in plane deformation vibration,[22] which shifts to lower frequencies as the weight of the metal atom increases. As this shift is relative to the central metal atom, this band is useful as a characteristic vibration frequency. A corresponding band shift has been observed in the infrared spectra of the phenyl halides.[23]

Young, *et al.*,[24] have described phenyl–silicon vibrations at 1429 and 1110 cm.$^{-1}$. These authors regard the absorption at 1429 cm.$^{-1}$ to be characteristic of the phenyl–silicon structure. This band, which is also present in phenyl compounds of the other IVth group elements, does, however, not shift as it should were it indeed characteristic of a specific phenyl–metal structure. Moreover, the band at 1429 cm.$^{-1}$ is situated in the region where aliphatic vibrations occur, which is apparent upon comparison of the spectra of tetraethyltin and phenyltriethyltin (Fig. 4). Since there are no interfering vibrations around 1100 cm.$^{-1}$ and as has already been noted the frequency of the absorption in this region is specifically dependent on the nature of the metal atom, the perturbed phenyl vibrations in the area between 1050 and 1120 cm.$^{-1}$ may be successfully used for the characterization of molecules containing different phenyl-IVth group metal bonds.[1,2]

A corresponding picture is presented by the compounds containing vinyl groups where the $=CH_2$ out-of-plane bending vibration is perturbed by the adjacent metal atom. This results in a characteristic band shift in the region of 940 to 960 cm.$^{-1}$ (Figs. 1 and 2). The following values are tabulated: triphenylvinylsilane 960 cm.$^{-1}$, diphenyldivinylsilane 960 cm.$^{-1}$, triphenylvinylgermane 952 cm.$^{-1}$, diphenyldivinylgermane 952 cm.$^{-1}$, triphenylvinyltin 950 cm.$^{-1}$, diphenyldivinyltin 950 cm.$^{-1}$ and triphenylvinyllead 942 cm.$^{-1}$. Since the position of this band is relative to the central metal atom it is useful as a characteristic vibration frequency.

In the compounds containing allyl groups the out-of-plane hydrogen deformation absorptions are noticed at 894 and 930 cm.$^{-1}$ characteristic of $CH_2=CHR$ where R must contain at least one carbon. No shift is noticed going from one metal atom to another.

The C–H stretching vibration for triphenylmethane has been proposed by Fox and Martin[25] to occur at 2890 cm.$^{-1}$, that of Si–H in the case of triphenylsilane has been reported at about 2135 cm.$^{-1}$,[26] a shift of 755 cm.$^{-1}$. The corresponding Ge–H frequency is reported to be about 2040 cm.$^{-1}$ [27] while the Sn–H frequency as observed in aliphatic tin hydrides is about 1820 cm.$^{-1}$.[28] The spectrum of triphenyltin hydride which shows the tin-hydrogen absorption occurring at 1825 cm.$^{-1}$ is presented in Fig. 4. In all cases the absorption is strong. Because the hydrogen atom is so light relative to the attached atom, it is obvious that as the weight of the adjacent atom increases the vibrational shift should decrease markedly.

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[22] D. H. Whiffen, *J. Chem. Soc.*, 1350 (1956).

[23] R. R. Randle and D. H. Whiffen, *Trans. Faraday Soc.*, **52**, 9 (1956); A. R. Katrizky and J. M. Lagonski, *J. Chem. Soc.*, 4155 (1958).

[24] C. W. Young, *et al.*, THIS JOURNAL, **70**, 3758 (1948).

[25] J. J. Fox and A. E. Martin, *Proc. Royal Soc.* (*London*), **A175**, 208, 234 (1940).

[26] A. Kaplan, THIS JOURNAL **76**, 5580 (1954).

[27] R. West, *ibid.*, **75**, 6080 (1953).

[28] D. R. Lide, Jr., *J. Chem. Phys.*, **19**, 1605 (1951); R. Mathis Noel, *et al.*, *Compt. rend.*, **243**, 251 (1956).

