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The Reaction between Dimethylmagnesium and Lithium Aluminum Hydride in Diethyl Ether and Tetrahydrofuran

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The reactions between lithium aluminum hydride and dimethylmagnesium in diethylether and THF have been studied in detail. Magnesium hydride as an insoluble solid was formed when $(CH_3)_2Mg$ *and* $LiAlH_4$ *were allowed to react in molar ratios varying from 4:1 to 1:2 in diethyl ether. The identity of the soluble bi-products,* $LiAlH_n(CH_3)_{4-n}$ $(n = 0, 1, 2, 3, 4)$*, was established by infrared and NMR spectral comparison with the products formed on redistribution of* $LiAlH_4$ *with* $LiAl(CH_3)_4$*. On the other hand, a clear solution containing a mixture of* CH_3MgH *and* $LiAl(CH_3)_4$ *was found in the reaction of* $(CH_3)_2Mg$ *with* $LiAlH_4$ *in THF in ratio 4:1. The reaction in 2:1 ratio yielded a mixture of* $CH_3Mg_2H_3$ *and* $LiAlH(CH_3)_3$ *in THF solution. Further addition of* $LiAlH_4$ *to* $(CH_3)_2Mg$ *in THF produced insoluble* MgH_2*. The course of these reactions has been justified by the help of IR and NMR analysis.*

Introduction

During the last few years, we have been engaged in a general study of the preparation and characterization of complex metal hydrides of beryllium [1], magnesium [2], copper [3, 4] and zinc [5, 6]. The preparation of these complexes involves the alkyl–hydrogen exchange in the reaction of an ether soluble 'ate' complex, $(M_nM'_mR_{2m+n})$ with $LiAlH_4$ resulting in the precipitation of the insoluble hydride, $(M_nM'_mH_{2m+n})$, leaving in solution the ether soluble $LiAlR_nH_{4-n}$. In an attempt to prepare $LiMgH_3$ by the reaction of $LiMg(CH_3)_3$ with $LiAlH_4$, a mixture of LiH, MgH_2 and $LiMgH_3$ was found. It was suggested that $LiAl(CH_3)_3$ was not stable in ether and dissociated into CH_3Li and $(CH_3)_2Mg$ which on reaction with $LiAlH_4$ produced LiH and MgH_2. In order to

$$LiH + MgH_2 \xleftarrow{LiAlH_4} CH_3Li + (CH_3)_2Mg \rightleftharpoons Li(Mg(CH_3)_3 \xrightarrow{LiAlH_4} LiMgH_3 \qquad (1)$$

justify our explanation, it has been considered worthwhile to perform the reactions between $(CH_3)_2Mg$ with $LiAlH_4$ in detail. Recently, during the course of the reactions of Ph_2Mg with $LiAlH_4$ in THF, we have observed the existence of complexes of the type PhMgH and $PhMg_2H_3$ in solution [7]. In a hope to find similar results in the reaction of $(CH_3)_2Mg$ with $LiAlH_4$, we have extended our efforts in THF solvent also.

Experimental

All the operations were performed under nitrogen at the bench using typical Schlenk tube techniques [8] or in a dry box equipped with a recirculating system using manganous oxide columns to remove oxygen and moisture.

Instrumentation

Infrared spectra were obtained using a Perkin-Elmer 621 spectrophotometer. Spectra of solutions were run in matched 0.10 mm path length KBr cells. Proton NMR spectra were obtained using Varian A-60 spectrometer.

Materials

Solvents were distilled immediately prior to use over $LiAlH_4$ (ether) or $NaAlH_4$ (THF). Dimethylmagnesium [9] was prepared by stirring a mixture of dimethylmercury with magnesium metal at room temperature for 24 hours followed by extraction of the crude reaction mixture with ether or THF. A solution of lithium aluminum hydride. $LiAlH_4$ (Ventron, Metal Hydrides Division) in diethyl ether or THF was prepared by refluxing overnight followed by filtration through a glass-fritted funnel using predried celite analytical filter-aid. The clear solution was standardized by aluminum and hydrogen gas analysis.

