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A Tin-119 NMR investigation of phosphine and phosphine oxide adducts of organotin chlorides

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Abstract

The stoichiometry and structure of phosphine and phosphine oxide adducts of Ph_3SnCl, R_2SnCl_2 (R = Et, Pr, Bu, *t*-Bu, and Ph), and $RSnCl_3$ (R = Bu and Ph) were studied with Sn-119 spectroscopy. The shift of the Sn-119 resonance to a lower frequency upon adduct formation, the multiplicity of the resonance, the variation of P-31-Sn-119 coupling with the nature of the substituent, and the change in structure of the peaks with concentration and temperature were all used to determine stoichiometry and structure. The organotin chloride adducts readily exchange with base or with other adducts. The diorganotin dichlorides form only 1:1 adducts with tributylphosphine (TBP), even at high base to acid ratios, but most form 1:2 adducts (as several geometric isomers) with phosphine oxides at mole ratios above 1:1. The lower dialkyltin dichlorides prefer to form 1:1 adducts (at 1:1 mole ratios) with TBP rather than tributylphosphine oxide (TBPO), whereas diphenyltin dichloride and di(*t*-butyl)tin dichloride prefer TBPO adduct formation. The reactions of the trihalides with TBP and TBPO are complicated by aryl transfer or displacement of chloride by base and consequent ion formation.

Keywords: Organotin adduct; Tin-119 chemical shift; Sn-119-P-31 coupling constant; Exchange

1. Introduction

Studies of adducts of organotin halides continue to provide fundamental information about both the Lewis acid–base model and the reactivity of organotin species. Previous work from our laboratory using both P-31 NMR and calorimetry indicated that organotin chlorides form predominantly 1:1 adducts in solution, with evidence of 1:2 and other adducts for both R_2SnCl_2 and $RSnCl_3$ [1–3]. The P-31 signals from various adducts were exchange averaged even at temperatures of −90 °C [1,2]. Because of the relatively high abundance and the large and distinctive range of chemical shifts of Sn-119, NMR studies of this nucleus have the potential to reveal a great deal of direct structural information, particularly in organotin adducts where the coordination number of tin is changing. Colton and Dakternieks have utilized Sn-119 NMR in several studies of adducts of organotin halides and have reported formation of only 1:1 adducts between diphenyl- and dimethyltin dichloride and tributyl- and tricyclohexylphosphine and both 1:1 and 1:2 adducts for the interaction of diphenyltin dichloride with tributylphosphine oxide (TBPO) [4]. Our study is designed to: (a) explore the relative reactivities of tributylphosphine (TBP) and TBPO with dialkyltin dichlorides and diphenyltin dichloride, (b) determine the relative reactivities of other bases with diphenyltin dichloride (chosen as a model dichloride), and (c) to reexamine the reaction of TBP and TBPO with phenyl- and butyltin trichloride.

2. Experimental

Organotin chlorides were obtained from Aldrich Chemical Co. or Gelest and were used without purification. Bases were dried over Drierite and then stored over type 4A molecular sieves. Solvents were obtained from Aldrich Chemical Co. and were dried over type 4A molecular sieves. Solutions were prepared under Ar using oven-dried glassware and 10 mm NMR tubes. Tin-119 spectra were obtained on a Varian Unity 300 at

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111.8 MHz using gated proton decoupling. For each acid–base pair, spectra were obtained at mole ratios of 1:0.5, 1:1, and 1:2 (acid to base, respectively). In many cases, additional mole ratios of 1:0.8, 1:1.5, and 1:4 were also obtained. Reproducibility of spectra was ascertained by obtaining spectra on additional sets of solutions for most acid–base pairs. When solubility permitted, the initial concentration of acid was 0.4 g in 4 ml of solvent. In many cases, several series of spectra were taken with delay times varying from 1 to 30 s.

