C(7)H(17)O(2) = 147·7° for compound (1) and O(2) and H(217) (2·58 Å) with O(2)H(217)C(17) = 170·5° for compound(2).

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Structure of 1,1,4,4-Tetrakis(phenylthio)butane

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Abstract. $C_{28}H_{26}S_4$, M_r = 490·8, monoclinic, $P2_1/n$, a = 9·834 (2), b = 13·692 (2), c = 10·261 (2) Å, β = 112·60 (2)°, V = 1275·5 Å^3, Z = 2, D_x = 1·28 g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ = 3·7 cm^{-1}, F(000) = 516. Final R = 0·030 for 2181 reflections with $I > 3\sigma(I)$. The molecule lies on a crystallographic inversion centre with the central S—CH—$(CH_2)_2$—CH—S moiety maximally extended. Mean distances are $C_{\rm arom}$—S 1·777 (2) and C_{sp^3}—S 1·828 (2) Å.

Introduction. In a recent communication (Cronin, Dilworth & McKervey, 1986) on new methods of organic synthesis employing α-chlorosulfides as reaction intermediates we described a convenient procedure whereby an alkyl phenyl sulfide is converted in a two-step sequence into a (phenylthio)acetal. The procedure was also applied to several $\alpha\omega$ bis sulfides. In a typical example, 1,4-bis(phenylthio)butane was treated with *N*-chlorosuccinimide to afford the corresponding bis(chlorosulfide) which on exposure to thiophenol with zinc chloride catalysis gave 1,1,4,4-tetrakis(phenylthio)butane (1), a compound previously prepared from 2,5-dimethoxytetrahydrofuran (Cohen, Ritter & Ouellette, 1982). For many years thioacetals and thioketals have been recognized as valuable intermediates in a wide variety of useful synthetic transformations. The crystal and molecular structure of a simple bifunctional thioacetal has not previously been recorded. Accordingly, having isolated good quality crystals of (1), we determined the crystal and molecular structure.

Experimental. Colorless crystals of (1), $C_{28}H_{26}S_4$, were grown from ethanol. Accurate cell data and orientation matrix were determined on a CAD-4

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Table 1. *Positional and thermal parameters with e.s.d.'s in parentheses*

	x	y	z	B (Å^2)
S1	0·46007 (4)	0·36369 (3)	0·70517 (4)	3·275 (8)
S2	0·25776 (4)	0·30243 (3)	0·41978 (4)	3·940 (9)
C1	0·3455 (2)	0·4566 (1)	0·7286 (1)	3·00 (3)
C2	0·4105 (2)	0·5336 (2)	0·8171 (2)	4·47 (4)
C3	0·3245 (2)	0·6043 (2)	0·8449 (2)	5·78 (5)
C4	0·1740 (2)	0·5991 (2)	0·7832 (2)	5·24 (4)
C5	0·1084 (2)	0·5235 (2)	0·6938 (2)	4·69 (4)
C6	0·1924 (2)	0·4511 (1)	0·6674 (2)	3·99 (4)
C7	0·4271 (2)	0·3706 (1)	0·5184 (1)	2·95 (3)
C8	0·4267 (2)	0·4738 (1)	0·4637 (1)	3·03 (3)
C9	0·2617 (2)	0·2972 (1)	0·2482 (2)	3·27 (3)
C10	0·3310 (2)	0·2207 (1)	0·2113 (2)	4·48 (4)
C11	0·3301 (2)	0·2158 (2)	0·0754 (2)	5·62 (5)
C12	0·2621 (2)	0·2861 (2)	−0·0210 (2)	5·72 (5)
C13	0·1926 (2)	0·3614 (2)	0·0145 (2)	5·32 (5)
C14	0·1911 (2)	0·3674 (1)	0·1482 (2)	4·24 (4)

Anisotropically refined atoms are given in the form of the isotropic equivalent thermal parameter defined as: $4/3[a^2B_{11} + b^2B_{22} + c^2B_{33} + ab\cos\gamma B_{12} + ac\cos\beta B_{13} + bc\cos\alpha B_{23}]$.

