indicate that ligands such as PdAdH normally exhibit *Dq* values between 1200 and 1400 cm.$^{-1}$ when in combination with divalent metal ions.[18] In the cobalt(II) case, the intervention of a strong non-cubic component may render invalid the calculation of the crystal field stabilization energy in terms of an octahedral model for the spin-paired (doublet) state.

(18) M. A. Robinson and D. H. Busch, unpublished results.

McPHERSON CHEMICAL LABORATORY R. CARL STOUFER
THE OHIO STATE UNIVERSITY DARYLE H. BUSCH
COLUMBUS 10, OHIO WAYNE B. HADLEY

RECEIVED MAY 29, 1961

A NEW ORGANOTIN-MAGNESIUM COMPOUND

Sir:

Organomagnesium compounds of group IV elements ($R_3M^{IV}MgX$) have thus far been reported for the element carbon. Gilman[1] and Seyferth[2] have reported negative results in attempts to synthesize an organomagnesium derivative of germanium. Silyl Grignard reagents, with silicon directly bonded to magnesium, have been postulated by other investigators[3]; however, no such reagents have been isolated.

As part of a program on the preparation of organometallics of group III, IV and V elements, we wish to report the preparation and some reactions of a novel organotin-magnesium compound. From its synthesis and reaction products the structure of this compound appears to be $[(C_6H_5)_3Sn]_2Mg$. It can be prepared at room temperature in tetrahydrofuran (THF) solvent by the reaction of triphenyltin chloride with excess magnesium turnings.

$$2(C_6H_5)_3SnCl + 2Mg \xrightarrow[C_2H_5Br]{THF} [(C_6H_5)_3Sn]_2Mg + MgCl_2 \quad (1)$$

The reaction was initiated by ethyl bromide. Without an initiator, no reaction was observed. No reaction took place when diethyl ether was substituted for tetrahydrofuran as solvent.

It appears that a two step process takes place. After the reaction has started, a white precipitate[4] is formed. Color Test I[5] is negative at this stage. With further stirring at room temperature, the white precipitate disappears and the reaction mixture turns grayish-green in color, and now gives an intense positive Color Test I.

$$2(C_6H_5)_3SnCl + Mg \xrightarrow[C_2H_5Br]{THF} (C_6H_5)_3SnSn(C_6H_5)_3 + MgCl_2 \quad (2)$$

$$(C_6H_5)_3SnSn(C_6H_5)_3 + Mg \xrightarrow[C_2H_5Br]{THF} [(C_6H_5)_3Sn]_2Mg \quad (3)$$

If the mixture was hydrolyzed shortly after the white precipitate is formed but prior to a positive Color Test I, the products identified were $(C_6H_5)_3SnSn(C_6H_5)_3$ and unreacted $(C_6H_5)_3SnCl$. To further substantiate the mode by which this organotin-magnesium compound is formed, hexaphenylditin was allowed to react with excess magnesium in tetrahydrofuran using ethyl bromide as the initiator. The white appearance[4] of the reaction mixture soon disappears with the formation of a grayish-green color, and a positive Color Test I. Hydrolysis of this solution with ammonium chloride solution yields triphenyltin hydride in 82% yield.

$$[(C_6H_5)_3Sn]_2Mg + 2H_2O \longrightarrow 2(C_6H_5)_3SnH + Mg(OH)_2 \quad (4)$$

Carbonation of this organometallic yielded hexaphenylditin as the product. A similar reaction takes place when triphenyltinlithium[6] is treated with carbon dioxide to yield only hexaphenylditin in 80–90% yield.

All the preparations of $[(C_6H_5)_3Sn]_2Mg$ described thus far have been carried out at room temperature. If, however, during the preparation the reaction is refluxed, the organotin-magnesium compound decomposes and hydrolysis yields no triphenyltin hydride.

Other reactions of this organotin-magnesium compound as well as the generality of this preparative method for other elements are currently under investigation.

DIRECTORATE OF MATERIALS & PROCESSES
POLYMER BRANCH CHRIST TAMBORSKI
NOMETALLIC MATERIALS LABORATORY
WRIGHT-PATTERSON AFB, OHIO EDWARD J. SOLOSKI[7]

RECEIVED JULY 28, 1961

---

(1) H. Gilman and C. W. Gerow, *J. Am. Chem. Soc.*, **78**, 5435 (1956).

(2) D. Seyferth, *ibid.*, **79**, 2738 (1957). Private communication to D. Seyferth from A. L. Allred.

(3) C. Eaborn, *J. Chem. Soc.*, 2755 (1955); T. G. Selin and R. West, *Tetrahedron*, **5**, 97 (1959); M. V. George, D. J. Peterson and H. Gilman, *J. Am. Chem. Soc.*, **82**, 403 (1960); W. Steudel and H. Gilman, *ibid.*, **82**, 6129 (1960); H. Gilman and A. G. Brook, *ibid.*, **76**, 278 (1954).

(4) The white appearance of the reaction mixture is due to the incomplete solubility of hexaphenylditin in the quantity of tetrahydrofuran solvent used.

(5) H. Gilman and F. Schulze, *ibid.*, **45**, 2002 (1925).

(6) C. Tamborski and F. E. Ford, unpublished studies; see also D. Blake, G. E. Coates and J. M. Tate, *J. Chem. Soc.*, 618 (1961); these investigators treated triphenyltinsodium with carbon dioxide and obtained an 80% yield of hexaphenylditin.

(7) University of Dayton, Research Institute, Dayton, Ohio.