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# STRUCTURES OF TETRAPHENYLTIN (REDETERMINATION) AND TETRA(*o*-TOLYL)TIN

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## Summary

Complete X-ray structure analyses of tetraphenyltin and tetra(*o*-tolyl)tin have been performed. The crystals are tetragonal ($P\bar{4}2_1c, Z = 2$). The introduction of a methyl group in the *ortho*-position causes lengthening of the Sn–C bond, while the whole molecular conformation and packing mode remain the same.

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## Introduction

During the systematic study of the crystal and molecular structures of organotin compounds it became nesessary to obtain precise data on the substances $R_4Sn$. So far, the structures of tetraphenyltin [1,2], tetra(*m*-tolyl)tin [3] and tetra(*p*-tolyl)tin [4] are known. In this study we report the results of X-ray structure analysis of tetra(*o*-tolyl)tin (II) and tetraphenyltin (I). For the latter we carried out an independent structure determination because of the inadequate (for our purposes) accuracy of the reported data [1,2].

## Experimental

The unit cell parameter determination (the structural class $P\bar{4}2_1c$, $Z = 2(\bar{4})$ for both I and II) and data collection were performed using a Syntex P$\bar{1}$ automatic diffractometer ($\lambda$(Mo–$K\alpha$), graphite monochromator, $\theta/2\theta$ scan method, $2\theta_{max} =$ 60° for I and 50° for II). The main crystallographic parameters are given in Table 1. The data were corrected for Lorentz and polarization effects and also for absorption. The structures was solved by heavy atom technique; coordinates of H atoms were localized from the difference syntheses. The refinement was performed by full-matrix least-squares using anisotropic (Sn,C) and isotropic (H) thermal parameters. The final $R$ values are 0.022 (I, 543 independent reflections with $I > 3\sigma$(I)) and 0.020 (II, 433 reflections). All calculations were made using the SHELXTL program incorpo-

TABLE 1

CRYSTALLOGRAPHIC DATA

| | I | II |
|---|---|---|
| $a$(Å) | 12.058(1) | 12.021(1) |
| $c$(Å) | 6.581(1) | 8.054(1) |
| $V$(Å$^3$) | 956.8(2) | 1163.8(2) |
| $\rho$(calc)(g/cm$^3$) | 1.48 | 1.38 |
| $\mu$(cm$^{-1}$)(Mo) | 13.5 | 11.2 |

rated in the system Nicolet-R3 on a Nova-3 computer. Table 2 presents atomic coordinates and thermal parameters $U_{eq}$ (Sn,C) and $U_{iso}$(H). The general view of the molecule II with the atom numbering system is shown in Fig. 1.

TABLE 2

ATOMIC COORDINATES WITH e.s.d.'s ($\times 10^4$ FOR C AND $\times 10^3$ FOR H) AND $U_{eq}$ ($\times 10^3$ FOR Sn AND C) AND $U$ ($\times 10^2$ FOR H) [a]

| | $x$ | $y$ | $z$ | $U_{eq}(U)$(Å$^2$) |
|---|---|---|---|---|
| Sn | 0 | 0 | 0 | 43(1) |
| | 0 | 0 | 0 | 47(1) |
| C(1) | 1451(4) | −190(4) | 1836(7) | 44(1) |
| | 1456(4) | 310(4) | 1483(7) | 49(2) |
| C(2) | 2330(4) | 542(4) | 1669(8) | 53(2) |
| | 2420(5) | −328(5) | 1134(8) | 60(2) |
| C(3) | 3264(4) | 418(5) | 2874(8) | 63(2) |
| | 3396(5) | −133(7) | 1963(9) | 76(3) |
| C(4) | 3341(4) | −413(5) | 4229(8) | 66(2) |
| | 3455(6) | 672(6) | 3135(10) | 78(3) |
| C(5) | 2471(5) | −1161(4) | 4437(8) | 63(2) |
| | 2541(7) | 1289(5) | 3518(9) | 73(3) |
| C(6) | 1539(4) | −1036(4) | 3259(8) | 53(2) |
| | 1521(5) | 1108(5) | 2711(8) | 58(2) |
| C(7) | – | – | – | – |
| | 529(6) | 1761(6) | 3231(9) | 87(3) |
| H(2) | 228(6) | 108(6) | 72(10) | 7(2) |
| | 230(6) | −99(6) | 27(9) | 6(2) |
| H(3) | 381(6) | 87(6) | 265(10) | 7(2) |
| | 400(6) | −60(6) | 164(9) | 9(2) |
| H(4) | 395(5) | −52(5) | 504(10) | 8(2) |
| | 402(6) | 74(6) | 374(9) | 7(2) |
| H(5) | 250(5) | −167(5) | 533(10) | 6(2) |
| | 253(6) | 181(6) | 439(9) | 5(2) |
| H(6) | 101(6) | −148(6) | 345(10) | 7(2) |
| | – | – | – | – |
| H(71) | – | – | – | – |
| | 10(6) | 194(6) | 249(9) | 10(2) |
| H(72) | – | – | – | – |
| | 66(6) | 247(6) | 387(9) | 10(2) |
| H(73) | – | – | – | – |
| | 3(6) | 146(6) | 385(9) | 17(2) |

[a] For each atom the first line corresponds to the structure I and the second one to the structure II.

