

**trans-1,2-Bis(methylthio)-1,2-bis(phenylthio)ethylene**

BY RITA GRØNBÆK HAZELL AND OVE K. LILLELUND

*Department of Inorganic Chemistry, Aarhus University, DK-8000 Århus C, Denmark*

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**Abstract.**  $C_{16}H_{16}S_4$ , monoclinic,  $P2_1/c$ ,  $a = 5.36$  (1),  $b = 8.47$  (1),  $c = 18.05$  (3) Å,  $\beta = 98.3$  (1)°,  $V = 810.9$  Å<sup>3</sup>,  $Z = 2$ ,  $D_c = 1.373$  g cm<sup>-3</sup>,  $\mu(\text{Mo } K\alpha) = 5.54$  cm<sup>-1</sup>. The structure was solved by direct methods and refined to  $R = 0.055$  for 1419 observed diffractometer data. The molecules are at centres of symmetry and therefore have the *trans* or *E* configuration.

**Introduction.** The title compound was prepared by Falsig & Iversen (1978) by electrolysis of diphenyl trithiocarbonate in the presence of a methylating agent in DMF. The reaction yielded a crystalline solid as well as an oil, and the two were expected to be *cis-trans* isomers. It was not possible to characterize them by spectroscopic means, so an X-ray investigation of the crystals was initiated and carried through although the original question was answered once the space group was known. Intensities for a hemisphere of reflexions with  $\sin \theta/\lambda \leq 0.7$  Å<sup>-1</sup> were collected by means of a diffractometer of the Arndt & Phillips (1961) type using graphite-monochromated Mo  $K\alpha$  radiation and a scintillation counter with pulse-height discrimination. 2030 independent reflexions were measured of which 1419 had  $I > 2\sigma(I)$  and were so far from the rotation axis of the reciprocal lattice that they could be expected to be measured reasonably well. The structure was solved by means of the program system *MULTAN*

(Germain, Main & Woolfson, 1971) and refined with the full-matrix least-squares program *LINUS* (Coppens & Hamilton, 1970). Positional and thermal parameters, anisotropic for the non-hydrogen atoms, isotropic for H atoms, were refined together with a scale factor and an isotropic extinction parameter. The latter was small; the minimum transmission was 0.77 with only four reflexions below 0.9. The final  $R$  was 0.055 ( $R_w = 0.061$ ) for 124 parameters. The coordinates are given in Table 1.\*

**Discussion.** Experience shows that compounds with the *trans* configuration around a double bond are usually more stable than the *cis* isomers, but when the difference between the substituents is small, or the substituents are far from the double bond, this may not be a reliable rule. Here, however, it does fit, and the main crystalline product is *trans*.

The structure of the molecule is shown in Fig. 1. The molecular packing is shown in Fig. 2. Bond lengths and angles are listed in Table 2. A rigid-body analysis of the benzene ring together with S(2) showed good agreement, so this model was used to correct the bond lengths for thermal vibration. For the central part of the molecule the riding-motion model was used, but is not satisfactory since the ethylenic bond cannot be treated in this way. The model is also obviously poor for the S(1)–C(2) bond because the S atom vibrates more than the methyl C in one direction and less in another, so that the correction is underestimated by at least 0.003 Å. It is likely that this bond is not significantly different from the 1.804 Å found in tetrakis(methylthio)ethylene (Collins & Davis, 1978*a*). The C=C bond is not significantly longer than a normal double bond and the C(*sp*<sup>2</sup>)–S and C(*ar*)–S bonds, when corrected for thermal motion, are all about the same length—the length expected for a C(*sp*<sup>2</sup>)–S distance. The C(*ar*)–S distance is at the longer end of the range given by Domenicano, Vaciago & Coulson (1975) and the angle at C(3) is correspondingly relatively large (119.9°). The benzene ring shows no significant deviations from

Table 1. *Fractional coordinates* ( $\times 10^4$ )

	<i>x</i>	<i>y</i>	<i>z</i>
S(1)	4828 (2)	2524 (1)	323 (1)
S(2)	8536 (2)	5161 (1)	771 (1)
C(1)	5714 (6)	4501 (4)	232 (2)
C(2)	7760 (12)	1497 (7)	462 (4)
C(3)	7903 (6)	4864 (4)	1707 (2)
C(4)	5893 (8)	5620 (5)	1953 (3)
C(5)	5538 (9)	5497 (7)	2698 (3)
C(6)	7195 (10)	4637 (6)	3191 (3)
C(7)	9184 (11)	3876 (6)	2948 (3)
C(8)	9547 (8)	3992 (5)	2201 (2)
H(4)	4566 (83)	6240 (47)	1603 (24)
H(5)	4140 (105)	6102 (66)	2867 (28)
H(6)	6984 (74)	4510 (51)	3683 (23)
H(7)	10480 (93)	3377 (53)	3286 (27)
H(8)	10932 (65)	3545 (41)	2029 (19)
H(21)	8717 (89)	1753 (56)	968 (28)
H(22)	7163 (113)	505 (84)	449 (31)
H(23)	8801 (118)	1764 (71)	91 (34)

\* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33720 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

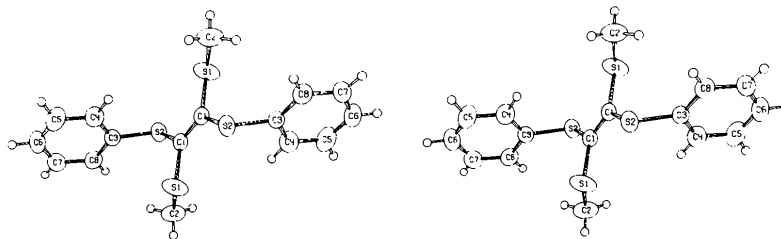


Fig. 1. Stereoscopic drawing (ORTEP, Johnson, 1965) of a molecule showing the numbering of the atoms.

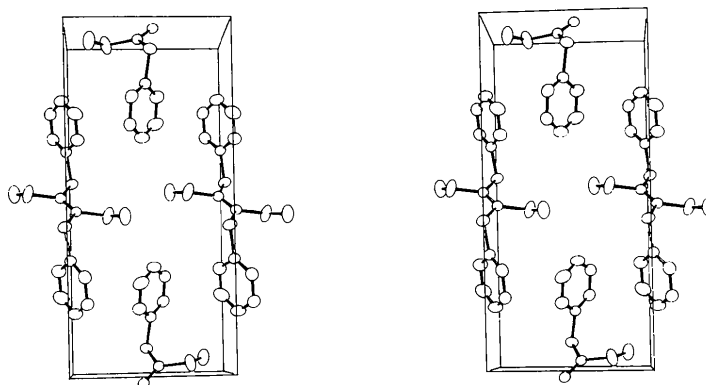


Fig. 2. Molecular packing of *trans*-1,2-bis(methylthio)-1,2-bis(phenylthio)ethylene.

Table 2. Bond lengths (Å) and angles (°)

	Uncor- rected	Corrected for thermal motion		
C(1)—S(1)	1.754 (4)	1.777	H(4)—C(4)	1.03 (5)
C(1)—S(2)	1.767 (4)	1.773	H(5)—C(5)	0.99 (6)
C(2)—S(1)	1.782 (6)	1.787	H(6)—C(6)	0.92 (4)
C(3)—S(2)	1.788 (4)	1.796	H(7)—C(7)	0.95 (5)
C(1)—C(1')	1.348 (7)		H(8)—C(8)	0.93 (4)
C(4)—C(3)	1.380 (6)	1.391	H(21)—C(2)	1.01 (5)
C(5)—C(4)	1.390 (6)	1.396	H(22)—C(2)	0.90 (7)
C(6)—C(5)	1.372 (7)	1.384	H(23)—C(2)	0.96 (7)
C(7)—C(6)	1.370 (7)	1.381		
C(8)—C(7)	1.395 (6)	1.401		
C(8)—C(3)	1.374 (5)	1.386		
S(1)—C(1)—S(2)	118.2 (2)		C(4)—C(3)—C(8)	119.9 (4)
S(1)—C(1)—C(1')	121.3 (4)		C(3)—C(4)—C(5)	119.9 (4)
S(2)—C(1)—C(1')	120.5 (4)		C(4)—C(5)—C(6)	120.1 (5)
C(1)—S(1)—C(2)	103.5 (3)		C(5)—C(6)—C(7)	120.2 (5)
C(1)—S(2)—C(3)	102.2 (2)		C(6)—C(7)—C(8)	120.0 (5)
S(2)—C(3)—C(4)	120.1 (3)		C(7)—C(8)—C(3)	119.9 (4)
S(2)—C(3)—C(8)	119.8 (3)			

regularity but here, as well as in tetrakis(phenylthio)ethylene (Collins & Davis, 1978*b*), the bonds furthest from the S atoms are the shortest. The phenyl groups are nearly perpendicular (88°) to the plane through the

four S atoms. In tetrakis(phenylthio)ethylene these angles are only 67 and 75°, and the two neighbouring rings are nearly parallel. In the present compound no parallel neighbouring rings are found.

The torsion angle C=C—S—Ph is 118.3° against 135 and 143° in the tetraphenyl compound and C=C—S—Me is 145.1° against 134 and 135° in the tetramethyl compound. In all cases this leads to similar non-bonding intramolecular distances. Only one intermolecular C—C distance less than 3.5 Å is found [C(5)—C(8)(1 + x, y, z) = 3.45 Å].

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