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SYNTHESIS, CRYSTAL STRUCTURE, AND TRANSESTERIFICATION REACTION OF THE β-PROPOXYCARBONYLETHYLTIN TRICHLORIDE COMPLEX WITH DIBENZYLSULFOXIDE

Laijin Tian [a], Bin Zhao [a] & Feng Li [a]
[a] Department of Chemistry, Qufu Normal University, Qufu, Shandong, 273165, People's Republic of China

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SYNTHESIS, CRYSTAL STRUCTURE, AND TRANSESTERIFICATION REACTION OF THE β-PROPOXYCARBONYLETHYLTIN TRICHLORIDE COMPLEX WITH DIBENZYLSULFOXIDE

Laijin Tian,* Bin Zhao, and Feng Li

Department of Chemistry, Qufu Normal University, Qufu,
Shandong 273165, People's Republic of China

ABSTRACT

Synthesis, crystal structure and transesterification reaction of the β-propoxy-carbonylethyltin trichloride complex with dibenzylsulfoxide have been reported and a possible mechanism for the transesterification has been suggested. A series of the complexes of β-alkoxycarbonylethyltin trichloride with dibenzylsulfoxide have been synthesized and characterized by means of elemental analyses, IR, and ^{1}H NMR spectra.

INTRODUCTION

β-Alkoxycarbonylethyltin trichlorides, the so-called estertin chlorides, have attracted considerable attention ever since their syntheses were first reported (1–10). Their complexes with neutral and ligand anions have been investigated

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*Corresponding author. E-mail: zhaobinl@ji-public.sd.cninfo.net

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extensively (2–9). However, less attention has been paid to the transesterifications of their complexes with neutral ligands (10). In this paper, we report the synthesis, crystal structure and transesterification reaction of the β-propoxycarbonylethyltin trichloride complex with dibenzylsulfoxide.

RESULTS AND DISCUSSION

The synthesis and transesterification reaction of β-propoxycarbonylethyltin trichloride complex with dibenzylsulfoxide (L) may be represented by the following equations:

$$n\text{-PrOCOCH}_2\text{CH}_2\text{SnCl}_3 + \text{L} \rightarrow \underset{(\mathbf{1})}{n\text{-PrOCOCH}_2\text{CH}_2\text{SnCl}_3\cdot\text{L}}$$

$$\underset{(\mathbf{1})}{n\text{-PrOCOCH}_2\text{CH}_2\text{SnCl}_3\cdot\text{L}} + \text{ROH} \rightarrow \underset{(\mathbf{2})\text{-}(\mathbf{10})}{\text{ROCOCH}_2\text{CH}_2\text{SnCl}_3\cdot\text{L}} + n\text{-PrOH}$$

$$\text{R} = \text{Me}\,(\mathbf{2}),\ \text{Et}\,(\mathbf{3}),\ i\text{-Pr}\,(\mathbf{4}),\ \text{allyl}\,(\mathbf{5}),\ n\text{-Bu}\,(\mathbf{6}),\ i\text{-Bu}\,(\mathbf{7}),$$
$$n\text{-amyl}\,(\mathbf{8}),\ i\text{-amyl}\,(\mathbf{9}),\ \text{cyclohexyl}\,(\mathbf{10})$$

The physical and analytical data of these complexes are listed in Table 1. They are colorless crystals and air-stable and soluble in benzene and in common polar organic solvents, such as ethanol, chloroform, acetone and nitrobenzene, but insoluble in the saturated hydrocarbons as hexane and petroleum ether. The molar conductance values (4.8–7.2 S cm^2 mol^{-1}) of the complexes in nitrobenzene at room temperature suggest that they are non-electrolytes (11).

Infrared Spectra

The characteristic IR bands of the complexes are listed in Table 2. The $\nu(\text{C}{=}\text{O})$ values of the complexes (1634–1650 cm^{-1}) exhibit a remarkable red-shift from that of the free fatty acid ester (ca. 1740 cm^{-1}). This indicates that the carbonyl oxygen is coordinated intramolecularly to the tin atom (3,12). Compared with $\nu(\text{S}{=}\text{O})$ of the free dibenzylsulfoxide (1028 cm^{-1}), the $\nu(\text{S}{=}\text{O})$ frequency of these complexes shifts to lower frequency (945–962 cm^{-1}), confirming its coordination to the tin via oxygen (13). The $\text{Sn} \leftarrow \text{O}$ stretching vibration is expected in the region (14,15) below 400 cm^{-1} and cannot be obtained using KBr disks. In chloroform solution, the $\nu(\text{C}{=}\text{O})$ and $\nu(\text{S}{=}\text{O})$ bands of the complexes are in the 1650–1660 and 950–966 cm^{-1} regions, respectively. Thus, it may be seen that all the complexes are six-coordinated to tin in the solid and in solution. Their structures are suggested in Figure 1.

