observations are in disagreement with those of Thilo and Friedrich,[5] who could not find any reaction between Ni($(DMG)_2$ and dimethyl sulfate, the slow decomposition observed might be due to slow decomposition of the dimethyl sulfate to more acidic products.

Brady and Muers[4] have shown that Ni$(DMG)_2$ did not yield methane with methylmagnesium iodide in amyl ether, and in organic solution[6] Cu$(DMG)_2$ does not react with methylmagnesium iodide.

All these data on reactivity of the three chelates seem to indicate first, that the hydroxyl groups in solid Cu$(DMG)_2$ are more reactive than those in Ni $(EMG)_2$ or Ni$(DMG)_2$ and, second, that in solution Cu$(DMG)_2$ is less reactive than in the solid and appears to be much like the crystalline nickel chelates. All three chelates appear to have similar strong hydrogen bonds in solution.

Nature of the OHO Bond.—The data on reactivity, coupled with the infrared evidence, would seem to give very substantial support to the prediction of Rundle and Banks[10] that the OHO bonds in Cu$(DMG)_2$ rearrange upon solution to become more strongly hydrogen bonded. The infrared data interpreted in terms of the "abnormal isotope effect"[23] indicate that solid Ni$(DMG)_2$ and Ni$(EMG)_2$ have symmetrical OHO and ODO bonds. Concerning the two different O–O bonds in solid Cu$(DMG)_2$, it is believed that both hydrogen and deuterium have asymmetrical arrangements in the longer O–O bond; whereas, hydrogen has a symmetrical and deuterium has an asymmetrical arrangement in the shorter O–O bond. Upon solution both OHO bonds in Cu$(DMG)_2$ appear to become identical, and the infrared data coupled with the unreactivity of the hydroxyl groups would seem to indicate that both deuterium and hydrogen exist in symmetrical arrangements. Thus, it would be expected that the O–O distance of Cu$(DMG)_2$ in solution should be significantly shorter than either of the O–O distances in the crystal and should be approximately the same as the O–O distances found in crystalline Ni$(DMG)_2$ and Ni$(EMG)_2$.

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Low-Frequency Infrared Studies of Some Tin(IV) Halide Adducts[1]

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The infrared spectra of the complexes $SnCl_4 \cdot 2L$ (L = CH_3CN, CH_2CHCN, C_6H_5CN), $SnBr_4 \cdot 2CH_3CN$, $SnX_4 \cdot$dipy (X = Cl, Br, I), and $SnX_4 \cdot 2DMF$ (X = Cl, Br) have been investigated in the 500–140-cm^{-1} region. The SnN stretching frequencies for the $SnCl_4 \cdot 2$(nitrile) and $SnX_4 \cdot$dipy adducts have been observed below the CsBr region. The SnCl stretching frequencies of all adducts occur in the 275–370-cm^{-1} region, whereas those of SnBr are located in the 195–250-cm^{-1} region. The assignments made in this investigation reverse those made recently by other workers for the SnCl and SnN stretching frequencies for the nitrile adducts and the SnCl and ligand vibrations for the DMF derivatives. Some special features of the spectra of $SnCl_4 \cdot 2CH_2CHCN$ in the NaCl region, particularly the observed crossover of the vinyl twist and wag frequencies, are discussed.

Introduction

Several papers have appeared in the past few years dealing with the low-frequency spectra of some nitrile adducts of stannic chloride.[2–5] Considerable controversy has arisen over the assignments of the bands below 500 cm^{-1} in the spectrum of $SnCl_4 \cdot 2CH_3CN$. Brown and Kubota[2] originally assigned the bands around 400 cm^{-1} as SnCl stretching modes, whereas those bands occurring between 350 and 300 cm^{-1} were assigned to the asymmetric and symmetric SnN stretching vibrations. Beattie, *et al.*,[3] however, showed that the assignments of Brown and Kubota were incorrect; they assigned the bands around 400 cm^{-1} as ligand vibrations (NCC bending modes) and the lower frequency bands as SnCl stretching vibrations. On the basis of a simple valence force field calculation, Beattie and Rule[4] predicted that the SnN stretching frequency for $SnCl_4 \cdot 2CH_3CN$ would occur below 265 cm^{-1}.

