From the residue of the first distillation was obtained a solid which was recrystallized from dry petroleum ether (b.p. 60–70°) to give 2.4 g (3.1 %) of triphenyltin chloride, m.p. 101–103° (mixed m.p. 103–105°).

Preparation of methyldiphenyltinlithium. To 7.9 g (0.024 mole) of methyldiphenyltin chloride and 0.52 g (0.075 g-atom) of finely cut lithium wire was added a small amount of tetrahydrofuran. Stirring was commenced, and an immediate exothermic reaction took place. Tetrahydrofuran (50 ml) was added, and the mixture rapidly became green-brown in color. After stirring for 3 h, titration showed a 93.7 % yield of the tinlithium reagent.

Acknowledgement

This research was supported in part by the United States Air Force under Contract No. AF 33(616)-6463 administered by Materials Laboratory, Wright Air Development Division, Dayton, Ohio.

Chemistry Department, Iowa State University,
Ames, Iowa (U.S.A.)

HENRY GILMAN
FRANK K. CARTLEDGE
SEE-YUEN SIM

1 D. WITTENBERG AND H. GILMAN, *J. Am. Chem. Soc.*, 80 (1958) 2677.
2 H. GILMAN, R. A. KLEIN AND H. J. S. WINKLER, *J. Org. Chem.*, 26 (1961) 2474.
3 H. GILMAN, F. K. CARTLEDGE AND S.-Y. SIM, *J. Organometal. Chem.*, 1 (1963) 8.
4 H. GILMAN AND F. K. CARTLEDGE, *J. Organometal. Chem.*, in press.
5 H. GILMAN AND F. K. CARTLEDGE, *J. Organometal. Chem.*, 3 (1965) 255.
6 H. GILMAN AND C. W. GEROW, *J. Am. Chem. Soc.*, 77 (1955) 5740; M. V. GEORGE, P. B. TALUKDAR, C. W. GEROW AND H. GILMAN, *J. Am. Chem. Soc.*, 82 (1960) 4562.
7 H. GILMAN, O. L. MARRS AND S.-Y. SIM, *J. Org. Chem.*, 27 (1962) 4232.
8 C. TAMBORSKI, F. E. FORD AND E. J. SOLOSKI, *J. Org. Chem.*, 28 (1963) 237.

Received May 7th, 1965

J. Organometal. Chem., 4 (1965) 332–334

An electrolytic preparation of methylaluminium diiodide

Methyl aluminium diiodide can be prepared by the reaction

$$3CH_3I + 2Al \longrightarrow (CH_3)_2AlI + CH_3AlI_2$$

but attempts to isolate the diiodide have proved very difficult; fractional distillation under reduced pressure have resulted in complete disproportionation of the diiodide[1].

$$2CH_3AlI_2 \longrightarrow (CH_3)_2AlI + AlI_3$$

The diiodide has been prepared by the following method[1]. The monoiodide was melted with aluminium triiodide at 170°.

$$(CH_3)_2AlI + AlI_3 \longrightarrow 2CH_3AlI_2$$

This was followed by fractional distillation under reduced pressure to remove the diiodide from the reaction mixture (considerable disproportionation occurred at this stage). The pure compound was obtained by repeated recrystallization from *n*-pentane.

J. Organometal. Chem., 4 (1965) 334–335

We wish to report a very simple preparation of the diiodide by electrolysis of a solution of aluminium triiodide in iodomethane using aluminium electrodes.

The solution of the triiodide was prepared *in situ* by warming a solution of iodine in iodomethane with aluminium wire. The resulting solution was electrolysed, using concentric coils of aluminium wire as the electrodes (applied potential: 12 volts). The current slowly rose until the diiodide began to crystallize from solution. The crystals which were obtained as colourless hexagonal plates formed on both electrodes, m.p. 67–68° (lit.[1] 68–71°). (Found: Al, 9.17; I, 84.71; CH_3, 5.22. CH_3AlI_2 calcd.: Al, 9.12; I, 85.80; CH_3, 5.07 %.) The aluminium and iodide were determined by conventional gravimetric methods and the methyl group was determined by reaction with water to produce a measured volume of methane

$$CH_3AlI_2 + 3H_2O \longrightarrow Al(OH)_3 + 2HI + CH_4$$

The filtered electrolyte reacted violently with water to yield methane together with a small amount of hydrogen (0.7 %)*.

The formation of hydrogen would seem to indicate the presence of monovalent aluminium which has been postulated as an intermediate in aqueous electrolysis using aluminium electrodes[2].

It is suggested that monovalent aluminium may be formed initially at the anode [while Al(III) discharges as metal on the cathode], but that this unstable species rapidly reacts with iodomethane to form the diiodide:

$$AlI + CH_3I \longrightarrow CH_3AlI_2$$

Acknowledgement

We wish to thank Northampton College of Advanced Technology, London for a maintenance grant to E.K.

The Chemistry Department,
Northampton College of Advanced Technology,
London (Great Britain)

J. R. CHADWICK
E. KINSELLA

1 A. VON GROSSE AND J. M. MAVITY, *J. Org. Chem.*, 5 (1940) 106.
2 A. W. DAVIDSON AND F. JIRIK, *J. Am. Chem. Soc.*, 72 (1950) 1700.

Received March 30th, 1965

---

* Identified by mass-spectrometric analysis performed by Messrs. G. GOUGH and B. H. STRINGER of Distillers Company Ltd. (Epsom).

Zero-valent nickel complexes of bis(phosphino)-o-carboranes

The recent synthesis[1] of *o*-carborane [1,2-dicarbaclovododecaborane(12)][2] provides a new basic molecular framework which, when appropriately substituted at the two carbon atoms, can act as a bi-functional ligand toward metals. Unsub-