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HEXAMETHYLDILEAD

III *. REACTION WITH METAL SALTS AND ORGANOMERCURIALS

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Summary

Hexamethyldilead reacts with various metal salts very rapidly in comparison with the rate of mixing of the reactants so that the product composition depends upon the order of addition and the competition between rapid reactions rather than any fundamental differences in mechanism.

Introduction

The most extensive set of reactions of a hexaalkyldilead so far reported are the following [1]:

$$(C_2H_5)_6Pb_2 + HgCl_2 \rightarrow 2\ (C_2H_5)_3PbCl + Hg \quad (1)$$

$$(C_2H_5)_6Pb_2 + 2HgCl_2 \rightarrow 2\ (C_2H_5)_3PbCl + Hg_2Cl_2 \quad (2)$$

$$(C_2H_5)_6Pb_2 + 2AgNO_3 \rightarrow 2\ (C_2H_5)_3PbCl + 2\ Ag \quad (3)$$

$$2\ (C_2H_5)_6Pb_2 + 2FeCl_3 \rightarrow 3\ (C_2H_5)_4Pb + PbCl_2 + 2\ FeCl_2 \quad (4)$$

$$2\ (C_2H_5)_6Pb_2 + Hg_2Cl_2 \rightarrow 3\ (C_2H_5)_4Pb + PbCl_2 + 2Hg \quad (5)$$

$$2\ (C_2H_5)_6Pb_2 + CuCl_2 \rightarrow 3\ (C_2H_5)_4Pb + PbCl_2 + Cu \quad (6)$$

$$2\ (C_2H_5)_6Pb_2 + 2Cu_2Cl_2 \rightarrow 3\ (C_2H_5)_4Pb + PbCl_2 + 2Cu \quad (7)$$

$$6\ (C_2H_5)_6Pb_2 + 3AuCl_3 \rightarrow 9\ (C_2H_5)_4Pb + 3PbCl_2 + Au \quad (8)$$

$$2\ (C_2H_5)_6Pb_2 + 4CuCl_2 \rightarrow (C_2H_5)_4Pb \text{ and } (C_2H_5)_3PbCl$$
$$PbCl_2 \text{ and } Cu_2Cl_2 \text{ and ``gas''} \quad (9)$$

These reactions are the stoichiometric results of the addition of liquid hexa-

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* For part II see ref. 7.

ethyldilead to methanol solutions of the various reagents. There appear to be two types of behaviour; Type A, where the product is $(C_2H_5)_3PbX$ [reactions 1—3] and Type B, where $(C_2H_5)_4Pb$ and $PbCl_2$ are formed [reactions 4—8]. Reaction 9 has some of the characteristics of both types but is in addition the only one in which gaseous products, presumably ethane and ethene and perhaps butane, are formed and ethyl groups are lost from lead. In each case the metal salt has suffered reduction.

It has been suggested [1] that there is a facile equilibrium:

$$(C_2H_5)_6Pb_2 \rightleftharpoons (C_2H_5)_4Pb + (C_2H_5)_2Pb$$

and that metal ions with unfilled electron shells react with diethyllead, whereas those with filled shells cleave hexaethyldilead. The above equilibrium has been shown to play no part in the acetolysis of hexaphenyldilead [2] and a report that it might be important was shown to be erroneous [3]. This mode of decomposition does seem to be responsible for the thermal decomposition of hexamethyldilead [4,5] but at a much slower rate than is appropriate to the reactions under consideration, and with no indication of reversibility.

The reactions of hexaalkyldileads with alkylmercuric salts have not previously been described, although decomposition of hexaethyldilead is reported to be catalysed by ethyl- and propyl-mercuric salts [6].

The investigations described below were undertaken to clarify the mechanisms of these reactions, their relationship to the reaction with trimethyllead chloride described in Part II [7], and their comparison with some similar reactions of hexamethylditin [8].

Experimental

Materials

Samples of pure hexamethyldilead were obtained as described in Part I [5]; tetramethyllead and trimethyllead chloride as in Part II [7]. The various metal salts were A.R. grade and employed without further purification. Methanol was AJAX UNIVAR "dried for non-aqueous titration". Methylmercuric chloride, purified by three sublimations, and trideutero-methylmercuric chloride were obtained as previously described [8].

Product examination

All the reactions of hexamethyldilead when conducted at concentrations suitable for PMR study had visible ceased within ca. 5 sec of initial mixing.

