NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB 1886.314-2020

National food safety standard - Food additives - Edetate Calcium Disodium

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1 Scope

This Standard is applicable to food additive - edetate calcium disodium generated by chelating disodium edetate and calcium (usually calcium carbonate or natural shells).

2 Chemical Name, Molecular Formula, Structural

Formula and Relative Molecular Mass

2.1 Chemical Name

Edetate calcium disodium

2.2 Molecular Formula

C10H12CaN2Na2O8 ? 2H2O

2.4 Relative Molecular Mass

410.30 (in accordance with the international relative molecular mass of Year 2014)

3.1 Sensory Requirements

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

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 Method summary Use the flame test to identify the sodium ions in the sample; use the oxalic acid test to identify the calcium ions in the sample; the residual ethylene diamine tetra-acetic acid ions in edetate calcium disodium can make the iron chloride solution fade, so as to identify the ethylene diamine tetra-acetic acid ions. Infrared spectroscopy is a qualitative analysis of edetate calcium disodium in accordance with the absorption bands of the ionic groups of the constituent substances within the infrared range (mid-infrared: 400 cm-1 ~ 4,000 cm-1).

A.2.2 Reagents and materials A.2.2.1 Ammonium hydroxide solution (6 mol/L): measure-take 400 mL of 28% ammonium hydroxide solution; use water to dilute it to 1,000 mL.

A.2.2.2 Ammonium oxalate solution (0.5 mol/L): take 3.5 g of ammonium oxalate monohydrate; dissolve it in water to 100 mL.

A.2.2.3 Ammonium thiocyanate solution (0.02 g/mL): weigh-take 2 g of ammonium thiocyanate; dissolve it in water to 100 mL.

A.2.2.4 Hydrochloric acid solution (3 mol/L): measure-take 226 mL of hydrochloric acid;

use water to dilute it to 1,000 mL.

A.2.2.5 Ferric chloride solution (1 mol/L): weigh-take 9 g of ferric chloride hexahydrate;

dissolve it in water to 100 mL.

A.2.2.6 Methyl red solution: weigh-take 100 mg of methyl red; dissolve it in 100 mL of ethanol; if necessary, filter it.

A.2.3 Instruments and equipment Infrared spectrophotometer.

A.2.4 Identification method A.2.4.1 Weigh-take 0.2 g of sample; prepare a sample solution (50 mg/mL). Take platinum wire; use hydrochloric acid solution to moisten it, then, dip it in the sample solution. Burn it in a colorless flame; the flame is bright yellow.

A.2.4.2 Take 5 mL of the sample solution (50 mg/mL) in A.2.4.1; add 2 drops of methyl red solution as an indicator; use ammonium hydroxide solution to adjust it to neutral.

Then, use hydrochloric acid solution to titrate the sample solution to acidic; add ammonium oxalate solution. Thus, white precipitate - calcium oxalate is generated.

The calcium oxalate is insoluble in acetic acid, but soluble in hydrochloric acid. Use hydrochloric acid solution to moisten it, then, dip it in the sample solution. Burn it in a colorless flame; the flame is yellow-red.

A.2.4.3 Take 5 mL of water into a test tube. Add 2 drops of ammonium thiocyanate solution and 2 drops of ferric chloride solution to obtain a dark red solution. Then, add

50 mg of sample. After mixing, the dark red color disappears.

A.2.4.4 See Appendix B for the infrared spectrum of edetate calcium disodium.

A.3 Determination of Edetate Calcium Disodium (C10H12CaN2Na2O8 ? 2H2O)

Content A.3.1 Method summary Edetate calcium disodium is under acidic conditions. Take diphenyl-carbazone as an indicator; the mercury nitrate solution is titrated to purple-red as the end point. Use the amount of consumed mercury nitrate to calculate the edetate calcium disodium content.

A.3.2 Reagents and materials A.3.2.1 Mercury nitrate solution.

A.3.2.2 Nitric acid solution.

A.3.2.3 Ferric ammonium sulfate solution: weigh-take 8 g of ferric ammonium sulfate dodecahydrate; dissolve it in water to 100 mL.

A.3.2.4 Ammonium thiocyanate standard titration solution (0.1 mol/L): weigh-take 8 g of ammonium thiocyanate; dissolve it in water to 1,000 mL. In accordance with the requirements of GB/T 601, calibrate it.

A.3.2.5 6% acetic acid solution: take 60 mL of glacial acetic acid or 166 mL of acetic acid; use water to dilute to 1,000 mL.

V2---the volume of mercury nitrate solution consumed during the titration process, expressed in (mL);

0.4103---each mL of mercury nitrate (1 mol/L) is equivalent to 0.4103 g of this product (C10H12CaN2Na2O8 ? 2H2O);

w---the mass of the sample, expressed in (g).

The test result shall be subject to the arithmetic mean value of the parallel determination results.

A.4 Magnesium Chelate Transfer Test A.4.1 Method summary Under alkaline conditions, the residual ethylene diamine tetra-acetic acid free monomers in the sample preferentially forms stable complexes with magnesium ions.

When excessive magnesium ions are complexed with acidic chrome black T, a deep wine-red complex is formed.

A.4.2 Reagents and materials A.4.2.1 Concentrated ammonia: 28% ammonia.

A.4.2.2 Acid mordant black test solution: weigh-take about 200 mg of acidic chrome black T and 2 g of hydroxylamine hydrochloride; dissolve in methanol to 50 mL. Filter it, then, store in a light-shielded container; use it within 2 weeks.

A.4.2.3 Magnesium acetate solution (0.1 mol/L): accurately weigh-take 1.1 g of magnesium acetate tetrahydrate; dissolve it in water to 100 mL.

A.4.3 Analytical procedures A.4.3.1 Preparation of buffer solution Accurately weigh-take 67.5 g of ammonium chloride; dissolve it in 200 mL of water.

Then, add 570 mL of concentrated ammonia; add water to dilute to 1,000 mL.

A.4.3.2 Test Accurately weigh-take 1 g of sample; dissolve it in 5 mL of water. Add 5 mL of buffer solution, then, add 5 drops of the acid mordant black test solution. Use magnesium acetate solution to titrate it, till it manifests a dark wine-red color. The required amount of magnesium acetate solution is <= 2 mL.

A.5 Determination of Aminotriacetic Acid A.5.1 Method summary 10 ug/mL.

A.5.5.4 Preparation of sample solution Accurately weigh-take 1.0 g of the sample; transfer it to a 100 mL volumetric flask. Use copper nitrate solution to dilute it to the scale, then, mix it well. If necessary, perform ultrasonic dissolution.

A.5.5.5 Preparation of sample spiking solution Accurately weigh-take 1.0 g of the sample; transfer it to a 100 mL volumetric flask. Add 100 uL of standard stock solution. Use copper nitrate solution to dilute it to the scale, then, mix it well. If necessary, perform ultrasonic dissolution.

A.5.5.6 System adaptability test Repeatedly inject the standard solution into the chromatograph for 3 times; record the peak response value. The relative standard deviation of the three determination results of the standard sample is <= 2.0 %; the resolution between aminotriacetic acid and edetate calcium disodium is >= 4.0.

A.5.5.7 Determination Under the reference chromatographic conditions in A.5.4, respectively take an equal volume (20 uL) of the sample spiking solution and sample solution for chromatographic analysis. Record the chromatogram; calculate the response value of aminotriacetic acid.

A.5.6 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 sample spiking solution and the sample solution.