PATENT SPECIFICATION

NO DRAWINGS

876,038

Date of Application and filing Complete Specification: *October* 13, 1959.
No. 34687/59.
Application made in Germany on October 13, 1958.
Complete Specification Published August 30, 1961.

Index at Acceptance: Class 1(2), E4X.
International Classification: C01b.

A process for the production of hydrazine hydrates.

COMPLETE SPECIFICATION

SPECIFICATION NO. 876,038

The inventor of this invention in the sense of being the actual deviser thereo within the meaning of Section 16 of the Patents Act, 1949, is Günter Henrich of Schoellerstrasse 25, Opladen, Germany, a German citizen.

THE PATENT OFFICE,
1st October, 1961.

DS 96751/1(3)/R.153 200 9/61 P

zine hydrate from dihydrazine sulphate in the presence of water.

The present invention provides a process for the production of hydrazine hydrate from dihydrazine sulphate in the presence of water, which comprises reacting an aqueous solution of dihydrazine sulphate with a hydroxide of an alkali metal or alkaline earth metal, or an alkaline earth metal oxide, the base being present in an amount ranging between 100% and 150% of the amount stoichiometrically equivalent to the sulphate, adding a water-soluble organic liquid in a quantity of 20 to 500% of the total water present at a temperature between 15 and 100°C., separating off the resulting metal sulphate and working up the remaining solution to hydrazine hydrate by distillation.

The solubility of the alkali metal or alkaline earth metal sulphate formed is lowered by adding the water-soluble organic liquid.

By the process of the invention, the dihydrazine sulphate solution is reacted with an alkali metal or alkaline earth metal hydroxide with subsequent addition of a readily volatile organic liquid. By maintaining the specific concentration and temperature conditions, it is surprisingly possible in this way for the alkali metal or alkaline earth metal sulphate, which is this way, the danger of mother liquor containing hydrazine occluded in water of crystallisation of the precipitate is avoided.

Sodium hydroxide and potassium hydroxide, in the form of 40 to 45% by weight aqueous solutions, as well as calcium oxide and hydroxide are especially suitable for reaction with dihydrazine sulphate solution. The base is present in such a quantity as to provide an excess of from 0 to 50% over the stoichiometric quantity required for reaction with the hydrazine sulphate, calculated on the sulphuric acid to be combined.

As organic liquids for the precipitation of the sulphate in question, it is possible to use any water-soluble organic liquid, more especially aliphatic alcohols and advantageously methyl, ethyl and propyl alcohol as well as dioxane. With the process of the present invention, the quantity of organic liquid should be from 20 to 500%, advantageously 50 to 200%, calculated on the quantity of water which is present. The temperature during the reaction with the base and also during treatment with the organic liquid is preferably between 15 and 100°C. It is advisable that the temperature, prior to separating out the metal sulphate, should be reduced to between 20 and 30°C. to lower the solubility of the metal sulphate, even if

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Price 75p

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We, FARBENFABRIKEN BAYER AKTIENGESELLSCHAFT, of Leverkusen-Bayerwerk, Germany, a body Corporate organised under the Laws of Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:

This invention relates to a process for the production of hydrazine hydrate.

According to known processes it is possible to obtain anhydrous hydrazine from dry hydrazine sulphate by reaction with pure ammonia. The present invention is concerned with a process for obtaining hydrazine hydrate from dihydrazine sulphate in the presence of water.

The present invention provides a process for the production of hydrazine hydrate from dihydrazine sulphate in the presence of water, which comprises reacting an aqueous solution of dihydrazine sulphate with a hydroxide of an alkali metal or alkaline earth metal, or an alkaline earth metal oxide, the base being present in an amount ranging between 100% and 150% of the amount stoichiometrically equivalent to the sulphate, adding a water-soluble organic liquid in a quantity of 20 to 500% of the total water present at a temperature between 15 and 100°C., separating off the resulting metal sulphate and working up the remaining solution to hydrazine hydrate by distillation.

The solubility of the alkali metal or alkaline earth metal sulphate formed is lowered by adding the water-soluble organic liquid.

