Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Chlorothiazide *N*,*N*-dimethylacetamide disolvate

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

[a]Solid-State Research Group, Strathclyde Institute of Pharmacy and Biomedical Sciences, The John Arbuthnott Building, University of Strathclyde, 27 Taylor Street, Glasgow G4 0NR, Scotland, and [b]WestCHEM, Department of Pure and Applied Chemistry, University of Strathclyde, 295 Cathedral Street, Glasgow G1 1XL, Scotland
Correspondence e-mail: alastair.florence@strath.ac.uk

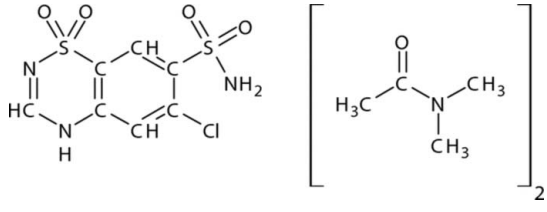
Received 28 March 2007; accepted 3 April 2007

Key indicators: single-crystal X-ray study; T = 123 K; mean σ(C–C) = 0.004 Å; disorder in solvent or counterion; R factor = 0.044; wR factor = 0.098; data-to-parameter ratio = 12.9.

Chlorothiazide forms a 1:2 solvate with *N*,*N*-dimethylacetamide (systematic name: 6-chloro-4*H*-1,2,4-benzothiadiazine-7-sulfonamide 1,1-dioxide *N*,*N*-dimethylacetamide disolvate), $C_7H_6ClN_3O_4S_2{\cdot}2C_4H_9NO$. The compound crystallizes with one chlorothiazide and two solvent molecules in the asymmetric unit, forming three intermolecular N—H···O hydrogen bonds.

Related literature

For details on experimental methods used to obtain this form, see: Florence *et al.* (2003, 2006). For previous studies on the anhydrous form of the title compound, see: Dupont & Dideberg (1970) and Shankland *et al.* (1997). Intermolecular interactions in polymorphs and solvates of the related thiazide diuretic hydrochlorothiazide have also been studied (Johnston *et al.*, 2007).



Experimental

Crystal data

$C_7H_6ClN_3O_4S_2{\cdot}2C_4H_9NO$
M_r = 469.96
Monoclinic, $P2_1/n$
a = 8.2037 (3) Å
b = 24.5226 (6) Å
c = 10.6443 (3) Å
β = 105.231 (2)°
V = 2066.16 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.43 mm^{-1}
T = 123 (2) K
0.25 × 0.12 × 0.06 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Absorption correction: none
15173 measured reflections
4061 independent reflections
2579 reflections with $I > 2\sigma(I)$
R_{int} = 0.094

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.098
S = 1.02
4061 reflections
315 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3N···O5	0.87 (3)	2.06 (3)	2.909 (4)	168 (3)
N3—H2N···O6^{i}	0.89 (3)	2.05 (3)	2.906 (14)	159 (3)
N2—H1N···O5^{ii}	0.79 (3)	1.98 (3)	2.759 (3)	170 (3)
N3—H2N···O7^{i}	0.89 (3)	1.74 (4)	2.628 (18)	171 (3)

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

Data collection: *COLLECT* (Nonius, 1998) 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) and *ORTEP-3* (Farrugia, 1997); 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 (http://www.cposs.org.uk).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2264).

References

Dupont, L. & Dideberg, O. (1970). *Acta Cryst.* B**26**, 1884–1885.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Florence, A. J., Baumgartner, B., Weston, C., Shankland, N., Kennedy, A. R., Shankland, K. & David, W. I. F. (2003). *J. Pharm. Sci.* **92**, 1930–1938.
Florence, A. J., Johnston, A., Fernandes, P., Shankland, N. & Shankland, K. (2006). *J. Appl. Cryst.* **39**, 922–924.
Johnston, A., Florence, A. J., Shankland, N., Kennedy, A. R., Shankland, K. & Price, S. L. (2007). *Cryst. Growth Des.* **7**, 705–712.
Nonius (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Shankland, K., David, W. I. F. & Sivia, D. S. (1997). *J. Mater. Chem.* **7**, 569–572.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

