

Related literature. Previous syntheses and structural studies of transition-metal complexes with tetraaza η^2 -phosphoranido ligands include (η^2 -cyclenphosphoranido)Mo(η^5 -C₅H₅)(CO)₂ (Lattman, Chopra, Cowley & Arif, 1986) and (η^2 -cyclamphosphoranido)Mo(η^5 -C₅H₅)(CO)₂ (Dupart, Grand, Pace & Riess, 1982). Other examples of η^2 -coordination of phosphoranido ligands have also been reported (Wachter, Mentzen & Riess, 1981; Jeanneaux, Grand & Riess, 1981).

This work was supported by the Robert A. Welch Foundation (ML and SSCC) and the Research Corporation (ML).

Acta Cryst. (1987). C43, 164–165

Structure of Triphenyl(4,4,5,5-tetramethyl-2-thioxo-1,3,2-dioxaphospholane-2-thiolato)tin

BY H. PREUT, V.-D. NGO AND F. HUBER

Fachbereich Chemie, Universität Dortmund, D-4600 Dortmund 50, Federal Republic of Germany

(Received 31 January 1986; accepted 28 July 1986)

Abstract. [Sn(C₆H₁₂O₂PS₂(C₆H₅)₃)]_n, $M_r = 561.3$, monoclinic, $P2_1/n$, $a = 14.511(6)$, $b = 11.536(7)$, $c = 15.435(14)$ Å, $\beta = 94.32(6)^\circ$, $U = 2576.5$ Å³, $Z = 4$, $D_x = 1.447$ Mg m⁻³, $\lambda(\text{Ag } K\alpha) = 0.56083$ Å, $\mu = 0.64$ mm⁻¹, $F(000) = 1136$, $T = 292(1)$ K. Final $R = 0.041$ for 1796 unique observed X-ray diffractometer data and 273 variables. Besides four atoms directly bound to Sn – three Sn–C(phenyl) bonds [mean bond distance 2.118(9) Å] and one Sn–S bond [2.436(3) Å] – there is a weak chelating interaction between the second S atom and Sn [Sn...S(2) 3.411(3) Å] and the coordination around Sn is intermediate between a tetrahedral and a trigonal-bipyramidal arrangement, with C(21) and S(2) [C(21)–Sn...S(2) 164.9(3)°] in the apical positions. Intermolecular distances exceed van der Waals distances.

Experimental. The compound was prepared from (C₆H₅)₃SnOH and HSP(S)(C₆H₁₂O₂) in ethanol. The precipitate was dried over P₄O₁₀. Colorless crystals from ethanol. M.p. 390–393 K. Crystal size: 0.16 × 0.22 × 0.13 mm. $\omega/2\theta$ scan. Scan speed: 5° min⁻¹ in θ . Nonius CAD-4 diffractometer, graphite-monochromated Ag $K\alpha$ radiation. Lattice parameters from least-squares fit with 25 reflexions up to $2\theta = 28.0^\circ$; four standard reflexions recorded every 2.5 h, only random deviations; 4441 reflexions, $1 \leq \theta \leq 18^\circ$, $-16 \leq h \leq 16$, $-1 \leq k \leq 12$, $0 \leq l \leq 17$, $[(\sin \theta)/\lambda]_{\max}$

References

- DUPART, J.-M., GRAND, A., PACE, S. & RIESS, J. G. (1982). *J. Am. Chem. Soc.* **104**, 2316–2318.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
- JEANNEAUX, F., GRAND, A. & RIESS, J. G. (1981). *J. Am. Chem. Soc.* **103**, 4272–4273.
- JOHNSON, C. K. (1976). ORTEPII. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee.
- LATTMAN, M., CHOPRA, S. K., COWLEY, A. H. & ARIF, A. M. (1986). *Organometallics*, **5**, 677–683.
- SHELDRICK, G. M. (1976). SHELX76. Program for crystal structure determination. Univ. of Cambridge, England.
- WACHTER, J., MENTZEN, B. F. & RIESS, J. G. (1981). *Angew. Chem. Int. Ed. Engl.* **20**, 284–285.

$= 0.55$ Å⁻¹; after averaging 3795 ($R_{\text{int}} = 0.024$) unique reflexions, 1796 with $I < 3\sigma(I)$, Lorentz–polarization correction and absorption correction *via* ψ scans; max./min. transmission 1.00/0.92; systematic absences conform to space group $P2_1/n$ (No. 14), structure solution *via* direct methods, ΔF syntheses and full-matrix least squares on F with 1796 reflexions; 273 refined parameters, anisotropic temperature factors for all non-H atoms, two common isotropic temperature

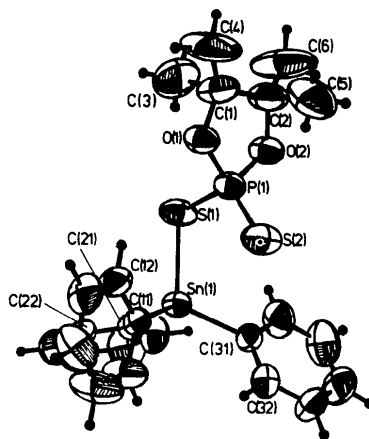


Fig. 1. General view of the molecule, showing atom numbering.

