If reaction is incomplete, and metallic cesium remains with the excess aluminum, treatment with diglyme will lead to orange-brown soluble cleavage products which finally form a diglyme-insoluble material as an impurity in the $CsAlH_4$.

Sodium Aluminum Tetraethyl.—To 6 g. (0.26 mole) of sodium was added 7 g. (0.26 mole) of activated aluminum powder in 125 ml. of toluene. To this mixture was added 2 ml. of triethylaluminum catalyst. The resulting mixture was allowed to react for 16 hr. at 160° and 2000 p.s.i. pressure of hydrogen and ethylene. Filtration of the reaction mass, followed by washing with hexane, yielded 23.06 g. of solids. The toluene filtrate was diluted to 500 ml. with hexane to precipitate additional solid product weighing 10.32 g. The yield of sodium aluminum tetraethyl was 77.5%.

Anal. Calcd. for $NaAl(C_2H_5)_4$: Na, 13.8; Al, 16.3; ethane evolved on hydrolysis, 24.08 mmoles/g. Found (fraction 1): Na, 14.6; Al, 16.2; ethane evolved, 23.8 mmoles/g.; (fraction 2): Na, 14.7; Al, 16.9; ethane evolved, 25.3 mmoles/g.

$NaAl(C_2H_5)_2H_2$.—This reaction was run exactly as described for the preparation of sodium aluminum tetraethyl except that the amount of ethylene added was controlled. The yield as solid product obtained in two fractions was 87%.

Anal. Calcd. for $NaAl(C_2H_5)_2H_2$: Na, 20.9; Al, 24.6; gas evolved on hydrolysis, 36.4 mmoles/g. Found (fraction 1): Na, 20.3; Al, 24.5; gas evolved, 35.3 mmoles/g.; (fraction 2): Na, 21.9; Al, 24.9; gas evolved, 37.3 mmoles/g.

Preparation of $LiAlH_4$ from $NaAlH_4$ and LiCl.—About 13 g. of commercial LiCl (Foote Mineral, 99±%; dried *in vacuo* at 100–120°) was charged to a 250-ml. Magne-Dash autoclave with an equimolar amount or slight excess (up to 4%) of crude $NaAlH_4$ (prepared by the direct synthesis in toluene), 0.5 mole % $LiAlH_4$ as reaction initiator, and 100 ml. of diethyl ether (distilled over $LiAlH_4$). After a 3-hr. reaction at a temperature preferably near room temperature (see Table IX), the resulting product solution of $LiAlH_4$ in ether was filtered and the solids were washed with fresh ether. Filtration difficulties were eliminated by using Celite as filter aid. Crystalline LiAlH was recovered from the solution in diethyl ether as follows: dry toluene (about 5 ml./g. of $LiAlH_4$) was added to the solution, and ether then was distilled from the mixture *in vacuo* (40–50° at 400–150 mm.). Crystalline $LiAlH_4$ precipitated during this procedure and finally a slurry in toluene was obtained.[11] The product then was separated by filtration and dried *in vacuo* at room temperature. The purity of the products (96–99%) was determined by elemental analysis (hydride content measured by gas evolution upon hydrolysis). The impurities consisted of 0.4–0.6% Na and 0.1% Cl.

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[11] The $LiAlH_4$ recovery is virtually quantitative, if the ether is sufficiently removed.

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Complex Aluminum Hydrides Containing Nitrogen, Phosphorus, and Arsenic

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Ammonia, phosphine, and arsine react with aluminum hydrides to evolve hydrogen and form a variety of complex hydrides. Under the proper conditions, definite compounds $LiAl(NH_2)_4$, $NaAl(NH_2)_4$, and $LiAl(PH_2)_4$ were prepared. The new compound $LiAl(PH_2)_4$ reacts with ethyl iodide to form ethylphosphine in high yield but reactions with silicon halogen compounds were less straightforward.

