NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB 1903.77-2025

National Food Safety Standard - Food Nutritional Fortification Substance - Ferrous Citrate

食品安全国家标准 食品营养强化剂 柠檬酸亚铁

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1 Scope

This standard applies to the food nutritional fortification substance ferrous citrate obtained by reacting ferrous sulfate with sodium carbonate to form ferrous carbonate and then reacting the ferrous carbonate with citric acid.

2 Molecular formula and relative molecular mass

2.1 Molecular formula. FeC6H6O7.

2.2 Relative molecular mass. 245.95, based on the 2022 international relative atomic masses.

3 Technical requirements

3.1 Sensory requirements. The sensory requirements shall comply with Table 1. Table 1 fixes the colour as pale greyish green or white and the state as powder or crystals. The test method given against both is the same: take a suitable amount of the sample, place it in a clean, dry white porcelain dish, and observe its colour and state under natural light.

3.2 Physical and chemical indexes. The physical and chemical indexes shall comply with Table 2. Table 2 fixes the following limits, each with the test method shown against it. Ferrous content, expressed as Fe, mass fraction not less than 20.0 percent, by A.3 of Annex A. Ferric iron, expressed as Fe, mass fraction not more than 3.0 percent, by A.4 of Annex A. Chloride, expressed as Cl, mass fraction not more than 0.2 percent, by A.5 of Annex A. Sulfate, expressed as SO4, mass fraction not more than 0.06 percent, by A.6 of Annex A. Lead, not more than 2.0 mg/kg, by the graphite furnace atomic absorption spectrometric method of GB 5009.75.

A.1 General provisions

Unless other requirements are stated, the reagents and the water used in this standard are analytically pure reagents and grade three water as laid down in GB/T 6682.

Unless other requirements are stated, the standard solutions, the standard solutions for the determination of impurities, and the preparations and products used in the tests are prepared as laid down in GB/T 601, GB/T 602 and GB/T 603.

Where the solvent used to prepare a solution is not stated, an aqueous solution is meant.

A.2 Identification test

A.2.1 Reagents and materials. Pyridine. Glacial acetic acid. Potassium ferricyanide solution: weigh 1 g of potassium ferricyanide and dissolve it in 10 mL of water; this solution is prepared fresh for use.

A.2.2.1 Identification of the citrate. Weigh 1 g of the sample, add 10 mL of water to make a sample solution of 100 mg/mL, add 15 mL of pyridine and 5 mL of glacial acetic acid and shake; the solution takes on a pale red colour.

A.2.2.2 Identification of the ferrous salt. Weigh 1 g of the sample, add 10 mL of water to make a sample solution of 100 mg/mL, and add a suitable amount of the potassium ferricyanide solution dropwise; a deep blue precipitate forms.

A.3 Determination of ferrous content, expressed as Fe

A.3.1 Outline of the method. In an acid medium the sample is titrated with cerium sulfate standard solution, the end point being shown by the 1,10-phenanthroline ferrous indicator solution.

A.3.2 Reagents and materials. Phosphoric acid. Sulfuric acid solution: measure 16 mL of sulfuric acid, add it to 100 mL of water, cool to room temperature, then dilute with water and make up to 1000 mL. Cerium sulfate standard volumetric solution: concentration of cerium sulfate 0.1 mol/L. 1,10-phenanthroline ferrous indicator solution: weigh 0.7 g of ferrous sulfate heptahydrate, dissolve it in 100 mL of water, add 2 drops of sulfuric acid and 0.15 g of 1,10-phenanthroline monohydrate, and mix; store this solution in a closed container.

A.3.3 Apparatus and equipment. Balance, readability 0.0001 g.

A.3.4 Procedure. Weigh about 0.4 g of the sample, to the nearest 0.0001 g, into 20 mL of the sulfuric acid solution, add 5 mL of phosphoric acid, dilute with 50 mL of water and shake to dissolve the sample. Add several drops of the 1,10-phenanthroline ferrous indicator solution and titrate with the cerium sulfate standard volumetric solution until the solution changes from red to pale blue, recording the volume of cerium sulfate standard volumetric solution consumed. Carry out a blank test at the same time.