We have examined the low-frequency spectra of acetonitrile and acrylonitrile derivatives of $M(CO)_6$ (M = Cr, Mo, W) and showed that for the acetonitrile derivatives, the NCC bending modes occur in the 390–400-cm^{-1} region,[6] in agreement with the assignments of Beattie, *et al.*[3]

However, Aggarwal and Singh,[5] on the basis of the low-frequency infrared spectra of some amide, urea, and aminobenzoic acid adducts of Sn(IV) halides, have reversed the assignments and support those of Brown and Kubota[2] for $SnCl_4 \cdot 2CH_3CN$. It was this paper which prompted us to design some definitive experi-

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[1] Presented at the 153rd National Meeting of the American Chemical Society, Miami Beach, Fla., April 1967.

[2] T. L. Brown and M. Kubota, *J. Am. Chem. Soc.*, **83**, 4175 (1961).

[3] I. R. Beattie, G. P. McQuillan, L. Rule, and M. Webster, *J. Chem. Soc.*, 1514 (1963).

[4] I. R. Beattie and L. Rule, *ibid.*, 3267 (1964).

[5] R. C. Aggarwal and P. P. Singh, *J. Inorg. Nucl. Chem.*, **28**, 1651 (1966).

[6] M. F. Farona, J. G. Grasselli, and B. L. Ross, *Spectrochim. Acta*, **23A**, 1875 (1967).

TABLE I

INFRARED SPECTRA (500–140 CM^{-1}) OF SnX_4 ADDUCTS[a]

Compound	ν, cm^{-1}			
	SnX	SnN	Ligand	Other bands
CH_3CN			377 s	
$SnCl_4 \cdot 2CH_3CN$	366 s (360 sh), 339 vs (332 sh), 305 s (301 sh)	222 m, 207 m	412 w, 401 w, 392 w	171 s, 161 s, 156 s, 150 s
$SnBr_4$ in CH_3CN			398 m, 377 (solvent)	
CH_2CHCN			362 (Raman), 242 w	
$SnCl_4 \cdot 2CH_2CHCN$	360 s (354 sh), 340 vs (333 sh), 306 s (302 sh)	204 w, 192 w	401 w	183 m, 173 m, 155 m
C_6H_5CN			462 w, 388 sh, 380 m, 345 vw, 306 s, b, 175 m	
$SnCl_4 \cdot 2C_6H_5CN$	357 s, b,[b] 345 vs, b,[b] 339 sh, s, b,[b] 304 m[b]	248 m, 220 m	491 m, 391 vw, 160 s	
dipy			429 w, 403 s, 170 m	
$SnCl_4 \cdot$dipy	337 s, sh, 326 s, 318 s, sh, 303 vw, sh, 282 m	254 m, 207 ms	445 w, 418 m, 367 vw, 356 vw, 160 m	
$SnBr_4 \cdot$dipy	236 s, 220 ms	260 ms, 195 m	444 m, 417 m, 365 vw	
$SnI_4 \cdot$dipy	186 ms, 176 m	252 ms, 198 ms	441 w, 416 m, 361 vw	
DMF			402 w, 351 s, 319 m	
$SnCl_4 \cdot 2DMF$	340 sh, 330 vs, 313 sh		426 s, 402 vs, 386 sh	
$SnBr_4 \cdot 2DMF$	249 s, b, 236 s, b, 216 s, b		422 s, 399 vs, 388 sh	174 m

[a] Abbreviations: s, strong; m, medium; w, weak; sh, shoulder; v, very; b, broad; dipy, 2,2′-dipyridine; DMF, N,N-dimethylformamide. Values in parentheses refer to $SnCl^{37}$ bands. [b] Ligand vibrations also in this region.

ments in order to make unambiguous assignments for the bands occurring in the low-frequency spectra of these compounds.

Experimental Section

The adducts of the Sn(IV) halides were prepared as described elsewhere.[2–5,7] The compounds have all been reported to possess the *cis* structure. Rigorously anhydrous conditions were employed in the preparation and sampling of the moisture-sensitive compounds. The spectra of these compounds were recorded immediately upon preparation.

The spectra in the 600–265-cm^{-1} region were recorded on a Perkin-Elmer 221 spectrophotometer equipped with a CsBr interchange. This region was also recorded on a Perkin-Elmer 521 grating infrared spectrophotometer using an expanded scale (2×). The spectra in the CsBr region were recorded as Nujol mulls between CsBr plates. The spectra below 265 cm^{-1} were recorded on a Beckman IR-11 spectrophotometer; the samples were recorded as Nujol mulls between polyethylene disks. Precision on these spectrophotometers is ±3 cm^{-1}. The NaCl region was calibrated with polystyrene, whereas water vapor served as the calibrating substance for the low-frequency regions.

