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# DIBROMO-SUBSTITUTED PROPYL- AND CYCLOHEXYL-TIN COMPOUNDS

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Functionally-substituted organotin compounds, containing substituents such as CN, COOH, $COOCH_3$ and $CONH_2$, are prepared by adding aryl- or alkyl-tin hydrides to the appropriate olefinic derivatives at 70–100°. According to Noltes and Van der Kerk[1], the reaction proceeds by an ionic mechanism. However, Neumann *et al.*[2] used the radical initiators—azobis(isobutyronitrile), $(CH_3)_2C(CN)N{=}NC(CN)(CH_3)_2$, and benzyl hyponitrite, $C_6H_5CH_2ON{=}NOCH_2C_6H_5$,—to catalyze the addition of alkyltin mono-, di- and tri-hydrides to various olefines. It therefore seems likely that both radical and ionic mechanisms are operating, depending on the participating unsaturated compound and whether an alkyl- or aryl-tin hydride is used in the reaction.

The organotin hydrides are prepared by reduction of the corresponding organotin halides with lithium aluminium hydride in ethereal solution[3], or with amalgamated aluminium and water[1].

It has now been shown by the author that organotin halides themselves, *e.g.* diphenyltin dibromide, (I), and triphenyltin bromide, (II), can easily be added to allylic bromides in the presence of pyridine.

Pyridine forms coordination compounds with (I) and (II); they have been prepared and investigated by Pfeiffer *et al.*[4] and Kocheshkov[5]. These complexes react with allyl bromide in ethanol at 80° with good yields of the addition products which contain dibromopropyl groups. The yields were even better using pyridine as reaction medium, as the adducts decompose slowly in boiling ethanol. When the allylic bromide is 3-bromocyclohexene, only pyridine is suitable as solvent. In ethanol, the dibromocyclohexyltin derivatives are apparently decomposed on boiling under reflux for 4 h. For example, diphenyltin dibromide reacted in this way, gives a product which on recrystallizing from ethanol, melts above 300°. This is probably diphenyldiethoxytin but it was not investigated further. Mack and Parker[6] state that the analogous dimethoxytin compound decomposes at 270° and has a tendency to polymerize. Triphenyltin bromide, reacts with 3-bromocyclohexene in ethanol under similar conditions, to give only an impure product which after recrystallization from ethanol still melted in the range 120–160°.

Figure 1 shows a reaction scheme of the possible additions of diphenyltin dibromide to allyl bromide and 3-bromocyclohexene. The four adducts, from diphenyltin dibromide (IV, VII) and triphenyltin bromide (IX, X), in which every bromine atom has been displaced by a propyl- or cyclohexyl-group, were isolated as

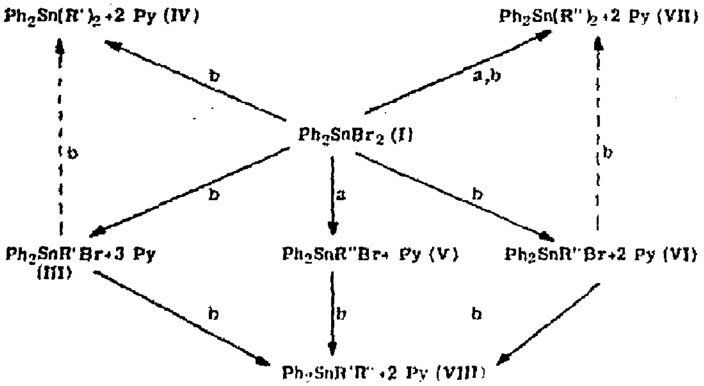


Fig. 1. Additions of diphenyltin dibromide to allyl bromide and 3-bromocyclohexene in the presence of pyridine. R' = 2,3- or 2,6-dibromocyclohexyl, R" = $CH(CH_2Br)_2$ or $CH_2CHBrCH_2Br$, Py = pyridine, a = ethanol and b = pyridine as reaction medium.

their pyridinium salts, containing one molecule of pyridine/dibromo-substituted propyl- or cyclohexyl-group. The question as to whether a 1,3-dibromoisopropyl- or a 1,2-dibromo-n-propyl-group is linked to the tin atom, has not yet been resolved, but will be the subject of further study. The monoadducts, III and VI, prepared in pyridine as solvent, contain 3 and 2 molecules of pyridine, respectively. The mixed addition compound, dibromopropyl-dibromocyclohexyldiphenyltin, VIII, on the other hand has 2 moles of pyridine/mole, as have the two single adducts, IV and VII.

