

Gang Xue, Wentao Yu and  
Qi Fang\*State Key Laboratory of Crystal Materials,  
Shandong University, Jinan 250100, Shandong  
Province, People's Republic of China

Correspondence e-mail: fangqi@icm.sdu.edu.cn

## Key indicators

Single-crystal X-ray study  
 $T = 293$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å  
 $R$  factor = 0.026  
 $wR$  factor = 0.068  
Data-to-parameter ratio = 25.8For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.

## 4,5-Methylenedithio-1,3-dithiol-2-one

The methylene C atom of the title compound,  $\text{C}_4\text{H}_2\text{OS}_4$ , deviates by 0.703 (2) Å from the mean plane of the eight other non-H atoms. The molecular packing is characterized by some short  $\text{S}\cdots\text{S}$  interactions between neighbouring molecules.

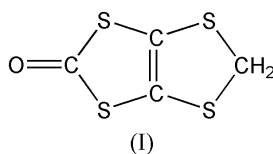
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## Comment

With the development of charge-transfer salts, single-component molecular conductors have attracted more and more attention for their structural novelty and electrical conductivity (Tanaka *et al.*, 2001). The title compound, (I), serves as the precursor of a kind of single-component molecular conductor. We report here the crystal structure of (I).



The bond lengths and angles of (I) (Fig. 1) are similar to those of an analogous compound 4,5-methylenedithio-1,3-dithiole-2-thione (Liu *et al.*, 2003). In (I),  $\text{C1}-\text{O1}$  at 1.2023 (16) Å is a typical  $\text{C}=\text{O}$  double bond and the  $\text{C2}-\text{C3}$  bond is a  $\text{C}=\text{C}$  double bond with a length of 1.3414 (17) Å. The six  $\text{S}-\text{C}$  bonds have lengths in the range 1.7324 (13)–1.7824 (14) Å, which are between those of typical single  $\text{S}-\text{C}$  and double  $\text{S}=\text{C}$  bonds. The  $\text{S3}-\text{C4}$  and  $\text{S4}-\text{C4}$  bonds, involving the methylene C atom, are typical single  $\text{S}-\text{C}$  single bonds, with lengths of 1.8110 (14) and 1.8123 (14) Å, respectively. Atom C4 deviates by 0.703 (2) Å from the least-squares plane of the eight other non-H atoms.

As shown in Fig. 2, there are some short  $\text{S}\cdots\text{S}$  interactions between neighbouring molecules. The  $\text{S1}\cdots\text{S1}(1-x, -y, 1-z)$ ,  $\text{S1}\cdots\text{S3}(2-x, -y, 1-z)$  and  $\text{S3}\cdots\text{S3}(2-x, -y, 1-z)$  contact distances are 3.4969 (6), 3.4848 (5) and 3.5906 (6) Å, respectively, which are shorter than the sum of the van der Waals radii. In addition to the above interactions, a weak  $\text{C4}-\text{H1B}\cdots\text{O1}(1+x, 1+y, z)$  interaction [ $\text{C}-\text{H} = 0.97$  Å,  $\text{H}\cdots\text{O} = 2.55$  Å,  $\text{C}\cdots\text{O} = 3.2808$  (19) Å and  $\text{C}-\text{H}\cdots\text{O} = 132^\circ$ ] is also observed.

## Experimental

4,5-Methylenedithio-1,3-dithiole-2-thione (Kato *et al.*, 1984) (0.5 g) and  $\text{Hg}(\text{CH}_3\text{COO})_2$  (1.2 g) were added to a chloroform–acetic acid mixture (3:1, 60 ml). The solution was stirred at room temperature for 20 min. The yellow precipitate was filtered off and the filtered solution was washed with water, with a saturated aqueous  $\text{NaHCO}_3$  solution and then again with water. It was then filtered to give a clear

solution to which was added methanol. After one week, pale yellow crystals were formed on slow evaporation of the solvent at room temperature.

Crystal data

$C_4H_2OS_4$   
 $M_r = 194.30$   
 Monoclinic,  $P2_1/c$   
 $a = 7.8361$  (6) Å  
 $b = 4.0090$  (3) Å  
 $c = 21.572$  (2) Å  
 $\beta = 97.586$  (7)°  
 $V = 671.76$  (10) Å<sup>3</sup>  
 $Z = 4$

$D_x = 1.921$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 Cell parameters from 39 reflections  
 $\theta = 5.2$ – $15.6$ °  
 $\mu = 1.32$  mm<sup>-1</sup>  
 $T = 293$  (2) K  
 Prism, pale yellow  
 $0.52 \times 0.44 \times 0.40$  mm

Data collection

Bruker  $P4$  diffractometer  
 $\omega$  scans  
 Absorption correction:  $\psi$  scan  
 (XSCANS; Bruker, 1996)  
 $T_{min} = 0.498$ ,  $T_{max} = 0.597$   
 3180 measured reflections  
 2140 independent reflections  
 1763 reflections with  $I > 2\sigma(I)$