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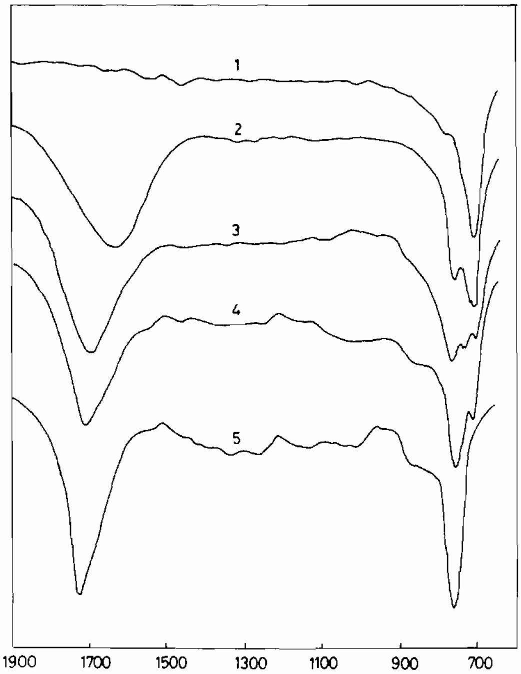

Fig. 1. Infrared spectra of supernatant solutions obtained by adding $LiAlH_4$ to $(CH_3)_2Mg$ in diethyl ether. $LiAlH_4$:$(CH_3)_2Mg$ ratio: (1) 1:2 (2) 2:3 (3) 1:1 (4) 2:1 (5) Pure $LiAlH_4$.

Lithium tetramethylaluminate ($LiAl(CH_3)_4$) [10] was prepared by the reaction of CH_3Li with $(CH_3)_3$-Al (Texas Alkyls, Inc.). Methyllithium was prepared by the reaction of excess lithium metal with dimethylmercury (Org-Met.) in diethylether (or THF) at −20 °C. Lithium metal was obtained as a 30% dispersion in petrolatum from Alfa-Ventron. The solution of $LiAl(CH_3)_4$ was standardized by aluminum analysis.

Analytical

Gas analysis was accomplished by hydrolysis of samples with methanol or HCl on a standard vacuum line equipped with a Toepler pump. Aluminum was determined by adding excess of EDTA and back-titrating with standard zinc acetate at pH-4 in 50% ethanol with dithiazone as an indicator. Magnesium was determined at pH-10 by EDTA titration using Eriochrome Black-T as an indicator. When aluminum was present, it was masked by complexation with triethanolamine.

Infrared Study of the Reaction of $LiAlH_4$ with $(CH_3)_2Mg$

(a) In Diethyl Ether

10 ml of 0.95 *M* solution of $(CH_3)_2Mg$ (9.6 mmol) in diethylether was diluted to 100 ml and was placed in a two-neck round bottom flask fitted with a condenser and a three-way stopcock. Increments (2.2 ml each time) of 1.10 *M* solution of $LiAlH_4$ (2.4 mmol) in diethyl ether were added *via* syringe under nitrogen to the magnetically stirred $(CH_3)_2Mg$ solution. After each addition, the solution was stirred for 15 minutes at room temperature and the precipitate formed was allowed to settle. Infrared spectra were obtained by withdrawing samples of the supernatant solution by syringe under nitrogen. The additions of $LiAlH_4$ were continued until the ratio of $LiAlH_4$ to the original $(CH_3)_2Mg$ was 2:1. The infrared spectra obtained in this way are shown in Fig. 1.