 © 2007 International Union of Crystallography doi:10.1107/S1600536807016388

supplementary materials

Acta Cryst. (2007). E**63**, o2422 [**doi:10.1107/S1600536807016388**]

Chlorothiazide *N*,*N*-dimethylacetamide disolvate

A. Johnston, A. J. Florence, P. Fernandes and A. R. Kennedy

Comment

Chlorothiazide (CT) is a thiazide diuretic drug that is known to crystallize in at least one non-solvated form (Dupont & Dideberg, 1970; Shankland *et al.*, 1997). The title compound, (I), was produced as part of an automated parallel crystallization study (Florence *et al.*, 2006) of CT as part of a wider investigation that couples automated parallel crystallization with crystal structure prediction methodology to investigate the basic science underlying the solid-state diversity of CT and the related thiazide diuretic, hydrochlorothiazide (Johnston *et al.*, 2007). The sample was identified as a novel form using multi-sample foil transmission X-ray powder diffraction analysis (Florence *et al.*, 2003). Subsequent manual recrystallization from a saturated *N,N*-dimethylacetamide solution (DMA) by slow evaporation at 278 K yielded samples of (I) suitable for single-crystal X-ray diffraction (Fig. 1).

Compound (I) crystallizes with one CT and two DMA molecules in the asymmetric unit, with one of the solvent molecules (residue C) disordered over two sites.

The structure contains three N—H···O bonds (Table 1), with all available hydrogen-bond donors in CT (N3—H3N, N3—H2N and N2—H1N) forming contacts to adjacent acetyl O-atoms of DMA. Contacts 1 and 3 combine to form an $R_2^4(20)$ motif (Etter, 1990) between CT and DMA residue B, whilst contact 2 connects DMA residue C to CT (Fig. 2).

Experimental

A single-crystal sample of the title compound, (I), was recrystallized from a saturated dimethylacetamide solution by isothermal solvent evaporation at 278 K.

Refinement

The three amine H atoms were found by difference synthesis and refined freely. All other H atoms were constrained to idealised geometry using riding models, with C—H = 0.98 Å and $U_{iso}(H) = 1.5U_{eq}(C)$ for CH_3 groups, and C—H =

0.95 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ for CH groups. One solvent molecule was modelled as disordered over two positions [occupancies refined to 0.58 (2):0.42 (2)] with shared C12 and C15 sites.

Figures

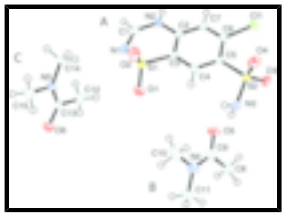


Fig. 1. The asymmetric unit of (I), showing 50% probablility displacement ellipsoids. Minor occupancy disordered atomic sites (residue C) have been omitted for clarity.

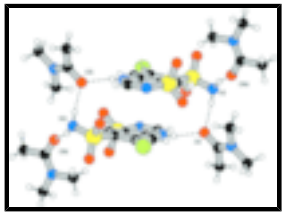


Fig. 2. The packing motif of (I), showing the centrosymmetric $R_2^4(20)$ motif between CT and DMA (residue B) molecules, as well as the N—H···O contact between CT and disordered DMA residue C. Hydrogen bonds are shown as dashed lines and minor disorder components have been omitted for clarity. [Symmetry codes:(i) 1 - *x*, 2 - *y*, 1 - *z*; (ii) 1/2 - *x*, 1/2 + *y*, 3/2 - *z*; (iii) 1/2 + *x*, 3/2 - *y*, -1/2 + *z*.]