[CONTRIBUTION FROM THE INSTITUTE FOR ORGANIC CHEMISTRY T.N.O., UTRECHT, HOLLAND]

Reactions of Phenyltin Hydrides with Vinyl and Allyl Derivatives of the IVth Main Group Elements

BY MALCOLM C. HENRY[1] AND JAN G. NOLTES

RECEIVED APRIL 16, 1959

The reactions of triphenyltin hydride and diphenyltin dihydride with vinyl and allyl derivatives of silicon, germanium, tin and lead have been studied. Organometallic compounds containing two and three metal atoms, these being various combinations of tin and silicon or germanium, have been obtained. Vinyl and allyl compounds of lead appeared to be reductively cleaved by phenyltin hydrides with formation of metallic lead. The infrared spectra of the addition compounds are presented and discussed. Separate absorption bands characteristic of each different metal atom present are shown to be useful for identification of these types of compounds.

The formation of tin-carbon bonds by the addition of organotin hydrides to compounds containing olefinic double bonds was first reported in 1956[2] and has been the subject of considerable study at our Institute since that time.[3a,b] Such addition reactions proceed in excellent yield in the absence of any added solvent or catalyst, especially when the double bond is activated by the presence of neighboring electron-attracting substituents. It is noteworthy that attempts at synthesis of organotin adducts using hydrocarbon solvents and peroxides or ultraviolet irradiation as a catalyst were unsuccessful,[4] whereas with the same react-

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[1] U. S. Army Research and Engineering Command, Natick, Mass.; work done in Utrecht under the auspices of a Secretary of the Army Research Fellowship.

[2] G. J. M. van der Kerk, J. G. A. Luijten and J. G. Noltes, *Chemistry & Industry*, 352 (1956).

[3] (a) G. J. M. van der Kerk, J. G. Noltes and J. G. A. Luitjen, *J. Appl. Chem.*, **7**, 356 (1957); (b) G. J. M. van der Kerk and J. G. Noltes, *ibid.*, **9**, 106 (1959).

[4] F. Fuchs and H. Gilman, *J. Org. Chem.*, **22**, 1009 (1957).

ants, namely, "triphenyltin hydride and *n*-octene-1," the expected adduct was obtained in good yield by carrying out the reaction in the absence of solvent or catalyst.[3a]

In this paper uncatalyzed reactions of organotin hydrides with various vinyl and allyl derivatives of silicon, germanium, tin and lead will be described. These were carried out in an attempt to synthesize compounds containing more than one tin atom as well as compounds containing silicon, germanium or lead in addition to tin.

Addition reactions of organometallic hydrides of silicon and germanium to allyl derivatives of silicon, germanium and tin leading to the formation of organometallic compounds containing more than one metal atom separated by a bridge of three carbon atoms have been described in the literature. The results are far from unequivocal. Using peroxide catalysis and a hydrocarbon solvent triphenylgermane reacts almost quantitatively with triphenylallylsilane[5] as does the same hydride with triphenyallylgermane[6] although in somewhat lower yield. No product was, however, isolated from the same reaction with triphenylallyltin.[7] Under the same conditions only reaction of triphenylsilane with triphenylallylsilane[5] afforded an addition product (1%), whereas with triphenylallylgermane[6] and triphenylallyltin[7] no products were isolated.

The reaction of triphenyltin hydride with the monovinyl compounds triphenylvinylsilane, triphenylvinylgermane and triphenylvinyltin gave the expected addition products I in good yield[8]

$$(C_6H_5)_3SnH + (C_6H_5)_3MCH{=}CH_2 \longrightarrow (C_6H_5)_3SnCH_2CH_2M(C_6H_5)_3$$

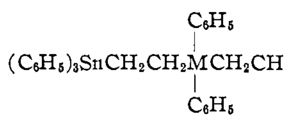
M = Si, Ge, Sn

The reaction between triphenyltin hydride and triphenylvinyllead yielded hexaphenyldistannane and metallic lead. Whenever one of the vinyl- or allyllead compounds was treated with a phenyltin mono- or dihydride the formation of metallic lead was noticed after a short heating period. The first traces of metallic lead formed appeared to catalytically accelerate the reductive degradation of the organolead compound. Other examples of reduction reactions involving triphenyltin hydride[9] and diphenyltin dihydride[10] have recently been reported.

