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4-(4-Methylbenzenesulfonamido)benzoic acid *N*,*N*-dimethylformamide monosolvate

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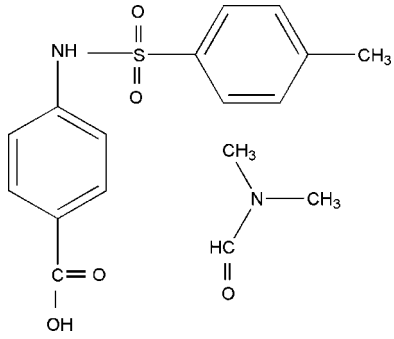
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.039; wR factor = 0.111; data-to-parameter ratio = 14.5.

In the title compound, $C_{14}H_{13}NO_4S\cdot C_3H_7NO$, the C—S—N—C torsion angle of −64.55 (17)° defines the folded conformation of the molecule. The dihedral angle between the benzene rings is 83.367 (6)°. In a crystal, molecules are linked into a chain along *a* axis through intermolecular N—H···O and O—H···O hydrogen bonds. There is also an intramolecular C—H···π interaction.

Related literature

N-protected amino acids possess an *R*-CONH-*R*′ group analogous to the structure of *O*-terminal peptides and proteins, see: Antolini *et al.* (1984); Menabue & Saladini (1988).



Experimental

Crystal data

$C_{14}H_{13}NO_4S\cdot C_3H_7NO$
M_r = 364.41
Monoclinic, $P2_1/n$
a = 8.0953 (10) Å
b = 25.151 (3) Å
c = 8.8840 (11) Å
β = 98.010 (1)°
V = 1791.1 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.21 mm^{-1}
T = 296 K
0.39 × 0.29 × 0.25 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.923, T_{max} = 0.949
9993 measured reflections
3336 independent reflections
2570 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.111
S = 1.04
3336 reflections
230 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

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

*Cg*1 is the centroid of the C1–C6 ring.

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.86	2.27	2.903 (2)	131
O2—H2*A*···O5^{ii}	0.82	1.79	2.598 (2)	168
C16—H16*C*···*Cg*1	0.96	2.98	3.576 (3)	121

Symmetry codes: (i) $x-1, y, z$; (ii) $-x+2, -y, -z+1$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KP2315).

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supporting information

Acta Cryst. (2011). E**67**, o1062 [doi:10.1107/S1600536811011949]

4-(4-Methylbenzenesulfonamido)benzoic acid *N*,*N*-dimethylformamide monosolvate

Miao-Ling Huang and Zhi-Yang Lin

S1. Comment

N-Protected amino acids possess *R*-CONH-*R′* group analogous to the structure of O-terminal peptides and proteins (Menabue, *et al.*, 1988, Antolini, *et al.*, 1984). The substitution of an Ar—SO_2-group on amine addes to 4-aminobenzeoic acid the coordination donors of three types-O, N donors from carboxyl, sulfoxyl and amine respectively, which may result in different coordination modes. In this paper, we synthesized the desired ligand in *N*,*N*-dimethylformamide solvent.

The title compound contains one *N*-*p*-tolysulfonyl-4-aminobenzoic acid molecule and a solvent N, *N*-dimethyl-formamide in the asymmetric unit (Fig. 1). The molecule has a C1—N1—S1—C8 of 64.552 (2) °, and the dihedral angle between the benzene rings is 83.367 (6) °. There are intermolecular hydrogen bonds between carboxylate group oxygen atoms, nitrogen atoms and solvent oxygen atoms of the types N—H···O and O—H···O generating a chain along *a* axis (Figs. 2 and 3, Table 1). There are intramolecular CH···π interactions between H(16 C) and H(17B) of solvent and the centre of two aromatic rings of the title molecule(C16—H16C···C1—C2—C3—C4—C5—C6 and C17—H17B···C8—C9—C10—C11—C12—C13; the distances of H to the centre of the aromatic rings are 2.982 (3) and 3.84 (3) Å, respectively).