3. Results and discussion

The results obtained from the tin-119 spectra are reported in Tables 1 and 2. Assignment of stoichiometry was based on the extent of the low frequency shift relative to the shift of the free acid. Generally, shifts of 200–250 ppm to low frequency are indicative of five-coordinate (1:1) adducts, with another 200 ppm shift to low frequency characteristic of six-coordinate (1:2) adducts [5–8]. For each acid–base pair, exchange of base was indicated by: (a) the presence of an ex-

Table 1
^{119}Sn Shifts and ^{31}P–^{119}Sn couplings in adducts [e]

Acid/base	Species present			NMR shifts		
	1:1 Mole ratio	1:2 Mole ratio	Free acid	1:1 Adduct	1:2 Adduct	1:3 Adduct
Et$_2$SnCl$_2$			127			
TBP	1:1	1:1		−176(d,207) [b,f]		
TBPO	1:1	1:2 (3 isomers)		−117(d,122) [b,f]	−306(b), −333, 368(b) [c]	
Pr$_2$SnCl$_2$			127			
TBP	1:1	1:1		−183(d,143) [a,f]		
TBPO	1:1	1:2 (2 isomers)		−125(d,143) [b,f]	−339(b), −376(b) [c]	
(*t*-Bu)$_2$SnCl$_2$			55			
TBP	1:1			−207(d,89) [c,f]		
TBPO	1:1, 1:2			−183(d,195) [b,f]	−241(t,217) [b]	
Bu$_2$SnCl$_2$			128			
TBP	1:1	1:1		−182(d, 195) [c,f]		
TBPO	1:1	1:2 (3 isomers)		−124(d,143) [b,f]	−308(b), −338(b), −374(d,172) [d,f]	
Ph$_2$SnCl$_2$			−32			
TBP	1:1	1:1		−285(d,818) [c]		
TBzP	1:1 (isomers)	1:1 (isomers)		−256(d,1065) −257(d,998) [f] −276(d,720) [c]		
TBPO	1:1, 1:2 (isomers)	1:1, 1:2 (isomers)		−274(d,156) [b,f]		−522(q,162) [c]
	1:3	1:3			471(t,154), 474(t,167) [c]	
TPPO	1:1	1:1		−258(d,134) [b,f]		
TEPO	1:1	1:1		−275(b) [c]		
DMSO	1:1	1:1, 1:2		−234	−398(b), −406 [c]	
Pyridine	1:1, 1:2	1:1, 1:2		−389 [c]	−360 [c]	
Ph$_3$SnCl			−45.3			
TBP	1:1 + Free acid			−285 in CDCl$_3$ (0.1 g in 5 ml)		
TBPO	1:1	1:1		−110(b)		
TPPO	Mainly acid			−70 moving to −114 from 1:2 to 1:6		
PhSnCl$_3$			−60.6			
TBP	See text					
TBzP	See text					
TBPO	1:1	1:1		−336(d,210) [f]		
		1:2 (isomers),1:3			556(dd,184,88) [f], 579 (t,201) [f], −581(t,201) [f]	−607(q,210)
TPPO	1:1	1:1,1:2		−335(vb)	< −500(vb)	

[a] At −60 °C in CDCl$_3$.
[b] At −110 °C in CD$_2$Cl$_2$.
[c] At −90 °C in CD$_2$Cl$_2$.
[d] At −100 °C in CD$_2$Cl$_2$.
[e] Shifts in ppm from Sn(CH$_3$)$_4$, couplings in Hz.
[f] Coupling constant obtained from peaks not completely resolved, b = broad (>400 Hz), d = doublet, t = triplet, q = quartet, dd = doublet of doublets.

Table 2
Relative acidities toward TBP and TBPO [a]

Acid	TBP	TBPO
Ph_2SnCl_2	42	56
$(t\text{-Bu})_2SnCl_2$	46	54
Bu_2SnCl_2	59	43
Pr_2SnCl_2	78	22
Et_2SnCl_2	69	28
$BuSnCl_3$	67	33

[a] Percent acid forming adduct with each base as obtained from integration of ^{119}Sn resonances.

changing acid peak (appearing between the free acid and the 1:1 adduct) at low mole ratios (for example, 1:0.5), (b) a shift to lower frequencies and a change in peak width upon the addition of more base, and (c) a decrease in peak width at lower temperatures and the appearance of coupling in the phosphine and phosphine oxide series at sufficiently low temperatures.

