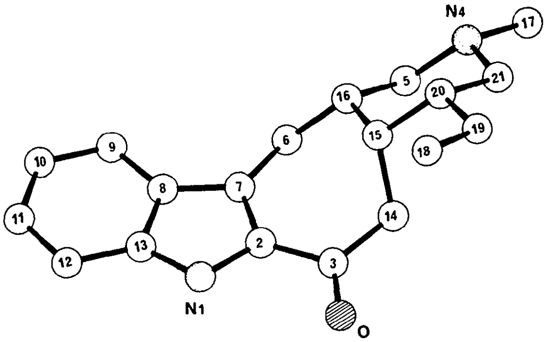

Fig. 1. Molecular structure and atom numbering. *ORTEP* (Johnson, 1965) was used for this drawing and for Fig. 3.

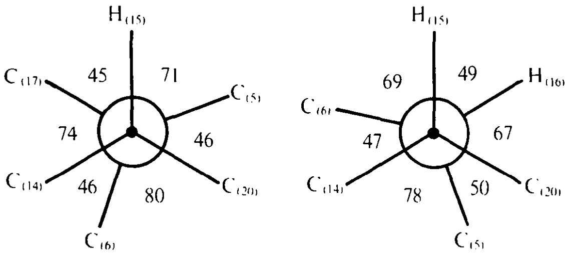

Fig. 2. Newman projections along C(15)—C(16). (*a*) Ervatamine. (*b*) This work.

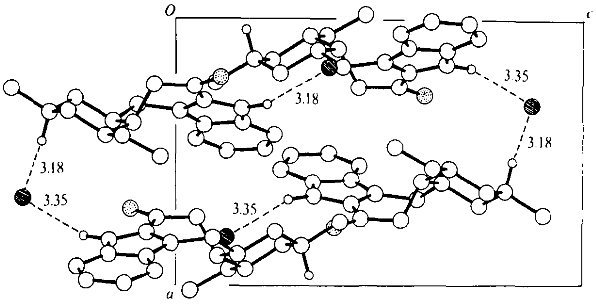

Fig. 3. Crystal structure viewed along **b**.

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Trimethyltin Methylsulphinate

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Abstract. $C_4H_{12}O_2SSn$, orthorhombic, $C222_1$; a = 17·569 (20), b = 12·293 (20), c = 8·240 (12) Å, U = 1778 Å^3, Z = 8, D_x = 1·81 g cm^{-3}, μ(Mo $K\alpha$) = 28·18 cm^{-1}. The structure was solved by the heavy-atom method and refined to an R of 0·043 for 719 unique two-circle diffractometer data. Planar trimethyltin groups are linked by disordered —O—S(Me)—O— units to form infinite helical chains along **c**, with trigonal bipyramidal geometry about Sn.

Introduction. Several tetraorganotin derivatives undergo insertion of SO_2 into the Sn—C bond to give stable, crystalline mono-inserted products which have been shown by IR and NMR studies to be aggregated in solution (Fong & Kitching, 1970). In view of the facility with which Sn increases its coordination number beyond four when a suitable bridging ligand is available, it seemed likely that these insertion compounds are polymeric in the solid state, and this has been confirmed by the structure of $Me_3SnOS(O)CH_2CCH$ (Ginderow & Huber, 1973) and by the determination reported here.

The sample was prepared by dissolving Me_4Sn in dry liquid SO_2 under vacuum. Evaporation of excess SO_2 left a white solid which was recrystallized from CCl_4 to afford a few plate-like crystals which were mounted in

Lindemann glass capillary tubes. Intensities were determined with an automated Stoe two-circle diffractometer, Mo $K\alpha$ radiation, graphite monochromator, and a crystal 0·04 × 0·25 × 0·11 mm (layers $h0$–$12l$). 1377 reflexions were measured in an approximately constant count mode; Lp and absorption corrections were applied, and equivalent reflexions averaged to give 719 unique observed data. a and c were obtained by a least-squares fit to the ω values of 141 $h0l$ reflexions, and b was obtained from diffractometer μ measurements.