S2. Experimental

A mixture of *N*-*p*-tolysulfonylchloride (1 mmol) and 4-aminobenzoic acid (1 mmol) in water(20 ml) was stirred at room temperature for 10 h. Then HCl (12 mol/*L*) was slowly added to the resulting solution. The mixture was stirred for 5 min and filtrated. The block colourless single crystals suitable for X-ray analysis were obtained by recrystallization from *N*,*N*-dimethylformamide. IR(KBr): 3423(*s*), 3198(*versus*), 3059(w), 2928(*s*), 2510(*m*), 1693(*versus*), 1635(*versus*), 1607(*s*), 1512(*m*), 1433(*m*), 1405(*s*), 1383(*s*), 1334(*s*), 1291(*s*), 1231(*m*), 1159(*versus*), 1092(*s*), 1021(w), 923(*s*), 862(*m*), 827(*m*), 765(*m*), 674(*s*), 5757(*s*), 547(*s*), 525(*m*), 503(w)cm^{-1}.

S3. Refinement

H atoms were placed in calculated positions and treated as riding on their parent atoms (C—H = 0.93–0.96 Å, N—H = 0.86 Å,*O*—H = 0.82 Å) and U_{iso}(H) = 1.2Ueq(C,*N*).

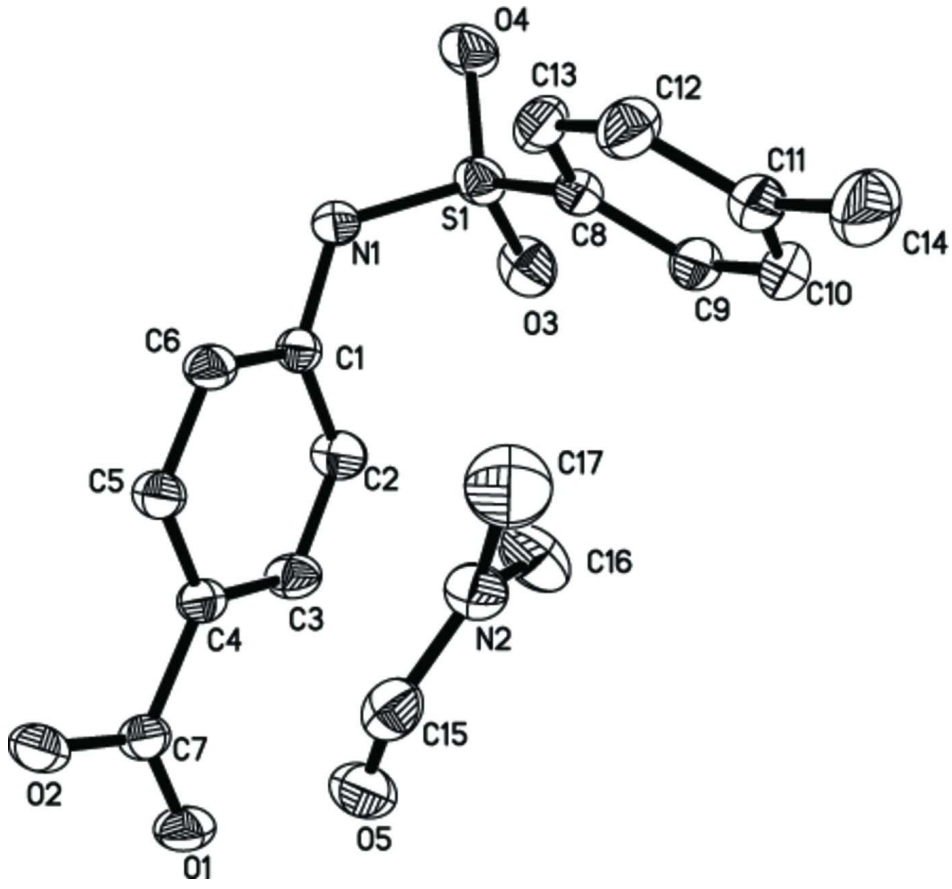

Figure 1

The *ORTEP* drawing of the title compound (I). Displacement ellipsoids are drawn at 30% probability level. All hydrogen atams have been omitted for clarity.