Introduction

Reactions between hydrides of the fifth group elements and lithium aluminum hydride have been reported by a number of investigators. Most of these reactions have been carried out in ethyl ether with the stoichiometry somewhat obscure. The equation for the reaction between the hydride and ammonia has been reported[1] as

$$2LiAlH_4 + 5NH_3 \longrightarrow [LiAlH(NH_2)_2]_2NH + 6H_2 \quad (1)$$

More work has been done with alkylated compounds. With primary amines one hydrogen is replaced rapidly

$$LiAlH_4 + 4RNH_2 \longrightarrow LiAl(RNH)_4 + 4H_2 \quad (2)$$

but eventually the second hydrogen does react.[2] Similar equations have been proposed for secondary amines and primary and secondary phosphines.[3,4]

In the present work the stoichiometry of the reactions of ammonia, phosphine, and arsine with aluminum hydrides has been further studied. The course of the reaction can be controlled and definite products containing nitrogen and phosphorus have been isolated.

Results and Discussion

Under the proper conditions alkali metal aluminum hydrides reacted with ammonia, phosphine, and arsine according to the equation

$$MAlH_4 + 4EH_3 \longrightarrow MAl(EH_2)_4 + 4H_2 \quad (3)$$

Products of the composition $MAl(EH_2)_4$ were prepared from ammonia and phosphine with lithium or sodium aluminum hydride. Arsine also reacted in the same manner but no attempt was made to isolate a complex aluminum dihydrogen arsenide product. On hydrolysis these aluminum compounds released the fifth group hydrides quantitatively according to the stoichiometry

$$MAl(EH_2)_4 + 4HOH \longrightarrow 4EH_3 + MAl(OH)_4 \quad (4)$$

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[1] A. E. Finholt, A. C. Bond, Jr., and H. I. Schlesinger, *J. Am. Chem. Soc.*, **69**, 1199 (1947).

[2] J. A. Krynitsky, J. E. Johnson, and H. W. Carhart, *ibid.*, **70**, 486 (1948).

[3] A. B. Burg and K. Modritzer, *J. Inorg. Nucl. Chem.*, **13**, 318 (1960).

[4] J. T. Yoke, Dissertation, University of Michigan, 1954.

Amides of the formula $NaAl(NH_2)_4$ and $KAl(NH_2)_4$ have been prepared by Bergstrom by the reaction of the alkali metals, aluminum, and ammonia.[5] The amides prepared from the hydrides have the same properties of decomposition under heat and solubility in liquid ammonia as those reported by Bergstrom.

Ammonia reacted quickly and simply according to eq. 3 in the absence of a solvent. In the presence of ethyl ether or diglyme the stoichiometry was dependent on the ratio of the moles of the reagents put in contact with each other. In the presence of a high ammonia to hydride ratio the stoichiometry followed eq. 3. In the presence of a high hydride to ammonia ratio, up to three moles of hydrogen were evolved rapidly for every mole of ammonia that reacted, suggesting that all three ammonia hydrogens can be displaced. In practice this was the situation at the start of all reactions because the volatility of ammonia precluded having a high concentration of this gas in solution at room temperature. It is not surprising therefore that more than one mole of hydrogen was evolved per mole of ammonia in the initial stages of all reactions. In some experiments, probably more complex aluminum nitrogen lattices were formed than indicated in eq. 1 since all three hydrogens of ammonia can react. If more ammonia was added to these complex products, hydrogen continued to be evolved but more slowly as the reaction product approached an aluminum to nitrogen ratio of 1:4. The slow reaction may involve a gradual conversion of Al–N–Al bonds to amide groups by the ammonia. On the other hand, when fresh lithium aluminum hydride was added to a product that was essentially (88%) $LiAl(NH_2)_4$, a rapid evolution of hydrogen occurred. This confirms the improbability of the reaction proceeding solely according to eq. 3 at the early part of any of the reactions. It is apparent that a wide variety of complex hydrides are possible since all of the hydrogens on the aluminum or the nitrogen may be replaced.

In diglyme and tetrahydrofuran, phosphine reacted with lithium aluminum hydride according to eq. 3 to form a soluble product. This differs from ammonia, which formed insoluble products and did not necessarily follow eq. 3. In these solvents there was no evidence of a variation from eq. 3 for phosphine even when the concentration of hydride was high and the concentration of phosphine was low. By contrast, in ethyl ether almost three moles of hydrogen were evolved for every mole of phosphine that reacted and an insoluble product formed. While diglyme and tetrahydrofuran solvate lithium aluminum hydride much more strongly than ethyl ether, there is no detailed explanation, as yet, for this solvent effect.

