stirring 0.5 hr., the solution was poured into 15 l. of water, and the product extracted with three portions of benzene. Removal of the solvent left an oil which readily crystallized, yield 105 g. (87%). An analytical sample, recrystallized from isopropanol and methanol melted at 77.5–78.0°.

Anal. Calcd. for $C_{18}H_{20}O_2Si$: C, 72.93; H, 6.80; Si, 9.48. Found: C, 72.63; H, 6.67; Si, 9.28.

The infrared spectrum showed a strong carbonyl band at 1680 cm.$^{-1}$; *p*-substitution at 842 cm.$^{-1}$; $=Si(CH_3)_2$ bands at 818 and 1248 cm.$^{-1}$; Si-C_6H_5 at 1112 and 1382 cm.$^{-1}$

Di[4-(1'-hydroxyethyl)phenyl]dimethylsilane. The sodium borohydride reduction of the diketone to the dialcohol proceeded as described above. Removal of the solvent yielded 91% of an oil which readily crystallized. An analytical sample, recrystallized from isopropanol, melted at 97–98°.

Anal. Calcd. for $C_{18}H_{24}O_2Si$: C, 71.95; H, 8.05; Si, 9.35. Found: C, 71.47; H, 8.19; Si, 9.78.

The infrared spectrum showed an associated O—H stretch strong at 3280 cm.$^{-1}$; *p*-substitution at 843 cm.$^{-1}$; $=Si(CH_3)_2$ at 816 and 1252 cm.$^{-1}$; Si-C_6H_5 at 1112 and 1390 cm.$^{-1}$

Attempted preparation of di(4-styryl)dimethylsilane. (a). By dehydration. Attempts to use the phosphorus pentoxide dehydration of Gilman *et al.*,[15] led to polymer formation. After passing the dialcohol over alumina at either 300° or 400° and 1 mm. pressure, only styrene, in the form of styrene dibromide, could be isolated from the products.

(*b*) *By Grignard synthesis.* A vigorous reaction took place on the addition of dimethyldichlorosilane to 4-styrylmagnesium chloride. Occasionally the reaction mixture polymerized to a rubbery mass at this stage. With carefully purified reagents, and addition of an antioxidant, the reaction mixture could be worked up as usual, so that solvent removal yielded a fluid liquid. Attempts to distill this material alone or with polymerization inhibitors led to immediate polymerization. The crude product consumed approximately 50% of the calculated amount of perbenzoic acid but no diepoxide could be isolated from the reaction. Addition of bromine produced a crystalline material, m.p. 134–135°, which had the following analysis: C, 41.12; H, 3.69; Br, 55.28%. In view of the apparent absence of silicon in this product, and the known case of cleavage of the phenyl-silicon bond by bromine,[16] this was not investigated further.

Acknowledgment. We wish to thank Mr. Murray Taylor of this laboratory for the microanalyses reported, and Mr. Harry Goldberg and Mrs. Katharine Haldorsen for measurements of infrared spectra.

SEATTLE, WASH.

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[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY, MASSACHUSETTS INSTITUTE OF TECHNOLOGY]

Trimethyl(iso)cyanogermane and Trimethyltin (Iso)cyanide[1]

DIETMAR SEYFERTH AND NORBERT KAHLEN

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Trimethyl(iso)cyanogermane, on the basis of its infrared spectrum and its physical and chemical properties consists of an equilibrium mixture of the normal and the isocyano isomers. It reacts with sulfur, forming trimethylisothiocyanatogermane, and with boron trifluoride to give $(CH_3)_3GeCN \cdot BF_3$. Trimethyltin (iso)cyanide has properties consistent with its formulation as a highly polar cyanide. It reacts with sulfur, giving trimethyltin thiocyanate, and does not form an adduct with boron trifluoride. The preparation of trimethyl(iso)cyanogermane and trimethyltin (iso)cyanide is described.

Trimethyl(iso)cyanosilane could have the normal cyanide structure, I, or the isocyanide structure II.

$$(CH_3)_3Si-C\equiv N(I) \quad (CH_3)_3Si-N=C(II)$$

The question of the correct structure has been the subject of much discussion in recent years[2-7] and it is now assumed, on the basis of infrared[6] and chemical[3,6] studies, that "trimethyl(iso)cyanosilane" consists of an equilibrium mixture of trimethylcyanosilane and trimethylisocyanosilane, the former isomer predominating at room temperature. We report here the results of an investigation concerning the preparation and properties of the germanium and tin analogs of trimethyl(iso)cyanosilane: trimethyl(iso)cyanogermane and trimethyltin (iso)cyanide.

