This invention relates to a process for the production of complex alkali aluminum alkyls or alkali aluminum alkyl hydrides.

It is known that aluminum alkyls form complex compounds with various substances. For example, such complexes are formed from aluminum alkyls and ethers, thioethers or tertiary amines, but also from the organic aluminum compounds and alkali halides, more especially alkali fluoroalkides, alkali cyanides, alkali hydrides and alkali alkyls. All these complexes can be produced by simple combination and perhaps also by joint heating of the complex formers and aluminum alkyls. These complex aluminum alkyl compounds have acquired considerable industrial interest, for example as electrolyte liquids in electrolysis processes, for example in the recovery of sodium by electrolysis or for the electrolytic production of metal alkyl compounds of the second to fifth groups of the Periodic System and in particular for the electrolytic production of lead tetraethyl compounds as described in the co-pending applications No. 548,862, filed November 25, 1955, and issued as U.S. Patent No. 2,985,568 on May 23, 1961, and 740,623, filed June 9, 1958, and issued as U.S. Patent No. 3,069,534 on December 18, 1962; as well as in the following applications which are now abandoned:

Serial No. 792,467, filed Feb. 11, 1959

Serial No. 792,614, filed Feb. 11, 1959

Serial No. 27,218, filed May 5, 1960

Serial No. 27,219, filed May 5, 1960

Serial No. 27,220, filed May 5, 1960

Among these compounds, it is more especially the complex compounds of aluminum trialkyl with alkali hydrides and alkali alkyls, i.e. the alkali aluminum trialkyl hydrides, for example Na[Al(C₂H₅)₃]H and alkali aluminum trialkyls, for example Na[Al(C₂H₅)₃] which have achieved particular importance as electrolytes, whether for the precipitation of metallic sodium, or for the electrolytic production of metal alkyls, for example for the production of lead tetraethyl. These two last mentioned groups of complex compounds are hereinafter designated as alkali aluminum alkyls.

In the production of such metal alkyls by electrolysis, more especially for obtaining lead tetraethyl, mixtures of metal alkyls and free aluminum trialkyl are formed as product of electrolysis product. These mixtures must firstly be separated into aluminum trialkyl and the desired metal alkyl and secondly it is essential in order that such an electrolysis process may be conducted economically, for the free aluminum alkyl to be converted to the complex compound used as electrolyte. In the simplest form, this conversion of the aluminum trialkyl is successfully achieved by direct reaction with sodium hydride or sodium alkyl. In practice, however, a series of difficulties arise. Thus, if the mixture to be reacted contains metal alkyl compounds sensitive to hydride, for example lead tetraethyl, the sodium hydride cannot be used for the complex formation, because otherwise this metal alkyl compound, that is to say, for example the lead tetraethyl, would decompose. A reaction with alkali alkyl is possible and can also be carried out practically. The exploitation of this possibility on a large industrial scale is however precluded by the fact that the alkali alkyls can only be prepared with difficulty, so that the provision of adequate quantities of alkali alkyls is only possible with practical difficulties, at least at the present time. The working up of the primary electrolysis products is therefore effected by way of other complex compounds of aluminum triethyl, but these compounds are not the aforementioned most important complex electrolytes.

Thus, for example, with the electrolytic production of lead tetraethyl, using an electrolyte of sodium-aluminum tetraethyl at the lead anode, a mixture of 1 mol of lead tetraethyl and 4 mols of aluminum triethyl is obtained. The lead tetraethyl can be separated out from this mixture in pure form by converting the aluminum triethyl into its complex compounds with sodium fluoride and/or sodium cyanide, as described in the patent application 40,134, filed July 1, 1960, and now abandoned ("Process for Separating Aluminum Triethyl From Other Metal Ethyl Compounds"). The lead tetraethyl is sparingly soluble in these compounds and can be separated out in liquid form.