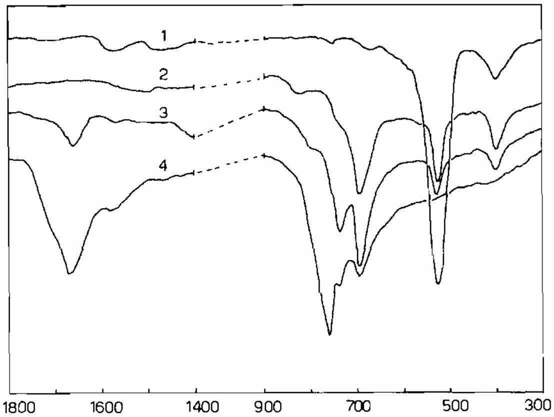

Fig. 2. Infrared spectra of supernatant solutions obtained by adding $LiAlH_4$ to $(CH_3)_2Mg$ in THF. $LiAlH_4$:$(CH_3)_2Mg$ ratio: (1) Pure $(CH_3)_2Mg$ (2) 1:4 (3) 1:2 (4) 1:1. Reaction time of 1 hr.

In a similar manner, the entire experiment was repeated by reversing the mode of addition using 1.0 *M* dimethylmagnesium and 0.65 *M* lithium aluminum hydride solutions.

(b) In THF

The above experiment was repeated in a similar manner using dimethylmagnesium and $LiAlH_4$ in THF. The infrared spectra of the supernatant solutions were recorded (Fig. 2).

Redistribution of $LiAlH_4$ and $LiAl(CH_3)_4$ in Diethylether and in THF

Reactions between $LiAlH_4$ and $LiAl(CH_3)_4$ were performed by mixing standard solutions of the reagents in ratios appropriate to produce $LiAlH(CH_3)_3$, $LiAlH_2(CH_3)_2$ and $LiAlH_3(CH_3)$. After stirring at room temperature for 1 hour, the infrared spectra were obtained on the resulting solution.

IR in Et_2O

νAl–H of (a) $LiAlH(CH_3)_3$ = 1680 cm^{-1}, (b) $LiAlH_2(CH_3)_2$ = 1706 cm^{-1}; (c) $LiAlH_3(CH_3)$ = 1725 cm^{-1}.

IR in THF

νAl–H of (a) $LiAlH(CH_3)_3$ = 1656 cm^{-1}, (b) $LiAlH_2(CH_3)_2$ = 1668 cm^{-1}, (c) $LiAlH_3(CH_3)$ = 1680 cm^{-1}. (Fig. 3).

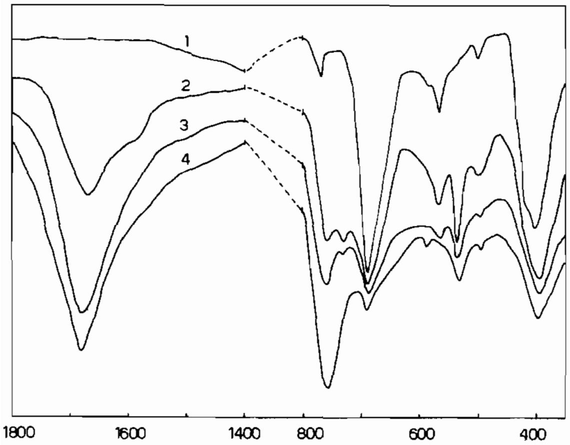

Fig. 3. Infrared spectra of (1) $LiAl(CH_3)_4$, (2) $LiAlH(CH_3)_3$, (3) $LiAlH_2(CH_3)_2$, (4) $LiAlH_3(CH_3)$ in THF.

Reactions Between $LiAlH_4$ and $(CH_3)_2Mg$ in Diethylether

(1) The Ratio of $LiAlH_4$ to $(CH_3)_2Mg$ = 1.0:4.0

A diethylether solution of $LiAlH_4$ (3.0 mmol in 10 ml) was added dropwise by syringe (under nitrogen flush) to a magnetically stirred solution of $(CH_3)_2Mg$ (12.0 mmol in 30 ml). A white precipitate formed immediately. After stirring for one hour at room temperature the mixture was filtered. The resulting solid was washed with diethylether and dried under vacuum at room temperature.

