USDA United States Department of Agriculture
Foreign Agricultural Service

GAIN Global Agricultural Information Network

Voluntary Report – Voluntary - Public Distribution **Date:** April 12, 2023

Report Number: CH2023-0058

Report Name: National Food Safety Standard of Food Nutrition Fortifier Sodium Iron EDTA Notified

Country: China - People's Republic of

Post: Beijing

Report Category: WTO Notifications, Sanitary/Phytosanitary/Food Safety, FAIRS Subject Report

Prepared By: FAS China Staff

Approved By: Adam Branson

Report Highlights:

On March 6, 2023, China notified an updated National Food Safety Standard for Food Nutrition Fortifier Sodium Iron EDTA to the World Trade Organization (WTO) under G/SPS/N/CHN/1274. The deadline for comment submission is May 5, 2023. The proposed date of entry into force is to be determined. Comments may be submitted by email to China's SPS Enquiry Point at sps@customs.gov.cn. This report provides an unofficial translation of the draft standard.

Summary:

On March 6, 2023, China notified an updated National Food Safety Standard of Food Nutrition Fortifier Sodium Iron EDTA to the WTO under G/SPS/N/CHN/1274. This standard applies to food nutrition fortifier sodium iron EDTA formed by reaction of ferric inorganic salt and EDTA sodium salt as raw materials. It mainly specifies technical requirements and testing methods of the food nutrition fortifier sodium iron EDTA.

The notified standard is an update of the current National Food Safety Standard of Food Additive Sodium Iron (III) ethylenediaminetetraacetate, trihydrate (GB 22557-2008) (link in Chinese), which went into effect in June 2009. Changes in this standard included the revised sensory requirements, new indicators for moisture content, free iron, chloride and sulfate, and the corresponding testing methods.

This report provides an unofficial translation of the draft standard. Comments may be submitted by email to China's SPS Enquiry Point at sps@customs.gov.cn.

BEGIN TRANSLATION

National Food Safety Standard

Food Nutrition Fortifier Sodium Iron EDTA

(Draft for comments)

Foreword

This standard replaces GB 22557-2008 Food additive Sodium iron (III) ethylenediaminetetraacetate, trihydrate. Compared with GB 22557-2008, the main changes in this standard are as follows:

- Revised sensory requirements and testing methods
- Added indicators of free iron, moisture, chloride and sulfate and their testing methods
- Added indicators and testing methods for lead and arsenic
- Added national standard reference of the testing method for pH value

1 Scope

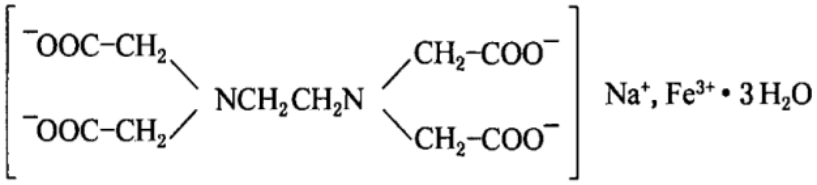
This standard is applicable to the food nutritional fortifier sodium iron EDTA produced by the reaction of inorganic iron salts and sodium EDTA as raw materials.

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

2.1 Chemical name
Sodium iron EDTA

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 (according to 2018 international relative atomic mass)

3 Technical Requirements

3.1 Sensory requirements
Sensory indicators should comply with provisions in the Table 1.

Table 1: Sensory Requirements

Item	Requirements	Testing Methods
Color	Light yellow to yellowish brown	Take an appropriate amount of the sample and place it on a clean, dry white porcelain plate. Under natural light, observe its color and status, and smell it.
Status	Powder or crystalline powder	
Smell	Odorless	

