C(16)	0.5107 (8)	0.1798 (9)	0.2126 (4)	0.067 (3)
C(17)	0.3561 (6)	0.3190 (7)	−0.0071 (3)	0.049 (2)
C(18)	0.4080 (8)	0.3676 (8)	−0.0646 (4)	0.067 (3)
C(19)	0.3531 (9)	0.4893 (10)	−0.1056 (4)	0.080 (3)
C(110)	0.2478 (9)	0.5659 (10)	−0.0903 (5)	0.083 (3)
C(111)	0.1950 (8)	0.5235 (9)	−0.0316 (5)	0.081 (3)
C(112)	0.2494 (8)	0.3986 (8)	0.0099 (4)	0.066 (3)

Table 2. *Selected geometric parameters* (Å, °)

Sn(1)—O(11)	2.162 (5)	Sn(1)—O(1)	2.156 (5)
Sn(1)—C(11)	2.107 (6)	Sn(1)—C(17)	2.113 (6)
Sn(1)—O(11^{i})	2.024 (4)	Cl(1)—C(2)	1.720 (8)
Cl(2)—C(2)	1.742 (8)	Cl(3)—C(2)	1.714 (7)
O(1)—C(1)	1.250 (8)	O(2)—C(1)	1.222 (9)
C(1)—C(2)	1.556 (10)		
O(11)—Sn(1)—O(1)	158.3 (2)	O(11)—Sn(1)—C(11)	96.2 (2)
O(1)—Sn(1)—C(11)	92.0 (2)	O(11)—Sn(1)—C(17)	96.4 (2)
O(1)—Sn(1)—C(17)	92.2 (2)	C(11)—Sn(1)—C(17)	133.9 (2)
O(11)—Sn(1)—O(11^{i})	70.6 (2)	O(1)—Sn(1)—O(11^{i})	87.7 (2)
C(11)—Sn(1)—O(11^{i})	113.8 (2)	C(17)—Sn(1)—O(11^{i})	112.1 (2)
Sn(1)—O(11)—Sn(1^{i})	109.4 (2)	Sn(1)—O(1)—C(1)	128.9 (5)
O(1)—C(1)—O(2)	128.1 (6)	O(1)—C(1)—C(2)	113.7 (6)
O(2)—C(1)—C(2)	118.2 (6)	Cl(1)—C(2)—Cl(2)	107.4 (4)
Cl(1)—C(2)—Cl(3)	112.6 (5)	Cl(2)—C(2)—Cl(3)	107.8 (4)
Cl(1)—C(2)—C(1)	107.6 (5)	Cl(2)—C(2)—C(1)	112.3 (5)
Cl(3)—C(2)—C(1)	109.2 (4)		

Symmetry code: (i) $1 - x, -y, -z$.

Dimeric μ-hydroxy-(trichloroacetato)diphenyltin, $[SnPh_2(O_2CCCl_3)OH]_2$, was prepared from $Ph_3SnO_2CCCl_3.MeOH$ (Alcock & Roe, 1989) by recrystallization from CCl_4/hexane by the liquid diffusion method. Crystals formed at the interface after 12 h and were used directly for structure determination. The formation of this dimer, rather than the tetrameric bis-μ-oxo complex, $\{[SnPh_2(O_2CCCl_3)]_2\}_2$ [Alcock & Roe, 1989, compound (2)], is apparently favoured by the presence of traces of water in the (nominally dry) solvents interacting with the relatively strong acid.

Scan speed was 2-15°(ω) min^{-1}, depending on the intensity of a 2 s prescan; backgrounds were measured at each end of the scan for 0.25 of the scan time. The data were rescaled to correct for the slight crystal decay. Reflections were processed using profile analysis. Systematic reflection conditions $h0l$, $l = 2n$ and $0k0$, $k = 2n$, indicated space group $P2_1/c$. Heavy atoms were located by the Patterson interpretation section of *SHELXTL-Plus* (Sheldrick, 1986) and the light atoms were then found by successive Fourier syntheses. Anisotropic displacement parameters were used for all non-H atoms. The hydroxy H atom was refined, with an O—H constraint of 0.85 (1) Å. Other H atoms were inserted at calculated positions and not refined; all were given fixed isotropic temperature factors, U = 0.07 Å^2. Computations were performed with *SHELXTL-Plus* on a DEC MicroVAX II.

