

*Preparation of n-decyltriphenylsilane.* A solution of 0.086 mole of *n*-decyllithium in 100 ml of diethyl ether was added to 25.35 g (0.086 mole) of chlorotriphenylsilane dissolved in 100 ml of ether at ice-bath temperature. After seven h of stirring at room temperature, Color Test I<sup>6</sup> was negative. After hydrolysis with water the reaction mixture was worked up in the usual manner. A portion of the product was lost mechanically during work-up. However, 8.72 g (25.3%) of crude *n*-decyltriphenylsilane, m.p. 64–69°, was obtained. One recrystallization from ethanol afforded pure product, m.p. 68–69.5° (mixed m.p.).

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- 1 H. GILMAN AND R. D. GORSICH, *J. Org. Chem.*, 22 (1957) 687.
- 2 H. GILMAN AND S. GRAY, *J. Org. Chem.*, 23 (1958) 1476.
- 3 H. GILMAN AND W. J. TREPKA, *J. Org. Chem.*, 27 (1962) 1418.
- 4 H. GILMAN AND B. J. GAJ, *J. Org. Chem.*, 22 (1957) 447.
- 5 H. GILMAN AND H. A. MCNINCH, *J. Org. Chem.*, 27 (1962) 1889.
- 6 H. GILMAN AND F. SCHULZE, *J. Am. Chem. Soc.*, 47 (1925) 2002.
- 7 R. L. BURWELL, JR., *Chem. Rev.*, 54 (1954) 615.
- 8 H. GILMAN AND A. H. HAUBEIN, *J. Am. Chem. Soc.*, 66 (1944) 1515.
- 9 H. GILMAN AND F. K. CARTLEDGE, *J. Organometal. Chem.*, 5 (1966) 48.
- 10 H. GILMAN, J. A. BEEL, C. G. BRANNEN, M. H. BULLOCK, G. E. DUNN AND L. S. MILLER, *J. Am. Chem. Soc.*, 71 (1949) 1499.

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### Isolation of a compound containing the trimethyltin cation dihydrate

In 1960 Okawara *et al.*<sup>1</sup> suggested, on the basis of the infrared spectra of trimethyltin fluoride and carboxylates, the existence of the planar trimethyltin cation,  $(\text{CH}_3)_3\text{Sn}^+$ , in the solid state. Although their interpretation was soon questioned by Beattie<sup>2</sup>, the interest of many organotin chemists has been, since then, focused on the unique structural properties of organotin derivatives. Several following investigations<sup>3–5</sup> have shown the polymeric structure for these compounds by the bridging of the anionic group, F or OOCR, to the trialkyltin group, resulting a pentacoordinated structure for the tin atom. Among similar investigations<sup>6–15</sup>, Clark<sup>15</sup> showed that trimethyltin perchlorate and nitrate form diammonia adducts, which on the basis of their infrared spectra were indicated to be ionic compounds formulated as  $[(\text{CH}_3)_3\text{Sn}(\text{NH}_3)_2]^+\text{X}^-$ , the tin atom still having a trigonal bipyramidal configuration.

In this present investigation we have isolated for the first time a compound containing the dihydrated trimethyltin cation. This novel compound was obtained by reaction of trimethyltin chloride and sodiumtetraphenylborate in water. The infrared spectrum of  $[(\text{CH}_3)_3\text{Sn}(\text{OH}_2)_2]^+\text{[B}(\text{C}_6\text{H}_5)_4]^-$  shows only one band in the 500–550  $\text{cm}^{-1}$  region, indicating a planar  $(\text{CH}_3)_3\text{Sn}$  arrangement<sup>1</sup>. The rest of the spectrum is

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almost consistent with that of sodium tetraphenylborate, except for the bands at  $3450\text{ cm}^{-1}$  (OH str.),  $1592\text{ cm}^{-1}$  ( $\text{H}_2\text{O}$  def.),  $791\text{ cm}^{-1}$  ( $\text{CH}_3$  rock.) and  $735\text{ cm}^{-1}$ . The origin of the last band is unknown, but we tentatively make the assignment as  $\text{H}_2\text{O}$  rocking vibration<sup>16</sup>.

