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2′,2′-(Ethane-1,2-diylidene)bis(2-hydroxybenzhydrazide) dimethyl sulfoxide disolvate

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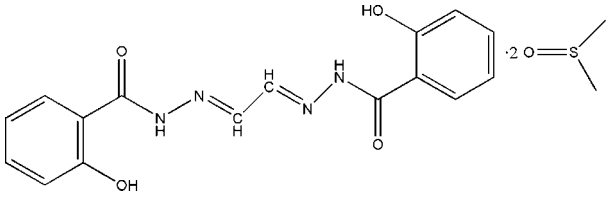
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.004 Å; R factor = 0.044; wR factor = 0.124; data-to-parameter ratio = 13.7.

The title molecule, $C_{16}H_{14}N_4O_4$, lies on a crystallographic inversion center and all bond lengths and angles show normal values. The crystal structure is stabilized by intermolecular hydrogen bonds.

Related literature

For related literature, see: Chandra & Sangeetika (2004); Pasini *et al.* (1996); Zurek & Karst (1997).



Experimental

Crystal data

$C_{16}H_{14}N_4O_4\cdot 2C_2H_6OS$
M_r = 482.57
Triclinic, $P\overline{1}$
a = 7.9739 (7) Å
b = 8.5125 (8) Å
c = 9.0215 (8) Å
α = 78.031 (1)°
β = 85.704 (1)°
γ = 85.295 (1)°
V = 595.97 (9) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.27 mm^{-1}
T = 294 (2) K
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: none
3262 measured reflections
2071 independent reflections
1684 reflections with $I > 2\sigma(I)$
R_{int} = 0.057

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.124
S = 1.09
2071 reflections
151 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O4^{i}	0.83 (3)	2.05 (3)	2.858 (2)	164 (3)
C6—H6···S1^{i}	0.93	2.77	3.636 (2)	156
C6—H6···O4^{i}	0.93	2.57	3.449 (3)	157
C8—H8···O4^{i}	0.93	2.44	3.213 (3)	141
O1—H1···O9	0.82	1.81	2.538 (2)	147
C9—H9B···N2	0.96	2.61	3.465 (3)	149

Symmetry code: (i) $x, y, z+1$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; 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: *SHELXTL*.

This work was supported by Hubei Education Government of China (grant No. 20040131).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2367).

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supplementary materials

Acta Cryst. (2007). E**63**, o2696 [**doi:10.1107/S1600536807019393**]

2',2'-(Ethane-1,2-diylidene)bis(2-hydroxybenzhydrazide) dimethyl sulfoxide disolvate

F.-H. Luo, S.-M. Shi, M.-N. Cao, C.-X. Cheng and Z.-Q. Hu

Comment

Bishydrazone ligands and complexs with glyoxal have been intensively studied, due to their interesting biological and material properties (Alessandro *et al.*, 1996; Gabriela & Uwe, 1997; Sulekh *et al.*, 2004). We report herein the synthesis and crystal structure of the title compound (I). The molecular structure of (I) (Fig. 1) has crystallographic inversion symmetry. In the crystal structure, intermolecular N–H–O hydrogen bonds link hydrazone molecules with two dimethyl sulfoxide solvent molecules, forming three component clusters. These clusters are further connected by weak intermolecular C–H–S and C–H–O hydrogen bonds (Fig. 2; Table 1).

Experimental

To a solution of salicylhydrazide (3.04 g, 20 mmol)in absolute ethanol (40 ml) a solution of glyoxal (40%)(1.55 g, 10 mmol) was added at 323 K .The mixture was left to react at reflux for 6 h, then the yellow product in the form of needle was filtered, washed with hot ethanol (20 ml portion) three times and dried in vacuo. Crystals suitable for X-ray diffraction were obtained from a dimethyl sulfoxide solution of (I) over a period of approximately three weeks.

Refinement

After their location in the difference map, H-atoms bonded to C and O were fixed geometrically at ideal positions and allowed to ride on the parent atoms with C—H = 0.93 - 0.96Å and O—H = 0.82 Å. The positional parameters of the H atom bonded to N1 were allowed to refine. All H atoms were included in the refinement with U_{iso}(H) = 1.2U_{eq}(C, N)or U_{iso}(H)= 1.5U_{eq}(C_{methyl} and O).

