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Journal of Organometallic Chemistry 590 (1999) 88–92

Journal of Organometallic Chemistry

Synthesis, spectroscopy and structure of [*N*-(2-carboxyphenyl)salicylideneiminato]dimethyltin(IV)

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Received 10 June 1999

Abstract

The novel dimethyltin complex with formula $Me_2Sn(2\text{-}OC_6H_4CH{=}NC_6H_4COO)$, compound **1**, involving the Schiff base derived from anthranilic acid and salicylaldehyde, has been synthesized and characterized by elemental analysis, IR, ^{1}H-, ^{13}C- and ^{117}Sn-NMR, ^{119}Sn Mössbauer spectroscopy and single-crystal X-ray diffraction structure analysis. The crystals of complex **1** are monoclinic, space group $P2_1/n$, $a = 8.907(5)$, $b = 12.044(7)$, $c = 14.062(9)$ Å, $\beta = 104.99(5)°$, $Z = 4$, $R_1 = 0.0296$, $wR_2 = 0.08$ for 1368 observed reflections. The crystal structure of **1** features centrosymmetric dimers disposed about a central Sn_2O_2 core. The tin environment is seven-coordinate if a moderately long contact distance Sn(1)–O(2a) (2.69(1) Å) and a longer one Sn(1)–O(3a) (3.03(1) Å) are included in the coordination list. In the basic monomeric structural unit, the tin configuration can be seen as a distorted square-pyramid with the imine nitrogen atom in apical position, the *N*-(2-carboxyphenyl)salicylideneimine dianion coordinating a single tin atom via the phenolate oxygen atom, the imine nitrogen atom and one of the carboxylate oxygen atoms. The distortion of the square pyramid is discussed in terms of a trend to trigonal-bipyramidal geometry.

Keywords: Dimethyltin complex; Schiff base; X-ray structure; NMR spectra

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1. Introduction

Studies on the coordination chemistry of amino-acid-derived Schiff bases ligating diorganotin(IV) centers have recently received some attention. The mode of coordination of *N*-arylidene-α-amino acids in diorganotin(IV) complexes is known [1–3]. The coordination chemistry of other tridentate ONO donor Schiff bases has also been described [4,5]. Moreover, in diorganotin complexes, amino-acid-derived Schiff bases have been used as coordinating ligands to form uncommon mixed dinuclear organotin species [3]. Diorganotin pyridine-2,6-dicarboxylates [6,7] and di-*n*-butyltin pyridine-2-phosphonate-6-carboxylate [8] exhibit a centrosymmetric dimeric structure with an Sn_2O_2 core. However, no study has been conducted so far on the coordination chemistry of the *N*-(2-carboxyphenyl)salicylideneimine dianion towards diorganotins so that the mode of interaction of such ligands with tin is so far unknown.

We report here the synthesis, characterization and structure of [*N*-(2-carboxyphenyl)salicylideneiminato]-dimethyltin(IV), an investigation lying in the frame of previous studies on diorganotin(IV) complexes of Schiff bases [9].

2. Experimental

2.1. Materials

All chemicals and reagents were of reagent grade quality. Dimethyltin dichloride (Fluka), salicylaldehyde

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PII: S0022-328X(99)00436-2

(Merck), anthranilic acid (s.d. fine chemicals, India) were used as received. Triethylamine (s.d. fine chemicals, India) was dried over KOH. Benzene (AR, thiophene-free) and petroleum ether (40–60°C) were dried by refluxing over freshly cut sodium. Methanol (Ranbaxy, India) was dried over CaO. All solvents were distilled prior to use. Other solvents were dried and purified by standard procedures.