The atoms were located from Patterson and difference syntheses; the MeS moiety was found to be disordered, and was refined with the site occupation factors constrained so that $k_a + k_b = 1$, where k_a is the value for both S(1*a*) and C(5*a*), and k_b for S(1*b*) and C(5*b*). k_a refined to 0·55 (2); common temperature factors were employed for S(1*a*) and S(1*b*), and for C(5*a*) and C(5*b*). Complex neutral-atom scattering factors were employed; the weighting scheme was $w = \Sigma[\sigma^2(N) + 0{\cdot}001N^2]^{-1}/(30{\cdot}13 + |F_o| + 0{\cdot}00551F_o^2)$, where N is the net peak count, and the summation is over equivalent reflexions. 58 parameters were varied independently in the full-matrix least-squares refinement, including interlayer scale factors, anisotropic temperature factors for Sn and isotropic temperature factors for the light atoms. To avoid correlation between the interlayer scale factors and the U_{22} components of the Sn temperature factors, the latter were constrained to be equal to the mean of U_{11} and U_{33}. The refinement converged to $R' = \Sigma w^{1/2}\Delta/\Sigma w^{1/2}|F_o| = 0{\cdot}046$ and $R = 0{\cdot}043$; refinement in the opposite enantiomorph produced no

Table 1. *Atom coordinates* (×10⁴) *and isotropic temperature factors* (Å² ×10³)

	x	y	z	U
Sn(1)	−1750 (1)	0	0	
Sn(2)	0	−3311 (1)	2500	
O(1)	−1760 (10)	−1757 (23)	576 (27)	133 (7)
O(2)	−1158 (9)	−3426 (15)	1283 (19)	106 (5)
C(1)	−2967 (15)	0	0	82 (8)
C(2)	−1142 (11)	253 (16)	2179 (25)	82 (6)
C(3)	0	−5024 (28)	2500	56 (7)
C(4)	460 (11)	−2454 (17)	482 (24)	83 (6)
Disordered atoms				
S(1*a*)	−1481 (7)	−2797 (7)	−66 (15)	57 (2)
S(1*b*)	−1763 (8)	−2859 (9)	621 (18)	57 (2)
C(5*a*)	−2038 (28)	−3236 (49)	−1397 (63)	109 (12)
C(5*b*)	−2261 (33)	−3624 (50)	−457 (71)	109 (12)

Table 2. *Anisotropic temperature factors* (Å² ×10³)

The temperature factor exponent takes the form: $-2\pi^2(U_{11}h^2a^{*2} + \cdots + 2U_{12}hka^*b^*)$.

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Sn(1)	51 (1)	63 (1)	73 (1)	13 (1)	0	0
Sn(2)	63 (1)	59 (1)	55 (1)	0	−5 (1)	0

Table 3. *Bond lengths* (Å)

Sn(1)–O(1)	2·209 (31)	Sn(2)–O(2)	2·272 (19)
Sn(1)–C(1)	2·137 (30)	Sn(2)–C(3)	2·104 (37)
Sn(1)–C(2)	2·113 (23)	Sn(2)–C(4)	2·128 (23)
Disordered group			
S(1*a*)–O(1)	1·467 (29)	S(1*b*)–O(1)	1·354 (30)
S(1*a*)–O(2)	1·468 (21)	S(1*b*)–O(2)	1·384 (20)
S(1*a*)–C(5*a*)	1·565 (53)	S(1*b*)–C(5*b*)	1·561 (59)

Table 4. *Bond angles* (°)

O(1)–Sn(1)–O(1′)	179·1 (12)	O(2)–Sn(2)–O(2′)	172·9 (8)
C(1)–Sn(1)–O(1)	89·6 (6)	C(3)–Sn(2)–O(2)	86·4 (6)
C(2)–Sn(1)–O(1)	88·0 (9)	C(4)–Sn(2)–O(2)	91·5 (8)
C(2)–Sn(1)–C(1)	120·4 (6)	C(4)–Sn(2)–C(3)	119·6 (7)
C(2)–Sn(1)–C(2′)	119·2 (11)	C(4)–Sn(2)–C(4′)	120·7 (12)
Disordered group			
O(2)–S(1*a*)–O(1)	108·3 (14)	O(2)–S(1*b*)–O(1)	120·7 (15)
C(5*a*)–S(1*a*)–O(1)	110·1 (24)	C(5*b*)–S(1*b*)–O(1)	126·1 (26)
C(5*a*)–S(1*a*)–O(2)	126·3 (25)	C(5*b*)–S(1*b*)–O(2)	110·6 (25)
S(1*a*)–O(1)–Sn(1)	140·4 (15)	S(1*a*)–O(2)–Sn(2)	130·4 (12)
S(1*b*)–O(1)–Sn(1)	169·2 (17)	S(1*b*)–O(2)–Sn(2)	146·2 (13)