Figures

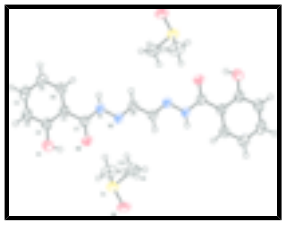

Fig. 1. Molecular structure of (I) showing 30% probability displacement ellipsoids. Unlabeled atoms are related by the symmetry operator (-x, -y, 2-z)

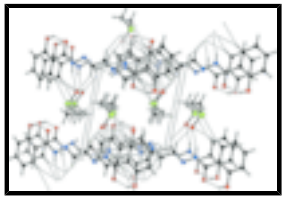

Fig. 2. Partial packing plot of (I) with dashed lines indicating hydrogen bonds.

2',2'-(Ethane-1,2-diylidene)bis(2-hydroxybenzohydrazide) dimethyl sulfoxide disolvate

Crystal data

$C_{16}H_{14}N_4O_4·2C_2H_6OS$	$Z = 1$
$M_r = 482.57$	$F_{000} = 254$
Triclinic, $P\overline{1}$	$D_x = 1.345$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 7.9739$ (7) Å	Cell parameters from 1568 reflections
$b = 8.5125$ (8) Å	$\theta = 2.3$–28.2º
$c = 9.0215$ (8) Å	$\mu = 0.27$ mm^{-1}
$\alpha = 78.031$ (1)º	$T = 294$ (2) K
$\beta = 85.704$ (1)º	Block, colorless
$\gamma = 85.295$ (1)º	0.30 × 0.20 × 0.20 mm
$V = 595.97$ (9) Å^3	

Data collection

Bruker SMART CCD diffractometer	1684 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.057$
Monochromator: graphite	$\theta_{max} = 25.0$º
$T = 294(2)$ K	$\theta_{min} = 2.3$º
φ and ω scans	$h = -9\rightarrow9$
Absorption correction: none	$k = -8\rightarrow10$
3262 measured reflections	$l = -10\rightarrow10$
2071 independent reflections	

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0532P)^2 + 0.1619P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.044$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.124$	$\Delta\rho_{max} = 0.30$ e Å^{-3}
$S = 1.09$	$\Delta\rho_{min} = -0.30$ e Å^{-3}
2071 reflections	Extinction correction: none
151 parameters	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	
Hydrogen site location: inferred from neighbouring sites	

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}
C1	0.2762 (3)	0.5595 (2)	0.9492 (2)	0.0389 (5)
C2	0.3586 (3)	0.6821 (3)	0.8469 (3)	0.0461 (6)
C3	0.4365 (3)	0.7962 (3)	0.9019 (3)	0.0567 (7)
H3	0.4924	0.8759	0.8348	0.068*
C4	0.4316 (3)	0.7921 (3)	1.0542 (3)	0.0560 (7)
H4	0.4839	0.8696	1.0897	0.067*
C5	0.3505 (3)	0.6748 (3)	1.1563 (3)	0.0537 (6)
H5	0.3480	0.6729	1.2599	0.064*
C6	0.2734 (3)	0.5608 (3)	1.1030 (3)	0.0470 (6)
H6	0.2178	0.4822	1.1719	0.056*
C7	0.2029 (3)	0.4338 (3)	0.8875 (2)	0.0397 (5)
C8	0.0311 (3)	0.0654 (2)	1.0270 (2)	0.0394 (5)
H8	0.0317	0.0630	1.1305	0.047*
C9	−0.0242 (4)	0.2384 (4)	0.5576 (3)	0.0732 (8)
H9A	−0.1146	0.3043	0.5067	0.110*
H9B	−0.0134	0.2670	0.6538	0.110*
H9C	−0.0480	0.1272	0.5735	0.110*
C10	0.2997 (4)	0.1336 (4)	0.5665 (3)	0.0784 (9)
H10A	0.2641	0.0265	0.5783	0.118*
H10B	0.2947	0.1632	0.6639	0.118*
H10C	0.4133	0.1376	0.5230	0.118*
N2	0.0853 (2)	0.1844 (2)	0.9302 (2)	0.0407 (4)
N1	0.1446 (2)	0.3054 (2)	0.9877 (2)	0.0391 (4)
O1	0.3692 (3)	0.6902 (2)	0.6955 (2)	0.0726 (6)
H1	0.3241	0.6141	0.6766	0.109*
O4	0.1500 (3)	0.1961 (2)	0.30909 (19)	0.0745 (6)
O9	0.1975 (2)	0.4444 (2)	0.74889 (18)	0.0603 (5)
S1	0.16523 (9)	0.26925 (8)	0.44551 (7)	0.0586 (3)
H1A	0.152 (3)	0.292 (3)	1.081 (3)	0.070*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0413 (12)	0.0325 (11)	0.0437 (12)	−0.0057 (9)	−0.0076 (9)	−0.0063 (9)
C2	0.0519 (14)	0.0374 (12)	0.0483 (14)	−0.0100 (10)	−0.0092 (10)	−0.0022 (10)
C3	0.0696 (17)	0.0377 (13)	0.0635 (17)	−0.0212 (12)	−0.0082 (13)	−0.0033 (11)
C4	0.0644 (16)	0.0352 (13)	0.0751 (19)	−0.0142 (11)	−0.0097 (13)	−0.0203 (12)
C5	0.0649 (16)	0.0505 (14)	0.0528 (15)	−0.0121 (12)	−0.0037 (12)	−0.0231 (12)
C6	0.0550 (14)	0.0420 (12)	0.0460 (13)	−0.0164 (11)	0.0030 (10)	−0.0107 (10)
C7	0.0426 (12)	0.0387 (12)	0.0389 (12)	−0.0095 (9)	−0.0074 (9)	−0.0061 (9)
C8	0.0492 (13)	0.0396 (12)	0.0315 (11)	−0.0138 (10)	−0.0060 (9)	−0.0067 (9)
C9	0.0708 (19)	0.105 (2)	0.0483 (16)	−0.0080 (16)	−0.0116 (13)	−0.0231 (15)
C10	0.088 (2)	0.087 (2)	0.0624 (18)	0.0104 (17)	−0.0126 (16)	−0.0243 (16)
N2	0.0519 (11)	0.0355 (10)	0.0375 (10)	−0.0138 (8)	−0.0063 (8)	−0.0080 (8)
N1	0.0492 (11)	0.0355 (9)	0.0351 (9)	−0.0162 (8)	−0.0052 (8)	−0.0065 (8)
O1	0.1101 (16)	0.0623 (12)	0.0456 (10)	−0.0445 (11)	−0.0098 (10)	0.0054 (9)
O4	0.1213 (17)	0.0750 (12)	0.0327 (9)	−0.0324 (12)	−0.0007 (10)	−0.0151 (9)
O9	0.0939 (13)	0.0549 (10)	0.0350 (9)	−0.0351 (9)	−0.0142 (8)	−0.0005 (7)
S1	0.0887 (5)	0.0536 (4)	0.0363 (4)	−0.0194 (3)	−0.0035 (3)	−0.0100 (3)