The monopyridinate of dibromopropyldiphenyltin bromide, V, is easily prepared by boiling stoichiometric amounts of the three reactants in ethanol under reflux. It adds to 3-bromocyclohexene in pyridine at 50° to produce the mixed adduct, VIII.

Compounds IV and VII could obviously be prepared from the monoadducts, III and VI, in a similar way to that described above but since these stepwise additions were not actually carried out, they have been indicated by broken arrows in Fig. 1.

In contrast with the above mentioned additions, cyclohexene does not react with diphenyltin dibromide, (I), in dry pyridine on heating for 18 h at 100°, the dipyridinate of (I) being recovered in theoretical yield. Allyl alcohol also does not react; no adduct was formed after heating in pyridine at 50–80° during 1 h (91.6% of I, 2 $C_5H_5N$ was recovered). Triphenyltin bromide did not add on to allyl alcohol or cyclohexene on boiling under reflux for a long period in ethanol in the presence of pyridine. The pyridinate of triphenyltin bromide, melting at 92–93°, was obtained in both cases.

## INFRARED SPECTRA

The IR-spectra were recorded on a Perkin–Elmer infrared spectrophotometer 21 with a sodium chloride prism. The spectrum of tetraphenyltin was taken both in admixture with potassium bromide in a pressed disc, and on a saturated solution in carbon tetrachloride. In the first case, sharp bands were obtained only between 700 and 1500 $cm^{-1}$. The solution gave marked peaks also in the region 1500–3100 $cm^{-1}$. The positions of the different bands are seen in Table 1.

TABLE 1

IR-SPECTRUM FOR TETRAPHENYLTIN IN KBr AND $CCl_4$

| *Frequency (cm$^{-1}$)* | *Intensity* | *Absorption type* |
|---|---|---|
| 3060 | m | =C–H stretching vibration |
| 1480 | m | C=C in-plane vibration |
| 1430 | s | C=C in-plane vibration |
| 1070 | s | effect of tin on Ph-group vibration |
| 1020 | m | C–H in-plane deformation |
| 992 | m | C–H in-plane deformation |
| 725 | s | C–H out-of-plane deformation |
| 692 | s | C–H out-of-plane deformation |

Noltes *et al.*[7] have investigated the spectra of the tetraphenyl-derivatives of the Group IV-elements and of tetraethyltin. They observed that a strong, sharp peak between 1110 and 1050 cm$^{-1}$ shifted in the series $Ph_4Si$ (1105 cm$^{-1}$), $Ph_4Ge$ (1090), $Ph_4Sn$ (1070) and $Ph_4Pb$ (1058). The band could not be detected in the spectra of tetraphenylmethane and tetraethyltin. The authors state that the metal–carbon linkage should not have any vibration absorption in the rock-salt region. They suggest that the band results from interference by the central metal atom on the vibration of the phenyl group.

The spectrum of diphenyltin dibromide in carbon tetrachloride and potassium bromide has the same absorption frequencies as tetraphenyltin. The same is true for triphenyltin bromide, which has eight sharp peaks.

Furthermore, $Ph_2SnBr_2$ shows six absorption bands of medium intensity in the 1650–2000 cm$^{-1}$ region, originating from overtones and combinations of frequencies for C–H out-of-plane deformations in monosubstituted benzene rings. These bands can also be observed in the spectra of $Ph_4Sn$ and $Ph_3SnBr$ but are weaker in intensity. Apparently the tin–bromine bond does not absorb between 700 and 3000 cm$^{-1}$. Johnson and Holum[8] mention briefly that the infrared spectra of triphenyltin chloride and bromide in chloroform are identical in the 650–3500 cm$^{-1}$ region, which confirms the observation.

