Acta Crystallographica Section C
**Crystal Structure
Communications**
ISSN 0108-2701

# ($p$-Nitrobenzoato)triphenyltin at 298 K

**R. Alan Howie,[a]* James L. Wardell,[a] Solange M. S. V. Wardell[a] and Philip J. Cox[b]**

[a]Department of Chemistry, University of Aberdeen, Meston Walk, Aberdeen AB24 3UE, Scotland, and [b]School of Pharmacy, The Robert Gordon University, Schoolhill, Aberdeen AB10 1FR, Scotland
Correspondence e-mail: r.a.howie@abdn.ac.uk

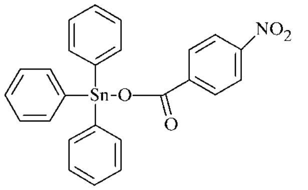
Received 15 March 2000
Accepted 22 March 2000

Data validation number: IUC0000086

The structure at 298 K described here, $[Sn(C_6H_5)_3(C_7H_4NO_4)]$, completely confirms the results at 173 K obtained previously [Weng, Das & Robinson (1990), *Malays. J. Sci.* **12**, 57]. In both structures, weak interaction between Sn and the carbonyl O atom of the benzoate group provides a distorted trigonal-pyramidal environment at the Sn atom derived from its pseudo-tetrahedral primary coordination in both molecules of the asymmetric unit.

## Comment

It was only upon completion of the solution and refinement of the 298 K structure reported here for (I) that a search of the Cambridge Structural Database (Allen & Kennard, 1993) by means of the *CSSR* component of the EPSRC's Chemical Database Service at Daresbury (Fletcher *et al.*, 1996) revealed the existence of the 173 K structure described by Weng *et al.* (1990; CSD No. 170773, CODEN RACFUW). Aside from the reversal in direction of cell edges *a* and *b* and the increased cell dimensions and thermal parameters at 298 K, the two structures are identical. Thus, making due allowance for the different labelling schemes, the description of the 298 K structure which follows applies equally to both structures.



(I)

The asymmetric unit consists of two independent molecules, *A* and *B*, whose atoms are labelled in an identical manner and distinguished by suffix *A* or *B*, except for the Sn atoms, which are distinguished by number (1 or 2). As a consequence, geometric parameters are given below in pairs with those for molecule *A* preceding the corresponding value in square brackets for molecule *B*.

With a few exceptions, the bond lengths and angles observed are unremarkable. However, in the $p$-nitrobenzoate groups, both the carboxylate and nitro substituents are rotated around the C—*X* bond (*X* = C or N) by 26.8 (6) [13.4 (6)°] and 19.2 (7)° [16.0 (7)°], respectively, to displace O from the plane of the benzene ring. Also of interest is the coordination of Sn (Table 1). The primary coordination by O1, C8, C14 and C20 is tetrahedral in nature but highly distorted, as evidenced for example by the O1—Sn—C20 angle. The distortion is perceived to be of the order of 11.7° [15.2°] towards a trigonal-bipyramidal configuration with the introduction of O2 axial and *trans* to C20, and O1, C8 and C14 equatorial. In molecule *A*, the axial Sn—C20 bond is appreciably longer than the equatorial Sn—C bonds, but a similar effect is not apparent in molecule *B*. With the inclusion of the Sn—O2 contact, the benzoate carboxylate group now acts in a bidentate manner, but with a bite angle of only 50.10 (11)° [51.78 (12)°] which, despite amelioration by the length of the Sn—O2 bond, renders the bipyramid extremely distorted also.

The two molecules in the asymmetric unit are clearly generally quite similar. However, comparison of the values given pairwise above and in Table 1 clearly shows that they are not identical. The differences between them are attributed to packing effects rather than fundamental structural differences.

## Experimental

The title compound was prepared from bis(triphenyl)tin oxide and $p$-nitrobenzoic acid (1:2 molar ratio) in toluene using a Dean–Stark separator. The residue, after removal of solvent, was recrystallized from chloroform/petroleum ether (333–353 K).

