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Monothiophosphorylation of Diorganotin Dichlorides. Synthesis and Characterization

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SYNTHESIS AND REACTIVITY IN INORGANIC AND METAL-ORGANIC CHEMISTRY Vol. 33, No. 2, pp. 183–194, 2003

Monothiophosphorylation of Diorganotin Dichlorides. Synthesis and Characterization

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ABSTRACT

Reactions of diorganotin dichlorides with ammonium dialkyl monothiophosphates in refluxing benzene in 1:2 molar ratio yield diorganotin bis(dialkyl monothiophosphate). These compounds are soluble in common organic solvents. Molecular weight determinations show that these compounds are associated in solution and the degree of association is concentration dependent. The new compounds have been characterized by elemental analyses as well as spectroscopic (IR, ¹H, ¹³C, ³¹P and ¹¹⁹Sn) studies. On the basis of these data, chelate structures have been proposed.

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INTRODUCTION

The coordination chemistry of tin(IV) is still an area of interest for investigation, and much work has been done in the chemistry of tin(IV) and organotin(IV) dithiophosphate derivatives. However, comparatively less attention has been paid to the corresponding monothiophosphate derivatives.^[1-13] In addition to its biological activity, monothiophosphate is capable of a variety of bonding modes (either through sulfur or oxygen). We report in the present paper the synthesis and characterization of some diorganotin bis(dialkyl monothiophosphates).

RESULTS AND DISCUSSION

The reactions of ammonium salts of dialkyl monothiophosphates with diorganotin chlorides in refluxing benzene in 1:2 molar ratio yield diorganotin bis(dialkyl monothiophosphates).

R = Me and R' = Et (1), Pr-*i* (2), Pr-*n* (3), Bu-*n* (4), Bu-*i* (5) and Ph (6); R = Bu and R' = Ph (7) and Bu-*n* (8); R = Ph and R' = Bu-*n* (9) and Pr-*i* (10).

These reactions occur readily at room temperature but to ensure completion, the reaction mixtures were refluxed for ~ 3 hours and the formed ammonium chloride was removed by filtration. The resulting compounds are colorless viscous liquids and are soluble in common organic solvents (Table 1). Molecular weight determinations show that these compounds are associated in chloroform and the degree of association is concentration dependent. Thus, Me₂Sn[O(S)P(OPr-*i*)₂]₂ is monomer in 2.1% (w/w) solution but becomes a dimer on increasing the concentration to 18.3%.

IR Spectra

Characteristic bands in the IR spectra of the present diorganotin bis(dialkyl monothiophosphates) have been assigned by comparison with the spectra of triorganotin derivatives (Table 2). The bands present in the regions 960–1070 cm⁻¹ and 710–790 cm⁻¹ are assigned to v((P)–O–C) and v(P–O–(C)) stretching vibrations, respectively.^[14] The most important feature is the absence of any peak due to v(P=O) in the region 1150–1250 cm⁻¹. All the spectra show a band of medium to strong intensity at

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Diorganotin bis(Dialkyl Monothiophosphates)

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	Re	actants (g)	4 4	ý	Analvsis %	found/(calc.)	
		ò				~ ~	
S. no.	$\begin{array}{l} R_2 SnCl_2 \\ (R=) \end{array}$	$(R'O)_2P(S)ONH_4$ (R' =)	Products (g)	Yield (%)	Tin	Sulfur	Mol. weight % found/(calc.)
E	Me (2.12)	Et (3.6)	Me ₂ Sn[OSP(OEt) ₂] ₂ (4.50)	86	24.61 (24.42)	13.48 (13.14)	476.5 (486.7)
(5)	Me (1.09)	Pr- <i>i</i> (2.13)	Me ₂ Sn[OSP(OPr- <i>i</i>) ₂] ₂	94	21.72 (21.90)	11.53 (11.79)	556.3 (542.7)
(3)	Me (1.17)	Pr- <i>n</i> (2.27)	$Me_{2}Sn[OSP(OPr-n)_{2}]_{2}$	96	21.64 (21.90)	12.04 (11.79)	549.2 (542.7)
(Me (1.21)	Bu-n (2.367)	$Me_2Sn[OSP(OBu-n)_2]_2$ (2.91)	06	20.13 (19.85)	10.29 (10.68)	605.3 (598.7)
(2)	Me (1.04)	Bu- <i>i</i> (2.29)	Me ₂ Sn[OSP(OBu- <i>i</i>) ₂] ₂ (2.56)	92	19.64 (19.85)	$10.84 \ (10.68)$	589.7 (598.7)
9	Me (1.14)	Ph (2.93)	Me ₂ Sn[OSP(OPh) ₂] ₂ (3.34)	95	17.23 (17.51)	9.77 (9.42)	689.5 (678.7)
6	Bu (1.25)	Ph (2.86)	$Bu_2Sn[OSP(OPh)_2]_2$ (3.60)	93	15.75 (15.50)	8.40 (8.34)	773.5 (766.7)
8	Bu (1.34)	Bu-n (2.63)	$Bu_2Sn[OSP(OBu-n)_2]_2$ (3.02)	89	18.78 (19.00)	10.63 (10.22)	630.9 (625.7)
6	Ph (1.53)	Bu-n (2.16)	$Ph_2Sn[OSP(OBu-n)_2]_2$ (2.95)	92	16.81 (16.45)	8.46 (8.85)	731.8 (722.7)
(10)	Ph (1.28)	Pr- <i>i</i> (1.59)	Ph ₂ Sn[OSP(OPr- <i>i</i>) ₂] ₂ 2.39	96	17.44 (17.72)	9.71 (9.54)	663.6 (670.7)