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

S1—C1	1·776 (2)	C5—C6	1·382 (3)
S1—C7	1·819 (2)	C7—C8	1·520 (2)
S2—C7	1·837 (1)	C8—C8^i	1·524 (2)
S2—C9	1·778 (2)	C9—C10	1·379 (3)
C1—C2	1·378 (2)	C9—C14	1·381 (2)
C1—C6	1·392 (2)	C10—C11	1·393 (3)
C2—C3	1·386 (3)	C11—C12	1·358 (3)
C3—C4	1·369 (3)	C12—C13	1·363 (3)
C4—C5	1·370 (3)	C13—C14	1·380 (3)

C1—S1—C7	103·89 (7)	S1—C7—C8	114·3 (1)
C7—S2—C9	101·87 (8)	S2—C7—C8	113·32 (8)
S1—C1—C2	118·6 (1)	C7—C8—C8^i	112·9 (1)
S1—C1—C6	122·2 (1)	S2—C9—C10	120·2 (1)
C2—C1—C6	119·1 (2)	S2—C9—C14	120·8 (1)
C1—C2—C3	120·3 (2)	C10—C9—C14	119·0 (2)
C2—C3—C4	120·4 (2)	C9—C10—C11	119·8 (2)
C3—C4—C5	119·7 (2)	C10—C11—C12	120·5 (2)
C4—C5—C6	120·7 (2)	C11—C12—C13	120·0 (2)
C1—C6—C5	119·7 (2)	C12—C13—C14	120·5 (2)
S1—C7—S2	108·10 (9)	C9—C14—C13	120·2 (2)

Symmetry code: (i) $1 - x$, $1 - y$, $1 - z$.

diffractometer by a least-squares treatment of the setting angles of 25 reflections in the range $10 < \theta < 15°$. Crystal dimensions 0·10 × 0·15 × 0·40 mm; intensities of reflection with indices h 0 to 12, k 0 to 17, l −12 to 12 with $2 < 2\theta < 54°$ were measured, ω–2θ scans, ω-scan width $(0{\cdot}60 + 0{\cdot}35\tan\theta)°$, graphite-monochromated Mo $K\alpha$ radiation; intensities of three reflections measured every two hours of exposure time showed no evidence of crystal decay. The space group was determined uniquely as $P2_1/n$ by the absences ($h0l$, $h + l = 2n + 1$; $0k0$, $k = 2n + 1$). 3183 reflections were measured of which 2790 were unique; 2181 reflections with $I > 3\sigma(I)$ were used in structural solution and refinement (R_{int} = 0·013). Data were corrected for Lorentz and polarization effects. With $Z = 2$ the molecule lies on an inversion centre. The structures was solved with the aid of *MULTAN*82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). Refinement was by full-matrix least-squares calculations on F, initially with isotropic and, finally, with anisotropic vibration parameters. Difference maps at various stages of the refinement showed maxima in positions consistent with the expected location of all the H atoms; in the final round of calculations, H atoms were positioned on geometric grounds (C—H 0·95 Å) and included (as riding atoms), but not refined in the calculations. The final cycles of refinement had 145 variable parameters, R = 0·030, wR = 0·046, goodness-of-fit 1·71, $w = 1/[\sigma^2F_o + 0{\cdot}03(F_o)^2]$. Maximum shift/e.s.d. in the last cycle was < 0·005; density in final difference map ±0·27 e Å^{-3}, no chemically significant features. Scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974). All calculations were performed on a PDP 11/73 computer using *SDP-Plus* (B. A. Frenz & Associates Inc., 1983).

Atomic coordinates and details of geometry are given in Tables 1 and 2.* Fig. 1 is a view of the molecule prepared with *ORTEP*II (Johnson, 1976).

