

## Diiodobis(4-methylpentan-2-onato- $C^4,O$ )tin(IV): a comparison with diiodobis(3-methoxy-3-oxopropyl- $C,O$ )tin(IV)

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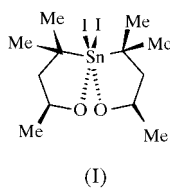
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The title compound,  $[\text{SnI}_2(\text{C}_6\text{H}_{11}\text{O})_2]$ , contains a six-coordinate tin centre as a consequence of intramolecular Sn—O interactions. The Sn—O bond lengths range between 2.428 (4) and 2.439 (4) Å.

### Comment

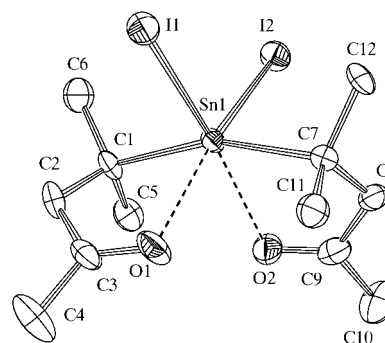
3-(Halostannyl)propanoic esters, so called estertin compounds, such as  $X_2\text{Sn}(\text{CH}_2\text{CH}_2\text{CO}_2R)_2$  and  $X_3\text{SnCH}_2\text{CH}_2\text{CO}_2R$  ( $X$  = halogen,  $R$  = alkyl or aryl), have been known for almost 25 years (Hutton & Oakes, 1976). The initial interest in these compounds as precursors of PVC stabilizers has long since waned, but interest persists in their coordination chemistry (Balasubramanian *et al.*, 1997, and references therein). (3-Halostannyl)alkanones, *e.g.*  $X_2\text{Sn}(\text{CR}_2\text{CH}_2\text{COR})_2$ , have been known for a similar period (Hutton & Oakes, 1976), but have been less extensively investigated, especially in regard to their coordination chemistry. Comparison of the structures of the title compound,  $\text{I}_2\text{Sn}(\text{CMe}_2\text{CH}_2\text{COMe})_2$ , (I), now reported, and that of  $\text{I}_2\text{Sn}(\text{CH}_2\text{CH}_2\text{CO}_2\text{Me})_2$ , (II), reported previously by Balasubramanian *et al.* (1997), allows the relative donor abilities of the intramolecular ester and ketone groups to be assessed.



Both (I) and (II) exist as molecular compounds, but with different degrees of octahedral distortion. The organic groups act as  $C,O$ -chelating ligands, with the O atoms *trans* to the *cis*-iodides. There are two independent molecules in the structure of (I). Selected geometric parameters for both compounds are listed in Table 1.

The ketone group in (I) is a stronger donor than the ester group in (II), as shown by the shorter Sn—O bonds and the longer Sn—I bonds. For comparison, Sn—I bond lengths in four-coordinate tetrahedral diorganotin diiodides are in the range 2.649–2.719 Å (Balasubramanian *et al.*, 1997). Longer Sn—I bond lengths [2.965 (1) and 3.009 (1) Å] were found in octahedral [ $(p\text{-MeC}_6\text{H}_4)\{2,6\text{-(Me}_2\text{NCH}_2)_2\text{C}_6\text{H}_3\text{-C,N,N''}\}\text{SnI}_2$ ], but here the iodides were in a *trans* arrangement (Jastrzebski *et al.*, 1991).

The chelate bite angles in (I) [74.66 (17)–75.15 (17)°] are slightly larger than those in (II) [73.9 (4) and 72.8 (3)°; Balasubramanian *et al.*, 1997]. The bite angles result in octahedral distortions and there are significant differences between the C—Sn—C angles in (I) and (II): 144.0 (5)° in (II), and 160.4 (2) and 163.1 (2)° in the two independent molecules of (I).



**Figure 1**

A view of molecule A of (I), showing the atom-labelling scheme used for both molecules of the asymmetric unit, which are differentiated in the text by suffix A or B. Displacement ellipsoids are at the 50% probability level and H atoms have been omitted for clarity.

