GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB 1886.235-2016

National Food Safety Standard -

Food Additives - citric acid

食品安全国家标准

食品添加剂 柠檬酸

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Issued by: National Health and Family Planning Commission of the PRC

Table of Contents

National Food Safety Standard - Food Additives - citric acid

1 Scope

This Standard is applicable to the food additives citric acid prepared by fermentation of starchy or sugary raw materials.

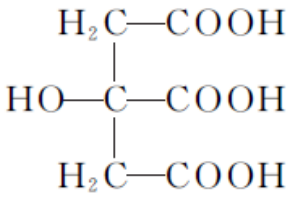
2 Molecular formulas, structural formulas, and relative molecular mass

2.1 Molecular formulas

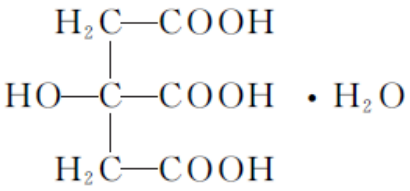
Citric acid anhydrous: $C_6H_8O_7$

Citric acid monohydrate: $C_6H_8O_7 \cdot H_2O$

2.2 Structural formulas



Citric acid anhydrous



Citric acid monohydrate

2.3 Relative molecular mass

Citric acid anhydrous: 192.13 (according to the 2013 international relative atomic mass)

Citric acid monohydrate: 210.14 (according to the 2013 international relative atomic mass)

3 Technical requirements

3.1 Sensory requirements

Appendix A

Inspection methods

A.1 Safety instruction

Some of the reagents used in the inspection methods of this Standard are toxic or corrosive. During operation, it shall take appropriate safety and protective measures.

A.2 General provisions

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

A.3 Identification test

A.3.1 Reagents and materials

A.3.1.1 Ethyl alcohol.

A.3.1.2 Diethyl ether.

A.3.1.3 Sulfuric acid solution: MEASURE and TAKE 29 mL of sulfuric acid; slowly INJECT it into approximately 700 mL of water; COOL and DILUTE to 1000 mL.

A.3.1.4 Mercury sulfate solution: WEIGH 5 g of mercury oxide; ADD 40 mL of water; slowly ADD 20 mL of sulfuric acid; STIR while adding; and ADD 40 mL of water to dissolve it.

A.3.1.5 Potassium permanganate solution: $c(\frac{1}{5}KMnO_4)$=0.1 mol/L.

A.3.1.6 Sodium hydroxide solution: 50 g/L.

A.3.1.7 Pyridine-acetic anhydride solution: MIX pyridine and acetic anhydride at 3:1 volume ratio.

A.3.2 Analytical procedures

A.3.2.1 The test piece is freely soluble in water, soluble in ethyl alcohol, slightly soluble in diethyl ether.

A.3.2.2 TAKE an appropriate amount of test piece in 25 mL enamel; IGNITE, it slowly decomposes; but caramel odor shall not be produced.

A.3.2.3 WEIGH 50 mg of the test piece; after dissolving it in water, DILUTE to 10 mL. PIPETTE 2 mL in 25 mL colorimetric tube; USE sodium hydroxide solution to adjust to neutral; ADD 1 drop of sulfuric acid solution and HEAT to boiling; ADD 1 drop of potassium permanganate solution; SHAKE well, purple fades; and ADD 1 drop of mercury sulfate solution, white precipitate is produced.

A.3.2.4 WEIGH 5 mg of the test piece in 25 mL colorimetric tube; ADD about 5 mL of pyridine-acetic anhydride solution; SHAKE, and a yellow to red or purple red solution is generated.

A.4 Determination of citric acid content

A.4.1 Method summary

In an aqueous medium, USE phenolphthalein as the indicator solution, and USE the standard titration solution of sodium hydroxide to titrate citric acid solution. According to the amount of the standard titration solution of sodium hydroxide consumed, the content of citric acid is calculated.

A.4.2 Reagents and materials

A.4.2.1 Standard titration solution of sodium hydroxide: c(NaOH)=0.5 mol/L.

A.4.2.2 Phenolphthalein indicator solution: 10 g/L.

A.4.2.3 Carbon dioxide-free water.

A.4.3 Analytical procedures

WEIGH 1 g of the test piece, accurate to 0.0001 g; PLACE it in a 150 mL conical flask; ADD 50 mL of carbon dioxide-free water to dissolve; ADD 3 drops of phenolphthalein indicator solution; and USE standard titration solution of sodium hydroxide to titrate to pink as the end-point. At the same time, CONDUCT blank test.

A.4.4 Result calculation

The mass fraction w_1 of citric acid content (calculated in citric acid anhydrous) in citric acid monohydrate is calculated according to the equation (A.1). The mass fraction w_2 of citric acid content in citric acid anhydrous is calculated according to the equation (A.2).