Results and Discussion

The infrared spectra of the compounds studied in this work are given in Table I.

Nitrile Derivatives.—The spectrum of acetonitrile in the CsBr region shows one band at 377 cm^{-1} which has been assigned to the NCC bending mode.[8] The molecule $SnCl_4 \cdot 2CH_3CN$ belongs to the point group C_{2v}, and simple group theoretical considerations predict three NCC bending modes to be infrared active ($A_1 + B_1 + B_2$). In fact, three weak bands are observed around 400 cm^{-1} (412, 401, 392 cm^{-1}). The adducts $SnCl_4 \cdot 2CH_2CHCN$ and $SnCl_4 \cdot 2C_6H_5CN$ show absorptions around 400 cm^{-1}, but the free ligands also show absorption near this region (362 and 380 cm^{-1} for free acrylonitrile and benzonitrile, respectively). The band at 362 cm^{-1} in acrylonitrile is Raman active, but upon coordination may become infrared active.

In order to show unambiguously that the bands in the 400-cm^{-1} region in the spectra of the nitrile adducts are ligand vibrations and are not due to SnCl stretches, the spectrum of $SnBr_4$ was recorded in the CsBr region in acetonitrile. The compound $SnBr_4 \cdot 2CH_3CN$ has been reported,[9,10] but it is stable only at low temperatures.

The complex which is formed between $SnBr_4$ and acetonitrile in solution is undoubtedly not the 1:2 addition compound, but probably is a 1:1 adduct, similar to that of $SnCl_4 \cdot 2CH_3CN$ in benzene solution.[3] Nevertheless, the spectrum in the 2300-cm^{-1} region shows a CN absorption not traceable to free acetonitrile and shifted about 33 cm^{-1} to higher frequencies. This shift to higher frequencies is characteristic of coordinated nitriles and clearly shows that some complexation has occurred between $SnBr_4$ and CH_3CN.

More important, however, is the fact that a medium-intensity band appears at 398 cm^{-1}. The spectrum of $SnBr_4$ in cyclohexane shows no absorption in this region, showing clearly that since this band is independent of the halogen, it must arise from a ligand vibration.

Further evidence showing that octahedral Sn(IV) complexes show no ν(SnCl) absorptions above 400 cm^{-1} is afforded from examination of the spectra of $SnX_4 \cdot$dipy and $SnX_4 \cdot 2DMF$ in this region. The spectra in the 350–450-cm^{-1} region of the $SnX_4 \cdot$dipy adducts (X = Cl, Br, I) are virtually identical, as are those of $SnX_4 \cdot 2DMF$ (X = Cl, Br) in the 400–425-cm^{-1} region. These bands are independent of the halogen, showing clearly that the absorptions are due to ligand vibrations and do not arise from SnCl stretching

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(7) R. C. Aggarwal and P. P. Singh, *J. Inorg. Nucl. Chem.*, **28**, 1665 (1966).

(8) R. J. H. Clark, *Spectrochim. Acta*, **21**, 955 (1965).

(9) P. Pfeiffer and O. Halperin, *Z. Anorg. Allgem. Chem.*, **87**, 335 (1914).

(10) N. A. Pushin, M. Ristic, I. Parchomenko, and J. Ubovic, *Ann.*, **553**, 278 (1942).