6-chloro-2*H*-1,2,4-benzothiadiazine-7-sulfonamide 1,1-dioxide *N*,*N*-dimethylacetamide disolvate

Crystal data

$C_7H_6ClN_3O_4S_2·2C_4H_9NO$	F_{000} = 984
M_r = 469.96	D_x = 1.511 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 8879 reflections
a = 8.2037 (3) Å	θ = 1.0–27.1º
b = 24.5226 (6) Å	μ = 0.43 mm^{-1}
c = 10.6443 (3) Å	*T* = 123 (2) K
β = 105.231 (2)º	Bar, colourless
V = 2066.16 (11) Å^3	0.25 × 0.12 × 0.06 mm
Z = 4	

Data collection

Nonius Kappa CCD area-detector diffractometer	2579 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.094
Monochromator: graphite	θ_{max} = 26.0º
T = 123(2) K	θ_{min} = 1.7º
φ and ω scans	*h* = −9→10
Absorption correction: none	*k* = −30→30
15173 measured reflections	*l* = −13→13
4061 independent reflections	

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.044$

$wR(F^2) = 0.098$

$S = 1.02$

4061 reflections

315 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

$w = 1/[\sigma^2(F_o^2) + (0.0359P)^2 + 1.0897P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.002$

$\Delta\rho_{max} = 0.34$ e Å^{-3}

$\Delta\rho_{min} = -0.39$ e Å^{-3}

Extinction correction: none

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Cl1	0.86503 (9)	1.06818 (3)	0.36097 (7)	0.0287 (2)	
S1	0.62335 (10)	0.87377 (3)	0.64178 (7)	0.0265 (2)	
S2	0.75667 (9)	0.95507 (3)	0.20371 (7)	0.0266 (2)	
O1	0.4740 (3)	0.84724 (8)	0.56543 (18)	0.0371 (6)	
O2	0.7733 (3)	0.84094 (8)	0.68134 (19)	0.0378 (6)	
O3	0.6779 (2)	0.99953 (8)	0.12360 (17)	0.0321 (5)	
O4	0.9323 (2)	0.94450 (9)	0.21843 (18)	0.0364 (5)	
N1	0.5850 (3)	0.89908 (9)	0.7715 (2)	0.0275 (6)	
N2	0.6902 (3)	0.98702 (10)	0.7444 (2)	0.0215 (6)	
N3	0.6539 (4)	0.90121 (12)	0.1483 (2)	0.0287 (6)	
C1	0.6280 (3)	0.94856 (12)	0.8083 (3)	0.0251 (7)	
H1	0.6136	0.9589	0.8907	0.030*	
C2	0.7069 (3)	0.97998 (11)	0.6192 (2)	0.0195 (6)	
C3	0.6686 (3)	0.93016 (10)	0.5563 (2)	0.0189 (6)	
C4	0.6775 (3)	0.92372 (10)	0.4284 (2)	0.0197 (6)	
H4	0.6451	0.8900	0.3849	0.024*	
C5	0.7329 (3)	0.96583 (11)	0.3640 (2)	0.0197 (6)	
C6	0.7786 (3)	1.01539 (10)	0.4309 (3)	0.0205 (6)	
C7	0.7605 (3)	1.02315 (10)	0.5543 (3)	0.0203 (6)	
H7	0.7844	1.0577	0.5953	0.024*	