3.2 Physical and chemical indicators
Physical and chemical indicators shall comply with provisions in Table 2.

Table 2: Physical and Chemical Indicators

Item		Indicator	Testing Methods
Iron content (Fe), w/%		12.5~13.5	A.4 in Appendix A
EDTA content [calculated as $C_{10}H_{14}N_2O_8$], w/%		65.5~70.5	A.5 in Appendix A
Moisture, w/%	≤	13.5	GB 5009.3 Method IV[a]
Free iron (calculated as Fe^{3+}), w/%	≤	0.05	A.6 in Appendix A
PH value (10g/L aqueous solution)		3.5~5.5	GB/T 9724
Water insoluble matter, w/%		0.1	GB/T 9738
Aminotriacetic acid, w/%	≤	0.1	A.7 in Appendix A
Chloride (in Cl), w/%	≤	0.06	A.8 in Appendix A
Sulfate (in SO_4), w/%	≤	0.06	A.9 in Appendix A
Lead (Pb)/(mg/kg)	≤	1.0	GB 5009.12 or GB 5009.75
Total arsenic (in As)/(mg/kg)	≤	1.0	GB 5009.11 or GB 5009.76
[a] sampling amount is 0.1 g			

Appendix A

Testing Methods

A.1 Safety Statement

Some reagents used in the testing methods of this standard are toxic or corrosive and appropriate safety and health measures should be taken during operation. If it splashes onto the skin, it should be immediately rinsed with water, and in severe cases, it should be treated in a hospital immediately. When using flammable materials, use of fire for heating is strictly prohibited.

A.2 General Provisions

Unless otherwise specified, the purity of the reagents used should be analytical pure. The standard titration solution, the standard solution for impurity determination, preparations and products should be prepared in accordance with the provisions of GB/T601, GB/T602, and GB/T 603, respectively. Level III water should be used in experiments that comply with the provisions in GB/T 6682. When the solvent used in the test is not specified, it refers to aqueous solution.

A.3 Identification Tests

A.3.1 Reagents and materials

A.3.1.1 Ammonium thiocyanate solution (8g/L): Accurately weigh 0.8g of ammonium thiocyanate, dissolve with water to a constant volume of 100 mL.

A.3.1.2 Hydrochloric acid solution (10%).

A.3.2 Instruments and equipment

A.3.2.1 Balance: shall be with a sensitivity of 0.0001g.

A.3.2.2 Ultraviolet visible spectrophotometer (1cm quartz cuvette).

A.3.3 Identification methods

A.3.3.1 Color reaction: Weigh 0.05 g of sample (accurate to 0.0001 mg), place it in a 10 mL glass tube, add 5 mL of water to dissolve, and add 1.0 mL of ammonium thiocyanate solution (A.3.1.1). The color of the solution should not change. Add 0.5 mL of hydrochloric acid solution (A.3.1.2), mix well and the solution should be dark red.

A.3.3.2 Ultraviolet absorption: Weigh 0.001 g of sample (accurate to 0.0001 g), dissolve with water and dilute to 50 mL, the concentration of this sample solution is 20 μg/mL. Measure with a 100 nm~700 nm scanning pattern and there should be maximum absorption at a wavelength of 256 ± 2 nm.

A.4 Determination of Iron Content

A.4.1 Methods summary

Under strong acidic conditions, the free Fe^{3+} reacts quantitatively with excess potassium iodide to form and precipitate I_2. The precipitated I_2 is titrated with a sodium thiosulfate standard titration solution. Calculate the iron content in the sample based on the titration consumption of the standard sodium thiosulfate 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 (10g/L): Accurately weigh 0.5g of soluble starch and add a small amount of water to make a paste. Pour in 50mL of boiling water while stirring, boil again, stir well and cool (prepare on site).

A.4.3 Instruments and equipment

Balance: the sensitivity is 0.0001g.

A.4.4 Analysis steps

Weigh 0.5 g of sample (accurate to 0.0001 g), dissolve it in 40 mL of water in an iodine measuring flask, and 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 with water and place it in a dark place for about 5 minutes. Remove and rinse the lid with a small amount of water. Titrate with standard sodium thiosulfate titration solution (A.4.2.3) close to end point (light yellow), add 2.0 mL of starch indicator solution (A.4.2.4) and continue titrating until the blue color disappears.

While testing the sample, titrate the blank solution without sample with a standard sodium thiosulfate titration solution.

