Fig. 2. Packing diagram for $(MeS)_2C=C(SMe)_2$. The unit-cell outline extends from $-\frac{1}{2}$ to $+\frac{1}{2}$ in each direction.

thio)ethylene (Jandal, Seip & Torgrimsen, 1976), 1·762 (5) and 1·817 (5) Å respectively.

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# Tetrakis(phenylthio)ethylene*

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**Abstract.** $(C_6H_5S)_2C=C(SC_6H_5)_2$, $(C_{14}H_{10}S_4)$, $M_r = 470·7$, orthorhombic, *Pbca*, $a = 9·537$ (2), $b = 19·418$ (4), $c = 12·462$ (3) Å, $V = 2307·7$ Å³, $Z = 4$, $D_x = 1·33$ ($-35°$C), $D_m = 1·30$ ($23°$C) g cm⁻³. Full-matrix least-squares refinement with 1610 reflections collected at $-35°$C on a Syntex $P2_1$ diffractometer yielded a final $R$ of 0·040. The C=C—S—Ph torsion angles are 135·3 and 142·9°. The conformation at

* Perheterosubstituted Ethanes and Ethylenes. II.

each end of the molecule is *gauche–gauche*, with the phenyl rings substantially on opposite sides of the ethylene plane.

**Introduction.** Single crystals of the title compound, TPTE, were obtained by recrystallization from benzene at 10°C. Preliminary X-ray diffraction experiments established the space group as *Pbca*. Intensity-data collection details are given in Table 1. Assignment of

standard deviations to the intensity data and data reduction were effected as described earlier (Riley & Davis, 1976).

Positions of all non-hydrogen atoms were determined by the symbolic addition method (*ASPASIA*: a local modification by J. Korp of *SORTE6* by A. Bednowitz) with $|E|$'s normalized by parity group. Refinement procedure, including treatment of H atoms, followed that described in part I of this series (Collins & Davis, 1978) with 1610 reflections (XPR = XSR =

Table 1. *Experimental summary*

Syntex $P2_1$ autodiffractometer equipped with a graphite mono-chromator and Syntex LT-1 low-temperature flow system
Radiation: Mo $K\alpha$, $\lambda = 0.71069$ Å
Mode: $\omega$-scan technique, recentered automatically after every 700 reflections
Scan range: symmetrically over 0.79° about the $K\alpha_{1,2}$ maximum
Scan rate: variable, 1.5 to 5.0° min⁻¹
Background: offset 1.0 and −1.0° in $\omega$ from $K\alpha_{1,2}$ maximum
Check reflections: four remeasured after every 96 reflections; analysis* of 32 sets of check reflections indicated that a decay correction was not required
2649 reflections measured, $4° \leq 2\theta \leq 55°$
Data-crystal dimensions: 0.25 × 0.28 × 0.57 mm
Data-crystal volume: 0.0357 mm³
Mosaic character: peak widths at half height, ~0.2° in $\omega$
Absorption coefficient: $\mu$(Mo $K\alpha$) = 4.10 cm⁻¹
Transmission-factor range: 0.904 to 0.914

* Henslee & Davis (1975).

Table 2. *Atomic coordinates* ($\times 10^4$) *for the non-hydrogen atoms, with e.s.d.'s in parentheses*

|        | $x$        | $y$      | $z$       |
|--------|-----------|----------|-----------|
| S(1)   | −2252 (1) | −46 (0)  | −299 (1)  |
| S(2)   | −142 (1)  | 1122 (0) | −50 (1)   |
| C(1)   | −514 (2)  | 231 (1)  | −73 (2)   |
| C(2)   | −3250 (3) | 502 (1)  | 542 (2)   |
| C(3)   | −4356 (3) | 861 (1)  | 102 (2)   |
| C(4)   | −5200 (3) | 1259 (2) | 750 (3)   |
| C(5)   | −4928 (3) | 1311 (2) | 1830 (3)  |
| C(6)   | −3819 (4) | 957 (2)  | 2258 (3)  |
| C(7)   | −2980 (3) | 551 (1)  | 1624 (2)  |
| C(8)   | −1255 (3) | 1464 (1) | −1050 (2) |
| C(9)   | −1306 (3) | 1202 (2) | −2069 (3) |
| C(10)  | −2170 (4) | 1491 (2) | −2830 (3) |
| C(11)  | −2948 (4) | 2061 (2) | −2578 (3) |
| C(12)  | −2886 (4) | 2332 (2) | −1568 (3) |
| C(13)  | −2048 (3) | 2034 (1) | −799 (3)  |
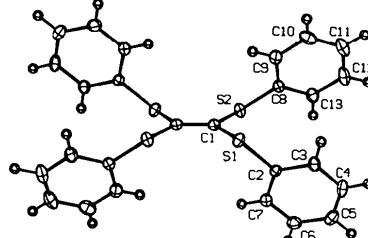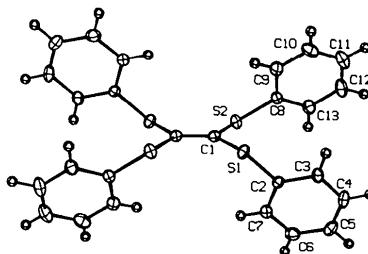