Table 1. Physical and Analytical Data for the Compounds

Compd. No.	Empirical Formula (Formula Weight)	Yield (%)	m.p. (°C)	Analysis, Found (Calculated)			
				C	H	S	Sn
(1)	$C_{20}H_{25}Cl_3O_3SSn$ (570.51)	87	125–6	42.28 (42.10)	4.32 (4.42)	5.49 (5.62)	20.57 (20.81)
(2)	$C_{18}H_{21}Cl_3O_3SSn$ (542.47)	89	150	39.87 (39.85)	3.76 (3.90)	5.94 (5.91)	21.84 (21.88)
(3)	$C_{19}H_{23}Cl_3O_3SSn$ (556.49)	79	123–4	41.03 (41.01)	4.06 (4.17)	5.68 (5.76)	21.28 (21.33)
(4)	$C_{20}H_{25}Cl_3O_3SSn$ (570.51)	63	146–8	42.08 (42.10)	4.19 (4.42)	5.57 (5.62)	20.68 (20.81)
(5)	$C_{20}H_{23}Cl_3O_3SSn$ (568.50)	84	113–4	42.13 (42.25)	4.06 (4.08)	5.66 (5.64)	20.62 (20.88)
(6)	$C_{21}H_{27}Cl_3O_3SSn$ (584.55)	80	117	43.09 (43.15)	4.47 (4.66)	5.33 (5.49)	20.42 (20.31)
(7)	$C_{21}H_{27}Cl_3O_3SSn$ (584.55)	76	120–1	42.96 (43.15)	4.48 (4.66)	5.51 (5.49)	20.05 (20.31)
(8)	$C_{22}H_{29}Cl_3O_3SSn$ (598.57)	78	124	44.08 (44.14)	4.89 (4.88)	5.32 (5.36)	19.77 (19.83)
(9)	$C_{22}H_{29}Cl_3O_3SSn$ (598.57)	76	98–9	43.85 (44.14)	4.63 (4.88)	5.35 (5.36)	19.52 (19.83)
(10)	$C_{23}H_{29}Cl_3O_3SSn$ (610.58)	68	172	45.15 (45.24)	4.76 (4.79)	5.30 (5.25)	19.08 (19.44)

Table 2. IR Spectral Data (cm^{-1}) for the Compounds[a]

Compd.	ν(C=O)	ν(C–O)	ν(S=O)	ν(Sn–C)
(1)	1638 vs	1226 s	960 vs	480 w
(2)	1649 vs	1236 s	959 vs	475 w
(3)	1642 vs	1248 s	945 vs	477 w
(4)	1636 vs	1231 s	962 vs	480 w
(5)	1639 vs	1222 s	945 vs	482 w
(6)	1640 vs	1221 s	950 vs	470 w
(7)	1648 vs	1244 s	950 vs	475 w
(8)	1634 vs	1232 s	948 vs	480 w
(9)	1634 vs	1230 s	946 vs	482 w
(**10**)	1650 vs	1233 s	950 vs	478 w

[a] vs = very strong, s = strong, w = weak.

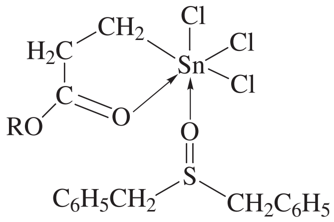

Figure 1. Suggested structures of the complexes.

^{1}H NMR Spectra

The ^{1}H NMR date of the β-alkoxycarbonylethyltin trichloride complexes with dibenzylsulfoxide in deuterochloroform are given in Table 3. These complexes exhibit two triplets centered at 2.64–2.74 and 1.78–1.84 ppm attributable to $COCH_2$ and CH_2Sn protons, respectively. The evidence of carbonyl group oxygen to tin coordination in these complexes is also obtained from the chemical shift values of the CH_nO ($n = 1$–3) protons of the alkoxyl group of the ester group. These proton signals show a downfield shift compared to those of the free fatty acid ester since coordination causes a deshielding effect on the CH_nO ($n = 1$–3) protons by the electron distribution (1,6,10). Compared with δ (CH_2S) of the free dibenzylsulfoxide (4.00 ppm), the protons in these complexes show a marked downfield shift ($\Delta\delta = \sim 0.10$ ppm) due to coordination of S=O with the tin atom thus decreasing the electron shielding of the CH_2S protons.

