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Chloro{μ-2-[(*E*)-1-(2-oxido-3-methylphenyl)-ethylideneamino]acetato}pentaphenylditin(IV)

Anthony Linden,[a]* Tushar S. Basu Baul[b] and Cheerfulman Masharing[b]

[a]Institute of Organic Chemistry, University of Zürich, Winterthurerstrasse 190, CH-8057 Zürich, Switzerland, and [b]Department of Chemistry, North-Eastern Hill University, NEHU Permanent Campus, Umshing, Shillong 793 022, India

Correspondence e-mail: alinden@oci.unizh.ch

Key indicators

Single-crystal X-ray study
T = 160 K
Mean σ(C–C) = 0.009 Å
R factor = 0.045
wR factor = 0.113
Data-to-parameter ratio = 23.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, [$Sn_2(C_6H_5)_5(C_{11}H_{11}NO_3)Cl$], is a dinuclear organotin adduct in which the two Sn atoms are bridged *via* the carboxylate O—C—O group of a 2-[(*E*)-1-(2-hydroxyaryl)alkylideneamino]acetate ligand. Each Sn atom has a distorted trigonal bipyramidal geometry, with the Ph_3SnCl moiety being less distorted.

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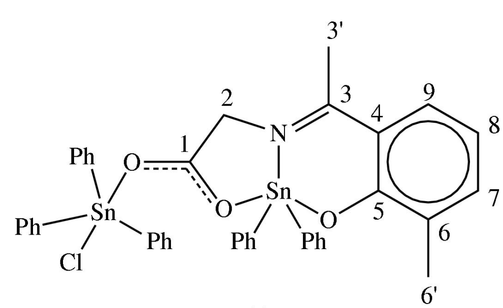
Comment

The title compound, (I), was prepared during an ongoing study of the coordination chemistry of organotin(IV) complexes of 2-[(*E*)-1-(2-hydroxyaryl)alkylideneamino]-acetates (L). These ligand systems generate a great variety of structural forms with R_2Sn– and R_3Sn– moieties (Dakternieks *et al.*, 1998; Basu Baul & Tiekink, 1999; Basu Baul *et al.*, 2001, 2002, 2003, 2005). A few examples of dinuclear organotin adducts of the type $R_2SnL{\cdot}R_2SnCl_2$ (R = Ph, ^{t}Bu; Khoo *et al.*, 1997; Dakternieks *et al.*, 1998) and $R_2SnL{\cdot}R_3SnCl$ (R = Ph; Dakternieks *et al.*, 1998; Basu Baul *et al.*, 2003) are known where two Sn atoms are bridged *via* the carboxylate O—C—O group of an L ligand. These considerations stirred our interest in the synthesis and structure of the title compound, (I), which has the $R_2SnL{\cdot}R_3SnCl$ (R = Ph) formulation.



(I)

The structure of (I) is virtually isomorphous with that of the $Ph_2SnL{\cdot}Ph_3SnCl$ adduct reported by Dakternieks *et al.* (1998). The only difference between the two compounds is the addition of the 3-methyl group on the benzene ring of the acetate ligand in (I). In all other respects, the two compounds and structures are the same and have similar coordination geometry at each Sn atom (Table 1). In (I), atom Sn1 has a distorted trigonal bipyramidal coordination geometry, with atoms O1 and O3 occupying axial positions and the O1—Sn—O3 angle distorted from linearity by 19.13 (13)°. The C10—Sn1—C11 angle is also about 14° wider than in an ideal trigonal bipyramid. Atom Sn1 lies 0.027 (1) Å out of the trigonal plane formed by atoms N1, C10 and C11 in the direction of atom O3. The geometry about atom Sn2 is also