O5	0.3012 (2)	0.90969 (8)	0.15126 (19)	0.0310 (5)	
N4	0.1638 (3)	0.83042 (10)	0.1039 (3)	0.0398 (7)	
C8	0.0491 (4)	0.91005 (12)	−0.0279 (3)	0.0350 (8)	
H8A	0.0794	0.9485	−0.0322	0.053*	
H8B	−0.0633	0.9074	−0.0124	0.053*	
H8C	0.0480	0.8921	−0.1103	0.053*	
C9	0.1766 (4)	0.88256 (13)	0.0817 (3)	0.0353 (8)	
C10	0.2902 (4)	0.80452 (13)	0.2124 (3)	0.0485 (9)	
H10A	0.3986	0.8021	0.1902	0.073*	
H10B	0.2519	0.7678	0.2274	0.073*	
H10C	0.3039	0.8265	0.2914	0.073*	
C11	0.0204 (4)	0.79663 (14)	0.0324 (3)	0.0557 (11)	
H11A	−0.0418	0.8160	−0.0461	0.084*	
H11B	−0.0550	0.7894	0.0882	0.084*	
H11C	0.0630	0.7620	0.0075	0.084*	
O6	0.2074 (16)	0.7032 (6)	0.7823 (13)	0.030 (2)	0.582 (18)
N5	0.4587 (10)	0.7349 (4)	0.8926 (6)	0.031 (2)	0.582 (18)
C12	0.2503 (4)	0.80107 (12)	0.7830 (3)	0.0365 (8)	
H12A	0.3069	0.8145	0.7189	0.055*	0.582 (18)
H12B	0.2763	0.8249	0.8594	0.055*	0.582 (18)
H12C	0.1274	0.8007	0.7440	0.055*	0.582 (18)
H12D	0.2826	0.8135	0.7054	0.055*	0.418 (18)
H12E	0.2352	0.8327	0.8351	0.055*	0.418 (18)
H12F	0.1442	0.7806	0.7566	0.055*	0.418 (18)
C13	0.3049 (12)	0.7419 (5)	0.8214 (7)	0.025 (2)	0.582 (18)
C14	0.577 (2)	0.7779 (6)	0.9476 (19)	0.039 (3)	0.582 (18)
H14A	0.5301	0.8132	0.9132	0.059*	0.582 (18)
H14B	0.6837	0.7717	0.9241	0.059*	0.582 (18)
H14C	0.5986	0.7778	1.0426	0.059*	0.582 (18)
C15	0.5077 (4)	0.67715 (13)	0.9299 (3)	0.0446 (9)	
H15A	0.4511	0.6649	0.9954	0.067*	0.582 (18)
H15B	0.6304	0.6749	0.9660	0.067*	0.582 (18)
H15C	0.4736	0.6538	0.8527	0.067*	0.582 (18)
H15D	0.4754	0.6685	1.0100	0.067*	0.418 (18)
H15E	0.6168	0.6960	0.9518	0.067*	0.418 (18)
H15F	0.5174	0.6434	0.8832	0.067*	0.418 (18)
O7	0.248 (3)	0.6938 (8)	0.7573 (19)	0.036 (3)	0.418 (18)
N6	0.3880 (17)	0.7667 (5)	0.8636 (9)	0.033 (3)	0.418 (18)
C16	0.371 (2)	0.7123 (5)	0.8437 (12)	0.032 (4)	0.418 (18)
C17	0.533 (3)	0.7924 (10)	0.955 (3)	0.063 (8)	0.418 (18)
H17A	0.5635	0.7716	1.0363	0.094*	0.418 (18)
H17B	0.5043	0.8298	0.9729	0.094*	0.418 (18)
H17C	0.6292	0.7929	0.9162	0.094*	0.418 (18)
H1N	0.706 (4)	1.0162 (12)	0.776 (3)	0.031 (9)*	
H2N	0.697 (4)	0.8701 (13)	0.187 (3)	0.041 (10)*	
H3N	0.546 (4)	0.9047 (12)	0.137 (3)	0.040 (10)*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0314 (4)	0.0265 (4)	0.0280 (4)	−0.0046 (3)	0.0075 (3)	0.0036 (3)
S1	0.0337 (5)	0.0215 (4)	0.0259 (4)	−0.0019 (4)	0.0107 (4)	−0.0002 (3)