Fig. 1. A stereoview of $(PhS)_2C=C(SPh)_2$ illustrating the atom-numbering scheme. Atoms are shown with 20% equiprobability ellipsoids. Hydrogen atoms are shown as 0.15 Å radius spheres.
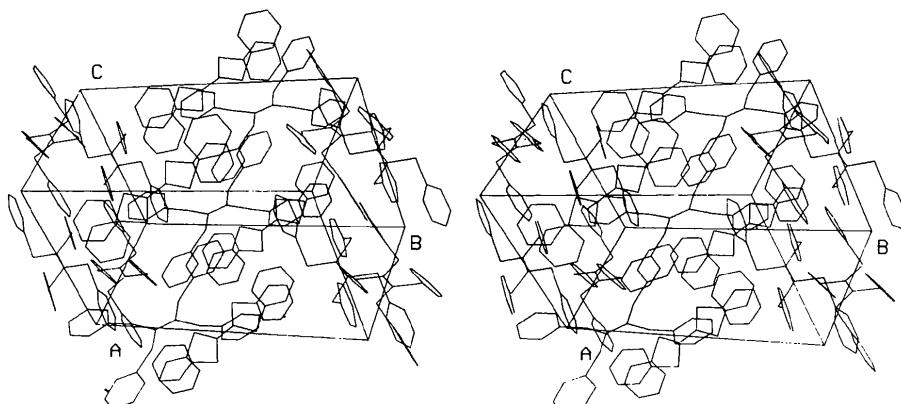


Fig. 2. Packing diagram for $(PhS)_2C=C(SPh)_2$. The unit-cell outline extends from $-\frac{1}{2}$ to $+\frac{1}{2}$ in each direction.

2·0). Convergence of this refinement* resulted in final values $R = 0.040$ and $R_w = 0.039$. A final difference map was featureless, with no peaks greater than 0·2 e Å$^{-3}$. A correction for extinction effects was not necessary. Atomic parameters are given in Table 2.

**Discussion.** A stereoview of the molecule illustrating the atom-numbering scheme is given in Fig. 1 and a packing diagram appears in Fig. 2. TPTE occupies a crystallographic inversion center and adopts a conformation similar to that of tetrakis(methylthio)ethylene, TMTE, (Collins & Davis, 1978). The difference in the C=C—S—Ph torsion angles (Table 3), 7·6°, indicates a greater deviation from local $2/m$ symmetry for TPTE than for TMTE. The C=C—S—Me torsion angles for TMTE differ by only 0·7°. Comparison of dihedral angles between the mean planes (Table 4) for the phenyl rings and the ethylene plane, 75·1 and 66·8°, also reveals a departure from local $2/m$ symmetry.

The S—Ph distances, 1·767 (3) and 1·770 (3) Å, are within the range, 1·769 (4) to 1·789 (3) Å, of S—Ph distances in several compounds summarized by

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* Lists of structure factors, non-hydrogen anisotropic thermal parameters, hydrogen atomic parameters and distances and angles involving hydrogens have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32885 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

### Table 3. *Metrical details*

Distances (Å) and angles (°) in ethylene group

| | | | |
|---|---|---|---|
| C(1)—C(1') | 1·342 (3) | C(1')—C(1)—S(1) | 120·3 (2) |
| C(1)—S(1) | 1·765 (2) | C(1')—C(1)—S(2) | 120·4 (2) |
| C(1)—S(2) | 1·767 (3) | S(1)—C(1)—S(2) | 119·3 (1) |
| S(1)—C(2) | 1·770 (3) | C(1)—S(1)—C(2) | 103·2 (1) |
| S(2)—C(8) | 1·767 (3) | C(1)—S(2)—C(8) | 103·6 (1) |
| S(1)—S(2) | 3·048 (1) | | |
| S(1)—S(2') | 3·126 (1) | | |