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71518 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU1059]

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Tris(2-methoxyphenyl)tin Iodide

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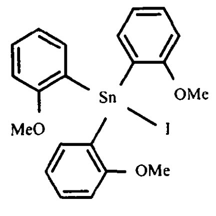
Abstract

The Sn atom in the title compound, $[SnI(C_7H_7O)_3]$, has a distorted tetrahedral geometry. There are indications of only weak oxygen–tin intramolecular interactions.

Comment

Crystal structures have been reported for a number of intramolecular complexes of triorganotin halides possessing suitably-sited donor groups. These groups include alcohol and ether groups, *e.g.* as in $ClPh_2SnCH_2CH_2CH_2OEt$ (Forrester, Howie, Ross, Low & Wardell, 1991), $IPh_2SnCH_2CH_2CH_2OH$ (Forrester, Garden, Howie & Wardell, 1992), *cis*-1-$ClMe_2Sn$-3-$PhCH_2O$(cyclohexane) (Ochiai *et al.*, 1988) and 3-*C*-(IBu_2SnCH_2)-1,2;5,6-di-*O*-isopropylidene-α-D-allofuranose (1) (Cox *et al.*, 1989). The most frequently reported chelate-ring sizes are generally five- and six-membered but four-membered rings, as in (1), can also be obtained.

In contrast to the compounds listed above, the geometry about Sn in $(2\text{-}MeOC_6H_4)_3SnI$ (2) is essentially tetrahedral with I—Sn—C angles ranging from



(2)

101.7 (2) to 110.3 (2)° [Σ(I—Sn—C) = 317.8 (6)°] and C—Sn—C angles ranging from 111.8 (3) to 113.6 (3)° [Σ(C—Sn—C) = 338.2 (9)°]; for a regular tetrahedral arrangement Σ(I—Sn—C) and Σ(C—Sn—C) should both be 328.5°. The Sn—I bond length is 2.713 (1) Å. This is similar to values reported for other tetrahedral triorganotin iodides, *e.g.* for $(Me_3SiCH_2)_3SnI$ [2.712 (6) Å (Zakharov, Petrov, Lebedev, Kuz'min & Belov, 1978)] and for $IPh_2Sn(CH_2)_4SnPh_2I$ [2.729 (3) Å (Cody & Corey, 1969)], but somewhat longer than the value reported for (*cyclo*-$C_6H_{11})_3SnI$ [2.54 (1) Å (Calogero, Ganis, Peruzzo, Tagliavini & Valle, 1981)]. Sn—I bond lengths in recognized five-coordinate trigonal bipyramidal triorganotin iodide complexes are greater, *e.g.* 2.764 (2) Å in (1) (Cox *et al.*, 1989), 2.811 (2) Å in $IPh_2SnCH_2CH_2CO_2Me$ (Harston *et al.*, 1991), 2.830 (7) Å in 1-MeO_2C-2-IMe_2Sn-1,4-cyclohexadiene (Kolb, Dräger & Jousseaume, 1991), 2.853 (3) and 2.946 (3) Å in $[1\text{-}IPh_2Sn\text{-}1,2,4\text{-triazole}]_4$ (Doidge-Harrison, 1992) and 2.857 (1) Å in $IPh_2SnCH_2CH_2CH_2OH$ (Forrester *et al.*, 1992).

In these and other trigonal bipyramidal R_3SnX complexes, the non-C donor atoms are in axial positions. The I—Sn···O angles and Sn···O separations in $(2\text{-}MeOC_6H_4)_3SnI$ (2) are 152.1 (1)° and 2.965 (7) Å [O(1)], 83.5 (1)° and 3.061 (7) Å [O(2)] and 80.6 (1)° and 3.045 (6) Å [O(3)]. The values, especially those associated with O(1), give indications of weak Sn···O intramolecular interactions. There is a significant difference in the values of the Sn—C11—C16 and Sn—C11—C12 valency angles, the smaller value for the latter allowing O(1) to approach closer to the Sn atom. The exocyclic valency angles involving Sn and each of the other two aryl rings are essentially the same.