The dipyridinate of diphenyltin dibromide shows, apart from some pyridine bands closely related to the characteristic frequencies of the benzene ring, three strong peaks. Two of these, at 1605 and 1215 cm$^{-1}$, are typical for pyridine and its substituted derivatives. The third band at 1450 cm$^{-1}$ is useful for the identification of coordination compounds between pyridine and aryltin halides containing one or two tin–nitrogen linkages. Gill *et al.*[9] investigated the infrared spectra of a large number of complexes between pyridine and the heavy metal halides (*e.g.* $NiI_2$ and $CuBr_2$). They found peaks at 1440–1460 cm$^{-1}$, which shifted to 1418–1422 cm$^{-1}$ in the spectra of pyridinium-hydro-salts [*e.g.* $PyHBF_4$, $(PyH)_2MnCl_4$ and $(PyH)_2CoCl_4$]. This pronounced frequency shift indicates that the absorption band originates from a stretching vibration of the nitrogen–metal (hydrogen) bond.

In the spectra of the two adducts of allyl bromide, VIII and IX, two bands at 1035 and 1005 cm$^{-1}$ which were present in the spectrum of $Ph_2SnBr_2 \cdot 2\ C_5H_5N$ and are connected with the C–H in-plane-deformation in pyridine, had disappeared. Instead, a rather strong absorption band was found at 950 cm$^{-1}$, probably of the same origin as the two peaks above. The shift to a lower frequency, if this is really so, must depend on the different electron density in coordinated and quaternized pyridine.

For the two addition compounds of 3-bromocyclohexene, IV and X, these deformation bands seem to have shifted further, to 900 $cm^{-1}$. As the frequency changes, the intensity decreases markedly compared with the phenyl band at 730 $cm^{-1}$. The monoadducts of 3-bromocyclohexene, III, and allyl bromide, VI, with diphenyltin dibromide, again absorb weakly at 1035 and 1005 $cm^{-1}$, and also have rather strong bands at 1450 $cm^{-1}$. The latter peak was attributed to Sn–N stretching vibrations in coordination compounds.

The three addition compounds of 3-bromocyclohexene, III, IV and X, also have absorption bands at 775–790 and 670 $cm^{-1}$. For all three compounds, the first band is rather weak, and the latter of medium intensity. They presumably are derived from the equatorial and axial C–Br bonds, respectively.

## PROTON MAGNETIC RESONANCE SPECTRA

The spectra were recorded with a Varian A 60 spectrometer operating at a frequency of 60 Mc/sec. The compounds were dissolved in carbon tetrachloride, deuterochloroform or dimethylsulfoxide (DMSO), and tetramethylsilane (TMS) was used as internal reference. The chemical shifts are reported as $\delta$-values corresponding to the definition $\delta = 10^6(H_{TMS} - H)/H_{TMS}$.

It has usually been difficult to assign the signals of the phenyl- and pyridine-protons to the various positions in the rings, with the exception of the pyridine $\alpha$-protons.

In the spectrum of diphenyltin dibromide I, in carbon tetrachloride, the proton signals appeared in the region 7.50–7.92 ppm. The proton frequency of benzene is 7.27 ppm. Consequently, the shift is in the same direction as when benzene is substituted with electron-attracting groups such as the nitro-group[10]. The similar shielding effects imply that the $SnBr_2$-substituent would act as a *meta*-directing group in an electrophilic substitution.

