Fig. 1. Stereoscopic view of the unit cell looking down the *b* axis with the *c* axis horizontal and the *a* axis vertical.
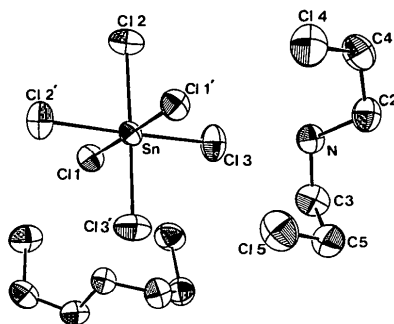

Fig. 2. Illustration of the structure of one formula unit of the title compound and the atom-numbering scheme. H atoms have been omitted.

anomalous-dispersion corrections, provided by *MolEN*, were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV) and Cromer & Mann (1968). The final equivalent isotropic thermal and positional parameters of non-H atoms are listed in Table 1.* Bond lengths and angles are presented in Table 2. All bond lengths and angles are in a normal range. Packing of the ions in a unit cell is shown in Fig. 1. Fig. 2 shows one formula unit including the atom-numbering scheme.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54990 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HH0578]

References

Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* A24, 321–324.
Enraf–Nonius (1990). *MolEN Structure Determination System.* Enraf–Nonius, Delft, The Netherlands.
Pettit, G. R. & Rawson, T. E. (1981). *Synthetic Commun.* 11, 179–184.
Walker, N. & Stuart, D. (1983). *Acta Cryst.* A39, 158–166.

[2-(Bromodimethylstannyl)ethyl]diphenylphosphine Sulfide

By Hans Preut, Bernd Godry and Terence N. Mitchell

Fachbereich Chemie der Universität Dortmund, Postfach 500500, D-4600 Dortmund 50, Germany

Abstract. $C_{16}H_{20}BrPSSn$, $M_r = 473.96$, orthorhombic, $P2_12_12_1$, $a = 10.535$ (2), $b = 10.936$ (2), $c = 16.500$ (3) Å, $V = 1901.0$ (6) Å^3, $Z = 4$, $D_x = 1.656$ Mg m^{-3}, $F(000) = 928$, $\lambda(Mo\ K\alpha) = 0.71073$ Å, $\mu = 3.61$ mm^{-1}, $T = 291$ (1) K, final $R = 0.032$ for 2900 unique observed $[F \geq 4.0\sigma(F)]$ diffractometer data. The atoms bound to Sn form a distorted trigonal bipyramid with S and Br in the apical [Sn—S 2.872 (2), Sn—Br 2.6504 (9) Å, S—Sn—Br 175.09 (4)°] and the C atoms in the equatorial positions [Sn—C 2.126 (7), 2.155 (6), 2.158 (5) Å, C—Sn—C 120.4 (2), 119.1 (2), 119.3 (3)°; C—Sn—Br 91.6 (1), 93.7 (2), 96.0 (2)°; C—Sn—S 84.5 (1), 85.8 (2), 88.5 (2)°]. The atoms bound to P form a slightly distorted tetrahedron with bond angles in the range 106.3 (3) to 112.5 (2)°. The five-membered ring has a half-chair conformation; the atoms P, S, Sn and C(3) are nearly coplanar. The molecules are separated by normal van der Waals contacts.

0108-2701/92/081491-03$06.00   © 1992 International Union of Crystallography

Experimental. The title compound was prepared by heating [2-(bromodimethylstannyl)ethyl]diphenylphosphine with sulfur in benzene for 2 h. Colourless