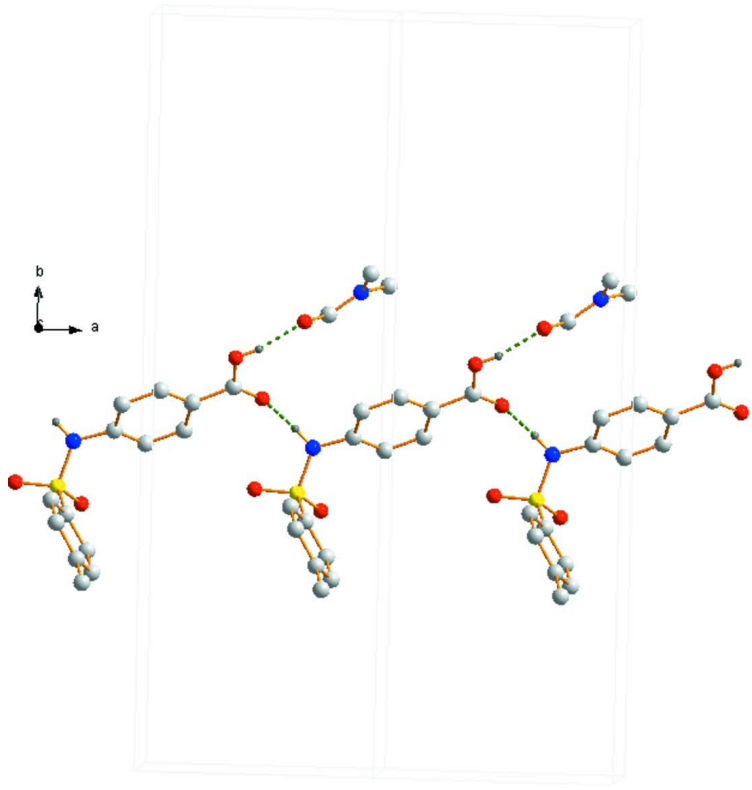

Figure 2

A view of the hydrogen bonds (dotted lines) in the crystal structure of the title compound (I).