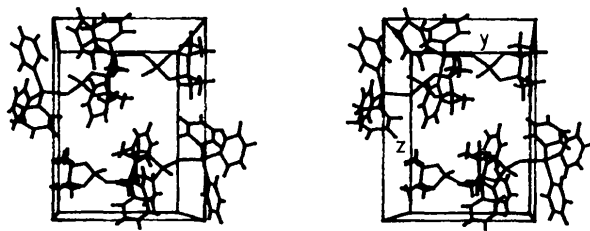


Fig. 2. Stereoscopic view of the unit cell.

Table 1. Fractional atomic coordinates and temperature factors ($\text{\AA}^2 \times 10^3$)

$$U_{eq} = (1/24)\pi^2 \sum_i \sum_j \beta_{ij} a_i \cdot a_j$$

	x	y	z	U_{eq}
Sn(1)	0.97341 (5)	0.39678 (6)	0.18983 (5)	3.67
S(1)	1.0434 (2)	0.5884 (3)	0.1855 (2)	5.36
S(2)	0.8372 (2)	0.6106 (3)	0.2638 (2)	6.08
P(1)	0.9434 (2)	0.6913 (3)	0.2309 (2)	4.34
O(1)	0.9227 (5)	0.7908 (6)	0.1604 (4)	5.2
O(2)	0.9968 (5)	0.7704 (6)	0.3020 (4)	5.2
C(1)	0.9637 (9)	0.898 (1)	0.1865 (8)	6.8
C(2)	0.9847 (9)	0.894 (1)	0.2820 (8)	7.1
C(3)	1.063 (1)	0.890 (1)	0.143 (1)	12.3
C(4)	0.920 (2)	0.996 (1)	0.145 (1)	14.9
C(5)	0.888 (1)	0.924 (1)	0.321 (1)	14.2
C(6)	1.058 (1)	0.961 (1)	0.325 (1)	14.6
C(11)	0.8645 (6)	0.3838 (9)	0.0921 (6)	3.8
C(12)	0.8398 (8)	0.475 (1)	0.0367 (7)	5.5
C(13)	0.7707 (9)	0.462 (1)	-0.0294 (7)	6.6
C(14)	0.7260 (8)	0.360 (1)	-0.0410 (8)	7.2
C(15)	0.7517 (9)	0.271 (1)	0.0102 (9)	8.5
C(16)	0.8207 (8)	0.281 (1)	0.0746 (8)	6.6
C(21)	1.0870 (6)	0.2932 (9)	0.1556 (6)	4.0
C(22)	1.0979 (7)	0.2569 (9)	0.0723 (7)	4.6
C(23)	1.1708 (8)	0.191 (1)	0.0518 (8)	6.7
C(24)	1.2349 (8)	0.157 (1)	0.1165 (9)	7.5
C(25)	1.2264 (8)	0.191 (1)	0.1980 (9)	7.4
C(26)	1.1529 (8)	0.257 (1)	0.2205 (7)	5.9
C(31)	0.9490 (7)	0.3481 (8)	0.3182 (6)	4.1
C(32)	0.8750 (8)	0.279 (1)	0.3332 (7)	5.5
C(33)	0.8605 (9)	0.243 (1)	0.4157 (8)	7.8
C(34)	0.921 (1)	0.271 (1)	0.4838 (8)	7.8
C(35)	0.9930 (9)	0.341 (1)	0.4703 (8)	8.1
C(36)	1.0088 (8)	0.380 (1)	0.3875 (7)	6.5

Table 2. Bond lengths (\AA) and bond angles ($^\circ$) with e.s.d.'s in parentheses