The corresponding reactions between triphenyltin hydride and the monoallyl compounds, when compared to those of the corresponding vinyl compounds, gave rather surprising results. With triphenylallyltin the expected addition product, 1,3-bis-triphenylstannylpropane, was isolated only in small yield[3b]; triphenylallylgermane and triphenylallylsilane yielded no products; and triphenylallyllead was reduced to metallic lead. Apparently the double bond in the vinyl compounds is activated by the neighboring triphenylmetal groups, presumably by a conjugative mechanism involving d_π–p_π bonding of the metal-to-carbon bond. The inactivation of the double bond in the allyl compounds is at present not well understood.

The reaction of triphenyltin hydride with di-phenyl divinylsilane and diphenyldivinylgermane in the molar ratio 2:1 afforded the expected addition products containing three metal atoms (II) in good yield

$$2(C_6H_5)_3SnH + (C_6H_5)_2M(CH{=}CH_2)_2 \longrightarrow$$



$$(C_6H_5)_3SnCH_2CH_2\overset{C_6H_5}{\underset{C_6H_5}{M}}CH_2CH_2Sn(C_6H_5)_3 \quad \text{II}$$

M = Si, Ge

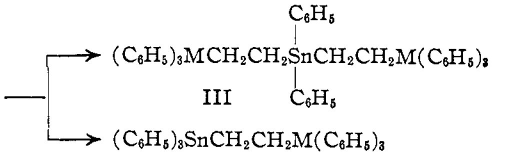
Reaction of two moles of triphenyltin hydride with diphenyldivinyltin afforded only 1,2-bis-triphenylstannylethane. This indicates that the two reactants must have entered into an exchange reaction involving either vinyl–hydrogen or vinyl–phenyl exchange. The resulting triphenylvinyltin reacts with the excess triphenyltin hydride to yield the isolated product.

The reactions of two molecules of triphenyltin hydride with the diallyl derivatives gave the expected results. No reaction was observed with diphenyldiallylsilane or diphenyldiallylgermane. However, reaction of triphenyltin hydride with diphenyldiallyltin afforded triphenylallyltin indicating that again either allyl–hydrogen or allyl–phenyl exchange must have occurred. Here the exchange product as a result of its low reactivity toward triphenyltin hydride could be isolated (compare the reaction with diphenyldivinyltin).

Finally reactions of diphenyltin dihydride with two molar equivalents of the monovinyl compounds were carried out, which gave abnormal results. Instead of the compounds containing three metal atoms (III), products of type I previously obtained by the reaction of triphenyltin hydride with the monovinyl compounds were isolated

$$(C_6H_5)_2SnH_2 + 2(C_6H_5)_3MCH{=}CH_2 \longrightarrow$$

M = Si, Ge, Sn



$$\longrightarrow (C_6H_5)_3MCH_2CH_2\overset{C_6H_5}{\underset{C_6H_5}{Sn}}CH_2CH_2M(C_6H_5)_3 \quad \text{III}$$

$$\longrightarrow (C_6H_5)_3SnCH_2CH_2M(C_6H_5)_3 \quad \text{I}$$

Occurrence of vinyl–hydrogen exchange may be ruled out in the case of M being silicon or germanium, since the exchange product triphenylsilane or triphenylgermane, respectively, could never give rise to the isolated products. It can not however be ruled out in the case of M being tin. Phenyl–hydrogen exchange leading to the formation of triphenyltin hydride, which thereupon reacts with the excess monovinyl compound of silicon, germanium or tin, may account for the reaction products. An alternative possibility is that the intermediately formed triphenyltin hydride results from disproportionation of diphenyltin dihydride according to

$$2(C_6H_5)_2SnH_2 \longrightarrow (C_6H_5)_3SnH + C_6H_5SnH_3$$

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[5] R. H. Meen and H. Gilman, *J. Org. Chem.*, **22**, 684 (1957).

