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The Schiff base 11-thiosemicarbazono-indeno[1,2-*b*]quinoxaline-8-carboxylic acid dimethyl sulfoxide solvate

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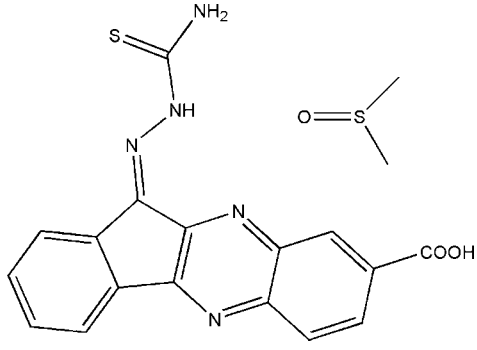
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.006 Å; H-atom completeness 65%; disorder in solvent or counterion; R factor = 0.067; wR factor = 0.198; data-to-parameter ratio = 13.6.

In the title Schiff base compound, $C_{17}H_{28}N_5O_2S\cdot C_2H_6OS$, all atoms in the Schiff base are nearly coplanar. There are two similar molecules of the Schiff base and two dimethyl sulfoxide solvent molecules in the asymmetric unit. The crystal packing is stabilized by N—H···O and N—H···N hydrogen-bond interactions. One solvent S atom is disordered over two positions in approximately a 4:1 ratio; the other solvent S atom is disordered over two positions in approximately a 7:1 ratio.

Related literature

For related literature, see: Che *et al.* (2006). For similar crystal structures, see; Javad *et al.* (2005) and Singh *et al.* (1978). For synthesis, see: Rao, Kumar & Rao (1984); Rao, Rao & Girisham (1984).



Experimental

Crystal data

$C_{17}H_{28}N_5O_2S\cdot C_2H_6OS$
M_r = 427.50
Monoclinic, $P2_1/c$
a = 14.876 (2) Å
b = 25.205 (3) Å
c = 11.0201 (15) Å
β = 95.775 (3)°
V = 4111.0 (10) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.29 mm^{-1}
T = 293 (2) K
0.43 × 0.15 × 0.15 mm

Data collection

Bruker SMART APEX2 CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.888, T_{max} = 0.958
21346 measured reflections
7290 independent reflections
4399 reflections with $I > 2\sigma(I)$
R_{int} = 0.051

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.198
S = 1.04
7290 reflections
535 parameters
18 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N7—H7*N*···N9	0.86	2.17	2.838 (4)	134
N2—H2···N4	0.86	2.12	2.807 (4)	137
N6—H6*B*···O5^{i}	0.86	2.09	2.820 (4)	143
N6—H6*A*···O2^{ii}	0.86	2.21	2.982 (5)	149
N1—H1*B*···O6^{iii}	0.86	2.36	3.131 (5)	150
N1—H1*A*···O3^{iv}	0.86	2.30	2.872 (4)	124
O4—H4···O6^{v}	0.82	1.83	2.642 (4)	170
O1—H1···O5^{vi}	0.82	1.78	2.591 (4)	168

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

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: *SHELXTL-Plus* (Sheldrick, 1990); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FL2127).

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supplementary materials

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The Schiff base 11-thiosemicarbazonoindeno[1,2-*b*]quinoxaline-8-carboxylic acid dimethyl sulfoxide solvate

X.-Q. Che and L.-B. Wang

Comment

The thiosemicarbazide Schiff-base and its complexes are particularly widely studied because they are not only organic colorants with excellent performance and antibacterial & antiviral biological activities, but also templates for studying biocatalytic activity (Singh *et al.*, 1978; Javad *et al.*, 2005), but there are few studies of thiosemicarbazide Schiff-base synthesis (Rao *et al.*, 1984a; Rao *et al.*, 1984 b). Recently, we have reported a crystal structure of the title compound (I) zinc complex (Che *et al.*, 2006). Here we present crystal structure of (I).

There are two similar molecules of (I) and two DMSO solvent molecules in the asymmetric unit (Fig. 1). Bond distances and angles are very similar to the our previously reported zinc complex of (I) (Che *et al.*, 2006). In the zinc complex of (I), each ligand (I) anion, which lost two H atoms on N2 and carboxyl O1, has two parts. One part, acting in tridentate chelating mode, coordinates to one Zn^{II} through N2, N4 and S1 , while another part coordinates to another Zn^{II} through a monodentate carboxyl group. In the present Schiff-base (I), the H atoms on N2 and O1 are not lost. The N2 atom is also in the cis position with respect to N4 in the quinoxaline ring. All atoms (except the H atoms) in the molecule are nearly planar (the mean deviation of the atoms from the least-squares plane is 0.0590 Å). The crystal packing of (I) is stabilized by intermolecular N—H···O and N—H···N hydrogen-bond interactions (Table 1).

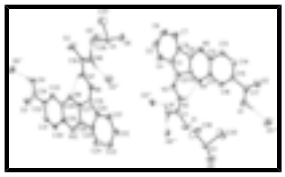
Experimental

All reagents were purchased (Adrich) and used without further purification. Compound (I) was synthesized according to the reference (Rao *et al.*, 1984a). Then compound (I) (0.698 g, 0.002 mol) was dissolved into 20 ml DMSO. After heating at 70 °C for 20 min, the mixture was allowed to cool and evaporate naturally. Yellow block crystals of (I) suitable for single-crystal X-ray diffraction were obtained by evaporating the mixture at room temperature for a period of two weeks. Analysis found: C 53.5, H 4.0, N 16.4, S 14.8%; $C_{19}H_{17}N_5O_3S_2$ requires: C 53.38, H 4.01, N 16.38, S 15.00%.