Table 3. ^{1}H NMR (δ, $CDCl_3$) Spectral Data for the Compounds[a]

Compd.	CH_nO ($n = 1 - 3$)	$COCH_2$	CH_2Sn	CH_2S
(1)	4.23 (t, 6.6)	2.70 (t, 7.5)	1.82 (t, 7.4)	4.11 (s)
(2)	3.88 (s)	2.70 (t, 7.6)	1.83 (t, 7.6)	4.10 (s)
(3)	4.33 (q, 7.0)	2.68 (t, 7.5)	1.81 (t, 7.5)	4.10 (s)
(4)	5.09 (m, 6.2)	2.65 (t, 7.2)	1.79 (t, 7.2)	4.10 (s)
(5)	4.74 (d, 5.6)	2.68 (t, 7.5)	1.78 (t, 7.0)	4.12 (s)
(6)	4.27 (t, 6.8)	2.69 (t, 7.8)	1.81 (t, 7.6)	4.11 (s)
(7)	4.02 (d, 6.4)	2.74 (t, 7.5)	1.84 (t, 7.8)	4.10 (s)
(8)	4.26 (t, 6.8)	2.69 (t, 7.5)	1.80 (t, 7.5)	4.11 (s)
(9)	4.31 (t, 6.8)	2.70 (t, 7.6)	1.83 (t, 7.6)	4.10 (s)
(10)	5.02 (m, 4.4)	2.64 (t, 7.2)	1.78(t, 7.2)	4.10 (s)

[a] The numbers in parentheses are the coupling constants in Hz.
d: Doublet; m: Multiplet; q = Quartet, s = Singlet, t = Triplet.

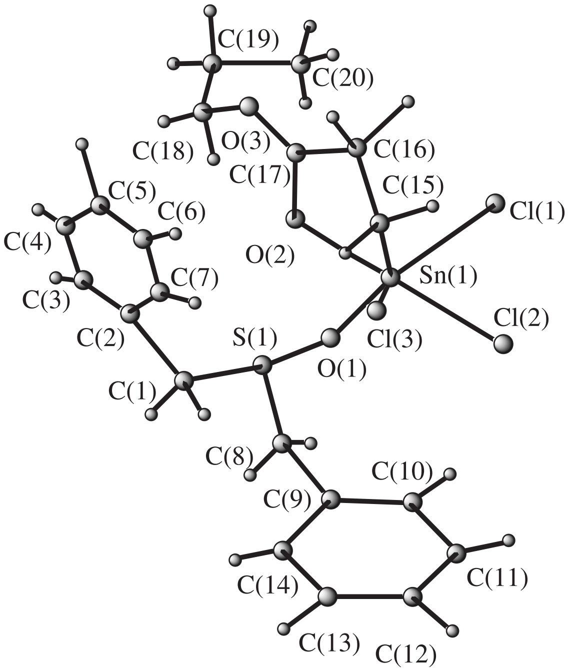

Figure 2. Molecular structure of (**1**).

Crystal Structure of *n*-PrOCOCH$_2$CH$_2$SnCl$_3$·L (1)

The molecular geometry and the atomic numbering scheme for **(1)** are shown in Figure 2. The selected bond lengths and bond angles are given in Table 4.

The complex exists as a discrete molecule which contains a five-membered chelate ring, formed via carbonyl oxygen to tin coordination. The tin atom is six-coordinated with the coordinating atoms Cl(1), Cl(2), Cl(3), O(1), O(2), and C(15) in a distorted octahedral arrangement. The bond angles Cl(1)–Sn(1)–O(1), Cl(2)–Sn(1)–O(2), and Cl(3)–Sn(1)–C(15) are, respectively, 170.5(1), 175.6(1), and 158.8(2)°, which are very close to those of reported for ROCOCH$_2$CH$_2$SnCl$_3$·D (D is an oxygen donor) (8,16). The Sn(1)–O(2) distance (2.343(7) Å) in complex **(1)** is comparable with the intramolecular Sn–O distances in other six-coordinated ROCOCH$_2$CH$_2$Sn compounds (Sn–O, 2.33–2.44 Å) (8,9,16). As expected, the Sn(1)–O(1) (2.188(4) Å) and Sn(1)–O(2) distances are both longer than the sum (2.06 Å) of the covalent radii of tin and oxygen and shorter than the sum (3.70 Å) of the Van der Waals' radii. The bond length Sn(1)–O(2) is longer than that