One reactant solution was rapidly added via a syringe to the rapidly stirred solution of the other reactant in a 5 ml sample bottle. A precipitate-free sample of the solution was then examined in the PMR spectrometer (JEOL PS-100 or MH-100) within 5 min of mixing. Reactions subsequent to the main reaction, see below, were then observed. (The results reported in [1] are for analysis after 5 h reaction). Precipitates were examined separately as indicated below.

Kinetic studies

(a) Tetramethyllead and methylmercuric chloride. Following the procedure

previously described [8] the reaction:

$$(CH_3)_4Pb + CH_3HgCl \xrightleftharpoons[2k_{-a}]{4k_a} (CH_3)_3PbCl + (CH_3)_2Hg \qquad (A)$$

was studied for methanol solutions at 36°C by PMR spectroscopy (Varian A-60). The second order rate constant, $4k_a$, was found to be $2.8(\pm 0.1) \times 10^{-2}\ M^{-2}\ s^{-1}$. (The reverse reaction and the equilibrium were undetectable so that $2k_{-a} \ll 1.5 \times 10^{-3}\ M^{-1}\ s^{-1}$).

(b) Trimethyllead chloride and mercuric chloride. The PMR spectral procedure was again employed to study:

$$(CH_3)_3PbCl + HgCl_2 \xrightarrow{3k_b} (CH_3)_2PbCl_2 + CH_3HgCl \qquad (B)$$

and we obtained $3k_b = 5.1(\pm 0.2) \times 10^{-2}\ M^{-1}\ s^{-1}$ (methanol, 36°C). The reverse reaction was not detectable.

(c) Tetramethyllead and mercuric chloride. This reaction occurs much too rapidly to be studied at the concentrations required for the PMR method. Several attempts were made to measure the rate constant by the method that Abraham [9,10] applied to the study of tetraalkyltins, but these were unsuccessful. Apparently, quenching the reaction with excess iodide was not complete, or perhaps other complexations interfered with the UV absorbances of the quenched samples.

Competition between initial concentrations of 0.181 M tetramethyltin and 0.084 M tetramethyllead for 0.062 M mercuric chloride gave only a trace of trimethyltin chloride. Comparison of trimethyllead chloride formed and tetramethyllead remaining showed 69% reaction so that a maximum of 7% of the tetramethyllead had been consumed. A minimum rate constant for tetramethyllead is thus ln 0.31/ln 0.93 = 16 times that of tetramethyltin which is $2.0 \times 10^{-1}\ M^{-1}\ s^{-1}$ at 30°C [8]. A lower limit is thus 30 $M^{-1}\ s^{-1}$ for $4k_c$. (A similar value is indicated from a competition between tetramethyllead and hexamethylditin, however the rate constant for the latter is not precisely known [8].)

The rate of reaction of tetramethyltin with mercuric chloride is ca. 10^4 greater than its rate of reaction with methylmercuric chloride [8]. A comparison of $4k_c$ with $4k_a = 3 \times 10^{-2}\ M^{-1}\ s^{-1}$, above, gives a rate ratio of $>10^3$ which appears to be quite reasonable.

(d) Hexamethyldilead and methylmercuric chloride. Kinetic studies were carried out following the UV absorbance of the substrate with methylmercuric chloride in excess to ensure first order kinetics. (This is the procedure described in Part II [7], and is necessitated by the absence of precise initial concentration information for the substrate.) In all experiments there was an initial fast reaction consistent with the presence of a fraction of a percent of mercuric chloride in the methylmercuric chloride. This has a disproportionate effect due to the large relative concentration of the reagent. The anomalous period of the reaction varied with the sample of the reagent. As indicated for a typical example, Fig. 1, the subsequent reaction shows good first order behaviour. The first order rate constants vary with methylmercuric chloride concentration in a manner suggesting that a small fraction of the reagent dissociates to form a more reactive species, solvated CH_3Hg^+, cf. [7,11]. The second order rate constant for the undissociated salt is 0.3 $M^{-1}\ s^{-1}$ (22°C).

