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Berkowitz

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[54] **PREPARATION OF LOW BULK DENSITY SODIUM DICHLOROISOCYANURATE DIHYDRATE**

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[52] **U.S. Cl.** ..... **260/248 C**

[51] **Int. Cl.** **C07d 55/38**

[58] **Field of Search** ..... 260/248 C

[56] **References Cited**

UNITED STATES PATENTS

3,035,056	5/1962	Symes	260/248
3,035,057	5/1962	Symes	260/248
3,452,012	6/1969	Langenhoff et al.	260/248
3,803,144	4/1974	Berkowitz	260/248

Primary Examiner—John M. Ford

[57] **ABSTRACT**

Process for producing a storage-stable, freeflowing, thermal-decomposition resistant, low bulk density sodium dichloroisocyanurate dihydrate by contacting a water-wet sodium dichloroisocyanurate dihydrate with a water miscible organic solvent and drying the solvent-wet material to remove the solvent from the material without removing the waters of hydration.

6 Claims, No Drawings

PREPARATION OF LOW BULK DENSITY SODIUM DICHLOROISOCYANURATE DIHYDRATE

This invention relates to the production of low bulk density sodium dichloroisocyanurate dihydrate from waterwet crystals of sodium dichloroisocyanurate dihydrate feed materials.

Dichloroisocyanurates are well-known materials which are widely used as a source of available chlorine in solid bleaching, sanitizing and detergent compositions. The sodium and potassium salts are the most widely used in laundering compositions because they are very soluble and easily removed by rinsing. The sodium dichloroisocyanurate compounds are known to exist in three forms, namely, the anhydrous form, the monohydrate (containing approximately 7.6% water of hydration by weight), and the dihydrate (containing approximately 14.1% water of hydration by weight). See Symes, U.S. Pat. Nos. 3,035,056 and 3,035,057.

One prior art procedure for producing sodium dichloroisocyanurate dihydrate involves the chlorination of trisodiumisocyanurate with gaseous chlorine. The dihydrate solids produced by chlorination are separated from the bulk of the aqueous phase by well-known methods for separating solids from liquids, and then dried to remove uncombined water to produce dry sodium dichloroisocyanurate. Dihydrates produced in this manner have bulk densities of approximately 35 pounds/cubic foot or 0.56 grams/cubic centimeter. These dihydrates are effective as a source of available chlorine when mixed with components of solid bleaching and detergent compositions having the same bulk density. However, when admixed with components of a low bulk density or fluffy type bleaching or detergent formulation, uniform formulations are not obtained since the formulations segregate during storage or shipping. This segregation results in non-homogeneous formulations which do not yield consistent sanitizing and/or bleaching action.

One attempt to produce preparations of bleaching, sterilizing and disinfecting compositions with varying bulk densities is disclosed by Brown et al in U.S. Pat. No. 3,293,188. Brown et al teach mixing a dichlorocyanurate (in acid or salt form) with a "synergistic carrier agent mixture" of sodium tripolyphosphate and sodium sulfate decahydrate. This mixture is heated and cooled to form a slurry or plastic mass, which is then dried at high temperatures, approximately 95°C and pulverized to form a powdered product with a "low bulk density" whose water content can vary from 7 to 30%. The bulk density of the final product is dependent upon the amount of water employed for combining the carrier agent and the dichlorocyanurate. Moisture contents between 5 and 15% by weight form dense homogeneous products upon drying. As the moisture contents are increased up to 50% by weight, compositions of progressively decreasing bulk densities are produced upon drying. The range of the bulk densities for the compositions produced in this manner are from 0.439 to 0.802 grams/cubic centimeter.

Brown et al's use of high temperatures (95°C) to dry their compositions, results in decomposition of the dichloroisocyanurate and in a product wherein the sodium dichloroisocyanurate component must be in the anhydrous and/or the monohydrate form. Such decomposition also results in lowering available chlorine content of the product. While the patentees state that their

products are "low bulk density" materials, these products, when admixed with low bulk density or fluffy type bleaching or detergent formulations, become segregated during shipping and storage. Segregation results in formulations that fail to yield consistent sanitizing and/or bleaching action.

It is the object of my invention to produce a storage-stable, free-flowing, thermal-decomposition resistant, low bulk density product without loss of available chlorine that will not cake, lump nor deliquesce when compounded and packaged, and which may be blended or admixed with low bulk density or fluffy type disinfecting, bleaching or detergent formulations.

