

# PATENT SPECIFICATION

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## COMPLETE SPECIFICATION

### Process for Obtaining Amino-Guanidine Bicarbonate

- I. THE DEPUTY MINISTER OF THE RUMANIAN MINISTRY OF PETROLEUM AND CHEMICAL INDUSTRY, of strada Scaune 1, Bucharest, Rumania, do hereby declare the invention, for which I pray that a patent may be granted to me, and the method by which it is to be performed, to be particularly described in and by the following statement:—
- 10 The present invention relates to a process for obtaining amino-guanidine bicarbonate from cyanamide and hydrazine acid sulphate.
- Amino-guanidine bicarbonate may be obtained in several ways. The known process starting from guanidine nitrate is not economic, having a low yield (73-75%) and requiring a great number of stages.
- 20 The other known processes, based upon the free condensation of cyanamide with hydrazine sulphate or hydrazine hydrate, work at a pH of 4-6, at a temperature of 90° or 100°C and with an aqueous solution of free cyanamide having a concentration of 3.5-4.5% by weight.
- Where the process is carried out at a pH of 4 and a reaction temperature of 90°C, the reaction rate is low, the reaction time long (about 24 hours) and the yield 85%.
- 30 Where it is carried out at a pH of 5-6 and a reaction temperature of 100°C, the reaction time diminishes to 6 hours (the reaction rate increases) and the yield is 80-90%. In the case of this known process, however, the disadvantage consists in the fact that at a temperature of 100°C the free cyanamide decomposes gradually with the formation of ammonia and the pH becomes alkaline (8.5). At this pH, decomposition of the amino-guanidine takes place too, which leads to a decrease in the yield to 80%.
- The present invention provides a process for obtaining amino-guanidine bicarbonate by the condensation of a solution of free cyanamide having a concentration of 3.5-4.5% by weight with hydrazine acid sulphate at a pH of 7.2-7.3 which is maintained throughout the reaction time, and by this means a conversion of 94-95% and yield of 95%, based on the hydrazine acid sulphate introduced into the reaction, are obtained. Amino-guanidine bicarbonate is then precipitated from the solution of amino-guanidine sulphate so obtained.
- For maintaining this constant pH in the reaction mass, it has been established as necessary to work in the presence of a buffer substance, preferably boric acid in a quantity of about 3% by weight based on the water and about 30% by weight based on the total quantity of hydrazine acid sulphate used. Further it has been found out that owing to the pH value of 7.2-7.3 employed, the reaction rate is at a maximum and thus the reaction time a minimum, while the decomposition of free cyanamide and amino-guanidine is much reduced, which conduces to a maximum conversion and yield.
- 70 The preferred procedure according to the invention, consists in the treatment of free cyanamide in 3.5-4.5% by weight aqueous solution with hydrazine acid sulphate in a molar ratio of 1.35:1, in the presence of boric acid in a proportion of about 1:3 by weight as compared with the hydrazine acid sulphate, at 100°C, for 30-60 minutes, followed by filtration to separate the inorganic salts and precipitation with sodium bicarbonate of the amino-guanidine bicarbonate obtained.
- One example of a process embodying the invention now follows:
- Example*
- 4,600 cc free cyanamide solution of about 85 4% concentration by weight, containing 189

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g. free 100% cyanamide (4.5 mols) with a pH of 5-6, and 433 g. hydrazine acid sulphate 100% (465.6 g. hydrazine acid sulphate 93% = 3.32 mols) are introduced into a reaction vessel. After adding these reactants, the pH of the aqueous solution is acid to Congo Red, 136 g. boric acid 100% (about 3% by weight against water) and 320 g. solid sodium bicarbonate are gradually added until the pH is 5-6 and the reaction mass takes the form of a solution.

The mass is heated under reflux for about 30 minutes and it is maintained at this temperature for 30-60 minutes until the sample shows a maximum conversion. When the reaction mass attains 100°C, the pH becomes 7.2-7.3, this value being maintained all the time of refluxing, due to the buffering action of the boric acid.

At the end of the reaction, the reaction mass is cooled, filtered to remove inorganic salts and the amino-guanidine sulphate solution thus obtained is treated with about 480 g. of sodium bicarbonate. It is cooled to under 10°C and after 3 hours the amino-guanidine bicarbonate precipitate is filtered off, 640 g. of cake with a content of about 400-408 g. amino-guanidine bicarbonate 100% and about 24-28 g. sodium bicarbonate are obtained. The conversion is 94-95% and the yield 95%.

The following advantages are obtained by the use of the invention:

1. The percentage of free cyanamide and that of degraded amino-guanidine is reduced to a minimum owing to the fact that a pH of 7.2-7.3 is employed, at which the reaction rate is high and the degradation rate is low. This allows a maximum yield of 95% based on the hydrazine acid sulphate undergoing the reaction to be obtained by

applying the invention. In the known processes a yield of 70-90% is obtained.

2. The reaction time is reduced to 30-60 minutes and a maximum conversion of 94-95% is effected. This is possible owing to the high reaction rate determined by the pH of 7.2-7.3 at which it is carried out. In the known processes a reaction time of 50-6-24 hours is required.

#### WHAT I CLAIM IS:—

1. A process for obtaining amino-guanidine bicarbonate which comprises the condensation of free cyanamide in 3.5-4.5% by weight aqueous solution with hydrazine acid sulphate at a pH value of 7.2-7.3 maintained by means of a buffer throughout the reaction time, followed by precipitation of amino-guanidine bicarbonate from the solution of amino-guanidine sulphate obtained.

2. A process according to claim 1 characterized in that the free cyanamide in solution is treated with hydrazine acid sulphate in a molar ratio of 1.35:1, in the presence of boric acid in the proportion of 1:3 by weight based on the hydrazine acid sulphate, at 100°C for 30-60 minutes, followed by filtration to remove the inorganic salts and the precipitation of amino-guanidine bicarbonate by means of sodium bicarbonate from the amino-guanidine sulphate solution obtained.

3. A process for obtaining amino-guanidine bicarbonate substantially as set forth in the example herein.

4. Amino-guanidine bicarbonate when prepared by a process according to any of claims 1 to 3.

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