S2	0.0223 (4)	0.0386 (5)	0.0189 (4)	−0.0005 (4)	0.0052 (3)	−0.0045 (3)
O1	0.0433 (14)	0.0351 (12)	0.0332 (12)	−0.0194 (11)	0.0107 (11)	−0.0067 (10)
O2	0.0448 (14)	0.0272 (11)	0.0444 (14)	0.0129 (10)	0.0169 (11)	0.0095 (10)
O3	0.0330 (13)	0.0418 (12)	0.0198 (11)	0.0004 (10)	0.0036 (9)	0.0048 (9)
O4	0.0199 (12)	0.0648 (15)	0.0259 (11)	0.0032 (10)	0.0084 (9)	−0.0102 (10)
N1	0.0322 (15)	0.0275 (14)	0.0246 (13)	−0.0023 (12)	0.0106 (11)	−0.0001 (11)
N2	0.0261 (15)	0.0167 (14)	0.0216 (14)	0.0010 (12)	0.0061 (11)	−0.0035 (12)
N3	0.0243 (18)	0.0359 (17)	0.0237 (14)	0.0013 (14)	0.0025 (13)	−0.0092 (13)
C1	0.0233 (17)	0.0341 (18)	0.0177 (15)	0.0035 (14)	0.0046 (13)	0.0004 (13)
C2	0.0180 (16)	0.0241 (16)	0.0149 (14)	0.0040 (13)	0.0016 (12)	−0.0016 (12)
C3	0.0165 (15)	0.0181 (14)	0.0223 (15)	0.0008 (12)	0.0055 (12)	−0.0021 (12)
C4	0.0162 (16)	0.0179 (15)	0.0234 (15)	0.0005 (12)	0.0026 (12)	−0.0068 (12)
C5	0.0141 (15)	0.0263 (16)	0.0170 (14)	−0.0006 (12)	0.0013 (12)	−0.0018 (12)
C6	0.0139 (15)	0.0204 (15)	0.0258 (16)	0.0015 (12)	0.0028 (12)	0.0026 (12)
C7	0.0200 (16)	0.0171 (15)	0.0211 (15)	0.0021 (12)	0.0009 (13)	−0.0024 (12)
O5	0.0272 (12)	0.0276 (11)	0.0373 (12)	−0.0051 (10)	0.0066 (10)	−0.0117 (10)
N4	0.0436 (18)	0.0343 (17)	0.0447 (17)	−0.0038 (14)	0.0175 (14)	−0.0015 (13)
C8	0.036 (2)	0.0411 (19)	0.0276 (17)	0.0008 (16)	0.0078 (15)	0.0015 (15)
C9	0.042 (2)	0.035 (2)	0.039 (2)	0.0099 (17)	0.0288 (18)	−0.0001 (16)
C10	0.049 (2)	0.036 (2)	0.058 (2)	0.0086 (17)	0.010 (2)	0.0145 (17)
C11	0.054 (3)	0.056 (2)	0.057 (2)	−0.034 (2)	0.015 (2)	−0.0084 (19)
O6	0.027 (5)	0.023 (5)	0.036 (4)	−0.004 (3)	0.003 (3)	0.001 (3)
N5	0.030 (4)	0.028 (5)	0.029 (3)	−0.013 (4)	0.000 (3)	−0.001 (3)
C12	0.048 (2)	0.0283 (18)	0.0331 (18)	0.0088 (16)	0.0103 (16)	0.0038 (14)
C13	0.031 (5)	0.022 (5)	0.025 (4)	−0.003 (5)	0.014 (3)	−0.004 (4)
C14	0.043 (7)	0.034 (8)	0.035 (5)	−0.020 (6)	0.001 (5)	0.001 (6)
C15	0.038 (2)	0.038 (2)	0.049 (2)	0.0049 (17)	−0.0038 (17)	0.0163 (17)
O7	0.032 (8)	0.022 (6)	0.044 (8)	−0.002 (5)	−0.007 (5)	−0.007 (4)
N6	0.048 (7)	0.024 (6)	0.027 (4)	−0.008 (6)	0.011 (5)	0.001 (4)
C16	0.048 (9)	0.021 (7)	0.032 (7)	−0.004 (8)	0.019 (7)	−0.003 (6)
C17	0.071 (15)	0.034 (10)	0.054 (12)	−0.005 (8)	−0.035 (10)	0.007 (8)

