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A PROTON NMR STUDY OF SOLVENT EFFECTS ON THE NATURE AND REACTIVITY OF ALKALI METAL TRIMETHYLTIN DERIVATIVES

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Summary

Variable temperature NMR studies on solutions of $LiSnMe_3$ and $KSnMe_3$ indicate that an equilibrium between contact and solvent separated ion pairs occurs for these species. Solvents investigated, in order of decreasing ability to solvate the alkali metal ion are: hexamethylphosphoramide (HMPA) > 1,2-dimethoxyethane (DME) > bis(β-ethoxyethyl) ether (BEEE) > tetrahydrofuran (THF), with the equilibrium shifted strongly to the right by HMPA and strongly to the left by THF. The addition of two moles of HMPA per mole of lithium to a THF solution of $LiSnMe_3$ shifts the equilibrium strongly to the right. A shift to the right also is observed on lowering the temperature; this effect is greatest for DME solutions.

$$M,SnMe_3 \rightleftharpoons M \| SnMe_3$$

Variations in $^2J(SnCH)$ for the $MSnMe_3$ species, as well as available IR data, give evidence that the structure of the SnC_3 fragment tends increasingly towards planarity with increasing solvation of the alkali metal.

Unlike the carbon and silicon analogs, the trimethylstannyllithium species does not appear to attack the solvent on heating but rather rearranges to form $SnMe_4$ and $LiSn(SnMe_3)_3$. The rate of thermal decomposition of $LiSnMe_3$ in THF and the yield of $LiSn(SnMe_3)_3$ both are increased by the addition of 0.5 to 2 moles of HMPA per mole of lithium.

Introduction

Although many reactions of alkali metal—tin derivatives have been studied [1], there is little information which deals with the exact nature of these species in solution or in the solid state. Recently PMR data have been reported for $LiSnH_3$ [2], while two studies have appeared on $LiSnMe_3$, in which widely ranging values of $^2J(SnCH)$ were recorded [3, 4]. In the first of these studies $^2J(SnCH)$

= 14 Hz was noted for a trimethylamine solution of $LiSnMe_3$, but in the later account values of $^2J(SnCH)$ = 5.2 and 7.5 Hz were reported for THF solutions of $LiSnMe_3$ with the coupling constant dependent on the mode of preparation of the $LiSnMe_3$ [4]. After consideration of both ^1H and ^7Li chemical shift data for the THF solutions of $LiEPh_3$ (E = Si, Ge, Sn, Pb), Cox et al. [5] argued that there is a decrease in the covalent character of the Li—E bond as the atomic number of E increases.

Deductions concerning the structure of stannyllithium compounds may be drawn from comparison of the Group IV analogues of tin. Lithium alkyls form aggregates which range from dimers to hexamers in hydrocarbon solutions [6] while in coordinating solvents the degree of aggregation is reduced [7]. The silyl derivatives, $MSiPh_3$ (M = Na, K, Rb, Cs), where there is less tendency to form electron deficient bonds, have been shown to exist as contact ion-pairs in THF solution while in the same solvent $LiSiPh_3$ has been reported to exist as an equilibrium mixture of contact and solvent-separated ion pairs [8-10].

Based on these studies and those of Cox et al. [5], one would anticipate that stannyllithium species should be monomeric and likely will exist in solution as an equilibrium between contact and solvent-separated ion pairs in coordinating solvents. However, no direct evidence has appeared concerning this postulate. Furthermore, many of the normal experiments used to characterize species such as $LiCMe_3$ [11] and $LiSiMe_3$ [12] are precluded from use in characterization of $LiSnMe_3$ since this compound undergoes decomposition, even at —78°, on removal of coordinating solvent [4]. Therefore, such tin compounds must be characterized in solution.

In the present study data are presented on the behavior of $LiSnMe_3$ in several strongly coordinating solvents which provide information concerning its structure in solution. This information should aid in understanding the reactions of this reagent and related species in the formation of tin—metal bonded compounds [13] and organic substitution reactions.