The aluminum triethyl is thus a decomposition product of the electrolyte used during the electrolysis. Consequently, if it is desired to operate economically, it must be supplied to the electrolyte again in the original form as sodium-aluminum tetraethyl. This can only be effected by the alkali fluoride or alkali cyanide complexes being again transformed into sodium aluminum tetraethyl.

Hitherto, there has been no other possibility than the thermal cracking of the corresponding complexes in vacuo, as described in respect of the alkali fluoride complex of aluminum alkyl, for example in Patent 2,844,615. With such a thermal cracking process, free aluminum trialkyl distills off and an alkali fluoride or alkali cyanide is left. In the case of the alkali fluoride complexes, this thermal cracking is only successful up to the so-called 1:1 complex compounds, for example of the formula NaF-Al₆R₁⁶. Thereafter, the free aluminum trialkyl which is distilled off could be reconverted with sodium hydride and the corresponding olefine or the alkali alkylene into the complex alkali aluminum alkyls.

The disadvantage of this process is that the thermal cracking is comparatively difficult and unpleasant. It is difficult to avoid overheating and particular care and attention is necessary if it is desired to obviate losses due to decomposition. Furthermore, as already indicated, the cracking only proceeds smoothly with the fluorides up to the 1:1 complex compounds. It follows from this that it is not possible simply to add alkali fluoride for separating out for example the lead tetraethyl, if it is desired to recover all the aluminum triethyl, but the 1:1 complex compound itself must be used for this purpose.

Another working up method for the said mixture of for example aluminum triethyl and lead tetraethyl consists in treating the mixture with a suitable tertiary amine, more especially tributylamine, as described in U.S. Patent 3,119,854, issued January 28, 1964 (Method of Separating Aluminum Triethyl From Other Metal Ethyl Compounds and to a Novel Aluminum Complex Compound).

The tributylamine compound of aluminum triethyl is solid at room temperature and is converted into the liquid by the cooling of lead tetraethyl; the necessity then exists of reverting this tributylamine compound of aluminum triethyl into the original electrolyte (sodium-aluminum tetraethyl). Problems of similar nature frequently arise in the course of modern electrolytic processes for the production of metal alkyls.

The complex compounds of the aluminum trialkyls
with alkali fluorides and alkali cyanides and with tertiary amines are very stable compounds. Those with tertiary amines are frequently even more stable than the pure alkali halide or cyanide complex. For example, the sodium fluoride complexes of aluminum triethyl are completely decomposed by added triethylamine, i.e. they are converted into a mixture of sodium fluoride and aluminum triethyl-triethylamine. Conversely, the ethers of the aluminum trialkyls, for example aluminum triethyl-diethyl etherate, can be split for example by potassium fluoride into potassium fluoride complexes of aluminum triethyl and free ether. It is not possible in practice to formulate any laws which regulate the stability of the various aluminum alkyl complexes. It was not possible to foresee how perhaps the sequence of the complex stability would be when comparing a series of substances which are capable of forming complexes.

The present invention is based on the unexpected and surprising fact that alkali hydrides in combination with the aluminum trialkyls, are stronger complex formers even than tertiary amines and the alkali fluorides and cyanides. It has surprisingly also been found that these very stable complexes can be split by alkali hydrides, the corresponding alkali aluminum alkyls then being formed.

Accordingly the present invention provides a process for the production of alkali aluminum alkyls or alkali aluminum alkyl hydrides, wherein other aluminum trialkyl complex or addition compounds are reacted with alkali hydrides if desired in the presence of olefins. Particularly suitable starting materials for this reaction are the alkali halide complexes, more especially the alkali fluoride complexes of the aluminum trialkyls, the alkali cyano complexes and the trialkylamine addition compounds of the aluminum trialkyls or mixtures containing these compounds.

In accordance with the invention the aluminum complex can be split with alkali hydrides, if necessary in the presence of olefins. In this embodiment of the invention it is the alkali aluminum alkyl complexes which are formed and not the aluminum alkyl hydride complex. It is preferable to use an olefin which corresponds to the alkyl residue. It is also preferred to use sodium hydride.

