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Key indicators

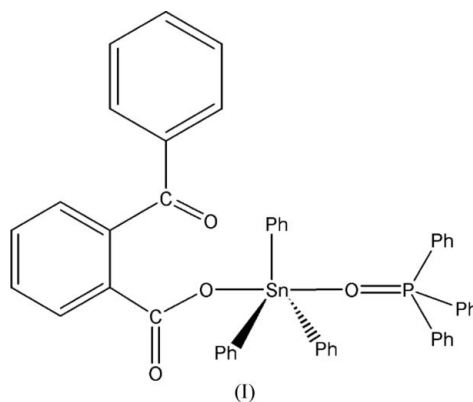
Single-crystal X-ray study
 $T = 173\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.007\text{ \AA}$
 R factor = 0.036
 wR factor = 0.074
Data-to-parameter ratio = 11.7For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.(2-Benzoylbenzoato- κO)triphenyl(triphenylphosphine
oxide- κO)tin(IV)

The structure of the title compound, $[\text{Sn}(\text{C}_6\text{H}_5)_3(\text{C}_{14}\text{H}_9\text{O}_3)(\text{C}_{18}\text{H}_{15}\text{OP})]$, consists of discrete molecules, in which the monodentate $\text{C}_6\text{H}_5\text{COC}_6\text{H}_4\text{COO}^-$ anion and OPPh_3 are coordinated to an SnPh_3 group, and the tin center is in a trigonal-bipyramidal environment. The axial and equatorial positions of the trigonal bipyramid are occupied by the two O atoms of OPPh_3 and RCOO^- and three *ipso* C atoms of the three phenyl rings, respectively.

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Comment

The structure of the title compound, (I), was determined as part of a continuation of our work on the elucidation of the structures of organotin derivatives including carboxylate ligands (Rénamy *et al.*, 2004). The interest in synthesizing new organotin derivatives is related to their biological and pharmaceutical applications (Davies & Smith, 1982) and, from a chemical point of view, to the diversity of structures that such compounds can form. The starting material in the synthesis of (I), $\text{C}_6\text{H}_5\text{COC}_6\text{H}_4\text{CO}_2\text{SnPh}_3$, (II), has already been crystallographically characterized (Khoo *et al.*, 1995); in its structure the ketone oxygens form bridges in a polymeric chain and the carboxylate carbonyl O atom is not involved in coordination. Triphenylphosphine oxide, OPPh_3 , has been extensively used as a ligand in transition metal complexes: e.g. Cambridge Structural Database (*MOGUL* Version 1.8, 2006 release; Allen, 2002) refcodes CLPOMN10 (Tomita, 1987), CLPOZN (Rose *et al.*, 1980) and CLTPOC (Margion *et al.*, 1976); its use has been extended to organotin(IV) chemistry (Nardelli *et al.*, 1976). Compound (I) has resulted from the reaction between 2-benzoylbenzoatotriphenyltin(IV) and triphenylphosphine oxide.



coordinated to an SnPh_3 group (Fig. 1), and the tin center is in a trigonal-bipyramidal environment (Table 1). The equatorial positions of the trigonal bipyramid around the tin center are occupied by the *ipso* C atoms (C1, C7 and C13) of the phenyl rings *A* (C1–C6), *B* (C7–C12) and *C* (C13–C18), and the axial sites by the O atoms, O1 and O2, of OPPh_3 and RCOO^- , respectively. The Sn1–O1 and Sn1–O2 bonds (Table 1) are non-equivalent. The sum of the C–Sn–C angles [359.07°] around the tin center is very close to 360°, while the O1–Sn1–O2 angle deviates slightly from linearity. The O1–P1 bond in OPPh_3 is of the order of those reported by Cunningham *et al.* (2000) in $X_2\text{SnPh}_2\text{OPPh}_3$ molecules [1.501 (2) Å for $X = \text{Cl}$ and 1.504 (4) Å for $X = \text{Br}$], while the Sn1–O1 bond and the P1–O1–Sn1 angle are slightly different from those reported for the former compounds [2.278 (2) Å, 2.287 (4) Å and 148.04 (13)°]. IR and Mössbauer spectroscopy also supported the structure of (I). The high value of $\Delta\nu = \nu_{\text{as}}\text{COO}^- - \nu_{\text{s}}\text{COO}^- = 301 \text{ cm}^{-1}$ is consistent with the presence of monodentate carboxylate groups (Basu Baul *et al.*, 2002), while the Mössbauer quadrupole splitting (3.26 mm s^{-1}) indicates a trigonal-bipyramidal environment around the tin center (Davies & Smith, 1982).