For triphenyltin chloride and TBP, a 1:1 mole ratio of acid to base produced both free acid and the trigonal bipyramidal, five-coordinate 1:1 adduct with a shift of − 285 ppm. The adduct peak is − 238 ppm to lower frequency of the free acid (− 47 ppm) and could not be examined at lower temperatures because of the insolubility of both the acid and the adduct. The observation of an adduct of triphenyltin chloride with TBP is contrary to our earlier results [1] and those of Colton and Dakternieks [4]. Additional drying of solvent and base gave the same results. With TBPO as base, there is evidence of a 1:1 adduct, whereas the equilibrium with triphenylphosphine oxide (TPPO) lies to the left so that at a mole ratio of 1:1, only free acid is observed. Additional base moves the peak to lower frequencies, indicative of the formation of small amounts of exchanging 1:1 adduct.

With the diorganotin dichlorides, TBP forms only a five-coordinate 1:1 adduct, even in solutions with mole ratios as high as 1:4 (acid to base). At mole ratios up to 1:1, the corresponding phosphine oxide, TBPO, forms a 1:1 adduct with the dialkyltin dichlorides which is converted completely to the 1:2 adduct at mole ratios of 1:2. As shown in Table 1, three peaks are present in the six-coordinate region for adduct formation at low temperatures with the diethyl- and dibutyltin dichlorides. These are likely due to the presence of at least three of the five possible octahedral isomers (see Fig. 1). At − 110 °C, exchange is still sufficiently rapid [4] to prevent the observation of two-bond coupling to P-31. For di(*t*-butyl)tin dichloride only one six-coordinate isomer is apparent. This is presumably a result of the destabilization of the *cis* isomers by the bulky *t*-butyl groups. That same hindrance presumably also slows exchange so that the two-bond coupling of 217 Hz can be seen in a triplet even at − 50 °C. The apparent equivalence of the two phosphorus atoms and the steric hindrance suggest that this isomer is one of the two isomers with *trans* ligands.

The reaction of diphenyltin dichloride with tribenzylphosphine in 1:1 and in 1:2 mole ratios produces three doublets (at − 80 °C) in the five-coordinate adduct region, which presumably correspond to three of the five possible trigonal bipyramidal isomers. Although all three of these isomers could contain the base on the equatorial position, the magnitude of the couplings and the chemical shifts suggest that the two higher frequency doublets ($J = 1065$, 998 Hz) are due to equatorial isomers, while the lower frequency doublet ($J = 720$ Hz) is due to an axial phosphorus [8,9].

Triethylphosphine oxide and TPPO, like the phosphines, form only five-coordinate adducts with diphenyltin dichloride. The TEPO adduct appears as a broad peak at − 275 ppm, while the TPPO adduct appears at − 90 °C as a doublet centered at − 258 ppm. The Sn-119 spectrum of diphenyltin dichloride with TBPO contains peaks in the five-coordinate and six-coordinate regions at mole ratios of 1:1 and higher. The 1:1 adduct appears as a broad peak at − 274 ppm down to − 50 °C (possibly indicative of the exchange between five-coordinate isomers) but as a doublet at − 90 °C. In the six-coordinate region at − 90 °C there are two triplets

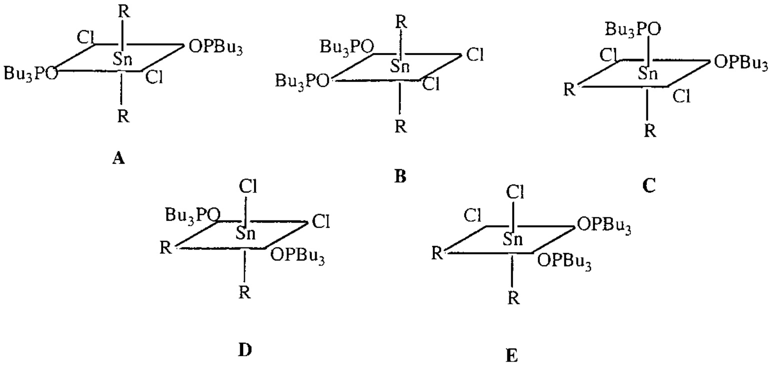

Fig. 1. Stereochemical isomers of dialkyltin dichloride and TBPO 1:2 adduct.