Each pair of exocyclic valency angles involving the O atoms O(1), O(2) or O(3), and phenyl ring C atoms shows considerable differences, *e.g.* 113.4 (7) and 124.5 (8)° for O(1). This appears to be a general feature of methoxyphenyl compounds [*e.g.* values are 115.7 (8) and 124.5 (8)° in $Ph_3SnCH_2SeC_6H_4OMe$-3 (Cox *et al.*, 1990)]. The exocyclic valency angle differences found in $(2\text{-}MeOC_6H_4)_3SnI$ are clearly not indicative of O→Sn coordination but probably arise to reduce steric repulsions between the OCH_3 group and the adjacent phenyl H atom.

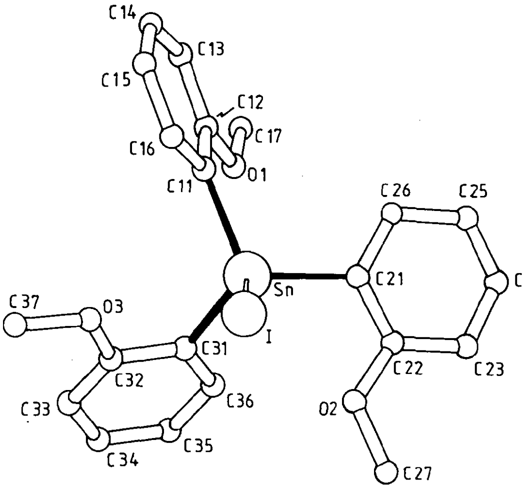


Fig. 1. View of the title structure showing the labelling of the non-H atoms.

Experimental

Crystal data

$[SnI(C_7H_7O)_3]$
$M_r = 566.99$
Monoclinic
$P2_1/n$
$a = 8.680\ (5)$ Å
$b = 28.352\ (20)$ Å
$c = 9.022\ (4)$ Å
$\beta = 99.81\ (4)°$
$V = 2188\ (2)$ Å^3
$Z = 4$
$D_x = 1.721$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 14 reflections
$\theta = 10\text{-}11.5°$
$\mu = 2.58$ mm^{-1}
$T = 293$ K
Prism
0.70 × 0.44 × 0.30 mm
Colourless

Data collection

Nicolet *P*3 diffractometer
ω scans
Absorption correction: none
6742 measured reflections
6375 independent reflections
4724 observed reflections [$I > 2.5\sigma(I)$]
$R_{\text{int}} = 0.064$

$\theta_{\text{max}} = 30.0°$
$h = -12 \rightarrow 12$
$k = 0 \rightarrow 39$
$l = 0 \rightarrow 12$
2 standard reflections monitored every 50 reflections intensity variation: ≃2%

Refinement

Refinement on *F*
$R = 0.064$
$wR = 0.058$
4724 reflections
115 parameters
H atoms riding on associated atoms
Unit weights applied

$(\Delta/\sigma)_{\text{max}} = 0.032$
$\Delta\rho_{\text{max}} = 1.22$ e Å^{-3}
$\Delta\rho_{\text{min}} = -1.20$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters* (Å^2)

