metal-organic papers

Bis[2,3-bis(4-chlorophenyl)propenoato-κ^2O,O']-dimethyltin(IV)

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The Sn atom in the title compound, $[Sn(CH_3)_2(C_{15}H_9Cl_2O_2)_2]$, has a highly distorted octahedral coordination that may be best described as skew-trapezoid planar for the carboxylates. The ligands are asymmetrically coordinated to the Sn atoms with Sn—O covalent bonds and dative bonds. The mean Sn—C distance for the methyl groups is 2.094 (6) Å, with a bent C—Sn—C angle of 145.77 (11)°.
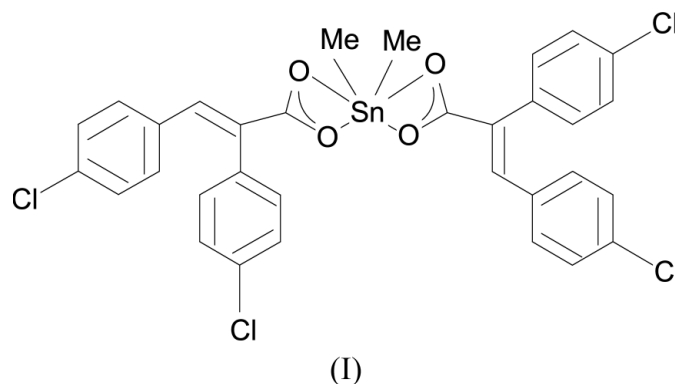
Comment

The production of organotin compounds, especially in the area of biocides and fungicides, has escalated since the early 1980's (Fent & Muller, 1991). Unlike non-toxic inorganic tin compounds, organotin species have been implicated in acute poisoning in humans (Rudel, 2003; Jiang *et al.*, 2000) and animals (Wester *et al.*, 1990; Martin *et al.*, 1989). Owing to their cytotoxicity, these compounds have been explored as anti-tumor agents (Gielen *et al.*, 2000). Several investigators have studied these biologically active organotin species with a coordination number greater than four (Di Stefano *et al.*, 2004; Mehring *et al.*, 1998; Teoh *et al.*, 1999; Carcelli *et al.*, 1995). The structural aspects of these biologically active ligands have been reviewed by several investigators (Nath *et al.*, 2001; Chandrasekhar *et al.*, 2002). Continuing our interest in the structural aspects of organotin carboxylates (Parvez, Ali, Bhatti *et al.*, 1999; Parvez, Ali, Mazhar, Bhatti & Choudhary, 1999; Parvez, Ali, Mazhar, Bhatti & Khokhar, 1999; Parvez *et al.*, 2000, 2002; Sadiq-ur-Rehman *et al.*, 2004), we now report the structure of bis[2,3-bis(4-chlorophenyl)propenoato-κ^2O,O']dimethyltin(IV), (I).

(I)

The structure of (I) is composed of discrete monomeric molecules (Fig. 1), in which hexacoordinated Sn atoms are bonded to two methyl groups and two 2,3-bis(4-chloro-
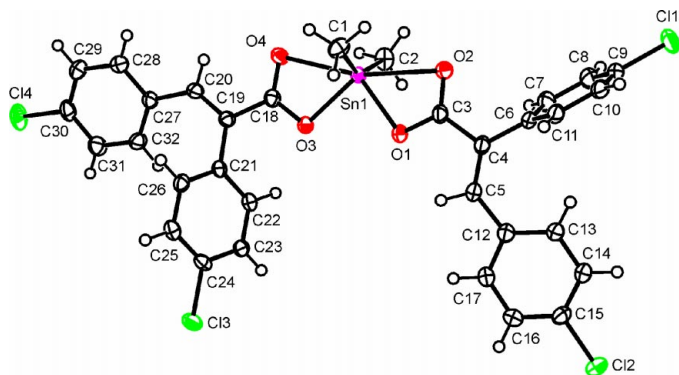
Figure 1
*ORTEP*II (Johnson, 1976) drawing of (I), with displacement ellipsoids drawn at 50% probability level.