Trimethyl(iso)cyanogermane. Tetra(iso)cyanogermane has been described,[6,8] as has (iso)cyanogermane.[9] No convincing proof was given for the postulated isocyanide structure of the latter. Two organogermanium (iso)cyanides, triethyl- and tri-*n*-

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[1] The "(iso)cyano" and "(iso)cyanide" nomenclature used in this paper has no structural implications when the iso prefix is in parentheses and merely indicates the presence of the CN and/or the NC grouping.

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propyl(iso)cyanogermane, have been reported,[10] but the question of their structure was not considered.

We used the method described[3] for the preparation of trimethyl(iso)cyanosilane in our synthesis of the germanium analog. Both chemical and physical properties of the germanium compound paralleled closely those reported for trimethyl(iso)cyanosilane.[3] Reaction with sulfur gave trimethylisothiocyanatogermane, $(CH_3)_3GeNCS$. This reaction, while being indicative of the presence of the (iso)cyanide structure, does not distinguish between the possibilities that the compound may be wholly the isocyanide, or that it may consist of an equilibrium mixture of both normal and isocyanide isomers. A comparison of the infrared spectrum of trimethyl(iso)cyanogermane with that of the analogous silicon compound (Fig. 1) showed that the germane also consists of a mixture of both isomers. The strong band at 2198 cm.$^{-1}$ in the spectrum of trimethyl(iso)cyanosilane has been assigned to the normal cyanide stretching frequency, while the weak band at 2105 cm.$^{-1}$ was assigned to the isocyanide bond.[6] The optical density of the $C{\equiv}N$ band was about 4.5 times as great as that of the $N{=}C$ band; these data were interpreted[6] as indicating that the normal cyano isomer predominates in a mixture of both isomers. The spectrum of trimethyl(iso)cyanogermane is quite similar, with a strong band at 2197 cm.$^{-1}$ and a much weaker absorption at 2100 cm.$^{-1}$, and the same interpretation, predominance of $(CH_3)_3Ge{-}C{\equiv}N$ in a mixture of both isomers, may be given in this case. However, on the basis of the available evidence, we do not consider the problem of the structures of trimethyl(iso)cyanosilane and trimethyl(iso)cyanogermane settled satisfactorily, and further experimental evidence seems desirable.

While sulfur appears to react only with the isocyanogermane in the mixture, continuously displacing the equilibrium $(CH_3)_3Ge{-}C{\equiv}N \rightleftharpoons (CH_3)_3Ge{-}N{=}C$ to the right, boron trifluoride apparently reacts with both isomers. When trimethyl(iso)cyanogermane and boron trifluoride diethyl etherate were mixed, stable, readily sublimable, white, crystalline $(CH_3)_3GeCN{\cdot}BF_3$ resulted. Its infrared spectrum showed two strong bands of about equal intensity at 2270 and 2230 cm.$^{-1}$ and a weak band at 2105 cm.$^{-1}$ The cause of the 2270–2230 cm.$^{-1}$ doublet is not known, but the presence of bands in the $C{\equiv}N$ and $N{=}C$ region suggests the tentative conclusion that both isomers act as donor molecules with boron trifluoride, and that formation of the N—B link results in an increase in the stretching frequency of the $C{\equiv}N$ bond. It may be noted that organic nitriles, such as acetonitrile,[11] form 1:1 adducts with boron trifluoride.

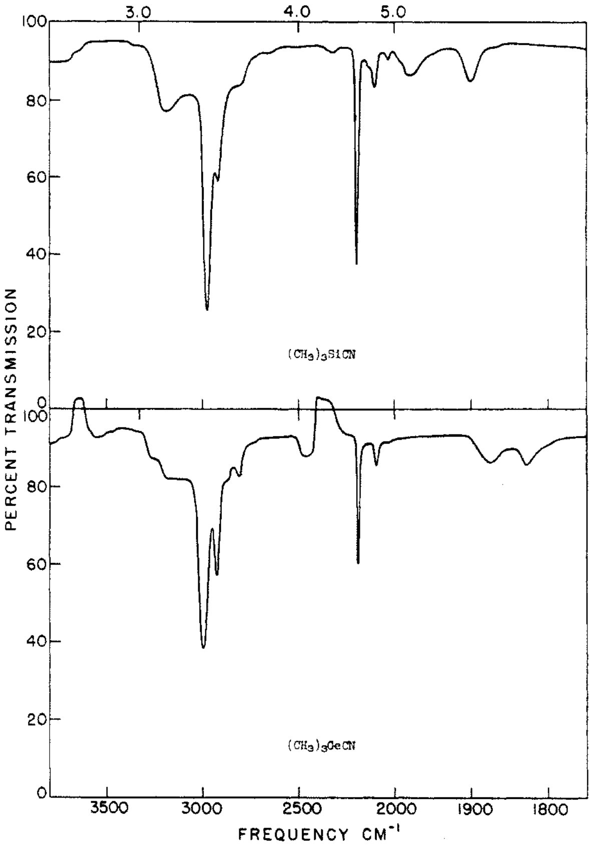

Fig. 1. Infrared spectra of trimethyl(iso)cyanosilane (liquid smear) and trimethyl(iso)cyanogermane (chloroform solution)