2.2. Physical measurements

Infrared spectra were recorded on a Perkin–Elmer 883 infrared spectrophotometer from 4000–200 cm^{-1} as KBr discs and were calibrated with respect to the 1601 cm^{-1} band of a polystyrene film. Tin was estimated gravimetrically as SnO_2 after decomposition with concentrated HNO_3. Carbon, hydrogen and nitrogen analyses were carried out on a Perkin–Elmer 2400 II elemental analyser. Melting points (uncorrected) were recorded on an electrical heating-coil apparatus. ^{1}H-NMR solution spectra were acquired on Bruker DRX250 and AMX500 instruments. ^{1}H chemical shifts were referenced to the standard Me_4Si scale from residual ^{1}H solvent resonances of chloroform ($CHCl_3$, 7.23 ppm). The ^{119}Sn chemical shift has been determined by a gradient pulsed ^{1}H–^{119}Sn HMQC experiment, as described elsewhere [10]. The CP-MAS NMR spectra were recorded on a Bruker DRX250 spectrometer, tuned to 89.15 and 62.93 MHz for ^{117}Sn and ^{13}C nuclei, respectively, under the same experimental conditions as described previously [11]. ^{117}Sn chemical shift referencing is toward tetracyclohexyltin taken as secondary reference at −97.35 ppm.

The Mössbauer spectra were recorded as described elsewhere [12].

2.3. X-ray crystal structure of $Me_2Sn(2\text{-}OC_6H_4CH{=}C_6H_4COO)$ (**1**)

A yellow crystal of $Me_2Sn(2\text{-}OC_6H_4CH{=}NC_6H_4\text{-}COO)$ (**1**), obtained from methanol, was mounted on a glass fiber. Cell dimension and intensity data for a crystal of **1** of dimension 0.30 × 0.20 × 0.20 mm were measured at 293(2) K on a Syntex $P2_1$ 4-circle diffractometer fitted with a graphite monochromated Mo–K_α radiation, $\lambda = 0.71073$ Å. A total of 1480 reflections in the θ range 2.26–20.04° were collected using ω scans. There were 1368 independent reflections ($R_{\text{int}} = 0.0317$), which were used in the subsequent structure determination and refinement. The stability of the crystal was checked by measuring standard reflections at fixed intervals during data collection: no significant loss of intensity was noted for the crystal. The data were corrected for Lorentz and polarization effects but not for absorption. The structure was solved by direct methods using SHELXTL-PLUS [13] and refined by a full-matrix least-squares procedure based on F^2 using SHELXL-93 [14]. Non-hydrogen atoms were refined with anisotropic displacement parameters and hydrogen atoms were included in their calculated position and refined with fixed isotropic displacement parameters. There were 1368 data, zero restraints and 193 parameters. The maximum difference peak and hole were 0.537 and −0.432 e Å^{-3}. The details of the structure solution and the refinement are given in Table 1.

Table 1
Crystal data and structure refinement of $Me_2Sn(2\text{-}OC_6H_4CH{=}NC_6H_4COO)$ (**1**)

Empirical formula	$C_{16}H_{15}NO_3Sn$
Formula weight	387.98
Temperature (K)	293(2)
Wavelength (Å)	0.71073
Crystal system	Monoclinic
Space group	$P2_1/n$
a (Å)	8.907(5)
b (Å)	12.044(7)
c (Å)	14.062(9)
β (°)	104.99(4)
Volume (Å^3)	1457(2)
Z	4
$D_{\text{calc.}}$ (Mg m^{-3})	1.769
μ (mm^{-1})	1.762
F(000)	768
Crystal size (mm)	0.3 × 0.2 × 0.2
θ (°)	2.26–20.04
Index ranges	$0 \le h \le 8$, $0 \le k \le 11$, $-13 \le l \le 13$
Reflections collected	1480
Independent reflections ($R_{\text{int}} = 0.0317$)	1368
Data/restraints/parameters	1368/0/193
Goodness-of-fit on F^2	1.072
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0296$, $wR_2 = 0.0800$
R indices (all data) [a]	$R_1 = 0.0343$, $wR_2 = 0.0825$
Extinction coefficient	0.0033(8)
Largest diff. peak and hole (e Å^{-3})	0.537 and −0.432

[a] $w = 1/[\sigma^2(F_o^2) + (0.0571P)^2 + 0.03P]$; $P = (F_o^2 + 2F_c^2]/3$.