at −471 and −474 ppm and a quartet at −522 ppm. The 1:1 peak at −274 ppm and 1:2 peaks at −471 and −474 ppm are in good agreement with those reported by Colton and Dakternieks [4]. The quartet at −522 ppm was not observed by these authors, although a quartet was reported for the TBPO adduct of diphenyltin dibromide and attributed to the ion $[Ph_2SnBr(TBPO)_3]^+$ [4]. We attribute the peaks at −471 and −474 ppm to two of the five possible octahedral six-coordinate 1:2 adducts and the quartet at −522 to the ion $[Ph_2SnCl(TBPO)_3]^+$ [4]. Following the reasoning of Colton and Dakternieks, we believe that this ion is formed by displacement of chloride by TBPO. The quartet is presumed to be a result of the equivalency of three TBPO ligands produced by exchange between two or more octahedral isomers.

DMSO appears to form a 1:1 adduct with Ph_2SnCl_2 at a mole ratio of 1:1 and 1:2 adducts at higher mole ratios. Only one fluxional isomer is observed for the five-coordinate adduct, while two fluxional isomers are observed for the 1:2, six-coordinate adducts. The Sn-119 spectrum for Ph_2SnCl_2 and pyridine shows a single broad peak between the 1:1 and 1:2 regions at all mole ratios for pyridine, suggesting that both 1:1 and 1:2 adducts are present and exchanging rapidly. Insolubility of the pyridine adducts precluded their study at low temperatures, due to instrumental time constraints.

In order to determine the affinity of the diorganotin dichlorides for TBP relative to TBPO, the Sn-119 NMR spectra of 1:0.4:0.4 mole ratios of the acid with both bases were obtained. At this mole ratio only the 1:1 adduct is observed for both TBP and TBPO, and the integrated ratio of the two adduct peaks is shown in Table 2. The table shows clearly the greater affinity of the smaller dialkyltin dichlorides for TBP and the greater affinity of di(*t*-butyl)- and diphenyltin dichloride for TBPO. The relative decrease in affinity for TBP with an increase in the size of the organo groups suggests that hindrance with the butyl groups on TBP increases more rapidly than the hindrance with the further removed butyl groups of TBPO. The table also shows that butyltin trichloride preferentially forms adducts with TBP. Because the trichloride should be a harder acid than dibutyltin dichloride and yet has a greater affinity for the soft TBP, it is probably not particularly productive to express these affinities using the hard–soft phenomenology.

Our previous work suggested that the organotin trichlorides showed a greater number of equilibria than the other organotin halides, in addition to being the strongest Lewis acids [1]. Indeed, our present Sn-119 study, as well as that of Colton and Dakternieks [10], shows that these systems produce reactions that we did not originally anticipate. The reaction of butyltin trichloride with TBP produces the 1:1 adduct at low base concentrations, but at concentrations greater than 1:1, the Sn-119 spectrum shows a triplet at −271 ppm ($J = 775$ Hz) and a doublet at −442 ppm ($J =$ ca. 2900 Hz) at temperatures of −90°C. We attribute these peaks, predominant at mole ratios of 1:2 (acid to base), to $BuSnCl_2(TBP)_2^+$ and $BuSnCl_4(TBP)^-$. The assignment of structure is based upon the chemical shift, the multiplicity, and the coupling constant. For example, the −442 ppm peak is in the six-coordinate region, being almost 440 ppm to low frequency of the acid, and the doublet suggests that only one phosphorus is attached to the tin. The one bond tin–phosphorus coupling seems to depend primarily on the number of electronegative groups attached to tin [9] (see below) and the coupling of almost 3000 Hz suggests that a large number of chlorines are present. We attribute the formation of these two ions to transfer of chloride from one $BuSnCl_3(TBP)$ adduct to another followed by complexation of $BuSnCl_2(TBP)^+$ with a molecule of TBP.

$$BuSnCl_3(TBP) + BuSnCl_3(TBP) \rightarrow BuSnCl_2(TBP)^+ + BuSnCl_4(TBP)^-$$

Alternatively, their formation could be a result of the nucleophilic displacement of chloride by TBP from $BuSnCl_3(TBP)_2$ thereby producing $BuSnCl_2(TBP)_2^+Cl^-$, followed by complexation of the chloride with $BuSnCl_3(TBP)$. Both hypotheses provide a rationalization for the occurrence of these ions only in solutions containing relatively high concentrations of base. There is also evidence for the presence of $BuSnCl_3(TBP)_2$ in solutions with a 1:2 mole ratio, where a triplet appears at −473 ppm ($J = 351$ Hz) in the six-coordinate region.