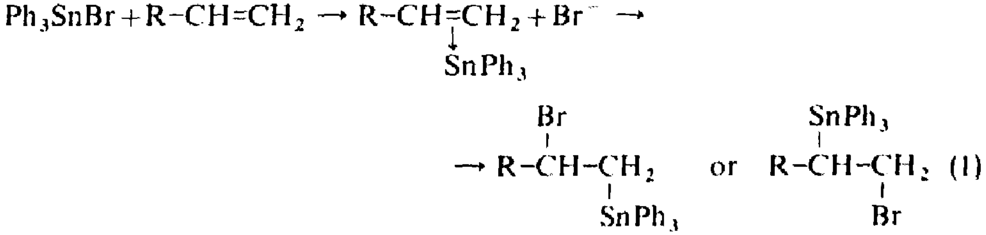
The dipyridinate of I was dissolved in both $CDCl_3$ and DMSO. In the first solvent, two shift regions were observed, *viz.* 8.92–9.00 and 7.32–8.09 ppm. From the integrated intensities, giving a proton ratio of 1 : 4, it may be concluded that the lower field strength region originates from the four $\alpha$-protons of pyridine, and that the latter region must be assigned to the six $\beta$- and $\gamma$-protons and to the ten phenyl protons. Schneider *et al.*[11], who analyzed the pyridine spectrum, found that the shift of the $\alpha$-protons (8.50 ppm) differs greatly from the shifts of the $\beta$- and $\gamma$-protons (6.99 and 7.36 ppm). When the strongly polar solvent DMSO was used, the $\alpha$-proton region was altered somewhat to 8.65–8.85 ppm. The limits of the signals of the sixteen residual protons of the complex were extended to 7.17 and 8.23 ppm. Integration showed the latter region to contain two multiplets with a proton ratio of 6 : 10, but their complicated nature resisted any calculation of the coupling constants.

In the spectra of the dibromocyclohexyltin derivatives, IV and X, recorded on solutions in DMSO, the $\alpha$-proton shift had changed further to higher $\delta$-values, *viz.* 9.30–9.53 and 9.12–9.33 ppm. An increased electron deficiency of pyridine will thus apparently cause the signal to appear at an even lower field strength. The protons in the 1-, 2- and 3-positions of the cyclohexane ring resonate in the region 5.50–6.70 ppm, but it cannot be said with certainty whether they occupy axial or equatorial positions. The width of the signals, however, indicates that the transition from the axial to the equatorial conformation of these protons is a slow process at 40°.

The two monoadducts, III and VI, of diphenyltin dibromide with 3-bromocyclohexene and allyl bromide, containing 3 and 2 molecules of pyridine, respectively, give two multiplets at 9.10–9.40 and 8.63–8.90 ppm. The proton ratios of the two compounds are 1 : 2 and 1 : 1. These regions of the α-protons can probably be assigned to pyridinium rings linked to the cyclohexyl and propyl groups, and to pyridine coordinated with the central tin atom.

## DISCUSSION

Two mechanisms for the addition of triphenyltin bromide to the double bond in olefine derivatives are conceivable. One possibility is that a π-complex is formed in the transition state between the triphenyltin group and the olefine (eqn. (1)). The



$$Ph_3SnBr + R{-}CH{=}CH_2 \rightarrow \underset{\overset{\downarrow}{SnPh_3}}{R{-}CH{=}CH_2} + Br^- \rightarrow R{-}\overset{\overset{Br}{|}}{CH}{-}\underset{\underset{SnPh_3}{|}}{CH_2} \text{ or } R{-}\overset{\overset{SnPh_3}{|}}{CH}{-}\underset{\underset{Br}{|}}{CH_2} \quad (1)$$

complex is then attacked in the *trans*-position by the bromide ion. This mechanism is analogous to that suggested for bromine addition to olefines. However, this reaction path is not very probable for two reasons. The formation of a π-complex should be sterically hindered by pyridine, the coordination compound of which with triphenyltin bromide is rather stable both in ethanol and pyridine. Steric hindrance would also be important if the tin–bromine bond should be completely dissociated in hot ethanol or pyridine, when the triphenyltin ion, suffering from electron deficiency, would very probably attract one or two pyridine molecules. Moreover, allyl bromide and allyl alcohol should have a similar ability to add triphenyltin bromide in ethanol at 80°, but this has been shown not to be so.

From the different reactivity of the allyl derivatives, and also of cyclohexene and 3-bromocyclohexene, towards the phenyltin bromides, it may therefore be postulated that the allyl cation is a reactive intermediate in these additions. In the absence of any kinetic measurements, no details of the reaction mechanism can be given.

