
Monoclinic, $P2_1/c$	$\mu = 5.20$ mm^{-1}
$a = 9.39703$ (16) Å	$T = 298$ K
$b = 7.28424$ (16) Å	Specimen shape: cylinder
$c = 21.9483$ (3) Å	$12 \times 0.7 \times 0.7$ mm
$\beta = 95.8020$ (11)°	Specimen prepared at 298 K
$V = 1494.67$ (6) Å^3	White
$Z = 4$	

Data collection

Bruker AXS D8 Advance diffractometer	430 measured reflections
Specimen mounting: 0.7 mm borosilicate capillary	$h = 0 \rightarrow 6$
	$k = -4 \rightarrow 4$
Specimen mounted in transmission mode	$l = -14 \rightarrow 14$
	$2\theta_{min} = 5.0, 2\theta_{max} = 60.0°$
Scan method: step	Increment in $2\theta = 0.014°$

Refinement

|	
Refinement on I_{net}	430 reflections
$R_p = 0.013$	126 parameters
$R_{wp} = 0.018$	Only H-atom coordinates refined
$R_{exp} = 0.013$	$w = 1/\sigma(Y_{obs})^2$
$R_B = 0.007$	$(\Delta/\sigma)_{max} = 0.004$
$S = 1.45$	$\Delta\rho_{max} = 0.6$ e Å^{-3}
Excluded region(s): 5.0 to 7.5 due to high low angle background and the absence of Bragg reflections.	$\Delta\rho_{min} = -0.7$ e Å^{-3}
Wavelength of incident radiation: 1.5406 Å	Preferred orientation correction: A spherical harmonics-based preferred orientation correction (Järvinen, 1993) was applied with *TOPAS* during the Rietveld refinement.
Profile function: fundamental parameters with axial divergence correction	
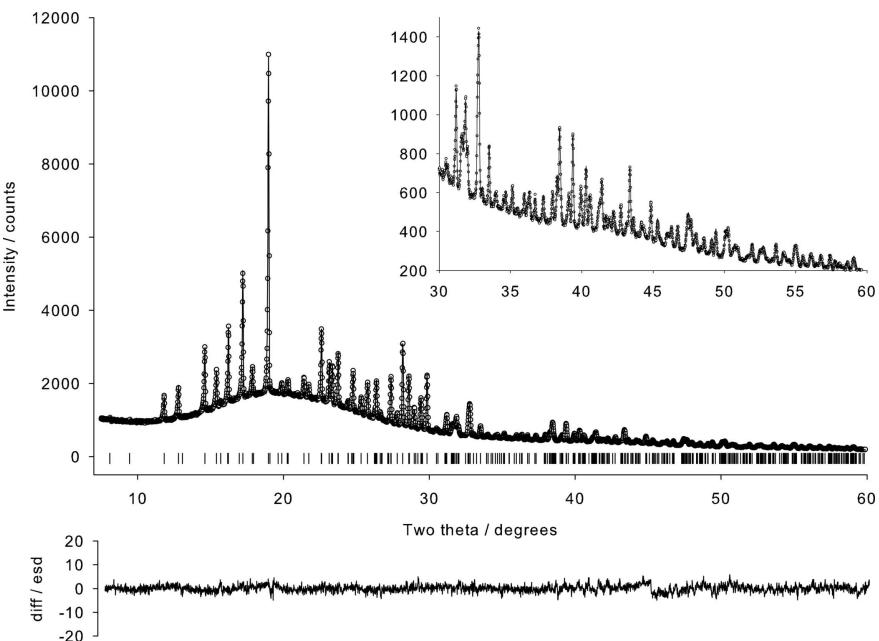
Figure 4
Final observed (points), calculated (line) and difference $[(y_{obs}-y_{calc})/\sigma(y_{obs})]$ profiles for the Rietveld refinement of the title compound.

Table 1
Selected geometric parameters (Å, °).

