NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB 1903.49-2020

National food safety standard - Food Nutritional Fortification Substance - Zinc Citrate Trihydrate

Issued on: September 11, 2020 **Implemented on: March 11, 2021**

Issued by: National Health Commission and State Administration for Market Regulation

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1 Scope

This Standard is applicable to food nutritional fortification substance - zinc citrate trihydrate, which is obtained through chemical synthesis and refining with citric acid and zinc oxide or zinc carbonate as the main raw materials.

2 Chemical Name, Molecular Formula, Structural

Formula and Relative Molecular Mass

2.1 Chemical Name

Zinc citrate trihydrate

2.2 Molecular Formula

C12H10O14Zn3 ? nH2O (n = 0, 2 or 3)

2.4 Relative Molecular Mass

574.34 (anhydride) (in accordance with the international relative atomic mass of Year 2018)

610.37 (dihydrate) (in accordance with the international relative atomic mass of Year 2018)

628.38 (trihydrate) (in accordance with the international relative atomic mass of Year 2018)

Appendix A Inspection Method

A.1 General Provisions When no other requirements are indicated, the reagents and water

used in this Standard refer to analytically pure reagents and Grade-3 water specified in GB/T 6682.

When no other requirements are indicated, the standard titration solutions, and standard solutions, preparations and products for impurity determination shall be prepared in accordance with the stipulations of GB/T 601, GB/T 602 and GB/T 603.

When the solvent used for solution preparation is not specified, it refers to aqueous solution in the tests.

A.2 Identification Test A.2.1 Reagents and materials A.2.1.1 Hydrochloric acid solution: 1 + 4.

A.2.1.2 Sulfuric acid solution: 5%.

A.2.1.3 Potassium ferrocyanide solution: 100 g/L. Prepare it right before use.

A.2.1.4 Potassium permanganate solution: 3.2 g/L.

A.2.1.5 Mercury sulfate solution: weigh-take 5 g of mercury oxide; firstly, add 40 mL of water, then, slowly add 20 mL of concentrated sulfuric acid; stir while adding it, then, add 40 mL of water; stir to dissolve it.

A.2.1.6 Pyridine-acetic anhydride: 3 + 1.

A.2.2 Identification method A.2.2.1 Solubility (anhydride and dihydrate)

Slightly soluble in water; soluble in hydrochloric acid solution.

A.2.2.2 Identification of zinc salt Weigh-take about 0.2 g of the sample, dissolve it in 20 mL of water; add newly prepared

2 mL of potassium ferrocyanide solution, which generates white precipitate. Separate the precipitate through centrifugation or filtration; add hydrochloric acid solution to the precipitate, and the precipitate will not dissolve.

A.2.2.3 Identification of citrate

Where, V1---the volume of the Ethylene diamine tetra-acetic acid (EDTA) standard titration solution consumed by the titration of the sample solution, expressed in (mL);

V0---the volume of the Ethylene diamine tetra-acetic acid (EDTA) standard titration solution consumed by the titration of the blank solution, expressed in (mL);

c1---the concentration of the Ethylene diamine tetra-acetic acid (EDTA) standard titration solution, expressed in (mol/L);

M1---the molar mass of zinc citrate trihydrate, expressed in (g/mol), [M1 (C12H10O14Zn3 ? 3H2O) = 209.46];

m1---the mass of the sample, expressed in (g);

1,000---conversion factor.

Take the arithmetic mean value of two parallel determination results as the report result.

The absolute difference between two parallel determination results shall be not greater than 0.3%.

A.4 Determination of Zinc (Zn) Content (calculated as dry basis)

A.4.1 Method summary Take chrome black T as the indicator solution; use ethylene diamine tetra-acetic acid (EDTA) standard titration solution to titrate the sample solution. In accordance with the amount of the ethylene diamine tetra-acetic acid standard titration solution being used, calculate the content of zinc calculated as Zn.

A.4.2 Reagents and materials A.4.2.1 Ammonia-ammonium chloride buffer solution (pH ? 10).

