Tabelle 3. *Beste Ebene durch verschiedene Atomgruppierungen*

Die angegebenen Ebenengleichungen beziehen sich auf das kristallographische Achsensystem und sind in Å normiert. Für jedes Atom ist der Abstand zur betreffenden Ebene in Å angegeben (das Vorzeichen bezieht sich auf die Ebene).

Molekül (I)

Ebene C(2)–C(3)–C(5)–C(6)

$$-0{\cdot}139x + 8{\cdot}018y - 7{\cdot}307z - 2{\cdot}764 = 0$$

C(2) 0,004 (20); C(3) –0,004 (20); C(5) 0,005 (19); C(6) –0,004 (20); P(1) 0,440 (10); C(4) 0,169 (19); Cr –1,662 (9)

Winkel zwischen Ebene C(2)–C(3)–C(5)–C(6) und

P(2)–C(2)–C(6)	23,6 (7)°
C(3)–C(4)–C(5)	165,1 (10)°

Molekül (II)

Ebene C(2)–C(3)–C(5)–C(6)

$$-3{\cdot}167x + 11{\cdot}73y - 1{\cdot}904z - 6{\cdot}385 = 0$$

C(2) –0,008 (17); C(3) 0,009 (17); C(5) –0,009 (17); C(6) 0,008 (17); P(1) –0,450 (17); C(4) –0,068 (17); Cr 1,663 (17)

Winkel zwischen Ebene C(2)–C(3)–C(5)–C(6) und

P(1)–C(2)–C(6)	–26,6 (1)°
C(3)–C(4)–C(5)	–174,3 (2)°

gemittelten C–C Abstände in den Phenylringen von 1,39 (3) Å in (I) und 1,399 (6) Å in (II) stimmen gut mit der C=C Bindungslänge in Benzol überein.

Die Cr–C(Carbonyl) und die C–O Bindungslängen, 1,78 (2) und 1,19 (2) Å in (I) bzw. 1,841 (4) und 1,150 (6) Å in (II), stimmen mit denen, die für andere Tricarbonylchrom Komplexe (Sim, 1967) gefunden werden, gut überein. Die C–Cr–C–Winkel [88,2 in (I) und 87,6° in (II)] liegen sehr nahe bei 90° und die Cr–C–O–Winkel sehr nahe bei 180° [173,9 in (I) und 177,4° in (II)].

Ich möchte mich bei Herrn Professor K. Dimroth von der Universität Marburg für die Überlassung der Kristalle bedanken.

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The Structure of μ-Oxo-bis[tribenzyltin(IV)]

BY CHRISTOPHER GLIDEWELL* AND DAVID C. LILES

Chemistry Department, University of St Andrews, St Andrews, Fife KY16 9ST, Scotland

Abstract. $[Sn_2(C_7H_7)_6O]$, $C_{42}H_{42}OSn_2$, $M_r = 800 \cdot 14$, rhombohedral, $R\bar{3}$, $a = 9 \cdot 667$ (2) Å, $\alpha = 84 \cdot 05$ (3)°, $U = 889 \cdot 7$ Å³, $Z = 1$, $D_c = 1 \cdot 493$ Mg m⁻³, $F(000) = 402$, $\lambda(\text{Mo } K\alpha) = 0 \cdot 71069$ Å, $\mu = 1 \cdot 311$ mm⁻¹. Final $R = 0 \cdot 0197$. The structure consists of molecules lying along the threefold axis of the unit cell, in which the SnOSn fragment is linear and centrosymmetric. The Sn–O distance is $1 \cdot 919$ (0) Å and the Sn–C distance $2 \cdot 167$ (2) Å.