Another difference between the group five hydrides was observed in reaction rates. Ammonia reacted more rapidly than arsine, which in turn reacted more rapidly than phosphine. While these observations need the support of precise kinetic studies they are in keeping with the relative electronegativities of Sanderson.[6] Thus relative to the central element the hydrogen is most positive in ammonia and least positive in phosphine. The most positive hydrogen should combine most readily with a negative hydride hydrogen, or, from the standpoint of a displacement reaction, ammonia is the strongest base and phosphine the weakest.

Solutions of aluminum hydride reacted with ammonia with a stoichiometry which also varied with the ratio of hydride to ammonia. No attempt was made to isolate products of a definite composition. Phosphine did not react with aluminum hydride solutions in ethyl ether or diglyme.

The alkali metal aluminum amides are soluble in liquid ammonia but in no other solvents that were tested. The lack of a good solvent was a restriction on investigation of the nucleophilic properties of these reagents. Undoubtedly one factor in the usefulness of lithium aluminum hydride is its solubility in ether solvents. It might be expected that the complex amides would give new or more clean cut substitution reactions just as lithium aluminum hydride does in contrast with lithium hydride. Unlike lithium aluminum hydride lithium tetraamidoaluminate did not react with ethyl iodide in the absence or presence of an ether medium. The diglyme soluble lithium tetrakis-(dihydrogenphosphido)-aluminate did give typical organic and inorganic substitution reactions. This reagent can be used to introduce PH groups into molecules but not enough experiments have been performed yet to properly evaluate the compound.

Lithium tetrakis-(dihydrogenphosphido)-aluminate reacted with ethyl iodide to give ethylphosphine and phosphine. Hydrolysis of the reaction product gave more ethylphosphine for a total yield of 97% based on ethyl iodide. Phosphine was evolved as a by-product either by ethyl iodide catalysis[4] or by base displacement of phosphine by ethylphosphine from the reagent. The latter hypothesis was confirmed as a possibility by reaction of ethylphosphine with the phosphido compound.

A reaction of silicon tetrachloride with lithium tetrakis-(dihydrogenphosphido)-aluminate was inconclusive. With trimethylsilyl chloride it was hoped that a replacement of the chloride would give high yields of mono-(trimethylsilyl)-phosphine. Actually a low yield of a mixture of mono and bis silyl compounds was obtained, no better than has been reported using the simple lithium hydrogen phosphides.[7]

Experimental

Materials.—Lithium aluminum hydride was purified by dissolving the commercial grade from Metal Hydrides in ethyl ether, filtering, and evaporating the solvent to give a 97% pure product. Sodium aluminum hydride, 98% purity, from Metal Hydrides, was used without further treatment. Aluminum hydride was prepared by adding aluminum chloride to an ethyl ether solution of lithium aluminum hydride.[1] Diglyme was added to aluminum

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[5] F. W. Bergstrom, *J. Am. Chem. Soc.*, **45**, 2788 (1923).

[6] R. T. Sanderson, "Chemical Periodicity," Reinhold Publishing Corp., New York, N. Y., 1960, pp. 192–193.

[7] G. W. Parshall and R. V. Lindsey, Jr., *J. Am. Chem. Soc.*, **81**, 6273 (1959).

hydride–ethyl ether solutions and the volatile ether was removed *in vacuo* to obtain diglyme solutions.

Diglyme, tetrahydrofuran, and ethyl ether were purified by distilling from lithium aluminum hydride. Ethyl iodide and amine solvents were dried over calcium hydride. Ammonia was treated with sodium before use.

Phosphine was generated by heating phosphorous acid.[8] This procedure gives excellent yields if water is removed constantly during the preparation. The phosphine was purified by repeated fractionation through traps held at −80°. Arsine was prepared by a reaction between arsenic trichloride and lithium aluminum hydride.

General Procedures.—Most of the experiments were performed using the conventional high vacuum line techniques. Gaseous products were identified by molecular weights and characteristic vapor pressures. Hydrogen was collected with a Töpler pump.

Analyses.—All hydride solutions were standardized by reaction with water and measurement of the volume of hydrogen evolved.