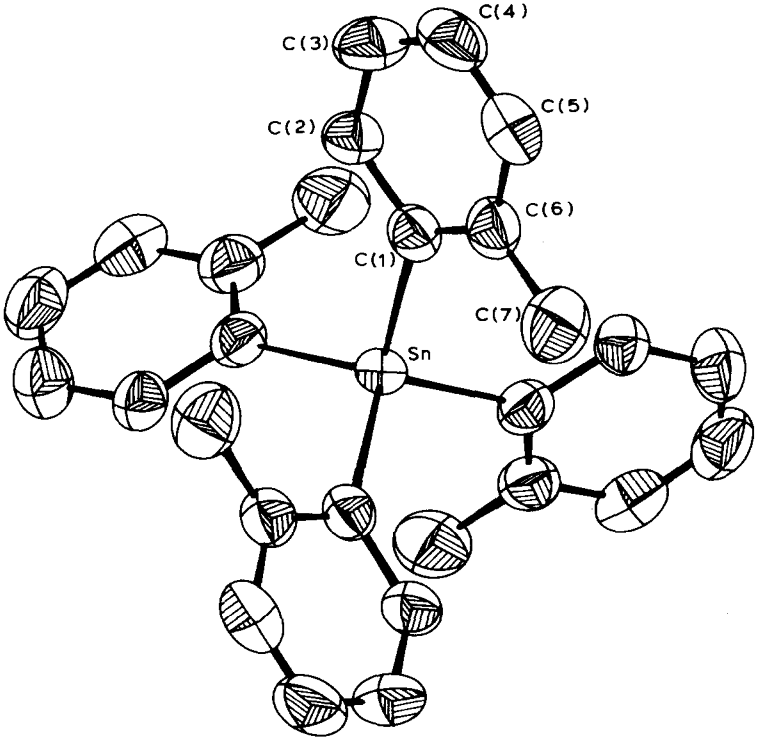

Fig. 1. An ORTEP drawing (50% probability) of tetra(*o*-tolyl)tin.

## Discussion

Bond lengths and valence angles with their e.s.d.'s are given in Tables 3 and 4 *. The interatomic distances C–H are in the limits 0.84–0.92 Å(I) and 0.82–1.07 Å(II). Generally, our results are in accordance with those presented earlier [1,2]. In the

TABLE 3

INTERATOMIC DISTANCES (Å)

| | I | | II | |
|---|---|---|---|---|
| Sn–C(1) | 2.139(4) | 2.144 | 2.152(5) | 2.160 |
| C(1)–C(2) | 1.384(6) | 1.391 | 1.418(8) | 1.429 |
| C(2)–C(3) | 1.385(7) | 1.389 | 1.370(9) | 1.375 |
| C(3)–C(4) | 1.344(8) | 1.351 | 1.354(11) | 1.361 |
| C(4)–C(5) | 1.390(8) | 1.398 | 1.361(10) | 1.371 |
| C(5)–C(6) | 1.374(8) | 1.377 | 1.405(10) | 1.410 |
| C(6)–C(1) | 1.388(7) | 1.395 | 1.380(8) | 1.387 |
| C(6)–C(7) | – | – | 1.489(9) | 1.500 |

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* In Table 3 the second column for each compound corresponds to the distances corrected for rigid body libration of the phenyl groups.

TABLE 4

VALENCE ANGLES (deg)

| | I | II |
|---|---|---|
| C(1)–Sn–C(1a) | 108.6(1) | 107.9(1) |
| C(1)–Sn–C(1b) | 111.2(2) | 112.6(3) |
| Sn–C(1)–C(2) | 120.9(3) | 117.5(4) |
| Sn–C(1)–C(6) | 121.5(3) | 124.4(4) |
| C(2)–C(1)–C(6) | 117.6(4) | 118.1(5) |
| C(1)–C(2)–C(3) | 120.6(5) | 120.7(6) |
| C(2)–C(3)–C(4) | 121.1(5) | 120.5(6) |
| C(3)–C(4)–C(5) | 119.8(5) | 120.4(7) |
| C(4)–C(5)–C(6) | 119.4(5) | 120.9(6) |
| C(5)–C(6)–C(1) | 121.6(5) | 119.3(6) |
| C(5)–C(6)–C(7) | – | 119.1(6) |
| C(1)–C(6)–C(7) | – | 121.5(6) |

molecule I the phenyl ring is planar (maximal deviation 0.007(5) Å), the Sn atom is in the plane (0.006 Å). In the molecule II the situation is somewhat different. The ring is also planar (maximum deviation 0.013(5) Å) but Sn and C(7) are out of the plane by 0.135 and −0.088(7) Å, respectively. This may be explained by the steric effects caused by the presence of a methyl group in the *ortho* position. This may also be the reason for the lengthening of the Sn–C distance in II with respect to I (Table 3). The angles between the planes of phenyl groups connected by the axis $\bar{4}$ are practically identical (88.5 and 119.1° in I and 88.0 and 118.9° in II).

It is interesting to note that during the transition from the phenyl substituents to *o*-,*m*- and *p*-tolyl ones the molecule $SnR_4$ retains its own $D_{2d}$ symmetry; however, the packing mode of the tetrahedrons differs: $SnPh_4$ and Sn(*o*-tolyl)$_4$ belong to the structural class $P\bar{4}2_1c, Z = 2(\bar{4})$ (the same as for the structures of $SiPh_4$ [5] and $GePh_4$[6]) whereas Sn(*p*-tolyl)$_4$ belongs to $I\bar{4}, Z = 2(\bar{4})$ and Sn(*m*-tolyl)$_4$ to $I4_1/a, Z = 4(\bar{4})$.

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