Refinement

H atoms were generated geometrically and refined as riding atoms with C—H= 0.93 Å (CH), C—H= 0.96 Å (CH_3), N—H= 0.86 Å (NH and NH_2), Uiso(H)= 1.2 times U_{eq}(C) or U_{eq}(N) for CH , NH and NH_2, Uiso(H)= 1.5 times U_{eq}(C) for CH_3. The S3 snd s4 atom in the solvent DMSO molecules were disordered with refined occupancies of 0.805, 0.195 and 0.871, 0.129, respectively. Standard DFIX restraints were used for the dimensions of the disordered DMSO. The S atoms of the minor orientation were refined with an overall U_{iso} value. The H atoms attached to the disordered DMSO molecules were not located. All other non-H atoms were refined anisotropically. The maximum positive peak of 0.42 e $Å^{-3}$ in the final difference electron density map is located 1.06 (1) Å from atom S4.

Figures



Scheme 1. Chemical structure.

Fig. 1. The asymmetric unit of (I) with the atom labeling scheme. Displacement ellipsoids are shown at the 30% probability level. Only the major orientation of the disordered DMSO molecule are shown. The hydrogen-bond interactions are drawn with dashed lines. Symmetry codes: (i) -x+1,y-1/2,-z+3/2 (ii) -x+1,-y+1,-z+2 (iii) -x,-y+1,-z+1 (iv) -x,-y+1,-z (v) x,y,z-1 (vi) x,-y+3/2,z+1/2.

11-thiosemicarbazonoindeno[1,2-b]quinoxaline-8-carboxylic acid dimethyl sulfoxide solvate

Crystal data

$C_{17}H_{28}N_5O_2S_1{\cdot}C_2H_6OS$	F_{000} = 1776
M_r = 427.50	D_x = 1.381 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 14.876 (2) Å	Cell parameters from 2329 reflections
b = 25.205 (3) Å	θ = 2.3–19.3º
c = 11.0201 (15) Å	μ = 0.29 mm^{-1}
β = 95.775 (3)º	T = 293 (2) K
V = 4111.0 (10) Å^3	Block, yellow
Z = 8	0.43 × 0.15 × 0.15 mm

Data collection

Bruker SMART APEX2 CCD diffractometer	7290 independent reflections
Radiation source: fine-focus sealed tube	4399 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.051
T = 293(2) K	θ_{max} = 25.1º
φ and ω scans	θ_{min} = 1.6º
Absorption correction: multi-scan (SADABS; Bruker, 2004)	h = −17→16
T_{min} = 0.888, T_{max} = 0.958	k = −23→30
21346 measured reflections	l = −12→13

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.067	$w = 1/[\sigma^2(F_o^2) + (0.0964P)^2 + 0.2132P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.198	$(\Delta/\sigma)_{max} < 0.001$
S = 1.04	$\Delta\rho_{max}$ = 0.42 e Å^{-3}
7290 reflections	$\Delta\rho_{min}$ = −0.21 e Å^{-3}

535 parameters
18 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
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 $F^2 > 2\text{sigma}(F^2)$ 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)
S3	0.24586 (9)	0.74512 (5)	0.94383 (12)	0.0586 (6)	0.805 (5)
S4	0.24527 (11)	0.29402 (6)	0.40349 (11)	0.0670 (7)	0.871 (5)
S3'	0.3110 (16)	0.7249 (8)	1.010 (2)	0.264 (10)*	0.195 (5)
S4'	0.1896 (15)	0.2639 (9)	0.4082 (17)	0.162 (9)*	0.129 (5)
N1	−0.0339 (2)	0.61471 (15)	0.6245 (3)	0.0728 (10)	
H1A	−0.0535	0.5838	0.6017	0.087*	
H1B	−0.0523	0.6424	0.5836	0.087*	
N2	0.0507 (2)	0.57418 (13)	0.7820 (3)	0.0549 (8)	
H2	0.0874	0.5761	0.8474	0.066*	
N3	0.0204 (2)	0.52559 (13)	0.7415 (3)	0.0530 (8)	
N4	0.13007 (19)	0.52554 (11)	0.9956 (3)	0.0450 (7)	
N5	0.1421 (2)	0.41785 (12)	1.0816 (3)	0.0582 (9)	
N6	0.5545 (2)	0.34561 (14)	0.3731 (3)	0.0725 (11)	
H6A	0.5718	0.3760	0.4021	0.087*	
H6B	0.5772	0.3171	0.4061	0.087*	
N7	0.4613 (2)	0.38960 (11)	0.2319 (3)	0.0518 (8)	
H7N	0.4222	0.3901	0.1688	0.062*	
N8	0.49081 (19)	0.43564 (12)	0.2853 (3)	0.0463 (8)	
N9	0.37777 (18)	0.45074 (11)	0.0357 (2)	0.0420 (7)	
N10	0.37666 (19)	0.56254 (11)	−0.0212 (3)	0.0465 (8)	
O1	0.2779 (2)	0.63300 (12)	1.3327 (3)	0.0755 (9)	
H1	0.2940	0.6551	1.3849	0.113*	
O2	0.3298 (2)	0.57660 (12)	1.4791 (3)	0.0819 (10)	
O3	0.1741 (2)	0.42962 (13)	−0.4565 (2)	0.0813 (10)	
O4	0.2167 (2)	0.36383 (11)	−0.3284 (2)	0.0666 (8)	
H4	0.1962	0.3455	−0.3862	0.100*	
O5	0.3139 (2)	0.78780 (12)	0.9794 (3)	0.0979 (12)	
O6	0.1697 (2)	0.29926 (12)	0.4872 (3)	0.0801 (9)	