Me$_2$Sn—CH$_2$—CH$_2$—PPh$_2$ + 1/8 S$_8$ ⟶
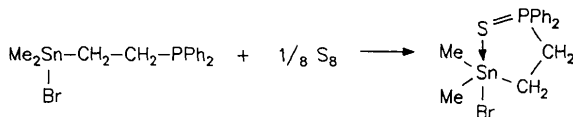
crystals were obtained by recrystallization of the crude product from hexane/methylene chloride, m.p. 390–391 K, yield 54%. A crystal of size ~ 0.18 × 0.40 × 0.28 mm was used. Its quality was checked by optical polarizing microscopy. The crystal was mounted on a glass fibre. D_m was not determined. Intensity data were collected with $\omega/2\theta$ scans, variable scan speed 1.5–15.0° min^{-1} in θ, scan width 1.2° + dispersion. A Nicolet R3m/V diffractometer with graphite-monochromated Mo Kα radiation was used for preliminary examinations and data collection. The lattice parameters were determined from a symmetry-constrained least-squares fit of 48 reflections with $2\theta_{max} = 29.9°$. ω scans of low-order reflections along the three crystal axes showed acceptable mosaicity. Six standard reflections (600, 040, 004, $\bar{6}$00, 0$\bar{4}$0, 00$\bar{4}$) were recorded every 300 reflections, only random deviations were detected during 71.13 h of X-ray exposure; 3782 reflections with $2.0 \le 2\theta \le 50.0°$ (13 391 accessible), $-13 \le h \le 13$, $0 \le k \le 14$, $0 \le l \le 20$ were measured. The data were corrected for Lorentz–polarization and for absorption effects (the latter via ψ-scan data, the max./min. transmission factors were 1.00/0.86), and averaged ($R_{int} = 0.022$) to 3366 unique reflections, 2900 of which had $F \ge 4.0\sigma(F)$. The systematic absences ($h00$) $h = 2n + 1$, ($0k0$) $k = 2n + 1$, ($00l$) $l = 2n + 1$ conform to space group $P2_12_12_1$. The structure was solved via a Patterson function and $\Delta\rho$ maps. It was refined (on F) using full-matrix least
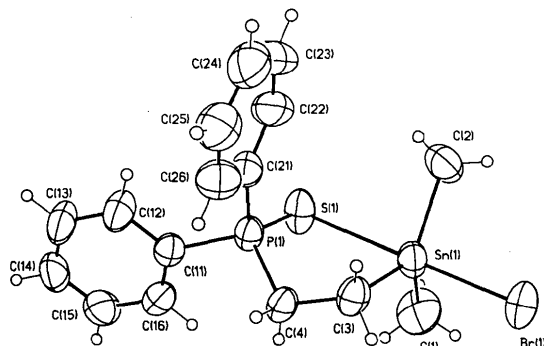

Fig. 1. General view (SHELXTL-Plus graphic) of the molecule, showing the atom-numbering scheme. Anisotropic ellipsoids represent 50% probability boundaries. H atoms are represented as spheres of arbitrary radii.

Table 1. *Atomic coordinates and equivalent isotropic displacement parameters* ($\text{Å}^2 \times 10^4$)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Sn(1)	0.89397 (4)	0.12253 (3)	0.22944 (2)	559
Br(1)	1.01974 (9)	0.06600 (8)	0.36342 (4)	1110
S(1)	0.7740 (1)	0.1724 (2)	0.07668 (8)	679
P(1)	0.9384 (1)	0.1690 (1)	0.01907 (7)	465
C(1)	0.8019 (7)	0.2795 (6)	0.2776 (4)	980
C(2)	0.7936 (6)	−0.0491 (5)	0.2259 (5)	817
C(3)	1.0684 (4)	0.1455 (5)	0.1625 (3)	548
C(4)	1.0631 (5)	0.2195 (5)	0.0855 (3)	505
C(11)	0.9354 (4)	0.2669 (5)	−0.0697 (3)	484
C(12)	0.9427 (6)	0.2182 (6)	−0.1469 (3)	685
C(13)	0.9342 (6)	0.2923 (6)	−0.2129 (3)	757
C(14)	0.9205 (6)	0.4160 (6)	−0.2036 (3)	728
C(15)	0.9113 (7)	0.4646 (6)	−0.1290 (4)	753
C(16)	0.9212 (5)	0.3902 (5)	−0.0608 (3)	651
C(21)	0.9817 (5)	0.0172 (5)	−0.0135 (3)	533
C(22)	0.8982 (7)	−0.0794 (6)	−0.0089 (4)	819
C(23)	0.937 (1)	−0.1947 (7)	−0.0304 (5)	1021
C(24)	1.056 (1)	−0.2151 (7)	−0.0555 (4)	959
C(25)	1.1402 (6)	−0.1221 (7)	−0.0635 (4)	808
C(26)	1.1040 (6)	−0.0055 (6)	−0.0425 (4)	689

