

Bis(2-carboethoxyethyl)diiodotin at 120 K

R. Alan Howie^{a*} and
Solange M. S. V. Wardell^b^aDepartment of Chemistry, University of Aberdeen, Meston Walk, Aberdeen AB24 3UE, Scotland, and ^bDepartamento de Química Orgânica, Instituto de Química, Universidade Federal Fluminense, 24020-150, Niterói, Rio de Janeiro, Brazil

Correspondence e-mail: r.a.howie@abdn.ac.uk

Key indicators

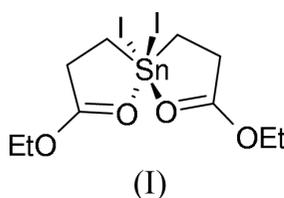
Single-crystal X-ray study
T = 120 K
Mean $\sigma(\text{C}-\text{C}) = 0.009 \text{ \AA}$
R factor = 0.038
wR factor = 0.077
Data-to-parameter ratio = 24.0For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the structure of the title compound, $[\text{Sn}(\text{C}_{10}\text{H}_{18}\text{O}_4)_2]$, Sn lies on a crystallographic twofold axis and the molecule therefore has C_2 molecular symmetry. As is usual for an ester-tin compound of this kind, the carbonyl O atom of the carboethoxy moiety bonds to Sn, creating a five-membered chelate ring and, due to the molecular symmetry, renders Sn six-coordinate.

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Comment

Among the 30 or so structures of ester-tin compounds presently available in the Cambridge Structural Database (CSD; Allen & Kennard, 1993), there are only three entries for bis(2-carboalkoxyethyl)dihalotin(IV) compounds, potentially closely analogous to the title compound, (I). In these analogues, all with space group $P2_1/n$, with no crystallographically imposed molecular symmetry and in that sense isostructural with one another but not with (I), the alkoxy component is, in every case, MeO as distinct from EtO in (I), and the halogen, X, is variously Cl, (III) (BCMESN01; Ng, 1993), I, (II) (NUKJOS; Balasubramanian *et al.*, 1997), or totally disordered Cl/Br (NUKKAF; Balasubramanian *et al.*, 1997). Also present, however, are data for the structure of the compound bis(β -amidoethyl)dichlorotin(IV), (IV) (AMESNC01; Marsh, 1997), in the space group $C2/c$ and to that extent isostructural with (I).



Owing to crystallographically imposed twofold axial symmetry, the asymmetric unit of (I) comprises, in addition to Sn, only one I (II) and a single complete 2-carboethoxyethyl ligand. The complete molecule is depicted in Fig. 1. The bond distances and angles involving Sn in (I) are given in Table 1 and the molecules of (I), (II), (III) and (IV) are compared in terms of the geometry of the five-membered chelate rings and other selected parameters in Table 2.

The distances and angles for (I) in Tables 1 and 2 are much as expected for a molecule of this kind. In Table 1, it is clear that Sn is six-coordinate in an octahedral environment with *trans* atom pairs O1/I1 and C1/C1ⁱ. The octahedron is distorted primarily, in addition to the inevitable differences of Sn—C, Sn—O and Sn—I bond lengths, by the *cis* I1—Sn1—I1ⁱ and O1—Sn1—C1 chelate bite angles.

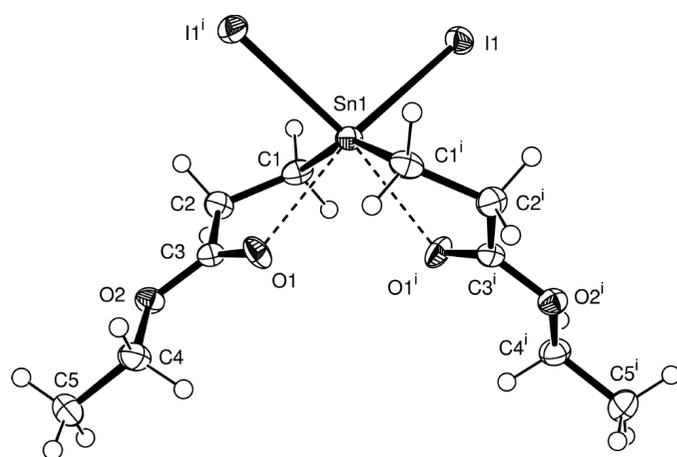


Figure 1
The molecule of (I). Non-H atoms are shown as 50% ellipsoids and H atoms as small circles. [Symmetry code (i) $-x, y, 3/2-z$].