Their infrared spectra have been examined.[12] An increase in the $C{\equiv}N$ stretching frequency similar to the one observed by us was noted when acetonitrile ($\nu_{C{\equiv}N}$ = 2248 cm.$^{-1}$) formed an adduct with boron trifluoride ($\nu_{C{\equiv}N}$ = 2359 cm.$^{-1}$). Also, a second band in the $C{\equiv}N$ region was observed in the case of the acetonitrile adduct. On the other hand, we found that organic isonitriles, such as ethyl and *tert*-butyl isonitrile, react with boron trifluoride to form brown tars, probably polymerization products of the isonitrile. Even a catalytic quantity of boron trifluoride etherate seemed sufficient to cause polymerization of ethyl isonitrile. In contrast to trimethyl(iso)cyanogermane, trimethyl(iso)cyanosilane reacts with boron trifluoride to give trimethylfluorosilane and polymeric $(BF_2CN)_n$ as final products.[7b] These apparently result from the decomposition of the initially formed, unstable $(CH_3)_3SiCN{\cdot}BF_3$. (Iso)cyanosilane and (iso)cyanogermane show a similar difference in their behavior toward boron triufloride, (iso)cyanosilane giving monofluorosilane,[7b] and (iso)cyanogermane yielding the stable adduct $H_3GeCN{\cdot}BF_3$.[9]

Trimethyltin(iso)cyanide. The first organotin (iso)cyanide, triethyltin (iso)cyanide, was prepared

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by Cahours[13] one hundred years ago by the reaction of silver cyanide with triethyltin bromide, and since then by other investigators[14,15] using different methods. We obtained trimethyltin (iso)cyanide, a new compound, by two different procedures: by the silver cyanide method, using benzene as a solvent, and by the reaction of aqueous potassium cyanide with trimethyltin iodide in ether, a procedure reported by Luijten and van der Kerk.[15] The products obtained by both methods were shown to be identical. Trimethyltin (iso)cyanide also reacts with sulfur, incorporating one atom of sulfur into the molecule. However, its physical properties indicate that it is much more polar than the silicon and germanium analogs, and therefore this reaction cannot be taken as conclusive evidence for the presence of the isocyanide structure, since it is known[16] that ionic cyanides react readily with sulfur at higher temperatures to form thiocyanates. Indeed, the trimethyltin(iso)cyanide-sulfur product was identical with trimethyltin thiocyanate prepared from trimethyltin chloride and sodium thiocyanate in ethanol. Additional indirect evidence that in the case of trimethyltin(iso)cyanide we are dealing with a molecule of much less covalent character is given by its failure to form a molecular addition compound with boron trifluoride. The infrared spectrum of trimethyltin(iso)cyanide shows one band in the $C{\equiv}N$ region at 2175 cm.$^{-1}$ and none in the isocyanide region. This does not permit us to distinguish between a covalent and a highly polar cyanide, nor does the absence of a second band in the $N{=}C$ region allow us to rule out the possible presence in very low equilibrium concentration of an (iso)cyanide isomer. Indeed, the isolation in very low yield of $(CH_3)_3Sn{-}N{=}C{-}Fe(CO)_4$ from the reaction of trimethyltin (iso)cyanide with iron pentacarbonyl showed that at least at 100° some $(CH_3)_3Sn{-}N{=}C$ was present.[17a] However, the chemical and physical properties of trimethyltin (iso)cyanide are more in line with those of a highly polar compound, $(CH_3)_3Sn^{\delta+}\text{-}CN^{\delta-}$, rather than those of a mixture of covalent isomers as in the case of the analogous silicon and germanium compounds. Evidence obtained by others also points to a polar cyanide structure for organotin cyanides. Thus, the preparation of triethyltin (iso)cyanide using the action of aqueous potassium cyanide on triethyltin bromide[15] is not consistent with the hydrolytically sensitive covalent isocyanide structure. Also the report by Beermann and Hartmann[17b] that triphenyltin (iso)cyanide in liquid ammonia forms weak complexes with cyanide ion of the type $Na[(C_6H_5)_3Sn(CN)_2]$ speaks for an ionic or at least highly polar structure for organotin cyanides.

