GB

NATIONAL STANDARD OF THE
PEOPLE’S REPUBLIC OF CHINA

GB 1903.72-2025

National Food Safety Standard - Food Additive - Sodium Iron (III) Ethylenediaminetetraacetate Trihydrate

食品安全国家标准 食品营养强化剂 乙二胺四乙酸铁钠

Issued on: March 16, 2025 **Implemented on: September 16, 2025**

Issued by: National Health Commission of the People’s Republic of China;
State Administration for Market Regulation.

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1 Scope

This Standard applies to sodium iron (III) ethylenediaminetetraacetate trihydrate, a food nutritional enhancer produced by the reaction of inorganic iron salts and Ethylenediaminetetraacetic acid sodium salt as raw materials.

2 Chemical Name, Molecular Formula, Structural Formula and Relative Molecular Mass

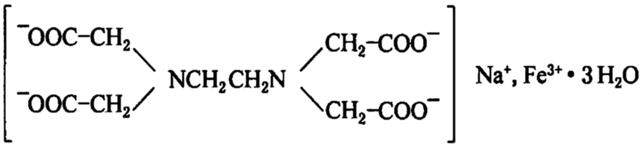
2.1 Chemical name

Sodium iron (III) ethylenediaminetetraacetate trihydrate

2.2 Molecular formula

$C_{10}H_{12}FeN_2NaO_8 \cdot 3H_2O$

2.3 Structural formula



2.4 Relative molecular mass

421.09 (by 2022 international relative atomic mass).

3 Technical Requirements

3.1 Sensory requirements

The sensory requirements shall comply with the provisions of Table 1.

Appendix A

Inspection Methods

A.1 Safety tips

Some of the reagents used in the test methods of this Standard are toxic or corrosive. Appropriate safety and health measures shall be taken during operation. If splashed on the skin, rinse with water immediately. In severe cases, treatment shall be given immediately. When using flammable materials, it is strictly forbidden to use open flames for heating.

A.2 General

Unless otherwise specified, the purity of the reagents used in this Standard shall be analytically pure. The used standard titration solutions, standard solutions for impurity determination, preparations and products shall be prepared in accordance with the provisions of GB/T 601, GB/T 602 and GB/T 603 respectively. The test water shall comply with the provisions of Grade 3 water in GB/T 6682. When the solution used in the test is not specified with which solvent it is prepared, it refers to an aqueous solution.

A.3 Identification test

A.3.1 Reagents and materials

A.3.1.1 Ammonium thiocyanate solution (8 g/L): weigh 0.8 g of ammonium thiocyanate (accurate to 0.000 1 g), dissolve it in water and make constant volume to 100 mL.

A.3.1.2 Hydrochloric acid solution ($V+V$): 1 + 9.

A.3.2 Instruments and equipment

A.3.2.1 Balance: sensitivity is 0.000 1 g.

A.3.2.2 UV-visible spectrophotometer (1 cm quartz cuvette).

A.3.3 Identification method

A.3.3.1 Color reaction: Weigh 0.05 g of specimen (accurate to 0.000 1 g); place in a 10 mL glass test tube; add 5 mL of water to dissolve; add 1.0 mL of ammonium thiocyanate solution (A.3.1.1); the color of the solution shall not change. Then add 0.5 mL of hydrochloric acid solution (A.3.1.2); mix well; the solution shall be dark red.

A.3.3.2 Ultraviolet absorption: Weigh 0.1 g of specimen (accurate to 0.000 1 g); dissolve in water and dilute to 100 mL; shake well; transfer 1 mL to a 50 mL volumetric flask; dilute with water and make constant volume; shake well. The concentration of the specimen solution is 20 μg/mL. Spectrophotometric determination is performed in the wavelength range of 190 nm ~

700 nm. The solution shall have a maximum absorption peak at a wavelength of 256 nm ± 2 nm.

A.4 Determination of iron content

A.4.1 Method summary

Under strong acidic conditions of sodium iron (III) ethylenediaminetetraacetate trihydrate, free Fe^{3+} reacts quantitatively with excess potassium iodide to generate and precipitate I_2, which is then titrated with sodium thiosulfate standard titration solution. The iron content in the specimen is calculated based on the titration consumption of sodium thiosulfate standard titration solution.

A.4.2 Reagents and materials

A.4.2.1 Potassium iodide.

A.4.2.2 Hydrochloric acid.

A.4.2.3 Sodium thiosulfate standard titration solution: $c(Na_2S_2O_3) = 0.1$ mol/L.

A.4.2.4 Starch indicator solution (10 g/L): Weigh 0.5 g of soluble starch and add a small amount of water to make a paste. Pour 50 mL of boiling water while stirring; boil and stir; then cool and set aside (prepare immediately before use).

A.4.3 Instruments and equipment

Balance: Sensitivity is 0.000 1 g.