2.4. Preparation of the Schiff base

The Schiff base has been synthesized by stirring at room temperature (r.t.) a 1:1 molar ratio mixture of anthranilic acid and salicylaldehyde in methanol following a reported method [15]. Its structure is given in Fig. 1.

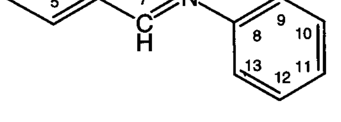

Fig. 1. Structure of the Schiff base H_2L.

2.5. Preparation and characteristics of the complex

To a stirred solution of *N*-(2-carboxyphenyl)-solicylideneimine (0.549 g, 2.28 mmol) and triethylamine in 30 ml dry methanol, a solution of Me_2SnCl_2 (0.500 g, 2.28 mmol) in 10 ml of dry methanol was added slowly at r.t. The mixture was then stirred at r.t. for 2 h. During this period, the yellow crystalline precipitate formed was filtered and washed with petroleum ether (40–60°C). Single crystals of **1** suitable for X-ray crystallography were obtained when the mother liquor was kept overnight. Yield: 0.65 g (74%); m.p. > 250°C.

Anal. data (%): Found: C, 49.48; H, 3.81; N, 3.44; Sn, 30.26. Calc. for $C_{16}H_{15}NO_3Sn$ (formula weight 387.98): C, 49.53; H, 3.89; N, 3.61; Sn, 30.59. IR (cm^{-1}): 1622 $\nu(OCO)_{asym}$, 1605 ν(C=N). ^{1}H-NMR ($CDCl_3$ solution, chemical shifts in ppm toward TMS, coupling constants in Hz): δ 6.86 (d, 7, 1H, H-2), 7.50 (ddd, 7, 7, 1, 1H, H-3), 6.84 (dd, 7, 7, 1H, H-4), 7.38 (d, 7, 1H, H-5), 8.44 (s, 1H, H-7, $^3J(^1H\text{-}^{119}Sn) = 39$), 8.30 (dd, 7, 2, 1H) and 7.02 (d, 7, 1H), H-10 & H-13, 7.50 (ddd, 7, 7, 1, 1H) and 7.56 (ddd, 7, 7, 2, 1H), H-11 & H-12, 0.81 (s, 6H, $^2J(^1H\text{–}^{117/119}Sn) = 77/80$, Sn–$CH_3$). ^{13}C-NMR (CPMAS): δ 147 (C-1, C-6, C-8, C-9), 136, 130, 125 (C-2, C-3, C-4, C-5, C-10, C-11, C-12, C-13), 115 (C-7), 173 (C-14), 10 (Sn–CH_3, $^1J(^{13}C\text{–}^{117/119}Sn) = 960$). ^{119}Sn-NMR ($CDCl_3$): δ −328. ^{117}Sn-NMR (CPMAS): δ −306.

Mössbauer spectrum (mm s^{-1}): QS = 3.89, IS = 1.32, Γ_1: 1.08, Γ_2: 1.14.

^{1}H parameters were assigned from chemical shift values and $^nJ(^1H\text{–}^1H)$ coupling patterns. Discrimination between H(2) and H(5) was achieved by comparison with the spectral data of organotin salicylaldoximates [10].

3. Results and discussion

3.1. Synthesis

The dimethyltin(IV) complex of the Schiff bases derived from amino acids and salicylaldehydes was previously prepared by refluxing a mixture of dimethyltin oxide, Me_2SnO, and the corresponding Schiff bases in toluene with the azeotropic removal of water [1,2]. Similar compound types were likewise prepared by the reaction between diorganotin dichlorides and the sodium/potassium salts of the respective Schiff bases [3].

