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## SYNTHESIS AND CHARACTERIZATION OF SOME ORGANOTIN(IV) COMPLEXES OF $\alpha\mbox{-}BENZOIN$ OXIME

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#### SYNTH. REACT. INORG. MET.-ORG. CHEM., 31(1), 157-165 (2001)

# SYNTHESIS AND CHARACTERIZATION OF SOME ORGANOTIN(IV) COMPLEXES OF $\alpha$ -BENZOIN OXIME

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#### ABSTRACT

Some di- and triorganotin(IV) complexes of  $\alpha$ -benzoin oxime of the general formulae R<sub>3</sub>SnL, R<sub>2</sub>SnCl<sub>2-n</sub>L<sub>n</sub>, and R<sub>2</sub>SnL have been synthesized by the reactions of the corresponding di- and triorganotin(IV) chlorides with the sodium salt of  $\alpha$ -benzoin oxime in the desired molar ratios. All of the compounds have been characterized by elemental analyses and spectral (IR, <sup>1</sup>H, <sup>13</sup>C, and <sup>119</sup>Sn NMR) studies.

#### **INTRODUCTION**

Complexes of organotin(IV) halides with N, O, and S donor ligands have received much attention during the last few years (1–4). The considerable developments over recent decades in the use of organotin compounds as reagents (5) or intermediates in organic synthesis (6,7) prompted the preparation of many new organotin compounds. Certain penta- and hexa-coordinated organotin(IV) complexes (8), a number of oximates (9), and complexes of titanium with  $\alpha$ -benzoin

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oxime (10) have been reported in the literature. A recent review deals with complexes of oximes and oximato species (11). Organotin compounds having the general formula  $R_n SnCl_{4-n}$  are biologically active (12–16) and have been used in carbohydrate and nucleoside chemistry (17). Thus, in view of the synthetic and biological importance of organotin(IV) compounds and in continuation of our recent report on organotin(IV) (18–21), we report here the synthesis and characterization of some new organotin(IV) complexes of  $\alpha$ -benzoin oxime.

#### **RESULTS AND DISCUSSION**

 $\alpha$ -Benzoin oxime was synthesized by the condensation of benzoin and hydroxylamine hydrochloride in 1:1 molar ratio (22,23). Triorganotin(IV) and diorganotin(IV) derivatives of  $\alpha$ -benzoin oxime with the general formulae R<sub>3</sub>SnL, R<sub>2</sub>SnL<sub>n</sub>Cl<sub>2-n</sub>, and R<sub>2</sub>SnL have been synthesized by the reaction of the corresponding triorganotin(IV) and diorganotin(IV) chlorides with the conjugate base of the ligand H<sub>2</sub>L (prepared in situ) by using sodium hydride in 1:1 and 1:2 molar ratios, respectively.



Similarly, diorganotin(IV) complexes of the type R<sub>2</sub>SnL have been synthesized from the dianion of the ligand.

All of these new complexes were characterized on the basis of satisfactory elemental analyses and spectral data. These compounds are more or less soluble

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in common organic and coordinating solvents like  $C_6H_6$ ,  $CHCl_3$ ,  $CH_2Cl_2$ ,  $CCl_4$ , DMF, and DMSO. Molecular weight determinations in dilute  $CHCl_3$  show the monomeric nature of these complexes.

#### **Infrared Spectra**

The IR spectral data of the complexes have been recorded in Nujol in the range of 4000–200 cm<sup>-1</sup>. The IR spectrum of the ligand (H<sub>2</sub>L) shows bands in the regions 3360–3240, 3482, and 1590 cm<sup>-1</sup> assignable to  $\nu$ (OH) of the CH–OH group,  $\nu$ (OH) of the =N–OH group and  $\nu$ (C=N), respectively. The disappearance of absorption bands and signals corresponding to  $\nu$ (OH) of the >CH–OH and =N–OH groups in the IR spectra of the complexes (7) and (8) suggest a cyclic structure. This fact is further supported by the appearance of new bands in the region 580–440 cm<sup>-1</sup>, which are ascribed to Sn–C and Sn–O stretching modes (24–26). A strong band at 1590 cm<sup>-1</sup> assignable to  $\nu$ (C=N) in the free ligand remains as such in the spectra of all the complexes, clearly indicating the noninvolvement of C=N in complexation (24). In compounds (1)–(6) that have been synthesized from the monoanion, the >CHOH group takes part in complexation, which is further supported by the shift of the  $\nu$ (OH) frequency to lower wave number (ca. 3130 cm<sup>-1</sup>) in the spectra of the complexes.