A.4.4 Analysis procedures

Weigh 0.5 g of specimen (accurate to 0.000 1 g); dissolve it in 40 mL of water in an iodine volume flask; add 3 g of potassium iodide (A.4.2.1) and 20 mL of hydrochloric acid (A.4.2.2); shake well. Seal the bottle stopper with water; place in a dark place for about 5 min; remove the stopper and rinse the stopper cover with a small amount of water; titrate with sodium thiosulfate standard titration solution (A.4.2.3) to the near end point (light yellow); add 2.0 mL of starch indicator solution (A.4.2.4); and continue titrating until the blue color disappears as the end point.

While measuring the sample, titrate the blank solution without sample with sodium thiosulfate standard titration solution (A.4.2.3).

A.4.5 Calculation of results

The mass fraction of iron w_1 is expressed in %, and is calculated according to Formula (A.1):

A.5.2.6 Calcium acetate monohydrate standard titration solution (0.25 mol/L): Weigh 44.0 g of calcium acetate monohydrate (A.5.2.1) (accurate to 0.0001 g); dissolve it in water; transfer it into a 1,000 mL volumetric flask; and make constant volume to the scale.

A.5.3 Instruments and equipment

A.5.3.1 Balance: Sensitivity is 0.000 1 g

A.5.3.2 pH meter.

A.5.4 Analysis procedures

A.5.4.1 Calibration of calcium acetate monohydrate standard titration solution: Weigh 2.0 g ~ 2.1 g of ethylenediaminetetraacetic acid (A.5.2.2) (accurate to 0.000 1 g) and place it in a 250 mL conical flask. Add 150 mL of water; then dissolve it with sodium hydroxide solution (A.5.2.4) and adjust the pH to 11.0~12.0; add 30 mg of hydroxynaphthol blue indicator (A.5.2.3); and titrate with calcium acetate monohydrate standard titration solution (A.5.2.6). When the solution changes from blue to red, it is the end point.

A.5.4.2 Titration operation: Weigh 0.8 g~1.0 g of specimen (accurate to 0.000 1 g); place in a 250 mL conical flask; and add 75 mL of distilled water to dissolve. Use triethanolamine (A.5.2.5) to adjust the pH of the solution to 9.0. After complexing and masking Fe^{3+}, use sodium hydroxide solution (A.5.2.4) to adjust the pH of the specimen solution to 12.5~13.0; and the solution becomes colorless and clear. Add 30 mg of hydroxynaphthol blue indicator (A.5.2.3); titrate with a calibrated calcium acetate monohydrate solution; and the titration endpoint is when the solution changes from blue to red.

While measuring the sample, titrate the blank solution without sample with the calibrated calcium acetate monohydrate standard titration solution.

A.5.5 Calculation of results

The molar concentration c_2 of the calcium acetate monohydrate standard titration solution is expressed in mol/L and is calculated according to Formula (A.2):

$$c_2 = \frac{m_2 \times 1\ 000}{V_3 \times M_2} \qquad \cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots (\text{A.2})$$

Where:

m_2 - mass of ethylenediaminetetraacetic acid (A.5.2.2), in g;

V_3 - volume of calcium acetate monohydrate standard titration solution (A.5.2.6) consumed for calibration, in mL;

M_2 - molar mass of ethylenediaminetetraacetic acid (A.5.2.2), in g/mol (M_2 = 292.24);

A.7.2.5 Phosphoric acid solution (1 mol/L): Measure 6.25 mL of phosphoric acid and dilute it to 100 mL with water.

A.7.2.6 Aminotriacetic acid standard sample: $C_6H_9NO_6$, purity>98.0%; CAS number: 139-13-9.

A.7.2.7 Aminotriacetic acid standard stock solution (10 mg/mL): Weigh 100 mg of aminotriacetic acid standard sample (A.7.2.6) (accurate to 0.000 1 g); transfer it to a 10 mL volumetric flask; dissolve it in water and add 0.5 mL of ammonia water; mix; make constant volume to the scale with water; and shake well.

A.7.2.8 Aminotriacetic acid standard solution (10 mg/L): Weigh 1 g of specimen (accurate to 0.000 1 g) and place in a 100 mL volumetric flask. Add 100 μL of aminotriacetic acid standard stock solution (A.7.2.7); dilute to scale with copper nitrate solution (A.7.2.4); and shake well. Ultrasonic treatment to obtain a standard solution.

A.7.3 Instruments and equipment

A.7.3.1 Balance: Sensitivity is 0.000 1 g.

A.7.3.2 High performance liquid chromatograph: With UV detector.

A.7.3.3 pH meter.

A.7.4 Analysis procedures

A.7.4.1 Specimen treatment

Weigh 1 g of specimen (accurate to 0.000 1 g); add 100 μL of ammonia solution (A.7.2.3) and 50 mL copper nitrate solution (A.7.2.4); treat with ultrasound; transfer to a 100 mL volumetric flask; make constant volume with copper nitrate solution (A.7.2.4); shake well; and obtain the specimen solution.