Introduction. As part of our study of organometallic oxo and hydroxo compounds, we have reported the structure of $O(Ph_3Sn)_2$ (Glidewell & Liles, 1978b), where the SnOSn angle is $137 \cdot 3$ (1)° and the Sn···Sn distance $3 \cdot 641$ (1) Å. Seeking further support for the view that the wide angles in μ-oxo compounds are primarily limited by the non-bonded $M \cdots M$ distances, we have determined the structure of the benzyl analogue $O[(PhCH_2)_3Sn]_2$ which turns out, unexpectedly, to contain a linear SnOSn fragment.

The compound was prepared in a single-step reaction from $SnBr_4$ (Bähr & Zoche, 1955).

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* To whom correspondence should be addressed.

Cell parameters were determined by a least-squares method from the reflecting positions of 24 reflections measured on a Philips PW 1100 automatic four-circle diffractometer with graphite-monochromatized Mo $K\alpha$ radiation. The intensities of 4825 reflections with $3 \leq \theta \leq 30°$ were measured by the $\omega/2\theta$ scan technique, with a scan width of $(0.84 + 0.1 \tan \theta)°$ in ω, a scan speed of $0.05°$ s^{-1} in ω and a maximum of two scans per reflection (a second scan was made if the total count obtained in the first scan was <100). Backgrounds were measured at both ends of the scan range for a time equal to (scan time)/2. Three standard reflections were measured every 5 hours and showed only small random deviations from their mean intensities. Lorentz and polarization corrections were applied but none for absorption.

Solution and refinement of the structure were carried out using *SHELX* 76 (Sheldrick, 1976).

The intramolecular Sn$\cdots$Sn distance was found from a Patterson synthesis. E statistics were inconclusive but tended to indicate a non-centrosymmetric space group. Consequently initial refinement was carried out in $R3$. The two independent Sn atoms were placed on the threefold axis symmetrically about $(\frac{1}{2},\frac{1}{2},\frac{1}{2})$. A difference synthesis yielded positions for all other non-hydrogen atoms. Full-matrix least-squares refinement with 2664 independent reflections having $F_o \geq 6\sigma(F_o)$, with individual isotropic temperature factors for all non-hydrogen atoms, and the position of Sn(1) fixed (to fix the origin) and Sn(2) and O constrained to lie on the threefold axis (62 variables), and a weighting scheme $w = [\sigma(F_o)]^{-2}$ yielded $R = \sum \Delta/\sum F_o$ ($\Delta = |F_o - F_c|$) = 0.0405, $R_w = \sum w^{1/2} \Delta/\sum w^{1/2} F_o = 0.0430$ and $R_G = (\sum w\Delta^2/\sum wF_o^2)^{1/2} = 0.0548$. The introduction of anisotropic temperature factors for the Sn atoms (64 variables) reduced R_G to 0.0540. The matrix was then blocked such that the O atom refined in every cycle, but the two crystallographically independent benzyl—Sn fragments refined in alternate cycles, to allow the position of Sn(1) to refine (but still constrained to lie on the threefold axis). Refinement (65 variables) reduced R_G to 0.0534. Further cycles with the matrix blocked as above, and with anisotropic temperature factors for all non-hydrogen atoms (136 variables) reduced R_G to 0.0429. At this stage a difference synthesis furnished positions for all the H atoms. Refinement with the matrix blocked as before and with separate common isotropic temperature factors for the phenyl and methylene H atoms in each of the two crystallographically independent benzyl groups (182 variables) reduced R to 0.0219, R_w to 0.0222 and R_G to 0.0259. At each stage of the refinement the reduction in R_G was significant at the 99.5% level (Hamilton, 1965). At this stage the refinement in $R3$ had converged; however, the geometry of the molecule was unsatisfactory with significant differences between chemically equivalent bond lengths, e.g. up to

0.093 Å (18 σ) for equivalent phenyl C—C bonds and 0.149 Å (18 σ) for the two Sn—O bonds.