Experimental

Reactions reported in this work were carried out either in a high vacuum system or in an argon-filled dry box scavenged by Na/K alloy. All NMR spectra were recorded on a Varian A-60-A spectrometer equipped with a V-6040 temperature controller. Temperatures were determined from the chemical shift differences in methanol or ethylene glycol according to the relationship reported by Van Geet [14]. Chemical shifts were determined by linear interpolation between audio frequency sidebands of the internal standard. Solvents were dried over Na/K alloy until the lasting blue color of the ketyl of benzophenone was formed (except for hexamethylphosphoramide, HMPA, which itself develops a lasting deep blue solution of alkali metal when sufficiently dry). After drying, all solvents were then distilled, degassed and stored in vacuo until used. The Sn_2Me_6 used was purchased from PCR, Inc., purified by distillation on the vacuum system, and stored in the dry box.

All alkali metal—tin species ($MSnMe_3$) were prepared in vacuo by reaction of excess lithium or potassium with solutions containing 0.25 ml of Sn_2Me_6 per 4 ml of solvent together with trace quantities of cyclopentane included as an internal standard for NMR measurements. Under these reactions conditions we found that neither diethyl ether nor *N,N,N',N'*-tetramethylethylenediamine (TMEDA) could be used successfully as solvents to produce pure $MSnMe_3$. However, when bis(β-ethoxyethyl) ether (BEEE), 1,2-dimethoxyethane (DME), hexamethylphosphoramide (HMPA) or tetrahydrofuran (THF) were used as solvents, the reaction proceeded generally as previously described for production of $LiSnMe_3$ in THF [4]. In the ethereal solvents, reaction was conducted for 2 h at −5 to −10°. In HMPA, the reaction was run for 2 h at 5° because of the high freezing point of this solvent.

A typical reaction was conducted in a Schlenk apparatus, which contained a teflon coated stirring bar, equipped with a stopcock for evacuation and access, and designed so that the reaction mixture could be filtered directly into NMR tubes or storage vessels. The reaction was started by placing 0.5 g (0.07 mol) of freshly prepared lithium chips in the reaction vessel, then 1.0 ml of Sn_2Me_6 (4.7 mol) was added by syringe, the vessel was closed and removed from the dry box and 16 ml of DME was condensed into it from the vacuum line. The apparatus was sealed off from the stopcock and the contents were carefully warmed to −10° and stirred for 2 h while the temperature was maintained between −10° to −5°. Within 10 min the solution turned greenish and then turned yellow. As the reaction proceeded, a black surface formed on the lithium and fine black sediment separated from the reaction mixture. These solid materials were removed by filtration through a medium porosity glass frit and the solutions then were stored in sealed ampules at −78°.

Purities of the compounds were determined from their NMR spectra. All solutions of $LiSnMe_3$ and $KSnMe_3$ exhibit a major peak at 0.4 ppm upfield from TMS with satellites due to tin—proton coupling clearly visible at ambient temperatures. Occasionally $SnMe_4$ appears as a minor side product (less than 5%) of the reaction.

Isolation of $LiSnMe_3$ complex

After lithium metal was placed in the side of the Schlenk tube which contained the stirring bar, 0.50 ml of Sn_2Me_6 (2.4 mol) and 3.4 ml of HMPA (20 mol) was added to the other side from a syringe. The solution was cooled with liquid nitrogen, the vessel evacuated, and 3.8 g of diethyl ether was distilled into it from the vacuum line. The reaction vessel then was warmed until the contents liquified and the solution was poured through the filter onto the lithium metal. As the reaction mixture approached −10 to −15°, the solution turned green almost instantly. After 2 h of stirring at that temperature the color had changed to brown and after 3.5 h had elapsed, the reaction mixture was worked up. Volatile components were removed on the vacuum line, leaving behind 4.0 g of a light yellow-brown solid which melted with some decomposition at 103-108°. Attempts to purify the solid by recrystallization proved unsatisfactory since the product was very soluble in donor solvents such as diethyl ether, but oiled out from toluene or cyclopentane. When the solid material was dissolved in DME or diethyl ether, the NMR spectrum showed the presence of approximately 4.5 moles of HMPA per mole of $LiSnMe_3$. The NMR spectrum of the diethyl ether solution of the solid material showed a singlet centered at δ = 2.00 ppm above cyclopentane with ^2J(SnCH) = 17.6 Hz. The isolated solid complex can be kept for weeks under an inert atmosphere at room temperature with no apparent decomposition.