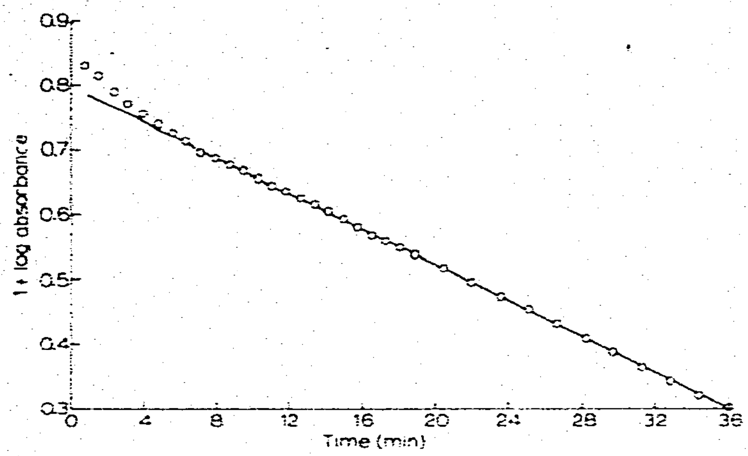

Fig. 1. First order plot for $(CH_3)_6Pb_2$ + CH_3HgCl.

Results

In the previous studies [1] liquid hexaethyldilead was added to a methanolic solution of the reagent. Both this and the reverse order of addition of solutions of the reactants were studied.

Addition of hexamethyldilead to mercuric chloride

(i) Mercurous chloride (indentified by the formation of mercury on treatment with ammonia) was immediately precipitated on addition of 2.5×10^{-5} mol of $(CH_3)_6Pb_2$ (in 0.25 ml methanol) to 6.5×10^{-5} mol of $HgCl_2$ (in 0.7 ml methanol). The solution contains trimethyllead chloride, dimethyllead dichloride and methylmercuric chloride. Reaction B was in progress and continued for some 30 min. Extrapolation indicates that the main reaction is predominantly:

$$(CH_3)_6Pb_2 + 2HgCl_2 \rightarrow 2(CH_3)_3PbCl + Hg_2Cl_2$$

although there could be some tetramethyllead formed and rapidly consumed by the excess mercuric chloride through reaction C.

(ii) On addition of 4.8×10^{-5} mol of the substrate (in 0.46 ml methanol) to 4.4×10^{-5} mol of $HgCl_2$ (in 0.48 ml methanol) both mercury and lead chloride (soluble in hot water, black precipitate of sulphide) are immediately produced. The solution after 5 min contains tetramethyllead (0.029 *M*), trimethyllead chloride (0.053 *M*), methylmercuric chloride (0.015 *M*), and dimethylmercury (0.014 *M*). Reaction A was observed to be in progress and continued until the methylmercuric chloride was consumed.

Addition of mercuric chloride to hexamethyldilead

(iii) A mixture of mercury and lead chloride was precipitated immediately on addition of 2.2×10^{-5} mol of $HgCl_2$ (in 0.27 ml methanol) to 4.5×10^{-5} mol of the substrate (in 0.53 ml methanol). The only product observable in solution was tetramethyllead.

(iv) The reaction of 3.6 × 10^{-5} mol of $HgCl_2$ (in 0.44 ml methanol) with 3.9 × 10^{-5} mol of the substrate (in 0.37 ml methanol) gave a similar immediate precipitate, but now the solution after 5 min contains tetramethyllead (0.040 *M*); trimethyllead chloride (0.034 *M*); methylmercuric chloride (0.022 *M*) and dimethylmercury (0.003 *M*). Reaction A was occurring and all the methylmercuric chloride was consumed during ca. 45 min.

Addition of hexamethyldilead to silver nitrate

(v) A black precipitate of silver was immediately produced when 5 × 10^{-5} mol of the substrate (in 0.3 ml methanol) were added in 1.1 × 10^{-4} mol of $AgNO_3$ (in 1 ml of 2% aqueous methanol). Only trimethyllead nitrate was observed in the solution. (N.B.: The rapid reactions 10a and 11 could have occurr-

$$(CH_3)_6Pb_2 + 2(CH_3)_3PbNO_3 \rightarrow 3(CH_3)_4Pb + Pb(NO_3)_2 \qquad (10a)$$

$$2(CH_3)_4Pb + 2AgNO_3 \rightarrow 2(CH_3)_3PbNO_3 + 2Ag + C_2H_6 \qquad (11)$$

ed to a small extent and the ethane product escaped detection.)

Addition of silver nitrate to hexamethyldilead

(vi) A similar immediate precipitation occurred on addition of 2.8 × 10^{-5} mol of $AgNO_3$ (in 0.35 ml 2% aqueous methanol) to 3.1 × 10^{-5} mol of the substrate (in 0.48 ml methanol). However, the solution now contains only tetramethyllead. Addition of further $AgNO_3$ solution led to the rapid formation of ethane (singlet at δ 0.85 ppm) at the expense of tetramethyllead according to reaction 11.