Cl1—C6	1.729 (3)	S2—C5	1.773 (2)
S1—O2	1.426 (3)	O5—C9	1.229 (3)
S1—O3	1.424 (3)	O6—C9	1.379 (3)
S1—N2	1.647 (2)	O6—C10	1.432 (3)
S1—C1	1.770 (2)	N1—C2	1.364 (2)
S2—O1	1.430 (3)	N1—C3	1.395 (2)
S2—O4	1.425 (4)	N2—C3	1.442 (2)
S2—N3	1.635 (2)		
O2—S1—O3	122.3 (2)	S1—N2—C3	114.00 (13)
O2—S1—N2	105.1 (2)	S1—C1—C4	119.05 (13)
O2—S1—C1	108.82 (19)	S1—C1—C2	119.79 (11)
O3—S1—N2	108.26 (19)	N1—C2—C7	120.31 (16)
O3—S1—C1	109.26 (19)	N1—C2—C1	121.89 (15)
N2—S1—C1	100.99 (11)	N1—C3—N2	112.9 (2)
O1—S2—O4	117.9 (3)	S2—C5—C4	117.37 (16)
O1—S2—N3	109.7 (3)	S2—C5—C6	124.82 (16)
O1—S2—C5	106.0 (2)	Cl1—C6—C7	116.92 (19)
O4—S2—N3	108.3 (2)	Cl1—C6—C5	121.60 (16)
O4—S2—C5	108.99 (19)	O5—C9—O6	117.66 (19)
N3—S2—C5	105.25 (16)	O5—C9—C8	129.20 (18)
C9—O6—C10	117.19 (17)	O6—C9—C8	113.14 (14)
C2—N1—C3	123.80 (15)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O6	0.94 (1)	2.48 (1)	3.052 (4)	119 (1)
N2—H2···O3^i	0.95 (1)	2.17 (1)	3.086 (3)	161 (1)
N3—H31···O1^{ii}	0.95 (1)	2.46 (1)	2.967 (6)	113 (1)
N3—H31···O5^{iii}	0.95 (1)	2.26 (1)	3.090 (3)	146 (1)
N3—H32···O5^{iv}	0.95 (1)	2.12 (1)	2.960 (4)	147 (1)
C7—H7···O2^v	0.95 (1)	2.41 (1)	3.336 (4)	165 (1)

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

The diffraction pattern indexed to a monoclinic cell [$M(20)$= 34.0, $F(20)$= 81.2; *DICVOL-91*; Boultif & Louer, 1991] and the space group $P2_1/c$ was assigned from volume considerations and a statistical consideration of the systematic absences (Markvardsen *et al.*, 2001). The data set was background subtracted and truncated to 52.2° 2θ for Pawley fitting (Pawley, 1981; χ^2_{Pawley} = 1.64) and the structure solved using the simulated annealing (SA) global optimization procedure, described previously (David *et al.*, 1998), that is now implemented in the *DASH* computer program (David *et al.*, 2001). The internal coordinate descriptions (including H atoms) of the molecules were constructed from standard bond lengths, angles and torsions where appropriate. The structure was solved using data to 52.23° 2θ, comprising 291 reflections. The structure was refined against data in the range 7.5 to 60.0° 2θ (430 reflections). The restraints were set such that bonds and angles did not deviate more than 0.01 Å and 1°, respectively, from their initial values during the refinement. Atoms C1, C2, C4, C5, C6, C7, H4, H7, Cl1 and S2 of HCT were restrained to be planar, as were atoms C8, C9, C10, O5 and O6 of the methyl acetate. The SA structure solution involved the optimization of one molecule of HCT plus one molecule of methyl acetate, totaling 13 degrees of freedom (six positional, six orientational and one torsional). All degrees of freedom were assigned random values at the start of the simulated annealing. The best SA solution had a favourable $\chi^2_{SA}/\chi^2_{Pawley}$ ratio of 5.2, a chemically reasonable packing arrangement and exhibited no significant misfit to the data. Prior to Rietveld refinement, atoms H31 and H32 (attached to N3) were set to positions which satisfied the hydrogen-bonding contacts within the structure. The solved structure was subsequently refined against data in the range 7.5–60.0° 2θ using a restrained Rietveld method (Rietveld, 1969) as implemented in *TOPAS* (Coelho, 2003), with the R_{wp} value falling to 0.0182 during the refinement. All atomic positions (including H atoms) for the structure of (I) were refined, subject to a series of restraints on bond lengths, angles and planarity. A spherical harmonics (8th order) correction of intensities for preferred orientation was applied in the final refinement (Järvinen, 1993). The observed and calculated diffraction patterns for the refined crystal structure are shown in Fig. 4. The atomic coordinates for all atoms are

taken from the software, and it is worth noting that the s.u. values derived from the Rietveld refinement are, in common with the majority of Rietveld refinements, significantly underestimated.

