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Metal and organometal complexes of phosphorus oxy- and thio-acids

III *. *O*-Alkyl *O*′-triorganostannyl phosphonates

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Abstract

O-Alkyl *O*′-triorganostannyl phosphonates, $R_3SnOP(O)(H)OR'$ (R = Me, Bu^n, Ph; R′ = Me and Et) have been obtained in excellent yield by the reactions of sodium salts of *O*-alkyl phosphonic acids with triorganotin chlorides. The triphenyltin derivatives are insoluble, high melting, white crystalline solids, whereas the trialkyltin derivatives are colourless, non-volatile, thermally and hydrolytically stable, viscous liquids, miscible with common organic solvents. Their molecular weights are concentration- as well as solvent-dependent. The IR and ^1H, ^{13}C, ^{31}P, and ^{119}Sn NMR spectral data are consistent with a polymeric structure involving planar triorganotin groups linked by phosphonate bridges. The two axial Sn–O–P bonds become equivalent as a result of strong P=O→Sn coordination.

Some reactions of the triorganostannyl alkyl phosphonates have also been studied.

Introduction

There is continuing interest in the chemistry of Sn–O–P-bonded compounds owing to their use as synthetic intermediates [2,3], (particularly in nucleoside [4] and carbohydrate chemistry [5]), as biocides [6], and as polymerisation catalysts [7,8]. They also present interesting structural possibilities because of the presence of both electron donor as well as electron acceptor sites in the molecule. Work in this area has been concerned mainly with triorganotin phosphates [7–12] and phosphinates [12–14] e.g., $(R_3SnO)_nP(O)(X)_{3-n}$; X = alkoxy or alkyl; $n = 1$ and 3, structural

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studies of which have revealed the bridging bidentate behaviour of phosphato and phosphinato moieties that gives rise to oligomeric or polymeric structures.

Surprisingly, studies on organotin phosphonates [12,15–18] have been confined to the derivatives of phenylphosphonic acid, and derivatives of the parent phosphonic acid, $HP(O)(OH)_2$, are still unknown. The present report deals with the synthesis, spectroscopic characterisation, and some reactions of *O*-alkyl *O'*-triorganostannyl phosphonates $(R_3SnO)(R'O)P(O)H$, a new series of Sn–O–P-bonded compounds.

Experimental

Tin was determined as SnO_2. Infrared spectra were recorded as Nujol mulls between CsI disks on a Perkin–Elmer spectrometer model 577 in the range 4000–200 cm^{-1}. The ^1H, ^{13}C, ^{31}P and ^{119}Sn NMR spectra were measured at 22 ° C on a JEOL FX 90Q spectrometer operated at 89.55, 22.49, 36.23, and 33.85 MHz respectively, with proton-decoupling. External D_2O was used as lock. The ^1H and ^{13}C NMR spectral data refer to carbon tetrachloride solutions with TMS as an external reference. The ^{31}P and ^{119}Sn NMR data refer to benzene solutions with aqueous H_3PO_4 or $SnMe_4$ as external reference. The concentrations for ^1H and ^{31}P NMR spectra were ~ 15–20%, and about 50% (w/w) for the ^{13}C and ^{119}Sn spectra. Molecular weights in chloroform and benzene were measured on a Knouer Vapour pressure osmometer.

Sodium *O*-alkyl phosphonates, NaOP(O)(H)OR (R = Me, Et) were obtained from dialkyl phosphonates [19], and were analytically and spectroscopically characterised. ^{31}P NMR: NaOP(O)(H)OMe in MeOH, δ 2.90, $^1J(^{31}P-^1H)$ 612.7 Hz; NaOP(O)(H)OEt in EtOH, δ 5.13, $^1J(^{31}P-^1H)$ 619.0 Hz.