#### Nuclear Magnetic Resonance Spectra

The <sup>1</sup>H, <sup>13</sup>C, and <sup>119</sup>Sn spectra of the complexes have been recorded in CDCl<sub>3</sub> and DMSO-d<sub>6</sub> using TMS as an internal standard and tetramethyltin as external standard respectively, and the observed chemical shifts are tabulated in Table 1. The <sup>1</sup>H NMR spectra of the complexes exhibit the usual features. A comparison of the <sup>1</sup>H NMR spectral data of the free ligand with the complexes exhibits the disappearance of the signal at  $\delta$ 10.6 ppm in compounds (1)–(6),

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**Table 1.** Spectral Data of the Complexes of Organotin( IV) Chloride with  $\alpha$ -Benzoin Oxime

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Compound		IR	$(cm^{-1})$		Z	IMR 8, ppm)	
No.	$\nu$ (C=N)	$\nu(OH)$	$\nu(Sn-O)$	$\nu(Sn \leftarrow Cl)$	H	13C	<sup>119</sup> Sn
(1)	1588 s	3164 m	578 sh	I	7.56–7.11 (m, 26H, Ph and OH);	126.8–142.8 (Ph), 86.5 (C–O),	-181.2
					3.12 (s, 1H, CH).	156.4 (C=N).	
(2)	1591 s	3186 m	576 m		7.40–7.08 (m, 11H, Ph and OH);	126.4–143.2 (Ph), 86.1 (C–O),	-182.4
					3.14 (s, 1H, CH);	156.4 (C=N), 9.46–27.2 ( <i>n</i> -Bu).	
					0.25–1.84 (m, 27H, <i>n</i> -C <sub>4</sub> H <sub>9</sub> ).		
(3)	1590 sh	3190  s	574 s	284 m	7.45–7.11 (m, 11H, Ph and OH);	126.8–142.5 (Ph), 86.3 (C–O),	-244.6
					3.08 (s, 1H, CH);	155.8 (C=N), 6.46, 6.42 (Me).	
					0.82 (s, 6H, CH <sub>3</sub> ).		
(4)	1585 s	3180  m	580 s	288 m	7.42–7.06 (m, 11H, Ph and OH);	126.1–142.7 (Ph), 86.9 (C–O),	-254.2
					3.04 (s, 1H, CH);	156.4 (C=N), 9.48–27.2 ( <i>n</i> -Bu).	
					0.38–1.71 (m, 18H, <i>n</i> -C <sub>4</sub> H <sub>9</sub> ).		
(5)	$1592  \mathrm{sh}$	3185 s	581 m		7.58–7.12 (m, 22H, Ph and OH);	126.5–142.8 (Ph), 86.2 (C–O),	-342.8
					3.10 (s, 2H, 2CH);	157.2 (C=N), 6.39, 6.34 (Me).	
					0.78 (s, 6H, CH <sub>3</sub> ).		
(9)	1585 s	3188 m	578 m		7.60–7.12 (m, 22H, Ph and OH);	126.2–143.0 (Ph), 86.3 (C–O),	-352.6
					3.06 (s, 2H, 2CH);	157.1 (C=N), 9.64–27.6 ( <i>n</i> -Bu).	
					0.28–1.68 (m, 18H, <i>n</i> -C <sub>4</sub> H <sub>9</sub> ).		
(2)	$1590  \mathrm{sh}$		584 s		7.56–7.14 (m, 10H, Ph);	126.7–142.7 (Ph), 86.2 (C–O).	135.2
					0.80 (s, 6H, CH <sub>3</sub> ).	157.1 (C=N), 6.38, 6.32 (Me)	
(8)	1591 s		582 m		7.58–7.14 (m, 10H, Ph);	126.9–143.1 (Ph), 86.2 (C–O)	132.8
					0.40–1.56 (m, 18H, <i>n</i> -C <sub>4</sub> H <sub>9</sub> ).	157.0 (C=N), 9.67–27.68 ( <i>n</i> -Bu).	