Data collection: *DIFFRAC* plus *XRD Commander* (Kienle & Jacob, 2003); cell refinement: *TOPAS* (Coelho, 2003); data reduction: *DASH* (David *et al.*, 2001); program(s) used to solve structure: *DASH*; program(s) used to refine structure: *TOPAS*; molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *enCIFer* (Cambridge Crystallographic Data Centre, 2004).

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supporting information

Powder study of hydrochlorothiazide–methyl acetate (1/1)

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(I)

Crystal data
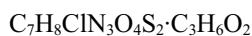
$C_7H_8ClN_3O_4S_2 \cdot C_3H_6O_2$	$Z = 4$
$M_r = 371.83$	$F(000) = 768.0$
Monoclinic, $P2_1/c$	$D_x = 1.652$ Mg m^{-3}
Hall symbol: -P 2ybc	Cu $K\alpha_1$ radiation, $\lambda = 1.54056$ Å
$a = 9.39703$ (16) Å	$\mu = 5.20$ mm^{-1}
$b = 7.28424$ (16) Å	$T = 298$ K
$c = 21.9483$ (3) Å	white
$\beta = 95.8020$ (11)°	cylinder, 12×0.7 mm
$V = 1494.67$ (6) Å^3	Specimen preparation: Prepared at 298 K

Data collection

Bruker AXS D8 Advance diffractometer	Specimen mounting: 0.7 mm borosilicate capillary
Radiation source: sealed X-ray tube, Bruker AXS D8	Data collection mode: transmission
	Scan method: step
Primary focussing, Ge 111 monochromator	$2\theta_{min} = 5.0°$, $2\theta_{max} = 60.0°$, $2\theta_{step} = 0.014°$

Refinement

Refinement on I_{net}	126 parameters
Least-squares matrix: selected elements only	72 restraints
$R_p = 0.013$	1 constraint
$R_{wp} = 0.018$	Only H-atom coordinates refined
$R_{exp} = 0.013$	Weighting scheme based on measured s.u.'s $1/\sigma(Y_{obs})^2$
$R_{Bragg} = 0.007$	
3929 data points	$(\Delta/\sigma)_{max} = 0.004$
Excluded region(s): 5.0 to 7.5 due to high low angle background and the absence of Bragg reflections.	Background function: Chebyshev polynomial
	Preferred orientation correction: A spherical harmonics-based preferred orientation correction (Järvinen, 1993) was applied with Topas during the Rietveld refinement.
Profile function: Fundamental parameters with axial divergence correction	

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

	x	y	z	$U_{iso}*/U_{eq}$
Cl1	0.8195 (3)	0.3882 (3)	−0.18148 (9)	0.0329 (5)*
S1	0.73715 (15)	1.05531 (19)	−0.00965 (7)	0.0329 (5)*
S2	0.72621 (16)	0.79646 (19)	−0.24031 (7)	0.0329 (5)*
O1	0.6906 (7)	0.9871 (3)	−0.24511 (19)	0.0329 (5)*
O2	0.8672 (3)	1.1557 (5)	0.00231 (19)	0.0329 (5)*