Reaction of trimethyltin chloride with sodium O-ethylphosphonate in 1 / 1 molar ratio

Sodium hydroxide (0.51 g) was dissolved in refluxing ethanol (~ 30 cm^3) and to this was added diethyl phosphonate (1.76 g). Trimethyltin chloride (2.54 g) was then added and the mixture again refluxed for ~ 2 h. Ethanol was distilled off, the residue was dissolved in benzene (~ 20 cm^3) and the solution was filtered to remove sodium chloride (0.73 g; 98%). The solvent was then removed and the residue kept under reduced pressure to give a colourless viscous liquid (**1**); yield 3.36 g, (97%). The product remained unchanged at 225 ° C and 0.05 mm Hg pressure.

Anal. Found: Sn, 44.10. $C_3H_{15}O_3PSn$ calc: Sn, 43.50%. ν_{max}: 2405s (P–H), 1200s (P=O), 1065s (C–O), 790s (P–O); ^1H NMR (CCl_4): δ 0.45 [9H, s, $^2J(^{119}Sn-^1H)$ 72 Hz, Sn-CH_3], 1.20 [3H, t, $^3J(^1H-^1H)$ 7 Hz, C-CH_3], 3.50–4.00(2H, m, -OCH_2), 6.32 [1H, d, $^1J(^{31}P-^1H)$ 665 Hz, P-H].

Other triorganotin *O*-alkylphosphonates were prepared similarly by the reactions of triorganotin chlorides with sodium ethyl phosphonate in ethanol (compound **3** and **5**) or with sodium methyl phosphonate in methanol (compound **2**, **4** and **6**). Pertinent data for individual reactions are given below:

$Me_3SnOP(O)(H)OMe$ (2). NaOH (0.99 g), MeOH (~ 25 cm^3), $(MeO)_2P(O)H$ (2.73 g), Me_3SnCl (4.95 g); yield 6.11 g (95%).

Anal. Found: Sn, 46.20. $C_4H_{13}O_3PSn$ calc: 45.90%. ν_{max}: 2420s (P–H), 1195s (P=O), 1070s (C–O), 800s (P–O); ^1H NMR (CCl_4): δ 0.22 [9H, s, $^2J(^{119}Sn-^1H)$ 73

Hz, Sn-CH_3], 3.25 [3H, d, $^3J(^{31}P-^1H)$ 12 Hz, -OCH_3], 6.57 [1H, d, $^1J(^{31}P-^1H)$ 653 Hz, P-H].

$Bu^n_3SnOP(O)(H)OEt$ (3). NaOH (0.96 g), EtOH(~ 30 cm^3), $(EtO)_2P(O)H$ (3.30 g), Bu_3SnCl (7.80 g); yield 9.18 g, (96%).

Anal. Found: Sn, 29.90. $C_{14}H_{33}O_3PSn$ calc: 29.80%. ν_{max}: 2400s (P–H), 1205s (P=O), 1070s (C–O), 780m (P–O); ^1H NMR (CCl_4): δ 0.40–1.80 (30H, m, Sn-Bu^n, -CH_3), 3.40–4.00 (2H, m, -OCH_2), 6.45 [1H, d, $^1J(^{31}P-^1H)$ 646 Hz, P-H)].

$Bu^n_3SnOP(O)(H)OMe$ (4). NaOH (0.50 g), MeOH (~ 30 cm^3) $(MeO)_2P(O)H$ (1.38 g), Bu_3SnCl (4.08 g); yield 4.62 g, (95%).

Anal. Found: Sn, 31.00. $C_{13}H_{31}O_3PSn$ calc: 30.85%. ν_{max}: 2410s (P–H), 1210s (P=O), 1075s (C–O), 800s (P–O); ^1H NMR (CCl_4): δ 0.50–1.75 (27H, m, Sn-Bu^n), 3.42 [3H, d, $^3J(^{31}P-^1H)$ 12Hz, -OCH_3], 6.47 [1H, d, $^1J(^{31}P-^1H)$ 646 Hz, P-H].