A refinement in $R\bar{3}$ was therefore tried, starting with mean parameter values from the final $R3$ refinement. Full-matrix least-squares refinement with the O atom fixed at $(\frac{1}{2},\frac{1}{2},\frac{1}{2})$ and the Sn atom constrained to lie on the threefold axis, with anisotropic temperature factors for all non-hydrogen atoms, and separate common temperature factors for the phenyl and methylene H atoms (92 variables) yielded $R = 0.0197$, $R_w = 0.0202$ and $R_G = 0.0248$, for 1569 independent reflections having $F_o \geq 6\sigma(F_o)$. The reductions in the R indices compared to the $R3$ refinement and the significant improvement in the agreement between chemically equivalent bond lengths led to the adoption of the centrosymmetric space group.

A final difference synthesis revealed no significant residual electron density, in particular around the O atom. Attempts to allow the O atom to refine off the threefold axis (*i.e.* as a disordered atom) to three or six positions each with occupancies of $\frac{1}{3}$ or $\frac{1}{6}$ respectively in $R\bar{3}$ or to six positions in $R3$ all led to unstable refinements with significant increases in R_G.

Complex neutral-atom scattering factors (Cromer & Mann, 1968; Doyle & Turner, 1968) were employed for all atoms.

Table 1. *Atom coordinates*

	x	y	z
Sn	0.3957 (0)	0.3957 (0)	0.3957 (0)
O	0.5000 (0)	0.5000 (0)	0.5000 (0)
C(1)	0.4912 (2)	0.1824 (2)	0.4044 (3)
C(11)	0.6459 (2)	0.1705 (2)	0.3722 (2)
C(12)	0.7088 (2)	0.1686 (2)	0.2356 (2)
C(13)	0.8534 (2)	0.1557 (2)	0.2096 (2)
C(14)	0.9369 (2)	0.1448 (2)	0.3167 (3)
C(15)	0.8759 (2)	0.1493 (2)	0.4523 (2)
C(16)	0.7324 (2)	0.1626 (2)	0.4793 (2)
H(1)	0.461 (3)	0.142 (3)	0.494 (3)
H(2)	0.442 (3)	0.143 (3)	0.333 (3)
H(12)	0.651 (3)	0.177 (3)	0.162 (3)
H(13)	0.897 (3)	0.156 (3)	0.115 (3)
H(14)	1.039 (3)	0.134 (3)	0.292 (3)
H(15)	0.926 (3)	0.146 (3)	0.532 (3)
H(16)	0.689 (3)	0.163 (3)	0.573 (3)

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

The superscript (i) refers to the symmetry position $1 - x$, $1 - y$, $1 - z$.

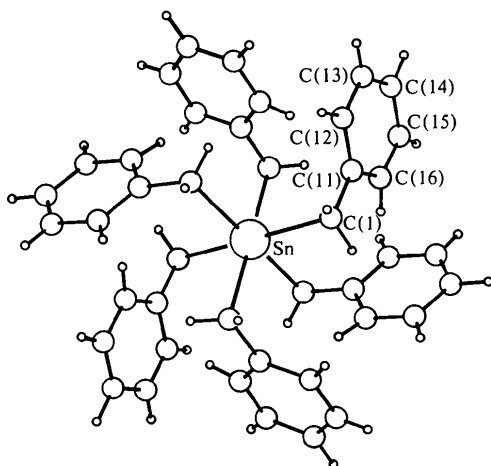|			
Sn—O	1.919 (0)	Sn—O—Sn^i	180.0 (0)
Sn—C(1)	2.167 (2)	O—Sn—C(1)	108.0 (1)
C(1)—C(11)	1.490 (3)	Sn—C(1)—C(11)	113.9 (1)
C(11)—C(12)	1.396 (3)	C(1)—C(11)—C(12)	122.1 (2)
C(12)—C(13)	1.389 (3)	C(1)—C(11)—C(16)	120.1 (2)
C(13)—C(14)	1.367 (3)	C(11)—C(12)—C(13)	120.5 (2)
C(14)—C(15)	1.384 (3)	C(12)—C(13)—C(14)	120.8 (2)
C(15)—C(16)	1.380 (3)	C(13)—C(14)—C(15)	119.3 (2)
C(16)—C(11)	1.389 (3)	C(14)—C(15)—C(16)	120.3 (2)
C(1)—H(1)	0.94 (3)	C(15)—C(16)—C(11)	121.2 (2)
C(1)—H(2)	1.01 (3)	C(16)—C(11)—C(12)	117.8 (2)
C(aryl)—H(mean)	0.96 (2)	C(aryl)—C(aryl)—H(mean)	120 (3)