A.7.4.2 Chromatographic reference conditions

Precolumn: C_8 chromatographic column (4.6 mm×10 mm, 5 μm).

Mobile phase: Measure 10 mL of 25% tetrabutylammonium hydroxide methanol solution (A.7.2.2); place in 200 mL of water; and then adjust the pH to 7.5±0.1 with 1 mol/L phosphoric acid. Transfer the solution to a 1,000 mL volumetric flask; add 90 mL of methanol (A.7.2.1); make constant volume to the scale with water; shake well; filter with a 0.45 μm filter membrane; degas and set aside.

Chromatographic column: C_{18} chromatographic column (4.6 mm×150 mm, 5 μm), or other equivalent chromatographic columns.

Detection wavelength: 244 nm.

Injection volume: 20 μL.

Flow rate: 2.0 mL/min.

A.7.4.3 Specimen analysis

Under the chromatographic reference conditions of A.7.4.2, perform chromatographic analysis on the standard solution and the specimen solution; record the chromatogram; and calculate the response value of aminotriacetic acid. Repeat the standard solution measurements for 3 times to take the average response value. The relative standard deviation of the 3 measurement results is ≤2.0%. The standard chromatogram of aminotriacetic acid is shown in Appendix B.

A.7.5 Judgment of results

The response value of the aminotriacetic acid chromatographic peak in the specimen solution does not exceed the difference between the response values of the aminotriacetic acid chromatographic peaks obtained from the standard solution and the sample solution, that is, the aminotriacetic acid content in the specimen is ≤0.1%.

The detection limit of this method is 20 mg/kg.

A.8 Determination of chloride (by Cl)

A.8.1 Method summary

In an acidic medium, use a silver electrode as the measuring electrode and a calomel electrode as the reference electrode; titrate with a silver nitrate standard titration solution; and determine the reaction endpoint with the help of a potential mutation.

A.8.2 Reagents

A.8.2.1 95% ethanol.

A.8.2.2 Saturated potassium nitrate solution.

A.8.2.3 Sodium hydroxide solution: 200 g/L.

A.8.2.4 Nitric acid solution ($V + V$): 2+3.

A.8.2.5 Standard silver nitrate titration solution: $c(AgNO_3) = 0.1$ mol/L.

A.8.2.6 Bromophenol blue indicator solution: 0.1% ethanol solution.

A.8.3 Instruments and equipment

A.8.3.1 Balance: Sensitivity is 0.000 1 g.

A.8.3.2 Potentiometric titrator (with silver electrode): Accuracy is 2 mV.

In an acidic medium, sulfate ions in the specimen react with barium ions to form barium sulfate precipitates, which are compared with the sulfate standard solution treated in the same way to perform a limit test.

A.9.2 Reagents

A.9.2.1 Hydrochloric acid solution ($V + V$): 1 + 4.

A.9.2.2 Sodium hydroxide solution: 160 g/L.

A.9.2.3 Barium chloride solution: 0.5 mol/L.

A.9.2.4 Zinc chloride solution: 1 mol/L.

A.9.2.5 Sulfate standard solution: Weigh 2.113 g of anhydrous sodium sulfate (accurate to 0.000 1 g) and dissolve it in water; place it in a 1,000 mL volumetric flask; and dilute to the scale. Each 1 mL of standard solution is equivalent to 1.430 mg of sulfate.

A.9.2.6 Solution A: Take 5 mL of barium chloride solution (A.9.2.3); add 55 mL of water and 20 mL of 95% ethanol solution to dilute; and then add 0.5 mL of sulfate standard solution (A.9.2.5) and mix well (prepare it before use).

A.9.2.7 Indicator solution: 0.25% p-nitrophenol solution.

A.9.3 Instruments and equipment

Balance: Sensitivity is 0.01 g.

A.9.4 Analysis procedures

Weigh 1.8 g of specimen (accurate to 0.01 g); add 30 mL of water to dissolve; slowly add 4.5 mL of sodium hydroxide solution (A.9.2.2); stir for 15 min; make constant volume to 50 mL; and filter. Take 20 mL of filtrate as specimen solution; place in a 50 mL colorimetric tube; add 2 drops of indicator solution (A.9.2.7); and titrate with hydrochloric acid solution (A.9.2.1) until the solution changes from yellow to colorless. Add 2 mL of zinc chloride solution (A.9.2.4), and titrate again with hydrochloric acid or sodium hydroxide solution to pH = 2.0. After making constant volume to 50 mL with water, add 5.0 mL of solution A (A.9.2.6) and mix well. Under a black background, observe axially and compare the turbidity of the specimen solution with the standard turbidity solution.

Standard turbidity solution: Measure 20 mL of water and place it in a 50 mL colorimetric tube. Treat it to pH = 2.0 at the same time as the specimen solution; add 300 µL of sulfate standard solution (A.9.2.5); make constant volume to 50 mL; and then add 5.0 mL of solution A (A.9.2.6); and mix well.

A.9.5 Judgment of results

------ The End ------