A notable feature of Table 2 is that, for (I) and (IV), the five-membered chelate rings are related by crystallographically imposed C_2 molecular symmetry and are therefore identical. This is not the case for the molecules of (II) and (III), where the chelate rings of the pair not only differ in puckering amplitude [Q_2 ; Cremer & Pople, 1975] but are, in fact, of opposite hand, as shown by the φ_2 parameters differing pairwise by almost exactly 180° in each case. The pucker of the chelate ring in (I) is virtually identical to that of ring 1 of (III) and ring pucker decreases from there in the order: ring 1 of (II), ring 2 of (III) and ring 2 of (II); it is least of all and virtually zero, due the planarity of the system, for (IV). Excepting (IV) for the reason just noted and ring 2 of NUKJOS (envelope on Sn1), the closest description (PLATON; Spek, 1990) of all of the chelate rings is twisted on the C1–Sn1 bond. The dihedral angle between the least-squares planes, calculated with unit weights applied to the ring atoms defining them, of the chelate rings of (I) is $86.85(11)^\circ$. Further features in Table 2 are the limited ranges of Sn–O distances [excluding the case of (IV)], O–Sn–X ($X = \text{Cl}$ or I) angles excluding the case of (I), and particularly the C–Sn–O chelate bite angles. Much greater variation is observed in the C1–Sn1–C1ⁱ bond angle or its equivalent [symmetry code: (i) $-x, y, -z+3/2$]. No intermolecular contacts of any significance are present in the structure of (I).

Experimental

Compound (I) was obtained as a result of halogen exchange with the corresponding chloride, a solution of which (2 mmol) and NaI (*ca* 20 mmol) in acetone (40 ml) was stirred at room temperature for 1 h. The resulting solution was rotary evaporated to dryness and the residue extracted with CHCl_3 (2×30 ml). The combined extracts were rotary evaporated once more and the product, (I), (m.p. 348–350 K) recrystallized from EtOH to yield crystals suitable for analysis.

Crystal data

[Sn(C₁₀H₁₈O₄)I₂]
 $M_r = 574.73$
Monoclinic, $C2/c$
 $a = 20.9399(10) \text{ \AA}$
 $b = 9.1673(5) \text{ \AA}$
 $c = 9.6144(5) \text{ \AA}$
 $\beta = 115.559(3)^\circ$
 $V = 1664.99(15) \text{ \AA}^3$
 $Z = 4$

$D_x = 2.293 \text{ Mg m}^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 2840 reflections
 $\theta = 2.9\text{--}27.5^\circ$
 $\mu = 5.24 \text{ mm}^{-1}$
 $T = 120(2) \text{ K}$
Prism, colourless
 $0.05 \times 0.02 \times 0.02 \text{ mm}$

Data collection

Enraf–Nonius KappaCCD area-detector diffractometer
 φ and ω scans
Absorption correction: multi-scan (SORTAV; Blessing, 1995, 1997)
 $T_{\min} = 0.113, T_{\max} = 0.142$
5078 measured reflections

1898 independent reflections
1390 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.060$
 $\theta_{\text{max}} = 27.5^\circ$
 $h = -27 \rightarrow 22$
 $k = -10 \rightarrow 11$
 $l = -12 \rightarrow 10$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.038$
 $wR(F^2) = 0.077$
 $S = 0.89$
1898 reflections
79 parameters

$w = 1/[\sigma^2(F^2)]$
H-atom parameters constrained
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 1.51 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -1.17 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters ($\text{\AA}, ^\circ$).