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^aAll compounds are colorless viscous liquids.

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Table 2. Some relevant IR spectral data^a (cm⁻¹) for diorganotin(IV) dialkyl mono-thiophosphates.

S. no.	Compound	$\nu[(P)-O-C]$	$\nu[P-O-(C)]$	v[P=S]
(1)	Me ₂ Sn[OSP(OEt) ₂] ₂	960 s	770 s	590 m
(2)	$Me_2Sn[OSP(OPr-i)_2]_2$	980 s	760 s	615 m
(3)	$Me_2Sn[OSP(OPr-n)_2]_2$	970 s	750 s	595 m
(4)	$Me_2Sn[OSP(OBu-n)_2]_2$	1010 s	780 s	590 m
(5)	$Me_2Sn[OSP(OBu-i)_2]_2$	1010 s	790 s	580 m
(6)	Me ₂ Sn[OSP(OPh) ₂] ₂	1070 s	715 s	670 m
(7)	$Bu_2Sn[OSP(OPh)_2]_2$	1070 s	710 s	690 m
(8)	$Bu_2Sn[OSP(OBu-n)_2]_2$	1010 s	790 s	590 m
(9)	$Ph_2Sn[OSP(OBu-n)_2]_2$	1010 s	780 s	680 m
(10)	$Ph_2Sn[OSP(OPr-i)_2]_2$	980 s	790 s	620 m

as = sharp and m = medium.

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 $\sim 600 \text{ cm}^{-1}$ (at $\sim 685 \text{ cm}^{-1}$ in diphenyl monothiophosphate derivatives) which we tentatively assign to v(P=S). The position of this band does not shed any light on the bonding pattern of the monothiophosphate ligands.^[13]

¹H NMR Spectra

The ¹H NMR spectra (Table 3) show the expected peak patterns and chemical shifts. The noteworthy observation is that the ²J(¹H-¹¹⁹Sn) values for dimethlyltin bis(diisopropyl and diphenyl monothiophosphate) derivatives are 80 and 78 Hz, respectively, and these coupling constant values appear to be concentration independent. These values are higher than those for penta-coordinated triorganotin monothiophosphate derivatives (~68), thus supporting to the presence of hexa-coordinated tin in the diorganotin derivatives.^[13]

¹³C NMR Spectra

The ¹³C NMR spectral data for these compounds are given in Table 4. The chemical shift values of diorganotin bis(dialkyl monothiophosphates) ($\sim 10\%$ solution in CHCl₃) are in the expected range. The alkoxy carbons show 2- and 3-bond coupling with the phosphorus atoms. The [¹J(¹³C-¹¹⁹Sn] values for Me₂Sn[O(S)P(OPr-*i*)₂]₂ and Me₂Sn[O(S)P(OPh)₂]₂ are 572.40 and 566.6 Hz, respectively, and correspond to hexa-coordinated

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	<i>Table 3.</i> ¹ H, ³¹ P, ¹¹⁹ Sn	NMR spectral data of diorge	anotin(IV) dialkyl monothiophosphates	in CDCl ₃ .	
S. no.	Compound	¹ J(¹³ C- ¹¹⁹ Sn) (Hz)	¹ H NMR	$^{31}\mathrm{p}$	119 Sn
(2)	Me ₂ Sn[OSP(OPr- <i>i</i>) ₂] ₂	80	1.28 (s, 6H, MeSn), 1 32 (d 1=7 Hz 24H Me)	50.9	- 130.5
(4)	Me ₂ Sn[OSP(OBu- <i>n</i>) ₂] ₂	79	4.35–4.88 (m, 4H, –CHO) 1.12 (s, 6H, MeSn),	47.8	- 87.4
			0.92 (t, J = 7Hz, 12H, Me), 1.23-1.84 (m, 16H, -CH ₂),		
			3.74-4.22 (m, 8H, -CH ₂ O)		
(9)	Me ₂ Sn[OSP(OPh) ₂] ₂	78	0.88 (s, 6H, MeSn),	43.6	-85.8
			6.90-7.52 (m, 20H, OPh)		
6	$Bu_2Sn[OSP(OPh)_2]_2$	80	0.81 (t, J=7 Hz, 6H, Me),	42.5	-97.8
			0.96–1.67 (m, 12H, –CH ₂),		
			6.99–7.52 (m, 20H, OPh)		
(6)	$Ph_2Sn[OSP(OBu-n)_2]_2$	I	0.92 (t, J=7 Hz, 12H, Me),	50.8	-305.1
			$1.05 - 1.76$ (m, 16H, $-CH_2$),		
			3.6-4.04 (m, 8H, -CH ₂ O),		
			7.08-8.00 (m, 10H, Ph)		
(10)	$Ph_2Sn[OSP(OPr-i)_2]_2$	I	1.31 (d, $J = 7$ Hz, 24 H, Me),	46.8	-302.0
			4.30-4.85 (m, 4H, -CHO),		
			7.08-8.1 (m, 10H, Ph)		