Discussion. The crystallographic inversion symmetry requires that the CH—CH$_2$—CH$_2$—CH carbon skeleton be fully *anti*. The conformation of the central S2—CH—CH$_2$—CH$_2$—CH—S2 fragment is also maximally extended with a torsion angle S2—C7—C(8)—C8^i of −174·2°. The two geminal benzene rings are asymmetrically disposed relative to C7; one ring (C1–C6) forms a dihedral angle of 115·6° with the C1—S1—C7 plane whereas the other (C9–C14) forms a dihedral angle of 91·4° with the C7—S2—C9 plane. Weak steric repulsion between vicinal benzene rings results in S1 and S2 being displaced 0·037 (1)

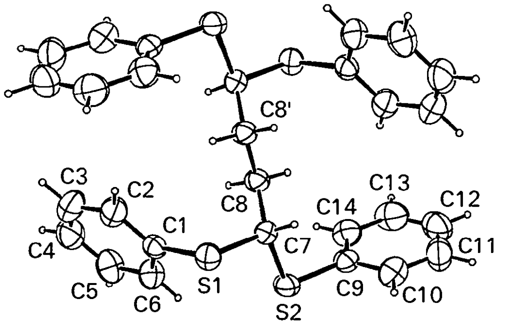

Fig. 1. A view of the (PhS)$_2$CHCH$_2$CH$_2$CH(SPh)$_2$ molecule showing the atomic numbering system.

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* Lists of structure factors, thermal parameters, calculated H-atom coordinates, mean-planes data and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52810 (28 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

and 0·091 (1) Å from the relevant benzene planes. The steric repulsion is also apparent from the bond angles about the thioacetal C atom C7 where the S—C—S angle of 108·10 (9)° is smaller than the C—C—S angle, mean value 113·8 (1)°. The mean S—C_{sp^3} distance [1·828 (2) Å] and the mean S—C_{arom} distance [1·777 (2) Å] are in agreement with those reported previously, *e.g.* 1·826 (3) and 1·776 (3) Å for these two bonds in tetraphenyl orthothiocarbonate (Kato, 1972). The S—C_{arom} bond length of 1·777 (2) Å is also in accord with the average value of 1·773 Å reported for phenyl sulfides (Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987). The bond angles at sulfur [average 102·9 (1)°] are less than tetrahedral as is usually found in simple sulfides. The various C—C bond lengths are in accord with the anticipated values. There are no untoward intermolecular contacts.

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Structure of (*E*)-2-Methyl-1,3-bis(2,4,5-trimethoxyphenyl)-1-pentene and 1-(2,4,5-Trimethoxyphenyl)-2-methyl-3-ethyl-4,6,7-trimethoxyindan* $C_{24}H_{32}O_6$: Two Asarone Dimers†

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Abstract. (*E*)-2-Methyl-1,3-bis(2,4,5-trimethoxyphenyl)-1-pentene, $C_{24}H_{32}O_6$, isomer (1), $M_r = 416·2$, monoclinic, $P2_1/n$, $a = 7·082$ (3), $b = 11·954$ (7), $c = 27·136$ (17) Å, $\beta = 94·14$ (4)°, $V = 2291$ (2) Å^3, $Z = 4$, $D_x = 1·21$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0·8$ cm^{-1}, $F(000) = 896$, $T = 298$ K, $R = 0·056$, $wR = 0·063$ for 2901 (71·4%) reflections with $F > 3\sigma(F)$. 1-(2,4,5-Trimethoxyphenyl)-2-methyl-3-ethyl-4,6,7-trimethoxyindan, isomer (2), $C_{24}H_{32}O_6$, $M_r = 416·2$, monoclinic, $P2_1/a$, $a = 17·281$ (5), $b = 7·701$ (1), $c = 18·057$ (6) Å, $\beta = 108·23$ (2)°, $V = 2282$ (1) Å^3, $Z = 4$, $D_x = 1·21$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 6·7$ cm^{-1}, $F(000) = 896$, $T = 298$ K, $R = 0·045$, $wR = 0·061$ for 2601 (90·3%) reflections with $F > 3\sigma(F)$. The X-ray structures of (1) and (2) confirm the structures previously assigned on the basis of chemical and NMR spectral evidence. Isomer (1) is non-planar. In isomer (2) the five-membered ring adopts an envelope conformation and the substituents at C(1) and C(3) are antiperiplanar to the methyl group at C(2). In both isomers the orientation of the trimethoxyphenyl substituent is determined by C—H···O intramolecular interactions. The packing in the crystal is entirely due to van der Waals forces.

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[*] IUPAC name: 1-ethyl-4,5,7-trimethoxy-2-methyl-3-(2,4,5-trimethoxyphenyl)indan.

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