A.4.2.2 Ethylene diamine tetra-acetic acid (EDTA) standard titration solution: c (EDTA)

= 0.05 mol/L.

A.4.2.3 Chrome black T indicator solution.

A.4.3 Analytical procedures Weigh-take 0.35 g of the sample (accurate to 0.0001 g) dried to a constant weight at 105 °C; add 60 mL of water and 10 mL of ammonia-ammonium chloride buffer solution;

shake to dissolve it. Add a little chrome black T indicator solution; use ethylene diamine tetra-acetic acid (EDTA) standard titration solution to titrate, until the solution turns from purple to pure blue. Meanwhile, conduct a blank test.

A.5.4 Result calculation The solubility of the sample shall be calculated in accordance with Formula (A.3):

Where, w3---the solubility of the sample, expressed in (g/100 mL);

m3---the mass of the sample, expressed in (g);

m4---the mass of the glass sand core funnel and the precipitate after drying, expressed in (g);

m5---the mass of the glass sand core funnel, expressed in (g).

Take the arithmetic mean value of parallel determination results as the determination result. The difference between two parallel determination results shall not exceed 2% of the arithmetic mean value.

A.6 Determination of Hydrochloric Acid Insoluble Matter A.6.1 Reagents and materials A.6.1.1 Hydrochloric acid solution: 6 mol/L.

A.6.1.2 Glass sand core funnel: with a pore size of 4 um ~ 9 um.

A.6.2 Instruments and equipment Electronic balance: with a division value of 1 mg.

A.6.3 Analytical procedures Weigh-take 5 g of the sample (accurate to 0.001 g); add 10 mL of hydrochloric acid solution and 50 mL of water. Magnetically heat it up and stir it for 30 min. Let the obtained solution pass through a glass sand core funnel, which is dried at 105 °C +/- 2 °C for 2 h, cooled down and weighed; conduct vacuum filtration. Divide 200 mL of water into 5 parts to rinse it. The precipitate is dried at 105 °C +/- 2 °C for 2 h, cooled down and weighed.

A.6.4 Result calculation The mass fraction w4 of hydrochloric acid insoluble matter shall be calculated in accordance with Formula (A.4):

A.8.1.2 Barium chloride solution: 250 g/L.

A.8.1.3 Potassium sulfate standard solution: after preparing it in accordance with GB/T 602, dilute it to the equivalent of 0.01 mg of sulfate ion per 1 mL.

A.8.2 Analytical procedures Weigh-take 0.10 g +/- 0.01 g of the sample; place it in a 50 mL Nessler colorimetric tube.

Add an appropriate amount of water and 2 mL of hydrochloric acid solution to dissolve it. Add 5 mL of barium chloride solution; use water to dilute it to 50 mL; shake it well;

place it in a dark place for 10 min. Under a black background, axially observe it;

compare the manifested turbidity with that of the standard turbidity solution.

Standard turbidity solution: measure-take 5 mL of sulfate standard solution and place it in a 50 mL colorimetric tube. Process it at the same time and in the same mode as the sample solution.

A.8.3 Result determination The manifested turbidity of the sample solution shall not be deeper than that of the standard turbidity solution. In other words, the sulfate in the sample is not greater than 0.05%.

A.9 Determination of Solution Clarity A.9.1 Reagents and materials A.9.1.1 Hydrazine sulfate: before use, dry it at 105 °C +/- 2 °C to a constant weight.

A.9.1.2 Urotropine solution: 10 %.

A.9.1.3 Turbidity standard stock solution: weigh-take 1.00 g of hydrazine sulfate; add water to dissolve and dilute it to 100 mL. After letting it stand for 6 h, evenly mix it with 100 mL of urotropine solution; shake it well; store it in the dark.

A.9.1.4 Turbidity standard intermediate solution: take 15.0 mL of the turbidity standard stock solution; use water to dilute it to 1,000 mL. When the obtained solution is determined at an optical path of 1 cm and a wavelength of 550 nm, its absorbance shall be 0.12 ~ 0.15. The storage time of the solution shall not exceed 48 h.