A.4.5 Calculation of Results

The mass fraction w_1 of iron, expressed in%, shall be calculated with formula (A.1):

$$w_1 = \frac{(V_1 - V_2) \times c_1 \times M_1}{m_1 \times 1000} \times 100 \qquad \text{(A.1)}$$

Where:

V_1 - consumed volume of standard sodium thiosulfate titration solution (A.4.2.3) by the sample, in milliliters (mL),

V_2 - consumed volume of standard sodium thiosulfate titration solution by the blank test (A.4.2.3), in milliliters (mL),

c_1 - concentration of standard sodium thiosulfate titration solution, in mol/L,

M_1 - molar mass of iron, in grams per mole (g/mol) (M1=55.85),

m_1 - mass of the sample, in grams (g),

1,000 - conversion factor.

The arithmetic mean of the two parallel determination results shall be taken as the determination result. Under repeatable conditions, the absolute difference between the two parallel measurement results shall not exceed 2% of the arithmetic mean.

A.5 Determination of EDTA content

A.5.1 Method summary

After masking $Fe^{3+}$with triethanolamine, in an alkaline solution with a pH of 12.5 to 13.0, titrate sodium iron EDTA with Ca^{2+} to form a stable complex with EDTA. The excess Ca^{2+} is then combined with a hydroxynaphthol blue indicator to form a complex. The titration end point is wine red. The content of EDTA is calculated from the consumption of Ca^{2+} titrant.

A.5.2 Reagents and materials

A.5.2.1 Calcium acetate monohydrate.

A.5.2.2 EDTA: control reagent.

A.5.2.3 Hydroxy naphthol blue indicator: according to hydroxy naphthol blue: sodium chloride = 1:100, which are mixed, ground and prepared.

A.5.2.4 Sodium hydroxide solution (500 g/L): Accurately weigh 50 g of sodium hydroxide, dissolve with water to a constant volume of 100 mL.

A.5.2.5 Triethanolamine.

A.5.2.6 Calcium acetate monohydrate titration solution (0.25 mol/L): Weigh 44.0g of calcium acetate monohydrate (accurate to 0.0001g), add water to dissolve, transfer to a 1,000mL volumetric flask and bring volume to the scale.

A.5.3 Instruments and equipment

A.5.3.1 Balance: shall be with a sensitivity of 0.0001 g.

A.5.3.2 pH meter.

A.5.4 Analysis steps

A.5.4.1 Calibration of standard calcium acetate monohydrate titration solution: weigh 2.0 g to 2.1 g of ethylenediaminetetraacetic acid (A.5.2.2) (accurate to 0.0001 g), place it in a 250 mL conical flask, and add 150 mL of water. Then dissolve it with sodium hydroxide solution (A.5.2.4) and adjust the pH to 11.0 to 12.0. Add 30 mg of hydroxynaphthol blue indicator (A.5.2.3) and titrate with standard calcium acetate monohydrate titration solution (A.5.2.6) until the solution changes from blue to red.

A.5.4.2 Titration operation: Weigh 0.8 g to 1.0 g of sample (accurate to 0.0001 g), place it in a 250 mL conical flask and add 75 mL of distilled water to dissolve it. Adjust the pH of the solution to 9.0 with triethanolamine (A.5.2.5) to mask Fe^{3+} and adjust the pH of the sample solution to 12.5~13.0 with sodium hydroxide solution (A.5.2.4) to make the solution colorless and clear. Add 30 mg of hydroxynaphthol blue indicator (A.5.2.3) and titrate with a calibrated calcium acetate monohydrate solution until the solution changes from blue to red.