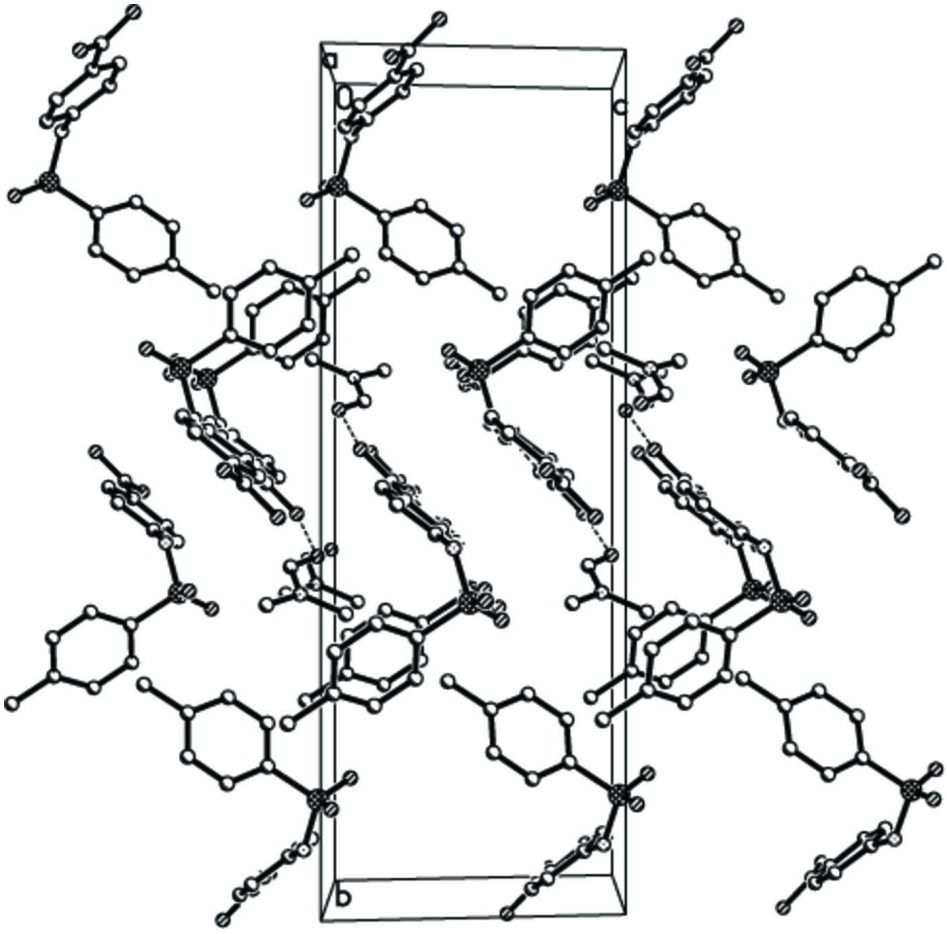

Figure 3

The crystal packing of the title compound (I), viewed along the *c* axis.

4-(4-Methylbenzenesulfonamido)benzoic acid *N*,*N*-dimethylformamide monosolvate

Crystal data

$C_{14}H_{13}NO_4S·C_3H_7NO$
M_r = 364.41
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 8.0953 (10) Å
b = 25.151 (3) Å
c = 8.8840 (11) Å
β = 98.010 (1)°
V = 1791.1 (4) Å^3
Z = 4
$F(000)$ = 768
D_x = 1.351 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3230 reflections
θ = 2.5–25.0°
μ = 0.21 mm^{-1}
T = 296 K
Block, colourless
0.39 × 0.29 × 0.25 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.923, T_{max} = 0.949
9993 measured reflections
3336 independent reflections
2570 reflections with $I > 2\sigma(I)$

R_{int} = 0.024
θ_{max} = 25.5°, θ_{min} = 2.5°
h = −9→9
k = −23→30
l = −10→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.111
S = 1.04
3336 reflections
230 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0517P)^2 + 0.5453P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 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}
C1	0.3556 (2)	0.05871 (7)	0.1256 (2)	0.0380 (4)
C2	0.4907 (2)	0.07414 (9)	0.0562 (2)	0.0498 (5)
H2	0.4753	0.0971	−0.0266	0.060*
C3	0.6483 (2)	0.05551 (9)	0.1098 (2)	0.0488 (5)
H3	0.7388	0.0665	0.0637	0.059*
C4	0.6733 (2)	0.02077 (8)	0.2312 (2)	0.0400 (4)
C5	0.5375 (2)	0.00633 (8)	0.3018 (2)	0.0449 (5)
H5	0.5529	−0.0167	0.3846	0.054*
C6	0.3800 (2)	0.02563 (8)	0.2509 (2)	0.0451 (5)
H6	0.2905	0.0164	0.3008	0.054*
C7	0.8422 (2)	−0.00147 (8)	0.2797 (2)	0.0452 (5)
C8	0.1600 (2)	0.17188 (8)	0.1824 (2)	0.0459 (5)
C9	0.2493 (3)	0.21904 (9)	0.1886 (3)	0.0560 (6)
H9	0.2980	0.2302	0.1052	0.067*
C10	0.2656 (3)	0.24944 (9)	0.3195 (3)	0.0632 (6)
H10	0.3253	0.2811	0.3230	0.076*
C11	0.1950 (3)	0.23365 (9)	0.4452 (3)	0.0602 (6)
C12	0.1080 (3)	0.18598 (10)	0.4370 (3)	0.0625 (6)
H12	0.0610	0.1745	0.5211	0.075*
C13	0.0896 (3)	0.15518 (9)	0.3078 (3)	0.0555 (6)