Sn1–C1	2.131 (5)	Sn1–I1	2.7877 (5)
Sn1–O1	2.518 (4)		
C1–Sn1–C1 ⁱ	152.0 (3)	O1–Sn1–I1 ⁱ	89.42 (9)
C1–Sn1–O1	73.55 (17)	C1–Sn1–I1	98.24 (15)
C1–Sn1–O1 ⁱ	84.99 (17)	O1–Sn1–I1	166.87 (9)
O1–Sn1–O1 ⁱ	79.80 (18)	I1–Sn1–I1 ⁱ	102.19 (2)
C1–Sn1–I1 ⁱ	99.21 (15)		

Symmetry code: (i) $-x, y, \frac{3}{2} - z$.

Table 2

Comparison of selected parameters of the molecular geometry ($\text{\AA}, ^\circ$) of (I), (II), (III) and (IV) of general formula $[\text{RC}(\text{O})\text{CH}_2\text{CH}_2]_2\text{SnX}_2$ with $R = \text{EtO}, \text{MeO}$ or NH_2 and $X = \text{Cl}$ or I.

	(I) ^a	(II) ^b Ring 1	(II) ^b Ring 2	(III) ^c Ring 1	(III) ^c Ring 2	(IV) ^d
Sn1–I1 ^e	2.7877 (5)	2.777 (2)	2.780 (2)	2.4107 (12)	2.4006 (11)	2.461
Sn1–O1	2.518 (4)	2.523 (7)	2.527 (8)	2.535 (3)	2.519 (3)	2.331
O1–Sn1–C1	73.55 (17)	72.89 (10)	79.93 (12)	72.8 (2)	73.9 (4)	78.7
I1–Sn1–O1	166.87 (9)	173.50 (19)	173.7 (2)	175.17 (6)	175.53 (5)	171.9
C1–Sn1–C1 ⁱ	152.0 (3)	144.0 (3)		144.49 (14)		161.0
C1–Sn1–O1–C3	–21.5 (4)	17.9 (7)	–6.6 (8)	–20.7 (2)	14.2 (2)	3.3
O1–Sn1–C1–C2	29.3 (4)	–26.9 (5)	6.3 (10)	29.7 (2)	–22.3 (3)	–0.58
Sn1–C1–C2–C3	–36.8 (6)	35.6 (8)	–6.6 (17)	–38.9 (3)	30.0 (4)	–1.7
C1–C2–C3–O1	17.3 (8)	–19.2 (14)	0 (2)	20.1 (5)	–17.2 (5)	5.2
C2–C3–O1–Sn1	7.7 (7)	–3.8 (12)	5.5 (13)	5.7 (4)	–2.2 (4)	–5.5
$Q(2)^f$	0.422 (5)	0.379 (8)	0.098 (10)	0.425 (3)	0.311 (3)	
$\varphi(2)^f$	339.5 (8)	155.4 (15)	358 (8)	337.4 (5)	154.2 (7)	

Notes: (a) $R = \text{EtO}, X = \text{I}$; (b) $R = \text{MeO}, X = \text{I}$ (NUKJOS; Balasubramanian *et al.* 1997); (c) $R = \text{MeO}, X = \text{Cl}$ (BCMESN01; Ng, 1993); (d) $R = \text{NH}_2, X = \text{Cl}$ (AMESNC01; Marsh, 1997), s.u.'s on coordinates unavailable in this CSD entry; (e) atom designations as for (I), symmetry code: (i) $-x, y, 3/2-z$; (f) puckering parameters as defined by Cremer & Pople (1975), with φ_2 calculated in every case for the ring atoms in the same sequence starting with Sn and then O followed by the three C atoms in cyclic order in order to obtain truly comparable values.

Anisotropic displacement parameters were refined for all non-H atoms. In the final stages of refinement, H atoms were introduced in calculated positions with C–H distances of 0.98 and 0.99 Å for methyl and methylene H, respectively, and refined with a riding model, with U_{iso} values of 1.5 and $1.2U_{\text{eq}}$ of the attached C atoms. The rotational position of the rigid body methyl group was also refined. Large peaks in the final difference map were always close to Sn and I and are attributed to ripple.

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: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 1990).

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