The complex formers originally combined in complex form with the aluminum alkyls and liberated by the alkali hydrides or alkali alkyls can be separated very easily from the reaction products according to the invention. Thus the liberated alkali fluorides and alkali cyanides crystallize out directly and can easily be separated from the molten or previously dissolved alkali aluminum alkyls in the usual manner, for example, by allowing them to settle and then decanting, by filtration or by centrifuging. If tertiary amines in the reaction mixture are liberated when carrying out the process according to the invention these amines can be easily removed by distillation, preferably in vacuo.

With a number of aluminum trialkyls, and more especially with aluminum triethyl, sodium hydride not only forms a 1:1 complex, but also a 1:2 complex with the composition NaH×2Al(C₂H₅)₃. This complex has been proved to be more stable than even the 1:1 complex of aluminum triethyl with sodium cyanide. Consequently, if 1 mol of sodium hydride is added to the compound NaN₃×2Al(C₂H₅)₃, sodium cyanide immediately precipitates. Sodium cyanide can also be precipitated from the 2:1 complex of aluminum triethyl and sodium cyanide by adding the compound Na₂Al(C₂H₅)₃H. In this second case, the complex compound of the sodium hydride with the aluminum triethyl operates approximately in the manner of a dissolved sodium hydride.

Even although the disadvantages of the thermal splitting of the complexes of aluminum trialkyl for recovering the free aluminum trialkyl or the alkali aluminum trialkyl are largely obviated by the previously described embodiment of the invention, it is still also possible for certain difficulties to arise in some cases using the above described embodiment of the invention. The direct treatment of the complex aluminum compounds with solid hydride is subjected to the substantial disadvantage that all reactions in which, under the action of a solid substance on a liquid, the first solid substance disappears and a second solid substance is formed. Under such circumstances, incrustation of the first solid substance by the second substance being separated out can readily occur, and this means that one difficulty in the present part of the reaction proceeds fairly quickly, a relatively long time may be necessary until a complete conversion has taken place.

In another embodiment of the invention, it is now possible also completely to eliminate these difficulties. In this embodiment use is made of the differing complex strengths of the different aluminum trialkyl complexes, but the initial complex compound is not directly transformed into the alkali aluminum tetraalkyl complexes, but the reaction is carried out in stages, i.e. an intermediate reaction is interposed. This intermediate reaction consists in initially adding to the alkali aluminum alkyl halide or other complex aluminum compounds an ether or a tertiary amine. Corresponding to the actual complex stabilities, an aluminum trialkyl etherate or an aluminum trialkyl trialkyl amine is split off from the less stable initial complex aluminum compounds. Simultaneously, either the liberated inorganic complex former is directly obtained or the so-called 1:1 complex compounds are obtained as second reaction compound. For example, the combination NaF×2Al(C₂H₅)₃ with triethylamine gives sodium fluoride and aluminum triethyl-triethylamine. From the corresponding combination of the compound KF×2Al(C₂H₅)₃ only 1 mol of alkali triethyl is split off as triethylamine, and the 1:1 complex compound KF×Al(C₂H₅)₃ is obtained. Ethers, such as diethyl ether, disopropyl ether, di-sec-butylerther or di-n-butylether, generally provide the 1:1 compounds as well as 1 molecule of the corresponding ethers from the complex fluorides of aluminum trialkyl. A condition for carrying out this form of the process is obviously that the complex initial aluminum compounds can be split by adding ether or tertiary amine. As already mentioned, predictions can scarcely be made here for the special case, but it is nevertheless possible from case to case to establish easily, by a small initial experiment, whether this form of the invention can be used. If such conversion takes place, it is then very easy to separate these ethers or trialkyl amines of the aluminum trialkyl from the cleavage products, and thereafter to convert them with alkali hydride or alkali hydride and olefine into the alkali aluminum alkyls while splitting off amine or ethers.