Addition of ferric chloride to hexamethyldilead

(vii) The orange colour of 1.0 × 10^{-4} mol of $FeCl_3$ (in 0.5 ml methanol) was rapidly discharged when added to 1.1 × 10^{-4} mol of the substrate (in 0.52 ml methanol) and lead chloride was precipitated. A thiocyanate test showed the absence of iron(III) and the addition of sodium cyanide precipitated iron(II) permitting a PMR spectrum to be taken. This showed only tetramethyllead was present in accordance with eq. 4.

Addition of hexamethyldilead to mercurous chloride

(viii) Since Hg_2Cl_2 is essentially insoluble in methanol, to a suspension of 2 × 10^{-5} mol in 0.6 ml methanol, was added $(CH_3)_6Pb_2$ (4.2 × 10^{-5} mol in 0.35 ml methanol). Mercury and lead chloride were precipitated and the solution contained tetramethyllead with a trace of trimethyllead chloride in accordance with eq. 5.

Addition of hexamethyldilead to cupric chloride

(xi) The green colour of 2.2 × 10^{-5} mol of $CuCl_2 \cdot 2H_2O$ (in 0.29 ml methanol) was immediately discharged on addition of 4.4 × 10^{-5} mol of hexamethyldilead (in 0.5 ml methanol) with the precipitation of white lead chloride and brown copper metal. The solution showed that only tetramethyllead was present (cf. 6).

(x) The addition of 9 × 10^{-5} mol of the substrate (in 0.57 ml methanol) to

8.2×10^{-5} mol of $CuCl_2 \cdot 2H_2O$ (in 0.5 ml methanol) was similarly rapid, however the initial precipitate was white and composed of lead chloride and cuprous chloride. The solution contained mostly tetramethyllead and also trimethyllead chloride, whose concentration increased over ca. 5 min at the expense of tetramethyllead. The cuprous chloride was rapidly converted to metallic copper while small peaks in the PMR spectrum arising from methane (δ 0.15 ppm) and ethane (0.85 ppm) were observed.

Addition of hexamethyldilead to cuprous chloride

(xi) 3.2×10^{-5} mol of hexamethyldilead (in 0.22 ml methanol) was added to a suspension of 2.1×10^{-5} mol of CuCl (in 0.6 ml methanol) to yield lead chloride, copper metal and tetramethyllead only, with some residual substrate (cf. eq. 7).

Reaction of hexamethyldilead with methylmercuric chloride

The main reaction is complete within the time of mixing at PMR concentrations (ca. 0.04 *M*) producing a white precipitate of lead chloride containing grey specks of mercury. The only products visible in the spectrum are tetramethyllead and dimethylmercury. Slower reactions then occur depending upon which reagent is in excess, methanolysis of hexamethyldilead or reaction A. However, this latter reaction is too slow to account for more than ca. 5% of the dimethylmercury present at the time of the first observation on the system.

The stoichiometry approximetely conforms to:

$$(CH_3)_6Pb_2 + 1.5\ CH_3HgCl \rightarrow 1.2(CH_3)_4Pb + 1.3(CH_3)_2Hg + 0.8\ PbCl_2 + 0.2\ Hg$$

although several experiments showed deviations from this equation.

When hexamethyldilead is reacted with trideutero-methylmercuric chloride the resonance of "dimethylmercury" is within experimental uncertainty half that observed in the above case. The product is thus CH_3HgCD_3 (a 1 : 1 mixture of $(CH_3)_2Hg$ and $(CD_3)_2Hg$ is possible but unlikely).

Discussion

At the concentrations employed for these and the previous studies [1], reaction 10, described in Part II [7] as having a second order rate constant of ca.

$$(CH_3)_6Pb_2 + 2(CH_3)_3PbCl \rightarrow 3(CH_3)_4Pb + PbCl_2 \qquad (10)$$

14 M^{-1} s^{-1} (25°C), will be a rapid reaction. (Reaction 10a will be even faster.) Trimethyllead chloride formed in the presence of hexamethyldilead will therefore not survive.