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

A.5.5 Calculation of results

Molar concentration C_2 of standard calcium acetate monohydrate titration solution, expressed in mol/L, is calculated according to formula (A.2):

$$c_2 = \frac{m_2 \times 1000}{V_3 \times M_2} \quad \text{(A.2)}$$

Where:

m_2 - mass of m^2 ethylenediaminetetraacetic acid, in grams (g),

V_3 - consumed volume of standard calcium acetate monohydrate titration solution for calibration, in milliliters (mL),

M_2 - molar mass of ethylenediaminetetraacetic acid, expressed in grams per mole (g/mol) (M_2=292.24),

The mass fraction of ethylenediaminetetraacetic acid is expressed in % and calculated according to formula (A.3):

$$w_2 = \frac{(V_4 - V_5) \times c_2 \times M_2}{m_3 \times 1000} \times 100 \quad \text{(A.3)}$$

Where:

V_4 - consumed volume of standard calcium acetate monohydrate titration solution by the sample, in milliliters (mL),

V_5 - consumed volume of standard calcium acetate monohydrate titration solution in the blank test, in milliliters (mL),

c_2 - molar concentration of standard calcium acetate monohydrate titration solution, in mol/L,

M_2 - molar mass of ethylenediaminetetraacetic acid, expressed in grams per mole (g/mol) (M_2=292.24),

m_3 - mass of the sample, in grams (g),

1,000 - conversion factor.

The arithmetic mean of the two parallel determination results shall be taken as the determination result. Under repeatable conditions, the absolute difference between the two parallel determination results shall not exceed 2% of the arithmetic mean.

A.6 Determination of free iron

A.6.1 Method summary

The non-chelated Fe^{3+} in the sodium iron EDTA sample reacts with sodium catechol-3,5-disulfonate and its absorbance is measured at a wavelength of 670 nm to calculate the content of free iron. A limit experiment is conducted for comparison.

A.6.2 Reagents and materials

A.6.2.1 Sodium catechol-3,5-disulfonate.

A.6.2.2 Standard iron solution: dilute with water to 0.025 mg of iron per mL after preparation according to GB/T 602.

A.6.2.3 Color developing agent: accurately weigh 1.0 g of sodium catechol-3,5-disulfonate, and add 100 mL of water to dissolve.

A.6.3 Instruments and equipment

A.6.3.1 Balance: shall be with a sensitivity of 0.0001g.

A.6.3.2 Spectrophotometer.

A.6.4 Analysis steps

Weigh 0.2 g of sample (accurate to 0.0001 g) and add water to dissolve to a constant volume of 20 mL. Take three test tubes and label the tubes as A, B and C respectively. Add 5 mL of sample solution to tube A and tube B, and add 4 mL of water and 1 mL of standard iron solution to tube C. Add 1 mL of chromogenic agent to tube A and tube C, and 1 mL of water to tube B. Measure the absorbance of solution in tube A at a wavelength of 670 nm using solution in tube B as control solution. Measure the absorbance of solution in tube C with water as a reference (see Table 3).

Table 3: Reagent Adding Amount

Name	Tube A	Tube B	Tube C
Solution	5 mL	5 mL	-
Water	-	1 mL	4 mL
Standard iron solution	-	-	1 mL
Chromogenic agent	1 mL	-	1 mL

A.6.5 Result determination

The absorbance of sample solution in tube A is not greater than that of solution in tube C, that is, the content of free iron in the sample does not exceed 0.05%.

A.7 Determination of aminotriacetic acid

A.7.1 Method summary

Under alkaline conditions, dissolve the aminotriacetic acid in the sample in copper nitrate solution to a constant volume and separate it by high-performance liquid chromatography, and test by ultraviolet detector. Qualitative analysis is performed by adding standard solution to the sample and the limit is determined.

A.7.2 Reagents and materials

A.7.2.1 Methanol: chromatographical pure

A.7.2.2 Tetrabutylammonium hydroxide - methanol solution (TBAOH): 25% (w/w).

A.7.2.3 Ammonia (5%): measure 0.5 mL of ammonia and dilute to 10 mL with water.

A.7.2.4 Copper nitrate solution (10 g/L): accurately weigh 10.0 g of copper nitrate and dissolve it with water to the constant volume of 1,000 mL.

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

A.7.2.6 Standard aminotriacetic acid sample: CAS No.: 139-13-9.

A.7.2.7 Standard stock solution of aminotriacetic acid (10 mg/mL): weigh 100 mg of aminotriacetic acid (accurate to 0.0001 g), transfer to a 10 mL volumetric flask and dissolve with water. Add 0.5 mL of ammonia, mix and bring volume to the scale with water.