Results and discussion

Properly prepared solutions of $MSnMe_3$ (M = Li or K) give rise to PMR spectra with the general appearance of those shown in Fig. 1 for $LiSnMe_3$ both in THF and in HMPA, which illustrate the extreme features of all $MSnMe_3$ spectra we have observed. Typically, an intense single line (83.9% total intensity) flanked by tin satellite peaks due to the presence of ^{117}Sn(7.6% natural abundance, I = 1/2) and ^{119}Sn(8.5% natural abundance, I = 1/2) is observed. Since the $^2J(^{117}$SnCH) and $^2J(^{119}$SnCH) are quite small and because the ratio $J(^{117}$SnCH)/$J(^{119}$SnCH) = 1.046, individual satellite peaks due to each of these isotopes usually cannot be resolved for the $MSnMe_3$ species.

Data plotted in Fig. 2 indicate the way in which proton chemical shift of $MSnMe_3$ is affected by solvent, counter ion M, and temperature. The increase in chemical shift for the methyl protons with a decrease in temperature parallels the previously measured increase in dielectric constant as a function of temperature for THF and DME [15]. Clearly the greatest effect on chemical shift as a function of temperature is shown by the DME solutions of $LiSnMe_3$. This behavior is similar to that observed for other systems in which equilibria between contact and solvent separated ion pairs have been reported [15, 16].

A plot of the coupling constant ^2J(SnCH), observed in $LiSnMe_3$, vs. temperature, given in Fig. 3, shows three additional features which are of interest: (*i*) the temperature dependence of ^2J(SnCH) is greater in the chelating ethers, DME and BEEE, than in THF, (*ii*) for DME solutions of $LiSnMe_3$, the magnitude of the coupling constant ^2J(SnCH) observed at higher temperatures approaches that observed for THF solutions of this species, while at low temperatures the magnitude of ^2J(SnCH) increases to values approaching those observed in HMPA

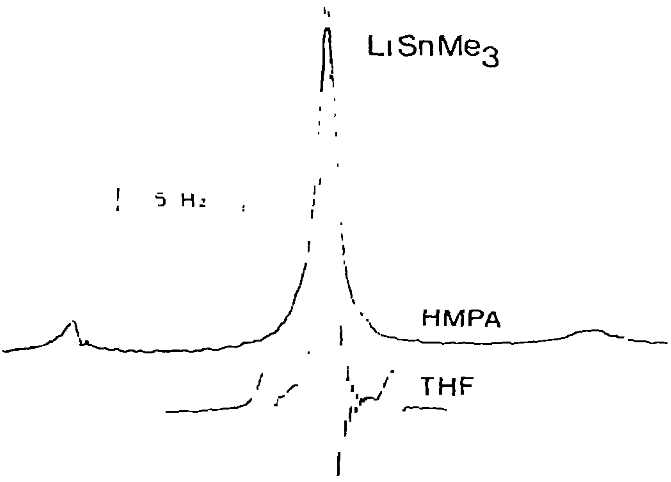


Fig. 1. The 60 MHz spectra of $LiSnMe_3$ in HMPA and THF solution showing tin—proton coupling.

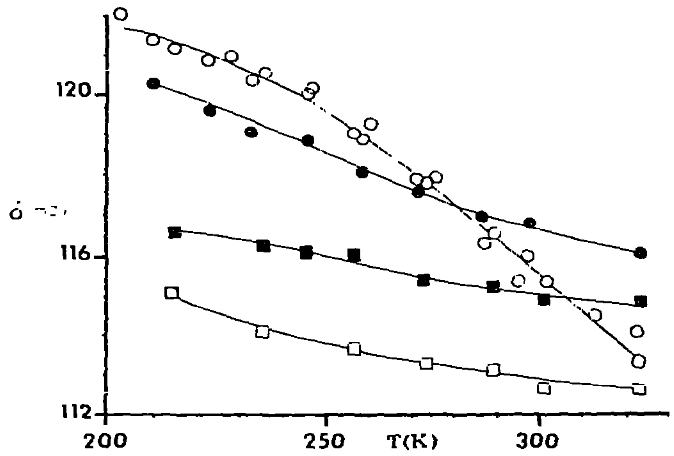


Fig. 2. Variation in the chemical shift, δ, (in Hz above internal cyclopentane) for the 60 MHz spectra of MSnMe$_3$ as a function of the temperature, counter ion (Li = ○, □; K = ●, ■) and solvent (DME = ●,○; THF = ■,□). All solutions were 0.6 *M*.