Fig. 1. The moelcule of O[(PhCH$_2$)$_3$Sn]$_2$ viewed down the threefold axis, showing the atom numbering.

The positional parameters are given in Table 1, bond lengths and angles in Table 2.* No correction for thermal motion was applied to the bond lengths.

Fig. 1 shows a view of the molecule down the three-fold axis, and the numbering of the atoms. In the tables, the H atoms of the phenyl rings are given the numbers of the C atoms to which they are bonded.

Discussion. The Sn—O distance, 1·919 (1) Å, is significantly shorter than that in the phenyl analogue, O(Ph$_3$Sn)$_2$, 1·955 (1) Å (Glidewell & Liles, 1978b), in which the SnOSn fragment is markedly non-linear. In a similar way the P—N distance in linear [N(PPh$_3$)$_2$]$^+$, 1·539 (2) Å, is significantly less than that in the non-

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34339 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

linear forms of this cation where the range of PNP angles is 134·6–141·8° and the range of P—N distances 1·570–1·586 Å with a mean of 1·575 (2) Å (Wilson & Bau, 1974). Apart from the single linear occurrence of the cation [N(PPh$_3$)$_2$]$^+$ (Wilson & Bau, 1974), the only other p-block analogue of the present molecule is the linear O(Ph$_3$Si)$_2$ (Glidewell & Liles, 1978a): however, amongst d-block species, O[(PhCH$_2$)$_3$Ti]$_2$ is isomorphous and isostructural with the compound described here (Stoeckli-Evans, 1974).

It has been suggested (Glidewell, 1978a,b) that in molecules or ions of general type O(MR_n)$_2$, linearity at the O atom may be expected if M is of low electro-negativity: when R is an electron donor, it will enhance the effect of the low electronegativity of M. The results reported here for O[(PhCH$_2$)$_3$Sn]$_2$ and those earlier reported for analogous O(Ph$_3$M)$_2$ (Glidewell & Liles, 1978a,b) are consistent with this suggestion.

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Tetrakis[{2-[(3-aminopropyl)amino]ethanolato}copper(II)] Dimalonate Pentahydrate

By Aarne Pajunen

Department of Inorganic Chemistry, University of Helsinki, Vuorikatu 20, SF-00100 Helsinki 10, Finland

Abstract. [Cu$_4$(C$_5$H$_{13}$N$_2$O)$_4$](C$_3$H$_2$O$_4$)$_2$.5H$_2$O, C$_{20}$H$_{52}$-Cu$_4$N$_8$O$_4^{4+}$.2C$_3$H$_2$O$_4^{2-}$.5H$_2$O, tetragonal, $I4_1/a$, $a = b = 15·749$ (16), $c = 34·407$ (19) Å, $Z = 8$, $D_c = 1·58$, $D_m = 1·60$ Mg m^{-3}, $R = 0·067$ for 2075 observed reflections. The structure contains tetranuclear complex cations with a cubane-type Cu$_4$O$_4$ core.

Introduction. The title compound is a member of a series of tetranuclear complexes formed by 2-[(3-aminopropyl)amino]ethanol and Cu^{II} (Nieminen, 1977; Nieminen & Pajunen, 1978; Pajunen & Nieminen, 1975). The blue plate-like crystals were prepared by a method described earlier (Nieminen & Pajunen, 1972).