This difference between seemingly analogous compounds of silicon and germanium on one hand, and of tin on the other is not unusual. A few examples may serve to illustrate this point. Thus it has been found[18] that the carbonyl stretching frequency in the infrared spectrum of organosilicon acetates is at 1715 cm.$^{-1}$, only slightly lower than the analogous absorption in organic esters. On the other hand, the carbonyl frequency in organotin esters is found in the region characteristic for acetate salts (1580 cm.$^{-1}$). As other examples may be cited the great difference in physical and chemical properties of organosilicon[19] and organotin[20] fluorides, the differences in the reactivity of organosilicon and organotin hydrides,[21] and the hydrolytic behavior of dimethyldichlorosilane,[22] dimethyldichlorogermane,[23] and dimethyltin dichloride.[24]

EXPERIMENTAL[25]

Starting materials. Trimethyliodogermane. The method of Lesbre and Mazerolles[26] was used in this preparation. To 41 g. (0.309 mole) of tetramethylgermane in a three necked flask equipped with a stirrer, a Dry Ice condenser, and a thermometer, was added 78.3 g. (0.309 mole) of iodine and 1 g. of aluminum powder. An exothermic reaction commenced. The temperature was kept at 40° by external cooling, and the reaction mixture was stirred at this temperature for 5 hr. In this time the originally brown solution became nearly colorless. Filtration was followed by fractional distillation to give methyl iodide and 60.5 g. (80%) of trimethyliodogermane, b.p. 57–57.5° at 55 mm., n_D^{24} 1.5159.

Anal. Calcd. for C_3H_9IGe: C, 14.73; H, 3.71. Found: C, 14.56; H, 3.68.

The colorless iodide was very light sensitive, rapidly becoming yellow on standing in daylight.

Trimethyltin iodide was prepared by iodine cleavage of tetramethyltin in benzene solution, using a previously

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described[27] method. Distillation gave an 88.3% yield of $(CH_3)_3SnI$, b.p. 69–70° at 14 mm.

Preparation of the (iso)cyanides. Trimethyl(iso)cyanogermane. To a stirred solution of 58.5 g. (0.239 mole) of trimethyliodogermane in 220 cc. of dry benzene was added in portions during 0.5 hr. 50 g. (0.37 mole) of dry silver cyanide. A mildly exothermic reaction, accompanied by formation of yellow silver iodide, was observed. The mixture was heated at reflux for 2.5 hr., cooled, and filtered. The residue was washed well with benzene, and the washings added to the filtrate. After removal of the benzene, distillation of the remaining liquid resulted in a fraction boiling at 150° which crystallized immediately. The translucent white needles, m.p. 38–38.5°, thus obtained liquefied immediately when in contact with moist air. They were soluble in ether, chloroform, and acetone. Yield: 24.2 g. (70.5%).

Anal. Calcd. for C_4H_9NGe: C, 33.43; H, 6.31; N, 9.75. Found: C, 33.66; H, 6.39; N, 9.66.

Trimethyltin (iso)cyanide. In a similar manner 42.5 g. (0.32 mole) of silver cyanide was added to a solution of 78.5 g. (0.27 mole) of trimethyltin iodide in 400 cc. benzene. The mixture was refluxed for 3 hr. and stirred at room temperature overnight. Filtration was followed by washing of the residue with benzene. The combined filtrate and washings were evaporated, giving 4 g. of white solid. Extraction of the filtration residue in a Soxhlet extractor with chloroform gave very light yellow solid. Recrystallization from chloroform of both solid fractions resulted in 31.5 g. (61.5%) of trimethyltin (iso)cyanide, m.p. 184.5–186° (in a sealed tube filled with nitrogen).

Anal. Calcd. for C_4H_9NSn: C, 25.31; H, 4.78; N, 7.38; Sn, 62.54. Found: C, 25.11; H, 4.98; N, 7.59; Sn, 62.51. Trimethyltin (iso)cyanide is sparingly soluble in benzene and ether, soluble in chloroform and very soluble in acetone. The substance has a distinctly unpleasant odor, but less intense than that of its germanium analog.

The method of Luijten and van der Kerk[15] was also used to prepare this compound. Four grams (20%) of white needles of trimethyltin (iso)cyanide, which after recrystallization from a chloroform-ether mixture melted at 183–184.5°, were obtained from 0.1 mole trimethyltin iodide. Mixed melting point with trimethyltin (iso)cyanide prepared by the silver cyanide method: 183°.

