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# 2,2′-Ethylenedioxydibenzaldehyde bis(thiosemicarbazone) bis(dimethyl sulfoxide)

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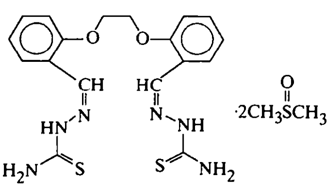
## Abstract

The two thiosemicarbazone moieties of the title compound, $C_{18}H_{20}N_6O_2S_2\cdot 2C_2H_6OS$, are positioned in a *trans* fashion. The molecule lies on a crystallographic twofold axis. Both O and S atoms of the dimethyl sulfoxide are disordered.

## Comment

Thiosemicarbazones belong to a large group of organic derivatives, the biological activities of which are a function of the parent aldehydes and ketones (Hagenbach & Gysin, 1952; Blanz & French, 1968; French *et al.*, 1974). Antitumour activity has been attributed to 2-ethoxybutyraldehyde bis(thiosemicarbazones) (Tosi, 1982) and thiosemicarbazones derived from diketones (Jones & McCleverty, 1970), which has led to interest in their coordination chemistry. As part of our continuing study of mixed NS donor ligands (Duan *et al.*, 1996; Tian *et al.*, 1997), we have determined the structure of the title compound, (I).



(I)

Fig. 1 shows the *ORTEP*II (Johnson, 1976) drawing of the title compound with the atom-numbering scheme. The C1—S1 bond distance of 1.689 (2) Å (Table 1) agrees well with those in related compounds (Tian *et al.*, 1997) and with the value for a C═S double bond (1.67 Å) reported by Allen *et al.* (1987). The thiosemicarbazone moiety shows an *E* configuration about the C2—N3 and C1—N2 bonds and the resulting plane (C2/N2/N3/C1/S1, mean deviation from the best plane 0.029 Å) is twisted relative to the phenyl ring (C3–C8) by 17.9 (1)°. This twisted conformation is comparable to that observed for formaldehyde thiosemicarbazone (twist angle 14.6°; Restivo & Palenik, 1970). The title molecule lies on a crystallographic $C_2$ axis and the two thiosemicarbazone moieties in the centrosymmetric molecule are, of course, equivalent and are positioned in a *trans* fashion, which minimizes their intramolecular interactions. The molecular packing pattern is stabilized by the incorporation of two dimethyl sulfoxide molecules, and both O and S atoms of the solvent are disordered.

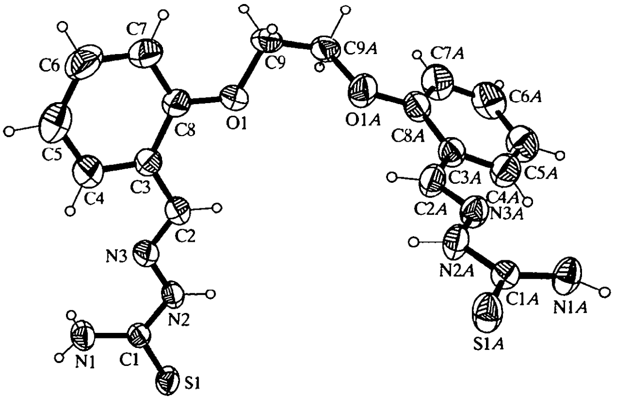


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as spheres of an arbitrary radius.

## Experimental

The title compound was synthesized by refluxing 2,2′-ethylenedioxydibenzaldehyde with thiosemicarbazide in a 1:2 molar ratio in ethanol for 3 h. Single crystals, suitable for X-ray diffraction, were obtained from a dimethyl sulfoxide solution.

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*Crystal data*

$C_{18}H_{20}N_6O_2S_2{\cdot}2C_2H_6OS$
$M_r = 572.78$
Monoclinic
$C2/c$
$a = 17.432\,(4)$ Å
$b = 17.339\,(4)$ Å
$c = 11.538\,(4)$ Å
$\beta = 124.49\,(2)°$
$V = 2874.3\,(13)$ Å$^3$
$Z = 4$
$D_x = 1.324$ Mg m$^{-3}$
$D_m$ not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 20 reflections
$\theta = 4.76$–$12.61°$
$\mu = 0.37$ mm$^{-1}$
$T = 293\,(2)$ K
Block
0.42 × 0.36 × 0.32 mm
Yellow

*Data collection*

Siemens *P*4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3953 measured reflections
2420 independent reflections
1920 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.030$

$\theta_{\rm max} = 25.03°$
$h = -14 \rightarrow 20$
$k = -15 \rightarrow 20$
$l = -13 \rightarrow 1$
3 standard reflections every 97 reflections
intensity decay: 3.3%

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.111$
$S = 1.024$
2417 reflections
182 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0533P)^2 + 1.7286P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.23$ e Å$^{-3}$
$\Delta\rho_{\rm min} = -0.23$ e Å$^{-3}$
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| S1—C1 | 1.689 (2) | N3—C2 | 1.272 (3) |
| N2—C1 | 1.353 (3) | C1—N1 | 1.315 (3) |
| N2—N3 | 1.373 (2) | C3—C2 | 1.463 (3) |
| C1—N2—N3 | 118.3 (2) | N1—C1—S1 | 122.6 (2) |
| C2—N3—N2 | 119.0 (2) | N2—C1—S1 | 120.6 (2) |
| N1—C1—N2 | 116.8 (2) | N3—C2—C3 | 118.9 (2) |

The H atoms of the main molecule were located from difference maps and their positions were refined isotropically, with $U_{\rm iso} = 1.2U_{\rm eq}$ of the parent atoms. Dimethyl sulfoxide H atoms were placed in calculated positions, with C—H = 0.96 Å and $U_{\rm iso} = 1.5U_{\rm eq}$ of the parent C atoms. The disorder of the dimethyl sulfoxide molecule was modelled with both S and O atoms occupying two sites having occupancy factors of 0.708 (2) and 0.292 (2). The methyl H atoms of dimethyl sulfoxide were likewise placed in two sets of sites with these occupancy factors.

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXTL* (Siemens, 1995). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1153). Services for accessing these data are described at the back of the journal.

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# Hydrogen bonding and C—H···O interactions in 2,2′-dihydroxy-5,5′-dimethoxybiphenyl-3,3′-dicarbaldehyde

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## Abstract

The title dialdehyde, $C_{16}H_{14}O_6$, crystallized in the centrosymmetric space group $P\bar{1}$ with one molecule in the asymmetric unit. In this structure, one intermolecular and two intramolecular hydrogen bonds occur; the $O_D\cdots O_A$ distances are 3.030 (2), 2.626 (2) and 2.662 (2) Å. In addition, ten significant C—H···O interactions have C···O distances ranging from 3.342 (2) to 3.741 (3) Å and C—H···O angles ranging from 117 to 167°. The 11 intermolecular interactions link a molecule directly to eight neighbors and form a richly three-dimensional network. The dihedral angle between the best-fit planes of the benzene rings within a molecule (biphenyl twist angle) is 79.00 (8)°. The dihedral angles