[6] H. Gilman and C. W. Gerow, THIS JOURNAL, **79**, 342 (1957).

[7] H. Gilman and J. Eisch, *J. Org. Chem.*, **20**, 763 (1955).

[8] For a discussion on the structure of addition products of organotin hydrides see J. G. Noltes and G. J. M. van der Kerk, "Functionally Substituted Organotin Compounds," Tin Research Institute, Greenford, England, 1958, p. 20.

[9] J. G. Noltes and G. J. M. van der Kerk, *Chemistry & Industry*, 294 (1959).

[10] H. G. Kuivila and O. F. Beumel, Jr., THIS JOURNAL, **80**, 3798 (1958).

Benzene and metallic tin also formed in these reactions may well originate from decomposition of the phenyltin trihydride. The uncatalyzed disproportionation of diphenylsilane to triphenylsilane and phenylsilane recently has been reported.[11]

Infrared Absorption Spectra.—The occurrence of a characteristic absorption band in the region of 1050–1120 cm.$^{-1}$ in the infrared spectra of IVth main group aromatic compounds has been previously discussed.[12,13] This band, a perturbed phenyl vibration, shifts characteristically to a longer wave length depending on whether the metal atom is silicon, germanium, tin or lead. In Fig. 1 the infrared spectra in the region of 8.5–9.5 μ of five compounds containing more than one metal atom are presented.

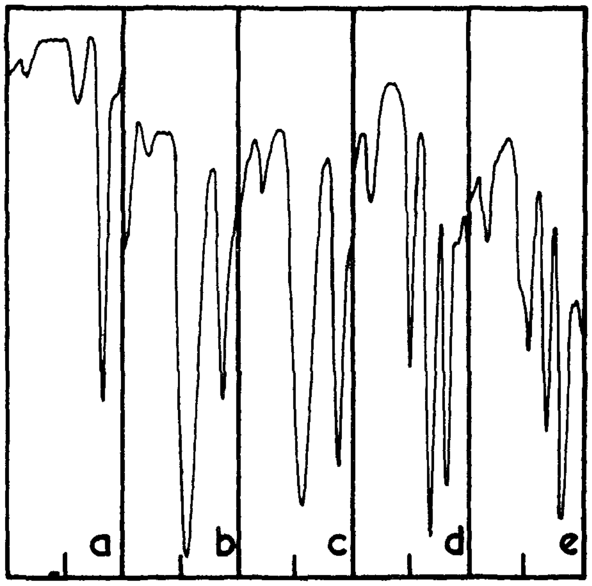

Fig. 1.—Infrared absorption spectra (8.5–9.5 μ) of: a, $Ph_3SnCH_2CH_2SnPh_3$; b, $Ph_3SnCH_2CH_2SiPh_3$; c, Ph_3Sn-$CH_2CH_2Si(Ph_2)CH_2CH_2SnPh_3$; d, $Ph_3SnCH_2CH_2GePh_3$; e, $Ph_3SnCH_2CH_2Ge(Ph_2)CH_2CH_2SnPh_3$.

It appears from these spectra that a compound containing two different metal atoms will display two distinctive phenyl absorption bands in its infrared spectrum. In 1,2-bis-triphenylstannylethane (a) only one phenyl vibration can be seen in this region, namely at 1068 cm.$^{-1}$. 1-Triphenylstannyl-2-triphenylsilylethane (b) and diphenyl-bis-[2-(triphenylstannyl)-ethyl]-silane (c) show absorption characteristic of phenyl bound to silicon at 1100 cm.$^{-1}$ and of phenyl bound to tin at 1070 cm.$^{-1}$. Similarly, in the spectrum of 1-triphenylstannyl-2-triphenylgermylethane (d) and diphenyl-bis-[2′-(triphenylstannyl)-ethyl]-germane (e) the phenyl(germanium) and phenyl(tin) vibrations (at 1085 and 1070 cm.$^{-1}$, respectively), can clearly be distinguished. It is interesting to note that the relative intensity of these bands depends on the number of metal atoms present. For example (b) contains one silicon atom and one tin atom each of which is attached to three phenyl groups. The band characteristic of phenyl bound to silicon is considerably stronger in intensity than the corresponding vibration characteristic of phenyl bound to tin. Comparison with the spectrum of (c) which compound contains two tin atoms attached to a total of six phenyl groups and one silicon atom attached to two phenyl groups reveals that the latter vibrations have almost the same intensity here. A comparison between the spectra of (d) and (e) shows an analogous phenomenon.

