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METHOD OF PRODUCING ALUMINUM BOROHYDRIDE

William H. Schechter, Zelienople, Pa., assignor, by mesne assignments, to Callery Chemical Company, Pittsburgh, Pa., a corporation of Pennsylvania

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This invention relates to the production of aluminum borohydride, $Al(BH_4)_3$.

Aluminum borohydride may be produced according to the equation:

$$3MBH_4 + AlX_3 \rightarrow Al(BH_4)_3 + 3MX$$

where M is an alkali metal, such as sodium (Na) or lithium (Li), and X is a halogen, such as chlorine (Cl) or bromine (Br). Heretofore this reaction has been performed by heating a quiescent dry mixture of the solid reactants. Experience has shown that in such practice materials of high purity are necessary, as well as a large excess of the aluminum halide. Furthermore, it has been found that reaction must be effected in thin layers but that even so foaming may take place, which is objectionable at least from an operating standpoint. Such prior practice is therefore undesirably uneconomical and is not adapted to quantity production of this compound.

It is among the objects of this invention to provide a method of making aluminum borohydride that is simple, is easily practiced and controlled, is adapted to quantity production, does not require high purity starting materials, and avoids disadvantages encountered in the prior art practice alluded to above.

Other objects will be recognized from the following specification.

I have discovered, and it is upon this that the invention is largely predicated, that the stated objects are obtained by vigorously agitating finely divided alkali metal borohydride in a closed container in contact with a finely dispersed aluminum halide, while excluding moisture from the system.

In one embodiment of the invention the necessary agitation of the two reactants is attained by the use of a ball mill in which the reaction may be performed in the dry state. To this end, the alkali metal borohydride is placed in the ball mill. It is initially finely subdivided, or that may be accomplished by operation of the ball mill. The aluminum halide may in either case be added with the alkali metal borohydride, or it may be passed as vapor into the finely divided borohydride while the ball mill is operated.

Preferably, and for the best results, however, the finely divided alkali metal borohydride is suspended in an inert liquid for contact with the aluminum halide either in a ball mill or, and most suitably, in an autoclave provided with a high speed stirrer to provide violent agitation.

The temperature at which the reaction occurs is not critical provided it does not exceed the decomposition temperature of aluminum borohydride. The reaction may be effected suitably at from ambient temperature to about 200° C., but for most purposes it is preferred to effect it between 100° and 150° C.

As the inert suspending liquid it is preferred to use liquid paraffin or mineral oils of high boiling point, in which aluminum borohydride is freely soluble but in which the alkali metal halide produced is substantially insoluble, which makes for ease of recovery of the desired

aluminum borohydride. Other aliphatic or aromatic hydrocarbons may be used providing their boiling points are above the temperature at which the reaction is effected. Ethers, even of high boiling point, are unsatisfactory for this purpose because they form complexes with aluminum borohydride.

As indicated above, moisture should be rigorously excluded from the system, and preferably air or other oxygen-containing gases likewise.

The reaction may be effected under super-atmospheric pressure or under vacuum, or at atmospheric pressure. Desirably nitrogen or other atmosphere inert to aluminum borohydride and the reactants is supplied to the system. Where the reaction is conducted above the boiling point of aluminum borohydride, about 44.5° C., it is preferred to maintain a current of inert sweep gas, e.g., N_2, through the system whereby the aluminum borohydride is removed continuously as it is formed, the vapor being then condensed to recover the product in liquid form.

Although any of the alkali metal borohydrides and any of the aluminum halides may be used in the practice of the invention, it is preferred to use sodium borohydride ($NaBH_4$) and aluminum chloride ($AlCl_3$) as affording the most inexpensive source of the aluminum borohydride. The aluminum halide may be added as a finely divided solid to the suspension of alkali metal halide, or a suspension of it may be mixed with a suspension of the alkali metal halide. Similarly, the aluminum chloride may be vaporized and fed continuously into the agitated suspension of the alkali metal halide.

A particular advantage of the method provided by this invention is that, contrary to prior practices, impure alkali metal borohydride may be used satisfactorily. Thus there may be used sodium borohydride containing sodium chloride or sodium metaborate ($NaBO_2$) with which the borohydride is associated in its production by various methods. For example, in application Serial No. 229,141, filed by me on May 31, 1951, there is disclosed the production of alkali metal borohydrides by reaction between an alkali metal hydride with a boron halide or an alkali metal borofluoride, which results in the production of a mixture of alkali metal borohydride and alkali metal halide. That application describes also reaction between alkali metal halide and boric oxide (B_2O_3), with production of a mixture of the alkali metal borohydride and $NaBO_2$. Such $NaBH_4$ and related reaction products, e.g., of alkali metal hydride and alkyl borate, may with economic advantage be used directly in the practice of the present invention, and if the alkali metal borohydride has been prepared in an inert liquid, such as mineral oil, as described in that application, the resultant suspension is ready for direct treatment with aluminum halide in accordance with the present invention.

In the practice of this invention it is advantageous to use a moderate excess of aluminum halide over that required by the equation given above.

According to the provisions of the patent statutes, I have explained the principle and mode of practicing my invention and have described what I now consider to represent its best embodiment. However, I desire to have it understood that, within the scope of the appended claims, the invention may be practiced otherwise than as specifically described.

I claim:

1. That method of producing aluminum borohydride which comprises the steps of violently agitating in an autoclave in the absence of air and moisture and at a temperature from ambient to about 200° C. a suspension of finely divided alkali metal borohydride in an inert liquid that is a solvent for aluminum borohydride and in which alkali metal halide produced during the reaction

is insoluble, and of boiling point substantially above that prevailing in the autoclave, in contact with an aluminum halide, whereby to form aluminum borohydride, and recovering the aluminum borohydride.

2. A method according to claim 1, said alkali metal borohydride being sodium borohydride, said halide being aluminum chloride, and said inert liquid being mineral oil.

3. A method according to claim 1, said liquid being mineral oil.

4. A method according to claim 1, said liquid being mineral oil and said temperature being from about 100° to 150° C.

5. That method of producing aluminum borohydride which comprises the steps of violently agitating in an autoclave in the absence of air and moisture and at a temperature from ambient to about 200° C. a suspension of finely divided alkali metal borohydride in an inert liquid that is a solvent for aluminum borohydride and in which alkali metal halide produced during the reaction is insoluble, and of boiling point substantially above that prevailing in the autoclave, in contact with an aluminum halide, whereby to form aluminum borohydride, maintaining a current of inert sweep gas through the autoclave to remove the aluminum borohydride as it is formed, and condensing and recovering the aluminum borohydride.

6. That method of producing aluminum borohydride which comprises the steps of contacting in a closed container an agitated suspension of finely divided alkali metal hydride in an inert liquid that is a solvent for aluminum borohydride and in which alkali metal halide produced during the reaction is insoluble, and of boiling point substantially above that prevailing in the container, with an agent of the group consisting of boron halides, alkyl borates, alkali metal borofluorides, and boric oxide, to form a suspension of reaction product comprising alkali metal borohydride and other product of the reaction, and then agitating the resultant suspension of said reaction product in the absence of moisture with an aluminum halide and thereby forming aluminum borohydride, and separating and recovering the aluminum borohydride.

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