Thus, considered as a whole, even if this form of the process requires an additional reaction stage, it can still provide considerable advantages. The aluminum trialkyl ethers or aluminum trialkyl trialkylamines are not solvents for alkali fluorides or cyanides. Even if the cleavage extends to below these salts, it is only necessary to pour off the liquid aluminum compounds from the solid phase or perhaps remove the said compounds by way of a filter candle. If the cleavage only extends to the 1:1 compounds, the ethers or trialkylamines can be separated with extreme ease by distillation, preferably a continuous vacuum distillation from the complex 1:1 compounds. It is true that distillation is necessary, as with the thermal splitting, but it is no longer a question of a splitting or cleavage of a complex compound, i.e. of the separation of free aluminum trialkyl from a dissociation equilibrium, which may be of the nature NaF×2Al(C₂H₅)₃→NaF×Al(C₂H₅)₃+Al(C₂H₅)₃ but simply of distilling off the aluminum trialkyl etherate or trialkyl amine, which has already been completely formed, from its mixture with the 1:1 complex.

The second stage in this form of the process according to the invention, i.e. the final regeneration to the complex
electrolytes, proceeds extremely quickly and easily, since it is now only necessary for the sodium hydride to dissolve during the reaction and no solid substance is separated out during this reaction.

If the alkali halide complex compounds and the alkali hydride used for the splitting or cleavage contain different alkali metals the cleavage generally takes place in such a way that the alkali metal with the highest atomic number remains in the alkali aluminum alkyl and the halide of the alkali metal with the lower atomic number is separated out.

As starting material for the process according to the invention, it is preferred to employ aluminum trialkyl complex compounds which originate from the working up of the electrolysis products from the electrolys Evelyn production of metal alkyls, and it is in fact preferred to employ aluminum trialkyl complex compounds which are free or substantially free from the metal alkyl compounds to be produced. The invention is further illustrated by the following examples:

Example 1

299 g. (1 mol) of aluminum triethyl tributylamine are added to 8 g. (1 mol) of lithium hydride in a dry 500 cc. flask filled with nitrogen and the reaction mixture is heated to a temperature of from 100 to 200° C. Over a period of 1 hour, all the lithium hydride has been dissolved. The reaction mixture is heated under a vacuum of 1 mm. Hg to 100° C. (measured in the reaction mixture) and tri-n-butyramine distills off in a quantity of 180 g. (=0.97 mol). The distillation residue is 122 g. of pure lithium-aluminum triethylhydride (=100% of the theoretical).

Example 2

150 cc. of a suspension of NaH in paraffin oil is added at 90° C. and while stirring to 215 g. (=1 mol) of aluminum triethyl-triethylamine in a 500 cc. flask filled with nitrogen. The content of sodium hydride in the suspension was fixed at 15.3 g. of NaH per 100 cc. of suspension. All the NaH is dissolved in a short time.

The reaction mixture is transferred under nitrogen into an autoclave with a reaction chamber having a capacity of 1 litre and heated to 160° C. Ethylene is introduced at a pressure of substantially 10 atm. The pressure in the autoclave is expediently kept constant by connecting the reaction vessel by way of a pressure-tight metal capillary tube to a larger storage vessel in which there is an ethylene pressure of 10 atm. Through mixing of the reaction components is assured by shaking the autoclave. Experience shows that the absorption of ethylene ceases after about 5 hours. In order to check this, the supply of ethylene is stopped and it is observed whether there is any further decrease in pressure in the sealed reaction vessel. The excess ethylene is blown off while hot and the liquid contents of the autoclave are emptied out under nitrogen or ethylene pressure while hot.

The reaction mixture is heated under a vacuum of 10 mm. Hg to 120° C., a total of 100 g. (=1 mol) of triethylamine being distilled off. The reaction mixture consists of two immiscible liquid phases, of which the lower phase solidifies on cooling (M.P. about 120° C.). It is practically pure sodium aluminum tetraethyl. The top phase is paraffin oil containing only a little sodium aluminum tetraethyl and can be immediately employed again for the preparation of a sodium hydride suspension.