### Experimental

Compound (I) was prepared by the halogen exchange reaction between  $\text{Cl}_2\text{Sn}(\text{CMe}_2\text{CH}_2\text{COMe})_2$  (Hutton & Oakes, 1976) and excess NaI in acetone solution. Crystals were obtained from a solution in EtOH. The compound decomposed on heating at 444–445 K.

#### Crystal data

$[\text{SnI}_2(\text{C}_6\text{H}_{11}\text{O})_2]$   
 $M_r = 570.79$   
Triclinic,  $P\bar{1}$   
 $a = 7.8655$  (2) Å  
 $b = 14.8464$  (4) Å  
 $c = 16.4602$  (4) Å  
 $\alpha = 108.0591$  (14)°  
 $\beta = 102.0352$  (14)°  
 $\gamma = 90.0908$  (14)°  
 $V = 1782.73$  (8) Å<sup>3</sup>

$Z = 4$   
 $D_x = 2.127$  Mg m<sup>-3</sup>  
Mo  $K\alpha$  radiation  
Cell parameters from 28 392 reflections  
 $\theta = 1.45\text{--}27.53^\circ$   
 $\mu = 4.888$  mm<sup>-1</sup>  
 $T = 150$  (2) K  
Plate, colourless  
0.12 × 0.10 × 0.05 mm

#### Data collection

Nonius KappaCCD area-detector diffractometer  
 $\varphi$  and  $\omega$  scans to fill the Ewald sphere  
Absorption correction: empirical (SORTAV; Blessing, 1997)  
 $T_{\min} = 0.692$ ,  $T_{\max} = 0.795$   
28 392 measured reflections

8156 independent reflections  
6019 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.069$   
 $\theta_{\text{max}} = 27.53^\circ$   
 $h = -10 \rightarrow 9$   
 $k = -19 \rightarrow 19$   
 $l = -21 \rightarrow 21$

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.042$   
 $wR(F^2) = 0.100$   
 $S = 0.998$   
 8156 reflections  
 320 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0464P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 2.36 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -1.62 \text{ e } \text{\AA}^{-3}$   
 Extinction correction: *SHELXL97*  
 (Sheldrick, 1997)  
 Extinction coefficient: 0.00079 (12)

Table 1

Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ) for (I) and (II).

	Compound (I)		Compound (II) <sup>a</sup>
	Molecule A	Molecule B	
Sn1—C1	2.219 (6)	2.215 (5)	2.123 (10)
Sn1—C7	2.220 (6)	2.218 (6)	2.144 (11)
Sn1—O1	2.428 (4)	2.439 (4)	2.522 (8)
Sn1—O2	2.438 (4)	2.435 (4)	2.528 (7)
Sn1—I1	2.8354 (5)	2.8551 (5)	2.7775 (12)
Sn1—I2	2.8572 (5)	2.8378 (5)	2.7801 (11)
C1—Sn1—O1	74.67 (18)	74.74 (18)	72.8 (3)
C7—Sn1—O2	74.66 (17)	75.15 (17)	73.9 (4)
O1—Sn1—O2	75.67 (15)	75.37 (14)	86.6 (3)
C1—Sn1—C7	160.4 (2)	161.1 (2)	144.0 (5)
O2—Sn1—I1	163.68 (11)	162.56 (11)	173.7 (2)
O1—Sn1—I2	163.11 (11)	163.49 (10)	173.5 (2)
I1—Sn1—I2	105.037 (16)	105.675 (16)	98.52 (3)

Notes: (a) Corresponding parameters for (II) (Balasubramanian *et al.*, 1997).

H atoms were placed in calculated positions and refined with a riding model. Maximum and minimum  $\Delta\rho$  were found 1.01  $\text{\AA}$  from I1B and 0.87  $\text{\AA}$  from Sn1B, respectively.

Data collection: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hooft, 1998); cell refinement: *DENZO* and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: GD1099). Services for accessing these data are described at the back of the journal.

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