Analysis of the solid showed it to contain Mg, Al and H in ratios of 1.00:0.08:2.13 and about 49% of the starting magnesium. An X-ray powder diffraction pattern of the solid was found to be similar to that of MgH_2. Analysis of the supernatant solution showed it to contain Li, Mg, Al, H and CH_3 in molar ratios of 1.04:2.03:1.00:0.00:7.92. Calcd. for $[LiAl(CH_3)_4 + 2(CH_3)_2Mg]$ = 1.00:2.00:1.00:0.00:8.00. The filtrate showed no νAl–H stretching in the IR spectrum.

(2) The Ratio of $LiAlH_4$ to $(CH_3)_2Mg$ = 1.0:2.0

This reaction was performed in a manner identical with that above. The precipitate, after isolating and drying, was found to contain Mg, H and Al in molar ratios of 1.00:2.12:0.07. The solid contained about 97% of the starting magnesium. The filtrate contained Li, Mg, Al, H and CH_3 in molar ratios of 1.03:0.06:1.00:0.00:4.02. $[LiAl(CH_3)_4]$, the infrared spectrum of the filtrates showed no Al–H stretching in the region 1650–1900 cm^{-1}.

(3) Molar Ratio of $LiAlH_4$ to $(CH_3)_2Mg$ = 0.67:1.00

The reaction of $LiAlH_4$ with $(CH_3)_2Mg$ in 0.67:1.00 molar ratio was performed in a similar manner as above. In this reaction about 96% of the starting magnesium was recovered in the precipitate which contained Mg, Al and H in molar ratios of 1.00:0.07:2.07. The filtrate contained Li, Mg, Al, H and CH_3 in molar ratios of 1.03:0.06:1.00:0.97:2.98. Calcd. for $LiAlH(CH_3)_3$ = 1.00:0.00:1.00:1.00:3.00. It gave an infrared band at 1680 cm^{-1} due to νAl–H stretching.

(4) Reaction of 1.0 $LiAlH_4$ with 1.0 $(CH_3)_2Mg$

In this reaction about 97% of the starting magnesium was recovered in the precipitate which gave an analysis of Mg, Al and H in molar ratios of 1.00:0.08:2.09. The filtrate contained Li, Mg, Al, H and CH_3 in molar ratios of 1.02:0.04:1.00: 1.99:2.01. The infrared spectrum of the solution showed a band at 1707 cm^{-1} due to νAl–H stretching (characteristic of $LiAlH_2(CH_3)_2$).

(5) $LiAlH_4$:$(CH_3)_2Mg$ = 2.0:1.0

In this reaction about 95.5% of the starting magnesium in the precipitate which contained Mg, Al and H in molar ratios of 1.00:0.08:2.13. The filtrate which showed νAl–H at 1725 cm^{-1}, contained Li, Mg, Al, H and CH_3 in molar ratios of 1.03:0.04:1.00:3.05:0.98 (corresponded to $LiAlH_3(CH_3)$).

Reaction of $LiAlH_4$ with $(CH_3)_2Mg$ in 1:4 Ratio in THF

4.0 mmoles of $LiAlH_4$ in THF (7.5 ml) were added dropwise to a magnetically stirred solution of $(CH_3)_2Mg$ (16.0 mmol) in 20 ml of THF. Reaction was exothermic and a clear solution remained even when it stirred for 1 hour. Infrared spectrum of this solution showed the absence of νAl–H and exhibited an absorption band at ~530 cm^{-1} characteristic of νMg–CH_3. NMR spectrum gave a singlet at 11.74 τ (due to methyl protons attached to magnesium) and a sextet centered at 11.32 τ (due to $LiAl(CH_3)_4$). The ratio of singlet to sextet was found to be 1.0:1.0. The analyses of the solution showed that it contained Li, Mg, Al, H and CH_3 in molar ratios of 1.02:4.03:1.00:3.97:8.02. Calcd. for $[LiAl(CH_3)_4 + 4CH_3MgH]$ 1.00:4.00:1.00:4.00:8.00.