Example 3

0.95 mol of sodium hydride suspension in Decalin (400 g. NaH/kg. suspension) are added to 270 g. (=1 mol) of the sodium fluoride-aluminum ethyl 1:2-compound at a temperature of from 80 to 100° C. After 30 minutes, the sodium hydride has dissolved. The Decalin is removed at 2 to 3 mm. Hg (bath temperature up to 120° C.), and the mixture of sodium-aluminum triethyl hydride and sodium fluoride-aluminum triethyl is obtained.

Example 4

1 mol of sodium hydride (solid or in Decalin suspension) is initially added to 1 mol of sodium fluoride-aluminum triethyl 1:2, as described in Example 3, then another 0.95 mol of sodium hydride is added, the mixture is placed in a shaker-type autoclave and treated at 180° C. for more than 20 hours with an ethylene pressure of 15 atm. After emptying out the contents of the autoclave, these mixtures can be separated at 150° C. in 2 hours by settling. The sodium-aluminum tetraethyl can be cleanly separated from the sodium fluoride which quickly settles on the bottom. After separation, the Decalin can be first of all extracted as a top layer above the sodium-aluminum tetraethyl and can be used again for preparing a fresh sodium hydride suspension.

Example 5

2 mols of sodium hydride are added to 1 mol of sodium cyanide-aluminum triethyl 1:2 at 120° C. while stirring. After 5 hours at 170° C., the sodium hydride has dissolved and 1 mol of sodium cyanide has separated out. The sodium cyanide is removed from the sodium-aluminum triethyl hydride by centrifuging and is washed with benzene to eliminate the residuals of organometallic substances.

Example 6

1.95 mols of sodium hydride are added to 1 mol of sodium hydride-aluminum triethyl 1:2. The mixture is placed under nitrogen pressure in a 500 cc. autoclave. In the stirrer-type autoclave, the mixture reacts at 170° C. and an ethylene pressure of 15 atm. in 4 hours to sodium-aluminum tetraethyl. The contents of the autoclave can be separated at 150° C. and the pure sodium-aluminum tetraethyl is extracted.

Example 7

1 mol of dry potassium hydride is added to 1 mol of potassium-aluminum tripropyl fluoride and heated while stirring to 90° C. After about 2 to 3 hours, potassium hydride is no longer present in the solid phase and instead an equivalent quantity of KF has precipitated. The potassium fluoride precipitate is allowed to settle at 90° C. and the clear supernatant liquid is siphoned off, this liquid being pure potassium-aluminum tripropyl fluoride. The yield of potassium-aluminum tripropyl fluoride is 190 g. (=97.9% of the theoretical).

Example 8

As described in the preceding example, 1 mol of potassium-aluminum tri-n-octyl fluoride is reacted at 90° C. with 1 mol of dry potassium hydride. The potassium hydride dissolves within a few hours and the equivalent quantity of potassium fluoride is precipitated. The precipitate is allowed to settle and the supernatant liquid, which is potassium-aluminum tri-n-octyl hydride, is siphoned off. The yield of this compound is 380 g. (=93% of the theoretical).

Example 9

24 g. (=1 mol) of sodium hydride are added to 257 g. (=1 mol) of aluminum triisobutyl trimethyl amine in a dry reaction vessel filled with nitrogen and gradually heated within approximately 2 hours and while stirring to 50 to 60° C. until all the NaH has dissolved. At the rate at which the NaH is dissolved, trimethylamine is distilled off from the reaction mixture, this trimethylamine being condensed at -20° C. in a receiver cooled to a low temperature and connected to the reaction vessel. After all the trimethylamine has distilled off, the reaction mixture consists of sodium-aluminum trisobutyl-hydride.
The yield of this complex compound is 222 g. (=100% of the theoretical) and the yield of trimethylamine is 55 g. (=93.5% of the theoretical).

