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## Key indicators

Single-crystal X-ray study  
T = 193 K  
Mean  $\sigma(\text{C}-\text{C}) = 0.007 \text{ \AA}$   
R factor = 0.042  
wR factor = 0.091  
Data-to-parameter ratio = 28.7For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.Hexameric isopropylloxotin acetate chloroform  
tetrasolvate

The molecular structure of the centrosymmetric hexanuclear title compound,  $[\text{PrSn}(\text{O})(\text{O}_2\text{CCH}_3)]_6 \cdot 4\text{CHCl}_3$  or  $[\text{Sn}_6\text{O}_6(\text{C}_3\text{H}_7)_6(\text{C}_2\text{H}_3\text{O}_2)_6] \cdot 4\text{CHCl}_3$ , adopts the 'drum' motif, in which two  $(\text{PrSnO})_3$  caps are linked *via* six  $\mu_3$ -oxo atoms and six acetate groups. The Sn atoms are in distorted octahedral environments, defined by a  $\text{CO}_3$  donor set for each of the three independent Sn atoms.

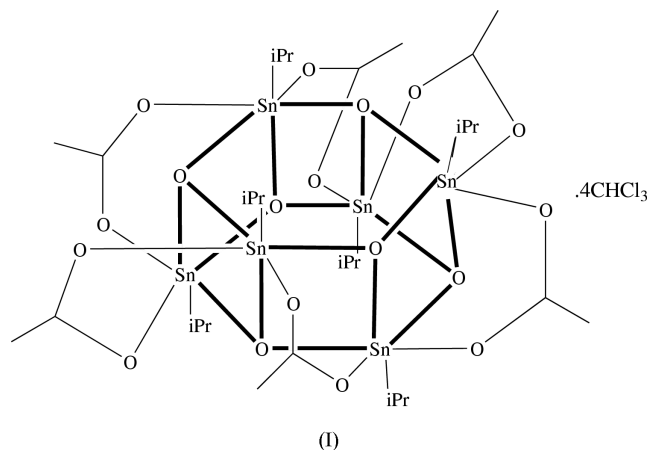
Received 10 May 2002

Accepted 17 May 2002

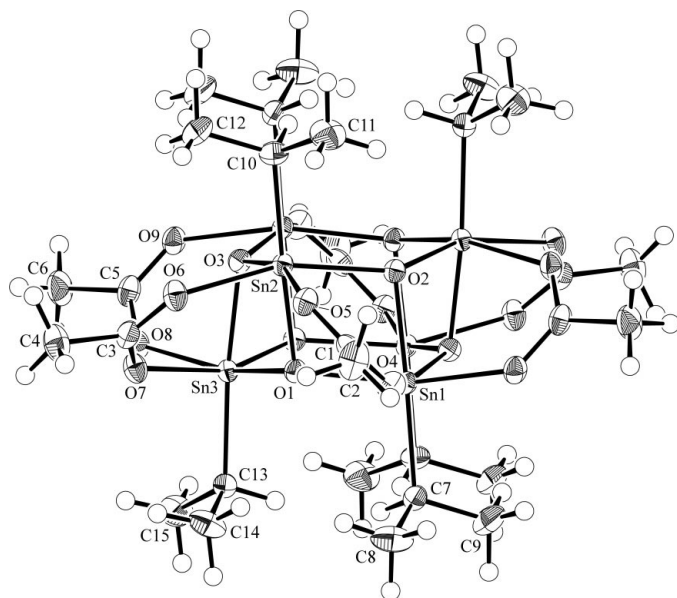
Online 24 May 2002

## Comment

The hydrolysis of organotin species is an area that continues to attract significant attention, owing to the many and varied applications of organotins (Davies, 1997). Monoorganotin species are particularly susceptible to hydrolysis (Holmes, 1989, 1999) and studies in this field have led to a fascinating array of structural motifs such as molecular footballs (Dakternieks *et al.*, 1994) and their deflated analogues (Zobel *et al.*, 2000). The title compound, (I), was isolated and characterized during on-going studies in this area and was shown crystallographically to be  $[\text{PrSn}(\text{O})(\text{O}_2\text{CCH}_3)]_6 \cdot 4\text{CHCl}_3$  (Fig. 1 and Table 1).



