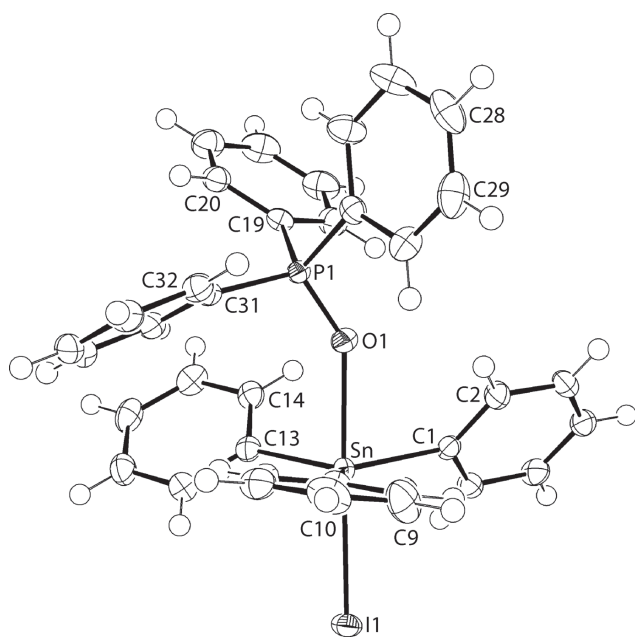


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Crystal structure of iodido-triphenyl-(triphenylphosphine oxide)tin(IV), $C_{36}H_{30}IOPSn$



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Abstract

$C_{36}H_{30}IOPSn$, monoclinic, $P2_1$ (no. 4), $a = 9.7251(6)$ Å, $b = 17.3609(11)$ Å, $c = 10.0542(6)$ Å, $\beta = 114.379(1)^\circ$, $V = 1546.16(17)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0113$, $wR_{ref}(F^2) = 0.0301$, $T = 296(2)$ K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Yellow prism
Size:	0.25 × 0.15 × 0.11 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	1.90 mm ⁻¹
Diffractometer, scan mode:	Bruker SMART APEX, ω
θ_{max} , completeness:	28.3°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	19627, 7642, 0.015
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 7603
$N(param)_{refined}$:	361
Programs:	Bruker [1], SHELX [2, 3], WinGX/ORTEP [4]

Source of material

All chemicals and solvents were used as purchased without purification. The melting point was determined on a Mel-temp II digital melting point apparatus and was uncorrected. The IR spectrum was obtained on a Bruker Vertex 70v FTIR Spectrometer from 4000 to 400 cm⁻¹.

Triphenyltin iodide was prepared from the reaction of triphenyltin chloride (Merck) with sodium iodide (Sigma-Aldrich) [5]. Triphenyltin iodide (0.48 g, 1 mmol) and triphenylphosphine oxide (Sigma-Aldrich; 0.27 g, 1 mmol) in chloroform (30 mL) were refluxed for 2 h. After filtration, the filtrate was evaporated slowly until yellow crystals were formed. The crystals were filtered, washed with a minimum amount of hexane and air-dried. Yield: 0.21 g (28%). M.pt: 370–372 K. IR (cm⁻¹) 1590 (m) $\nu(C-C)$, 1116 (m) $\nu(P-O)$, 1070 (m) $\nu(C-O)$, 446 (w) $\nu(Sn-O)$.

Experimental details

The C-bound H atoms were geometrically placed ($C-H = 0.95$ Å) and refined as riding with $U_{iso}(H) = 1.2U_{eq}(C)$. Owing to poor agreement, one reflection, i.e. (3 3 1), was omitted from the final cycles of refinement. The absolute structure was determined based on differences in the Friedel pairs included in the data set.

Comment

The crystal and molecular structures of the title compound, $Ph_3SnI \cdot O=PPh_3$, (I), were determined as part of an on-going investigation of oxide adducts of organotin species, such as phosphineoxide [6], arsineoxide [7] and sulphoxide [8, 9].