The compound reported here has been synthesized from dimethyltin(IV) dichloride, Me_2SnCl_2 and the Schiff base in methanol in the presence of triethylamine as a Lewis base in 10% molar excess (Eq. (1)). The synthesized complex precipitated from the reaction mixture.

$$Me_2SnCl_2 + H_2L + 2Et_3N \xrightarrow[\text{room temperature}]{\text{methanol}} Me_2SnL + 2Et_3NH^{(+)}Cl^{(-)} \quad (1)$$

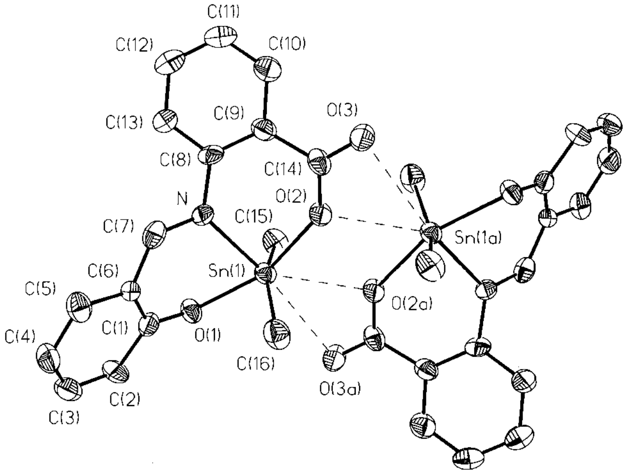

Fig. 2. Molecular structure and atomic numbering scheme for $Me_2Sn(2\text{-}OC_6H_4CH{=}NC_6H_4COO)$ (**1**). Symmetry operation a is $-x, -y, 1-z$.

where H_2L = tridentate Schiff base.

The compound has been characterized by elemental analysis, IR, ^{1}H-NMR in solution, and CP-MAS ^{13}C- and ^{117}Sn-NMR in the solid state as well as by ^{119m}Sn Mössbauer spectroscopy (see Section 2). Solubility is too low in usual solvents for getting meaningful ^{13}C- and ^{119}Sn-NMR in solution, but the ^{119}Sn chemical shift could be determined by $^1H\text{–}^{119}Sn$ HMQC NMR spectroscopy [10]. The structure of the compound has been determined by X-ray diffraction.

Although this reaction has been carried out in methanol at room temperature, this compound could be prepared as well in other solvents such as benzene or toluene. The reaction time could be reduced by carrying out the reaction at higher temperature. The resulting yellow compound is stable under atmospheric conditions.

3.2. Description of the X-ray structure of $Me_2Sn(2\text{-}OC_6H_4CH{=}NC_6H_4COO)$ (1)

The molecular structure of **1** is shown in Fig. 2. Selected bond lengths and angles are given in Table 2. The tin environment is seven-coordinate if a moderately long contact distance Sn(1)–O(2a) (2.69(1) Å) and an even longer one Sn(1)–O(3a) (3.03(1) Å) are included in the coordination list. The crystal structure of **1** can then be compared with some diorganotin pyridine-2,6-dicarboxylates [8] where the tin atom is always seven-coordinate in a centrosymmetric arrangement leading to a Sn_2O_2 core; the overall tin geometry is based on a distorted pentagonal bipyramid with the carbons in apical positions *trans* to one another, though C–Sn–C = 155.1(3)° is rather small compared with 168.2(3)° in [8]. The deviation of Sn from the least-squares plane (mean deviation 0.042 Å) is only 0.056 Å, about four times larger than the deviations reported in [8]. The O–Sn–O angle in the four-membered

Table 2
Selected bond lengths (Å) and angles (°) of $Me_2Sn(2\text{-}OC_6H_4CH{=}NC_6H_4COO)$ (**1**)