$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{iso}/B_{eq}†
I	0.99379 (7)	0.86848 (2)	0.04163 (7)	4.76 (3)†
Sn	1.28070 (6)	0.866838 (16)	−0.03950 (5)	3.103 (18)†
C11	1.2325 (8)	0.8953 (3)	−0.2603 (8)	3.29 (12)
C12	1.3543 (10)	0.8940 (3)	−0.3436 (9)	4.03 (14)
C13	1.3363 (11)	0.9106 (3)	−0.4900 (11)	4.87 (18)
C14	1.1945 (12)	0.9300 (4)	−0.5521 (12)	5.3 (2)
C15	1.0734 (11)	0.9341 (3)	−0.4714 (11)	4.98 (18)
C16	1.0933 (10)	0.9159 (3)	−0.3244 (10)	4.13 (15)
O1	1.4905 (8)	0.8756 (2)	−0.2654 (8)	5.56 (14)
C17	1.6229 (15)	0.8708 (5)	−0.3404 (14)	7.1 (3)
C21	1.3477 (10)	0.7939 (3)	−0.0366 (9)	4.07 (15)
C22	1.3760 (11)	0.7708 (3)	0.0985 (11)	4.65 (17)
C23	1.4250 (14)	0.7228 (4)	0.1051 (14)	6.4 (2)
C24	1.4406 (17)	0.7027 (5)	−0.0270 (18)	8.7 (4)
C25	1.4085 (18)	0.7229 (6)	−0.1589 (18)	8.8 (4)
C26	1.3603 (14)	0.7716 (4)	−0.1689 (14)	6.5 (3)
O2	1.3537 (8)	0.7957 (2)	0.2180 (8)	5.37 (14)
C27	1.3786 (17)	0.7744 (5)	0.3632 (17)	8.5 (3)
C31	1.4403 (8)	0.9075 (2)	0.1119 (8)	3.04 (11)
C32	1.4084 (9)	0.9552 (3)	0.1326 (9)	3.68 (13)
C33	1.5150 (11)	0.9830 (3)	0.2253 (11)	4.79 (17)
C34	1.6521 (12)	0.9635 (4)	0.2987 (12)	5.3 (2)
C35	1.6859 (11)	0.9168 (3)	0.2797 (11)	4.72 (17)
C36	1.5786 (9)	0.8887 (3)	0.1860 (8)	3.46 (13)
O3	1.2697 (7)	0.9699 (2)	0.0535 (7)	4.63 (12)
C37	1.2268 (13)	1.0184 (4)	0.0664 (12)	5.7 (2)

Table 2. *Selected geometric parameters* (Å, °)

I—Sn	2.713 (1)	O1—C17	1.436 (14)
Sn—C11	2.124 (7)	C22—O2	1.329 (12)
Sn—C21	2.147 (9)	O2—C27	1.425 (16)
Sn—C31	2.115 (7)	C32—O3	1.356 (10)
C12—O1	1.373 (11)	O3—C37	1.435 (12)
I—Sn—C11	101.7 (2)	Sn—C21—C22	118.6 (6)
I—Sn—C21	105.8 (2)	Sn—C21—C26	119.7 (7)
I—Sn—C31	110.3 (2)	C21—C22—O2	116.1 (8)
C11—Sn—C21	112.8 (3)	C23—C22—O2	123.8 (9)
C11—Sn—C31	113.6 (3)	C22—O2—C27	120.1 (9)
C21—Sn—C31	111.8 (3)	Sn—C31—C32	119.4 (5)
Sn—C11—C12	116.6 (5)	Sn—C31—C36	121.5 (5)
Sn—C11—C16	125.0 (6)	C31—C32—O3	113.8 (7)
C11—C12—O1	113.4 (7)	C33—C32—O3	125.9 (8)
C13—C12—O1	124.5 (8)	C32—O3—C37	118.0 (7)
C12—O1—C17	118.8 (8)		

Compound (2), m.p. 428-430 K, was prepared from (2-$MeOC_6H_5)_4Sn$ and I_2 in $CHCl_3$ and recrystallized from ethyl acetate/petroleum ether (b.p. 333-353 K) mixture.

Data collection and cell refinement: Nicolet *P*3 software. Data collection used ω scan rates of 1.0 ($I_p < 150$) to 29.3° ($I_p > 2500$) min^{-1} with a constant scan width of 0.6° and background counts taken at ±1° in ω, where I_p is the prescan intensity. Data reduction: local program (Aberdeen). Structure solution, structure refinement and preparation of material for publication: *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989). Isotropic displacement parameters were used for all but the Sn and I atoms.

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Lists of structure factors, anisotropic displacement parameters for Sn and I, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71539 (31 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU1066]

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$[(C_5H_5N)_2H]_3[PW_{12}O_{40}]$

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Abstract

The crystal structure of tris(dipyridinium) dodecatungstophosphate has been determined. The $[PW_{12}O_{40}]^{3-}$ heteropolyanion, having T_d symmetry, is disordered by the $\bar{3}$ symmetry. The pyridine molecules are paired around H^+ forming $(C_5H_5N)—H^+—(NC_5H_5)$ cations with their molecular planes almost parallel to the *ab* plane of the crystal.

Comment

The authors have reported previously that the anhydrous solid 12-tungsto- and molybdophosphates absorb polar organic solvents such as alcohols, ethers and amines,