Diorganotin bis(Dialkyl Monothiophosphates)

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Table 4. Some ¹³C NMR spectral data of diorganotin(IV) dialkyl monothiophosphates in CHCl₃.

				R_2Sn c	arbons		Chemical shift (ô nnm)
S. no.	Compound	¹ J(¹³ C- ¹¹⁹ Sn) (Hz)	C(1)	C(2)	C(3)	C(4)	or carbons
(2)	$Me_2Sn[O(S)P(OPr-i)_2]_2$	572.4	I	I	I	11.47	72.08 d (J = 6.10 Hz) (OCH), 23.64 t (J = 4.89), Me
(9)	Me ₂ Sn[O(S)P(OPh) ₂] ₂	566.6	I	I	I	9.32	149.97 d (J = 8.55), (1), 121.12 d (J = 4.88), (2), 120.47 $e^{-(2)}$, 125 $e^{-(4)}$
(ع	Bu ₂ Sn[O(S)P(OPh) ₂] ₂	561.3	29.35	26.67	26.04	13.35	150.24 d (J = 8.53), (1), 121.26 d (J = 4.88), (2), 129.54 s (3), 125.58 s (4)
(6)	Ph ₂ Sn[O(S)P(OBu-n) ₂] ₂	6.006	139.78	135.12	128.16	130.02	66.92 d (J = 6.10) (OCH ₂), 31.31 d (J = 7.73) (OCH ₂); 17.95 s (CH ₂). 12.89 s (Me
(10)	Ph ₂ Sn[O(S)P(OPr- <i>i</i>) ₂] ₂	942.3	140.32	135.28	128.17	130.02	72.02 d $(J = 6.10)$ (OCH), 22.92 t $(J = 4.81)$ (Me)

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Diorganotin bis(Dialkyl Monothiophosphates)

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tin atoms in the complexes.^[15] These values are similar to those of dialkyltin bis(dialkyl dithiophosphates).

The corresponding $[{}^{1}J({}^{13}C-{}^{119}Sn]$ values for diphenyltin bis(diisopropyl and dibutyl monothiophosphates) could also be determined (after 4200 scans) and are 942.5 and 900.8 Hz, respectively, which is significantly higher than the reported values for triphenyltin carboxylates^[16] (~650 Hz) or diphenyltin chloride^[17] (786.8 Hz). These data are thus consistent with a hexa-coordinated state of tin in the diphenyl complexes also.

³¹P NMR Spectra

Proton decoupled ³¹P NMR spectra in CDCl₃ (Table 3) show only one peak for each compound in the region 42.5–50.9 ppm. These chemical shift values are indicative of bidentate attachment of the thiophosphato moieties to the tin atom.^[18]

¹¹⁹Sn NMR Spectra

¹¹⁹Sn NMR chemical shifts for diorganotin bis(dialkyl monothiophosphates) recorded in chloroform (Table 3) do not appear to change significantly with a change in the concentration. A Me₂Sn[O(S)P(OPr-*i*)₂]₂ solution of the concentration 2.2% (w/w) shows a signal at -130.5 ppm. This value is almost in the same range as that reported for R₂Sn[S₂P(OR')₂]₂ (~ -160 ppm) and thus is consistent with hexa-coordinated tin atoms.^[19] It may be recalled that in the case of Me₃SnO(S)P(OPr-*i*) which has a penta-coordinated structure, the chemical shift is 59.9 ppm.

Interestingly, the ¹¹⁹Sn shifts in organotin monothiophosphates appear to be affected by the nature of organic groups present in thiophosphate moieties. Thus, the values for $R_2Sn[O(S)P(OPh)_2]_2$ are in the range of -85ppm which are appreciably lower than the corresponding value for O,Odiisopropyl dithiophosphate derivatives.