S1	0.06694 (10)	0.67658 (5)	0.77726 (13)	0.0971 (5)
S2	0.45768 (10)	0.28637 (5)	0.21016 (13)	0.0931 (5)
C1	0.0248 (3)	0.61963 (16)	0.7224 (4)	0.0575 (11)
C2	0.0437 (2)	0.48613 (16)	0.8122 (4)	0.0497 (10)
C3	0.0197 (3)	0.43073 (17)	0.7843 (4)	0.0589 (11)
C4	−0.0273 (3)	0.40903 (19)	0.6791 (4)	0.0700 (13)
H4A	−0.0483	0.4306	0.6137	0.084*
C5	−0.0416 (3)	0.3553 (2)	0.6751 (6)	0.0921 (17)
H5	−0.0719	0.3402	0.6056	0.111*
C6	−0.0116 (4)	0.3230 (2)	0.7735 (6)	0.0954 (18)
H6	−0.0233	0.2867	0.7690	0.114*
C7	0.0357 (3)	0.34383 (18)	0.8793 (5)	0.0808 (14)
H7	0.0554	0.3221	0.9450	0.097*
C8	0.0523 (3)	0.39833 (16)	0.8825 (4)	0.0585 (11)
C9	0.0995 (2)	0.43138 (15)	0.9757 (4)	0.0517 (10)
C10	0.0951 (2)	0.48527 (15)	0.9334 (3)	0.0473 (9)
C11	0.1742 (2)	0.51258 (14)	1.1071 (3)	0.0432 (9)
C12	0.1806 (3)	0.45922 (15)	1.1484 (3)	0.0509 (10)
C13	0.2272 (3)	0.44885 (17)	1.2644 (4)	0.0619 (11)
H13	0.2332	0.4141	1.2923	0.074*
C14	0.2633 (3)	0.48922 (17)	1.3356 (4)	0.0577 (11)
H14	0.2935	0.4816	1.4116	0.069*
C15	0.2557 (2)	0.54232 (16)	1.2961 (3)	0.0498 (9)
C16	0.2118 (2)	0.55346 (15)	1.1823 (3)	0.0489 (9)
H16	0.2072	0.5884	1.1553	0.059*
C17	0.2914 (3)	0.58507 (17)	1.3783 (4)	0.0581 (11)
C18	0.4934 (3)	0.34274 (15)	0.2781 (3)	0.0549 (10)
C19	0.4676 (2)	0.47900 (14)	0.2286 (3)	0.0414 (8)
C20	0.4953 (2)	0.53187 (14)	0.2728 (3)	0.0437 (9)
C21	0.5442 (2)	0.54663 (16)	0.3814 (3)	0.0529 (10)
H21	0.5641	0.5213	0.4393	0.063*
C22	0.5626 (3)	0.59962 (17)	0.4015 (4)	0.0605 (11)
H22	0.5948	0.6102	0.4742	0.073*
C23	0.5337 (3)	0.63772 (18)	0.3143 (4)	0.0695 (12)
H23	0.5472	0.6733	0.3295	0.083*
C24	0.4850 (3)	0.62332 (16)	0.2053 (4)	0.0619 (11)
H24	0.4658	0.6488	0.1474	0.074*
C25	0.4657 (2)	0.57004 (14)	0.1849 (3)	0.0454 (9)
C26	0.4162 (2)	0.54262 (14)	0.0815 (3)	0.0424 (9)
C27	0.4160 (2)	0.48737 (14)	0.1083 (3)	0.0407 (8)
C28	0.3352 (2)	0.47041 (14)	−0.0723 (3)	0.0408 (8)
C29	0.3351 (2)	0.52530 (14)	−0.0997 (3)	0.0430 (9)
C30	0.2910 (2)	0.54193 (15)	−0.2124 (3)	0.0514 (10)
H30	0.2896	0.5778	−0.2324	0.062*
C31	0.2506 (2)	0.50632 (17)	−0.2914 (3)	0.0526 (10)
H31	0.2218	0.5182	−0.3652	0.063*
C32	0.2509 (2)	0.45140 (15)	−0.2652 (3)	0.0478 (9)
C33	0.2926 (2)	0.43428 (15)	−0.1562 (3)	0.0438 (9)
H33	0.2928	0.3983	−0.1374	0.053*