It is apparent that the perturbed phenyl vibrations may serve as a valuable aid to structure identification and confirmation of this type of compound.

Experimental[14]

Starting Materials.—Triphenyltin hydride and diphenyltin dihydride were prepared just prior to carrying out the addition experiments using established procedures.[15] The preparation of the vinyl and allyl derivatives has been published elsewhere.[13]

1-Triphenylstannyl-2-triphenylsilylethane.—A mixture of 5.0 g. (0.017 mole) of triphenylvinylsilane and 7.0 g. (0.020 mole) of triphenyltin hydride was heated *in vacuo* at 80° for 4.5 hr. The reaction mixture had then almost solidified. The mixture was dissolved in chloroform. Upon addition of acetone, 5.8 g. (54%) of glittering platelets with m.p. 203–205° settled out of solution. Recrystallization from dimethylformamide raised the m.p. to 207–208°.

Anal. Calcd. for $C_{38}H_{34}SiSn$: C, 71.60; H, 5.39; Si + Sn, 23.02. Found: C, 71.53; H, 5.70; Si + Sn, 23.13.

1-Triphenylstannyl-2-triphenylgermylethane.—A mixture of 6.0 g. (0.018 mole) of triphenylvinylgermane and 7.6 g. (0.022 mole) of triphenyltin hydride was heated *in vacuo* at 70° for 6 hr. The solid reaction product was recrystallized from benzene yielding 3.78 g. (32%) of needles with m.p. 210–211°.

Anal. Calcd. for $C_{38}H_{34}GeSn$: C, 66.92; H, 5.03; Ge + Sn, 28.05. Found: C, 66.91; H, 5.22; Ge + Sn, 28.31.

Bis-1,2-triphenylstannylethane.—A mixture of equimolecular amounts of triphenylvinyltin (7.5 g., 0.02 mole) and triphenyltin hydride (7.0 g.) was heated *in vacuo* at 70° for 4 hr. The solid reaction product was recrystallized from benzene and from acetone yielding 7.1 g. (50%) of crystals with m.p. 208–209°. A mixed m.p. with an authentic sample[3] showed no depression.

Reaction of Triphenyltin Hydride with Triphenylvinyl lead.—Equimolecular amounts of triphenyltin hydride (0.90 g., 0.0025 mole) and triphenylvinyllead (1.16 g.) were heated together under nitrogen at 70°. The color of the mixture gradually turned from colorless to canary yellow, until suddenly a lead mirror was formed on the bottom of the flask. The contents of the flask were then taken up in hot benzene. The metallic lead was isolated by filtration and weighed 0.26 g. (52%). The filtrate was taken to dryness and the residue recrystallized from acetone. Crystals with m.p. 231–233° were obtained. A mixed m.p. with an authentic sample of hexaphenyldistannane showed no depression, yield 0.55 g. (60%).

Diphenyl-bis-[2′-(triphenylstannyl)-ethyl]-silane.—A mixture of 4.72 g. (0.02 mole) of diphenyldivinylsilane and 14.0 g. (0.04 mole) of triphenyltin hydride was heated *in vacuo* at 80° for 4.5 hr. The reaction mixture had then nearly solidified. The solid was recrystallized from ligroin and from dimethylformamide; yield 16.0 g. (86%), m.p. 143–144°.

Anal. Calcd. for $C_{52}H_{48}SiSn_2$: C, 66.56; H, 5.15; Si + Sn, 28.30. Found: C, 66.67; H, 5.39; Si + Sn, 28.57.