Lithium was determined by flame photometry, aluminum by the standard 8-hydroxyquinoline procedure, and nitrogen by the Kjeldahl method. Phosphorus was determined as phosphine since in all analyses it was completely converted to this form on hydrolysis of the phosphorus compounds. Completeness of the removal of phosphorus as phosphine was checked by spectrophotometric analyses of the hydrolyzed solution.[9]

Preparation of Sodium Tetraamidoaluminate.—A large excess of ammonia, about 125 mmoles, was condensed with liquid nitrogen onto 6.61 mmoles of sodium aluminum hydride. The reagents were warmed and frozen several times while hydrogen was removed with a Töpler pump. After 20 min., 23.8 mmoles of hydrogen was collected. A chlorobenzene bath (−46°) was placed around the reaction vessel and the mixture was held at this temperature for 11 hr. with occasional stirring. The ammonia then was removed with final pumping for 1 hr. at room temperature to leave a white solid. The total amount of hydrogen evolved was 25.5 mmoles, indicating a 96% reaction according to eq. 3. The weight of product was 0.758 g. (calcd. 0.761). On hydrolysis the product gave 0.25 mmole of hydrogen. Ammonia was evolved but was readsorbed into the solution, which was acidified to retain nitrogen for a Kjeldahl determination.

Anal. Calcd. for $NaAl(NH_2)_4$: Al, 23.7; N, 49.1. Found: Al, 23.3; N, 46.4.

Quantities up to 7 g. were prepared by this procedure. Lithium aluminum hydride reacted in the same way as the sodium compound with liquid ammonia.

Reaction of Sodium Aluminum Hydride with Ammonia in Diglyme.—The stoichiometry of this reaction was established in diglyme and tetrahydrofuran. In a typical experiment 11.4 mmoles of ammonia was condensed onto a solution of 5.30 mmoles of sodium aluminum hydride in 26.2 g. of diglyme. The mixture was warmed to room temperature and a white precipitate formed on stirring. After 30 min. 14.6 mmoles of hydrogen was evolved. More ammonia, 42.2 mmoles, was added in several batches and hydrogen was removed periodically over a total of 6 hr. reaction time. The partial pressure of ammonia was maintained at 400 to 700 mm. The total hydrogen evolved during the reaction was 20.8 mmoles; 32.9 mmoles of ammonia was recovered, indicating that 20.7 mmoles had reacted.

Reaction of Lithium Aluminum Hydride with Ammonia in Ether Solvents.—Three experiments illustrate the effect of reagent ratios.

A large excess of ammonia, roughly 100 mmoles, was condensed onto 12.5 mmoles of lithium aluminum hydride dissolved in 12.8 g. of ethyl ether. At temperatures between −80 and −46° a rapid reaction took place with evolution of hydrogen and formation of a white solid. Most of the reaction occurred in a few minutes but it was allowed to proceed for 4 hr. at −46° and the rate of evolution gradually tapered off; 49.8 mmoles of hydrogen was collected, conforming to eq. 3. Ether and ammonia were removed *in vacuo* at room temperature and a white solid weighing 1.25 g. (calcd. 1.22) was obtained. After hydrolysis, analyses for Al and N gave a 1:3.9 molar ratio although the percentages indicated some impurities in the product. A small amount of hydrogen, 0.2 mmole, was given off in the hydrolysis.

Anal. Calcd. for $LiAl(NH_2)_4$: Al, 27.6; N, 57.3. Found: Al, 26.2; N, 52.8.

A similar experiment at room temperature showed the same stoichiometry.

A second experiment was carried out in 24 g. of diglyme with 13.2 mmoles of lithium aluminum hydride and 23.2 mmoles of ammonia. In 2 hr. the reaction had almost ceased, the ammonia was used up, a white precipitate formed, and 39.1 mmoles of hydrogen was evolved. The ratio of hydrogen evolved to ammonia reacted was 1.7. A large excess of ammonia was added and hydrogen evolution immediately began again slowly. In 2 hr. 8.7 mmoles of hydrogen was collected. At this point all ammonia was removed with pumping. Fresh lithium aluminum hydride, 13.5 mmoles, was added *in vacuo* and a rapid evolution of hydrogen began. In less than 30 min. 16.3 mmoles of hydrogen was collected.

In a third experiment, 11.3 mmoles of ammonia reacted with 21.9 mmoles of lithium aluminum hydride in 43 g. of diglyme. The reaction was extremely rapid at room temperature with evolution of hydrogen. This continued even after all the gaseous ammonia had apparently reacted. In 4 hr. 33.4 mmoles of hydrogen was evolved and the reaction had almost ceased. The amount of hydrogen evolved was three times the amount of ammonia which had reacted.