### *Crystal data*

$[Sn(C_6H_5)_3(C_7H_4NO_4)]$
$M_r = 516.10$
Triclinic, $P\overline{1}$
$a = 8.949$ (11) Å
$b = 14.302$ (16) Å
$c = 17.530$ (16) Å
$\alpha = 94.89$ (8)°
$\beta = 83.79$ (8)°
$\gamma = 96.24$ (9)°
$V = 2211$ (4) Å$^3$
$Z = 4$
$D_x = 1.550$ Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 14 reflections
$\theta = 10.0$–11.5°
$\mu = 1.187$ mm$^{-1}$
$T = 298$ (2) K
Block, colourless
0.50 × 0.40 × 0.20 mm

### *Data collection*

Nicolet *P*3 diffractometer
$\theta$–$2\theta$ scans
Absorption correction: $\psi$ scan (North *et al.*, 1968)
$T_{\min} = 0.621$, $T_{\max} = 0.789$
10 232 measured reflections
10 230 independent reflections
7111 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.055$
$\theta_{\max} = 27.56°$
$h = 0 \rightarrow 11$
$k = -18 \rightarrow 18$
$l = -22 \rightarrow 22$
2 standard reflections every 50 reflections intensity decay: none

### *Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.088$
$S = 1.019$
10 230 reflections
559 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0345P)^2 + 1.0146P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.55$ e Å$^{-3}$
$\Delta\rho_{\min} = -0.58$ e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Sn1—O1*A* | 2.074 (3) | Sn2—O1*B* | 2.077 (3) |
| Sn1—C14*A* | 2.106 (4) | Sn2—C14*B* | 2.115 (4) |
| Sn1—C8*A* | 2.117 (4) | Sn2—C8*B* | 2.123 (4) |
| Sn1—C20*A* | 2.125 (4) | Sn2—C20*B* | 2.128 (4) |
| Sn1—O2*A* | 2.874 (4) | Sn2—O2*B* | 2.778 (4) |
| | | | |
| O1*A*—Sn1—C14*A* | 106.84 (14) | O1*B*—Sn2—C14*B* | 106.41 (14) |
| O1*A*—Sn1—C8*A* | 110.64 (14) | O1*B*—Sn2—C8*B* | 114.31 (15) |
| C14*A*—Sn1—C8*A* | 117.42 (15) | C14*B*—Sn2—C8*B* | 115.88 (16) |
| O1*A*—Sn1—C20*A* | 95.64 (15) | O1*B*—Sn2—C20*B* | 95.45 (15) |
| C14*A*—Sn1—C20*A* | 109.71 (16) | C14*B*—Sn2—C20*B* | 111.12 (16) |
| C8*A*—Sn1—C20*A* | 114.19 (17) | C8*B*—Sn2—C20*B* | 111.76 (16) |
| O1*A*—Sn1—O2*A* | 50.10 (11) | O1*B*—Sn2—O2*B* | 51.78 (12) |
| C14*A*—Sn1—O2*A* | 86.81 (14) | C14*B*—Sn2—O2*B* | 85.70 (14) |
| C8*A*—Sn1—O2*A* | 81.19 (15) | C8*B*—Sn2—O2*B* | 83.20 (15) |
| C20*A*—Sn1—O2*A* | 145.61 (12) | C20*B*—Sn2—O2*B* | 146.96 (12) |

Data collection: *Nicolet P*3 *Software* (Nicolet, 1980); cell refinement: *Nicolet P*3 *Software*; data reduction: *RDNIC* (Howie, 1980); program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); software used to prepare material for publication: *SHELXL*97 (Sheldrick, 1997).

## References

Allen, F. H. & Kennard, O. (1993). *Chem. Des. Autom. News*, **8**, 31–37.

Fletcher, D. A., McMeeking, R. F. & Parkin, D. (1996). *J. Chem. Inf. Comput. Sci.* **36**, 746–749.

Howie, R. A. (1980). *RDNIC*. University of Aberdeen, Scotland.

Nicolet. (1980). *Nicolet P*3/*R*3 *Data Collection Operator's Manual*. Nicolet XRD Corporation, 10061 Bubb Road, Cupertino, CA 95014, USA.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.

Weng, N. S., Das, V. G. K. & Robinson, W. T. (1990). *Malays. J. Sci.* **12**, 57.

**e180** R. Alan Howie *et al.* • [$Sn(C_6H_5)_3(C_7H_4NO_4)$] *Acta Cryst.* (2000). C**56**, e179–e180