solutions, and (*iii*) in both DME and THF solvents ^2J(SnCH) increases as the temperature decreases.

From these observations one may deduce that in solution there are at least two species present in rapid equilibrium with each other and that the relative concentrations of these species are determined both by temperature and solvent. This conclusion is borne out by the changes in product distribution with temperature observed by Wiberg et al. [17], in the reaction between LiSnR$_3$and R$_3$SiCl, and also by the observed changes in the stereochemistry of the reaction between LiSnR$_3$ and alkyl halides on changing solvents from THF to DME to tetraglyme [18].

When one compares Fig. 3 and 4 the variation in ^2J(SnCH) as a function of both solvent and temperature is seen to be smaller for potassium than for lithium. Extrapolation of the plots in these figures indicates that there are three

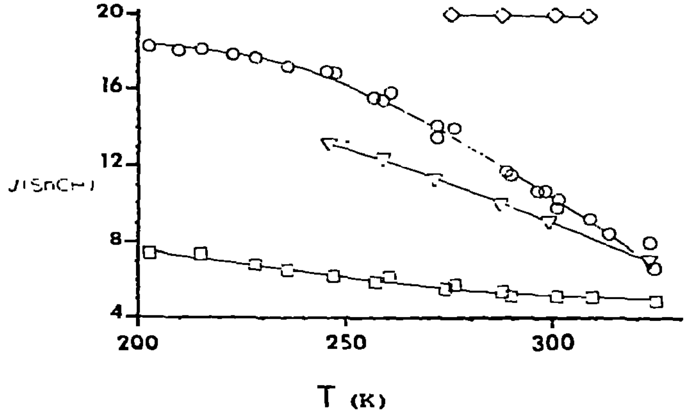


Fig. 3. Variation of the tin—proton coupling constant, ^2J(SnCH), for LiSn(CH$_3$)$_3$ as a function of temperature and solvent. (HMPA, ◇; DME, ○; BEEE, ▽; THF, □). The coupling constants are in Hz and the measurements were made on 0.6 *M* solutions.

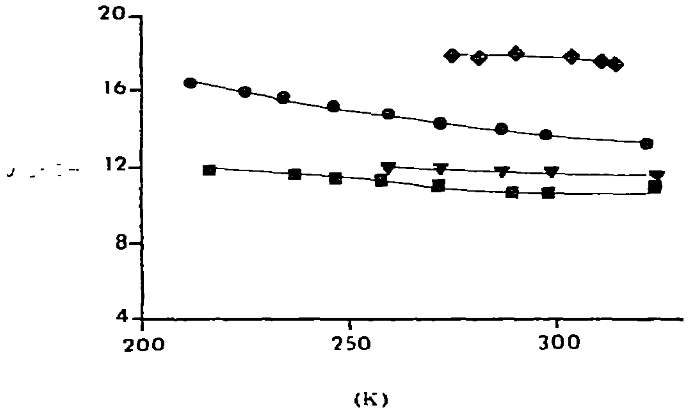


Fig. 4. Variation in the tin—proton coupling constant, ^2J(SnCH) for KSn(CH$_3$)$_3$ as a function of temperature and solvent (HMPA, ◆; DME, ●; BEEE, ▼; THF, ■). The coupling constants are in Hz and the measurements were made on 0.6 *M* solutions.

characteristic values for the coupling constants: (*i*) ^2J(SnCH) = 18-20 Hz for solvent separated ion pairs, (*ii*) ^2J(SnCH) ≈ 11 Hz for KSnMe$_3$ contact ion pairs, and (*iii*) ^2J(SnCH) ≈ 5 Hz for LiSnMe$_3$ contact ion pairs.