Example 10
2 mols of sodium hydride are added at 130° C. to 1 mol of potassium fluoride-aluminum triethyl 12 and stirred for 5 hours. 1 mol of sodium fluoride is precipitated and this is separated out by allowing it to settle at 90° C. The supernatant mixture contains 1 mol of potassium-aluminum triethyl-hydride as well as 1 mol of sodium-aluminum triethyl hydride.

Example 11
202 g. of dry trimethylamine are carefully added while stirring at room temperature to 270 g. of NaF *2Al(C₂H₅)₃
The precipitated sodium fluoride is allowed to settle and it is separated from the formed aluminium triethyl-triethylamine by decanting. The sodium fluoride can be washed with a little benzene and after removing the last traces of benzene in vacuo, it can be obtained in pure form with a yield of 100% of the calculated quantity, and in addition there are obtained 220 g. of aluminum triethyl-triethylamine, i.e. 98% of the theoretical. This is further processed in accordance with Example 2.

Example 12
102 g. of diisopropylether are added at room temperature to 270 g. of NaF *2Al(C₂H₅)₃. The homogeneous liquid mixture is thereafter heated in vacuo at 10° C. Hg to 100 to 130° C. (measured in the liquid) and 220 g. of aluminum triethyl disopropylether distill off. The distillation residue consists of 156 g. of NaAl(C₂H₅)₃F.
The result remains the same if 204 g. of disopropylether are used instead of 102 g. The 102 g. of disopropylether can be distilled off from the reaction mixture before it is placed under vacuum. The further processing of the distillate is in accordance with Example 2. 102 g. of disopropylether distill off during the heating with sodium hydride in paraffin oil.
Instead of disopropylether, it is also possible to use diethyl ether, but in this case the danger of an other loss is greater than with the disopropylether, which has a higher boiling point.
In this example, only half of the aluminum triethyl contained in the 270 g. of NaF *2Al(C₂H₅)₃ is utilized. With the main use of the present process, which is perhaps the separation of lead tetrachloride and aluminum triethyl, this is unimportant. It only means that 4 mols of NaF *Al(C₂H₅)₃ and not two mols of NaF must be added to the mixture of Pb(C₂H₅)₄+4Al(C₂H₅)₃.

Example 13
260 g. of di-n-butyl ether are carefully added at about 50° C. to 277 g. of NaCNC *2Al(C₂H₅)₃. Distillation is carried out at a pressure of 10 to 15 mm. from an oil bath, the temperature of which rises slowly from 120 to 200° C. 470 to 480 g. of aluminum triethyl dibutyl etherate are obtained as distillate and 49 g. of solid sodium cyanide as residue. The distillate is further processed according to Example 2 and dibutylether appears instead of triethylamine. Otherwise, the entire procedure is the same.

Example 14
286 gms. (=1 mol) of the compound KP *2Al(C₂H₅)₃ are mixed at 130° C. with 24 gms. (=1 mol) of sodium hydride (suspended in 200 ml. of paraffinum liquidum) while stirring. After 50 minutes, all of the sodium hydride has dissolved. The reaction mixture is treated for 4 hours at 180° C. with ethylene of 15 atmospheres in a shaking autoclave of 1 liter capacity.