A drum motif is found for (I), as has been found previously for several other derivatives [see reviews, Tiekink (1991, 1994)]. The centrosymmetric structure may be described as being comprised of two  $(\text{PrSnO})_3$  rings that are linked in two ways (see below). The deviations from planarity in the six-membered rings are relatively minor, so that the respective deviations of atoms Sn1, Sn2<sup>i</sup>, Sn3, O1, O2<sup>i</sup> and O3<sup>i</sup> from their least-squares planes are 0.129 (1), 0.120 (1), 0.127 (1), -0.130 (3), -0.123 (3) and -0.124 (3) Å [symmetry code: (i)  $-x, -y, 1-z$ ]. Each of the bridging-oxo atoms of each ring connects an Sn atom of the adjacent ring, leading to the



**Figure 1**  
The molecular structure and crystallographic numbering scheme for (I). Displacement ellipsoids are shown at the 50% probability level (Johnson, 1976).

presence of six  $\mu_3$ -oxo groups. The girth of the drum may be thought of as six  $\text{Sn}_2\text{O}_2$  rectangles, arranged so as to form a tube. Further links between the rings are afforded by six bidentate bridging acetate bridges, each of which spans, diagonally, an  $\text{Sn}_2\text{O}_2$  rectangle. The isopropyl groups are directed above and below the  $(\text{PrSnO})_3$  rings. Each Sn atom exists in a distorted octahedral geometry, defined by three O atoms derived from three  $\mu_3$ -O atoms, two carboxylate O atoms and a C atom of the isopropyl substituent.

The most significant intermolecular contacts are of the type C—H...O and involve the chloroform molecules of solvation. The closest such contact, 2.34 Å, occurs between C17—H17 and O8, so that C17...O8 is 3.278 (6) Å and the angle subtended at H17 is 156°. A similar contact is found for the second chloroform molecule, so that C16—H...O9<sup>ii</sup> is 2.54 Å, C16...O9<sup>ii</sup> is 3.421 (6) Å and the angle at H16 is 146° [symmetry code: (ii) 1—x, -y, 1—z].

## Experimental

A saturated aqueous solution of ammonium acetate (30 ml) was added to a suspension of  ${}^i\text{Pr}_2\text{SnO}$  which contained 5%  ${}^i\text{PrSn}(\text{O})(\text{OH})$  (0.56 g, 2.5 mmol) in 1,4-dioxane (30 ml), and the mixture was stirred at reflux for 20 h. The 1,4-dioxane was removed *in vacuo* and chloroform (30 ml) added. The residue was extracted with chloroform (3 × 15 ml), the combined extracts were dried over  $\text{Na}_2\text{SO}_4$  and the solvent removed *in vacuo* to yield  ${}^i\text{Pr}_2\text{Sn}(\text{O}_2\text{CCH}_3)_2$  (0.72 g, 89%). Colourless crystals of the title compound were obtained by slow evaporation of a chloroform/hexane solution (1:3). m.p. > 583 K (decomposition).

## Crystal data

$[\text{Sn}_6\text{O}_6(\text{C}_3\text{H}_7)_6(\text{C}_2\text{H}_3\text{O}_2)_6]\cdot 4\text{CHCl}_3$   
 $M_r = 1898.39$   
 Monoclinic,  $P2_1/c$   
 $a = 13.2736$  (14) Å  
 $b = 13.5405$  (16) Å  
 $c = 17.3572$  (19) Å  
 $\beta = 92.007$  (3)°  
 $V = 3117.7$  (6) Å<sup>3</sup>  
 $Z = 2$

$D_x = 2.022$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 Cell parameters from 25440 reflections  
 $\theta = 1.5$ – $30.1$ °  
 $\mu = 2.94$  mm<sup>-1</sup>  
 $T = 193$  (2) K  
 Block, colourless  
 0.20 × 0.10 × 0.05 mm

## Data collection

Bruker AXS SMART CCD diffractometer  
 $\omega$  scans  
 Absorption correction: multi-scan (SADABS; Bruker, 2000)  
 $T_{\min} = 0.683$ ,  $T_{\max} = 0.863$   
 25440 measured reflections

9084 independent reflections  
 6796 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.051$   
 $\theta_{\max} = 30.1$ °  
 $h = -17 \rightarrow 18$   
 $k = -15 \rightarrow 19$   
 $l = -24 \rightarrow 20$

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.042$   
 $wR(F^2) = 0.091$   
 $S = 1.01$   
 9084 reflections  
 316 parameters

H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0368P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 1.33$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.71$  e Å<sup>-3</sup>

**Table 1**

Selected geometric parameters (Å, °).

