

Crystal Structure of a Binuclear Tin Complex with an Sn-Sn Bond

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Summary Di- μ -acetato-bis(diphenyltin)(Sn-Sn) has a slightly distorted trigonal-bipyramidal configuration with two phenyl groups and a tin atom occupying equatorial positions, and oxygen atoms in axial sites.

PREVIOUS studies on diacyloxyditin compounds in solution¹ have indicated a monomeric acetate-bridged structure. We have studied, by X-ray methods, the solid state structure of di- μ -acetato-bis(diphenyltin)(Sn-Sn) $[\text{Ph}_4\text{Sn}_2(\text{OOCMe})_2]$, prepared as described in refs. 2 and 3. Crystals of $[\text{Ph}_4\text{Sn}_2(\text{OOCCH}_3)_2]$ are triclinic, with $a = 8.843$, $b = 17.828$, $c = 8.755$ Å, $\alpha = 88.28^\circ$, $\beta = 76.50^\circ$, $\gamma = 98.97^\circ$, $U = 1322.5$ Å³, $M = 663.9$, $Z = 2$, $D_c = 1.66$ g cm⁻³; space group $P\bar{1}$. Intensity data were measured with Cu- K_α radiation using a Siemens four-circle automatic diffractometer, ca. 3300 reflexions (3100 observed) ($\theta \leq 60^\circ$) being collected. The structure was solved by a combination of sharpened Patterson and Fourier syntheses, and direct methods. The trial structure, with anisotropic vibrations of tin atoms, was refined by least-squares. The R index is now 0.11 and refinement is continuing.

The crystals consist of chemically equivalent molecules which differ only in the rotation of their phenyl groups. There is a crystallographic centre of symmetry half way between the tin atoms. The bond lengths (Å) and angles ($^\circ$), standard deviation ca. 2° , for the "inner core" of the complex are shown in the Figure. In each molecule the two bonded tin atoms are held by two bridging acetate ligands, the tin atoms being five-co-ordinated in a slightly distorted trigonal bipyramidal configuration. The major distortions are the O-Sn-O angle and the three equatorial angles.

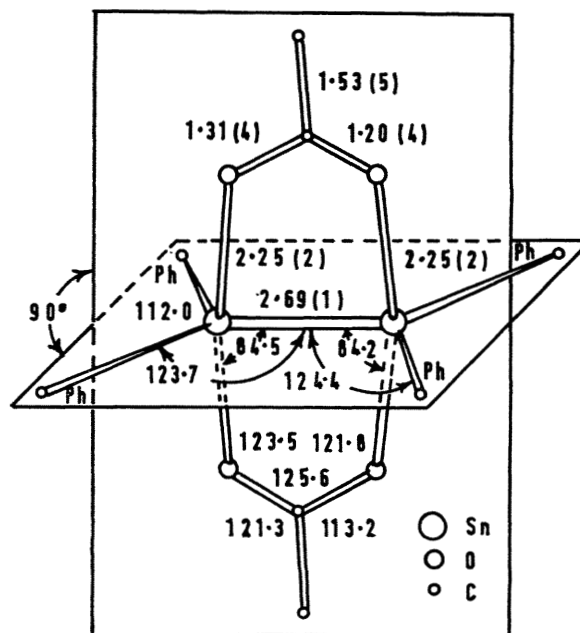


FIGURE. Co-ordination about the tin atoms in di- μ -acetato-bis(diphenyltin)(Sn-Sn).

The Sn-Sn bond length is appreciably smaller than the sum of the covalent radii (2.80 Å)⁴. The equatorial plane contains the four Sn-C bonds, with the mean value (2.15

± 0.02 Å) characteristic of the Ph-Sn bond;⁵ also the plane of the acetate groups is normal to the equatorial plane in agreement with the generalisation that, in trigonal bipyramidal structures, the more electronegative atoms occupy the axial sites. However the Sn-O bond is unusually long.^{7,8}

The difference in the C-O bonds in the bridging acetate

group is similar to that found in the μ -acetato-uranyl compound.⁹

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