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(54) **Method for making B-form aluminum trimetaphosphate**

(57) Aluminum trimetaphosphate in the B-form is prepared by reacting aluminum dihydrogen phosphate with ammonia and heating the reaction product so that the crystal structure of the heat-treated material is predominantly in the B-form.

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SPECIFICATION

Method for making B-form aluminum trimetaphosphate

The present invention relates generally to the art of making $Al(PO_3)_3$ and more particularly to the preparation of the B-form of aluminum trimetaphosphate.

In U.S. Patent No. 3,445,257, Hlock et al disclose an improved hardener for water glass cements. The hardener is a condensed aluminum phosphate prepared by subjecting acid aluminum orthophosphates consisting of P_2O_5 and Al_2O_3 in a ratio of 1.1 to 3 to a two stage thermal treatment. The aluminum orthophosphate is prepared by adding alumina to phosphoric acid.

In U.S. Patent No. 3,943,231, Wasel-Nielen et al disclose a process of spray drying an aluminum orthophosphate solution containing P_2O_5 and Al_2O_3 in a ratio greater than 1.5 at a temperature greater than 250°C to effect direct transformation into amorphous condensed aluminum phosphates.

Crystalline condensed aluminum phosphates are described in the literature by D'Yvoire, who identified a cyclic aluminum tetrametaphosphate, the stable A-form of $Al(PO_3)_3$, and four long-chain polyphosphates, the B, C, D and E forms of $Al(PO_3)_3$. According to D'Yvoire, these crystalline condensed aluminum metaphosphates are produced by reacting P_2O_5 and Al_2O_3 in a molar ratio of 4 to 15 over several hours. Mixtures of aluminum metaphosphates, including both the A and B forms, have been found in available hardeners.

An alternate process for making condensed aluminum phosphates involves reacting soluble linear or cyclic condensed alkali metal polyphosphates with soluble aluminum salts in water.

According to the present invention a method is provided for preparing aluminum trimetaphosphate in the B-form comprising the steps of:

a. reacting aluminum dihydrogen phosphate with ammonia; and
b. heating the reaction product to a sufficient temperature for a sufficient time that the crystal structure of the heat-treated material is predominantly the B-form.

One embodiment of the present invention involves the preparation of B-form aluminum trimetaphosphate by the addition of concentrated ammonium hydroxide to a solution of aluminum dihydrogen phosphate, $Al(H_2PO_4)_3$, to effect the formation of a white precipitate which is directly converted to the B-form of aluminum trimetaphosphate, $Al(PO_3)_3$, by a single elevated temperature treatment. The resultant B-form aluminum trimetaphosphate is useful as a hardener in inorganic paint or cement compositions.

The aluminum dihydrogen phosphate solution of the present invention is preferably a concentrated solution, most preferably an aqueous solution comprising about 50 percent solids. The solution is preferably maintained at ambient temperature. To the solution of aluminum dihydrogen phosphate is added concentrated ammonium hydroxide, preferably a solution comprising about 30 percent NH_3, in an amount sufficient to form a white precipitate from the phosphate solution.

The precipitate which results from the combined solutions may comprise both crystalline and amorphous material. The crystalline material may comprise mixtures of ammonium phosphates and ammonium aluminum phosphates. The precipitate is subjected to a single elevated temperature treatment which results in the formation of B-form aluminum trimetaphosphate. The treatment time varies inversely with the treatment temperature. Preferably, the temperature is at least about 500°C but less than about 900°C with treatment times varying from several hours to several minutes. Thus, for example, the precipitate may be heated to a temperature of about 540°C for a period of 1 to 2 hours or to about 650°C for a period of about 30 to 60 minutes or to about 750°C for a period of about 10 to 30 minutes. The precipitate may be dried prior to the heat treatment.

The present invention is illustrated by the specific Examples which follow.

Example I

To 30 grams of a 48 percent solution of aluminum dihydrogen phosphate is added a 60 percent solution of ammonium hydroxide until the pH reaches about 9. The white precipitate formed by the combination of the two solutions is filtered, dried and heated to 540°C for 2 hours. The product is about 90 percent of the B-form aluminum trimetaphosphate as identified by X-ray diffraction analysis.

Example II

The following components are added together in a ball mill.

$Al(H_2PO_4)_3$, 48% solution	240 grams
NH_4OH, 60% solution	122 milliliters
Water	130 milliliters

The mixture is ground, dried and treated at 650°C for 1 hour. The product is identified by X-ray diffraction analysis to be about 80 percent of the B-form aluminum trimetaphosphate.

Example III

Ammonium hydroxide solution is added to aluminum dihydrogen phosphate solution which is

continuously agitated. A damp white precipitate is formed from the combination of the following solutions.

$Al(H_2PO_4)_3$, 50% solution	430 grams
NH_4OH, 60% solution	160 milliliters

The reaction product is treated at 750°C. for 30 minutes resulting in the formation of predominantly the B-form of aluminum trimetaphosphate.

The above examples are offered to illustrate the present invention. Other time-temperature cycles can be used to obtain the B-form of aluminum trimetaphosphate, as well as other concentrations and ratios of reactants. The acid aluminum phosphate may be reacted with ammonia gas rather than a solution. Other metal cations such as chromium, iron and boron may be substituted for aluminum. The scope of the invention is defined by the following claims.

CLAIMS

1. A method for preparing aluminum trimetaphosphate in the B-form comprising the steps of:
   a) reacting aluminum dihydrogen phosphate with ammonia; and
   b) heating the reaction product to a sufficient temperature for a sufficient time that the crystal structure of the heat-treated material is predominantly the B-form.
2. A method according to claim 1, wherein step (a) comprises adding solution of ammonium hydroxide to a solution of aluminum dihydrogen phosphate to form a white precipitate.
3. A method according to claim 2, wherein the solution of ammonium hydroxide contains about 30% NH_3.
4. A method according to claim 2 or 3, wherein the solution of aluminum dihydrogen phosphate contains about 50% solids.
5. A method according to claim 3 or 4, wherein the precipitate is heated in step (b) to a temperature of at least about 500°C but less than about 900°C.
6. A method according to claim 5 wherein the precipitate is heated to a temperature of about 540°C for a period of about 1 to 2 hours.
7. A method according to claim 5 wherein the precipitate is heated to a temperature of about 650°C for a period of about 30 to 60 minutes.
8. A method according to claim 5, wherein the precipitate is heated to a temperature of about 750°C for a period of about 10 to 30 minutes.

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