### Experimental

To a hot solution of sodium tetraphenylborate (3 g) in water (40 ml) was added trimethyltin chloride (1.7 g) solution in water (40 ml) with gentle stirring. The white precipitate which resulted was filtered on glass filter, washed repeatedly with water and dried over calcium chloride under vacuum. Yield, 3.7 g; m.p.,  $80\text{--}84^\circ$ . (Found: C, 62.24; H, 6.25.  $\text{C}_{27}\text{H}_{33}\text{BO}_2\text{Sn}$  calcd.: C, 62.48; H, 6.41 %.) The compound is soluble in acetone, ethanol and diethyl ether, but insoluble in benzene, cyclohexane and chloroform. The compound decomposes slowly in air, but is rather stable in water or under vacuum.

The infrared spectra were measured in nujol mull using Hitachi EPI-2G Grating Spectrometer. The relevant infrared frequencies are shown in Table I.

TABLE I

INFRARED VIBRATIONAL FREQUENCIES<sup>a</sup> OF TRIMETHYLTIN TETRAPHENYLBORATE DIHYDRATE,  $[(\text{CH}_3)_3\text{Sn}(\text{OH})_2]_2^+ [\text{B}(\text{C}_6\text{H}_5)_4]_2^-$

Frequencies ( $\text{cm}^{-1}$ )	Assignment
3450 (s)	OH str.
1592 (m)	$\text{H}_2\text{O}$ def. <sup>b</sup>
791 (m)	$\text{CH}_3$ rock.
735 (s)	$\text{H}_2\text{O}$ rock.
562 (s)	$\text{SnC}_3$ asym. str.

<sup>a</sup> Those frequencies due to tetraphenylborate anion were excluded by comparison with the spectrum of sodium tetraphenylborate. <sup>b</sup> Overlapping with the vibration due to the phenyl group.

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- 1 R. OKAWARA AND E. G. ROCHOW, *J. Am. Chem. Soc.*, **82** (1960) 3285.
- 2 I. R. BEATTIE AND T. GILSON, *J. Chem. Soc.*, (1961) 2585.
- 3 M. J. JANSSEN, J. G. A. LUIJTEN AND G. J. M. VAN DER KERK, *Rec. Trav. Chim.*, **82** (1963) 90.
- 4 R. OKAWARA AND M. OHARA, *J. Organometal. Chem.*, **1** (1964) 360.
- 5 H. C. CLARK, R. J. O'BRIEN AND J. TROTTER, *J. Chem. Soc.*, (1964) 2332.
- 6 H. KRIEGSMANN, H. HOFFMANN AND S. PISCHTSCHAN, *Z. Anorg. Allgem. Chem.*, **315** (1962) 283.
- 7 R. OKAWARA AND K. YASUDA, *J. Organometal. Chem.*, **1** (1964) 356.
- 8 N. KASAI, K. YASUDA AND R. OKAWARA, *J. Organometal. Chem.*, **3** (1965) 76.
- 9 R. OKAWARA, B. J. HATHAWAY AND D. E. WEBSTER, *Proc. Chem. Soc.*, (1963) 13.
- 10 H. C. CLARK AND R. J. O'BRIEN, *Inorg. Chem.*, **2** (1963) 740.
- 11 K. YASUDA AND R. OKAWARA, *J. Organometal. Chem.*, **3** (1965) 76.
- 12 J. G. A. LUIJTEN, M. J. JANSSEN AND G. J. M. VAN DER KERK, *Rec. Trav. Chim.*, **81** (1962) 202.
- 13 B. J. HATHAWAY AND D. E. WEBSTER, *Proc. Chem. Soc.*, (1963) 14.
- 14 I. R. BEATTIE AND G. P. MCQUILLAN, *J. Chem. Soc.*, (1963) 1519.
- 15 H. C. CLARK AND R. J. O'BRIEN, *Inorg. Chem.*, **2** (1963) 1020.
- 16 K. NAKAMOTO, *Infrared Spectra of Inorganic and Coordination Compounds*, Wiley, New York, London, 1963, sect. III-3.

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