Reaction of $LiAlH_4$ with $(CH_3)_2Mg$ in 1:2 Ratio in THF

To 10 ml of 0.85 *M* $(CH_3)_2Mg$ solution in THF (8.5 mmol) was added dropwise 5.3 ml of 0.80 *M* solution of $LiAlH_4$ in THF (4.24 mmol) with stirring at room temperature, producing a clear solution. The infrared spectrum of this solution exhibited an absorption band at 1656 cm^{-1} characteristic of Al–H stretching in $LiAlH(CH_3)_3$ in THF and a band at ~530 cm^{-1} due to νMg–CH_3. Analysis of the solution showed that it contained Li, Mg, Al, H and CH_3 in molar ratios of 1.03:2.02:1.00:4.04:3.97 (corresponded to $LiAl(CH_3)_3 + (CH_3)Mg_2H_3$).

Reaction of $LiAlH_4$ with $(CH_3)_2Mg$ in 1:1 Ratio in THF

Addition of 6.5 mmol in $LiAlH_4$ in THF (12 ml) to a well stirred solution of $(CH_3)_2Mg$ (6.5 mmol) in THF (15 ml), resulted in an insoluble solid. The reaction mixture was stirred for 1 hour and the insoluble solid was filtered washed with THF and dried *in vacuo*. Analysis of the solid revealed that it contained Mg, H and Al in ratios 1.00:1.98:0.06. It contained about 90% of the starting magnesium. Filtrate of the reaction showed in the infrared spectrum, a band at 1670 cm^{-1} characteristic of Al–H stretching in $LiAlH_2(CH_3)_2$ in THF. Analysis of the solution revealed that it contained Li, Mg, Al, H and CH_3 in molar ratios of 1.02:0.07:1.00:2.04:2.01 (corresponded to $LiAlH_2(CH_3)_2$).

Preparation of CH_3MgH by the Reaction of $(CH_3)_2Mg$ with MgH_2 in THF

4.0 mmol of $(CH_3)_2Mg$ in THF (15 ml) was allowed to react with 4.0 mmol of MgH_2 slurry in THF (12 ml) with continued stirring at room temperature. A clear solution resulted within 15 minutes. The solution was analyzed which showed that it contained Mg, H and CH_3 in molar ratios 1.00:0.96:1.03.

Reaction of CH_3MgH with $LiAlH_2(CH_3)_2$ in THF in 2:1 Ratio

4.0 mmol of a THF (15 ml) solution of $LiAlH_2(CH_3)_2$ was prepared by the redistribution reaction of $LiAlH_4$ (2.0 mmol) with $LiAl(CH_3)_4$ (2.0 mmol) in THF. Addition of the 4.0 mmol of $LiAlH_2(CH_3)_2$ to a magnetically stirred solution of CH_3MgH (4.0 mmol) in THF resulted in a clear solution. Infrared spectrum of this solution showed a band at 1657 cm^{-1} characteristic of $LiAlH(CH_3)_3$ in THF and the band at 428 cm^{-1} suggested the presence of Mg–CH_3 bond. *Anal.*: Calcd. for ($LiAlH(CH_3)_3$ + $CH_3Mg_2H_3$) = Li:Mg:Al:H:CH_3 = 1.00:2.00:1.00:4.00:4.00. Found: 1.04:1.98:1.00:3.96:4.04.

Reaction of $(CH_3)_2Mg$ with MgH_2 in 3:1 Ratio in THF

Addition of 6.0 mmol of $(CH_3)_2Mg$ in 17 ml THF to a magnetically stirred slurry of MgH_2 (2.0 mmol) in THF (10 ml), resulted in a clear solution within 1 hour. Analysis showed that it contained Mg, H and CH_3 in ratios 1.00:1.45:0.52 (corresponded to $CH_3Mg_2H_3$). NMR spectrum gave a singlet at 11.72 τ (3.51 ppm upfield to THF).

