

(2*RS*,5*SR*)-2,5-Diferrocenyl-1,6-dibenzoyl-3,4-dithiahexane

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## Key indicators

Single-crystal X-ray study

$T = 293\text{ K}$

Mean  $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$

$R$  factor = 0.041

$wR$  factor = 0.099

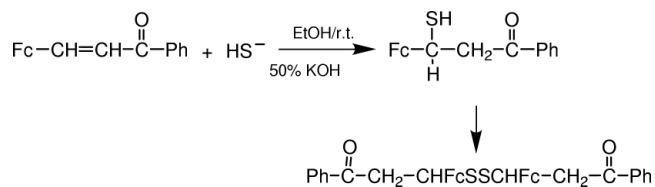
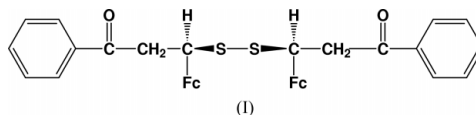
Data-to-parameter ratio = 17.2

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

The title compound,  $\text{C}_{38}\text{H}_{34}\text{Fe}_2\text{O}_2\text{S}_2$ , is a novel disulfide. The S—S bond distance is 2.0189 (11) Å. There are two chiral C atoms in the molecule, with relative stereochemistry *RS* and *SR*. Pairs of molecules form centrosymmetric dimers through weak C—H···S interactions which are, in turn, linked into two-dimensional layers by further weak C—H···S interactions.

## Comment

The preparation of organic disulfides has attracted some attention due to their variable applications, such as antifungal and antiproliferative reagents (Langler *et al.*, 2001), self-assembled monolayers (Chen *et al.*, 2001), protection reagent of hair against UV light (Smith & Penna, 1987), lubricating oil additives (Wachal & Kulczynski, 1988), stabilizers of photographic dye (Krishnamurthy, 1988) and regulators of molecular weight (Schiller & Schiller, 1989). In general, organic disulfides can be obtained by the catalytic oxidation of thiols using various catalysts and oxidants (Raghavan *et al.*, 2001; Zhong & Guo, 2001; Yu & Zhang, 2001). The title compound, (I) (Fc = ferrocenyl), was obtained by the nucleophilic addition of  $\text{H}_2\text{S}$  to 1-phenyl-3-ferrocenyl-1-oxo-propene, followed by autooxidation of the intermediate in anhydrous ethanol and KOH at room temperature (see Scheme).



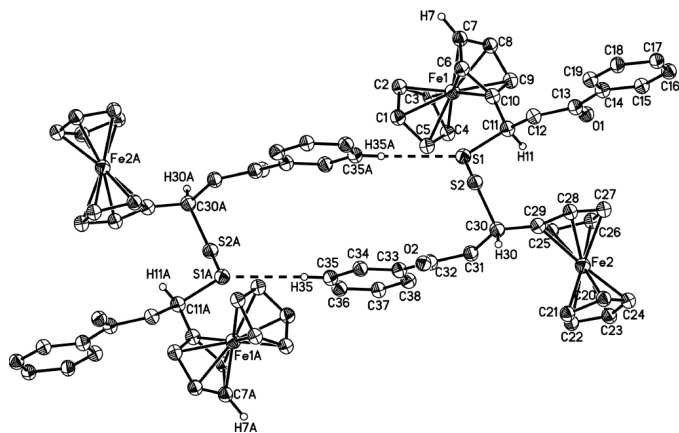
The title molecule is *meso*, having two chiral C atoms with relative stereochemistry *R* and *S* or *S* and *R*, respectively. The torsion angle C11—S1—S2—C30 is 86.69 (13)°; the conformation appears to be stable and this is close to a torsion angle of 90° which corresponds to minimum repulsion between the lone pairs of electrons on adjacent S atoms (Pauling, 1949). From theoretical calculations (Ohsaku & Allinger, 1988) it can be seen that, in similar compounds, the torsion angles for C—

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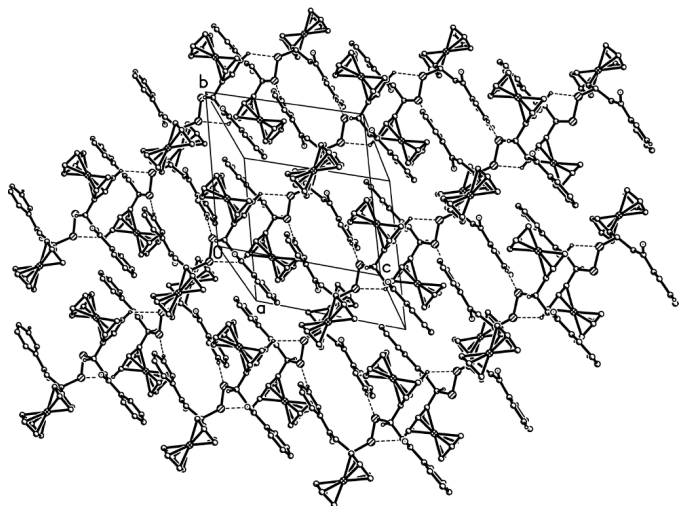
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**Figure 1**

View of a centrosymmetric pair of molecules of the title compound, shown with 30% probability displacement ellipsoids. All H atoms are omitted except for H7, H35, H11, and H30 and their symmetry equivalents. Weak H...S interactions are shown as dashed lines and the atoms labelled A are at symmetry position  $-x, 1-y, 1-z$ .