Sn1—O1	2.081 (3)	Sn2—O5	2.171 (3)
Sn1—O2	2.087 (3)	Sn2—O6	2.166 (3)
Sn1—O3 <sup>i</sup>	2.096 (3)	Sn2—C10	2.148 (4)
Sn1—O4	2.172 (3)	Sn3—O1	2.096 (3)
Sn1—O9 <sup>i</sup>	2.180 (3)	Sn3—O2 <sup>i</sup>	2.086 (3)
Sn1—C7	2.147 (5)	Sn3—O3	2.083 (3)
Sn2—O1	2.073 (3)	Sn3—O7	2.166 (3)
Sn2—O2	2.103 (3)	Sn3—O8	2.177 (3)
Sn2—O3	2.087 (3)	Sn3—C13	2.142 (4)
O1—Sn1—O2	78.08 (11)	O1—Sn3—O7	88.63 (11)
O1—Sn1—O3 <sup>i</sup>	105.45 (11)	O1—Sn3—O8	160.61 (11)
O1—Sn1—O4	87.76 (11)	O1—Sn3—C13	96.74 (14)
O1—Sn1—O9 <sup>i</sup>	158.91 (11)	O2 <sup>i</sup> —Sn3—O3	78.48 (11)
O1—Sn1—C7	99.73 (14)	O2 <sup>i</sup> —Sn3—O7	157.70 (11)
O2—Sn1—O3 <sup>i</sup>	78.19 (11)	O2 <sup>i</sup> —Sn3—O8	85.68 (11)
O2—Sn1—O4	87.63 (11)	O2 <sup>i</sup> —Sn3—C13	101.11 (14)
O2—Sn1—C7	176.09 (14)	O3—Sn3—O7	87.17 (12)
O2—Sn1—O9 <sup>i</sup>	88.13 (11)	O3—Sn3—O8	88.45 (11)
O3 <sup>i</sup> —Sn1—O4	157.93 (12)	O3—Sn3—C13	174.24 (14)
O3 <sup>i</sup> —Sn1—O9 <sup>i</sup>	86.93 (11)	O7—Sn3—O8	76.88 (11)
O3 <sup>i</sup> —Sn1—C7	99.41 (15)	O7—Sn3—C13	94.85 (15)
O4—Sn1—O9 <sup>i</sup>	75.68 (12)	O8—Sn3—C13	97.25 (14)
O4—Sn1—C7	95.55 (15)	Sn1—O1—Sn2	100.79 (12)
O9 <sup>i</sup> —Sn1—C7	94.85 (15)	Sn1—O1—Sn3	131.30 (14)
O1—Sn2—O2	77.87 (11)	Sn2—O1—Sn3	100.05 (12)
O1—Sn2—O3	78.33 (11)	Sn1—O2—Sn2	99.63 (12)
O1—Sn2—O5	87.59 (11)	Sn1—O2—Sn3 <sup>i</sup>	99.98 (11)
O1—Sn2—O6	86.84 (12)	Sn2—O2—Sn3 <sup>i</sup>	133.42 (13)
O1—Sn2—C10	177.03 (15)	Sn1 <sup>i</sup> —O3—Sn2	132.72 (15)
O2—Sn2—O3	103.38 (11)	Sn1 <sup>i</sup> —O3—Sn3	99.77 (12)
O2—Sn2—O5	86.21 (11)	Sn2—O3—Sn3	100.02 (12)
O2—Sn2—O6	159.38 (12)	Sn1—O4—C1	128.9 (3)
O2—Sn2—C10	99.76 (15)	Sn2—O5—C1	130.7 (3)
O3—Sn2—O5	160.70 (12)	Sn2—O6—C3	131.2 (3)
O3—Sn2—O6	86.71 (11)	Sn3—O7—C3	129.0 (3)
O3—Sn2—C10	100.62 (14)	Sn3—O8—C5	130.4 (3)
O5—Sn2—O6	79.33 (12)	Sn1 <sup>i</sup> —O9—C5	129.5 (3)
O5—Sn2—C10	94.06 (15)	O4—C1—O5	125.8 (4)
O6—Sn2—C10	95.89 (15)	O6—C3—O7	125.3 (4)
O1—Sn3—O3	77.90 (11)	O8—C5—O9	125.2 (4)
O1—Sn3—O2 <sup>i</sup>	104.68 (11)		

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

H atoms were placed in geometrically calculated positions and were included in the final refinement in the riding-model approximation. The largest residual electron-density peak is located in the vicinity of atom Sn1.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SHELXTL* (Bruker, 2000); program(s) used to solve structure: *PATY* in *DIRDIF92* (Beurskens *et al.*, 1992); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2000).

The Australian Research Council is thanked for support.

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