The difference between the coupling constants observed for the lithium and potassium contact ion pairs can be ascribed to the difference in size of the cations, and therefore of the coulombic interaction between them and the SnMe$_3^-$ moiety. The minor difference between the observed coupling constants (18 Hz for KSnMe$_3$ vs. 20 Hz for LiSnMe$_3$) indicates that a greater interaction occurs between the tightly bound lithium complex and the SnMe$_3^-$ anion than for the corresponding potassium species. This behavior is typical for systems which involve alkali metal contact ion-pair solvent-separated ion pair equilibria [19].

From the NMR data one also may deduce the relative coordinating ability of the solvents in this study as HMPA > DME > BEEE > THF. This order is similar to that reported for solutions of LiSnPh$_3$ in which it was established that the order was HMPA > DMSO > DME > TMEDA > THF [20], but differs from the established order for the coordinating ability of polyethers, R(OCH$_2$CH$_2$)$_n$OR, which normally increases with increasing *n* [21]. The greater solvating ability of DME in the present system may be rationalized readily since two molecules of either DME or BEEE would be required to form tetracoordinate lithium and the greater steric requirements of BEEE would tend to reduce the stability of the complex, thus giving rise to the observed order.

We also attempted to verify the postulated equilibria and the order of solvating ability above by using HMPA as a cryoscopic solvent [22] but could not obtain consistently reproducible results. From the data given in Table 1 and those plotted in Fig. 5, it is apparent that addition of small quantities of HMPA to 0.6 *M* LiSnMe$_3$ solutions in THF markedly affects the nature of the solvated species. The variation in NMR parameters in the system suggests solvation of the alkali metal by HMPA rather than by THF. Thus, after 2 moles of HMPA have been added per mole of lithium, the LiSnMe$_3$ has been converted from the contact ion pairs to solvent separated ion pairs. As more HMPA is added, both

TABLE 1

THE EFFECT OF HMPA ON PRODUCTS OF THE THERMAL DECOMPOSITION OF $LiSnMe_3$ in THF

J(SnCH) at 38° for $LiSnMe_3$	Mol. % of:			Heating periods[a]					
				1		1 + 2		1 + 2 + 3	
	$LiSnMe_3$	HMPA	THF	A[b]	B[c]	A	B	A	B
5.0	4.6	0	95.4	0		33		100	40
7.8	4.5	2.3	93.2	100	90				
11.2	4.35	4.9	90.7	100	90				
17.4	4.1	11.0	84.9	80		100	85	100	95
17.9	3.9	15.5	80.7	50		80		100	70
18.0	3.7	19.2	77.1	20		50		100	55
17.6	3.25	28.9	67.8	0				60	>20
17.8	2.6	42.7	54.7					10	> 5

[a] Heating periods are as follows: 1 = 2 h at 80°; 2 = 1.3 h at 94°; 3 = 17 h at 94°. [b] A = % $LiSnMe_3$ decomposed based on mol % Sn. [c] B = % Sn as $LiSn(SnMe_3)_3$.

^2J(SnCH) and $\delta(SnCH_3)$ approach a constant value. The chemical shift of the doublet due to the HMPA protons is decreased to a nearly constant value as a result of its coordination to lithium from that of free HMPA until a 2/1 ratio is reached. Then as excess HMPA (more than 2 HMPA per Li) is added, the chemical