H13	0.0303	0.1234	0.3047	0.067*
C14	0.2119 (4)	0.26663 (12)	0.5889 (4)	0.0878 (9)
H14A	0.1492	0.2989	0.5700	0.132*
H14B	0.3273	0.2750	0.6199	0.132*
H14C	0.1699	0.2469	0.6679	0.132*
C15	0.7728 (3)	0.09910 (9)	0.6138 (2)	0.0540 (6)
H15	0.7882	0.0853	0.7119	0.065*
C16	0.6101 (3)	0.15217 (11)	0.4239 (3)	0.0717 (7)
H16A	0.7017	0.1442	0.3698	0.108*
H16B	0.5986	0.1900	0.4319	0.108*
H16C	0.5092	0.1375	0.3702	0.108*
C17	0.5200 (4)	0.13996 (13)	0.6762 (4)	0.0936 (10)
H17A	0.5620	0.1273	0.7761	0.140*
H17B	0.4173	0.1221	0.6400	0.140*
H17C	0.5005	0.1776	0.6802	0.140*
N1	0.18945 (19)	0.07351 (7)	0.06580 (18)	0.0446 (4)
H1	0.1149	0.0489	0.0558	0.054*
N2	0.6410 (2)	0.12933 (7)	0.5741 (2)	0.0518 (4)
O1	0.96596 (17)	0.01262 (6)	0.22823 (18)	0.0589 (4)
O2	0.84373 (17)	−0.03862 (7)	0.38401 (19)	0.0628 (4)
H2A	0.9385	−0.0505	0.4047	0.094*
O3	0.2361 (2)	0.15441 (7)	−0.08607 (17)	0.0661 (5)
O4	−0.04468 (18)	0.12891 (6)	−0.03477 (18)	0.0621 (4)
O5	0.87652 (19)	0.08779 (7)	0.53101 (19)	0.0625 (4)
S1	0.13037 (6)	0.13338 (2)	0.01515 (6)	0.04889 (17)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0357 (10)	0.0379 (10)	0.0400 (10)	0.0011 (8)	0.0036 (8)	−0.0049 (8)
C2	0.0447 (12)	0.0610 (14)	0.0444 (11)	0.0025 (10)	0.0084 (9)	0.0134 (10)
C3	0.0369 (11)	0.0612 (14)	0.0501 (12)	−0.0020 (9)	0.0124 (9)	0.0063 (10)
C4	0.0336 (10)	0.0438 (11)	0.0424 (10)	−0.0017 (8)	0.0044 (8)	−0.0040 (9)
C5	0.0387 (11)	0.0504 (12)	0.0459 (11)	0.0025 (9)	0.0073 (8)	0.0092 (9)
C6	0.0348 (11)	0.0506 (12)	0.0517 (12)	0.0004 (9)	0.0122 (9)	0.0067 (9)
C7	0.0353 (10)	0.0476 (12)	0.0522 (12)	−0.0024 (9)	0.0046 (8)	−0.0041 (9)
C8	0.0364 (11)	0.0419 (12)	0.0582 (12)	0.0028 (8)	0.0025 (9)	0.0042 (9)
C9	0.0480 (13)	0.0471 (13)	0.0736 (15)	−0.0006 (10)	0.0112 (11)	0.0114 (11)
C10	0.0536 (14)	0.0423 (13)	0.0934 (18)	−0.0078 (10)	0.0091 (13)	−0.0037 (12)
C11	0.0488 (13)	0.0527 (14)	0.0794 (16)	−0.0030 (11)	0.0103 (11)	−0.0146 (12)
C12	0.0592 (14)	0.0635 (15)	0.0682 (15)	−0.0128 (12)	0.0207 (11)	−0.0104 (12)
C13	0.0540 (13)	0.0482 (13)	0.0659 (14)	−0.0130 (10)	0.0142 (11)	−0.0056 (11)
C14	0.083 (2)	0.0776 (19)	0.106 (2)	−0.0165 (16)	0.0240 (17)	−0.0388 (17)
C15	0.0571 (14)	0.0541 (14)	0.0479 (12)	−0.0114 (11)	−0.0028 (10)	0.0104 (10)
C16	0.0728 (17)	0.0799 (18)	0.0596 (14)	0.0280 (14)	−0.0006 (12)	0.0042 (13)
C17	0.091 (2)	0.102 (2)	0.100 (2)	0.0006 (17)	0.0533 (18)	−0.0015 (18)
N1	0.0352 (9)	0.0432 (10)	0.0532 (10)	−0.0005 (7)	−0.0017 (7)	−0.0013 (7)
N2	0.0465 (10)	0.0578 (11)	0.0517 (10)	0.0023 (8)	0.0088 (8)	0.0015 (8)