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suggesting the deprotonation of =N–OH and complexation with tin metal (25,26). The >CH–OH group in the ligand appears at  $\delta$  5.61 ppm, shifts downfield (to  $\delta$  6.20 ppm) in the complexes, indicating its deshielding and coordination through the oxygen of the >CH–OH group to tin metal in compounds (1)–(6). The resonance due to the phenyl moiety at  $\delta$  7.32–7.15 ppm remains almost unaffected in all the complexes. The resonances due to butyl tin protons are observed in the region  $\delta$  0.70–1.16 ppm (J = 8 Hz), which is due to terminal methyl protons of butyl group. The methyl protons of dimethyl tin(IV) derivatives appear as a sharp singlet at  $\delta$  0.88 ppm. The coupling constants provide valuable information about the hybridization state of tin (21,24). The <sup>2</sup>J(<sup>119</sup>Sn–<sup>1</sup>H) values in the range 54–68 Hz are indicative of four-coordinate tin in compounds (7) and (8). The tin–proton coupling constants, <sup>2</sup>J(<sup>119</sup>Sn–<sup>1</sup>H) in compounds (1)–(6) have been found at  $87 \pm 1$  Hz, which is indicative of a higher coordination number (five and six) of tin (24), suggesting the bidentate behaviour of the ligand.

The <sup>13</sup>C NMR spectra of the complexes show the expected number of signals. The  $\delta$  10–15 ppm downfield signal shifts of carbons attached to oxygen indicate oxygen coordination with the tin atom. The R groups attached to tin display a single resonance for chemically equivalent carbons. The <sup>1</sup>J(<sup>119</sup>Sn–<sup>13</sup>C) values occur in the range for four-(7), five- and six-coordinated tin (27,28).

The <sup>119</sup>Sn NMR chemical shifts of these complexes have been observed in the range  $\delta$  132.8 to –352.6 ppm and suggest that these compounds contain four-(7), five- and six-coordinated tin (28).

#### EXPERIMENTAL

All reactions were carried out under a nitrogen atmosphere and analytical grade chemicals were used. Solvents used were purified and dried according to standard procedures (29). The progress of the reaction and purity was checked by TLC on silica gel. The ligand was synthesized by the literature method (22,23). Elemental anslyses were performed by the Central Drug Research Institute, Lucknow. Tin and chloride in the complexes were determined gravimetrically or messenger's method, respectively (30).

IR spectra were recorded on a Perkin–Elmer model 377 spectrometer in the range 4000–200 cm<sup>-1</sup>. NMR spectra were recorded on a Bruker AC200 instrument at 200, 74, and 50.32 MHz for <sup>1</sup>H, <sup>119</sup>Sn, and <sup>13</sup>C, respectively at the National Chemical Laboratory, Pune. Chemical shifts are in ppm and relative to TMS for <sup>1</sup>H and <sup>13</sup>C, and tetramethyltin for <sup>119</sup>Sn as an internal standard, respectively.

α-Benzoin oxime: m.p. 151°C
<sup>1</sup>H NMR (CDCl<sub>3</sub>): δ 10.6 (s, 1H, N–OH); 7.32–7.15 (m, 10H, Ph); 5.61 (s, 1H, OH); 2.95 (s, 1H, CH).