$Ph_3SnOP(O)(H)OEt$ (5). NaOH (0.57 g), EtOH (~ 30 cm^3), $(EtO)_2P(O)H$ (1.97 g), Ph_3SnCl (5.50 g); yield 5.11 g, (78%, from benzene); m.p. 182–183 °C.

Anal. Found: Sn, 26.30. $C_{20}H_{21}O_3PSn$ calc: Sn, 25.90%. ν_{max}: 2460m (P–H), 1150(P=O), 1080s (C–O), 865m (P–O).

$Ph_3SnOP(O)(H)OMe$ (6). NaOH (0.36 g), MeOH (~ 25 cm^3), $(MeO)_2P(O)H$ (0.99 g), Ph_3SnCl (3.46 g); yield 3.06 g, (76%, from benzene); m.p. 218 °C.

Anal. Found: Sn, 26.90. $C_{19}H_{19}O_3PSn$ calc: Sn, 26.70%. ν_{max}: 2410m (P–H), 1175s (P=O), 1070s (C–O). 875s (P–O):

Compound **1** was also prepared by first isolating pure NaOP(O)(H)OEt and then heating it with trimethyltin chloride. The analytical and spectroscopic data for this sample were identical with those for the above.

Results and discussion

O-Alkyl-*O′*-triorganostannyl phosphonates have been prepared by the reactions of sodium *O*-alkyl phosphonates (made in situ by basic hydrolysis of dialkylphosphonates) with triorganotin chloride:

$$R_3SnCl + NaOP(O)(H)OR' \xrightarrow{R'OH + benzene} R_3SnOP(O)(H)OR' + NaCl$$

(**1**) R = Me, R′ = Et; (**2**) R = Me, R′ = Me; (**3**) R = Bu^n, R′ = Et; (**4**) R = Bu^n, R′ = Me; (**5**) R = Ph, R′ = Et; (**6**) R = Ph, R′ = Me

Removal of sodium chloride followed by evaporation of the solvent yielded the desired product.

The trimethyltin and tributyltin derivatives are colourless non-volatile viscous liquids, thermally stable up to 225 °C under vacuum, and miscible with common organic solvents, like benzene, toluene, chloroform and carbon tetrachloride. The triphenyltin derivatives, on the other hand, are sharp melting white crystalline solids, sparingly soluble in benzene (from which they can be recrystallised) and insoluble in other common organic solvents (because of this their ^1H, ^{13}C and ^{119}Sn NMR spectra could not be recorded).

The infrared spectra of these compounds show a strong band in the region 2460–2400 cm^{-1} that can be assigned to the P–H stretching vibration. This band is not appreciably shifted from its position in the spectrum of $(MeO)_2P(O)H$ (at 2427 cm^{-1}) or $(EtO)_2P(O)H$ (at 2425 cm^{-1}) [20]. The strong phosphoryl band appears in the range 1210–1150 cm^{-1}, and is thus shifted substantially to lower frequency (by

55–115 cm^{-1}) from its position for dialkyl phosphonates [e.g., $(EtO)_2P(O)H$, 1265 cm^{-1}] [20]. This indicates involvement of phosphoryl oxygen in coordination to tin atoms. If the $\nu(P{=}O)$ shift is taken to reflect the strength of $P{=}O \rightarrow Sn$ dative bond, it can be concluded that Ph_3Sn is a much stronger electron acceptor than Bu_3Sn or Me_3Sn groups, the latter two being almost equal in this respect. A similar conclusion was reached previously during studies on complexation of triorganotin halides [21]. Other characteristic absorptions of the phosphonato moieties in the IR spectra are in the regions 1080–1065 cm^{-1} and 875–780 cm^{-1}, and may be assigned to ν((P)–O–C) and ν(P–O–(C)) [20]. The peaks due to Sn–C stretching vibrations usually appear in the region 500–600 cm^{-1}, and the number of peaks in the trimethyltin derivatives can be correlated with the geometry of the Me_3Sn moiety [22]. Unfortunately, there are strong absorptions in this region in the spectra of dialkyl phosphonates as well as in those of their sodium salts, making assignment of the Sn–C stretching vibrations very difficult.