Type A behaviour reported for hexaethyldilead added to mercuric chloride at 1 : 1 and 1 : 2 ratios is only observed by us when hexamethyldilead is in excess and, in contrast, type B behaviour is observed for the reverse order of addition. In general both trimethyllead chloride and tetramethyllead are products and the situation is far more complex than was previously indicated.

In the systems involving mercuric chloride its reaction with hexamethyldilead certainly has the largest rate constant of any of the possible reactions. It would be expected to be at least as great as that for tetramethyllead and mercuric chloride (> 30 M^{-1} s^{-1}). However, when the rates of reactions are comparable with or faster than the mixing of reagents the consequences of inhomogeneity need to be considered. (A rate constant of 10^2 M^{-1} s^{-1} at initial concentrations of 0.04 *M* in each reactant means that their half-lives are only 0.25 s.)

If an element of added hexamethyldilead enters the bulk of the solution and reacts as in eq. 12 then the product trimethyllead chloride will diffuse into a

$$(CH_3)_6Pb_2 + HgCl_2 \rightarrow 2(CH_3)_3PbCl + Hg \qquad (12)$$

solution of mercuric chloride and react negligibly slowly (Reaction B). Further hexamethyldilead will react as above until the mercuric chloride concentration has fallen sufficiently to allow the slower reacting product trimethyllead chloride to compete with it. The difference between experiments *i* and *ii* is that in the former case there is ample mercuric chloride present to react with hexamethyldilead.

On the other hand, when mercuric chloride enters the solution the product of reaction 12 diffuses into regions rich in hexamethyldilead allowing reaction 10 to take place. When hexamethyldilead is in excess most of the trimethyllead chloride can be consumed without effective competition from the inadequately mixed mercuric chloride.

Allowing for composition changes due to subsequent reactions, i.e. reaction A, the result of experiment *ii* corresponds to 40% consumption of hexamethyldilead by trimethyllead chloride and 60% by mercuric chloride. (This was "improved" to 25% and 75% by addition of a concentrated solution of hexamethyldilead in 0.05 ml of benzene, but the partition ratio 0 : 100% corresponding to type A behaviour reported for addition of liquid hexaethyldilead could not be matched.)

The reaction of a suspension of mercurous chloride, experiment *viii*, most probably arises from a low concentration of mercuric chloride formed through:

$$HgCl_2 + Hg \rightleftharpoons Hg_2Cl_2 \; (K = 170) \qquad (12)$$

There will be no effective competitor for trimethyllead chloride so that reaction 12 is followed by reaction 10 giving Type B behaviour.

The case of silver nitrate is quite similar to that of mercuric chloride although the change from type A or type B behaviour with change in order of addition of the reagents is more clear cut. Reaction 13 is anticipated to be very fast since for hexaethylditin in ethanol 3×10^5 M^{-1} s^{-1} is reported [13].

$$(CH_3)_6Pb_2 + 2AgNO_3 \rightarrow 2(CH_3)_3PbNO_3 + 2Ag \qquad (13)$$

If reaction of the reagent with hexamethyldilead is not sufficiently faster than the reaction of trimethyllead chloride, then irrespective of the order of addition type B behaviour will be observed. This seems to be the case for ferric chloride, cupric chloride and especially, like mercurous chloride, the sparingly soluble cuprous chloride.

The observation of type A or B behaviour is thus not a feature of the mechanism of the primary reaction of the hexaalkyldilead with the metal salt but rather a consequence of ineffective reagent mixing and the competition between fast reactions.

In all cases the primary reaction appears to involve Pb—Pb bond cleavage for otherwise one would anticipate that alkyl groups would be lost from lead. When Pb—C cleavage occurs hydrocarbon products are obtained as in the case of tetraalkylleads reacting with silver salts [14,15] and with copper salts [16]. Such reactions are observed in our experiments only when tetramethyllead has been formed, and this would seem to be the case for reaction 9 of the previous study [1].

In the reaction of hexamethyldilead with methylmercuric chloride there is a rate controlling step which is first order in the two reactants, but the stoichiometry is complex and, in particular, non-integral. This was also found to be the case for the corresponding reaction of hexamethylditin [8]. In the latter case experiments with trideutero-methylmercuric chloride demonstrated that exclusive Sn—Sn cleavage occurred in the rate step, and that the products arose from competing reactions of transient methyltrimethylstannylmercury. The observed rate was found to be 29 times greater than that anticipated for Sn—CH_3cleavage, based upon the finding that Sn—CH_3 reactions in hexamethylditin and tetramethyltin have similar rates [17].