	Reactants in	Gram (mr	nol)		Droduct			Mol			2 I 20	(Felo	
Compound	Organotin( IV)			Molar	Physical	Yield	M.p	Wt.		Analysis:	% Found	(Lalcu.)	
No.	Chlorides	Ligand	NaH	Ratio	State	(%)	()°C)	F/(C)	С	Н	Z	$\mathbf{Sn}$	CI
(1)	Ph <sub>3</sub> SnCl 0.8030 (2.08)	0.4735 (2.08)	0.050 (2.08)	1:1:1	C <sub>32</sub> H <sub>27</sub> NO <sub>2</sub> Sn Ph <sub>3</sub> SnHL white solid	52	225–230	562 (576)	66.52 (66.67)	4.38 (4.69)	2.37 (2.43)	20.42 (20.60)	
(2)	<i>n</i> -Bu <sub>3</sub> SnCl 0.6781 (2.08)	0.4735 (2.08)	0.050 (2.08)	1:1:1	C <sub>26</sub> H <sub>39</sub> NO <sub>2</sub> Sn <i>n</i> -Bu <sub>3</sub> SnHL white solid	50	128–130	508 (516)	60.37 (60.47)	7.48 (7.56)	2.66 (2.71)	22.88 (23.00)	
(3)	Me <sub>2</sub> SnCl <sub>2</sub> 0.7780 (3.54)	0.8049 (3.54)	0.085 (3.54)	1:1:1	C <sub>16</sub> H <sub>18</sub> NO <sub>2</sub> SnCl Me <sub>2</sub> SnHLCl vellow solid	56	176–180	506 (470)	46.68 (46.83)	4.28 (4.39)	3.35 (3.41)	28.78 (28.95)	8.42 (8.66)
(4)	<i>n</i> -Bu <sub>2</sub> SnCl <sub>2</sub> 0.6330 (2.08)	0.4735 (2.08)	0.050 (2.08)	1:1:1	C <sub>22</sub> H <sub>30</sub> NO <sub>2</sub> SnCl n-Bu <sub>2</sub> SnHLCl white solid	51	96-98	472 (495)	53.11 (53.33)	6.11 (6.06)	2.70 (2.83)	23.72 (23.98)	7.08 (7.17)
(5)	Me <sub>2</sub> SnCl <sub>2</sub> 0.3982 (1.81)	0.8238 (3.62)	0.087 (3.62)	1:2:2	C <sub>30</sub> H <sub>30</sub> N <sub>2</sub> O <sub>4</sub> Sn Me <sub>2</sub> Sn(HL) <sub>2</sub> ninkinsh hrown	54	174–176	588 (601)	59.62 (59.90)	4.87 (4.99)	4.56 (4.66)	19.58 (19.75)	
(9)	<i>n</i> -Bu <sub>2</sub> SnCl <sub>2</sub> 0.3165 (1.04)	0.4735 (2.08)	0.050 (2.08)	1:2:2	$C_{36}H_{42}N_2O_4Sn$ $n-Bu_2Sn(HL)_2$ reddish hrown	48	110–115	671 (685)	62.98 (63.07)	6.05 (6.13)	4.20 (4.09)	17.15 (17.33)	
( <b>1</b> )	Me <sub>2</sub> SnCl <sub>2</sub> 0.4576 (2.08)	0.4735 (2.08)	0.100 (4.16)	1:1:2	$C_{16}H_{17}NO_2Sn$ $Me_2SnL_2$	62	203–205	362 (374)	51.24 (51.34)	4.42 (4.55)	3.64 (3.74)	31.50 (31.74)	
(8)	<i>n</i> -Bu <sub>2</sub> SnCl <sub>2</sub> 0.6330 (2.08)	0.4735 (2.08)	0.100 (4.16)	1:1:2	$C_{22}H_{29}NO_2Sn$ $n-Bu_2SnL_2$ cream solid	55	80-82	470 (458)	57.48 (57.64)	6.24 (6.33)	3.00 (3.06)	25.72 (25.91)	l







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IR (Nujol, cm<sup>-1</sup>):  $\nu$ (C=N) 1590,  $\nu$ (OH) 3360–3240,  $\nu$ (N–OH) 3482. Analysis found: C, 73.84; H, 5.54; N, 5.96. Calcd. for C<sub>14</sub> H<sub>13</sub> NO<sub>2</sub> (227): C, 74.01%; H, 5.73%; N, 6.17%.

#### Reaction Between Triphenyltin(IV) Chloride and the Sodium Salt of α-Benzoin Oxime in 1:1 Molar Ratio

A quantity of 0.050 g (2.08 mmol) of sodium hydride and 15 mL of isopropanol were placed in a two neck round bottom flask fitted with a dried and cooled water condenser and a dropping funnel. The mixture was stirred for about 30 min till a clear solution of sodium isopropoxide was obtained. Then 0.474 g (2.08 mmol) of  $\alpha$ -benzoin oxime was added to the sodium isopropoxide solution. The mixture was refluxed for 2 h. The light yellow solution changed to brown after 2 h. Then a solution of 0.803 g (2.08 mmol) of triphenyltin(IV) chloride in benzene was added slowly by a dropping funnel at room temperature. The colour of the mixture changed to milky white. It was further refluxed for 2–3 h to ensure the completion of the reaction, checked by TLC. The desired product was isolated by filtering off the precipitated sodium chloride and evaporation of the solvent under reduced pressure. The product was further purified by crystallization from benzene–petroleum ether (40–60°C) mixture (20 mL each).

All other organotin(IV) derivatives of  $\alpha$ -benzoin oxime were synthesized analogously. The pertinent data for this and other derivatives are listed in Table 2.

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