Geometric parameters (Å, °)

Cl1—C6	1.735 (3)	C10—H10A	0.9800
S1—O1	1.436 (2)	C10—H10B	0.9800
S1—O2	1.437 (2)	C10—H10C	0.9800
S1—N1	1.619 (2)	C11—H11A	0.9800
S1—C3	1.748 (3)	C11—H11B	0.9800
S2—O3	1.4297 (19)	C11—H11C	0.9800
S2—O4	1.431 (2)	O6—C13	1.24 (2)
S2—N3	1.595 (3)	N5—C13	1.300 (16)
S2—C5	1.786 (3)	N5—C14	1.45 (2)

N1—C1	1.295 (3)	N5—C15	1.498 (11)
N2—C1	1.339 (3)	C12—C13	1.541 (12)
N2—C2	1.386 (3)	C12—H12A	0.9783
N2—H1N	0.79 (3)	C12—H12B	0.9770
N3—H2N	0.89 (3)	C12—H12C	0.9843
N3—H3N	0.87 (3)	C12—H12D	0.9800
C1—H1	0.9500	C12—H12E	0.9800
C2—C3	1.389 (3)	C12—H12F	0.9800
C2—C7	1.397 (4)	C14—H14A	0.9800
C3—C4	1.392 (4)	C14—H14B	0.9800
C4—C5	1.381 (4)	C14—H14C	0.9800
C4—H4	0.9500	C15—H15A	0.9800
C5—C6	1.409 (4)	C15—H15B	0.9800
C6—C7	1.373 (4)	C15—H15C	0.9800
C7—H7	0.9500	C15—H15D	0.9800
O5—C9	1.280 (4)	C15—H15E	0.9800
N4—C9	1.310 (4)	C15—H15F	0.9801
N4—C11	1.476 (4)	O7—C16	1.26 (3)
N4—C10	1.477 (4)	N6—C16	1.35 (2)
C8—C9	1.506 (4)	N6—C17	1.47 (3)
C8—H8A	0.9800	C17—H17A	0.9800
C8—H8B	0.9800	C17—H17B	0.9800
C8—H8C	0.9800	C17—H17C	0.9800
O1—S1—O2	116.70 (13)	N4—C9—C8	120.3 (3)
O1—S1—N1	109.17 (13)	N4—C10—H10A	109.5
O2—S1—N1	107.95 (12)	N4—C10—H10B	109.5
O1—S1—C3	109.28 (12)	H10A—C10—H10B	109.5
O2—S1—C3	108.26 (13)	N4—C10—H10C	109.5
N1—S1—C3	104.83 (12)	H10A—C10—H10C	109.5
O3—S2—O4	119.20 (13)	H10B—C10—H10C	109.5
O3—S2—N3	107.38 (14)	N4—C11—H11A	109.5
O4—S2—N3	108.05 (15)	N4—C11—H11B	109.5
O3—S2—C5	108.13 (12)	H11A—C11—H11B	109.5
O4—S2—C5	106.33 (12)	N4—C11—H11C	109.5
N3—S2—C5	107.20 (13)	H11A—C11—H11C	109.5
C1—N1—S1	121.2 (2)	H11B—C11—H11C	109.5
C1—N2—C2	123.2 (2)	C13—N5—C14	125.8 (14)
C1—N2—H1N	117 (2)	C13—N5—C15	115.5 (12)
C2—N2—H1N	119 (2)	C14—N5—C15	118.4 (8)
S2—N3—H2N	115 (2)	C13—C12—H12A	110.1
S2—N3—H3N	113 (2)	C13—C12—H12B	110.9
H2N—N3—H3N	115 (3)	H12A—C12—H12B	109.9
N1—C1—N2	128.1 (3)	C13—C12—H12C	107.3
N1—C1—H1	116.0	H12A—C12—H12C	109.3
N2—C1—H1	116.0	H12B—C12—H12C	109.4
N2—C2—C3	120.4 (2)	C13—C12—H12D	113.1
N2—C2—C7	120.3 (2)	C13—C12—H12E	131.8
C3—C2—C7	119.3 (2)	H12D—C12—H12E	109.5
C2—C3—C4	120.5 (2)	C13—C12—H12F	76.6