Bond lengths			
Sn(1)–C(15)	2.102(7)	Sn(1)–C(16)	2.103(6)
Sn(1)–O(1)	2.135(4)	Sn(1)–O(2)	2.187(4)
Sn(1)–O(2a)	2.69(1)	Sn(1)–O(3a)	3.03(1)
Sn(1)–N	2.230(5)	N–C(7)	1.304(7)
N–C(8)	1.435(8)	O(1)–C(1)	1.304(7)
O(2)–C(14)	1.282(7)	O(3)–C(14)	1.217(7)
Bond angles			
C(15)–Sn(1)–C(16)	155.1(3)	C(15)–Sn(1)–O(1)	90.8(2)
C(16)–Sn(1)–O(1)	91.0(2)	C(15)–Sn(1)–O(2)	97.7(2)
C(16)–Sn(1)–O(2)	90.1(2)	O(1)–Sn(1)–O(2)	157.2(2)
C(15)–Sn(1)–N	103.6(2)	C(16)–Sn(1)–N	101.1(2)
O(1)–Sn(1)–N	80.1(2)	O(2)–Sn(1)–N	77.4(2)
C(7)–N–Sn(1)	119.6(4)	C(8)–N–Sn(1)	123.6(4)
C(1)–O(1)–Sn(1)	122.9(4)	C(14)–O(2)–Sn(1)	138.8(4)
O(3)–C(14)–O(2)	121.6(6)	O(3)–C(14)–C(9)	120.3(6)

core around the center of inversion is 64.9(2)° compared with 65.6(2)° in [8].

These similarities are surprising remembering that the extremely long contact distance of 3.03 Å to O(3a) was included in the coordination list of Sn. If O(3a) is omitted from the coordination list the question arises at which distance the coordination sphere should be cut off. The maximum distance gap criterion [16] might be helpful in this case: the maximum distance gap is 0.46 Å between Sn(1)–O = 2.23 Å and Sn(1)–O(2a) = 2.69 Å; the gap between the Sn(1)–O(2a) and Sn(1)–O(3a) distances is smaller by 0.34 Å only. With O(2a) and O(3a) omitted from the coordination list a pentacoordinated Sn remains. In the latter view, the coordination polyhedron may be regarded as a strongly distorted tetragonal pyramid, but a discussion in terms of a distorted trigonal bipyramid seems to be even more interesting: the deviation of Sn from the basal plane defined by N, C(15) and C(16) is only 0.037(4) Å. Similar diorganotin(IV) complexes derived from *N*-2-hydroxyarylidene-α-amino acids [1–3] and *N*-(2-hydroxyphenyl)salicylideneimine [4,5] have distorted trigonal–bipyramidal structure. However, the C–Sn–C angle of 155°, too small in comparison with the ideal 180° angle of regular pentagonal bipyramidal structures, is now too large when compared with the angles of about 125° observed in the trigonal basal planes of the trigonal–bipyramidal structures. The structure of **1** may thus be described as a compromise between the dimeric pentagonal–bipyramidal structure type and the monomeric trigonal bipyramidal ones.

The strong distortion from regular symmetry is seen in the departure from 180° of the angles O(1)–Sn(1)–O(2) [157.2(2)°] and C(15)–Sn(1)–C(16) (155.1(3)°) and from 90° of C(15)–Sn(1)–N (103.6(2)°), C(16)–Sn(1)–N (101.1(2)°), O(1)–Sn(1)–N (80.1(2)°) and O(2)–Sn(1)–N (77.4(2)°). This distortion is mainly due to the rigidity of chelate rings, together with the large covalent radius of tin(IV), as well as the very different lengths of the Sn–N (2.230(5)Å) and Sn–O (2.69(1) Å) bonds *trans* to one another. The bite angles O(1)–Sn(1)–N [80.1(2)°] and O(2)–Sn(1)–N (77.4(2)°) are comparable to those reported for other organotin(IV) complexes containing six-membered chelate rings with oxygen and nitrogen donor atoms [1,3,15] and 80.19(6), 78.98(7)° for *n*-Bu_2Sn-(Vanophen) [17] (H_2Vanophen = *N*,*N′*-bis(3-methoxysalicylaldehyde)-1,2-phenylenediimine).