In contrast CD_3HgCl becomes converted into CD_3HgCH_3 in its reaction with hexamethyldilead. This could be taken to indicate the initial reaction involves Pb—CH_3 cleavage with subsequent or concerted formation of dimethyllead as discussed in Part II [7]. Competing reactions between dimethyllead and methylmercuric chloride, demethylation (eq. 15a) and insertion and decomposition (eq. 15b), would give the observed stoichiometric result with $\alpha = 0.6$, i.e.

$$\begin{aligned}&(CH_3)_6Pb_2 + RHgCl \rightarrow [(CH_3)_5Pb_2Cl] + RHgCH_3\\ &\qquad\qquad\qquad\qquad\quad \hookrightarrow (CH_3)_2Pb + (CH_3)_3PbCl \qquad (14)\end{aligned}$$

$$(CH_3)_6Pb_2 + 2(CH_3)_3PbCl \rightarrow 3(CH_3)_4Pb + PbCl_2 \qquad (10)$$

$$\alpha(CH_3)_2Pb + 2\alpha RHgCl \rightarrow \alpha PbCl_2 + 2\alpha RHgCH_3 \qquad (15a)$$

$$\begin{aligned}&(1-\alpha)(CH_3)_2Pb + (1-\alpha)RHgCl \rightarrow [(1-\alpha)(CH_3)_2RPbHgCl]\\ &\qquad\qquad\qquad\qquad\qquad\qquad\qquad \downarrow\\ &\qquad\qquad\qquad\qquad (1-\alpha)(CH_3)_2RPbCl + (1-\alpha)Hg \qquad (15b)\\ &\qquad\qquad\qquad\qquad\qquad \downarrow \tfrac{1}{2}(1-\alpha)(CH_3)_6Pb_2\\ &(1-\alpha)(CH_3)_3PbR + \tfrac{1}{2}(1-\alpha)(CH_3)_4Pb + \tfrac{1}{2}(1-\alpha)PbCl_2\end{aligned}$$

$\alpha : (1-\alpha) = 1.5 : 1$. This would mean that the observed rate constant is that for Pb—CH_3 cleavage which, when statistically corrected to 0.05 M^{-1} s^{-1}, is 6.7 times greater than that for the corresponding reaction of tetramethyllead. This must be compared with the rates of Pb—CH_3 cleavage by trimethyllead chloride and trimethyltin chloride which are respectively 130 and 12 times faster for hexamethyldilead than tetramethyllead [7]. Most important, however, is the certainty that all reactions subsequent to reaction 14 are faster.

It is evident, however, that the Pb—CH_3 cleavage reaction need not be the initial step (reaction 16), but rather that the reaction parallels that of hexamethylditin. In that case the Pb—Pb cleavage rate, 0.15 M^{-1} s^{-1}, is 20 times the rate of Pb—CH_3 cleavage in tetramethyllead and the ratio is quite similar to that for the tin substrates [8]. Mercury is then envisaged to arise from the decomposition (reaction 17a) of the intermediate, methyl trimethylplumbyl mercury, in competition with its reaction with methylmercuric chloride (reaction 17b), which must be unusually fast.

$$(CH_3)_6Pb_2 + RHgCl \rightarrow (CH_3)_3PbHgR + (CH_3)_3PbCl \qquad (16)$$

$$(CH_3)_3PbCl + \tfrac{1}{2}(CH_3)_6Pb_2 \rightarrow \tfrac{3}{2}(CH_3)_4Pb + \tfrac{1}{2}PbCl_2 \qquad (10)$$

$$(CH_3)_3PbHgR \rightarrow (1-\beta)(CH_3)_3PbR + (1-\beta)Hg \qquad (11a)$$

$$\xrightarrow{2\beta RHgCl} 3\beta RHgCH_3 + \beta PbCl_2 \qquad (17b)$$

The observed stoichiometry would be obtained for $\beta = 0.7$, i.e. $\beta : (1-\beta) \simeq 2 : 1$, although this must be an average value since the competition between reactions 17a and 17b will change as the methylmercuric chloride concentration changes. To some extent this could be the origin of our problems in establishing the stoichiometry of this reaction. On the other hand we have been unable to account for the variations in the manner employed in the case of methyltrimethylstannyl mercury, and they seem to have much of the character of random experimental errors. Substantial variations in the ratio of reactants do not yield useful information since methanolysis or reaction A become important.

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