In the ^1H NMR spectra of *O*-alkyl *O′*-triorganostannyl phosphonates, the *O*-alkyl protons show the usual 3-bond ^1H–^{31}P coupling of ~ 12 Hz [23]. The hydrogen atom directly attached to phosphorus, on the other hand, shows a coupling of the order of 650–680 Hz. The tin proton coupling constants $^2J(^1H–^{119}Sn)$ in trimethylstannyl phosphonates are 73.2 and 72.0 Hz, for *O*-methyl and *O*-ethyl derivatives, respectively, values which are consistent with the presence of pentacoordinate tin in these compounds [24].

The ^{13}C NMR spectra (Table 1) show tin–carbon coupling constants, $^1J(^{13}C–^{119}Sn)$, in the range 460–550 Hz, which again indicate the presence of five-coordinate tin [25,26].

Only one peak was observed in proton decoupled ^{31}P NMR spectra of these compounds (Table 2) in the range −0.73 to −4.23 ppm. Substitution of an ethyl group in triethylphosphate by a trimethylstannyl group has been reported to shift the ^{31}P signal by about 8 ppm upfield ($\delta(EtO)_3PO$, −0.96 ppm; $Me_3SnOP(O)(OEt)_2$, −9.1 ppm) [9]. A similar 9–15 ppm upfield shift of ^{31}P signal is observed in these cases also when the values for $(RO)_2P(O)H$ and $R_3SnOP(O)H(OR')$ are compared [$(MeO)_2P(O)H$, 11.0; $(EtO)_2P(O)H$, 8.0 ppm] [27]. The ^{31}P signals at ambient temperature do not show satellites due to coupling with ^{119}Sn except for $Bu_3SnOP(O)H(OEt)$, for which there is a value of 105 Hz for $^2J(^{31}P–^{119}Sn)$. The values of one bond ^{31}P–^1H coupling constants (655 ± 10 Hz) (from proton coupled ^{31}P NMR spectra) are almost same as those from the ^1H NMR spectra, with slight

Table 1

^{13}C NMR shifts in CCl_4 (δ (ppm))

Compound	$OSnR_3$ carbons	$^1J(^{13}C–^{119}Sn)$ (Hz)	OR′ carbons
1	1.83	546.9	16.19 (CH_3), 59.96 (CH_2)
2	1.56	508.8	54.93
3	19.94(C(1)), 27.95(C(2)) 27.03(C(3)), 13.66(C(4))	464.9	16.34 (CH_3), 60.03 (CH_2)
4	20.58(C(1)), 27.95(C(2)), 26.97(C(3)), 13.75(C(4))	502.6	53.69 (CH_3)

Table 2

^{31}P and ^{119}Sn NMR shifts (δ in ppm) in benzene

Compound	$\delta(^{31}P)$	$^{1}J(^{31}P-^{1}H)$ (Hz)	$\delta(^{119}Sn)$
1	−2.83	651	−41.07
2	−1.61	660	−34.93
3	−1.10	657	−41.88
4	−3.56	645	−57.15
5	−4.23	667	[a]
6	−0.73	664	[a]

[a] Could not be obtained due to low solubility.

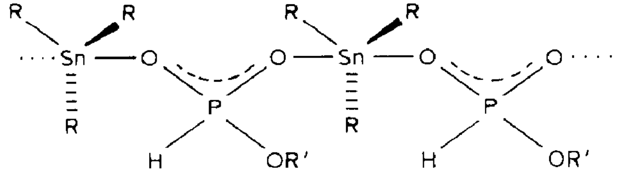
changes (5–15 Hz) in some cases. These values are slightly less than those for dialkyl phosphonates $^{1}J(^{31}P-^{1}H)$: $(MeO)_2P(O)H$, 700 Hz; $(EtO)_2P(O)H$, 670 Hz [27].