Distances (Å) and angles (°) involving phenyl groups

| | | | |
|---|---|---|---|
| C(2)—C(3) | 1·378 (4) | C(7)—C(2)—C(3) | 119·8 (2) |
| C(3)—C(4) | 1·380 (4) | C(2)—C(3)—C(4) | 119·8 (3) |
| C(4)—C(5) | 1·376 (5) | C(3)—C(4)—C(5) | 120·4 (3) |
| C(5)—C(6) | 1·369 (5) | C(4)—C(5)—C(6) | 119·3 (3) |
| C(6)—C(7) | 1·374 (5) | C(5)—C(6)—C(7) | 121·0 (3) |
| C(7)—C(2) | 1·377 (4) | C(6)—C(7)—C(2) | 119·6 (3) |
| | | S(1)—C(2)—C(3) | 118·7 (2) |
| | | S(1) C(2) C(7) | 121·4 (2) |
| C(8)—C(9) | 1·369 (4) | C(13)—C(8)—C(9) | 119·4 (3) |
| C(9)—C(10) | 1·377 (5) | C(8)—C(9)—C(10) | 120·6 (3) |
| C(10)—C(11) | 1·369 (5) | C(9)—C(10)—C(11) | 119·8 (3) |
| C(11)—C(12) | 1·367 (6) | C(10)—C(11)—C(12) | 120·0 (3) |
| C(12)—C(13) | 1·376 (5) | C(11)—C(12)—C(13) | 120·3 (3) |
| C(13)—C(8) | 1·378 (4) | C(12)—C(13)—C(8) | 119·9 (3) |
| | | S(2)—C(8)—C(9) | 122·5 (2) |
| | | S(2)—C(8)—C(13) | 118·1 (2) |

Torsion angles (°)

| | |
|---|---|
| C(1')—C(1)—S(1)—C(2) | 135·3 |
| C(1')—C(1)—S(2)—C(8) | 142·9 |
| C(2)—S(1)----S(2)—C(8) | 80·5 |

### Table 4. *Least-squares planes and deviations* (Å) *from the planes*

(a) The six phenyl carbon atoms C(2–7):
$(0.5982)X + (0.7832)Y + (-0.1693)Z + (1.2034) = 0^*$

| | | | |
|---|---|---|---|
| S(1) | −0·088 (1) | | |
| C(2) | −0·002 (2) | | |
| C(3) | 0·006 (3) | H(3) | 0·05 (2) |
| C(4) | −0·006 (3) | H(4) | 0·00 (3) |
| C(5) | 0·000 (3) | H(5) | −0·01 (3) |
| C(6) | 0·004 (3) | H(6) | 0·02 (3) |
| C(7) | −0·002 (3) | H(7) | 0·01 (3) |

(b) The six phenyl carbon atoms C(8–13):
$(0.7537)X + (0.5950)Y + (-0.2792)Z + (-1.1580) = 0$

| | | | |
|---|---|---|---|
| S(2) | 0·051 (1) | | |
| C(8) | −0·003 (3) | | |
| C(9) | 0·011 (3) | H(9) | −0·03 (3) |
| C(10) | −0·011 (3) | H(10) | −0·05 (3) |
| C(11) | 0·000 (3) | H(11) | −0·03 (3) |
| C(12) | 0·007 (3) | H(12) | 0·00 (3) |
| C(13) | −0·003 (3) | H(13) | −0·07 (3) |

(c) The ethylene plane composed of atoms C(1), S(1), and S(2) and restricted to pass through the origin:
$(0.1714)X + (-0.0173)Y + (-0.9850)Z + (0.0000) = 0$

| | |
|---|---|
| S(1) | 0·000 (1) |
| S(2) | 0·000 (1) |
| C(1) | −0·002 (2) |

Dihedral angles (°) between planes

| | |
|---|---|
| (a) and (c) | 75·2 |
| (b) and (c) | 66·8 |
| (a) and (b) | 15·4 |

* $X$, $Y$ and $Z$ are in Å units in an axis system parallel to the unit-cell axes.

Domenicano, Vaciago & Coulson (1975). The C(7)—C(2)—C(3) and C(13)—C(8)—C(9) angles, 119·8 (2) and 119·4 (3)°, respectively, agree with the mean value for this endocyclic angle given by the above workers, 119·7 (3)°.

No unusually short intramolecular or intermolecular contacts are observed in TPTE.

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