Sn(1)—C(11)	2.106 (8)	P(1)—O(1)	1.595 (7)
Sn(1)—C(31)	2.115 (9)	O(1)—C(1)	1.42 (1)
Sn(1)—C(21)	2.134 (9)	O(2)—C(2)	1.47 (1)
Sn(1)—S(1)	2.436 (3)	C(1)—C(3)	1.63 (2)
Sn(1)—S(2)	3.411 (3)	C(1)—C(4)	1.42 (2)
S(1)—P(1)	2.039 (4)	C(1)—C(2)	1.48 (2)
S(2)—P(1)	1.903 (4)	C(2)—C(5)	1.61 (2)
P(1)—O(2)	1.583 (7)	C(2)—C(6)	1.43 (2)
C(11)—Sn(1)—C(31)	118.9 (3)	C(2)—O(2)—P(1)	111.7 (6)
C(11)—Sn(1)—C(21)	109.6 (4)	O(1)—C(1)—C(2)	107.7 (9)
C(11)—Sn(1)—S(1)	109.7 (3)	O(1)—C(1)—C(3)	102 (1)
C(31)—Sn(1)—C(21)	105.7 (4)	O(1)—C(1)—C(4)	114 (1)
C(31)—Sn(1)—S(1)	111.5 (3)	C(2)—C(1)—C(3)	107 (1)
C(21)—Sn(1)—S(1)	99.8 (3)	C(2)—C(1)—C(4)	122 (1)
P(1)—S(1)—Sn(1)	102.1 (1)	C(3)—C(1)—C(4)	104 (1)
O(1)—P(1)—O(2)	96.6 (4)	O(2)—C(2)—C(1)	104.6 (9)
O(1)—P(1)—S(2)	114.8 (3)	O(2)—C(2)—C(5)	103 (1)
O(1)—P(1)—S(1)	106.7 (3)	O(2)—C(2)—C(6)	111 (1)
O(2)—P(1)—S(2)	117.5 (3)	C(1)—C(2)—C(5)	104 (1)
O(2)—P(1)—S(1)	104.5 (3)	C(1)—C(2)—C(6)	122 (1)
S(2)—P(1)—S(1)	114.8 (2)	C(5)—C(2)—C(6)	110 (1)
C(1)—O(1)—P(1)	112.4 (6)		
S(2)...Sn(1)—C(11)	82.4 (3)	S(2)...Sn(1)—C(31)	74.7 (3)
S(2)...Sn(1)—C(21)	164.9 (3)	S(2)...Sn(1)—S(1)	66.8 (8)

factors for H atoms [one group for C(methyl), one group for C(phenyl)]; H atoms in geometrically calculated positions (C—H 0.95 \AA), $w^{-1} = [\sigma^2(I) + (0.060F^2)^2]^{1/2}$, $S = 1.02$, $R = 0.041$, $wR = 0.049$, $\Delta/\sigma = 0.01$, largest peak in final ΔF map = $\pm 0.6 (2) e \text{\AA}^{-3}$; complex neutral-atom scattering factors from *International Tables for X-ray Crystallography* (1974); programs: *Enraf-Nonius Structure Determination Package* (Frenz, 1981), *ORTEP* (Johnson, 1976), *POP1* (van de Waal, 1976), *MULTAN82* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982).

The structure of the title compound is shown in Fig. 1 and in a stereoview in Fig. 2. Positional parameters and equivalent values of the anisotropic temperature factors are given in Table 1, * bond lengths and angles in Table 2. The unit cell contains four molecules. The four atoms directly bound to Sn form a distorted tetrahedron. Besides these bonds there is an Sn...S(2) [3.411 (3) \AA] interaction, widening the bond angle C(11)—Sn(1)—C(31) to 118.9 (3) $^\circ$ and reducing the angle S(1)—Sn(1)—C(21) to 99.8 (3) $^\circ$. The atom S(2) extends the coordination polyhedron around Sn to be intermediate between tetrahedral and trigonal-bipyramidal. The Sn atom deviates 0.183 (1) \AA from the plane through the atoms S(1), S(2), C(21).

Related literature. Molloy, Hossain, van der Helm, Zuckerman & Haiduc (1979); Molloy, Hossain, van der Helm, Zuckerman & Mullins (1981); Preut, Ngo & Huber (1986).

* 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 43293 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- FRENZ, B. A. (1981). *Enraf-Nonius Structure Determination Package*, 4th ed. Version 18. College Station, Texas.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Tables 2.2B and 2.3.1. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
- JOHNSON, C. K. (1976). *ORTEP*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee.
- MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1982). *MULTAN82. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- MOLLOY, K. C., HOSSAIN, M. B., VAN DER HELM, D., ZUCKERMAN, J. J. & HAIDUC, I. (1979). *Inorg. Chem.* **18**, 3507.
- MOLLOY, K. C., HOSSAIN, M. B., VAN DER HELM, D., ZUCKERMAN, J. J. & MULLINS, F. P. (1981). *Inorg. Chem.* **20**, 2172.
- PREUT, H., NGO, V.-D. & HUBER, F. (1986). *Acta Cryst.* **C42**, 809–811.
- WAAL, B. W. VAN DE (1976). *POP1* program – version of September 1976. *Plot of Packing Program*. Twente Univ. of Technology, Enschede, The Netherlands.