C34	0.2103 (3)	0.41428 (18)	−0.3589 (3)	0.0554 (11)
C35	0.2086 (7)	0.7220 (3)	1.0792 (7)	0.180 (4)
C36	0.3075 (5)	0.6881 (3)	0.9071 (8)	0.159 (3)
C37	0.2878 (4)	0.2273 (2)	0.4219 (5)	0.114 (2)
C38	0.1898 (4)	0.2855 (2)	0.2531 (4)	0.0961 (17)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S3	0.0611 (10)	0.0390 (8)	0.0715 (10)	−0.0050 (6)	−0.0128 (7)	0.0021 (6)
S4	0.0797 (12)	0.0654 (10)	0.0545 (8)	−0.0086 (7)	−0.0003 (7)	−0.0078 (6)
N1	0.075 (3)	0.078 (3)	0.060 (2)	0.000 (2)	−0.0169 (19)	0.0062 (19)
N2	0.050 (2)	0.057 (2)	0.0539 (19)	−0.0005 (16)	−0.0135 (15)	−0.0034 (16)
N3	0.0436 (19)	0.058 (2)	0.057 (2)	−0.0015 (16)	0.0012 (16)	−0.0157 (17)
N4	0.0408 (18)	0.0445 (18)	0.0500 (18)	0.0012 (14)	0.0062 (14)	−0.0036 (15)
N5	0.057 (2)	0.046 (2)	0.074 (2)	−0.0028 (16)	0.0180 (19)	0.0067 (18)
N6	0.092 (3)	0.052 (2)	0.066 (2)	0.0160 (19)	−0.024 (2)	0.0004 (17)
N7	0.060 (2)	0.0424 (19)	0.0503 (18)	−0.0031 (15)	−0.0105 (15)	−0.0024 (15)
N8	0.0474 (19)	0.0464 (19)	0.0440 (17)	0.0010 (15)	−0.0007 (14)	−0.0046 (15)
N9	0.0411 (17)	0.0447 (18)	0.0398 (16)	0.0014 (14)	0.0030 (14)	−0.0022 (14)
N10	0.0443 (18)	0.0463 (18)	0.0490 (18)	0.0007 (14)	0.0047 (15)	0.0026 (15)
O1	0.087 (2)	0.065 (2)	0.070 (2)	0.0067 (17)	−0.0174 (17)	−0.0044 (16)
O2	0.103 (3)	0.081 (2)	0.0570 (18)	−0.0100 (18)	−0.0135 (18)	0.0059 (16)
O3	0.095 (2)	0.100 (2)	0.0446 (17)	−0.0271 (18)	−0.0159 (16)	0.0102 (16)
O4	0.082 (2)	0.065 (2)	0.0500 (16)	−0.0034 (16)	−0.0073 (15)	−0.0073 (15)
O5	0.120 (3)	0.055 (2)	0.108 (3)	−0.0283 (18)	−0.040 (2)	0.0101 (18)
O6	0.101 (3)	0.080 (2)	0.0588 (17)	0.0047 (18)	0.0047 (17)	−0.0224 (16)
S1	0.1048 (11)	0.0569 (8)	0.1182 (11)	−0.0131 (7)	−0.0446 (9)	0.0123 (7)
S2	0.1140 (12)	0.0498 (7)	0.1095 (11)	−0.0076 (7)	−0.0181 (9)	−0.0135 (7)
C1	0.048 (2)	0.065 (3)	0.057 (2)	0.006 (2)	−0.0068 (19)	0.006 (2)
C2	0.035 (2)	0.054 (3)	0.061 (2)	−0.0012 (18)	0.0104 (18)	−0.014 (2)
C3	0.037 (2)	0.063 (3)	0.079 (3)	−0.0070 (19)	0.016 (2)	−0.021 (2)
C4	0.055 (3)	0.073 (3)	0.084 (3)	−0.012 (2)	0.013 (2)	−0.030 (3)
C5	0.075 (4)	0.088 (4)	0.114 (5)	−0.023 (3)	0.018 (3)	−0.045 (4)
C6	0.091 (4)	0.067 (3)	0.134 (5)	−0.030 (3)	0.039 (4)	−0.043 (4)
C7	0.080 (3)	0.060 (3)	0.108 (4)	−0.016 (3)	0.032 (3)	−0.013 (3)
C8	0.050 (3)	0.053 (3)	0.074 (3)	−0.008 (2)	0.019 (2)	−0.011 (2)
C9	0.039 (2)	0.047 (2)	0.072 (3)	−0.0016 (17)	0.021 (2)	−0.003 (2)
C10	0.035 (2)	0.049 (2)	0.059 (2)	−0.0006 (17)	0.0090 (18)	−0.0066 (19)
C11	0.038 (2)	0.048 (2)	0.046 (2)	0.0015 (16)	0.0119 (17)	0.0011 (18)
C12	0.049 (2)	0.051 (2)	0.055 (2)	0.0004 (19)	0.0156 (19)	0.0034 (19)
C13	0.065 (3)	0.060 (3)	0.063 (3)	0.001 (2)	0.016 (2)	0.020 (2)
C14	0.054 (3)	0.069 (3)	0.051 (2)	0.000 (2)	0.009 (2)	0.014 (2)
C15	0.041 (2)	0.060 (3)	0.049 (2)	−0.0007 (18)	0.0080 (18)	0.0031 (19)
C16	0.042 (2)	0.052 (2)	0.053 (2)	0.0022 (18)	0.0105 (18)	0.0032 (19)
C17	0.053 (3)	0.069 (3)	0.052 (2)	0.004 (2)	0.005 (2)	0.004 (2)
C18	0.063 (3)	0.046 (2)	0.055 (2)	0.002 (2)	0.003 (2)	0.0006 (19)
C19	0.036 (2)	0.049 (2)	0.0389 (19)	−0.0010 (16)	0.0036 (16)	−0.0031 (17)