O1	0.0336 (8)	0.0704 (11)	0.0740 (10)	−0.0024 (7)	0.0121 (7)	0.0075 (8)
O2	0.0371 (8)	0.0679 (11)	0.0827 (11)	0.0070 (7)	0.0059 (8)	0.0237 (9)
O3	0.0747 (11)	0.0672 (11)	0.0587 (9)	0.0091 (8)	0.0173 (8)	0.0190 (8)
O4	0.0481 (9)	0.0665 (11)	0.0659 (10)	0.0111 (7)	−0.0123 (7)	0.0033 (8)
O5	0.0456 (9)	0.0688 (11)	0.0723 (10)	0.0096 (7)	0.0055 (8)	0.0127 (8)
S1	0.0447 (3)	0.0511 (3)	0.0492 (3)	0.0066 (2)	0.0007 (2)	0.0071 (2)

Geometric parameters (Å, º)

C1—C6	1.381 (3)	C12—C13	1.375 (3)
C1—C2	1.384 (3)	C12—H12	0.9300
C1—N1	1.425 (2)	C13—H13	0.9300
C2—C3	1.380 (3)	C14—H14A	0.9600
C2—H2	0.9300	C14—H14B	0.9600
C3—C4	1.380 (3)	C14—H14C	0.9600
C3—H3	0.9300	C15—O5	1.224 (3)
C4—C5	1.388 (2)	C15—N2	1.317 (3)
C4—C7	1.485 (3)	C15—H15	0.9300
C5—C6	1.380 (3)	C16—N2	1.442 (3)
C5—H5	0.9300	C16—H16A	0.9600
C6—H6	0.9300	C16—H16B	0.9600
C7—O1	1.211 (2)	C16—H16C	0.9600
C7—O2	1.315 (2)	C17—N2	1.450 (3)
C8—C13	1.386 (3)	C17—H17A	0.9600
C8—C9	1.386 (3)	C17—H17B	0.9600
C8—S1	1.762 (2)	C17—H17C	0.9600
C9—C10	1.382 (3)	N1—S1	1.6243 (17)
C9—H9	0.9300	N1—H1	0.8600
C10—C11	1.382 (3)	O2—H2A	0.8200
C10—H10	0.9300	O3—S1	1.4266 (16)
C11—C12	1.387 (3)	O4—S1	1.4291 (15)
C11—C14	1.512 (3)		
C6—C1—C2	119.62 (17)	C12—C13—H13	120.3
C6—C1—N1	118.66 (16)	C8—C13—H13	120.3
C2—C1—N1	121.62 (17)	C11—C14—H14A	109.5
C3—C2—C1	120.08 (18)	C11—C14—H14B	109.5
C3—C2—H2	120.0	H14A—C14—H14B	109.5
C1—C2—H2	120.0	C11—C14—H14C	109.5
C2—C3—C4	120.81 (18)	H14A—C14—H14C	109.5
C2—C3—H3	119.6	H14B—C14—H14C	109.5
C4—C3—H3	119.6	O5—C15—N2	124.8 (2)
C3—C4—C5	118.68 (17)	O5—C15—H15	117.6
C3—C4—C7	119.61 (17)	N2—C15—H15	117.6
C5—C4—C7	121.68 (18)	N2—C16—H16A	109.5
C6—C5—C4	120.87 (18)	N2—C16—H16B	109.5
C6—C5—H5	119.6	H16A—C16—H16B	109.5
C4—C5—H5	119.6	N2—C16—H16C	109.5