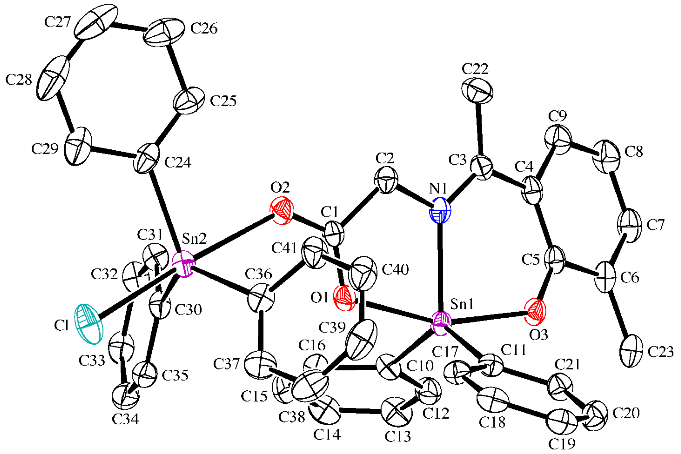


Figure 1
View of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms have been omitted for clarity.

distorted trigonal bipyramidal, with atoms Cl and O2 defining the axial positions, but the O1—Sn—O3 angle is distorted from linearity by only 3.76 (9)°. Atom Sn2 lies 0.224 (1) Å out of the trigonal plane formed by atoms C24, C30 and C36 in the direction of the Cl atom. The O2—Sn bond is about 0.24 Å longer than the O1—Sn1 bond and the carboxylate C—O distances are inversely related. The carboxylate C—O bonds are not completely delocalized, with the C1—O2 bond having much more double-bond character than the C1—O1 bond.

Experimental

Ph_2SnL was prepared by reacting Ph_2SnCl_2 and LHK as described by Basu Baul *et al.* (2001). A hot anhydrous benzene solution (10 ml) of Ph_3SnCl (0.20 g, 0.51 mmol) was added dropwise to a hot stirred benzene solution (20 ml) containing Ph_2SnL (0.25 g, 0.52 mmol) and refluxed for 1 h. The volatiles were removed *in vacuo*, the yellow mass was washed several times with hexane and filtered. The dried residue was dissolved in benzene and filtered to remove any particles. The filtrate was allowed to evaporate at room temperature, which afforded yellow crystals of (I) (52% yield, m.p. 457–458 K). Analysis calculated for $C_{41}H_{36}ClNO_3Sn_2$: C 57.03, H 4.20, N 1.62%; found: C 57.10, H 4.10, N 1.60%; IR (KBr, cm^{-1}): 1626 $\nu(OCO)_{asym}$, 1235 ν[Ph-(C=O)]; ^{1}H NMR ($CDCl_3$, 250.13 MHz): δ 7.83 [*m*, 4H, Sn—Ph^a(*ortho*-)], 7.67 [*m*, 6H, Sn—Ph^b(*ortho*-)], 7.48–7.35 [*m*, 17H, Sn—Ph^{a,b}(*meta*- and *para*-), H-7 and H-9], 6.70 (*t*, 1H, H-8), 4.29 (*s*, 2H, H-2), 2.63 (*s*, 3H, H-3′), 2.45 (*s*, 3H, H-6′); ^{13}C NMR ($CDCl_3$, 62.89 MHz): δ 182.4 (C-1), 170.6 (C-3), 164.8 (C-5), 137.8 [Sn—Ph^a(*ipso*-)], 137.5 [Sn—Ph^b(*ipso*-)], 136.7 (C-7), 136.4 [Sn—Ph^a(*ortho*-)], 136.1 [Sn—Ph^b(*ortho*-)], 131.5 (C-9), 130.7 [Sn—Ph^a(*para*-)], 130.4 [Sn—Ph^b(*para*-)], 129.1 [Sn—Ph^b(*meta*-)], 128.9 [Sn—Ph^a(*meta*-)], 128.6 (C-4), 119.4 (C-6), 117.4 (C-8), 53.6 (C-2), 23.0 (C-3′), 16.8 (C-6′) (*a* and *b* represent signals due to Sn—Ph_2 and Sn—Ph_3, respectively, and refer to the scheme for ligand assignment); ^{119}Sn NMR ($CDCl_3$, 89.12 MHz): δ −51, −351. The ^{119}Sn NMR data indicate the presence of two tin centres both having trigonal bipyramidal geometry in the structure (Dakternieks *et al.*, 1998; Basu Baul *et al.*, 2003). X-ray diffraction quality crystals were grown by slow evaporation of a solution of (I) in acetone.