C20	0.039 (2)	0.046 (2)	0.046 (2)	0.0025 (17)	0.0063 (17)	−0.0060 (18)
C21	0.051 (2)	0.057 (3)	0.050 (2)	−0.0055 (19)	0.0042 (19)	−0.0063 (19)
C22	0.056 (3)	0.062 (3)	0.063 (3)	−0.005 (2)	0.003 (2)	−0.016 (2)
C23	0.061 (3)	0.054 (3)	0.092 (3)	−0.007 (2)	0.001 (2)	−0.019 (3)
C24	0.058 (3)	0.050 (3)	0.076 (3)	0.004 (2)	−0.003 (2)	0.000 (2)
C25	0.039 (2)	0.045 (2)	0.053 (2)	0.0015 (16)	0.0066 (17)	−0.0046 (18)
C26	0.036 (2)	0.043 (2)	0.049 (2)	0.0047 (16)	0.0069 (17)	0.0021 (17)
C27	0.035 (2)	0.045 (2)	0.043 (2)	0.0020 (16)	0.0082 (16)	−0.0019 (17)
C28	0.037 (2)	0.053 (2)	0.0333 (19)	0.0033 (16)	0.0069 (16)	0.0030 (16)
C29	0.041 (2)	0.047 (2)	0.042 (2)	0.0038 (17)	0.0078 (17)	0.0026 (17)
C30	0.054 (2)	0.051 (2)	0.049 (2)	0.0052 (19)	0.0083 (19)	0.0083 (19)
C31	0.047 (2)	0.074 (3)	0.037 (2)	0.004 (2)	0.0025 (18)	0.008 (2)
C32	0.040 (2)	0.063 (3)	0.041 (2)	−0.0038 (18)	0.0080 (17)	0.0012 (19)
C33	0.042 (2)	0.051 (2)	0.039 (2)	−0.0021 (17)	0.0023 (16)	0.0004 (17)
C34	0.048 (2)	0.082 (3)	0.037 (2)	−0.010 (2)	0.0069 (18)	0.001 (2)
C35	0.239 (10)	0.180 (8)	0.134 (6)	−0.072 (7)	0.082 (7)	0.012 (6)
C36	0.131 (6)	0.094 (5)	0.251 (9)	0.032 (4)	0.014 (6)	−0.070 (6)
C37	0.127 (5)	0.107 (5)	0.109 (4)	0.054 (4)	0.013 (4)	0.018 (4)
C38	0.126 (5)	0.110 (4)	0.048 (3)	0.014 (3)	−0.007 (3)	−0.007 (3)

Geometric parameters (Å, °)

S3—O5	1.502 (3)	C3—C4	1.403 (6)
S3—C35	1.743 (7)	C4—C5	1.371 (7)
S3—C36	1.774 (6)	C4—H4A	0.9300
S4—O6	1.530 (3)	C5—C6	1.393 (7)
S4—C38	1.788 (5)	C5—H5	0.9300
S4—C37	1.800 (6)	C6—C7	1.402 (7)
S3'—C36	1.46 (2)	C6—H6	0.9300
S3'—O5	1.62 (2)	C7—C8	1.395 (6)
S3'—C35	1.77 (2)	C7—H7	0.9300
S4'—O6	1.301 (19)	C8—C9	1.448 (5)
S4'—C37	1.721 (19)	C9—C10	1.435 (5)
S4'—C38	1.794 (19)	C11—C16	1.403 (5)
N1—C1	1.324 (5)	C11—C12	1.420 (5)
N1—H1A	0.8600	C12—C13	1.416 (5)
N1—H1B	0.8600	C13—C14	1.362 (6)
N2—C1	1.357 (5)	C13—H13	0.9300
N2—N3	1.364 (4)	C14—C15	1.408 (5)
N2—H2	0.8600	C14—H14	0.9300
N3—C2	1.289 (5)	C15—C16	1.383 (5)
N4—C10	1.304 (4)	C15—C17	1.472 (5)
N4—C11	1.373 (4)	C16—H16	0.9300
N5—C9	1.316 (5)	C19—C20	1.464 (5)
N5—C12	1.368 (5)	C19—C27	1.477 (5)
N6—C18	1.318 (5)	C20—C21	1.388 (5)
N6—H6A	0.8600	C20—C25	1.404 (5)
N6—H6B	0.8600	C21—C22	1.377 (5)
N7—C18	1.354 (4)	C21—H21	0.9300

