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2,2′-Dithiodisalicylic acid dimethylformamide disolvate

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In the title compound, $C_{14}H_{10}O_4S_2 \cdot 2C_3H_7NO$, the dithiodisalicylic acid is hydrogen bonded to dimethylformamide solvent molecules. Within the dithiodisalicylic acid, the two benzene rings are nearly perpendicular to each other, with a dihedral angle of 77.77 (10)°.

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.045
wR factor = 0.133
Data-to-parameter ratio = 14.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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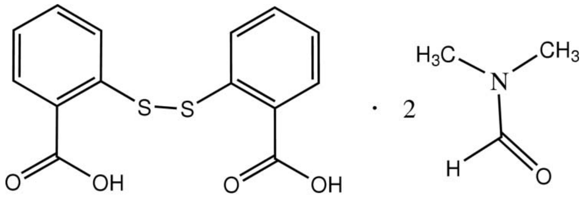
Comment

As part of our ongoing investigation of structures containing flexible ligands (Cai *et al.*, 2001), we report here the structure of the title compound, (I).



(I)

The molecular structure of (I) is shown in Fig. 1. The structure of the dithiodisalicylic acid molecule is similar to that found in dithiodisalicylic acid tetrahydrofuran solvate, (II) (Kresinski & Fackler, 1994). The S1—S2 bond distance in (I) (Table 1) is slightly shorter than the value of 2.051 (5) Å found in (II), and the dihedral angle of 77.77 (10)° between the two benzene planes in (I) is comparable with the values of 80.4 (6)

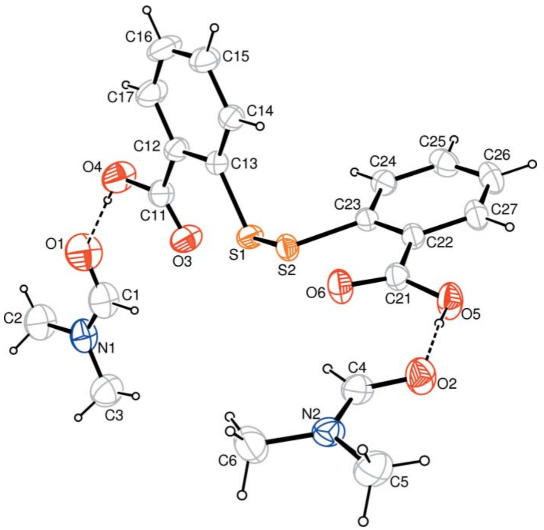


Figure 1
The structure of the asymmetric unit of (I), shown with 30% probability displacement ellipsoids (arbitrary spheres for H atoms). Dashed lines indicate hydrogen bonds.

and 73.8 (4)° found in (II). The C13—S1—S2—C23 torsion angle in (I) is 90.42 (11)°.

O—H···O hydrogen bonding occurs between dithiodisalicylic acid and dimethylformamide solvent molecules in the crystal structure (Fig. 1 and Table 2).

Experimental

Powdered sulfur (3.4 g) was added slowly to an aqueous solution (50 ml) of sodium sulfide (7.8 g) with stirring at 343 K. After stirring for 30 min, an aqueous solution (50 ml, 10 mol l^{-1}) of sodium hydroxide was added. The mixture was stirred for a further 30 min, then an aqueous solution (100 ml) of 2-(chlorodiazenyl)benzoic acid (36 g) was slowly added to the mixture at 278 K. The reaction mixture was stirred for 2 h at room temperature. The product was separated by adding concentrated hydrochloric acid (20 ml). Brown crystals of (I) were obtained by recrystallization from a dimethylformamide solution (yield: 85%).

Crystal data

$C_{14}H_{10}O_4S_2\cdot 2C_3H_7NO$
M_r = 452.53
Triclinic, $P\overline{1}$
a = 7.554 (3) Å
b = 7.710 (3) Å
c = 20.046 (8) Å
α = 83.486 (7)°
β = 84.933 (6)°
γ = 83.057 (7)°
V = 1148.1 (8) Å^3

Z = 2
D_x = 1.309 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4617 reflections
θ = 2.1–25.1°
μ = 0.27 mm^{-1}
T = 293 (2) K
Prism, brown
0.28 × 0.22 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
8419 measured reflections
4103 independent reflections

3085 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.020
$\theta_{\rm max}$ = 25.2°
$h = -9 \rightarrow 9$
$k = -9 \rightarrow 9$
$l = -24 \rightarrow 23$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.133$
S = 1.03
4103 reflections
275 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0692P)^2 + 0.3654P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max}$ = 0.002
$\Delta\rho_{\rm max}$ = 0.47 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.13 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—S2	2.0419 (12)	S2—C23	1.790 (2)
S1—C13	1.783 (2)		
C13—S1—S2	105.55 (9)	C23—S2—S1	105.73 (8)

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4D···O1	0.84	1.72	2.558 (4)	172
O5—H5D···O2	0.89	1.70	2.587 (3)	174

Hydroxyl H atoms were located in a difference Fourier map and refined as riding in their as-found relative positions, with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(O). Methyl H atoms were placed in calculated positions (C—H = 0.96 Å) and the torsion angles refined to fit the electron density [$U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C)]. Other H atoms were placed in idealized positions (C—H = 0.93 Å) and refined in the riding mode, with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C).

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 2000); software used to prepare material for publication: *SHELXTL*.

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