I have discovered that a storage-stable, free-flowing, thermal-decomposition resistant, low bulk density sodium dichloroisocyanurate dihydrate can be prepared from waterwet crystals of sodium dichloroisocyanurate dihydrate by contacting the water-wet dihydrate crystals with a water miscible organic solvent, then drying the solvent containing material at temperatures below about 48°C to remove the contacting liquid and not the waters of hydration. Quite unexpectedly, a sodium dichloroisocyanurate dihydrate product is produced having a bulk density at least four times lighter than sodium dichloroisocyanurate dihydrate prepared in the conventional manner.

The term "low bulk density" sodium dichloroisocyanurate dihydrate used to define the product of the invention means a sodium dichloroisocyanurate dihydrate having a bulk density of about 0.1 to about 0.3 grams/cubic centimeter.

Sodium dichloroisocyanurate dihydrate may be produced by any conventional manner and used in the process of the invention. The dihydrate feed material may be in either dry or water-wet form. When employing dry dihydrate, it is advantageous to dissolve the dry crystals in water in order to dissolve the crystalline agglomerates. The solution containing the dissolved crystals is then evaporated and/or cooled, e.g. to below 12°C, to reform the crystals. The reformed crystals are then separated from the aqueous solution, mixed with the contacting liquid, and dried.

Precipitated sodium dichloroisocyanurate dihydrate produced by either conventional means or by dissolving crystalline agglomerates discussed above may be separated from the bulk of the aqueous reaction solution by any well-known means, such as by filtration, centrifugation, decantation or the like.

The treatment with the contacting liquid is carried out by adding the organic solvent to the precipitate and contacting and mixing the precipitate with the organic solvent. The organic solvent should be added almost immediately to the separated precipitate to remove any residual reaction solution. Such contacting, results in the formation of low bulk density crystals which are approximately 3 × 180 microns in size. If the mother liquor is allowed to remain on the crystals during separation, the crystals will break and agglomerate, resulting in crystals of higher bulk densities that are about 6 × 30 microns in size. If the precipitate is allowed to dry, it will cake in such a manner that complete contact with the solvent will be impossible.

The contacting liquid may be added completely at one time or added in small portions, each portion being drained completely before the next one is added. Generally, it is more efficient to employ two small portions of contacting liquid than one portion of the same total

volume. When employing two or more portions of contacting liquid, the contacting liquid may be reused with fresh contacting liquid. Thorough mixing of the precipitate and contacting liquid is necessary if the full benefits of the invention are to be realized.

The contacting liquid used for contacting the water-wet dihydrate crystals must be an organic solvent that is miscible with water. The boiling point of the solvent should be relatively low, that is less than about 48°C, so that drying temperatures below about 48°C will remove the solvent from the dihydrate but not the waters of hydration. If an organic solvent having a boiling point about 48°C is desired, drying must be conducted under vacuum to assure drying at below 48°C. The solvent should also be chemically inert in the presence of the dihydrate. Examples of organic solvents which may be employed in my invention are acetone, methanol, tetrahydrofuran, and acetonitrile.

After the dihydrate crystals have been thoroughly mixed with the contacting liquid, the dihydrate crystals are dried. Drying may be carried out by any known manner, such as by a warm air stream or in a vacuum oven.

Drying temperatures below about 48°C are employed, and preferably between 25° and 35°C. Temperatures slightly above 48°C may be employed if losses of minor amounts of water of hydration can be tolerated. Temperatures above about 66°C are avoided in order to prevent the formation of the anhydrous salt. Drying temperatures are employed which will remove the organic solvent retained by the precipitate but which will not remove the sodium dichloroisocyanurate waters of hydration.

The low bulk density sodium dichloroisocyanurate crystals of the invention have an average size of 3 × 180 microns and a bulk density of about 0.1 to about 0.3 grams/cubic centimeter, preferably about 0.13 to about 0.25 grams/cubic centimeter. A particularly preferred bulk density is on the order of 0.13 grams/cubic centimeter. Upon heating, both waters of hydration are lost in one step at temperatures between 48° and 66°C. These low bulk density crystals have the same excellent storage-stability and free-flowing properties as the conventionally prepared sodium dichloroisocyanurate dihydrate. The inventive crystals are also resistant to thermal decomposition, a property which is an especial advantage of the dihydrate material.