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
I1	−0.18870(2)	0.38832(2)	0.05116(2)	0.01789(4)
Sn	0.06869(2)	0.46646(2)	0.26278(2)	0.01190(3)
P1	0.41973(6)	0.52683(3)	0.57287(5)	0.01174(9)
O1	0.28159(17)	0.53146(8)	0.43105(15)	0.0159(3)
C1	−0.0002(2)	0.57881(11)	0.1729(2)	0.0134(4)
C2	0.1057(2)	0.63274(12)	0.1717(2)	0.0172(4)
H2	0.207877	0.620464	0.213908	0.021*
C3	0.0596(3)	0.70497(12)	0.1077(2)	0.0189(4)
H3	0.130991	0.739977	0.105447	0.023*
C4	−0.0915(3)	0.72471(12)	0.0477(2)	0.0184(4)
H4	−0.121905	0.772898	0.005251	0.022*
C5	−0.1982(2)	0.67209(13)	0.0510(2)	0.0188(4)
H5	−0.299745	0.685555	0.013131	0.023*
C6	−0.1525(2)	0.59922(12)	0.1113(2)	0.0157(4)
H6	−0.224476	0.563736	0.110407	0.019*
C7	0.2293(2)	0.40758(11)	0.2079(2)	0.0137(4)
C8	0.2589(3)	0.43316(12)	0.0904(2)	0.0201(4)
H8	0.205208	0.474620	0.034470	0.024*
C9	0.3677(3)	0.39717(14)	0.0569(3)	0.0263(5)
H9	0.386785	0.414805	−0.021231	0.032*
C10	0.4484(3)	0.33520(13)	0.1386(3)	0.0239(5)
H10	0.522026	0.311647	0.116083	0.029*
C11	0.4189(2)	0.30838(13)	0.2541(2)	0.0207(4)
H11	0.472112	0.266432	0.308709	0.025*
C12	0.3097(2)	0.34435(12)	0.2881(2)	0.0169(4)
H12	0.289962	0.325986	0.365368	0.020*
C13	0.0288(2)	0.43000(12)	0.4461(2)	0.0145(4)
C14	0.0249(2)	0.48489(12)	0.5459(2)	0.0171(4)
H14	0.035243	0.536809	0.529250	0.021*
C15	0.0055(2)	0.46269(15)	0.6704(2)	0.0210(4)
H15	0.001474	0.499811	0.735392	0.025*
C16	−0.0077(2)	0.38541(15)	0.6973(2)	0.0223(4)
H16	−0.018538	0.370565	0.781366	0.027*
C17	−0.0049(3)	0.32990(13)	0.5983(2)	0.0216(4)
H17	−0.014614	0.278016	0.615726	0.026*
C18	0.0126(3)	0.35237(12)	0.4730(2)	0.0191(4)
H18	0.013509	0.315252	0.406657	0.023*
C19	0.3874(2)	0.57723(11)	0.7135(2)	0.0139(4)
C20	0.4537(2)	0.55538(12)	0.8599(2)	0.0172(4)
H20	0.518933	0.513437	0.888888	0.021*
C21	0.4219(3)	0.59649(14)	0.9625(2)	0.0232(5)
H21	0.463779	0.581182	1.059846	0.028*
C22	0.3271(3)	0.66081(14)	0.9197(3)	0.0258(5)
H22	0.307009	0.688628	0.988693	0.031*
C23	0.2636(3)	0.68299(13)	0.7753(3)	0.0250(5)
H23	0.200758	0.725860	0.747214	0.030*
C24	0.2928(3)	0.64181(12)	0.6717(3)	0.0196(4)
H24	0.249452	0.657063	0.574326	0.024*
C25	0.5760(2)	0.57320(11)	0.5535(2)	0.0148(4)
C26	0.6857(3)	0.61284(13)	0.6683(3)	0.0226(5)
H26	0.681089	0.614469	0.758768	0.027*
C27	0.8024(3)	0.65006(14)	0.6487(3)	0.0278(5)
H27	0.875730	0.676635	0.725757	0.033*
C28	0.8090(3)	0.64737(13)	0.5136(3)	0.0265(5)
H28	0.886108	0.672876	0.499563	0.032*