C5—C6—C1	119.87 (17)	H16A—C16—H16C	109.5
C5—C6—H6	120.1	H16B—C16—H16C	109.5
C1—C6—H6	120.1	N2—C17—H17A	109.5
O1—C7—O2	123.18 (18)	N2—C17—H17B	109.5
O1—C7—C4	123.84 (19)	H17A—C17—H17B	109.5
O2—C7—C4	112.97 (16)	N2—C17—H17C	109.5
C13—C8—C9	119.9 (2)	H17A—C17—H17C	109.5
C13—C8—S1	119.26 (16)	H17B—C17—H17C	109.5
C9—C8—S1	120.86 (17)	C1—N1—S1	124.87 (13)
C10—C9—C8	119.7 (2)	C1—N1—H1	117.6
C10—C9—H9	120.2	S1—N1—H1	117.6
C8—C9—H9	120.2	C15—N2—C16	120.48 (18)
C11—C10—C9	121.2 (2)	C15—N2—C17	122.1 (2)
C11—C10—H10	119.4	C16—N2—C17	117.4 (2)
C9—C10—H10	119.4	C7—O2—H2A	109.5
C10—C11—C12	118.0 (2)	O3—S1—O4	119.31 (10)
C10—C11—C14	121.7 (2)	O3—S1—N1	109.82 (9)
C12—C11—C14	120.3 (2)	O4—S1—N1	104.71 (9)
C13—C12—C11	121.7 (2)	O3—S1—C8	107.75 (10)
C13—C12—H12	119.1	O4—S1—C8	108.51 (9)
C11—C12—H12	119.1	N1—S1—C8	106.00 (9)
C12—C13—C8	119.4 (2)		

C6—C1—C2—C3	1.5 (3)	C10—C11—C12—C13	−0.8 (4)
N1—C1—C2—C3	−174.66 (19)	C14—C11—C12—C13	179.6 (2)
C1—C2—C3—C4	1.0 (3)	C11—C12—C13—C8	0.3 (4)
C2—C3—C4—C5	−2.2 (3)	C9—C8—C13—C12	0.6 (3)
C2—C3—C4—C7	175.86 (19)	S1—C8—C13—C12	−177.99 (18)
C3—C4—C5—C6	0.9 (3)	C6—C1—N1—S1	135.20 (17)
C7—C4—C5—C6	−177.16 (19)	C2—C1—N1—S1	−48.6 (2)
C4—C5—C6—C1	1.7 (3)	O5—C15—N2—C16	−0.3 (4)
C2—C1—C6—C5	−2.9 (3)	O5—C15—N2—C17	178.1 (2)
N1—C1—C6—C5	173.46 (18)	C1—N1—S1—O3	51.58 (18)
C3—C4—C7—O1	7.1 (3)	C1—N1—S1—O4	−179.18 (15)
C5—C4—C7—O1	−174.9 (2)	C1—N1—S1—C8	−64.55 (17)
C3—C4—C7—O2	−172.33 (19)	C13—C8—S1—O3	−170.90 (16)
C5—C4—C7—O2	5.7 (3)	C9—C8—S1—O3	10.6 (2)
C13—C8—C9—C10	−0.8 (3)	C13—C8—S1—O4	58.62 (19)
S1—C8—C9—C10	177.71 (17)	C9—C8—S1—O4	−119.92 (17)
C8—C9—C10—C11	0.2 (3)	C13—C8—S1—N1	−53.38 (19)
C9—C10—C11—C12	0.6 (4)	C9—C8—S1—N1	128.09 (17)
C9—C10—C11—C14	−179.9 (2)		

Hydrogen-bond geometry (Å, º)

Cg1 is the centroid of the C1–C6 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.86	2.27	2.903 (2)	131

O2—H2*A*···O5^{ii}	0.82	1.79	2.598 (2)	168
C16—H16*C*···*Cg*1	0.96	2.98	3.576 (3)	121

Symmetry codes: (i) $x-1, y, z$; (ii) $-x+2, -y, -z+1$.