Crystal data

$[Sn_2(C_6H_5)_5(C_{11}H_{11}NO_3)Cl]$
M_r = 863.39
Triclinic, $P\overline{1}$
a = 11.6984 (2) Å
b = 13.0662 (2) Å
c = 13.9410 (3) Å
α = 111.5066 (9)°
β = 90.6470 (9)°
γ = 115.4904 (9)°
V = 1752.94 (6) Å^3
Z = 2
D_x = 1.636 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 47226 reflections
θ = 2.0–30.0°
μ = 1.54 mm^{-1}
T = 160 (1) K
Prism, yellow
0.15 × 0.15 × 0.08 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ and ω scans with κ offsets
Absorption correction: multi-scan (Blessing, 1995)
T_{min} = 0.759, T_{max} = 0.881
57965 measured reflections
10272 independent reflections
8257 reflections with $I > 2\sigma(I)$
R_{int} = 0.060
θ_{max} = 30.1°
h = −16 → 16
k = −18 → 18
l = −19 → 19

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.113
S = 1.20
10271 reflections
436 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + 10.8098P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.29 e Å^{-3}
$\Delta\rho_{min}$ = −1.06 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0045 (3)

Table 1
Selected geometric parameters (Å, °).

Sn1—O3	2.057 (3)	Sn2—C30	2.132 (4)
Sn1—C11	2.116 (4)	Sn2—C24	2.153 (5)
Sn1—C10	2.119 (4)	Sn2—O2	2.415 (3)
Sn1—N1	2.160 (4)	Sn2—Cl	2.4610 (13)
Sn1—O1	2.172 (3)	O1—C1	1.276 (5)
Sn2—C36	2.117 (5)	O2—C1	1.239 (5)
O3—Sn1—C11	91.89 (15)	C30—Sn2—C24	111.18 (18)
O3—Sn1—C10	94.61 (15)	C36—Sn2—O2	83.24 (16)
C11—Sn1—C10	134.26 (17)	C30—Sn2—O2	84.30 (14)
O3—Sn1—N1	84.27 (13)	C24—Sn2—O2	84.51 (16)
C11—Sn1—N1	112.60 (15)	C36—Sn2—Cl	93.48 (14)
C10—Sn1—N1	113.09 (16)	C30—Sn2—Cl	96.33 (13)
O3—Sn1—O1	160.87 (13)	C24—Sn2—Cl	98.69 (15)
C11—Sn1—O1	94.66 (15)	O2—Sn2—Cl	176.24 (9)
C10—Sn1—O1	93.64 (15)	C1—O1—Sn1	117.5 (3)
N1—Sn1—O1	76.61 (13)	C1—O2—Sn2	134.2 (3)
C36—Sn2—C30	128.01 (18)	C5—O3—Sn1	128.3 (3)
C36—Sn2—C24	117.50 (18)		

The methyl groups were constrained to an ideal geometry (C—H = 0.98 Å), with U_{iso}(H) = 1.5U_{eq}(C), but were allowed to rotate freely about the parent C—C bonds. All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.95–0.99 Å and with U_{iso}(H) = 1.2U_{eq}(C). One low angle reflection was partially obscured by the beam stop and was omitted from the refinement. The largest peak of residual electron density is 1.02 Å from atom H15, but is merely the highest peak in a high background of noise in the difference Fourier map. The deepest electron-density hole lies 0.80 Å from atom Sn2.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); 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, 2003).

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