C2—C3—S1	119.8 (2)	H12D—C12—H12F	109.5
C4—C3—S1	119.50 (19)	H12E—C12—H12F	109.5
C5—C4—C3	120.7 (2)	O6—C13—N5	122.4 (15)
C5—C4—H4	119.7	O6—C13—C12	121.0 (9)
C3—C4—H4	119.7	N5—C13—C12	116.6 (12)
C4—C5—C6	118.2 (2)	N5—C15—H15A	109.5
C4—C5—S2	119.8 (2)	N5—C15—H15B	109.5
C6—C5—S2	121.9 (2)	H15A—C15—H15B	109.5
C7—C6—C5	121.5 (2)	N5—C15—H15C	109.5
C7—C6—Cl1	117.8 (2)	H15A—C15—H15C	109.5
C5—C6—Cl1	120.7 (2)	H15B—C15—H15C	109.5
C6—C7—C2	119.7 (2)	N5—C15—H15D	108.7
C6—C7—H7	120.2	N5—C15—H15E	77.3
C2—C7—H7	120.2	H15A—C15—H15E	123.3
C9—N4—C11	123.1 (3)	N5—C15—H15F	135.8
C9—N4—C10	119.2 (3)	H15D—C15—H15F	109.6
C11—N4—C10	117.5 (3)	H15E—C15—H15F	109.2
C9—C8—H8A	109.5	C16—N6—C17	124.0 (18)
C9—C8—H8B	109.5	O7—C16—N6	120 (2)
H8A—C8—H8B	109.5	N6—C17—H17A	109.5
C9—C8—H8C	109.5	N6—C17—H17B	109.6
H8A—C8—H8C	109.5	H17A—C17—H17B	109.5
H8B—C8—H8C	109.5	N6—C17—H17C	109.2
O5—C9—N4	119.5 (3)	H17A—C17—H17C	109.5
O5—C9—C8	120.2 (3)	H17B—C17—H17C	109.5
O1—S1—N1—C1	−132.9 (2)	O4—S2—C5—C4	−98.6 (2)
O2—S1—N1—C1	99.3 (2)	N3—S2—C5—C4	16.8 (3)
C3—S1—N1—C1	−15.9 (3)	O3—S2—C5—C6	−51.5 (2)
S1—N1—C1—N2	7.4 (4)	O4—S2—C5—C6	77.7 (2)
C2—N2—C1—N1	5.0 (5)	N3—S2—C5—C6	−167.0 (2)
C1—N2—C2—C3	−4.2 (4)	C4—C5—C6—C7	−3.4 (4)
C1—N2—C2—C7	175.1 (3)	S2—C5—C6—C7	−179.7 (2)
N2—C2—C3—C4	177.0 (2)	C4—C5—C6—Cl1	174.9 (2)
C7—C2—C3—C4	−2.3 (4)	S2—C5—C6—Cl1	−1.4 (3)
N2—C2—C3—S1	−7.6 (4)	C5—C6—C7—C2	4.5 (4)
C7—C2—C3—S1	173.1 (2)	Cl1—C6—C7—C2	−173.8 (2)
O1—S1—C3—C2	133.0 (2)	N2—C2—C7—C6	179.1 (2)
O2—S1—C3—C2	−98.9 (2)	C3—C2—C7—C6	−1.6 (4)
N1—S1—C3—C2	16.1 (3)	C11—N4—C9—O5	177.2 (3)
O1—S1—C3—C4	−51.5 (2)	C10—N4—C9—O5	2.3 (4)
O2—S1—C3—C4	76.6 (2)	C11—N4—C9—C8	−4.9 (4)
N1—S1—C3—C4	−168.4 (2)	C10—N4—C9—C8	−179.7 (3)
C2—C3—C4—C5	3.4 (4)	C14—N5—C13—O6	−176.7 (11)
S1—C3—C4—C5	−172.1 (2)	C15—N5—C13—O6	−3.0 (10)
C3—C4—C5—C6	−0.5 (4)	C14—N5—C13—C12	5.5 (12)
C3—C4—C5—S2	175.8 (2)	C15—N5—C13—C12	179.2 (4)
O3—S2—C5—C4	132.3 (2)	C17—N6—C16—O7	−175.2 (17)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3N···O5	0.87 (3)	2.06 (3)	2.909 (4)	168 (3)
N3—H2N···O6^{i}	0.89 (3)	2.05 (3)	2.906 (14)	159 (3)
N2—H1N···O5^{ii}	0.79 (3)	1.98 (3)	2.759 (3)	170 (3)
N3—H2N···O7^{i}	0.89 (3)	1.74 (4)	2.628 (18)	171 (3)