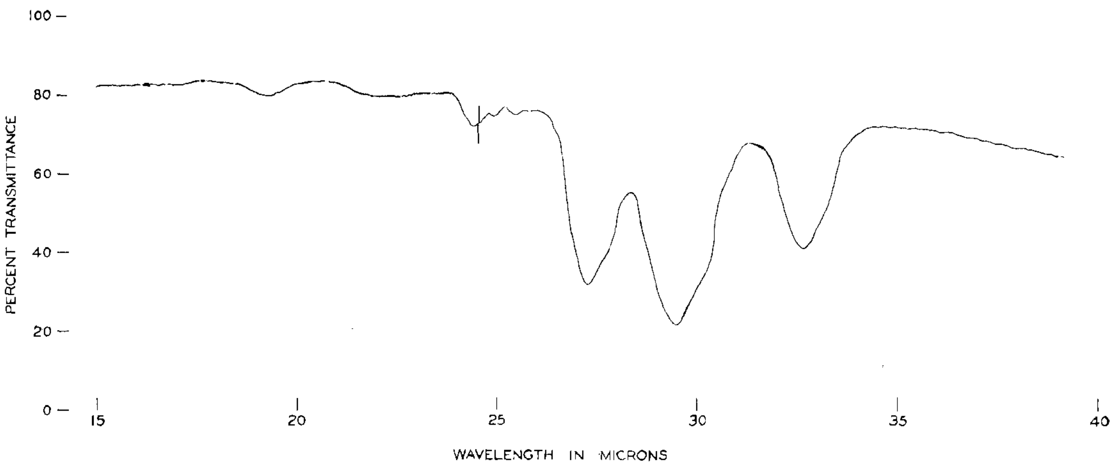

Figure 1.—Low-frequency spectrum of $SnCl_4 \cdot 2CH_3CN$.

modes. Add to the evidence presented above the fact that spectra of the acetonitrile complexes of $TiCl_4$, $TiCl_3$, VCl_4, VBr_4, and VCl_3 show absorptions in the 400–425-cm^{-1} region,[8] and it must be concluded that the assignment made above is correct.

Bands appearing in the 300–370-cm^{-1} region in the spectra of all the $SnCl_4$ adducts, but missing in the spectra of corresponding $SnBr_4$ and SnI_4 adducts, are assigned to SnCl stretching modes. An interesting feature which lends strong support for these assignments is the observation that bands in the 300–370-cm^{-1} region of the spectra of $SnCl_4 \cdot 2L$ (L = CH_3CN, CH_2CHCN) exhibit shoulders on the low-frequency side. These bands are due to $SnCl^{37}$ vibrations (relative abundance $Cl^{35}:Cl^{37}$ = 0.755:0.245). Tin has several isotopes, and the over-all effect is to broaden the SnCl stretching bands so that these shoulders are not clearly defined. Nonetheless, the bands assigned to SnCl stretching modes are not symmetrical; the asymmetry of the bands is the result of broadening on the low-frequency sides (see Figure 1). Under high resolution and an expanded scale (2×) the shoulders are more clearly observed approximately 6 cm^{-1} lower than the $SnCl^{35}$ bands. This phenomenon has been observed for MnCl stretches in the spectrum of $Mn(CO)_5Cl$, *inter alia*, and the $MnCl^{37}$ shoulder is observed 6 cm^{-1} below the $MnCl^{35}$ band.[11]

If one compares the ν(MBr)/ν(MCl) for a variety of corresponding metal bromide and chloride complexes, it is found that this ratio is consistently about 0.70–0.76.[11,12] The ratios of the ν(SnBr)/ν(SnCl) in $SnX_4 \cdot$dipy and $SnX_4 \cdot 2$DMF are found to vary between 0.70 and 0.73, lending further support to the assignments of the SnCl stretching frequencies made above.

Since all bands in the CsBr region of the spectra of the nitrile adducts of $SnCl_4$ are accounted for as due to ligand or SnCl vibrations, the SnN stretching frequencies must occur below the CsBr region, as Beattie, *et al.*, proposed. Examination of the spectra of the $SnCl_4 \cdot$2(nitrile) derivatives in the 140–265-cm^{-1} region reveals two bands for each derivative not traceable to ligand vibrations. These bands, are, therefore, assigned to the asymmetric and symmetric SnN stretching modes (Table I).

Dipyridine Derivatives.—In addition to the ligand vibrations in the 350–450-cm^{-1} region mentioned above for each complex, the bands which are dependent on the halogen are assigned to the SnX stretching vibrations as follows. For $SnCl_4 \cdot$dipy, the bands in the 280–340-cm^{-1} region are assigned to ν(SnCl); for $SnBr_4 \cdot$dipy, SnBr stretching frequencies are found in the 220–240-cm^{-1} region and SnI stretching modes are observed in the 175–190-cm^{-1} region. Comparison of ν(SnI)/ν(SnBr) in this work reveals a ratio of about 0.80. This is in accord with the value around 0.80 normally found for this ratio.[12]

Bands below the CsBr region which are essentially independent of the halogen and not traceable to free dipyridine vibrations are assigned to the asymmetric and symmetric SnN stretching modes. These are as follows: 254 and 207 cm^{-1}, $SnCl_4 \cdot$dipy; 260 and 195 cm^{-1}, $SnBr_4 \cdot$dipy; 252 and 198 cm^{-1}, $SnI_4 \cdot$dipy. These bands are correlated in the line drawing in Figure 2 where the dotted lines connect the corresponding ν(SnN) bands of the three complexes.