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(11) H. Gilman and D. H. Miller, *J. Org. Chem.*, **23**, 326 (1958).

(12) J. G. Noltes, M. C. Henry and M. J. Janssen, *Chemistry & Industry*, 298 (1959).

(13) M. C. Henry and J. G. Noltes, THIS JOURNAL, **82**, 555 (1960).

(14) All reactions involving triphenyltin hydride and diphenyltin dihydride were carried out in an atmosphere of dry oxygen-free nitrogen or *in vacuo*. Melting points (Kofler block) are uncorrected. Compounds containing different metal atoms were analyzed gravimetrically by determining the total weight of the metallic oxides obtained on ignition. Only the total metal content is given. Infrared spectra were obtained with a Perkin–Elmer Infracord spectrophotometer.

(15) G. J. M. van der Kerk, J. G. Noltes and J. G. A. Luijten, *J. Appl. Chem.*, **7**, 366 (1957).

Diphenyl-bis-[2′-(triphenylstannyl)]-germane.—Upon mixing 2.81 g. (0.01 mole) of diphenyldivinylgermane and 7.0 g. (0.02 mole) of triphenyltin hydride under nitrogen an exothermal reaction occurred. The reaction mixture became viscous and after two hours had become a solid glass. After heating at 70° for 1 hour, the solid was twice recrystallized from ligroin; white needles with m.p. 139–142°, yield 6.5 g. (66%).

Anal. Calcd. for $C_{52}H_{48}GeSn_2$: C, 63.53; H, 4.92; Ge + Sn, 31.54. Found: C, 63.87; H, 5.01; Ge + Sn, 31.66.

Reaction of Triphenyltin Hydride with Diphenyldivinyltin.—When a mixture of 6.0 g. (0.018 mole) of diphenyldivinyltin and 12.6 g. (0.036 mole) of triphenyltin hydride was heated under nitrogen at 90°, the reaction mixture gradually solidified. Gas was evolved and metallic tin separated. The solid was recrystallized from benzene, acetone and dimethylformamide, respectively. A solid with m.p. 202–203° identified as 1,2-bis-triphenylstannyl ethane was obtained. A mixed m.p. with an authentic sample was not depressed and infrared spectra were superimposable; yield 6.8 g. (52%).

Anal. Calcd. for $C_{38}H_{34}Sn_2$: Sn, 32.61; mol. wt., 728. Found: Sn, 32.73; mol. wt. (Rast), 710.

Reaction of Diphenyltin Dihydride with Triphenylvinylsilane.—A mixture of 3.43 g. (0.012 mole) of triphenylvinylsilane and 1.65 g. (0.006 mole) of diphenyltin dihydride was heated in an atmosphere of nitrogen at 70° for 5 hr. and at 100° for 12 hr. After two hours at the latter temperature evolution of gas set in and metallic tin began to separate. The gas was collected by condensation and shown to be benzene. The reaction mixture was taken up in light petroleum and the insoluble part was filtered (1.7 g. which melted at *ca.* 180°). Solution in hot dimethylformamide and filtration yielded 170 mg. (24%) of metallic tin. Water was added dropwise to the dimethylformamide solution and the precipitated solid, after filtration, recrystallized from benzene–petroleum ether yielding 0.92 g. (24%) of 1-triphenylstannyl-2-triphenylsilylethane with m.p. 201–204°. A mixed m.p. with an authentic sample showed no depression and the infrared spectra were superimposable.

Reaction of Diphenyltin Dihydride with Triphenylvinylgermane.—A mixture of 7.60 g. (0.023 mole) of triphenylgermane and 3.03 g. (0.011 mole) of diphenyltin dihydride was heated under nitrogen at 70° for 5 hr. and at 100° for 12 hr. Gas (identified as benzene) was evolved and metallic tin separated. From the semi-solid reaction product there was isolated 0.26 g. (20%) of metallic tin and 2.7 g. of a solid with m.p. *ca.* 190°. After several recrystallizations from ligroin and dimethylformamide the m.p. was raised to 208–210°. The solid was identified as 1-triphenylstannyl-2-triphenylgermylethane. A mixed m.p. with an authentic sample showed no depression and the infrared spectra were superimposable; yield 1.60 g. (21%).