N7—N8	1.354 (4)	C22—C23	1.396 (6)
N7—H7N	0.8600	C22—H22	0.9300
N8—C19	1.289 (4)	C23—C24	1.387 (6)
N9—C27	1.314 (4)	C23—H23	0.9300
N9—C28	1.383 (4)	C24—C25	1.387 (5)
N10—C26	1.321 (4)	C24—H24	0.9300
N10—C29	1.379 (4)	C25—C26	1.466 (5)
O1—C17	1.316 (5)	C26—C27	1.424 (5)
O1—H1	0.8200	C28—C33	1.402 (5)
O2—C17	1.215 (5)	C28—C29	1.416 (5)
O3—C34	1.216 (4)	C29—C30	1.409 (5)
O4—C34	1.316 (5)	C30—C31	1.349 (5)
O4—H4	0.8200	C30—H30	0.9300
S1—C1	1.656 (4)	C31—C32	1.414 (5)
S2—C18	1.668 (4)	C31—H31	0.9300
C2—C3	1.466 (5)	C32—C33	1.364 (5)
C2—C10	1.471 (5)	C32—C34	1.478 (5)
C3—C8	1.403 (6)	C33—H33	0.9300
O5—S3—C35	106.3 (3)	C14—C13—C12	120.8 (4)
O5—S3—C36	106.8 (3)	C14—C13—H13	119.6
C35—S3—C36	98.0 (4)	C12—C13—H13	119.6
O6—S4—C38	105.7 (2)	C13—C14—C15	121.1 (4)
O6—S4—C37	106.5 (2)	C13—C14—H14	119.4
C38—S4—C37	96.7 (3)	C15—C14—H14	119.4
C36—S3'—O5	117.3 (15)	C16—C15—C14	119.3 (4)
C36—S3'—C35	110.0 (14)	C16—C15—C17	121.2 (4)
O5—S3'—C35	100.0 (12)	C14—C15—C17	119.5 (4)
O6—S4'—C37	123.7 (15)	C15—C16—C11	120.7 (4)
O6—S4'—C38	116.8 (14)	C15—C16—H16	119.7
C37—S4'—C38	99.4 (10)	C11—C16—H16	119.7
C1—N1—H1A	120.0	O2—C17—O1	123.4 (4)
C1—N1—H1B	120.0	O2—C17—C15	122.8 (4)
H1A—N1—H1B	120.0	O1—C17—C15	113.9 (3)
C1—N2—N3	122.1 (3)	N6—C18—N7	116.1 (3)
C1—N2—H2	119.0	N6—C18—S2	124.5 (3)
N3—N2—H2	119.0	N7—C18—S2	119.3 (3)
C2—N3—N2	115.8 (3)	N8—C19—C20	123.9 (3)
C10—N4—C11	114.5 (3)	N8—C19—C27	130.2 (3)
C9—N5—C12	114.7 (3)	C20—C19—C27	105.8 (3)
C18—N6—H6A	120.0	C21—C20—C25	120.8 (3)
C18—N6—H6B	120.0	C21—C20—C19	129.6 (3)
H6A—N6—H6B	120.0	C25—C20—C19	109.6 (3)
C18—N7—N8	119.8 (3)	C22—C21—C20	118.5 (4)
C18—N7—H7N	120.1	C22—C21—H21	120.7
N8—N7—H7N	120.1	C20—C21—H21	120.7
C19—N8—N7	117.1 (3)	C21—C22—C23	120.9 (4)
C27—N9—C28	113.9 (3)	C21—C22—H22	119.5
C26—N10—C29	114.3 (3)	C23—C22—H22	119.5
C17—O1—H1	109.5	C24—C23—C22	120.9 (4)

C34—O4—H4	109.5	C24—C23—H23	119.5
S3—O5—S3'	47.4 (9)	C22—C23—H23	119.5
S4'—O6—S4	46.1 (11)	C23—C24—C25	118.4 (4)
N1—C1—N2	116.6 (4)	C23—C24—H24	120.8
N1—C1—S1	125.0 (3)	C25—C24—H24	120.8
N2—C1—S1	118.5 (3)	C24—C25—C20	120.4 (3)
N3—C2—C3	124.3 (4)	C24—C25—C26	131.5 (4)
N3—C2—C10	130.0 (3)	C20—C25—C26	108.1 (3)
C3—C2—C10	105.6 (4)	N10—C26—C27	122.7 (3)
C8—C3—C4	121.0 (4)	N10—C26—C25	129.2 (3)
C8—C3—C2	109.6 (4)	C27—C26—C25	108.1 (3)
C4—C3—C2	129.4 (4)	N9—C27—C26	124.8 (3)
C5—C4—C3	118.3 (5)	N9—C27—C19	126.9 (3)
C5—C4—H4A	120.8	C26—C27—C19	108.3 (3)
C3—C4—H4A	120.8	N9—C28—C33	118.1 (3)
C4—C5—C6	121.1 (5)	N9—C28—C29	121.6 (3)
C4—C5—H5	119.5	C33—C28—C29	120.3 (3)
C6—C5—H5	119.5	N10—C29—C30	119.3 (3)
C5—C6—C7	121.5 (5)	N10—C29—C28	122.7 (3)
C5—C6—H6	119.2	C30—C29—C28	117.9 (3)
C7—C6—H6	119.2	C31—C30—C29	120.6 (4)
C8—C7—C6	117.5 (5)	C31—C30—H30	119.7
C8—C7—H7	121.2	C29—C30—H30	119.7
C6—C7—H7	121.2	C30—C31—C32	121.8 (3)
C7—C8—C3	120.5 (4)	C30—C31—H31	119.1
C7—C8—C9	131.0 (4)	C32—C31—H31	119.1
C3—C8—C9	108.5 (4)	C33—C32—C31	118.8 (3)
N5—C9—C10	122.4 (4)	C33—C32—C34	122.2 (4)
N5—C9—C8	129.5 (4)	C31—C32—C34	118.9 (3)
C10—C9—C8	108.1 (4)	C32—C33—C28	120.6 (3)
N4—C10—C9	124.2 (4)	C32—C33—H33	119.7
N4—C10—C2	127.6 (3)	C28—C33—H33	119.7
C9—C10—C2	108.2 (3)	O3—C34—O4	123.2 (4)
N4—C11—C16	118.7 (3)	O3—C34—C32	122.0 (4)
N4—C11—C12	121.5 (3)	O4—C34—C32	114.8 (3)
C16—C11—C12	119.8 (3)	S3—C35—S3'	42.0 (8)
N5—C12—C13	119.0 (4)	S3'—C36—S3	44.5 (10)
N5—C12—C11	122.6 (3)	S4'—C37—S4	37.3 (8)
C13—C12—C11	118.3 (4)	S4—C38—S4'	36.7 (8)
C1—N2—N3—C2	−174.0 (3)	N7—N8—C19—C27	−3.4 (5)
C18—N7—N8—C19	170.9 (3)	N8—C19—C20—C21	−4.5 (6)
C35—S3—O5—S3'	61.9 (11)	C27—C19—C20—C21	179.2 (3)
C36—S3—O5—S3'	−42.0 (10)	N8—C19—C20—C25	174.6 (3)
C36—S3'—O5—S3	61.0 (15)	C27—C19—C20—C25	−1.7 (4)
C35—S3'—O5—S3	−57.7 (9)	C25—C20—C21—C22	0.4 (5)
C37—S4'—O6—S4	62.5 (16)	C19—C20—C21—C22	179.4 (4)
C38—S4'—O6—S4	−61.5 (13)	C20—C21—C22—C23	−0.6 (6)
C38—S4—O6—S4'	54.8 (12)	C21—C22—C23—C24	0.4 (6)
C37—S4—O6—S4'	−47.3 (12)	C22—C23—C24—C25	0.0 (6)