DMF Derivatives.—In an effort to be consistent in their assignments, Aggarwal and Singh[5] assigned the bands in the 400–430-cm^{-1} region as SnCl stretching modes in the spectrum of $SnCl_4 \cdot 2$DMF. This assignment was based on their observation that the bands in this region for $SnBr_4 \cdot 2$(1,1-dimethylurea) and $SnBr_4 \cdot$2(urea) are "absent or negligibly weak" when compared to the spectra of the corresponding $SnCl_4$ adducts. We have examined the spectra of $SnCl_4 \cdot 2$DMF and $SnBr_4 \cdot 2$DMF and found, however, that the spectra of these two complexes in the 385–425-cm^{-1} region are virtually identical. It is true that the bands of the bromide derivative are weaker than those of the chloride in that region; however, by no means are they "negligibly weak." Furthermore, the bands in the 310–340-cm^{-1} region of the spectrum of $SnCl_4 \cdot 2$DMF

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[11] R. J. H. Clark, *Record Chem. Progr.* (Kresge-Hooker Sci. Lib.), **26**, 269 (1965).

[12] F. A. Cotton, R. M. Wing, and R. A. Zimmerman, *Inorg. Chem.*, **6**, 11 (1967).

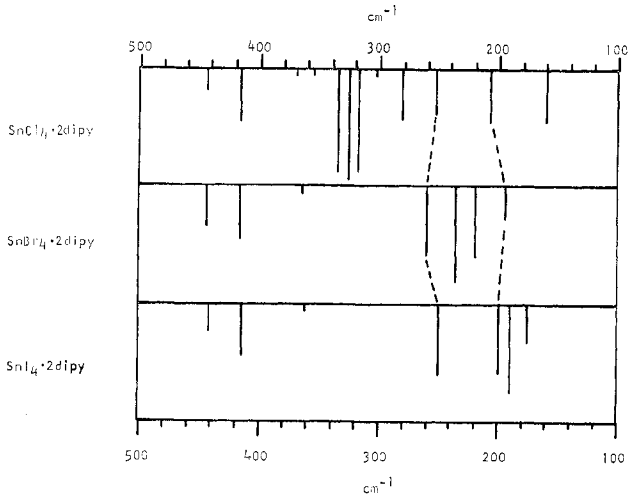

Figure 2.—Infrared spectra of tin(IV) halide adducts of 2,2'-dipyridine.

shift to lower frequencies (215–250 cm^{-1}) for the corresponding bromide. These bands are dependent on the halogen and are therefore assigned as SnX stretching modes; this assignment results in a ν(SnBr)/ν(SnCl) ratio of about 0.73, in accord with data already presented (*vide supra*).

The spectral results of the DMF derivatives of $SnCl_4$ and $SnBr_4$ render invalid all the low-frequency assignments made by Aggarwal and Singh[5] for the amide derivatives of $SnCl_4$, and it is suggested that the spectra of the amide complexes of SnX_4 be reexamined.

NaCl Region of $SnCl_4 \cdot 2CH_2CHCN$.—One special feature of the spectrum of $SnCl_4 \cdot 2CH_2CHCN$ deserves comment. Substituted vinyl compounds normally exhibit out-of-plane hydrogen deformations at about 990 and 910 cm^{-1} (twist and wag, respectively). The twist frequency has been shown to decrease with increasing inductive power of the substituent, whereas the wag frequency is sensitive to, and increases with, increasing resonance effects.[13] Associated with the wag frequency is its relatively strong first overtone, which is generally found at frequencies slightly higher than twice the fundamental frequency. An overtone is not observed for the twist vibration.