A.7.2.8 Standard aminotriacetic acid solution (10 mg/L): weigh 1 g of sample (accurate to 0.0001 g) and place it in a 100 mL volumetric flask. Add 100 µL of standard stock solution (A.7.2.6), dilute to scale with copper nitrate solution (A.7.2.4) and shake well. The standard solution is obtained by ultrasonic treatment.

A.7.3 Instruments and equipment

A.7.3.1 Balance: with a sensitivity of 0.0001g.

A.7.3.2 High performance liquid chromatography: equipped with ultraviolet detector

A.7.3.3 pH meter

A.7.4 Analysis steps

A.7.4.1 Sample preparation

Weigh 1 g of sample (accurate to 0.0001 g), add 100 µ L of ammonia aqueous solution (A.7.2.3) and add 50 mL of copper nitrate solution (A.7.2.4). Perform ultrasonic treatment and transfer to a 100 mL volumetric flask. Finally, dilute volume to the scale with copper nitrate solution (A.7.2.4), shake well and obtain the sample solution.

A.7.4.2 Chromatographic reference conditions

Pre column: C_8 chromatographic column (4.6 mmx10 mm, 5 µm).

Mobile phase: measure 10 mL of 25% tetrabutylammonium hydroxide - methanol solution (A.7.2.2) in 200 mL of water and adjust the pH to 7.5 ± 0.1 with 1 mol/L phosphoric acid. Transfer the solution to a 1,000 mL volumetric flask and add 90 mL of methanol. Dilute volume to the scale with water and shake well. Filter with a 0.45 µm filter membrane, degass and put aside.

Chromatographic column: C_{18} chromatographic column (4.6 mmx150 mm, 5 µm) or other equivalent chromatographic column.

Detection wavelength: 244 nm.

Injection volume: 20 µL.

Flow rate: 2.0 mL/minute.

A.7.4.3 Sample analysis

Perform chromatographic analysis of the standard solution and sample solution under the reference chromatographic conditions of A.7.4.2, record the chromatograms and calculate the response value of aminotriacetic acid. Repeat the testing of standard solution for three times and take the average response values. The relative standard deviation of the three tests is not more than 2.0%. The resolution between aminotriacetic acid and sodium iron EDTA is not less than 4.0. The standard chromatogram of aminotriacetic acid is as shown in Appendix B.

A.7.5 Result determination

The response value of the chromatographic peak of aminotriacetic acid in the sample solution shall not exceed the difference between the response values of the chromatographic peak of aminotriacetic acid obtained from the standard solution and the sample solution (i.e., the content of aminotriacetic acid in the sample is not more than 0.1%).

Detection limit: the minimum detection limit for the determination of aminotriacetic acid in sodium iron EDTA using this method is 20 mg/kg.

A.8 Determination of chloride (calculated as Cl)

A.8.1 Method summary

In an acidic medium, using a silver electrode as the measuring electrode and a calomel electrode as the reference electrode, titrate with a standard silver nitrate titration solution and determine the reaction endpoint by means of potential jump.

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: 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: with a sensitivity of 0.0001g.

A.8.3.2 Potentiometer: with an accuracy of 2mV/grid.

A.8.3.3 Reference electrode: double liquid-connected saturated calomel electrode, filled with saturated potassium chloride solution.

A.8.3.4 Measuring electrode: silver electrode.

A.8.4 Analysis steps

Weigh 5 g of the sample (accurate to 0.0001 g), dissolve it with water and add 1 drop of bromophenol blue indicator solution. Adjust the color of the solution to exactly yellow with sodium hydroxide solution or nitric acid solution and transfer it to a 100 mL volumetric flask. Dilute with water to volume of scale and shake well. This is solution A.

Accurately transfer 20 mL of the above solution A into a 50 mL beaker, titrate with standard silver nitrate titration solution and determine the titration end point using a silver reference electrode.

While testing the sample, titrate the blank solution without sample with a standard silver nitrate titration solution.