Table 2. *Bond distances* (Å), *bond angles* (°), *torsion angles* (°), *least-squares planes and dihedral angles* (°)

Sn(1)—Br(1)	2.6504 (9)	S(1)—P(1)	1.977 (2)
Sn(1)—S(1)	2.872 (2)	P(1)—C(4)	1.797 (5)
Sn(1)—C(1)	2.126 (7)	P(1)—C(11)	1.814 (5)
Sn(1)—C(2)	2.155 (6)	P(1)—C(21)	1.803 (6)
Sn(1)—C(3)	2.158 (5)	C(3)—C(4)	1.508 (7)

C(2)—Sn(1)—C(3)	120.4 (24)	S(1)—P(1)—C(11)	111.2 (2)
C(1)—Sn(1)—C(3)	119.1 (2)	S(1)—P(1)—C(4)	109.9 (2)
C(1)—Sn(1)—C(2)	119.3 (3)	C(11)—P(1)—C(21)	107.9 (2)
S(1)—Sn(1)—C(3)	84.5 (1)	C(4)—P(1)—C(21)	106.3 (3)
S(1)—Sn(1)—C(2)	85.8 (2)	C(4)—P(1)—C(11)	108.9 (2)
S(1)—Sn(1)—C(1)	88.5 (2)	Sn(1)—C(3)—C(4)	117.5 (3)
Br(1)—Sn(1)—C(3)	91.6 (1)	P(1)—C(4)—C(3)	112.1 (4)
Br(1)—Sn(1)—C(2)	93.7 (2)	P(1)—C(11)—C(16)	119.9 (4)
Br(1)—Sn(1)—C(1)	96.0 (2)	P(1)—C(11)—C(12)	121.0 (4)
Br(1)—Sn(1)—S(1)	175.09 (4)	P(1)—C(21)—C(26)	119.9 (4)
Sn(1)—S(1)—P(1)	91.87 (6)	P(1)—C(21)—C(22)	121.9 (5)
S(1)—P(1)—C(21)	112.5 (2)		

Sn(1)—S(1)—P(1)—C(4)	−30.5 (2)	S(1)—Sn(1)—C(3)—C(4)	22.8 (4)
S(1)—P(1)—C(4)—C(3)	55.7 (4)	C(3)—Sn(1)—S(1)—P(1)	7.2 (2)
Sn(1)—C(3)—C(4)—P(1)	−50.8 (4)		

No. Plane through atoms   Equation of the plane χ^2
1   C(11),C(12),C(13),C(14),C(15),C(16) $-0.9933x - 0.115y - 0.010z = -10.12$ Å   8.7
2   C(21),C(22),C(23),C(24),C(25),C(26) $-0.303x + 0.167y - 0.9384z = -2.88$ Å   15.3
Dihedral angle: 73.1 (2)

Possible hydrogen bonds

Donor···Acceptor	H···Acceptor	Donor—H···Acceptor
C(22)···S(1)	H(22a)···S(1)	C(22)—H(22a)···S(1)
3.360 (7)	2.850 (7)	114.2 (6)

squares with anisotropic displacement parameters for all non-H atoms and a common isotropic displacement parameter for the H atoms, which were placed in geometrically calculated positions (C—H 0.96 Å). 183 parameters were refined. Weights $w = 1.0/[\sigma^2(F) + (0.000215F^2)]$ led to a featureless analysis of variance in terms of $\sin\theta$ and F_o. The refinement converged to $S = 1.23$, $R = 0.032$, $wR = 0.033$, $(\Delta/\sigma)_{max} = 0.25$ (no extinction correction).

