Crystal Structure of Tetraphenyltin(IV) (a Redetermination)

Lutz M. Engelhardt, Wing-Por Leung, Colin L. Raston and Allan H. White

Department of Physical and Inorganic Chemistry, University of Western Australia, Nedlands, W.A. 6009.

Abstract

The crystal structure of tetraphenyltin(iv) has been redetermined at 295(1) K, full-matrix least-squares refinement yielding a residual of 0.031 for 557 independent 'observed' reflections. The tin-phenyl carbon distance is 2.143(5) Å, with the angles subtended at the tin atom being 108.9(2) and 110.5(2)°.

In the course of a series of structural studies on tin(IV)/alkyl systems in progress in this laboratory, it became apparent that the standard tin(IV)-phenyl carbon distances available for tetraphenyltin(IV), being derived from film or limited diffractometer data, were of limited precision and inappropriate for comparative purposes in the present context. Accordingly, a redetermination of the crystal structure of tetraphenyltin(IV) has been carried out at 295 K; although the results differ only trivially from those previously reported,^{1,2} they offer a significant improvement in precision. As described previously, the molecule has $\overline{4}$ symmetry, with only one phenyl ring being crystallographically independent. We find the carbon atom skeleton of the ring to be planar ($\sigma 0.004$ Å, $\chi^2 1.15$), with distances lying in the range 1.38(1)-1.41(1) Å and angles within the ring $118.5(5)-121.2(6)^{\circ}$; Sn-C-C angles are 121.7(4), $119.8(4)^{\circ}$. About the tin atom, Sn-C is 2.143(5) Å; the two independent C-Sn-C angles are $110.5(2)^{\circ}$ (about the $\overline{4}$ axis) and $108.9(2)^{\circ}$.

Crystallography

Crystal data.— $C_{24}H_{20}Sn$, M 427·1, tetragonal, space group $P\bar{4}2_1c$ (D_2^4 , No. 114), a 12·068(3), $c 6 \cdot 558(2)$ Å, U 955·1(4) Å³, D_m , D_c (ref.¹) 1·49, 1·485 g cm⁻³, Z 2, monochromatic Mo K α radiation, $\lambda 0.71069$ Å, $\mu 13.5$ cm⁻¹. Specimen size 0·07 by 0·14 by 0·32 mm. T 295(1) K.

Structure determination.—A unique data set was measured to $2\theta_{max} = 60^{\circ}$. A Syntex P2₁ fourcircle diffractometer was used in conventional $2\theta/\theta$ scan mode yielding 814 independent reflections, 557 of which with $I > 3\sigma(I)$ were considered 'observed' and used in the full-matrix least-squares refinement after analytical absorption correction. For the non-hydrogen atoms, $(x, y, z, U_{ij}$ (anisotropic)) were refined; for the hydrogen atoms (x, y, z, U(isotropic)) were refined. At convergence residuals were (R, R') 0.031, 0.038, reflection weights being $(\sigma^2(F_0) + 0.0005(F_0)^2)^{-1}$. Neutral atom scattering factors were used, those from the non-hydrogen atoms being corrected for anomalous

¹ Chieh, P. C., and Trotter, J., J. Chem. Soc. A, 1970, 912.

² Akhmed, N. A., and Aleksandrov, G. G., Zh. Strukt. Khim., 1970, 11, 891.

| Atom | x | У | z | $10^{3}U_{(11)}$ | $10^{3}U_{22}$ | $10^{3}U_{33}$ | $10^{3}U_{12}$ | $10^{3}U_{13}$ | $10^{3}U_{23}$ |
|------|------------|-----------|------------|------------------|----------------|----------------|----------------|----------------|----------------|
| Sn | 0 | 0 | 0 | 33.9(2) | 33.9(2) | 39·0(3) | | | _ |
| C(1) | 0.0195(4) | 0.1446(5) | 0.1862(8) | 35(4) | 39(3) | 35(2) | 0(2) | 6(2) | 6(2) |
| C(2) | 0.1051(6) | 0.1537(6) | 0.3271(11) | 41(3) | 54(4) | 49(3) | 0(3) | -8(3) | -2(3) |
| H(2) | 0.155(6) | 0.090(7) | 0.353(11) | 71(23) | | | | | |
| C(2) | 0.1169(6) | 0.2476(7) | 0 4465(11) | 59(4) | 64(4) | 39(4) | - 17(4) | - 5(3) | -11(3) |
| H(3) | 0.170(7) | 0:261(8) | 0.572(14) | 107(31) | | | | | |
| C(4) | 0.0435(7) | 0.3348(6) | 0 4256(11) | 71(4) | 44(4) | 50(3) | - 8(3) | 13(4) | -14(3) |
| H(4) | 0.040(5) | 0.399(5) | 0.494(9) | 54(18) | | | | | |
| C(5) | -0.0425(7) | 0.3270(5) | 0.2880(12) | 71(4) | 35(3) | 61(4) | 14(3) | 1(4) | -2(3) |
| H(5) | -0.088(5) | 0.376(5) | 0.284(11) | 48(20) | | | | | |
| C(6) | -0.0550(5) | 0.2335(5) | 0.1660(10) | 48(3) | 38(3) | 46(3) | 5(3) | -8(3) | - 1(3) |
| H(6) | -0.116(5) | 0.230(5) | 0.074(10) | 35(16) | | | | | |

| Table 1. Atom coordinates and thermal parameter | Table 1. | Atom | coordinates | and | thermal | parameter |
|---|----------|------|-------------|-----|---------|-----------|
|---|----------|------|-------------|-----|---------|-----------|

U in Å². The form of the anisotropic thermal parameters is

| $exp(-2\pi^2)$ | (IIh | $^{2}a^{*2} +$ | • • • - | 211. | L16*c*)) |
|-----------------|----------------|----------------|---------|-------|----------|
| $C_{A}O(-2\pi)$ | $U_{11}U_{11}$ | и – | · · · • | イムしつつ | h m c H |

dispersion (f', f'').³ Computation used the x-RAY 76 program system⁴ implemented on a Perkin-Elmer 3240 computer by S. R. Hall. Material deposited consists of Tables of structure factor amplitudes.[†] Table 1 lists the atom coordinates. Atom numbering follows that of ref.¹ and of systematic nomenclature whereby the carbon attached to the metal is numbered one.

Manuscript received 15 July 1982

† Copies are available on application to the Editor-in-Chief, Editorial and Publications Service, CSIRO, P.O. Box 89, East Melbourne, Vic. 3002.

³ Ibers, J. A., and Hamilton, W. C., (Eds) 'International Tables for X-Ray Crystallography' Vol. 4 (Kynoch Press: Birmingham 1974).

⁴ Stewart, J. M., (Ed.) 'The x-RAY System—Version of March, 1976' Technical Report TR-446, Computer Science Center, University of Maryland, U.S.A.