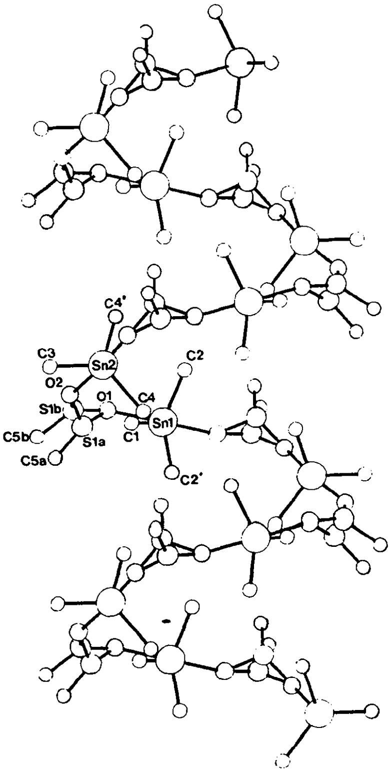

Fig. 1. The structure of trimethyltin methylsulphinate, illustrating the SMe-group disorder and a helix parallel to **c**.

significant changes in the *R* indices. Final atomic coordinates and thermal parameters are given in Tables 1 and 2, and the resulting bond lengths and angles in Tables 3 and 4.[*]

Discussion. Trimethyltin moieties are linked by disordered —O—S(Me)—O— groups to form infinite helical chains along **c**, with approximately trigonal bipyramidal geometry at Sn (Fig. 1). There are two crystallographically independent Me$_3$Sn groups, each arranged with one of the Sn—C bonds along a crystallographic twofold axis so that the group is exactly planar and the Sn atom is coordinated symmetrically by the O atoms to which it is bonded. Symmetrical bridging is also found in Me$_3$SnOS(O)CH$_2$CCH [Ginderow & Huber (1973); Sn—O 2·306 (10) and 2·296 (10) Å] where, however, the chairs adopt a zigzag rather than helical conformation. The Sn—O distances in the methylsulphinate are similar to those in trimethyltin methoxide [2·20 (2) and 2·26 (2) Å; Domingos & Sheldrick (1974)], but shorter than the values quoted above for the —OS(O)CH$_2$CCH derivative; this, and the relatively short Sn—C bonds, may arise from librational effects. Subsequent inspection of the X-ray photographs revealed the presence of weak diffuse low-order reflexions corresponding to a *P* rather than a *C* lattice, indicating some short-range order in the conformation adopted by the disordered SMe groups.

We are grateful to the Science Research Council for providing the diffractometer and for financial support to RT. The calculations were performed on the Cambridge University IBM 370/165 computer with programs written by GMS. Fig. 1 was drawn with *PLUTO* written by Dr W. D. S. Motherwell.

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[*] A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31977 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1 NZ, England.

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Methyl(triphenylphosphine)gold

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Abstract. $C_{19}H_{18}AuP$, monoclinic, $P2_1/n$, $a = 17{\cdot}29$ (1), $b = 11{\cdot}27$ (1), $c = 8{\cdot}92$ (1) Å, $\beta = 104{\cdot}5$ (1)°, $U = 1682$ Å^3, $Z = 4$, $D_x = 1{\cdot}88$ g cm^{-3}. There is essentially linear coordination of gold [P—Au—C = 179·1 (8)°] with Au—C 2·124 (28) and Au—P 2·279 (8) Å. The structure was refined to an *R* of 0·092 for 1867 unique diffractometer data.

Introduction. Previous crystallographic studies of Au$^{\rm I}$ complexes containing Au—C bonds (Bellon, Manassero & Sansoni, 1969; Corfield & Shearer, 1967; Baker & Pauling, 1972) have involved *sp* or *sp*^{2} hydridized C, in which π-bonding between Au$^{\rm I}$ and C is possible. The Au$^{\rm I}$—C(*sp*^{3}) distance in the title compound provides a suitable reference bond length in which π-bonding is absent.

Colourless crystals were obtained by recrystallization from benzene/light petroleum of a sample obtained by standard methods (Tamaki & Kochi, 1973), and mounted in Lindemann glass capillary tubes. Intensities were measured on a Stoe automated two-circle diffractometer, with Mo $K\alpha$ radiation ($\lambda = 0{\cdot}71069$ Å) and a graphite crystal monochromator. Cell dimensions were obtained by a least-squares fit to 652 *hk*0 and 444 *h*0*l* diffractometer zero-layer ω-angle measurements. The crystals possessed large mosaic spreads and the data were clearly of mediocre quality; only the 00*l* data (layers *hk*0–10) were used for structure refinement. Lp and empirical absorption corrections were applied. 2580 reflexions were measured; averaging equivalent reflexions gave 1867 reflexions with $I > 3\sigma(I)$ based on counting statistics.

The atoms were located from Patterson and difference syntheses; the structure was refined by blocked-