N3—N2—C1—N1	2.1 (6)	C23—C24—C25—C20	−0.2 (6)
N3—N2—C1—S1	−178.7 (3)	C23—C24—C25—C26	179.5 (4)
N2—N3—C2—C3	−178.7 (3)	C21—C20—C25—C24	0.0 (5)
N2—N3—C2—C10	2.9 (6)	C19—C20—C25—C24	−179.2 (3)
N3—C2—C3—C8	−177.5 (4)	C21—C20—C25—C26	−179.8 (3)
C10—C2—C3—C8	1.3 (4)	C19—C20—C25—C26	1.0 (4)
N3—C2—C3—C4	3.0 (6)	C29—N10—C26—C27	0.3 (5)
C10—C2—C3—C4	−178.3 (4)	C29—N10—C26—C25	−179.7 (3)
C8—C3—C4—C5	0.7 (6)	C24—C25—C26—N10	0.3 (7)
C2—C3—C4—C5	−179.8 (4)	C20—C25—C26—N10	−179.9 (3)
C3—C4—C5—C6	1.0 (7)	C24—C25—C26—C27	−179.7 (4)
C4—C5—C6—C7	−1.2 (8)	C20—C25—C26—C27	0.1 (4)
C5—C6—C7—C8	−0.3 (7)	C28—N9—C27—C26	−0.6 (5)
C6—C7—C8—C3	2.0 (6)	C28—N9—C27—C19	−179.0 (3)
C6—C7—C8—C9	−179.0 (4)	N10—C26—C27—N9	0.2 (5)
C4—C3—C8—C7	−2.2 (6)	C25—C26—C27—N9	−179.9 (3)
C2—C3—C8—C7	178.2 (3)	N10—C26—C27—C19	178.8 (3)
C4—C3—C8—C9	178.6 (3)	C25—C26—C27—C19	−1.2 (4)
C2—C3—C8—C9	−1.0 (4)	N8—C19—C27—N9	4.4 (6)
C12—N5—C9—C10	0.8 (5)	C20—C19—C27—N9	−179.6 (3)
C12—N5—C9—C8	179.4 (4)	N8—C19—C27—C26	−174.2 (3)
C7—C8—C9—N5	2.5 (7)	C20—C19—C27—C26	1.8 (4)
C3—C8—C9—N5	−178.4 (4)	C27—N9—C28—C33	179.7 (3)
C7—C8—C9—C10	−178.7 (4)	C27—N9—C28—C29	0.7 (5)
C3—C8—C9—C10	0.3 (4)	C26—N10—C29—C30	−179.8 (3)
C11—N4—C10—C9	1.0 (5)	C26—N10—C29—C28	−0.2 (5)
C11—N4—C10—C2	179.7 (3)	N9—C28—C29—N10	−0.3 (5)
N5—C9—C10—N4	−1.8 (6)	C33—C28—C29—N10	−179.3 (3)
C8—C9—C10—N4	179.3 (3)	N9—C28—C29—C30	179.3 (3)
N5—C9—C10—C2	179.3 (3)	C33—C28—C29—C30	0.3 (5)
C8—C9—C10—C2	0.5 (4)	N10—C29—C30—C31	179.3 (3)
N3—C2—C10—N4	−1.2 (6)	C28—C29—C30—C31	−0.4 (5)
C3—C2—C10—N4	−179.8 (3)	C29—C30—C31—C32	−0.1 (6)
N3—C2—C10—C9	177.6 (4)	C30—C31—C32—C33	0.6 (5)
C3—C2—C10—C9	−1.0 (4)	C30—C31—C32—C34	−176.3 (3)
C10—N4—C11—C16	−178.1 (3)	C31—C32—C33—C28	−0.7 (5)
C10—N4—C11—C12	0.5 (5)	C34—C32—C33—C28	176.2 (3)
C9—N5—C12—C13	179.3 (3)	N9—C28—C33—C32	−178.8 (3)
C9—N5—C12—C11	0.6 (5)	C29—C28—C33—C32	0.2 (5)
N4—C11—C12—N5	−1.4 (5)	C33—C32—C34—O3	−179.2 (4)
C16—C11—C12—N5	177.2 (3)	C31—C32—C34—O3	−2.4 (6)
N4—C11—C12—C13	180.0 (3)	C33—C32—C34—O4	0.7 (5)
C16—C11—C12—C13	−1.4 (5)	C31—C32—C34—O4	177.5 (3)
N5—C12—C13—C14	−177.4 (4)	O5—S3—C35—S3'	−62.6 (10)
C11—C12—C13—C14	1.3 (6)	C36—S3—C35—S3'	47.7 (10)
C12—C13—C14—C15	−0.2 (6)	C36—S3'—C35—S3	−70.8 (14)
C13—C14—C15—C16	−0.9 (6)	O5—S3'—C35—S3	53.3 (9)
C13—C14—C15—C17	176.7 (4)	O5—S3'—C36—S3	−51.0 (12)
C14—C15—C16—C11	0.7 (5)	C35—S3'—C36—S3	62.3 (12)

