NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB 1903.63-2023

National food safety standard - Food nutritional fortification - Calcium glycerophosphate

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Table of Contents

1 Scope

This Standard applies to calcium glycerophosphate, the food nutritional enhancer obtained by neutralizing glycerophosphate with calcium hydroxide or calcium carbonate.

2 Chemical name, molecular formula, structural formula and

relative molecular mass

2.1 Chemical name

Calcium glycerophosphate

2.2 Molecular formula

C3H7CaO6P

2.4 Relative molecular mass

210.14 (according to the 2018 international relative atomic mass)

3.1 Sensory requirements

Sensory requirements shall comply with the requirements in Table 1.

Annex A Inspection methods

A.1 General The reagents and water used in this standard refer to analytically pure reagents and grade 3 water specified in GB/T 6682 unless otherwise specified. Standard

solutions, standard solutions for impurity determination, preparations and products used in the test are prepared in accordance with the provisions of GB/T 601, GB/T602 and GB/T603 unless otherwise specified. Solutions used in the test refer to aqueous solutions unless otherwise specified.

A.2 Identification test A.2.1 Reagents and materials A.2.1.1 Nitric acid.

A.2.1.2 Molybdic acid.

A.2.1.3 Ammonia water.

A.2.1.4 Hydrochloric acid solution: 3.65 g/L.

A.2.1.5 Dilute nitric acid solution: Take 105 mL of nitric acid (A.2.1.1) and dilute to 1000 mL with water.

A.2.1.6 Ammonium molybdate solution: Weigh 6.5 g of molybdic acid (A.2.1.2). Add

14 mL of water and 14.5 mL of ammonia water (A.2.1.3). Shake to dissolve. Cool.

While stirring, slowly add to a mixture of 32 mL of cooled dilute nitric acid solution (A.2.1.5) and 40 mL of water. Let stand for 48 h. Filter. Take the filtrate.

A.2.2 Instruments and equipment A.2.2.1 Balance: sensitivity is 0.001 g.

A.2.2.2 Platinum wire: 1 piece.

A.2.3 Identification methods A.2.3.1 Solubility test Weigh about 1.0 g of the specimen. Add 10 mL of water <=5°C to dissolve. Boil. White crystals will precipitate.

A.2.3.2 Identification of ammonium molybdate Weigh 0.1 g (accurate to 0.001 g) of the specimen. Dissolve it in 10 mL of water and

10 mL of dilute nitric acid solution (A.2.1.5). Add 5 mL of ammonium molybdate solution (A.2.1.6). Boil until a yellow precipitate is produced.

A.2.3.3 Identification of calcium Take a platinum wire. Wet it with hydrochloric acid solution (A.2.1.4), and dip it into the specimen to take sample. Burn it in a colorless flame, and the flame will appear brick red.

A.2.3.4 Infrared identification Take an appropriate amount of dry sample. Place it in an agate mortar and grind it evenly.

Put it into a mold, press it tightly and scan it. The infrared spectrum of the sample shall be consistent with the infrared spectrum of the calcium glycerophosphate standard.

Refer to Figure B.1 in Annex B for the infrared spectrum of the calcium glycerophosphate standard.

A.3 Determination of calcium glycerophosphate content (on a dry basis)

A.3.1 Method summary In the test solution, calcium carboxylic acid is used as an indicator and titrated with EDTA-2Na standard titration solution. The content is calculated based on the amount of EDTA-2Na standard titration solution consumed.

A.3.2 Reagents and materials A.3.2.1 Sodium chloride.

A.3.2.2 Calcium carboxylate indicator.

A.3.2.3 Sodium hydroxide.

A.3.2.4 Calcium indicator: Weigh 10 g of sodium chloride (A.3.2.1) that has been dried at 105 degrees C~110 degrees C for 2 h. Grind it into a fine powder in a mortar. Then add 0.1 g of calcium carboxylate indicator (A.3.2.2). Grind it into a fine powder. Mix well.

A.3.2.5 Hydrochloric acid solution (1+1): Add concentrated hydrochloric acid to an equal volume of water.