When diphenyltin dibromide reacts with allyl bromide in the molar ratio 1 : 1, the addition product, VI, (Fig. 1) probably combines with pyridine in two ways, as indicated by the IR- and PMR-spectra. One molecule is coordinated to the tin atom because this still has some electron-attracting power, depending on the partial ionic character of the tin–bromine bond and also to some extent on the inductive effect of the bromine substituents of the propyl group. The second pyridine molecule is attached to the 2- or 3-position of the propyl chain, or alternatively, to the 1- or 3-position of the isopropyl group. On the addition of this intermediate to another molecule of allyl bromide, no coordinated pyridine is found in the product, VII. Organotin compounds with four tin–carbon bonds have no longer any ability to form coordination complexes[4]. Conversely, bis(dibromopropyl)diphenyltin contains only one pyridine/propyl group. As the absorption band at 1450 cm$^{-1}$, assigned to the Sn–N stretching vibration, was not observed in its infrared spectrum, this compound must be a pyridinium salt in the usual sense. It can be concluded that the inductive effect of the bromine substituents alone in the propyl groups is insufficient to promote coordination. In the monoadduct of diphenyltin dibromide and 3-bromocyclohexene, III, the PMR spectrum indicates that the ratio of the coordinated to carbon-bound pyridine is 2 : 1. Apparently, the bivalent forces of the tin atom are stronger in compound III than in the propyltin derivative, VI.

The bulkiness of the triphenyltin group and the bromine atoms in dibromocyclohexyltriphenyltin, X, should certainly force them to be bound mainly in equatorial positions. A substituent in the axial position, with the exception of hydrogen, and possibly fluorine, interferes sterically with the axial hydrogens in 3-positions. Thus tert-butylcyclohexane and its derivatives, in which the butyl group strongly prefers the equatorial position, are "conformationally pure" for this reason[12] and X almost certainly has the same property. The infrared spectra of the cyclohexane derivatives, III, IV and X, however, indicate that they also should contain axial C–Br bonds.

## EXPERIMENTAL

Carbon and hydrogen analyses were carried out at the microanalytical laboratory of the "Max-Planck-Institut für Kohlenforschung" Mülheim/Ruhr in Germany. The bromine analyses were made electrometrically with silver nitrate. 0.2–0.3 g of the sample was hydrolysed with 5 ml of 1 *M* sodium hydroxide on a boiling water bath for 30 min. The solution was diluted to 150 ml and 4–5 ml of acetic acid and 2 ml of a 2 % starch solution were added. The solution was titrated with 0.1 *N* silver nitrate, using a silver electrode and a saturated calomel electrode as reference. The reference solution of saturated potassium chloride was connected with the test solution by a salt bridge of ammonium nitrate in agar.

Diphenyltin dibromide, I, and triphenyltin bromide, II, were prepared by disproportionation between tetraphenyltin and tin tetrabromide by the methods of Van der Kerk and Luijten[13] (I) and Pedley and Skinner[14] (II). 3-bromocyclohexene was synthesized by bromination of cyclohexene with *N*-bromosuccinimide in carbon tetrachloride[15] and distilled at 57°/8 mm. Allyl bromide was used without further purification. Pyridine was dried over potassium hydroxide pellets and distilled. Ethanol contained less than 1 % water and was not further purified.

Compounds III–VIII (Fig. 1), dibromopropyltriphenyltin monopyridinate, IX, and dibromocyclohexyltriphenyltin monopyridinate, X, have been prepared either in pyridine (method A) or in ethanol (method B) as solvent. For simplicity, only one example of each method is described in detail below. The method used, yields, and analytical data of every compound are given in Table 2.

### *Method A*

Diphenyltin dibromide (2.0 g, 4.6 mmoles) was suspended in 7 g of pyridine. 3-bromocyclohexene (0.75 g, 4.6 mmoles) in 3 g of pyridine was added and the mixture warmed for 90 min at 50° on a water-bath. The mixture was cooled, 25 ml of ether were