Geometric parameters (Å, °)

C1—C6	1.388 (3)	C8—C8^{i}	1.439 (4)
C1—C2	1.412 (3)	C8—H8	0.9300
C1—C7	1.482 (3)	C9—S1	1.761 (3)
C2—O1	1.350 (3)	C9—H9A	0.9600
C2—C3	1.386 (3)	C9—H9B	0.9600
C3—C4	1.365 (4)	C9—H9C	0.9600
C3—H3	0.9300	C10—S1	1.767 (3)
C4—C5	1.377 (3)	C10—H10A	0.9600
C4—H4	0.9300	C10—H10B	0.9600
C5—C6	1.374 (3)	C10—H10C	0.9600
C5—H5	0.9300	N2—N1	1.375 (2)
C6—H6	0.9300	N1—H1A	0.83 (3)
C7—O9	1.238 (2)	O1—H1	0.8200
C7—N1	1.356 (3)	O4—S1	1.5065 (17)
C8—N2	1.274 (3)		
C6—C1—C2	117.9 (2)	N2—C8—H8	120.6
C6—C1—C7	123.6 (2)	C8^{i}—C8—H8	120.6
C2—C1—C7	118.5 (2)	S1—C9—H9A	109.5
O1—C2—C3	117.9 (2)	S1—C9—H9B	109.5
O1—C2—C1	122.2 (2)	H9A—C9—H9B	109.5
C3—C2—C1	119.8 (2)	S1—C9—H9C	109.5
C4—C3—C2	120.3 (2)	H9A—C9—H9C	109.5
C4—C3—H3	119.8	H9B—C9—H9C	109.5
C2—C3—H3	119.8	S1—C10—H10A	109.5
C3—C4—C5	121.0 (2)	S1—C10—H10B	109.5