The reaction of $BuSnCl_3$ with TBPO is somewhat simpler. At low base concentrations only the 1:1 adduct is present (see Table 1). However, at mole ratios of 1:1.2 both the 1:1 and the 1:2 adducts (with three of the five octahedral isomers) are indicated at −110 °C. A quartet at −556 ppm is probably due to the presence of $BuSnCl_2(TBPO)_3^+Cl^-$. As indicated above, this species can be rationalized by displacement of chloride in the 1:2 adduct by TBPO. The same species are also present at mole ratios of 1:2. The ion $BuSnCl_2(TBPO)_3^+$ cannot produce symmetrically equivalent TBPOs, but the TBPOs may be in sufficiently similar environments to have very similar couplings to tin.

The reaction of $PhSnCl_3$ with TBP at a mole ratio of 1:0.25 shows a small amount of 1:1 adduct (doublet at −292 ppm ($J = 1230$ Hz)). At a mole ratio of 1:0.5, singlets at −27 and −61 ppm, as well as a triplet at −574 ppm ($J = 2423$ Hz), are present at room temperature. The peak at −61 ppm is almost surely due to $PhSnCl_3$ and the peak at −27 ppm can be attributed to Ph_2SnCl_2 [11]. These assignments are corroborated by the fact that the $PhSnCl_3$ peak proved to be concentration independent. The presence of Ph_2SnCl_2 is likely

Table 3
Summary of species assignments and ^{119}Sn shifts (ppm) and coupling constants (Hz) for TBP adducts of butyl and phenyl acids [a]

$BuSnCl_3$	7.7	$PhSnCl_3$	−60.6
Bu_2SnCl_2L	−182 (195)	Ph_2SnCl_2L	−285 (818)
$BuSnCl_3L$	−227 (1004)	$PhSnCl_3L$	−292 (1230)
$BuSnCl_2L_2^+$	−271 (775)	$PhSnCl_2L_2^+$	−314 (1130)
$BuSnCl_4L^-$	−442 (3000)	$PhSnCl_4L^-$	−500 (3140)
$BuSnCl_3L_2$	−473 (351)		
$SnCl_4L$	−407 (2000)		
$SnCl_4L_2$	−574 (2423)		

[a] L = TBP.

due to aryl transfer, sometimes referred to as disproportionation:

$$2PhSnCl_3 \rightarrow Ph_2SnCl_2 + SnCl_4$$

This hypothesis is supported by the triplet, which is identical in both shift and coupling to that observed in a solution of $SnCl_4$ with TBP in a 1:2 mole ratio (a peak at −407 ppm (J = ca. 2000) can be attributed to the $SnCl_4$(TBP) adduct). Thus, the $SnCl_4$ produced in the aryl transfer is therefore present as the $SnCl_4(TBP)_2$ adduct. At a mole ratio of 1:1, the species $PhSnCl_3$(TBP) (−285 ppm (d), $J = 1330$ Hz), Ph_2SnCl_2, and $SnCl_4(TBP)_2$ are present. At a mole ratio of 1:2 a large triplet at −314 ppm ($J = 1130$ Hz) and a doublet at −500 ppm ($J = 3120$ Hz) can be attributed to the presence of $PhSnCl_2(TBP)_2^+$ and *trans*-$[PhSnCl_4(TBP)]^-$, presumably produced through the mechanisms discussed above. The peak for *trans*-$[PhSnCl_4(TBP)]^-$ agrees well with the chemical shift and coupling constant reported by Colton and Dakternieks [10]. The absence of Ph_2SnCl_2 or any of its adducts, as well as adducts of $SnCl_4$, suggests that the aryl transfer is reversed at higher base concentrations. A decrease in the intensity of the $SnCl_4(TBP)_2$ and Ph_2SnCl_2 peaks at a mole ratio of 1:1.5 is consistent with this interpretation.