Reaction of $CH_3Mg_2H_3$ with $LiAlH_4$ in 1:1 Ratio in THF

To a well stirred solution of $CH_3Mg_2H_3$ (4.0 mmol) in THF (18 ml) was added dropwise a solution of $LiAlH_4$ (4.0 mmol) in THF (8.5 ml). The reaction mixture was stirred at room temperature for 1 hour resulting in an insoluble white solid. The solid was filtered, washed with THF and dried *in vacuo*. Analysis of the solid showed that it contained Mg and H in ratio 1.00:2.02. The filtrate's analysis revealed that it contained Li, Al, H, CH_3 and Mg in ratio 1.02:1.00:2.02:2.03:0.07. Infrared spectrum showed a band at 1680 cm^{-1} characteristic of Al–H stretching in $LiAlH_3CH_3$.

Results and Discussion

Addition of $LiAlH_4$ to $(CH_3)_2Mg$ in 1:4, 1:2, 2:3, 1:1 and 2:1 in diethyl ether, or the corresponding inverse addition, resulted in the precipitation of MgH_2. The course of the reaction was checked by infrared study of the supernatant solution resulting after each incremental addition.

When $(CH_3)_2Mg$ was added to $LiAlH_4$ in four equal increments such that the ratio of total magnesium to aluminum was 1:2, 1:1, 3:2 and 2:1 after each of the reactions proceeded in a stepwise fashion exchanging methyl groups on magnesium for hydrogen on aluminum as shown in eqns. 1–4.

$$(CH_3)_2Mg + 2LiAlH_4 \longrightarrow MgH_2 + 2LiAl(CH_3)H_3 \qquad (1)$$

$$(CH_3)_2Mg + 2LiAl(CH_3)H_3 \longrightarrow MgH_2 + 2LiAl(CH_3)_2H_2 \qquad (2)$$

$$(CH_3)_2Mg + 2LiAl(CH_3)_2H_2 \longrightarrow MgH_2 + 2LiAl(CH_3)_3H \qquad (3)$$

$$(CH_3)_2Mg + LiAl(CH_3)_3H \longrightarrow MgH_2 + 2LiAl(CH_3)_4 \qquad (4)$$

Support for these reactions proceeding as shown above was provided by the elemental analysis of the products and by the infrared spectra of the supernatant solutions, which were found to be identical to that of $LiAl(CH_3)_nH_{4-n}$ in diethyl ether prepared by allowing $LiAl(CH_3)_4$ to redistribute with $LiAlH_4$ in appropriate stoichiometry (eqns. 5–7).

$$LiAlH_4 + 3LiAl(CH_3)_4 \longrightarrow 4LiAl(CH_3)_3H \qquad (5)$$

$$LiAlH_4 + LiAl(CH_3)_4 \longrightarrow 2LiAl(CH_3)_2H_2 \qquad (6)$$

$$3LiAlH_4 + LiAl(CH_3)_4 \longrightarrow 4LiAl(CH_3)H_3 \qquad (7)$$

When $LiAlH_4$ was added to a solution of $(CH_3)_2Mg$ in diethylether in 1:2 molar ratio, the reaction proceeded according to eqn. 8:

$$LiAlH_4 + 2(CH_3)_2Mg \longrightarrow 2MgH_2 + LiAl(CH_3)_4 \qquad (8)$$

Further addition of $LiAlH_4$ to this reaction mixture resulted in the redistribution of $LiAlH_4$ and the $LiAl(CH_3)_4$ formed in the reaction and $LiAl(CH_3)_nH_{4-n}$ was formed as diethylether soluble byproduct according to eqns. 5–8).