The ^{119}Sn NMR spectra of the trialkylstannyl phosphonates in benzene (Table 2) show chemical shifts in the range −34.93 to −57.15 ppm, indicating that they refer to pentacoordinate tin in these compounds [12,28]. These values are also comparable to those reported for triorganotin dialkyl phosphates, $R_3SnOP(O)(OR')_2$ (R,R′, δ = Me, Me, −32; Me, Bu, −39; Bu, Me, −55 and Bu, Bu, −61 ppm) [8]. In comparison with the ^{119}Sn shifts for other 5-coordinated triorganotin compounds, these values are substantially upfield, and indicate the stronger coordination from phosphonate and phosphate groups to tin. The signals are broad (with half-height width of ~ 10 ppm), thus showing the occurence of exchange at room temperature. Detailed NMR studies at low temperature are required to throw light on this process.

The tin–phosphorus coupling could not be resolved at room temperature except in the case of $Bu_3SnOP(O)H(OMe)$ (in benzene), for which, the tin signal appears as a triplet with a J value of 102 Hz. The tin–phosphorus coupling through oxygen has been reported in the literature to be observable only at low temperatures, with the J values quite different for coupling to 4- and 5-coordinated tin atoms (83–85 and 102–115 Hz, respectively). A few reported values are: $Me_3SnOP(O)Me_2$ (from ^{31}P NMR in THF at −60 °C, 102 Hz); $(Bu_3SnO)_2P(O)Me$ (from ^{31}P NMR in THF at −50 °C, 83 and 110 Hz) and $(Bu_3SnO)_2P(O)Ph$ (from ^{119}Sn NMR in toluene at −50 °C, 85 and 114 Hz) [12,14]. The values obtained in the present investigation (from ^{119}Sn NMR of **1** and ^{31}P NMR of **2**) are thus indicative of coupling to a 5-coordinate tin atom. The triplet nature of the tin signal shows that the tin atom is coupled to two almost equivalent phosphorus atoms.

It is noteworthy that the rate of exchange of phosphonate moieties appears to depend on the nature of the solvent. Thus the ^{119}Sn signal of $Me_3SnOP(O)H(OEt)$, which is broad in benzene, is very sharp in CH_2Cl_2. Broadening in the latter case starts when the temperature is lowered to −30 °C, and the shape is similar to that for benzene solutions only at −70 °C. (The temperature could not be lowered further)

The non-volatility and high thermal and hydrolytic stability of *O*-trialkylstannyl-*O*-alkyl phosphonates indicate an associated polymeric structure of the following type, and this is supported by the spectroscopic data:



In the above structure, with planar R_3Sn moieties and bridging phosphonato groups, the two axial Sn–O–P bonds become equivalent as indicated by the IR (ν(P=O)) and NMR (^{119}Sn–^{31}P coupling) spectra. Such associated structures are common for a number of triorganotin esters of bidentate inorganic oxyacids, e.g.; phosphate [8,9,12], carbonate [29] and sulphinate [30] etc.

As indicated by the molecular weight data (Table 3) for compounds **1** and **3** in chloroform and benzene solutions of varying concentrations, the extent of polymerisation of *O*-triorganostannyl *O*-alkyl phosphonates in solution depends on the concentration, the nature of solvent, and the nature of the organotin group. The observations can be summarised as:

(a) The molecular association increases with increasing concentration, indicating the dynamic nature of the system in solution. Similar concentration-dependent association has been noted earlier for triorganotin dialkyl phosphates [8] and phosphinates [14].

(b) At a given concentration, the degree of association is higher in benzene than in chloroform.

(c) At a given concentration, the trimethylstannyl derivative is more highly associated than the tributylstannyl derivative.