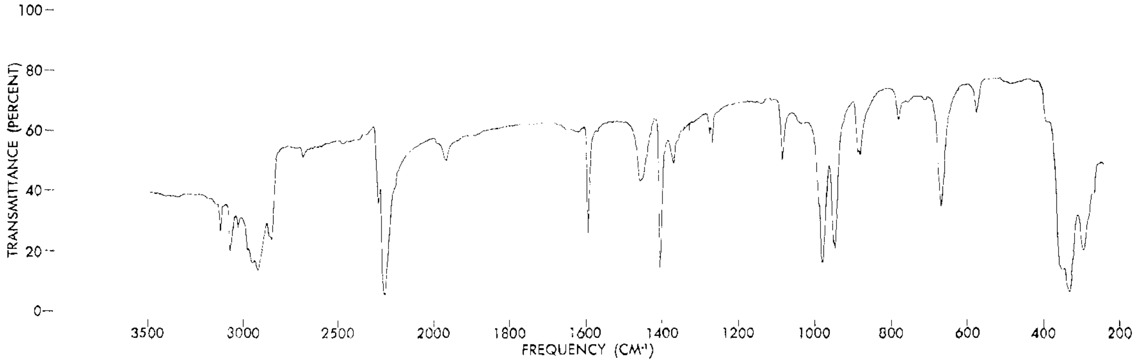

Figure 3.—Infrared spectrum of $SnCl_4 \cdot 2CH_2CHCN$.

For acrylonitrile, the resonance and inductive effects are such that the twist and wag frequencies are exactly superimposed at about 968 cm^{-1}. For coordinated acrylonitrile in the spectrum of the complex $SnCl_4 \cdot 2CH_2CHCN$, two bands are observed in the 900–1000-cm^{-1} region, at 978 and 947 cm^{-1}. Whereas it would appear that the twist and wag frequencies have returned toward their normal positions for substituted vinyl compounds when acrylonitrile is coordinated, this is apparently not the case.

If one considers the charge distribution for acrylonitrile coordinated to tin, *i.e.*, $CH_2{=}CHCN^+Sn^-$, then the inductive power of the nitrile group should be even greater in the coordinated than in the free acrylonitrile case. This suggests that the twist frequency should be lower for coordinated than for uncoordinated acrylonitrile and that the 947-cm^{-1} band is the twist frequency. The 978-cm^{-1} band must therefore be the wag frequency. Examination of the spectrum in the 1900–2000-cm^{-1} region reveals the first overtone of the wag vibration to occur at 1968 cm^{-1}, slightly more (12 cm^{-1}) than twice the 978-cm^{-1} band. It must be concluded, therefore, that a crossover between the wag and twist vibrations has occurred; the bands at 978 and 947 cm^{-1} are assigned to the wag and twist vibrational modes, respectively.

The fact that the wag vibration has increased from 968 cm^{-1} for free acrylonitrile to 978 cm^{-1} for the coordinated nitrile suggests that resonance effects have increased as well as inductive effects. This means that there must be some interaction of the filled nitrogen p orbitals with the empty 4d orbitals on tin, and the two resonance forms contributing to the electronic structure would be: $CH_2{=}CHC{\equiv}N{\rightarrow}Sn \leftrightarrow {}^+CH_2CH{=}C{=}N\overrightarrow{=}Sn^-$.

In light of the above resonance forms, it would be expected that the C=C stretching mode for coordinated acrylonitrile should occur at a lower frequency than that of free acrylonitrile. Examination of the spectrum of $SnCl_4 \cdot 2CH_2CHCN$ in the 1600-cm^{-1} region reveals a C=C stretching frequency at 1595 cm^{-1}, whereas that of free acrylonitrile occurs at 1605 cm^{-1}. All data, therefore, are consistent with the formulation and assignments made above and reveal that a cross-

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(13) W. J. Potts and R. A. Nyquist, *Spectrochim. Acta*, **15**, 679 (1959).

over of the twist and wag frequencies has occurred. The spectrum of $SnCl_4 \cdot 2CH_2CHCN$ (3500–265 cm^{-1}) is shown in Figure 3.

This same splitting of the vinyl out-of-plane hydrogen deformations has been observed in the spectrum of acrylonitrile complexes of some transition metals,[14] and the reported frequencies are similar to those found for $SnCl_4 \cdot 2CH_2CHCN$. In light of the evidence presented above, it is likely that an analogous situation exists for the transition metal complexes.

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[14] R. J. Kern, *J. Inorg. Nucl. Chem.*, **25**, 5 (1963).