The aryl transfer was also noted by Colton and Dakternieks, who proposed the scheme [10]:

$$2PhSnCl_3 + 3TBP \rightarrow Ph_2SnCl_2(TBP) + SnCl_4(TBP)_2$$

Our study of the Ph_2SnCl_2 system shows that the doublet observed in the $PhSnCl_3$ system is due not to Ph_2SnCl_2(TBP), but instead to the 1:1 adduct of $PhSnCl_3$. It is important to note that the coupling of 1330 Hz for the doublet at −285 ppm is considerably larger than the coupling of 818 Hz observed for Ph_2SnCl_2(TBP).

The reaction of $PhSnCl_3$ with TBPO at a mole ratio of 1:1 and a temperature of −90 °C shows a doublet at −336 ppm ($J = 215$ Hz). At a mole ratio of 1:2, the 1:1 adduct remained and three multiplets appeared in the 1:2 adduct region at temperatures of both −90 and −110 °C. These peaks appeared at −556 (doublet of doublets, $J = 189$, 88 Hz), −580 (t, $J = 201$ Hz), and −580 ppm (t, $J = 192$ Hz) and are in good agreement with peaks observed by Colton and Dakternieks, who attributed them to three of the five possible octahedral isomers [10]. A reproducible quartet at −607 ppm ($J = 220$ Hz) may be the result of the cation $PhSnCl_2(TBPO)_3^+$. As in the butyl analog, the TBPOs may be in sufficiently similar environments to have very similar couplings to tin.

Finally, Table 3 provides a comparison of the shifts and couplings for the assigned structures. The effect of the number of chlorines on the couplings is nicely illustrated by both the butyl and phenyl series. Of course, the structural assignments were based, in part, on the assumption that the couplings are some function of the number of electronegative groups attached to tin. The greater electronegativity of chlorine than phosphorus is presumably responsible for the primary dependence on chlorine. The data in Table 3 also show what appears to be an anomalously low coupling in the Bu_2SnCl_2(TBP) adduct, also observed by Colton and Dakternieks [4]. We attribute this to the possible equatorial location of the TBP in this adduct. The difference between equatorial couplings and axial couplings has been well documented [8,9].

In summary we conclude that: (a) the organotin chloride adducts readily exchange with base or with other adducts and that this exchange can be slowed by decreasing the relative amount of base, decreasing concentrations, or by decreasing the temperature, (b) the composition of the solution changes, in some cases dramatically, with a change in the initial mole ratios of base to acid, (c) Ph_3SnCl forms a 1:1 adduct with TBP, contrary to earlier reports, (d) the diorganotin dichlorides form only 1:1 adducts with TBP, even at high base to acid ratios, but form 1:2 adducts with TBPO at mole ratios above 1:1, (e) a series of octahedral isomers can be detected for the 1:2 adducts, except for di(*t*-butyl)tin dichloride, (f) Ph_2SnCl_2 forms only 1:1 adducts with triethylphosphine oxide and TPPO, but both 1:1 and 1:2 adducts with TBPO, DMSO, and pyridine, (g) the lower dialkyltin dichlorides prefer to form 1:1 adducts (at 1:1 mole ratios) with TBP rather than TBPO, whereas diphenyltin dichloride and di(*t*-butyl)tin dichloride prefer TBPO adduct formation, (h) the reaction of $BuSnCl_3$ with TBP produces both 1:1 and 1:2 adducts, but is complicated by displacement of chloride by base and consequent ion formation, (i) the reaction of $PhSnCl_3$ with TBP leads to aryl transfer and formation of $PhSnCl_3$(TBP), Ph_2SnCl_2, and $SnCl_4(TBP)_2$ at 1:1 mole ratio, but at higher mole ratios, only ionic species derived from $PhSnCl_3$ adducts are present, and (j) the reaction of $PhSnCl_3$ with TBPO produces both 1:1 and 1:2 adducts (with visible isomers) and probably the species $PhSnCl_2(TBPO)_3^+$.

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