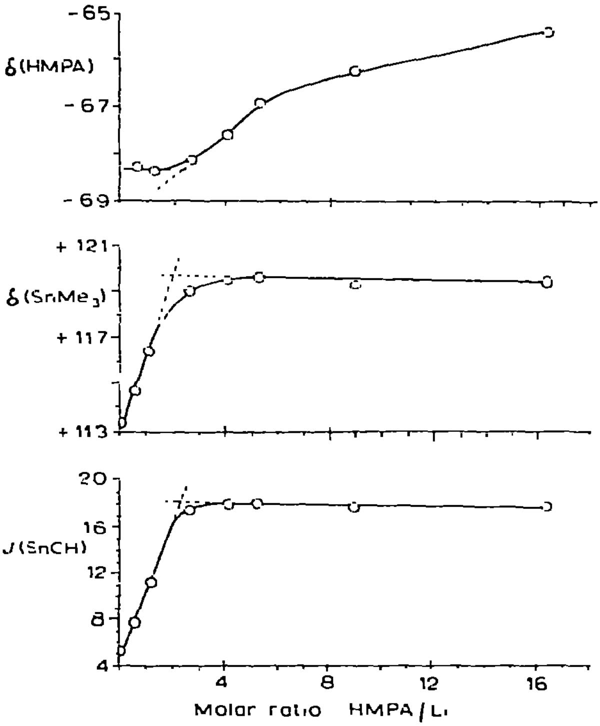
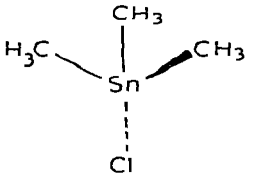


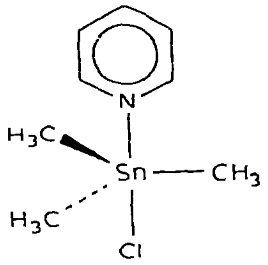
Fig. 5. Variation of the NMR parameters for $LiSn(CH_3)$ and HMPA in THF solvent as a function of the molar ratio HMPA/$LiSn(CH_3)_3$. The δ values are in Hz relative to internal cyclopentane while ^2J(SnCH) is also reported in Hz.

shift of ligand protons increases toward the value observed in pure HMPA suggesting rapid exchange between free and coordinated ligand molecules. The tendency for Li^+ to be four coordinated suggests that each HMPA molecule occupies two sites in the solvent separated ion pair to give the fully coordinated derivative in the 2/1 complex. It should be noted, however, that for fluorenyllithium it has been suggested that a solvent separated ion pair forms upon the addition of just one mole of dimethyl sulfoxide (DMSO) per mole of lithium [19]. In addition, it has been reported that HMPA acts only as a monodenate ligand in its complexes with alkylaluminum compounds [23]. Thus, it is not clear at this time what the exact nature of the coordination is in the HMPA-solvated species.

A comparison of the variations in the coupling constant ^2J(SnCH) with chemical shift shows that the absolute magnitude of the coupling constant increases as the methyl groups shift to higher fields. High field shifts of the methyl resonance on $SnMe_3$ species have been correlated with increased electron density on the tin moiety. This trend, the increase in coupling with increased electron density, is opposite to that normally observed in Me_3SnX species. It is of particular interest since the ^2J(SnCH) is normally governed by Fermi contact interactions [24] which here would suggest that the *s* character in the Sn—C bond is increased on formation of the more negatively charged tin moiety. This is opposite to the effect predicted from Bent's rule [25] which would suggest less *s* character in this bond. A similar change in coupling constant has been noted by Drago [26] who rationalized the increase in ^2J(SnCH) for Me_3SnCl on complex formation of the tin by pyridine using the following arguments. The uncomplexed tin is in a tetrahedral environment with hybridized tin as indicated in I. On complex formation, the tin becomes five coordinate with sp^3d hybridization as seen in II. The tin—carbon bonds in II are postulated to involve essentially sp^2 type tin orbitals, whereas, in I, these bonds are formed with sp^3 type tin orbitals [26]. The increase in *s* character from sp^3 to sp^2 is paralleled by large increases in ^2J(SnCH), illustrating the importance of orbital hybridization on the coupling constant.