$R_{int} = 0.023$   
 $\theta_{max} = 31.0$ °  
 $h = -1 \rightarrow 11$   
 $k = -5 \rightarrow 1$   
 $l = -31 \rightarrow 31$   
 3 standard reflections  
 every 97 reflections  
 intensity decay: none

Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.026$   
 $wR(F^2) = 0.068$   
 $S = 1.04$   
 2140 reflections  
 83 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0336P)^2 + 0.107P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{max} = 0.001$   
 $\Delta\rho_{max} = 0.33$  e Å<sup>-3</sup>  
 $\Delta\rho_{min} = -0.22$  e Å<sup>-3</sup>  
 Extinction correction: SHELXTL  
 Extinction coefficient: 0.061 (2)

Table 1 Selected geometric parameters (Å, °).

C1–O1	1.2023 (16)	C2–S3	1.7504 (12)
C1–S1	1.7721 (14)	C3–S2	1.7324 (13)
C1–S2	1.7824 (14)	C3–S4	1.7559 (13)
C2–C3	1.3414 (17)	C4–S3	1.8110 (14)
C2–S1	1.7343 (13)	C4–S4	1.8123 (14)
O1–C1–S1	123.84 (11)	C2–C3–S4	117.16 (10)
O1–C1–S2	122.96 (11)	S2–C3–S4	124.97 (7)
S1–C1–S2	113.20 (7)	S3–C4–S4	108.83 (7)
C3–C2–S1	118.08 (10)	C2–S1–C1	95.44 (6)
C3–C2–S3	117.33 (10)	C3–S2–C1	95.38 (6)
S1–C2–S3	124.59 (7)	C2–S3–C4	91.04 (6)
C2–C3–S2	117.86 (10)	C3–S4–C4	91.02 (6)

The H atoms were positioned geometrically and allowed to ride on their attached atoms, with C–H = 0.97 Å and  $U_{iso}(H) = 1.2U_{eq}(C)$ .

Data collection: XSCANS (Bruker, 1996); cell refinement: XSCANS; data reduction: XSCANS; program(s) used to solve structure: SIR97 (Altomare *et al.*, 1999); program(s) used to refine structure: SHELXTL (Bruker, 1997); molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

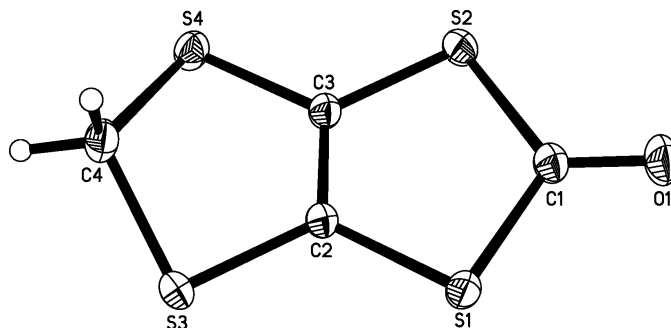


Figure 1 The molecular structure of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme.

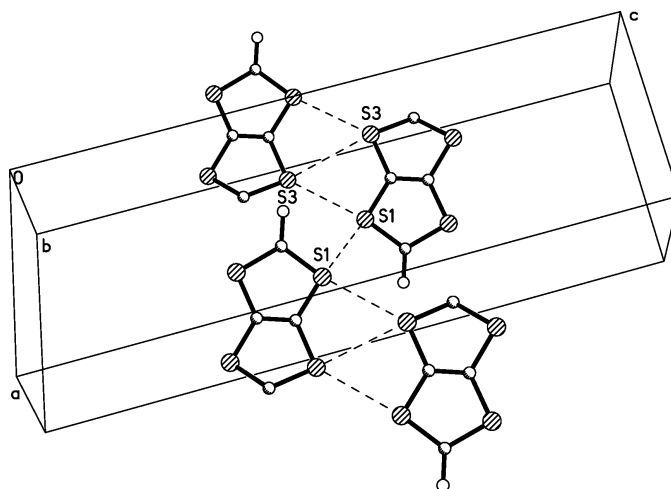


Figure 2 A packing diagram of the title compound. Dashed lines denote S...S short contacts.

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References

Altomare, A., Burla, M. C., Camalli, M., Cascarano, G., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.  
 Bruker (1996). XSCANS. Version 2.2. Bruker AXS Inc., Madison, Wisconsin, USA.  
 Bruker (1997). SHELXTL. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.  
 Kato, R., Kobayashi, A., Sasaki, Y. & Kobayashi, H. (1984). *Chem. Lett.* pp. 993–996.  
 Liu, Z., Yu, W.-T., Tao, X.-T. & Jiang, M.-H. (2003). *Z. Kristallogr. New Cryst. Struct.* **218**, 543–544.  
 Tanaka, H., Okano, Y., Kobayashi, H., Suzuki, W. & Kobayashi, A. (2001). *Science*, **291**, 285–287.