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C29	0.7012(3)	0.60682(15)	0.3994(3)	0.0268(5)
H29	0.707261	0.604502	0.309591	0.032*
C30	0.5847(3)	0.56979(13)	0.4182(2)	0.0210(4)
H30	0.512351	0.542733	0.341236	0.025*
C31	0.4753(2)	0.42993(11)	0.6328(2)	0.0146(4)
C32	0.5937(2)	0.39505(13)	0.6113(2)	0.0172(4)
H32	0.653135	0.423939	0.577333	0.021*
C33	0.6229(3)	0.31698(13)	0.6407(2)	0.0196(4)
H33	0.700655	0.293458	0.624792	0.023*
C34	0.5357(3)	0.27412(13)	0.6940(2)	0.0209(4)
H34	0.555849	0.221999	0.713991	0.025*
C35	0.4189(2)	0.30857(13)	0.7176(2)	0.0198(4)
H35	0.361168	0.279690	0.753584	0.024*
C36	0.3885(2)	0.38644(13)	0.6871(2)	0.0163(4)
H36	0.310302	0.409697	0.702874	0.020*

A search of the crystallographic literature [10] reveals there are no reports of simple R₃SnI·O=PR₃ adducts.

The molecular structure of (I) is shown in the figure (70% displacement ellipsoids). The tin atom exists within a C₃IO donor set defined by the three ipso-carbon atoms of the phenyl substituents [range of Sn–C bond lengths: Sn–C7 = 2.122(2) Å to Sn–C1 2.138(2) Å], an iodide [Sn–I1 = 2.8699(2) Å] and the phosphineoxide-O1 atom [2.3517(15) Å]. The resultant penta-coordinated geometry is based on a trigonal bipyramid with electronegative substituents occupying axial positions; the I1–Sn–O1 axial angle is 178.43(4)°. The tin-bound organo substituents occupy equatorial positions with the range of C–Sn–C angles being 117.73(8)° for C1–Sn–C7, to 119.98(8)° for C1–Sn–C13. The Sn atom lies 0.1910(12) Å out of the C₃ equatorial plane in the direction of the I1 atom. The P1–O1 bond length is 1.5044(15) Å and the Sn–O1–P1 angle is 146.39(9)°. When the molecule is viewed down the spine, the configuration of the two sets of phenyl rings is staggered, allowing for the bent Sn–O–P angle.

The molecular packing of (I) is largely devoid of directional interactions. The only notable interaction is an end-on Sn–I···π(phenyl) interaction [Sn–I1···Cg(C31–C36)]ⁱ: I1···Cg(C31–C36)]ⁱ = 3.8518(8) Å with angle at I1 = 154.14(2)° for symmetry operation (i) −1 + *x*, *y*, −1 + *z*. The mode of association leads to the formation of a linear supramolecular chain along [1 0 1].

A further evaluation of the molecular packing was made by calculating the Hirshfeld surfaces and full and delineated two-dimensional fingerprint plots for (I) using Crystal Explorer 17 [11] and literature protocols [12]. The two most dominant surface contacts, comprising over 90% of all contacts, are H···H [64.4%] and C···H/H···C [23.1%].

The contacts involving iodide are I···H/H···I [6.9%] and I···C/C···I [2.0%], with the only remaining contacts being of the type C···C [0.7%].

The crystal structures of the chloride (II) [13] and bromide (III) [14] analogues of (I) are known. Crystals of the reported (I)–(III) are not isostructural. In (II), C–H···π(phenyl) and C–H···Cl contacts are noted. However, in (III), no directional interactions are apparent.

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