Symmetry codes: (i) *x*+1/2, −*y*+3/2, *z*−1/2; (ii) −*x*+1, −*y*+2, −*z*+1.

Fig. 1

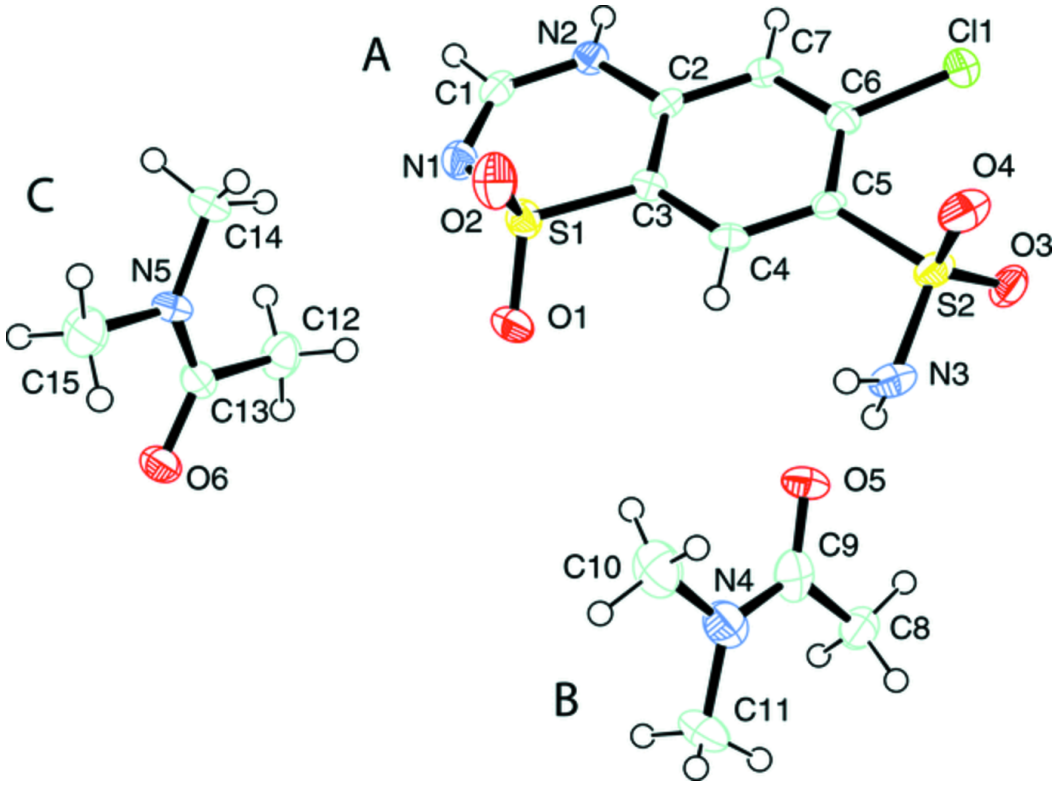

A
N2
C7
Cl1
C1
C2
C6
N1
O2
S1
C3
C5
O4
O3
C4
S2
O1
N3
C
C14
N5
C12
C15
C13
O6
O5
C9
C10
N4
C8
B
C11

Fig. 2