Reaction of Diphenyltin Dihydride with Triphenylvinyltin.—Gas evolution (benzene) and formation of metallic tin was observed upon heating a mixture of 10.60 g. (0.028 mole) of triphenylvinyltin and 3.85 g. (0.014 mole) of diphenyltin dihydride under nitrogen at 70° for 5 hr. and at 100° for 12 hr. From the semi-solid reaction mixture 2.75 g. (27%) of 1,2-bis-triphenylstannylethane with m.p. 201–204° was isolated. A mixed m.p. with an authentic sample showed no depression and the infrared spectra were superimposable.

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[CONTRIBUTION FROM THE INSTITUTE FOR ORGANIC CHEMISTRY T.N.O., UTRECHT, HOLLAND]

Synthesis of Some New Six-membered Organometallic Ring Systems

BY MALCOLM C. HENRY[1] AND JAN G. NOLTES

RECEIVED APRIL 16, 1959

New organometallic aliphatic heterocycles, *viz.*, the 1-stanna-4-silacyclohexane, the 1-stanna-4-germanacyclohexane and the 1,4-distannacyclohexane ring systems have been synthesized by means of addition reactions involving diphenyltin dihydride and the appropriate unsaturated reactants. Complexes of these compounds with several cyclic solvents were isolated.

Cyclic organotin compounds were described, in 1917, by Grüttner, *et al.*,[2] who prepared cyclopentamethylenedimethyltin and cyclopentamethylenediethyltin by the reaction of pentamethylenedimagnesium bromide with the appropriate dialkyltin dihalide. This method has been applied to the synthesis of cyclopentamethylene derivatives of the other IVth group elements silicon,[3] germanium[4a] and lead.[4b] Although the number of known organotin compounds recently has considerably increased, only one new heterocyclic system containing tin in the ring has been reported since that time. Kuivila, *et al.*,[5] prepared heterocycles in the dibenzocyclo heptadiene series containing tetra- and bivalent tin in the seven-membered ring. This method of synthesis involved the reaction of *o,o′*-dilithiodibenzyl with diphenyltin dichloride or stannic chloride.

In connection with a program involving the synthesis of organometallic compounds containing one of the IVth group elements silicon, germanium or lead in addition to tin,[6] reactions of diphenyltin dihydride with diphenyldivinyl derivatives of silicon, germanium and lead were studied. Since triphenyltin hydride had been found to add to these compounds in good yield,[6] such reactions might be expected to yield organometallic polymers of type II.

Reaction of diphenyltin dihydride with diphenyldivinylsilane afforded a crystalline product (m.p. 134–135°) with molecular weight (Rast determination) and analytical data calculated for the mono-addition product I (M = Si). Examination of the infrared spectrum revealed the presence of C_6H_5Sn and C_6H_5Si in a 1:1 ratio; there appeared to be no absorption bands which could be attributed to Sn–H

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[1] U. S. Army Research and Engineering Command, Natick, Mass.; work done in Utrecht under the auspices of a Secretary of the Army Research Fellowship.

[2] G. Grüttner, E. Krause and M. Wiernik, *Ber.*, **50**, 1549 (1917).

[3] A. Bygden, *ibid.*, **48**, 1236 (1915); G. Grüttner and M. Wiernik, *ibid.*, **48**, 1473 (1915).

[4] (a) R. Schwarz and W. Reinhardt, *ibid.*, **65**, 1743 (1932); (b) G. Grüttner and E. Krause, *ibid.*, **49**, 2666 (1916).

[5] H. G. Kuivila and O. F. Beumel, Jr., THIS JOURNAL, **80**, 3250 (1958).

[6] M. C. Henry and J. G. Noltes, *ibid.*, **82**, 558 (1960).