Interestingly, when $LiAlH_4$ was reacted with $(CH_3)_2Mg$ in THF in 1:4 molar ratio, a clear solution remained even after stirring for 1 hour. Infrared spectrum of the solution showed the absence of Al–H stretching in the region ~1700 cm^{-1}; however, the band at 530 cm^{-1} present in the infrared spectrum of $(CH_3)_2Mg$ in THF due to $Mg–CH_3$ [11] stretching, was still there indicating the presence of $Mg–CH_3$ bond. Since MgH_2 is practically insoluble in THF, the reaction course observed in ether (eqn. 9) is undoubtedly not involved here.

$$4(CH_3)_2Mg + LiAlH_4 \xrightarrow{Et_2O} 2MgH_2 + 2(CH_3)_2Mg + LiAl(CH_3)_4 \qquad (9)$$

NMR spectrum of the reaction mixture gave a singlet at 11.74 τ (3.53 ppm upfield from THF multiplet) and a sextet centered at 11.32 τ. The upfield singlet at 11.74 τ was almost at the position of $(CH_3)_2Mg$ and the sextet corresponded to that of $LiAl(CH_3)_4$ [12]. The 1:1 ratio of singlet to sextet suggested the number of methyl groups attached to magnesium are the same as that attached to aluminum. The infrared and NMR data favor the following pathway (eqn. 10) in THF.

$$4(CH_3)_2Mg + LiAlH_4 \xrightarrow{THF} 4CH_3MgH + LiAl(CH_3)_4 \qquad (10)$$

When $LiAlH_4$ was added further to the above reaction mixture so as to reach $LiAlH_4$ to $(CH_3)_2Mg$ in 1:2 molar ratio, a clear solution remained. Infrared spectrum of the solution showed a band at 1658 cm^{-1} due to Al–H stretching suggesting the presence of $LiAl(CH_3)_3H$ in THF and also showed a band at 526 cm^{-1} characteristic of $Mg–CH_3$. The infrared data favor the following pathway (eqns. 10–12).

$$4(CH_3)_2Mg + LiAlH_4 \longrightarrow 4CH_3MgH + LiAl(CH_3)_4 \qquad (10)$$

$$LiAl(CH_3)_4 + LiAlH_4 \longrightarrow 2LiAl(CH_3)_2H_2 \qquad (11)$$

$$4CH_3MgH + 2LiAl(CH_3)_2H_2 \longrightarrow 2CH_3Mg_2H_3 + 2LiAl(CH_3)_3H \qquad (12)$$

Further support for these reactions proceeding as shown above is provided by the fact that $CH_3Mg_2H_3$ and $LiAl(CH_3)_3H$ were formed according to the eqn. 13 when $LiAl(CH_3)_2H_2$ was added to a sample of $(CH_3)_2MgH$. $(CH_3)_2MgH$ was prepared independently by the reaction of MgH_2 with $CH_3)_2Mg$ in THF.

$$MgH_2 + (CH_3)_2Mg \xrightarrow{THF} 2CH_3MgH \qquad (13)$$

Further addition of $LiAlH_4$ to the products of the eqn. (13) produced insoluble MgH_2. The reaction (eqn. 14) could be proposed, for the reaction of $(CH_3)_2Mg$ with $LiAlH_4$ in 1:1 molar ratio in THF, on the basis of elemental analysis, and infrared spectrum which showed the presence of Al–H stretching band at 1670 cm^{-1} characteristic of $LiAlH_2(CH_3)_2$ in THF. However, the less amount of MgH_2 as insoluble solid and the presence of magnesium in the supernatant solution could be explained by an equilibrium reaction.

$$2(CH_3)_2Mg + 2LiAlH_4 \longrightarrow CH_3Mg_2H_3 + LiAlH_2(CH_3)_2 + LiAlH_3(CH_3) \rightleftharpoons 2MgH_2 + 2LiAlH_2(CH_3)_2 \qquad (14)$$

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