It would be of interest to correlate the changes in molecular association with the changes in the NMR parameters by carrying out the studies under the same experimental conditions. However, this approach is limited by the fact that molecular weights are determined in dilute solution (< 20% w/w) whereas for the NMR data, particularly in the case of ^{119}Sn and ^{13}C nuclei, much higher concentrations

Table 3

Molecular weight and NMR data

Compound	Solvent	Concentration (g/kg)	Mol. weight obtained	Molecular association	NMR		
					$\delta(^{31}P)$ (ppm)	$^1J(^{31}P-^1H)$ (Hz)	$\delta(^{119}Sn)$ (ppm)
$Me_3SnOP(O)(H)OEt$							
	Chloroform	11.08	803	2.94			
	Chloroform	89.05	2163	7.93			
	Chloroform	151.36	2213	8.11			
	benzene	88.13	5783	21.21			
$Bu_3SnOP(O)(H)OEt$							
	Chloroform	18.74	763	1.91	−2.097	644.6	–
	Chloroform	98.79	1062	2.66	−2.579	647.8	−44 [a]
	Chloroform	163.91	1097	2.75	−2.623	644.6	−46 [a]
	Chloroform	431.80	[b]	–	–	–	−40 [a]
	benzene	36.43	1137	2.85	−3.451	647.8	
	benzene	184.12	2613	6.55			

[a] Broad signal. [b] Molecular weight could not be determined because of the increased density of the solution.

are needed. From the ^{31}P and ^{119}Sn NMR data shown in Table 3, it appears that the change in molecular association is not reflected in the chemical shift values, and the latter do not, in fact, show any appreciable change. This probably means that even a considerable change in molecular association does not significantly change, the magnetic environment around the nuclei. It must be remembered that, like the molecular weight values, the chemical shifts, are also average values for a mobile system.

The oligomeric units of triorganostannyl alkyl phosphonates (**1**–**6**) may possess an open chain and/or a cyclic structure, and NMR data do not throw light on this aspect. One of the terminal tin atoms in an open chain structure should be 4-coordinate and one of the phosphorus atoms should likewise be monodentate. However, no additional signal could be observed in the ^{119}Sn or ^{31}P NMR spectra at room temperature, and this could be due to the fast exchange and to the presence of cyclic structures. It is noteworthy that splitting of the ^{31}P signal at low temperatures has been observed for $Me_3SnOP(O)(OEt)_2$ (−80 °C) [9] and $Me_3SnOP(O)Me_2$ (−60 °C) [14], and explained on the basis of the presence of open chain as well as cyclic structures in the case of the former, and of slowing down of the exchange between terminal and bridging phosphonate moieties in the case of the latter.

Crystal structure determinations have revealed that whereas $Ph_3SnOP(O)(OPh)_2$ [10] forms a cyclic hexamer, the phosphonate $Me_3SnOP(O)(OH)Ph$ [16] is a helical polymer. Attempts to obtain suitable crystals of triphenyltin *O*-alkyl phosphonates during the present investigations for an X-ray diffraction study were unsuccessful.

Triorganostannyl *O*-alkyl phosphonates are not hydrolysed by water. They do react, however, with refluxing ethanolic NaOH with cleavage of the Sn–OP rather than the C–OP bond, as indicated by the fact that ^{31}P NMR spectrum of the product mixture formed from $Bu_3SnOP(O)(OEt)H$ and NaOH (1/1) can be attributed to $NaOP(O)(OEt)H$ (δ 5.13 ppm).

$$R_3SnOP(O)(OR')H + NaOH \rightarrow R_3SnOH + NaOP(O)(OR')H$$

The $P{=}O \rightarrow Sn$ coordination in triorganostannyl alkyl phosphonates appears to be quite strong, and the linkage is not cleaved by pyridine (there is no change in ^{31}P and ^{119}Sn NMR in pyridine solution). These compounds do not undergo some of the typical reactions shown by alkyl phosphonates (e.g., bromination or reaction with dialkylamines in CCl_4). The differences are probably due to the fact that the tin compounds cannot give the phosphite form, $(R_3SnO)(RO)POH$, as an intermediate.

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