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Aluminum 2,2,2-Trichloroethoxide. Preparation and Properties

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In the present study, aluminum 2,2,2-trichloroethoxide was prepared for the first time by the reaction of triethylaluminum with 2,2,2-trichloroethanol carried out in ethyl ether. The properties of aluminum 2,2,2-trichloroethoxide were studied in comparison with the usual aluminum alkoxides such as aluminum ethoxide, isopropoxide, and *t*-butoxide. By the determination of molecular weight in solution and nmr spectral studies, aluminum 2,2,2-trichloroethoxide has been shown to be monomeric in donor solvents such as dioxane and dimeric in methylene chloride and in benzene. Dissociation to monomer in donor solvents distinguishes aluminum 2,2,2-trichloroethoxide from the usual aluminum alkoxides which are in associated form even in donor solvents. A stronger accepting power of aluminum 2,2,2-trichloroethoxide as a Lewis acid has also been demonstrated by the infrared spectra of its donor–acceptor complexes with pyridine and xanthone. These two prominent characters of aluminum 2,2,2-trichloroethoxide have been ascribed, at least partly, to the electron-withdrawing effect of the three chlorine atoms of the trichloroethoxy group.

Introduction

For many years aluminum alkoxide has been studied in terms of physicochemical properties and structural features as well as the catalytic behavior in organic synthetic reactions and in the polymerization of aldehydes and alkylene oxides.

The present paper describes the studies on aluminum 2,2,2-trichloroethoxide, $Al(OCH_2CCl_3)_3$, which has not been prepared until the present study. $Al(OCH_2CCl_3)_3$ was prepared successfully by the reaction of triethylaluminum with 2,2,2-trichloroethanol. The properties of $Al(OCH_2CCl_3)_3$, particularly the degree of association in solution and the Lewis acid character, have been compared with those of the usual aluminum trialkoxides of unsubstituted alcohols.

Experimental Section

Materials.—Solvents, triethylaluminum ($Al(C_2H_5)_3$), and other reagents were carefully purified by the usual procedures. 2,2,2-Trichloroethanol, Cl_3CCH_2OH, was synthesized by the reduction of trichloroacetaldehyde (chloral) with $Al(C_2H_5)_3$ in ether solution according to the procedure given by Meerwein, *et al.*,[1] which was purified by repeated distillation, bp 66° (26 mm).

Synthesis of $Al(OCH_2CCl_3)_3$.—Under nitrogen atmosphere, 0.53 mole of Cl_3CCH_2OH was dissolved in 150 ml of ethyl ether at $-78°$, to which a solution of 0.17 mole of $Al(C_2H_5)_3$ in 50 ml of ethyl ether was added slowly with vigorous stirring and cooling during 1 hr. Then the reaction system was allowed to warm slowly to room temperature and was finally heated to reflux for 2 hr. From the reaction system a white powder precipitated, which was washed repeatedly with cold ethyl ether, dried *in vacuo*, and recrystallized three times from benzene. The yield of $Al(OCH_2CCl_3)_3$ (purified) was about 50%. *Anal.* Calcd for $C_6H_6Cl_9OAl$: Al, 5.71. Found: Al, 5.74. The trichloroethoxy group of the product was quantitatively analyzed by the determination of Cl_3CCH_2OH by vpc in the acid hydrolysis mixture of the product. The analyses of aluminum and the Cl_3CCH_2O group showed the Cl_3CCHO/Al ratio to be 2.97, supporting the formula $Al(OCH_2CCl_3)_3$. $Al(OCH_2CCl_3)_3$ decomposed without melting above 100°.

Isolation of Aluminum 2,2,2-Trichloroethoxide–Pyridine (1:1) Complex.—Under nitrogen atmosphere, 1.015 g (12.8 mmoles) of pyridine was added at room temperature to a solution of 2.19 g (4.65 mmoles) of $Al(OCH_2CCl_3)_3$ in 10 ml of benzene, and the mixture was stirred for 1 hr. Then the volatile materials were removed *in vacuo* (8×10^{-3} mm) at 50° for 10 hr. A white powdery matter was left, which was shown by nmr spectrum to be a 1:1 aluminum 2,2,2-trichloroethoxide–pyridine complex.

Nmr Measurements.—At room temperature, the nmr spectrum was measured by using a Varian 60-Mc (A-60) spectrometer. All samples were prepared under nitrogen atmosphere, and tetramethylsilane was used as the standard.

Results and Discussion

In the present study, $Al(OCH_2CCl_3)_3$ was prepared for the first time by means of the reaction of $Al(C_2H_5)_3$ with Cl_3CCH_2OH carried out in ether. The employment of ether as the reaction solvent is highly recom-

[1] H. Meerwein, G. Hinz, and H. Majert, *J. Prakt. Chem.*, **147**, 236 (1936).