(I)



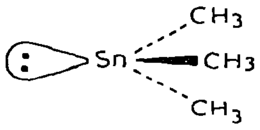
(II)

On the basis of our studies, we propose the equilibrium shown in eqn. 1 in which the solvent-stabilized contact ion pairs, III, may interact further for the solvent-separated ion pair IV. The structure of the anion in IV may be represented

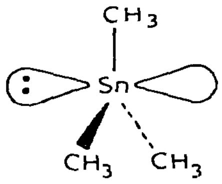
$$B_nM^+, SnMe_3^- \underset{-yB}{\overset{+yB}{\rightleftharpoons}} [B_{n+y}M] \parallel [SnMe_3]^- \qquad (1)$$

(III) (IV)

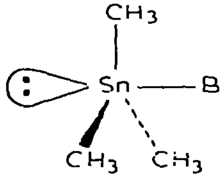
by structure V, VI, or VII. As noted previously, structure V would lead to a



(V)



(VI)



(VII)

decrease in ^2J(SnCH). Either structure VI or VII would lead to increased s character in the equatorial tin orbitals used for Sn—C bond formation and therefore would lead to increased values of ^2J(SnCH) as suggested by Drago [26]. The observed coupling constant will then be given by $J_{obs} = \Sigma t_a J_a + t_b J_b \ldots t_i J_i$ where t_i represents the time spent in environment i with coupling constant J_i.

The large increase in ^2J(SnCH) on addition of the strong base, HMPA, strongly supports formation of a planar $SnMe_3$ fragment in the presence of this complexing agent, but does not permit a choice to be made between the possible structures VI and VII.

The equilibria given adequately describe the behavior of the $LiSnR_3$ species in solution, but we also attempted to provide some information concerning the structure of the HMPA complex of $LiSnMe_3$ which was first isolated in this study. It has been shown that the appearance of two Sn—C vibrational modes is characteristic of a non-planar structure for the SnC_3 fragment while a single Sn—C vibrational mode would be characteristic for a planar arrangement [27]. Further these modes can be estimated to fall below 500 cm^{-1} from the correlation reported between ^2J(SnCH) and ν(Sn—C) [28]. The infrared spectrum of a Nujol mull of the complex showed only a single absorption at 450 cm^{-1}, which disappeared on exposure to air, that could definitely be assigned to ν(Sn—C). This observation, however, was inconclusive for structural assignment because of the other broad absorptions which occur in this region of the spectrum.

Another aspect of the chemistry of $MSnR_3$ species which has not been made clear in the past is the reactivity toward solvents. Such reactivity of the other Group IV derivatives is much better known. Lithium alkyls typically act as very strong bases by proton abstraction with ether solvents [29] but lithium silyl derivatives react with both THF and DME by nucleophilic displacement [30, 31]. One report states that tin derivatives also react by the latter route [32], but this appears to be an incorrect quotation of work previously reported by Gilman [33]. Our observations on the decomposition of $MSnMe_3$ in ethereal solvents indicate that nucleophilic displacement does not occur. In a BEEE solution of $LiSnMe_3$, for example, there is no evidence for the formation of $EtSnMe_3$ even after heating 10 h at 80°, but resonances assignable to $SnMe_4$ and $LiSn(SnMe_3)_3$ are present after such a period in this solvent as well as in THF or DME solutions. The formation of $LiSn(SnMe_3)_3$ had been observed previously as a decomposition product in the attempted isolation of $LiSnMe_3$ from THF solution at reduced temperature [4].

In order to investigate the effect of added HMPA on the decomposition of $LiSnMe_3$ in THF, the studies shown in Table 1 were carried out. Several conclusions may be drawn from the results obtained. First, there is no evidence for formation of products such as $Me_3Sn(CH_2)_4OLi$, which would result from nucleophilic attack on solvent, in any of these solutions covering wide ranges of alkali metal solvation. The main decomposition products observed are $SnMe_4$ and $LiSn(SnMe_3)_3$. Second, variation of neither the quantity of $LiSnMe_3$ decomposed nor that of relative quantities of $LiSn(SnMe_3)_3$ produced closely correspond to the changes observed in $^2J(SnCH)$. Third, as indicated by the second and third experiments, the addition of 0.5 to 2 moles of HMPA per $LiSnMe_3$ to THF stock solution strongly catalyzed formation of $LiSn(SnMe_3)_3$, yet the addition of larger quantities of HMPA appeared to slow its formation. As a result of this study, facile preparation of the $LiSn(SnMe_3)_3$ species (heating THF solution of $LiSnMe_3$ for 2 h at 80° in the presence of HMPA catalyst) is now possible. This should allow useful studies on $LiSn(SnMe_3)_3$ to be carried out in the future.

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