**Figure 2**

A view of the two-dimensional layers in the crystal structure of the title compound, viewed along the  $a$  axis. All H atoms are omitted except for H7, H35, H11, H30 and their symmetry equivalents.

C—S—S range from 29 to 150°, but the C—S—S—C angle remains near 90°. In the title molecule, the C—C—S—S torsion angles range from 25.7 (2) to 150.97 (15)°. The bond distance for S—S is 2.0189 (11) Å. A similar structure has been reported (Krein & Aizenshtat, 1993), which has a C—S—S—C torsion angle of 84.58 (2)° and an S—S distance of 2.027 (3) Å. In the title structure, pairs of molecules form centrosymmetric dimers (Fig. 1) through weak C—H...S interactions; these dimers are, in turn, linked into two-dimensional layers by further weak C—H...S interactions (Fig. 2, Table 1).

## Experimental

H<sub>2</sub>S was passed into a solution of 1-phenyl-3-ferrocenyl-1-oxopropene (2.5 g, 7.9 mmol) in anhydrous ethanol (150 ml) at room temperature until it was saturated. To the reactants was added a solution of 50% KOH (0.5 ml), then H<sub>2</sub>S was continuously passed

into it for 40 h. The resulting yellow precipitate was filtered, washed until neutral with water and dried. The crude product was recrystallized from benzene–petroleum ether to give 4.8 g of the title compound as orange needles.

## Crystal data

C<sub>38</sub>H<sub>34</sub>Fe<sub>2</sub>O<sub>2</sub>S<sub>2</sub>  
*M<sub>r</sub>* = 698.47  
 Triclinic,  $P\bar{1}$   
*a* = 11.492 (2) Å  
*b* = 11.806 (2) Å  
*c* = 12.511 (3) Å  
 $\alpha$  = 109.01 (3)°  
 $\beta$  = 93.90 (3)°  
 $\gamma$  = 99.56 (3)°  
*V* = 1568.9 (7) Å<sup>3</sup>

*Z* = 2  
*D<sub>x</sub>* = 1.479 Mg m<sup>-3</sup>  
 Mo *K*α radiation  
 Cell parameters from 25 reflections  
 $\theta$  = 2.0–25.8°  
 $\mu$  = 1.09 mm<sup>-1</sup>  
*T* = 293 (2) K  
 Block, orange  
 0.35 × 0.25 × 0.25 mm

## Data collection

Bruker *P4* diffractometer  
 $\omega$  scans  
 Absorption correction:  $\psi$  scan  
 (SHELXTL; Bruker, 1997)  
*T*<sub>min</sub> = 0.73, *T*<sub>max</sub> = 0.76  
 6839 measured reflections  
 6839 independent reflections  
 6778 reflections with  $I > 2\sigma(I)$

$\theta_{\max}$  = 27.0°  
*h* = -14 → 14  
*k* = -15 → 14  
*l* = 0 → 15  
 3 standard reflections  
 every 97 reflections  
 intensity decay: none

## Refinement

Refinement on  $F^2$   
*R*[ $F^2 > 2\sigma(F^2)$ ] = 0.041  
*wR*( $F^2$ ) = 0.099  
*S* = 1.05  
 6840 reflections  
 397 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.04P)^2 + 1.55P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.70 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.68 \text{ e \AA}^{-3}$

**Table 1**

Hydrogen-bonding geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C12—H12A...S2	0.97	2.60	3.240 (3)	124
C31—H31A...S1	0.97	2.84	3.337 (3)	113
C35—H35...S1 <sup>i</sup>	0.93	2.79	3.692 (3)	163
C7—H7...S2 <sup>ii</sup>	0.98	2.91	3.708 (3)	139

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

All H atoms bonded to C atoms were placed in calculated positions, with C—H distances ranging from 0.93 to 0.98 Å, and included in the refinement in the riding-model approximation, with  $U_{\text{iso}} = 1.2U_{\text{eq}}$  of the carrier atom.

Data collection: XSCANS (Bruker, 1997); cell refinement: XSCANS; data reduction: SHELXTL (Bruker, 1997); program(s) used to solve structure: SHELXTL; program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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