NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB 1903.53-2021

National food safety standard - Food Nutrient Fortifier - D-calcium pantothenate

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State Administration for Market Regulation

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1 Scope

This Standard applies to the food nutrient fortifier D-calcium pantothenate, which is obtained by acylation reaction of beta-alanine calcium and D-pantoate lactone.

2 Chemical name, molecular formula, structural

formula and relative molecular mass

2.1 Chemical name

D-(+)-N-(2,4-dihydroxy-3,3-dimethylbutyryl)-beta-alanine calcium

2.2 Molecular formula

C18H32CaN2O10

2.4 Relative molecular mass

476.54 (according to the international relative atomic mass in 2018)

3.1 Sensory requirements

Sensory requirements shall be in accordance with Table 1.

Appendix A Inspection methods

A.1 General provisions The reagents and water used in this Standard refer to analytical reagents and grade-3 water that is specified in GB/T 6682, when other requirements are

not indicated. The standard solution, the standard solutions, preparations and products for impurity determination, which are used in the test, are all prepared in accordance with the provisions of GB/T 601, GB/T 602, and GB/T 603, when no other requirements are specified. The solution used in the test, if not indicated which solvent is used, refers to aqueous solution.

A.2 Identification test A.2.1 Reagents and materials A.2.1.1 Sodium hydroxide solution: 43 g/L.

A.2.1.2 Copper sulfate solution: 125 g/L.

A.2.1.3 Phenolphthalein indicator solution: 10 g/L.

A.2.1.4 Hydrochloric acid solution: c(HCl) = 1 mol/L.

A.2.1.5 Ferric trichloride solution: 90 g/L.

A.2.1.6 Ammonium oxalate solution: 35 g/L.

A.2.1.7 Glacial acetic acid.

A.2.1.8 Hydrochloric acid.

A.2.1.9 Potassium bromide: spectrally pure, dry product.

A.2.2 Identification method A.2.2.1 Weigh about 50 mg of the sample; add 5 mL of sodium hydroxide solution (A.2.1.1); shake; add 2 drops of copper sulfate solution (A.2.1.2), and it appears blue-purple.

A.2.2.2 Weigh about 50 mg of the sample; add 5 mL of sodium hydroxide solution (A.2.1.1); shake; boil for 1 min; let cool; add 1 drop of phenolphthalein indicator solution (A.2.1.3); add hydrochloric acid solution (A.2.1.4) until the solution fades; then, add another 0.5 mL of hydrochloric acid solution (A.2.1.4);

add 2 drops of ferric trichloride solution (A.2.1.5), and it appears bright yellow.

A.2.2.3 The identification reaction of the calcium salt in the aqueous solution of this product: Weigh 0.5 g of the sample; add 5 mL of water to dissolve; add ammonium oxalate solution (A.2.1.6), and a white precipitate appears; the precipitate is insoluble in glacial acetic acid; but it is soluble in hydrochloric acid.

A.2.2.4 Infrared spectroscopy: use potassium bromide pellet technique, to test according to GB/T 6040. The infrared spectrum of the sample shall be consistent with the infrared spectrum of the D-calcium pantothenate standard.

Refer to Figure B.1 in Appendix B for the standard infrared spectrum of D-calcium pantothenate.

A.2.2.5 Clarity: Weigh about 1.00 g of the sample; add 20 mL of water to dissolve; the solution is clear and colorless.

A.2.2.6 Solubility: easily soluble in water and glycerin; slightly soluble in ethanol;

insoluble in trichloromethane or ether.

A.3 Determination of D-calcium pantothenate content (on a dry basis)

A.3.1 Method summary Use high performance liquid chromatography for determination, C18 chromatographic column for separation, UV detector for detection, and the peak area external standard method to quantitatively calculate the content of D-calcium pantothenate in the sample.

A.3.2 Reagents and materials A.3.2.1 Acetonitrile: chromatographically pure.

A.3.2.2 Disodium hydrogen phosphate.

A.3.2.3 Sodium hydroxide solution: 43 g/L.