Figure 2
*ORTEP*II (Johnson, 1976) drawing of the cell contents of (I). H atoms have been omitted.

phenyl)propenoate ligands. The geometry around the Sn atom is highly distorted octahedral and may best be described as skew-trapezoid planar for the carboxylates. The Sn atom lies only 0.0394 (10) Å from the plane formed by the two carboxylate groups and the two methyl groups lie above and below this plane. The mean Sn—C distance of 2.094 (6) Å and the C—Sn—C angle of 145.77 (11)° agree with the corresponding distances and angles reported earlier (Gibson *et al.*, 1997; Parvez *et al.*, 1997; Hans *et al.*, 2002). The propenoate ligands are asymmetrically coordinated to the Sn atoms, with short Sn—O covalent bonds [2.116 (2) and 2.127 (2) Å] and long dative bonds [2.544 (2) and 2.491 (2) Å]. These bond distances are very similar to the corresponding distances reported in some similar organotin compounds (Hans *et al.*, 2002; Stocco *et al.*, 1996; Gielen *et al.*, 1998; Gibson *et al.*, 1997; Parvez *et al.*, 1997, 2000; Ramirez *et al.*, 2002). The orientation of the benzene rings in the two ligands differs significantly. Rings C6–C11 and C12–C17 are inclined at an angle of 81.94 (8)° with respect to each other, while the corresponding angle in the second ligand (between rings C21–C26 and C27–C32) is 59.66 (9)°. The propenoate moieties, *viz.* O1/O2/C3/C4/C5 and O3/O4/C18/C19/C20, are individually planar, with maximum deviations of 0.036 (2) and 0.029 (2) Å for atoms C4 and C19, respectively. Benzene rings C6–C11 and C12–C17 are inclined at 71.13 (7) and 11.80 (18)°, respectively, to the mean plane of the propenoate moiety O1/O2/C3/C4/C5. For the second ligand, the corresponding angles between the planes of the benzene rings C21–C26 and C27–C32 and the propenoate moiety O3/O4/C18/C19/C20 are 52.60 (10) and 24.78 (15)°, respectively. The molecular dimensions in the ligands are comparable to those reported previously (Sadiq-ur-Rehman *et al.*, 2004).

Experimental

The silver salt of 2,3-bis(4-chlorophenyl)propenoic acid (2.0 g, 5.0 mmol) was refluxed for 9–10 h with dimethyltin dichloride (0.55 g, 2.5 mmol) in a 2:1 molar ratio. The solvent used was dry chloroform (60 ml) contained in a two-necked round-bottomed flask (250 ml), equipped with a water conden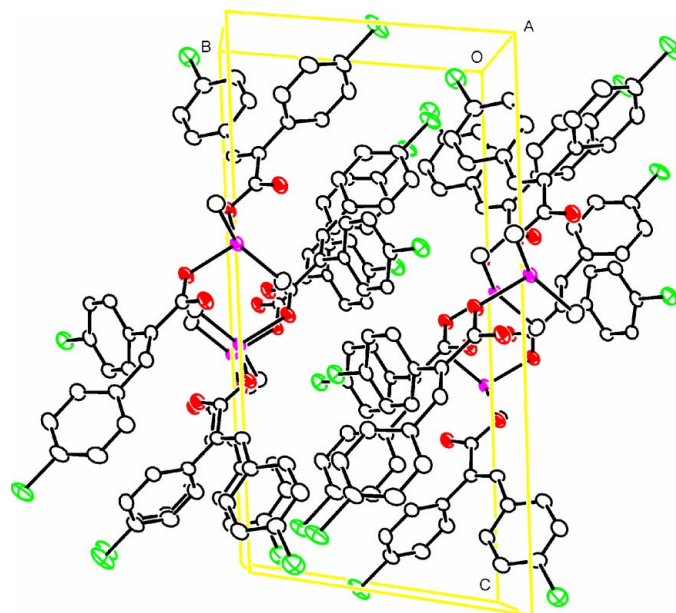ser and magnet bar. It was then allowed to stand overnight at room temperature. The silver salt formed was filtered off and the solvent was evaporated under reduced pressure. The resultant solid mass was recrystallized from a chloroform and *n*-hexane (4:1) mixture. Crystals suitable for single-crystal X-ray analysis were grown by dissolving 0.5 g of the compound in a minimum amount of chloroform (5.0 ml) to which a few drops of *n*-hexane were added. Slow evaporation of the solvent at room temperature over several days yielded fine crystals, which were then subsequently washed with *n*-hexane (yield = 80%; m.p. = 473–476 K).