TABLE 2

PREPARATION OF DIBROMOPROPYL- AND DIBROMOCYCLOHEXYL-TIN COMPOUNDS
ALL MELTING POINTS ARE UNCORRECTED

| *Compound* | *Method* | *Reactants (g)* | *Solvent (ml)* | *Yield (%)* | *M.p. (°C)* | *Calcd.* | | | *Found* | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | *C* | *H* | *Br* | *C* | *H* | *Br* |
| III | A | 2.0 I<br>0.75 bch[a] | 10 | 57.3 | 128–131 | 47.69 | 4.12 | 28.85 | 45.77 | 4.28 | 28.62 |
| IV | A | 2.0 I<br>1.5 bch | 10 | 75.8 | 166<br>(dec.) | 44.72 | 4.20 | 35.01 | 45.15 | 4.13 | 35.29<br>34.74 |
| V | B | 2.0 I<br>0.56 ab[b]<br>0.37 Py[c] | 20 | 44.5 | 152–159<br>(dec.) | — | — | 37.89 | — | — | 36.84 |
| VI | A | 2.0 I<br>0.56 ab | 10 | 42.6 | 128–131 | 42.18 | 3.54 | 33.68 | 42.52 | 3.75 | 33.16 |
| VIIa | A | 2.0 I<br>1.12 ab | 10 | 59.7 | 170–171 | 40.37 | 3.63 | 38.38 | 41.67 | 3.80 | 38.00 |
| VIIb | B | 2.0 I<br>1.12 ab<br>0.73 Py | 25 | 67.5 | 166–167[d] | — | — | — | — | — | — |
| VIIIa | A | 0.63 VI<br>0.16 bch | 3 | 78.1 | 147–149<br>(dec.) | 42.65 | 3.93 | 36.63 | 43.56 | 4.03 | 35.95 |
| VIIIb | A | 1.0 III<br>0.18 ab | 3 | 76.4 | 146–149[e]<br>(dec.) | — | — | — | — | — | — |
| VIIIc | A | 0.5 V<br>0.13 bch | 2.5 | 35.8 | 144–148[e]<br>(dec.) | — | — | — | — | — | — |
| IXa | A | 2.0 II<br>0.56 ab | 10 | 56.1 | 129–130 | 49.57 | 4.00 | 25.37 | 49.49 | 4.27 | 25.27 |
| IXb | B | 2.0 II<br>0.56 ab<br>0.37 Py | 8 | 47.8 | 129–130[f] | — | — | — | — | — | |
| X | A | 2.0 II<br>0.75 bch | 5 | 57.7 | 131–132<br>(dec.) | 51.98 | 4.36 | 23.85 | 52.51 | 4.49 | 23.41 |

[a] bch = 3-bromocyclohexene; [b] ab = allyl bromide; [c] Py = pyridine; [d] no depression with VIIa; [e] no depression with VIIIa; [f] no depression with IXa.

added, and the flask kept at −20° overnight. The crude product was filtered, washed with 25 ml of ether and recrystallized twice from 20 and 15 ml of ethanol; yield 2.2 g (57.3%) of salmon-red crystals of dibromocyclohexyldiphenyltin bromide tripyridinate, III, m.p. 128–131°. (Found: C, 45.77; H, 4.28; Br, 28.62. $C_{33}H_{34}Br_3N_3Sn$ calcd.: C, 47.69; H, 4.12; Br, 28.85%.)

### *Method B*

Diphenyltin dibromide (2.0 g) was suspended in 15 ml of ethanol and allyl bromide (0.56 g, 4.6 mmoles) and pyridine (0.37 g, 4.6 mmoles) in 5 ml of ethanol were added. On boiling, a colourless solution was obtained; it was boiled under reflux for 3 h. The solution was cooled slowly and kept overnight at −20°. A snow-white substance was filtered and recrystallized from 12 ml of ethanol. Yield, 1.3 g (44.5%) of dibromopropyldiphenyltin bromide monopyridinate, V, which melted to a viscous oil at 152–159°. (Found: Br, 36.84. $C_{20}H_{20}Br_3NSn$ calcd.: Br, 37.89%.)

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## SUMMARY

A number of dibromo-substituted propyl- and cyclohexyl-tin derivatives have been prepared by the addition of diphenyltin dibromide and triphenyltin bromide to allyl bromide and 3-bromocyclohexene in the presence of pyridine. The infrared and proton magnetic resonance spectra of some of the new compounds have been recorded and interpreted.

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