A.3.5 Calculation of the result. The mass fraction w1 of the ferrous content, expressed as Fe, is calculated by formula (A.1). In the formula: c1 is the concentration of the cerium sulfate standard volumetric solution, in moles per litre; V1 is the volume of cerium sulfate standard volumetric solution consumed in titrating the sample solution, in millilitres; V2 is

the volume consumed in titrating the blank solution, in millilitres; M is the molar mass of iron, in grams per mole, taken as 55.845; m is the mass of the sample, in grams; and 1000 is the volume conversion factor. The test result is taken as the arithmetic mean of parallel determinations, kept to two decimal places. The absolute difference between two independent results obtained under repeatability conditions shall not exceed 2 percent of their arithmetic mean.

A.4 Determination of ferric iron, expressed as Fe

A.4.1 Reagents and materials. Potassium iodide. Hydrochloric acid. Sodium thiosulfate standard volumetric solution: concentration of sodium thiosulfate 0.1 mol/L. Starch indicator solution: 10 g/L.

A.4.2 Apparatus and equipment. Balance, readability 0.0001 g.

A.4.3 Procedure. Weigh about 2 g of the sample, to the nearest 0.0001 g, dissolve it in 100 mL of water and 10 mL of hydrochloric acid, add 3 g of potassium iodide, shake well and leave it in the dark for 5 min. Add 0.5 mL of the starch indicator solution and titrate with the sodium thiosulfate standard volumetric solution until the blue colour of the solution disappears, recording the volume of sodium thiosulfate standard volumetric solution consumed. Carry out a blank test at the same time.

A.4.4 Calculation of the result. The mass fraction w2 of ferric iron, expressed as Fe, is calculated by formula (A.2). In the formula: c2 is the concentration of the sodium thiosulfate standard volumetric solution, in moles per litre; V3 is the volume of sodium thiosulfate standard volumetric solution consumed in titrating the sample solution, in millilitres; V4 is the volume consumed in titrating the blank solution, in millilitres; M is the molar mass of iron, in grams per mole, taken as 55.845; m is the mass of the sample, in grams; and 1000 is the volume conversion factor. The test result is taken as the arithmetic mean of parallel determinations, kept to two decimal places. The absolute difference between two independent results obtained under repeatability conditions shall not exceed 5 percent of their arithmetic mean.

A.5 Determination of chloride, expressed as Cl

A.5.1 Reagents and materials. Nitric acid solution: 1 plus 9 by volume. Silver nitrate solution: 17 g/L. Chloride standard solution: 0.01 mg/mL.

A.5.2 Apparatus and equipment. Balance, readability 0.01 g.

A.5.3 Procedure. Weigh 0.1 g of the sample, to the nearest 0.01 g, into a mixture of 2 mL of the nitric acid solution and 25 mL of water, heating if necessary to dissolve it, then dilute with water and make up to 100 mL. Take 10 mL of that solution into a colour comparison tube, add 1 mL of the silver nitrate solution, dilute to 50 mL with water, shake well and leave in the dark for 5 min. Compare the turbidity of the sample tube with that of the standard

tube; the turbidity of the sample tube shall not be deeper than that of the standard tube, in which case the mass fraction of chloride in the sample, expressed as Cl, is not more than 0.2 percent. Preparation of the standard tube: accurately take 2 mL of the chloride standard solution and treat it in the same way and at the same time as the sample tube.

A.6 Determination of sulfate, expressed as SO4

A.6.1 Reagents and materials. Hydrochloric acid solution: 1 plus 3 by volume. Barium chloride solution: 250 g/L. Sulfate standard solution: 0.1 mg/mL.

A.6.2 Apparatus and equipment. Balance, readability 0.01 g.

A.6.3 Procedure. Weigh 0.5 g of the sample, to the nearest 0.01 g, into a colour comparison tube, add 1 mL of the hydrochloric acid solution, dilute with water to 30 mL to 40 mL, then add 1 mL of the barium chloride solution, dilute to 50 mL with water, shake well and leave in the dark for 5 min. Compare the turbidity of the sample tube with that of the standard tube; the turbidity of the sample tube shall not be deeper than that of the standard tube, in which case the mass fraction of sulfate in the sample, expressed as SO4, is not more than 0.06 percent. Preparation of the standard tube: accurately take 3 mL of the sulfate standard solution and treat it in the same way and at the same time as the sample tube.