A.8.5 Calculation of Results

As for the mass fraction w_3 of chloride (calculated as Cl), expressed as a mass percentage, is calculated according to formula (A.4):

$$w_3(\%)=\frac{(V_6-V_7)\times c_3\times 0.03545\times 5}{m_4}\times 100 \qquad \text{(A.4)}$$

Where:

c_3 - concentration of standard silver nitrate titration solution, mol/L,

V_6 - consumed volume of standard silver nitrate titration solution in titration, in milliliters (mL),

V_7 - consumed volume of standard silver nitrate titration solution in blank titration, in milliliters (mL),

m_4 - value of sample mass, in grams (g),

5 - dilution ratio,

0.03545 - the mass of chloride in grams (calculated as Cl) equivalent to 1.00 mL of standard silver nitrate titration solution.

A.9 Determination of sulfate (calculated as SO_4)

A.9.1 Method summary

In an acidic medium, the sulfate ions and barium ions in the sample generate barium sulfate precipitation, which are compared with the standard sulfate solution processed by the same method and a limit experiment shall be carried out.

A.9.2 Reagents

A.9.2.1 Hydrochloric acid solution: 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 Standard sulfate solution: 2.113 mg/mL anhydrous sodium sulfate, with each mL equivalent to 1.430 mg sulfate.

A.9.2.6 Solution A: take 5 mL of barium chloride solution, add 55 mL of water and 20 mL of 95% ethanol solution to dilute and then add 0.5 mL of standard sulfate solution to mix well (prepare when needed).

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

A.9.3 Instruments and equipment

Balance: with a sensitivity of 0.01g.

A.9.4 Analysis steps

Weigh 1.8 g of sample (accurate to 0.01 g), add 30 mL of water to dissolve it and slowly add 4.5 mL of sodium hydroxide solution (A.9.2.2). Stir for 15 minutes, bring to a constant volume of 50 mL then filter it. Take 20 mL of filtrate as the sample solution and place it in a 50 mL colorimetric tube. Add two drops of the 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 bringing to a constant volume of 50 mL with water, add 5.0 mL of solution A (A.9.2.6) and mix well. Under a black background, when viewed axially, the turbidity of the sample solution is compared to that of the standard turbidimetric solution.

Standard turbidimetric solution: measure 20 mL of water and place it in a 50 mL colorimetric tube. At the same time and with the same process method of the sample solution to get a solution with a pH value of 2.0. Add 300 µl of standard sulfate solution (A.9.2.5) and bring it to a constant volume of 50 mL. Add 5.0 mL of solution A (A.9.2.6) and mix well.

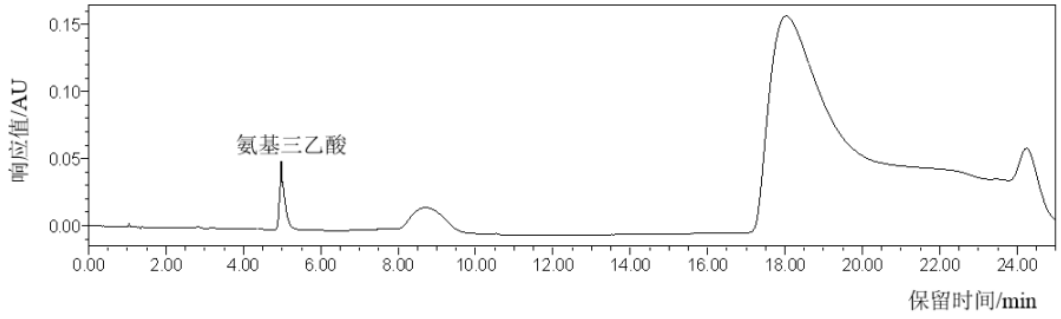
A.9.5 Result determination

The turbidity of the sample solution should not be deeper than the standard turbidimetric concentration, that is, the sulfate content in the sample should not be greater than 0.06%.

Appendix B

Standard Reference Chromatogram of Aminotriacetic Acid

Figure B.1: Standard Reference Chromatogram of Aminotriacetic Acid



氨基三乙酸: Aminotriacetic acid

保留时间: Retention time

Attachments:

No Attachments.