Preparation of Lithium Tetrakis-(dihydrogenphosphido)-aluminate (LAP).—The preparation was carried out on a small scale to determine the stoichiometry of the reaction and on a large scale to get enough material for analysis and study.

Phosphine, 30.7 mmoles, was condensed *in vacuo* onto a solution of 3.89 mmoles of lithium aluminum hydride in 12.3 g. of diglyme. The reactants were warmed to room temperature and were stirred for 19 hr. Over this period hydrogen evolution was slow, phosphine was taken up, and the solution turned yellow. Several times during this period the reactants were frozen out in a −196° bath for short periods of time while hydrogen was removed. At the end of the run 16.7 mmoles of phosphine was recovered, indicating that 14.0 mmoles had reacted (90%). Hydrolysis of the diglyme solution yielded 13.1 mmoles of phosphine (93% recovery) plus some hydrogen. A similar experiment performed for a shorter period of time established that the amount of hydrogen evolved during the reaction was in a 1:1 ratio to the amount of phosphine which had reacted.

On a larger scale 270 cc. of a 1.5% solution of lithium aluminum hydride in diglyme was placed in a liter flask. Phosphine was introduced *in vacuo* to about 600 mm. pressure and the mixture was stirred. At intervals the phosphine was trapped with liquid nitrogen, the non-condensable material was removed, and more phosphine was added. The reaction was allowed to proceed for 152 hr. Portions of this solution were used for analyses and the remainder in other studies.

An 11.717-g. sample of the large scale preparation was hydrolyzed to yield 13.3 mmoles of phosphine and 0.2 mmole of hydrogen. Analysis of the hydrolyzed solution gave 3.61 mmoles of lithium and 3.64 mmoles of aluminum. Phosphorus in the hydrolyzed solution was negligible. The empirical formula indicated by these results was $Li_{1.00}Al_{1.01}(PH_2)_{3.70}$.

Attempts to Isolate Pure LAP.—Evaporation of diglyme solutions at room temperature resulted in decomposition with evolution of phosphine. Addition of ethyl ether or benzene to a diglyme solution did not precipitate a pure product.

A reaction between lithium aluminum hydride and phosphine carried out in tetrahydrofuran gave the same ratio of hydrogen evolved to phosphine reacting (1:1) as was true in diglyme solu-

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(8) D. R. Martin and R. E. Dial, *J. Am. Chem. Soc.*, **72**, 852 (1950).

(9) W. R. Simmons and J. H. Robertson, *Anal. Chem.*, **22**, 1177 (1950).

tions. Attempts to remove this more volatile solvent gave finely divided yellow crystals which gradually turned orange in spots. Phosphine evolved steadily during the evaporation. The orange material was not soluble in diglyme.

No reaction was observed between liquid phosphine and lithium aluminum hydride at the boiling point of phosphine.

Reaction of Lithium Aluminum Hydride with Phosphine in Ethyl Ether.—Phosphine, 16.3 mmoles, was added to 4.68 mmoles of lithium aluminum hydride dissolved in 12.0 g. of ethyl ether. The reaction was carried out with stirring at 0° for 20 hr. and was continued for another 100 hr. at room temperature. A white precipitate slowly formed during the reaction and hydrogen was evolved steadily. At the end of the reaction period 11.6 mmoles of phosphine was recovered and thus 4.70 mmoles had reacted. During the reaction 12.4 mmoles of hydrogen was collected using a Töpler system. The ratio of hydrogen evolved to phosphine reacted was 2.6. Analysis of the white insoluble product in this and similar runs did not give consistent, simple, empirical formulas.

Reaction of Lithium Aluminum Hydride with Arsine.—Arsine, 16.1 mmoles, was condensed onto 11.8 mmoles of lithium aluminum hydride dissolved in 24.3 g. of diglyme. On warming to room temperature the solution turned yellow-green and gas bubbles formed. After 2 hr. 6.87 mmoles of hydrogen was collected. The 9.37 mmoles of recovered unreacted arsine indicated that 6.68 mmoles had reacted. This ratio of hydrogen evolved to arsine used up is 0.97. No attempt was made to push the reaction to completion. Hydrolysis of the reaction mixture gave 6.60 mmoles of arsine (99% recovery).