The correctness of the space-group choice was checked by using *MISSYM* (Le Page, 1987). The η refinements (Rogers, 1981) gave, with $wR = 0.033$, $\eta = 1.02\,(3)$ and confirm the proposed chirality. The largest peaks in the final $\Delta\rho$ map were $\pm 1.0\,(2)\,e\,\text{Å}^{-3}$. Atomic scattering factors for neutral atoms and real and imaginary dispersion terms were taken from *International Tables X-ray Crystallography* (1974, Vol. IV). The programs used were *PARST* (Nardelli, 1983), *SHELXTL-Plus* (Sheldrick, 1987), *PCK*83 (Williams, 1984), *PLATON* (Spek, 1982) and *MISSYM* (Le Page, 1987). The molecule and the numbering scheme are shown in Fig. 1. Positional parameters and the equivalent values of the anisotropic displacement parameters for the non-H atoms are given in Table 1.* Selected bond lengths and bond angles, torsion angles, least-squares

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, complete bond distances and bond angles and a stereoscopic view of the unit cell have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54956 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL0519]

planes, dihedral angles and possible H bonds are given in Table 2.

Related literature. The crystal structure of $(CH_3)_2Sn(Br)(CH_2)_2P(O)Ph'Bu$ is described by Weichmann, Mügge, Grand & Robert (1982) and literature on triorganotin halides with a pentacoordinated tin centre resulting from the intramolecular coordination of a donor function is given there.

References

Le Page, Y. (1987). *J. Appl. Cryst.* **20**, 264–269.
Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.
Rogers, D. (1981). *Acta Cryst.* A**37**, 734–741.
Sheldrick, G. M. (1987). *SHELXTL-Plus*, release 3.4. *An Integrated System for Solving, Refining, and Displaying Crystal Structures from Diffraction Data.* For Nicolet R3m/V crystallographic systems. Univ. of Göttingen, Germany.
Spek, A. L. (1982). *The EUCLID Package.* In *Computational Crystallography*, edited by D. Sayre, p. 528. Oxford: Clarendon Press.
Weichmann, H., Mügge, C., Grand, A. & Robert, J. (1982). *J. Organomet. Chem.* **238**, 343–356.
Williams, D. E. (1984). *PCK*83. *A Crystal and Molecular Packing Analysis Program.* Univ. of Louisville; QCPE Program No. 481, Dept. of Chemistry, Indiana Univ., Indiana, USA.

Structure of *trans*-Carbonylmethylbis(tri-*p*-tolylphosphine)iridium(I), *trans*-[Ir(CO)(CH₃){P(*p*-C₆H₄CH₃)₃}₂]

By Thomas S. Janik

Department of Chemistry, State University of New York at Fredonia, Fredonia, NY 14068, USA

and Melvyn Rowen Churchill, Ronald F. See, Sherri L. Randall, Jeffrey M. McFarland and Jim D. Atwood

Department of Chemistry, State University of New York at Buffalo, Buffalo, NY 14214, USA

Abstract. [Ir(CO)(CH₃){P(C₇H₇)₃}₂], C₄₄H₄₅IrOP₂, $M_r = 843.9$, orthorhombic, *Pna*2₁, $a = 21.654\,(3)$, $b = 10.581\,(1)$, $c = 16.820\,(1)$ Å, $V = 3853.8\,(6)$ Å³, $Z = 4$, $D_x = 1.455\,\text{g cm}^{-3}$, $\lambda(\text{Mo }K\bar{\alpha}) = 0.71073$ Å, $\mu(\text{Mo }K\alpha) = 35.62\,\text{cm}^{-1}$, $F(000) = 1696$, $T = 295$ K, $R = 5.73$, $wR = 5.01\%$ for 3527 unique data and $R = 3.08$, $wR = 3.82\%$ for 2291 data with $F_o > 6.0\sigma(F_o)$. The central Ir(I) atom has a square-planar coordination environment in which Ir—P(1) = 2.305 (3), Ir—P(2) = 2.302 (3), Ir—CH₃ = 2.206 (13) and Ir—CO = 1.867 (17) Å.

Experimental. We have been involved in the structural characterization of a number of simple complexes of Ir^I (Churchill, Fettinger, Rappoli & Atwood, 1987, and references therein). Among the complexes characterized was *trans*-[Ir(CO)(CH₃)(PPh₃)₂] (Rees, Churchill, Li & Atwood, 1985). This structure suffered from disorder with the methyl and carbonyl ligands scrambled on a statistical basis about the crystallographic inversion center. We now report the results of an X-ray diffraction study of the analogous compound *trans*-[Ir(CO)(CH₃){P(*p*-