Conventionally prepared sodium dichloroisocyanurate dihydrate crystals are in agglomerated form and have an approximate size of 6 × 30 microns and bulk densities above 0.56 grams/cubic centimeter. Upon heating, waters of hydration are released in two steps. The first water of hydration is removed at temperatures between 65° and 70°C. The second water of hydration is removed at temperatures between 105° and 110°C.

The following examples are given not by way of limitation, but merely as descriptive of the invention.

EXAMPLE 1–A

Prior Art Preparation of Sodium Dichloroisocyanurate Dihydrate

A 20% by weight aqueous slurry of dichloroisocyanuric acid was prepared and fed into a reactor. A 50% sodium hydroxide solution was prepared and was simultaneously fed into the same reactor at a sufficient rate to maintain the pH at 6.8. The temperature of the reaction mixture was maintained between 20° and 25°C by a water cooled heat exchanger. The resulting slurry containing precipitated sodium dichloroisocyanurate dihydrate was filtered through a medium porosity funnel to separate the salt from the aqueous medium. The separated salt was then gently dried at about 40°C in a warm air stream for approximately 24 hours. The resultant product was a free-flowing, white crystalline sodium dichloroisocyanurate dihydrate material. Available chlorine was assayed at 55.2%, theoretical 55.4%. The dihydrate's bulk density was 35 pounds/cubic foot (0.56 grams/cubic centimeter).

EXAMPLE 1–B

Process of the Invention

Sodium dichloroisocyanurate dihydrate was produced and recovered according to Example 1 except that no drying step was carried out. Immediately after filtration, the water-wet dihydrate crystals were thoroughly contacted and mixed with acetone. The acetone-wet salt product was then dried at about 30°C in a warm air stream for approximately 24 hours. The resultant product was a fluffy, free-flowing, white crystalline product identified as sodium dichloroisocyanurate dihydrate material having an available chlorine assay of 55.2%; theoretical 55.4%. The dihydrate had a bulk density of 8.1 pounds/cubic foot (0.13 grams/cubic centimeter).

EXAMPLE 2

Horizontal Flame Propagation Test

Samples from Examples 1–A and 1–B were tested according to Procedure RI7593 of the U.S. Bureau of Mines. The samples were placed in a 1 × 2 × 7 inch bed and heated at one end with a propane torch. The rate of horizontal flame propagation was timed and measured. The products from Examples 1–A and 1–B showed no flame propagation and could not be ignited.

The invention being thus described, it will be obvious that the same may be varied in many ways. Such variations are not to be regarded as a departure from the spirit and scope of the invention, and all such modifications are intended to be included within the scope of the following claims.

What is claimed is:

1. A process for the production of a storage-stable, free-flowing, thermal-decomposition resistant, low bulk density sodium dichloroisocyanurate dihydrate which comprises contacting water-wet sodium dichloroisocyanurate dihydrate with a water miscible organic solvent, drying the solvent treated sodium dichloroisocyanurate at a temperature less than about 48°C to remove the organic solvent and not the waters of hydration, and recovering a low bulk density, sodium dichloroisocyanurate dihydrate having a bulk density of about 0.1 to about 0.3 grams/cubic centimeter.

2. The process of claim **1** wherein the water miscible organic solvent is acetone.

3. The process of claim **2** wherein the drying temperature is between 25° and 35°C.

4. Sodium dichloroisocyanurate dihydrate having a bulk density of about 0.1 to about 0.3 grams/cubic centimeter.

5. Sodium dichloroisocyanurate dihydrate having a bulk density of about 0.13 to about 0.25 grams/cubic centimeter.

6. Sodium dichloroisocyanurate dihydrate having a bulk density of 0.13 grams/cubic centimeter.

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UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

PATENT NO. : 3,888,855

DATED : June 10, 1975

INVENTOR(S) : Sidney Berkowitz

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Abstract, line 1, "freeflowing" should read --free-flowing--.

Column 1, line 6, "waterwet" should read --water-wet--.

Column 2, line 17, "waterwet" should read --water-wet--.

Column 3, line 13, "about" should read --above--.

Signed and Sealed this

eighth Day of *June 1976*

[SEAL]

Attest:

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Attesting Officer

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Commissioner of Patents and Trademarks