Reaction of Aluminum Hydride with Ammonia.—Ammonia, 14.9 mmoles, was condensed onto a solution of 13.1 mmoles of aluminum hydride in 20.1 g. of diglyme. On warming and stirring a white precipitate formed and hydrogen was evolved. After 2 hr. at room temperature, the rate of gas evolution had become very slow. The hydrogen collected in this period was 23.6 mmoles. Unreacted ammonia amounted to 1.5 mmoles. Hydrolysis of the reaction mixture gave 14.3 mmoles of hydrogen.

As with lithium aluminum hydride, the stoichiometry of this reaction was different in other experiments where the reagent ratio was changed.

Very little or no reaction occurred in a few hours time when phosphine was put in contact with aluminum hydride either in diglyme or in diethyl ether.

Stability of the Amides.—There was no sharp decomposition point but the lithium and sodium amidoaluminates evolved ammonia above 50°. A sample of the lithium salt lost 29% of the available nitrogen as ammonia in 150 hr. at temperatures between 170° and 210°.

Solubilities.—The only solvent found for the amides was ammonia, as was observed by Bergstrom. These compounds were insoluble in ethyl ether, tetrahydrofuran, dioxane, diglyme, and triethylamine. Lithium tetraamidoaluminate appeared to dissolve slightly in *n*-butylamine and isopropylamine but the pure product could not be recovered on evaporation. An exchange reaction of amine for ammonia probably occurred. Reactions also took place with the evolution of ammonia with possible solvents containing carbonyl groups.

The phosphorus and arsenic tetrakis compounds were soluble in the ethers in which they may be prepared: diglyme and tetrahydrofuran.

Reaction of LAP with Ethyl Iodide.—Ethyl iodide, 13.1 mmoles, was condensed onto a solution of 7.1 mmoles of LAP dissolved in 27 g. of diglyme. As the solution was stirred for 12 hr. at room temperature, a solid formed, the liquid became yellow, and phosphine, 12.4 mmoles, and ethylphosphine, 9.1 mmoles, were evolved. The reaction mixture was hydrolyzed to yield 4.1 mmoles of phosphine and 3.6 mmoles of ethylphosphine. The total ethyl group accounted for as ethylphosphine was 12.7 mmoles (97%).

Two variations of this reaction were tried. In one experiment the gaseous products were constantly removed. The results were the same as above. In another experiment a diglyme solution of 3.43 mmoles of LAP was dropped slowly onto 24.6 mmoles of ethyl iodide dissolved in diglyme; 3.0 mmoles of phosphine and 0.8 mmole of ethylphosphine were evolved in 30 min.

Reaction of LAP with Ethylphosphine.—Ethylphosphine, 8.7 mmoles, was condensed onto a solution of 3.51 mmoles of LAP in 13.5 g. of diglyme. At the end of 7 hr. at room temperature, 1.0 mmole of phosphine was collected.

Reaction of LAP with Trimethylsilylchloride.—Trimethylsilylchloride, 21.9 mmoles, was condensed onto 11.4 mmoles of LAP dissolved in 38.5 g. of diglyme. The mixture was warmed to room temperature. Volatile materials were removed *in vacuo* after 9 hr. and again after 20 hr. Phosphine, 13.8 mmoles, was isolated plus a small amount of a mixture of less volatile materials that were not readily fractionated on the vacuum line. Molecular weights and vapor pressures indicated that the mixture contained mono-(trimethylsilyl)-phosphine and bis-(trimethylsilyl)-phosphine.

Reaction of LAP with Silicon Tetrachloride.—Silicon tetrachloride, 12.9 mmoles, was condensed onto a solution containing 19.9 mmoles of LAP in 62 g. of diglyme. On warming to room temperature a bright yellow color developed. After 3 hr. 26.8 mmoles of phosphine was collected and after 18 hr. 3.4 mmoles more of phosphine was isolated but no other volatile materials. The liquid had separated into light yellow solution and a viscous, orange, more dense phase. No simple materials were isolated from either phase. They both contained aluminum, silicon, and phosphorus.

Reaction of Lithium Tetraamidoaluminate with Ethyl Iodide.—Ethyl iodide, 37.7 mmoles, was added to a slurry of 8.0 mmoles of lithium tetraamidoaluminate in 10 cc. of diglyme. No ethylamine was formed in 2 days at room temperature. A similar reaction was run without diglyme and with equally negative results.

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