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Table 4. Selected Bond Lengths (Å) and Bond Angle (°) for (**1**)

Bond Length			
Sn(1)–Cl(1)	2.452(3)	C(1)–C(2)	1.504(9)
Sn(1)–Cl(2)	2.387(3)	C(8)–C(9)	1.494(9)
Sn(1)–Cl(3)	2.368(3)	C(15)–C(16)	1.513(11)
Sn(1)–O(1)	2.188(4)	C(16)–(17)	1.92(12)
Sn(1)–O(2)	2.343(7)	C(17)–O(2)	1.189(9)
Sn(1)–C(15)	2.129(7)	C(17)–O(3)	1.309(10)
S(1)–0(1)	1.521(4)	O(3)–C(18)	1.440(11)
S(1)–C(1)	1.809(7)	C(18)–C(19)	1.493(15)
S(1)–C(8)	1.835(6)	C(19)–C(20)	1.346(17)
Bond Angles			
Cl(1)–Sn(1)–Cl(2)	96.2(1)	Sn(1)–O(1)–S(1)	123.9(3)
Cl(1)–Sn(1)–Cl(3)	88.8(1)	Sn(1)–C(15)–C(16)	108.7(5)
Cl(1)–Sn(1)–(1)	170.5(1)	Sn(1)–O(2)–C(17)	110.8(5)
Cl(1)–Sn(1)–O(2)	88.2(1)	O(1)–S(1)–C(1)	101.8(3)
Cl(1)–Sn(1)–C(15)	94.6(2)	O(1)–S(1)–C(8)	105.6(3)
Cl(2)–Sn(1)–Cl(3)	96.5(1)	C(1)–S(1)–C(8)	100.8(3)
Cl(2)–Sn(1)–O(1)	90.8(1)	S(1)–C(1)–C(2)	108.1(4)
Cl(2)–Sn(1)–O(2)	175.6(1)	S(1)–C(8)–C(9)	114.5(5)
Cl(2)–Sn(1)–C(15)	103.9(2)	C(15)–C(16)–C(17)	113.4(6)
Cl(3)–Sn(1)–O(1)	84.1(1)	O(2)–C(17)–C(16)	122.2(7)
Cl(3)–Sn(1)–O(2)	83.11(1)	O(2)–C(17)–O(3)	125.2(8)
Cl(3)–Sn(1)–(1)–C(15)	158.8(2)	O(3)–C(17)–C(16)	112.6(7)
O(1)–Sn(1)–O(2)	84.8(2)	O(3)–C(18)–C(19)	116.5(9)
O(1)–Sn(1)–C(15)	89.9(3)	C(17)–O(3)–C(18)	117.8(6)
O(2)–Sn(1)–C(15)	76.1(3)	C(18)–C(19)–C(20)	110.9(10)

of Sn(1)–O(1), and shows that carbonyl O(2) coordination to tin is weaker than sulfinyl O(1) coordination.

Transesterification of (1)

When the β-propoxycarbonylethyltin trichloride complex with dibenzylsulfoxide is refluxed in an alcohol, it is readily converted into the corresponding alkoxycarbonyl analogs with yields of 63–89%.We attribute the occurrence of this novel transesterification to intramolecular Lewis acid catalysis by the electrophilic $SnCl_3$ group. The coordination of the ester carbonyl oxygen to the tin atom polarized the carbonyl bond, thereby making the carbonyl carbon more susceptible to attack by a nucleophilic reagent such as an alcohol. A possible mechanism for the transesterification is suggested in the Figure 3. The transesterification