Crystal data

[Sn(CH₃)₂(C₁₅H₉Cl₂O₂)₂]	$Z = 2$
$M_r = 733.00$	$D_x = 1.625$ Mg m⁻³
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 8.4740$ (11) Å	Cell parameters from 12446
$b = 10.2770$ (12) Å	reflections
$c = 19.472$ (4) Å	$\theta = 3.1$–27.3°
$\alpha = 95.817$ (6)°	$\mu = 1.25$ mm⁻¹
$\beta = 99.748$ (7)°	$T = 173$ (2) K
$\gamma = 114.116$ (8)°	Prism, colorless
$V = 1497.7$ (4) Å³	$0.14 \times 0.12 \times 0.10$ mm

Data collection

Nonius KappaCCD diffractometer	5570 reflections with $I > 2\sigma(I)$
ω and φ scans	$R_{int} = 0.032$
Absorption correction: multi-scan	$\theta_{max} = 27.3°$
(*SORTAV*; Blessing, 1997)	$h = -10 \rightarrow 10$
$T_{min} = 0.845$, $T_{max} = 0.886$	$k = -13 \rightarrow 13$
12446 measured reflections	$l = -25 \rightarrow 25$
6683 independent reflections	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0343P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.032$	$+ 0.4509P]$
$wR(F^2) = 0.075$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} = 0.01$
6683 reflections	$\Delta\rho_{max} = 0.43$ e Å⁻³
372 parameters	$\Delta\rho_{min} = -0.68$ e Å⁻³
H-atom parameters constrained	

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Sn1—C1	2.089 (3)	Cl2—C15	1.742 (3)
Sn1—C2	2.100 (3)	Cl3—C24	1.743 (2)
Sn1—O1	2.116 (2)	Cl4—C30	1.737 (3)
Sn1—O3	2.127 (2)	O1—C3	1.297 (3)
Sn1—O4	2.491 (2)	O2—C3	1.246 (3)
Sn1—O2	2.544 (2)	O3—C18	1.294 (3)
Cl1—C9	1.743 (3)	O4—C18	1.249 (3)
C1—Sn1—C2	145.77 (11)	C1—Sn1—O2	88.61 (9)
C1—Sn1—O1	101.90 (9)	C2—Sn1—O2	83.70 (9)
C2—Sn1—O1	100.97 (10)	O1—Sn1—O2	55.55 (6)
C1—Sn1—O3	101.12 (9)	O3—Sn1—O2	139.83 (6)
C2—Sn1—O3	106.20 (9)	O4—Sn1—O2	164.02 (5)
O1—Sn1—O3	84.27 (6)	C3—O1—Sn1	101.33 (14)
C1—Sn1—O4	89.28 (9)	C3—O2—Sn1	82.90 (14)
C2—Sn1—O4	89.19 (9)	C18—O3—Sn1	100.18 (14)
O1—Sn1—O4	140.23 (6)	C18—O4—Sn1	84.58 (14)
O3—Sn1—O4	56.03 (6)		

The H atoms were located in difference Fourier syntheses, but were included in the refinement at geometrically idealized positions, with C—H = 0.95 and 0.98 Å and U_{iso} = 1.5 (methyl) and 1.2 (aromatic) times U_{eq} of the atoms to which they are bonded. The final difference map was free of any chemically significant features.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *HKL DENZO* (Otwinowski & Minor, 1997); data reduction: *SCALE-PACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SAPI*91 (Fan, 1991); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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