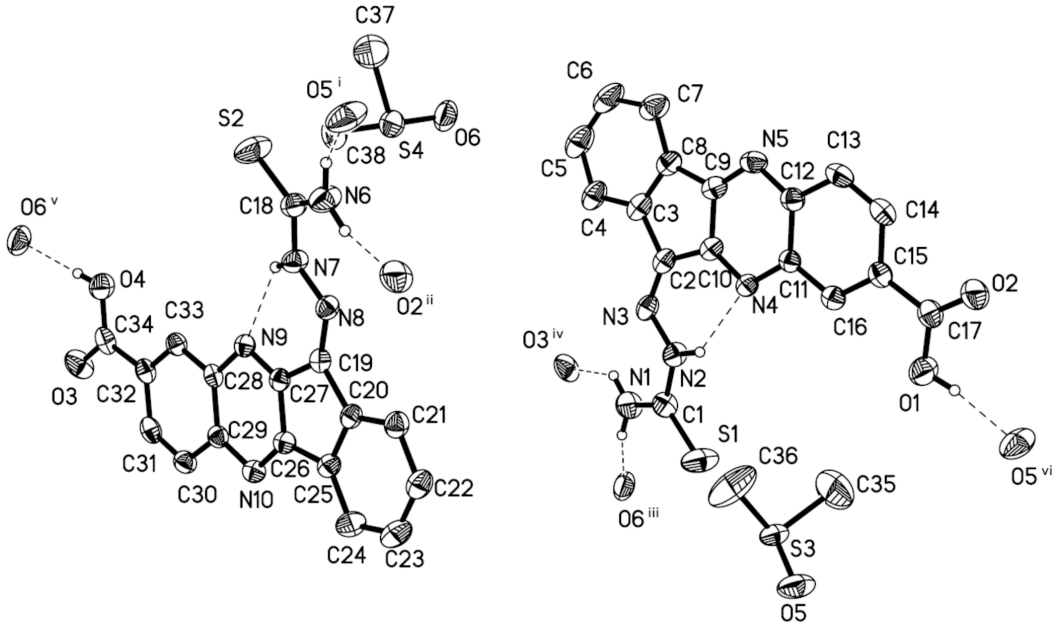
C17—C15—C16—C11	−176.8 (3)	O5—S3—C36—S3'	51.1 (12)
N4—C11—C16—C15	179.0 (3)	C35—S3—C36—S3'	−58.7 (12)
C12—C11—C16—C15	0.4 (5)	O6—S4'—C37—S4	−63.7 (18)
C16—C15—C17—O2	179.6 (4)	C38—S4'—C37—S4	67.7 (10)
C14—C15—C17—O2	2.1 (6)	O6—S4—C37—S4'	41.4 (10)
C16—C15—C17—O1	−0.5 (5)	C38—S4—C37—S4'	−67.2 (11)
C14—C15—C17—O1	−178.1 (3)	O6—S4—C38—S4'	−45.6 (10)
N8—N7—C18—N6	−1.5 (5)	C37—S4—C38—S4'	63.7 (10)
N8—N7—C18—S2	−179.0 (3)	O6—S4'—C38—S4	64.9 (16)
N7—N8—C19—C20	−178.8 (3)	C37—S4'—C38—S4	−70.7 (11)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N7—H7N···N9	0.86	2.17	2.838 (4)	134
N2—H2···N4	0.86	2.12	2.807 (4)	137
N6—H6B···O5^{i}	0.86	2.09	2.820 (4)	143
N6—H6A···O2^{ii}	0.86	2.21	2.982 (5)	149
N1—H1B···O6^{iii}	0.86	2.36	3.131 (5)	150
N1—H1A···O3^{iv}	0.86	2.30	2.872 (4)	124
O4—H4···O6^{v}	0.82	1.83	2.642 (4)	170
O1—H1···O5^{vi}	0.82	1.78	2.591 (4)	168

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

Fig. 1


C37
O5 i
S2
C38
S4
O6
C18
N6
O6 v
O4
N7
O2 ii
C33
C34
N8
N9
C19
O3
C32
C28
C27
C20
C21
C29
C31
C26
C22
C30
N10
C25
C24
C23
C6
C7
C5
C8
N5
C13
C9
C12
C4
C3
C14
C2
C10
C15
N4
C11
O2
N3
C16
C17
O3 iv
N1
N2
O1
C1
S1
C36
C35
O5 vi
O6 iii
S3
O5