C3—C4—H4	119.5	H10A—C10—H10B	109.5
C5—C4—H4	119.5	S1—C10—H10C	109.5
C6—C5—C4	119.1 (2)	H10A—C10—H10C	109.5
C6—C5—H5	120.4	H10B—C10—H10C	109.5
C4—C5—H5	120.4	C8—N2—N1	116.44 (18)
C5—C6—C1	121.8 (2)	C7—N1—N2	117.79 (18)
C5—C6—H6	119.1	C7—N1—H1A	124 (2)
C1—C6—H6	119.1	N2—N1—H1A	118 (2)
O9—C7—N1	121.09 (19)	C2—O1—H1	109.5
O9—C7—C1	121.12 (19)	O4—S1—C9	106.46 (13)
N1—C7—C1	117.78 (19)	O4—S1—C10	106.43 (13)
N2—C8—C8^{i}	118.7 (2)	C9—S1—C10	98.08 (15)
C6—C1—C2—O1	179.4 (2)	C7—C1—C6—C5	176.5 (2)
C7—C1—C2—O1	1.5 (3)	C6—C1—C7—O9	174.6 (2)
C6—C1—C2—C3	1.5 (3)	C2—C1—C7—O9	−7.6 (3)
C7—C1—C2—C3	−176.4 (2)	C6—C1—C7—N1	−6.5 (3)
O1—C2—C3—C4	−179.1 (2)	C2—C1—C7—N1	171.3 (2)
C1—C2—C3—C4	−1.1 (4)	C8^{i}—C8—N2—N1	−179.4 (2)
C2—C3—C4—C5	0.3 (4)	O9—C7—N1—N2	2.5 (3)
C3—C4—C5—C6	−0.1 (4)	C1—C7—N1—N2	−176.43 (17)
C4—C5—C6—C1	0.6 (4)	C8—N2—N1—C7	−179.9 (2)
C2—C1—C6—C5	−1.3 (3)		

Symmetry codes: (i) −*x*, −*y*, −*z*+2.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···O4^{ii}	0.83 (3)	2.05 (3)	2.858 (2)	164 (3)
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C9—H9B···N2	0.96	2.61	3.465 (3)	149

Symmetry codes: (ii) *x*, *y*, *z*+1.

Fig. 1

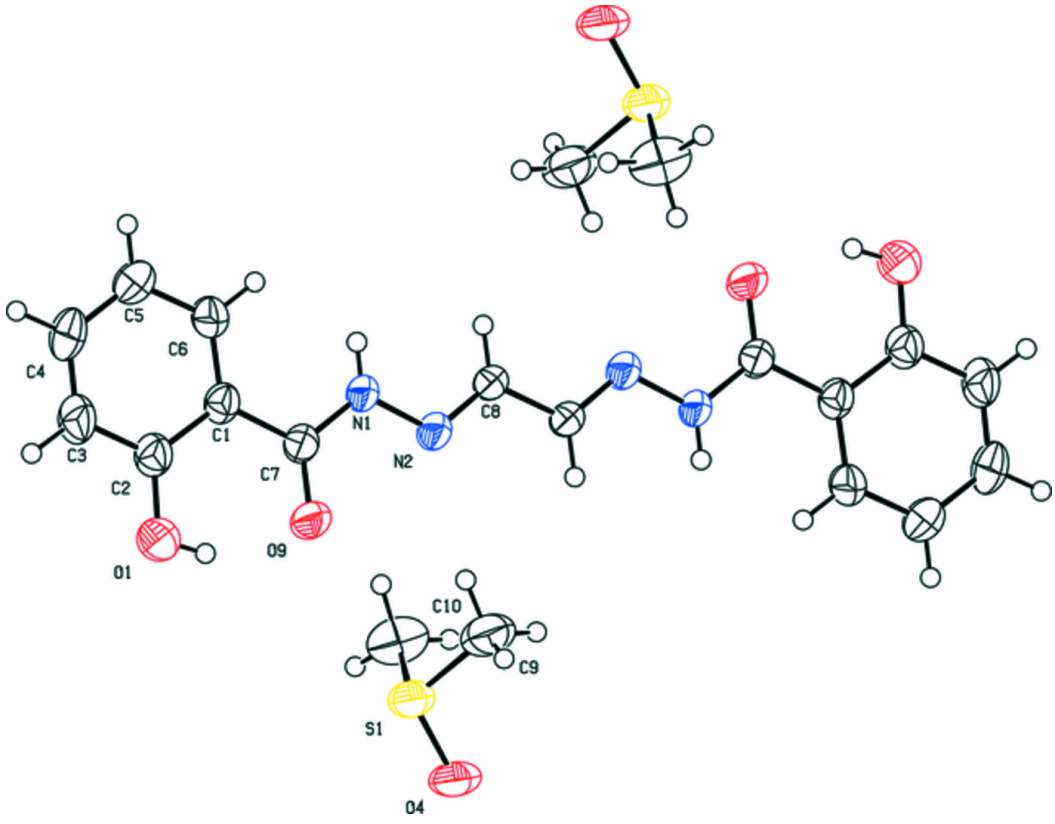

Fig. 2