Anal. Found: C, 24.90; H, 4.77; N, 7.26.

Infrared spectra of both samples, measured in chloroform solution, were superimposable.

Reactions with sulfur. Trimethyl(iso)cyanogermane. Nine grams (0.063 mole) of trimethyl(iso)cyanogermane was heated to 170° under nitrogen and 2.02 g. (0.063 mole) of sulfur was added, small portions at a time. After each addition the sulfur was allowed to dissolve completely before the next portion was added. During 5 hr. the temperature was increased to 185°. A clear, colorless liquid remained upon completion of the reaction. Distillation gave 10 g. (90.5%) of trimethylisothiocyanatogermane, b.p. 64–66° at 3 mm., 191.5–193° at atmospheric pressure, n_D^{22} 1.4960. A small amount of sulfur remained as a distillation residue.

Anal. Calcd. for C_4H_9NSGe: C, 27.96; H, 5.16; N, 7.97; S, 18.23. Found: C, 27.45; H, 5.36; N, 8.26; S, 18.06.

Since it has been shown conclusively that the product of the reaction between trimethyl(iso)cyanosilane and sulfur is trimethylisothiocyanatosilane,[28] it was assumed that our product was trimethylisothiocyanatogermane.

Trimethyltin (iso)cyanide. Sulfur (0.450 g., 0.014 mole) and trimethylin (iso)cyanide (2.5 g., 0.0133 mole) were heated under nitrogen for 1 hr. at 150–160°. The melt slowly darkened and a brown solid deposited. The cooled reaction mixture was extracted well with chloroform. Evaporation of the solvent and recrystallization of the residue from benzene gave 1.5 g. (51%) of white needles of trimethyltin thiocyanate, m.p. 105.5–106°. Sublimation raised the m.p. to 108.5°.

Anal. Calcd. for C_4H_9NSSn: C, 21.65; H, 4.09; N, 6.31; S, 14.44. Found: C, 21.82; H, 4.05; N, 6.26; S, 14.28.

Trimethyltin thiocyanate. A method previously described[29] was used to prepare trimethyltin thiocyanate in 86% yield from trimethyltin chloride. Recrystallization from benzene followed by sublimation at reduced pressure gave pure material, m.p. 108–108.5°.

Anal. Found: C, 21.83; H, 4.04; N, 6.45; S, 14.68.

The compound obtained from the trimethyltin (iso)cyanide-sulfur reaction was shown to be identical with the product from this reaction by mixed melting point 108°. The infrared spectra of both compounds, measured in chloroform solution, were identical in all respects. Trimethyltin thiocyanate is easily soluble in chloroform and acetone, difficultly soluble in ether.

Reactions with boron trifluoride. Trimethyl(iso)cyanogermane. A solution of 1.2 g. (8.35 millimoles) of trimethyl(iso)cyanogermane in 1.19 g. (8.35 millimoles) of redistilled boron trifluoride diethyl etherate was allowed to stand at room temperature for 2 hr. The ether then was removed *in vacuo* at 25°, leaving a residue of white crystalline $(CH_3)_3GeCN \cdot BF_3$. The compound, m.p. 85–87° (sealed tube, under nitrogen) sublimes readily *in vacuo* at 40°, is soluble in acetone and ether, less soluble in benzene, and immediately liquefies in moist air. Yield: 1.5 g. (85%).

Anal. Calcd. for $C_4H_9F_3NBGe$: C, 22.71; H, 4.29; N, 6.62. Found: C, 22.81; H, 4.47; N, 6.74.

The compound's infrared spectrum was measured in chloroform solution.

Trimethyltin (iso)cyanide. To 1 g. (5.3 millimoles) of trimethyltin (iso)cyanide was added 3 g. (21 millimoles) of boron trifluoride diethyl etherate. The tin compound dissolved completely. The solution was evaporated after 4 hr., leaving as residue only unchanged trimethyltin (iso)cyanide. The same experiment carried out at 70° during 12 hr. resulted in decomposition of the tin compound, giving a viscous brown oil.

Ethyl isonitrile. Seven-tenths grams of ethyl isonitrile (0.013 mole) was cooled with liquid nitrogen and then allowed to warm slowly. At its melting point, 1.2 g. of boron trifluoride diethyl etherate was added. An immediate, exothermic reaction resulted in a brown tar. A similar tar formation was observed when a drop of boron trifluoride diethyl etherate was added to a dilute solution of ethyl isonitrile in benzene. *tert*-Butyl isonitrile reacted in a similar fashion, but not as vigorously. These reactions were not investigated further.

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CAMBRIDGE 39, MASS.

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