The resultant mixture crystallizes upon cooling below 60° C. and the crystals can be separated from the paraffin oil by decanting the latter. There are obtained 338 gms. of a 1:1 molar mixture of K[Al(C₂H₅)₂F] and Na[Al(C₂H₅)₄].
We claim:
1. Process for the production of alkali-metal-aluminum-alkyl-hydride-complex compounds, which comprises reacting a complex of aluminum-trialkyl with a member selected from the group consisting of alkali-metal-halides, alkali-metal-cyanides, and trialkyl amines, with an alkali-metal-hydride, and recovering the alkali-metal-aluminum-alkyl-hydride-complex compound formed.
2. Process for the production of alkali-metal-aluminum-tetraalkyl-complex compounds, which comprises reacting a complex of aluminum-trialkyl with a member selected from the group consisting of alkali-metal-halides, alkali-metal-cyanides, and trialkyl amines, with an alkali metal hydride and recovering the resulting product with an olefin, and recovering the alkali-metal-aluminum-tetraalkyl compound formed.
3. Process for the production of alkali-metal-tetraalkyl-complex compounds, which comprises the steps of (1) reacting a complex of aluminum-trialkyl with a member selected from the group consisting of alkali-metal-halides, alkali-metal-cyanides, and trialkyl amines, with an alkali metal hydride to thereby obtain an alkali-metal-alkyl-aluminum-hydride, thereafter (2) reacting the latter compound with olefin, and recovering the alkali-metal-alkyl-aluminum-tetraalkyl compound formed.
4. Process according to claim 2, wherein said complex alkali metal hydride and said olefin are contacted in a reaction zone and said reactions occur simultaneously.
5. Process according to claim 1, in which the starting aluminum-trialkyl-complex compound is in the form of a complex with an alkali-metal-halide.
6. Process according to claim 5, in which the starting aluminum-trialkyl-complex compound is in the form of a complex with an alkali-metal-cyanide.
7. Process according to claim 1, in which the starting aluminum-trialkyl-complex compound is in the form of a complex with an alkali-metal-fluoride.
8. Process according to claim 1, in which the starting aluminum-trialkyl-complex compound is in the form of a complex with a trialkylamine.
9. Process according to claim 1 which comprises effecting said reaction with stoichiometric amounts of said aluminum-trialkyl-complex and alkali-metal-hydride reactants.
10. Process for the production of alkali-metal-aluminum-alkyl-hydride-complex compounds, which comprises reacting a complex of aluminum-trialkyl with a member selected from the group consisting of alkali-metal-halides and alkali-metal-cyanides with a member selected from the group consisting of ethers and tertiary amines, to form the corresponding trialkyl-aluminum-complex with the second-mentioned member, and thereafter reacting the latter with an alkali-metal-hydride, and recovering the alkali-metal-aluminum-alkyl-hydride compound formed.
11. Process according to claim 10, wherein a tertiary amine is reacted with the first-mentioned complex to form an aluminum trialkyl aminate and liberate a compound selected from the group consisting of alkali metal halides and alkali metal cyanides, said tertiary amine is separated from the liberated compound and is thereafter reacted with said alkali metal hydride.
12. Process for the production of alkali-metal-aluminum-tetraalkyl-complex compounds, which comprises reacting a complex of aluminum-trialkyl with a member selected from the group consisting of alkali-metal-halides and alkali-metal-cyanides with a member selected from the group consisting of ethers and tertiary amines, to form the corresponding trialkyl-aluminum-complex with the second-mentioned member, and thereafter reacting
the latter with an alkali-metal-hydride and an olefin, and recovering the alkali-metal-aluminum-tetraalkyl compound formed.

13. In the process for separating mixtures of aluminum-trialkyl and other metal-alkyl compounds of the second to fifth group of the Periodic System by treating the mixture with an alkali-metal-halide, whereby there is formed a complex of the aluminum-trialkyl with said alkali-metal-halide, and separating the complex thus formed from the metal-alkyl, the improvement which comprises reacting the aluminum-trialkyl-alkali-halide-complex with an alkali-metal-hydride and an olefin, to form an alkali-metal-aluminum-tetraalkyl complex.

14. Process according to claim 13, wherein the mixture to be separated consists of aluminum-triethyl and another metal-alkyl compound of the second to fifth group of the Periodic System.

15. Process of claim 12 wherein the first-mentioned complex is NaF·2Al(C₂H₅)₃, and said complex is reacted with an ether to form NaF·Al(C₂H₅)₃ and the aluminum triethyl etherate, and the etherate is separated from the NaF·Al(C₂H₅)₃, and the separated etherate is reacted with the alkali metal hydride and an olefin.

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