O3	0.6053 (3)	1.1419 (4)	−0.0305 (2)	0.0329 (5)*
O4	0.8458 (3)	0.7336 (7)	−0.26960 (15)	0.0329 (5)*
N1	0.8265 (6)	0.6705 (2)	0.02849 (8)	0.0329 (5)*
N2	0.71647 (17)	0.9472 (3)	0.05475 (8)	0.0329 (5)*
N3	0.58695 (16)	0.6729 (2)	−0.26508 (15)	0.0329 (5)*
C1	0.7665 (3)	0.8685 (2)	−0.05842 (6)	0.0329 (5)*
C2	0.8055 (5)	0.6976 (2)	−0.03324 (6)	0.0329 (5)*
C3	0.82476 (17)	0.8111 (2)	0.07153 (8)	0.0329 (5)*
C4	0.7449 (4)	0.8932 (2)	−0.12102 (7)	0.0329 (5)*
C5	0.7565 (3)	0.7489 (3)	−0.16085 (8)	0.0329 (5)*
C6	0.7971 (6)	0.5770 (3)	−0.13567 (9)	0.0329 (5)*
C7	0.8207 (4)	0.5514 (2)	−0.07365 (6)	0.0329 (5)*
H3A	0.8089 (12)	0.7606 (14)	0.1100 (5)	0.0443*
H3B	0.9134 (11)	0.8732 (16)	0.0795 (4)	0.0443*
H7	0.8452 (13)	0.4329 (15)	−0.0578 (5)	0.0443*
H4	0.7194 (16)	1.0106 (13)	−0.1369 (6)	0.0443*
H2	0.6190 (12)	0.9181 (15)	0.0575 (5)	0.0443*
H1	0.8462 (12)	0.5493 (16)	0.0418 (5)	0.0443*
H31	0.5006 (13)	0.7193 (15)	−0.2523 (4)	0.0443*
H32	0.6101 (12)	0.5465 (17)	−0.2687 (5)	0.0443*
C8	0.87362 (18)	0.21591 (19)	0.16847 (7)	0.0329 (5)*
C9	0.71849 (18)	0.2591 (2)	0.15411 (8)	0.0329 (5)*
C10	0.54815 (18)	0.4566 (2)	0.09788 (8)	0.0329 (5)*
O5	0.6140 (2)	0.1839 (4)	0.17260 (13)	0.0329 (5)*
O6	0.69391 (19)	0.4044 (3)	0.11414 (10)	0.0329 (5)*
H8A	0.8818 (12)	0.1090 (16)	0.1947 (5)	0.0443*
H8B	0.9172 (12)	0.3172 (16)	0.1893 (5)	0.0443*
H8C	0.9118 (13)	0.1916 (15)	0.1315 (5)	0.0443*
H10A	0.5049 (13)	0.4878 (13)	0.1326 (5)	0.0443*
H10B	0.5005 (13)	0.3589 (16)	0.0758 (5)	0.0443*
H10C	0.5494 (13)	0.5610 (16)	0.0707 (5)	0.0443*

Geometric parameters (Å, º)

Cl1—C6	1.729 (3)	N3—H31	0.947 (12)
S1—O2	1.426 (3)	C1—C4	1.380 (2)
S1—O3	1.424 (3)	C1—C2	1.396 (2)
S1—N2	1.647 (2)	C2—C7	1.402 (2)
S1—C1	1.770 (2)	C4—C5	1.379 (3)
S2—O1	1.430 (3)	C5—C6	1.406 (3)
S2—O4	1.425 (4)	C6—C7	1.370 (2)
S2—N3	1.635 (2)	C3—H3A	0.947 (11)
S2—C5	1.773 (2)	C3—H3B	0.949 (11)
O5—C9	1.229 (3)	C4—H4	0.945 (10)
O6—C9	1.379 (3)	C7—H7	0.950 (11)
O6—C10	1.432 (3)	C8—C9	1.494 (2)
N1—C2	1.364 (2)	C8—H8A	0.967 (11)
N1—C3	1.395 (2)	C8—H8B	0.940 (12)