By the process of the invention, the dihydrazine sulphate solution is reacted with an alkali metal or alkaline earth metal hydroxide with subsequent addition of a readily volatile organic liquid. By maintaining the specific concentration and temperature conditions, it is surprisingly possible in this way for the alkali metal or alkaline earth metal sulphate, which is sparingly soluble under these conditions, to be separated out in a substantially quantitative manner in the form of a granular precipitate which is free from water of crystallisation and which can be very easily filtered, it also being possible for the said granular precipitate to be washed easily. In the case of the precipitation of sodium sulphate, it has surprisingly been found that the conversion points of the sodium sulphate to the heptahydrate or decahydrate forms, which are difficult to filter, are lowered from 32° and 24°C., respectively, to below 15°C. when the separation is effected under the conditions according to the present invention. In this way, the danger of mother liquor containing hydrazine occluded in water of crystallisation of the precipitate is avoided.

Sodium hydroxide and potassium hydroxide, in the form of 40 to 45% by weight aqueous solutions, as well as calcium oxide and hydroxide are especially suitable for reaction with dihydrazine sulphate solution. The base is present in such a quantity as to provide an excess of from 0 to 50% over the stoichiometric quantity required for reaction with the hydrazine sulphate, calculated on the sulphuric acid to be combined.

As organic liquids for the precipitation of the sulphate in question, it is possible to use any water-soluble organic liquid, more especially aliphatic alcohols and advantageously methyl, ethyl and propyl alcohol as well as dioxane. With the process of the present invention, the quantity of organic liquid should be from 20 to 500%, advantageously 50 to 200%, calculated on the quantity of water which is present. The temperature during the reaction with the base and also during treatment with the organic liquid is preferably between 15 and 100°C. It is advisable that the temperature, prior to separating out the metal sulphate, should be reduced to between 20 and 30°C. to lower the solubility of the metal sulphate, even if

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higher temperatures are possible in this case.

The separation of the hydrazine hydrate, after the deposition of metal sulphate, from the organic liquid is readily possible by using distillation methods, so that the further working up no longer presents any difficulties.

The invention is further illustrated by the following Example:

EXAMPLE

162 Kg. of 50% aqueous dihydrazine sulphate solution are mixed with 84 kg. of 50% NaOH solution at 18°C. in a stirrer-type vessel. 200 Kg. of methanol are thereafter added to this mixture in another stirrer-type vessel, and the suspension formed is cooled to 20°C. and filtered. By this means, 71 kg. of sodium sulphate are separated out and washed with methanol. The filtrate, consisting of 200 kg. of methanol, 50 kg. of hydrazine hydrate, some sodium hydroxide and 123 kg. of water is almost completely evaporated in an evaporator, the vapour being conducted into an independently heated fractionating column. Methanol is extracted at the top of the column and water from the middle thereof, while pure hydrazine hydrate is recovered from the bottom.

WHAT WE CLAIM IS:

1. A process for the production of hydrazine hydrate from dihydrazine sulphate in the presence of water, which comprises reacting an aqueous solution of dihydrazine sulphate with a hydroxide of an alkali metal or alkaline earth metal or an alkaline earth metal oxide, the base being present in an amount ranging between 100% and 150% of the amount stoichiometrically equivalent to the sulphate, adding a water-soluble organic liquid in a quantity of 20 to 500% of total water present at a temperature between 15 and 100°C., separating off the resulting metal sulphate and working up the remaining solution to hydrazine hydrate by distillation.

2. A process as claimed in claim 1, wherein the water-soluble organic liquid is present in a quantity of from 50 to 200% of the total water present in the reaction mixture.

3. A process as claimed in claims 1 or 2, wherein the base is sodium hydroxide or potassium hydroxide in the form of a 40 to 45% by weight aqueous solution.

4. A process as claimed in claim 1, wherein said water-soluble organic liquid is an aliphatic alcohol or dioxane.

5. A process as claimed in claim 4, wherein the aliphatic alcohol is methyl-, ethyl- or propyl alcohol.

6. A process as claimed in any of claims 1 to 5, wherein the reaction mixture is cooled to 20-30°C. before the metal sulphate is filtered.

7. A process as claimed in claim 1 substantially as herein described with reference to the Example.

8. Hydrazine hydrate when prepared by a process as claimed in any of the preceding claims.

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Agents for the Applicants.

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