None of the six-membered chelate rings is planar as seen from the following torsion angles: Sn(1)–O(2)–C(14)–C(9) (1.1°), Sn(1)–N–C(8)–C(9) (−40.6°), Sn(1)–N–C(7)–C(6) (−17.0°), Sn(1)–O(1)–C(1)–C(6) (36.5°).

The Sn(1)–O(2) bond length (2.187(4) Å) is larger than the Sn(1)–O(1) one (2.135(4) Å). Such a difference in bond lengths has been found in other similar complexes [1–3]. These Sn–O bonds are slightly longer than in other organotin(IV) complexes derived from ONO donor tridentate Schiff bases [1–5]. The Sn–N bond length of 2.230(5) Å is comparable to those observed for $Ph_2Sn(OC_6H_4CH{=}NC_6H_4O)$ [4], $[Vin_2Sn(2\text{-}OC_6H_4C(CH_3){=}NCH_2COO)\cdot OH_2]$ [3] and the six-coordinate complex Ph_2Sn(NAPPDI) [18] (H_2NAPPDI=*N*,*N′*-bis(2-hydroxy-1-naphthaldehyde)-1,2-phenylenediimine). This Sn–N bond is longer than in other organotin(IV) complexes derived from ONO donor tridentate Schiff bases [1,3] and shorter than in the six-coordinate complexes Me_2Sn-(Salen) [19] and R_2Sn(Vanophen) (R = Ph, *n*-Bu, Me) [9] (H_2Salen = *N*,*N′*-bis(salicylaldehyde)ethylenediimine).

The $Sn–C_{methyl}$ bond lengths (2.102(7) and 2.103(6) Å) are much shorter than the $Sn–C_{methyl}$ bond lengths in Me_2SnCl_2 (2.21(8) Å) [20], which is expected as an increase in coordination number of dimethyltin(IV) derivatives is accompanied by a decrease in Sn–C bond lengths, because tin 5s electron density is preferentially concentrated into bonds with carbon.

3.3. Structure of 1 in solution

On the basis of Lockhart's equation [Me–C–Me $= 0.0161(|\,{}^2J(^{119}Sn–^1H)|)^2 - 1.32\,{}^2J(^{119}Sn–^1H)\,|) + 133.4$] [21], the C(15)–Sn(1)–C(16) angle is estimated to ca. 130°. The ^{119}Sn chemical shift, as determined by $^1H–^{119}Sn$ HMQC spectroscopy [10], is −328 ppm, a value which is close to the δ value of −306 ppm in the solid state. Despite some discrepancy between the C–Sn–C angle from X-ray data and the empirical estimation in solution, the similarity of the solid and solution state tin chemical shifts suggest no major change of the structure upon dissolution, the dimeric structure remaining apparently unaffected.

4. Supplementary information

Supplementary information in available from the Cambridge Crystallographic Data Center.

Acknowledgements

Financial support from UGC (New Delhi) and DST, Government of India, is gratefully acknowledged. We thank Professor Dr B. Mahieu for the Mössbauer spectra, the 'Ministère de l'Education Nationale du Luxembourg' (grant nos. BFR93/051, BPU96/130 and BPU97/138, M.K.), the 'Action Lions Vaincre le Cancer du Luxembourg' (M.K.), the Belgian 'Nationaal Fonds voor Wetenschappelijk Onderzoek' (N.F.W.O. grant no. G.0054.96, M.G.), and the Fund for Scientific Research Flanders (Belgium, grant no. G.0192.98, R.W., M.B.) for financial support.

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