Experimental

$\text{C}_6\text{H}_5\text{COC}_6\text{H}_4\text{CO}_2\text{SnPh}_3$, (II), was obtained from the condensation of 2-benzoylbenzoic acid ($\text{C}_6\text{H}_5\text{COC}_6\text{H}_4\text{CO}_2\text{H}$; 1.54 g, 6.81 mmol) with triphenyltin(IV) hydroxide (Ph_3SnOH ; 2.50 g, 6.81 mmol) in ethanol (50 ml). Compound (I) was obtained by reacting an ethanol solution of (II) (99.99%) with OPPh_3 (0.87 mmol) in a 1:1 ratio. The mixture was stirred for several hours and slow solvent evaporation gave crystals suitable for X-ray analysis.

Crystal data

$[\text{Sn}(\text{C}_6\text{H}_5)_3(\text{C}_{14}\text{H}_9\text{O}_3)(\text{C}_{18}\text{H}_{15}\text{OP})]$	$Z = 4$
$M_r = 853.47$	$D_x = 1.393 \text{ Mg m}^{-3}$
Monoclinic, Cc	Mo $K\alpha$ radiation
$a = 13.5072 (10) \text{ \AA}$	$\mu = 0.71 \text{ mm}^{-1}$
$b = 14.7298 (10) \text{ \AA}$	$T = 173 (2) \text{ K}$
$c = 20.481 (2) \text{ \AA}$	Prism, colorless
$\beta = 93.22 (5)^\circ$	$0.13 \times 0.10 \times 0.08 \text{ mm}$
$V = 4068.4 (6) \text{ \AA}^3$	

Data collection

Nonius KappaCCD diffractometer	5944 independent reflections
ω scans	4977 reflections with $I > 2\sigma(I)$
Absorption correction: none	$R_{\text{int}} = 0.036$
10419 measured reflections	$\theta_{\text{max}} = 30.1^\circ$

Refinement

Refinement on F^2	$(\Delta/\sigma)_{\text{max}} = 0.003$
$R[F^2 > 2\sigma(F^2)] = 0.036$	$\Delta\rho_{\text{max}} = 1.48 \text{ e \AA}^{-3}$
$wR(F^2) = 0.074$	$\Delta\rho_{\text{min}} = -0.81 \text{ e \AA}^{-3}$
$S = 1.04$	Extinction correction: <i>SHELXL97</i>
5944 reflections	Extinction coefficient: 0.00064 (13)
506 parameters	Absolute structure: Flack (1983)
H-atom parameters constrained	with 5949 Friedel pairs
$w = 1/[\sigma^2(F_o^2) + (0.0296P)^2 + 1.3481P]$	Flack parameter: 0.083 (17)
where $P = (F_o^2 + 2F_c^2)/3$	

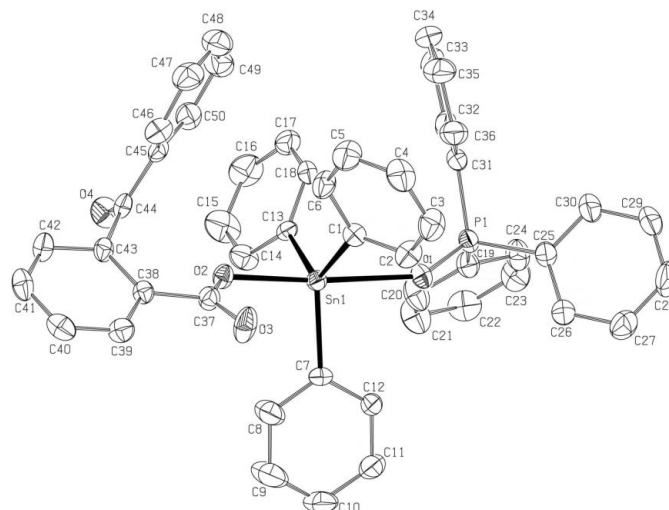


Figure 1

The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted.

Table 1

Selected geometric parameters (Å, °).

Sn1–C1	2.122 (4)	P1–O1	1.511 (3)
Sn1–C7	2.124 (4)	O2–C37	1.288 (5)
Sn1–C13	2.137 (4)	O3–C37	1.229 (5)
Sn1–O2	2.168 (2)	O4–C44	1.213 (5)
Sn1–O1	2.402 (3)		
C1–Sn1–C7	125.57 (16)	O2–Sn1–O1	175.46 (11)
C1–Sn1–C13	120.99 (15)	O1–P1–C25	113.13 (19)
C7–Sn1–C13	112.51 (15)	O1–P1–C19	110.27 (18)
C1–Sn1–O2	95.53 (14)	C25–P1–C19	105.33 (19)
C7–Sn1–O2	97.05 (14)	O1–P1–C31	112.29 (18)
C13–Sn1–O2	86.40 (13)	C25–P1–C31	106.16 (19)
C1–Sn1–O1	82.65 (13)	C19–P1–C31	109.3 (2)
C7–Sn1–O1	87.38 (13)	P1–O1–Sn1	146.43 (18)
C13–Sn1–O1	90.97 (12)	C37–O2–Sn1	119.3 (2)

H atoms were positioned geometrically, with C–H = 0.95 Å, and constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The highest residual electron-density lies 1.97 Å from atom C14.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *DENZO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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