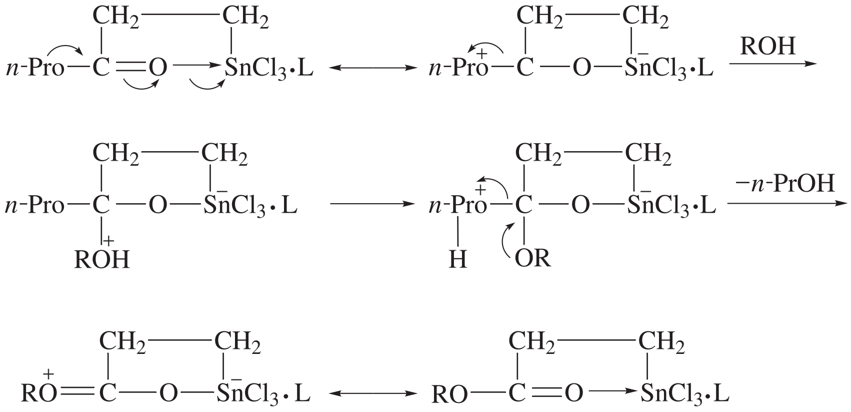

Figure 3. Proposed mechanism for the transesterification of **(1).**

provided a convenient way for preparing the β-alkoxycarbonylethyltin trichloride complexes.

EXPERIMENTAL

Materials and Physical Measurements

All chemicals were of reagent grade and were used without further purification. β-Propoxycarbonylethyltin trichloride and dibenzylsulfoxide were prepared according to leterature methods (6,17). Carbon, hydrogen, nitrogen, and sulfur analyses were obtained using a Perkin Elmer 2400 Series II elemental analyser, and tin was determined titrimetrically as Sn^{2+}. Melting points were measured on a TX4-100A microscopic melting point apparatus. IR spectra were recorded on an Alpha Centauri FT-IR spectrophotometer using KBr discs in the range 4000–400 cm^{-1}. ^1H NMR spectral data were collected using a Bruker AC-80 spectrometer or a Varian Unity-400 spectrometer with $CDCl_3$ as solvent and TMS as internal standard.

Synthesis of n-$PrOCOCH_2CH_2SnCl_3 \cdot L$ (1)

A solution of dibenzylsulfoxide (L) (1.15 g, 5 m mol) dissolved in benzene (30 mL) was added dropwise to a stirred solution of β-propoxycarbonylethyltintrichloride (1.70 g, 5 m mol) in benzene(30 mL), and the mixture was refluxed for an hour and concentrated to ca. 20 mL by evaporation under reduced pressure. The colorless crystals of **(1)** formed in the solution were filtered and recrystallized from benzene–petroleum ether (b.p. 60–90°C) (1:1, v/v) and dried in vacuo. Yield is 2.48 g.

Transesterification of n-PrOCOCH$_2$CH$_2$SnCl$_3$·L(1)

A solution of (**1**) (2.85 g, 5 m mol) dissolved in 60 mL ROH was refluxed for 2 h, and then concentrated slowly to ca. 15 mL by evaporation under reduced pressure. The resultant white solid was recrystallized from benzene–petroleum ether (b.p. 60–90°C) (1:1, v/v) and dried in vacuo. The yields and melting points of the obtained compounds are shown in Table 1.

Crystal Structure Determination for (1)

A colorless crystal with dimensions 0.15 × 0.26 × 0.58 mm was used for the single crystal determination. Data were collected on a Nicolet R3m/E four-circle diffractometer with graphite monochromated Mo K_α radiation (0.71073 Å), using the $\theta/2\theta$ scan technique ($2.2 \leq \theta \leq 25.0°$). 2516 independent reflections were measured of which 2250 had $I > 2.0\sigma(I)$ and were considered to be observed. The data were corrected for Lorentz and polarization factors but not for absorption. The crystal parameters were as follows: $C_{20}H_{25}Cl_3O_3SSn$, $M = 570.5$, orthorhombic, space group $P2_12_12_1$, $a = 10.62(1)$, $b = 14.27(2)$, $c = 16.35(1)$ Å, $V = 2477.8(3)$ Å^3, $Z = 4$, $D_c = 1.44$ Mgm^{-3}, $F(000) = 1084$, $\mu(\mathrm{Mo}K_\alpha) = 1.451$ mm^{-1}, $\lambda = 0.71073$ Å, $T = 298(2)$ K.

The structure was solved by the SHELXS-86 program (18) and full-matrix least-squares refinement on F^2 was performed using SHELXL-93 (19). Anisotropic displacement parameters were employed for non-H atoms and H-atoms were included in the model at their calculated positions and refined isotropically. The refinement converged to a final $R = 0.0336$, $wR = 0.0382$ and goodness-of-fit = 1.04. The maximum and mnimum residual electron density peaks in the final difference map were 0.552 and −0.470 e Å^{-3}, respectively. All calculations were performed on an Eclipse S/140 computer using the SHELXTL program (20).

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