N2—C3	1.442 (2)	C8—H8C	0.937 (11)
N1—H1	0.942 (12)	C10—H10A	0.927 (11)
N2—H2	0.948 (11)	C10—H10B	0.948 (12)
N3—H32	0.951 (12)	C10—H10C	0.967 (12)
O2—S1—O3	122.3 (2)	S2—C5—C6	124.82 (16)
O2—S1—N2	105.1 (2)	C4—C5—C6	117.75 (16)
O2—S1—C1	108.82 (19)	Cl1—C6—C7	116.92 (19)
O3—S1—N2	108.26 (19)	C5—C6—C7	121.48 (19)
O3—S1—C1	109.26 (19)	Cl1—C6—C5	121.60 (16)
N2—S1—C1	100.99 (11)	C2—C7—C6	120.57 (17)
O1—S2—O4	117.9 (3)	N1—C3—H3A	109.4 (6)
O1—S2—N3	109.7 (3)	N1—C3—H3B	113.9 (7)
O1—S2—C5	106.0 (2)	N2—C3—H3B	107.7 (7)
O4—S2—N3	108.3 (2)	H3A—C3—H3B	103.6 (9)
O4—S2—C5	108.99 (19)	N2—C3—H3A	108.9 (7)
N3—S2—C5	105.25 (16)	C1—C4—H4	119.4 (8)
C9—O6—C10	117.19 (17)	C5—C4—H4	119.3 (8)
C2—N1—C3	123.80 (15)	C2—C7—H7	119.7 (7)
S1—N2—C3	114.00 (13)	C6—C7—H7	119.8 (7)
C3—N1—H1	119.6 (7)	O5—C9—O6	117.66 (19)
C2—N1—H1	116.6 (7)	O5—C9—C8	129.20 (18)
C3—N2—H2	119.4 (7)	O6—C9—C8	113.14 (14)
S1—N2—H2	111.1 (7)	C9—C8—H8A	108.2 (7)
S2—N3—H31	112.7 (7)	C9—C8—H8B	107.8 (7)
H31—N3—H32	125.5 (10)	C9—C8—H8C	108.0 (7)
S2—N3—H32	112.2 (7)	H8A—C8—H8B	109.8 (10)
C2—C1—C4	121.11 (14)	H8A—C8—H8C	110.4 (10)
S1—C1—C4	119.05 (13)	H8B—C8—H8C	112.6 (10)
S1—C1—C2	119.79 (11)	O6—C10—H10A	110.4 (7)
C1—C2—C7	117.79 (12)	O6—C10—H10B	108.6 (8)
N1—C2—C7	120.31 (16)	O6—C10—H10C	107.0 (7)
N1—C2—C1	121.89 (15)	H10A—C10—H10B	112.4 (10)
N1—C3—N2	112.9 (2)	H10A—C10—H10C	110.4 (9)
C1—C4—C5	121.25 (16)	H10B—C10—H10C	108.0 (10)
S2—C5—C4	117.37 (16)		
O2—S1—N2—C3	−65.2 (2)	C2—N1—C3—N2	38.8 (6)
O3—S1—N2—C3	162.64 (19)	S1—N2—C3—N1	−61.7 (3)
C1—S1—N2—C3	47.94 (18)	C2—C1—C4—C5	2.1 (5)
O2—S1—C1—C2	92.7 (3)	S1—C1—C2—N1	−2.1 (6)
O2—S1—C1—C4	−89.8 (3)	S1—C1—C4—C5	−175.4 (3)
O3—S1—C1—C2	−131.6 (3)	C4—C1—C2—C7	−0.6 (6)
O3—S1—C1—C4	46.0 (3)	S1—C1—C2—C7	176.9 (3)
N2—S1—C1—C2	−17.6 (3)	C4—C1—C2—N1	−179.6 (4)
N2—S1—C1—C4	159.9 (2)	C1—C2—C7—C6	−0.3 (6)
O1—S2—C5—C4	−1.5 (4)	N1—C2—C7—C6	178.8 (5)
O1—S2—C5—C6	−178.7 (4)	C1—C4—C5—C6	−2.7 (5)

O4—S2—C5—C4	126.4 (3)	C1—C4—C5—S2	179.9 (3)
O4—S2—C5—C6	−50.9 (4)	S2—C5—C6—Cl1	−1.0 (6)
N3—S2—C5—C4	−117.7 (3)	S2—C5—C6—C7	179.0 (3)
N3—S2—C5—C6	65.1 (4)	C4—C5—C6—Cl1	−178.3 (3)
C10—O6—C9—C8	−179.70 (16)	C4—C5—C6—C7	1.8 (6)
C10—O6—C9—O5	0.5 (3)	Cl1—C6—C7—C2	179.7 (4)
C3—N1—C2—C1	−6.3 (8)	C5—C6—C7—C2	−0.3 (7)
C3—N1—C2—C7	174.7 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O6	0.94 (1)	2.48 (1)	3.052 (4)	119 (1)
N2—H2···O3[i]	0.95 (1)	2.18 (1)	3.086 (3)	161 (1)
N3—H31···O1[ii]	0.95 (1)	2.46 (1)	2.967 (6)	113 (1)
N3—H31···O5[iii]	0.95 (1)	2.26 (1)	3.090 (3)	146 (1)
N3—H32···O5[iv]	0.95 (1)	2.12 (1)	2.960 (4)	147 (1)
C7—H7···O2[v]	0.95 (1)	2.41 (1)	3.336 (4)	165 (1)

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