A.3.2.6 Sodium hydroxide solution (10 mol/L): Weigh 400 g of sodium hydroxide (A.3.2.3) and dilute to 1000 mL with water.

A.3.2.7 Disodium ethylenediaminetetraacetic acid standard titration solution: c (EDTA)

= 0.05 mol/L.

A.3.3 Instruments and equipment A.5.2 Instruments and equipment Balance: sensitivity is 0.0001 g.

A.5.3 Analysis steps Weigh 1 g of the specimen (accurate to 0.0001 g). Place it in 100 mL of carbon dioxide-free water. Add 2 drops of phenolphthalein indicator (A.5.1.7). If the solution is pink, add hydrochloric acid solution (A.5.1.5) until it is colorless. Record the volume of hydrochloric acid solution (A.5.1.5) added. If the solution is colorless, add sodium hydroxide solution (A.5.1.6) until the solution is pink. Record the volume of sodium hydroxide added.

A.5.4 Calculation of results The volume of hydrochloric acid or sodium hydroxide consumed in titration per gram of sample is taken as the measurement result.

The absolute difference between two independent determination results obtained under repeatability conditions shall not exceed 10% of the arithmetic mean.

A.6 Free glycerol and alcohol-soluble substances A.6.1 Reagents and materials Absolute ethanol.

A.6.2 Instruments and equipment A.6.2.1 Balance: sensitivity 0.0001 g.

A.6.2.2 Constant temperature drying oven: accuracy is 70 degrees C+/-2 degrees C.

A.6.2.3 Constant temperature water bath.

A.6.3 Analysis steps Weigh 1 g of the specimen (accurate to 0.0001 g). Add 25 mL of

anhydrous ethanol.

Shake for 2 min. Filter. Wash the residue with 5 mL of anhydrous ethanol. Combine the filtrate and washings. Evaporate to dryness on a water bath. Place in a constant temperature drying oven. Dry the residue at 70 degrees C+/-2 degrees C for 1 h.

A.6.4 Result calculation The mass fraction w3 of free glycerol and alcohol-soluble matter in calcium glycerophosphate is calculated according to formula (A.3).

Where, m - Mass of the residue after drying, in grams (g);

m0 - Mass of the specimen, in grams (g);

The calculation result is expressed to 1 decimal place.

The absolute difference between two independent determination results obtained under repeatability conditions shall not exceed 0.5% of the arithmetic mean.

A.7 Determination of phosphate A.7.1 Reagents and materials A.7.1.1 Nitric acid.

A.7.1.2 Molybdic acid.

A.7.1.3 Ammonia water.

A.7.1.4 Potassium dihydrogen phosphate.

A.7.1.5 Dilute nitric acid solution: Take 105 mL of nitric acid (A.7.1.1). Dilute it to 1000 mL with water.

A.7.1.6 Ammonium molybdate solution: Weigh 6.5 g of molybdic acid (A.7.1.2). Add

14 mL of water and 14.5 mL of ammonia water (A.7.1.3). Shake to dissolve. Cool.

While stirring, slowly add to the cooled mixture of 32 mL of nitric acid (A.7.1.1) and

40 mL of water. Let stand for 48 h. Filter. Take the filtrate.

A.7.1.7 Phosphate standard stock solution: Accurately weigh 0.192 g of potassium dihydrogen phosphate (A.7.1.4). Dilute to 100 mL with water.

A.7.1.8 Phosphate standard working solution: Pipette 3 mL of phosphate standard stock solution (A.7.1.7). Add dilute nitric acid solution (A.7.1.5) to 100 mL.

A.7.2 Instruments and equipment Balance: sensitivity is 0.0001 g.

A.7.3 Analysis steps Weigh 1 g of the specimen (accurate to 0.0001 g). Place in a 25 mL Nessler colorimetric tube. Add 10 mL of dilute nitric acid solution (A.7.1.5) to dissolve. Add 10 mL of ammonium molybdate solution (A.7.1.6). Shake well. Let stand for 10 min. Compare with the control solution made from 10 mL of phosphate standard working solution (A.7.1.8). The turbidity of the sample solution is not darker than that of the standard solution, that is, the phosphate content in the sample is <=0.04%.