¹¹⁹Sn chemical shift values for corresponding diphenyltin (diisopropyland dibutylmonothiophophates) were observed at -302.00 and -305.00ppm. These values are consistent with the values for diphenyltin bis(dialkyl and alkylene dithiophosphates),^[19] which contain hexa-coordinated tin atom.

The C-Sn-C bond angles for dimethyltin bis(diisopropyl and diphenyl dithiophosphates), calculated on the basis of $[{}^{1}J({}^{13}C-{}^{119}Sn]$ values, are 138.6° and 128.3°, respectively.^[15]

From the molecular weight, various spectroscopic data and yields, valuable information regarding structural features of diorganotin bis(dialkyl

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Figure 1. Suggested monomeric structure of diorganotin bis(dialkyl monothiophosphate). R = Me and R' = Et (1), Pr-i (2), Pr-n (3), Bu-n (4), Bu-i (5) and Ph (6); R = Bu and R' = Ph (7) and Bu-n (8); R = Ph and R' = Bu-n (9) and Pr-i (10).

monothiophosphates) may be obtained and the conclusion are summarized below:

- 1. Diorganotin bis(dialkyl monothiophosphates) in very dilute solutions are monomeric. The ¹¹⁹Sn chemical shifts as well as ${}^{1}H^{-119}Sn$ coupling constants obtained for the same concentrations confirm hexa-coordination of tin and, therefore, a *trans*-octahedral structure for the monomeric species may be suggested as shown in Figure 1.
- 2. In concentrated solutions, these derivatives are present as dimeric species but the spectroscopic features, in general, do not change. The dimer, therefore, may have the following structure in Figure 2, containing both chelating and bridging thiophosphate moieties. The presence of only one ³¹P NMR signal indicates a fast exchange between the bridging and chelating groups and obviously low-temperature NMR data would be required for these complexes.



Figure 2. Suggested dimeric structure of diorganotin bis(dialkyl monothiophosphate). R = Me and R' = Et (1), Pr-i (2), Pr-n (3), Bu-n (4), Bu-i (5) and Ph (6); R = Bu and R' = Ph (7) and Bu-n (8); R = Ph and R' = Bu-n (9) and Pr-i (10).

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Diorganotin bis(Dialkyl Monothiophosphates)



Figure 3. Suggested polymeric structure of diorganotin bis(dialkyl monothiophosphate). R = Me and R' = Et (1), Pr-i (2), Pr-n (3), Bu-n (4), Bu-i (5) and Ph (6); R = Bu and R' = Ph (7) and Bu-n (8); R = Ph and R' = Bu-n (9) and Pr-i (10).

- 3. Although molecular weights in solutions of higher than 18% concentration could not be determined, it seems reasonable to assume that the extent of oligomerisation should increase with concentration and in the neat form, these compounds probably are present as polymers of the following type in Figure 3. On the basis of the Möassbauer data for a few diorganotin bis(monothio-phosphates), Nasser and Zuckerman^[17] have also suggested a similar structure.
- 4. The *trans*-octahedral environment around tin in all of the above species is considerably distorted as indicated by the calculated C-Sn-C bond angles. It may not be out of place to mention here that x-ray crystal structures of diphenyltin bis(dithiophosphate)^[20,21] and dimethyltin bis(tetramethylethylene)^[22] show distorted *trans*-octahedral structures with 135° and 133.9° (C-Sn-C) bond angles, respectively. Our calculated bond angles being in the same range also support the above structure.

EXPERIMENTAL

IR spectra were recorded as Nujol mulls using a Perkin–Elmer 2000 FT IR spectrometer in the range 4000–200 cm⁻¹ and CsI cells. NMR spectra were recorded on a Bruker AC 250 FT spectrometer NMR using TMS as internal references for ¹H and ¹³C. ³¹P and ¹¹⁹Sn spectra were recorded using H₃PO₄ or Me₄Sn as external references, respectively. Molecular weights were determined using a Knaur vapour pressure osmometer.

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Stringent precautions were taken to exclude moisture. The ammonium salts of monothiophosphoric acids were prepared by the literature methods.^[23] Sulfur was determined by Messenger's method as barium sulfate and tin as tin oxide (Table 1).

Reactions of Diorganotin Dichlorides with Diisopropyl Monothiophosphate Salts in 1:2 Molar Ratio

A benzene (20 mL) solution of diorganotin dichloride (1.09 g, 4.9 mmol) was added to a suspension of ammonium dialkyl monothiophosphates (2.13 g, 9.9 mmol) in the same solvent (20 mL). The mixture was refluxed for \sim 3 hours. NH₄Cl which precipitated during the reaction was filtered the solvent was removed under reduced pressure and diorganotin dialkyl monothiophsphates were obtained as colorless, viscous liquids. Synthetic and analytical data are listed in Table 1.

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