A.3.2.4 Sodium dihydrogen phosphate solution: Weigh 3.2 g of sodium dihydrogen phosphate (A.3.2.2) into a 1 L volumetric flask; use water to dilute to the mark; mix well. Use sodium hydroxide solution (A.3.2.3) to adjust pH to 5.5.

A.3.2.5 D-sodium pantoate (CAS: 60979-68-2): content >=97.0%.

A.3.2.6 D-calcium pantothenate standard (CAS:137-08-6): content >=99.0%.

A.3.3 Instruments and apparatuses A.4.2.2 Ethylene Diamine Tetraacetic Acid standard titration solution: c(EDTA)

= 0.05 mol/L.

A.4.2.3 Calcium purpurin indicator: Take 0.1 g of calcium purpurin; add 10 g of anhydrous sodium sulfate; grind evenly, to get it.

A.4.3 Analysis steps Weigh 0.5 g of the sample (accurate to 0.000 1 g); add 100 mL of water to dissolve; add 15 mL of sodium hydroxide solution (A.4.2.1) and about 0.1 g of calcium purpurin indicator (A.4.2.3); use Ethylene Diamine Tetraacetic Acid standard titration solution (A.4.2.2) to titrate, until the solution turns from purple to pure-blue.

A.4.4 Calculation results The mass fraction w2 of calcium content (on a dry basis) is calculated according to Formula (A.2).

Where:

V2 - the volume of the Ethylene Diamine Tetraacetic Acid standard titration solution that is consumed by the sample solution, in milliliters (mL);

c3 - the concentration of the Ethylene Diamine Tetraacetic Acid standard titration solution, in moles per liter (mol/L);

M - the molar mass of calcium, in grams per mole (g/mol), M(Ca) = 40.08;

m2 - mass of the sample, in grams (g);

X3 - loss on drying of the sample, mass fraction, %.

Express the calculation result to one decimal place. The absolute difference of two independent test results under repeatability cannot exceed 0.6% of the arithmetic mean value.

A.5 Determination of specific rotation A.5.1 Instruments and apparatuses A.5.1.1 Polarimeter.

A.5.1.2 Polarimetry tube.

A.5.2 Analysis steps potassium mercuric iodide test solution (A.7.1.2); leave it for 1 min; if there is no turbidity, it means that it passes the test.

A.8 Determination of alkalinity A.8.1 Reagents and materials A.8.1.1 Hydrochloric acid solution: 0.1 mol/L.

A.8.1.2 Phenolphthalein indicator solution: 10 g/L.

A.8.2 Analysis steps Weigh about 1.0 g of the sample (accurate to 0.01 g); add 20 mL of carbon dioxide-free water to dissolve; immediately add 1 mL of hydrochloric acid solution (A.8.1.1) and 0.05 mL of phenolphthalein indicator solution (A.8.1.2); if there is no pink color within 5 s, the test is passed.

A.9 Determination of loss on drying Weigh 2.0 g ~ 5.0 g of the sample (accurate to 0.000 1 g); the following operations are the same as the Method 1 of GB 5009.3-2016.

A.10 Determination of chloride A.10.1 Reagents and materials A.10.1.1 Nitric acid A.10.1.2 Hydrochloric acid standard titration solution: 0.001 4 mol/L.

A.10.1.3 Nitric acid solution: Take 105 mL of nitric acid (A.10.1.1); add water to dilute to 1 000 mL; shake well.

A.10.1.4 Silver nitrate solution: 0.1 mol/L.

A.10.2 Analysis steps Weigh about 2.5 g of the sample (accurate to 0.000 1 g) into a 50 mL volumetric flask; add 30 mL of boiling water to dissolve; dilute to the mark, as the test liquid.

Take 5 mL of the test solution; add 10 mL of boiled water; add 6 mL of nitric acid solution (A.10.1.3); mix well, pour the mixed solution into a colorimetric tube that contains 1 mL of silver nitrate solution (A.10.1.4); shake well, as the sample solution. Take another 1 mL of hydrochloric acid standard titration solution (A.10.1.2); add 14 mL of boiled water; add 6 mL of nitric acid solution (A.10.1.3);

mix well; pour the mixed solution into a colorimetric tube that contains 1 mL of silver nitrate solution (A.10.1.4); shake well, as the control solution. After placing