A.8.1 Method summary

Under acidic conditions, the sulfate ion in citric acid solution and barium chloride solution generate barium sulfate precipitation. COMPARE it with the standard solution treated in the same method; and PERFORM a limit test.

A.8.2 Reagents and materials

A.8.2.1 Hydrochloric acid solution: 6 mol/L. PIPETTE 54 mL of hydrochloric acid; DILUTE to 100 mL.

A.8.2.2 Barium chloride solution: 250 g/L.

A.8.2.3 Acetic acid solution: MEASURE and TAKE 298 mL of acetic acid; DILUTE to 1000 mL.

A.8.2.4 Ethyl alcohol solution: MEASURE and TAKE 300 mL of absolute ethyl alcohol; USE water to dilute to 1000 mL.

A.8.2.5 Standard stock solution of sulfate: $c(SO_4)$ = 0.1 g/L.

A.8.2.6 Standard solution I of sulfate: $c(SO_4)$ = 0.01 g/L. PIPETTE 10 mL of standard stock solution of sulfate; USE ethyl alcohol solution to dilute to 100 mL.

A.8.2.7 Standard solution II of sulfate: $c(SO_4)$ = 0.015 g/L. PIPETTE 15 mL of standard stock solution of sulfate; USE ethyl alcohol solution to dilute to 100 mL.

A.8.3 Analytical procedures

WEIGH 1 g of test piece, accurate to 0.01 g; PLACE it in a 50 mL colorimetric tube with stopper; ADD 15 mL of water to dissolve; ADD 1 mL of barium chloride solution, and 1 mL of standard solution of sulfate; SHAKE, LET it stand for 1 min; ADD 1 mL of hydrochloric acid solution and 0.5 mL of acetic acid solution; SHAKE well, LET it stand for 5 min. PIPETTE 10 mL of standard solution of sulfate in a 50 mL colorimetric tube with stopper; ADD 5 mL of water; and starting from “ADD 1 mL of barium chloride solution”, TREAT it the same way as the test piece. The turbidity of the test piece tube shall not be deeper than that of the standard tube.

Note: Standard solution I of sulfate for citric acid anhydrous; standard solution II of sulfate for citric acid monohydrate.

A.9 Determination of chloride

A.9.1 Method summary

A.10.2.5 Standard solution of oxalic acid: 0.01 g/L. WEIGH 0.175 g of oxalic acid ($C_2H_2O_4 \cdot 2H_2O$); USE water to dissolve and dilute to 500 mL. PIPETTE 4 mL of this solution; and USE water to dilute to 100 mL.

A.10.3 Analytical procedures

WEIGH 0.4 g (accurate to 0.01 g) of the test piece in a 25mL colorimetric tube with stopper; ADD 4 mL of water, 3 mL of hydrochloric acid, and 1 g of zinc granule; BOIL it for 1 min; PLACE it for 2 min; TRANSFER it into a test tube containing 0.25 mL of phenylhydrazine hydrochloride solution; HEAT to boiling; quickly COOL; POUR into a 25 mL colorimetric tube with stopper; ADD 0.25 mL of equal volume of hydrochloric acid and potassium ferricyanide solution; SHAKE; and PLACE for 30 min. TAKE 4 mL of standard solution of oxalic acid; and starting from “3 mL of hydrochloric acid and 1 g of zinc granule”, TREAT it the same way as the test piece. The pink generated in the test piece tube shall not be deeper than that in the standard tube.

A.11 Determination of calcium salt

A.11.1 Method summary

In acetic acid medium, trace calcium ions and oxalate ions form insoluble calcium oxalate suspensions. COMPARE it with the standard solution treated in the same method; and PERFORM a limit test.

A.11.2 Reagents and materials

A.11.2.1 Acetic acid solution: 2 mol/L. MEASURE and TAKE 118 mL of glacial acetic acid; USE water to dilute to 1000 mL.

A.11.2.2 Ammonium oxalate solution: 40 g/L.

A.11.2.3 Standard solution of calcium: c(Ca)=0.01 g/L. WEIGH 2.5 g of the calcium carbonate which is baked at 105 °C ~ 110 °C to a constant mass; ADD 12 mL of 6 mol/L acetic acid (MEASURE and TAKE 354 mL of glacial acetic acid; USE water to dilute to 1000 mL) to dissolve; ADD water to dilute to 1000 mL. PIPETTE 1 mL of this solution; and ADD water to dilute to 100 mL.

A.11.3 Analytical procedures

WEIGH 0.5 g (accurate to 0.01 g) of the test piece; PLACE it in a 25 mL colorimetric tube; ADD 15 mL of water to dissolve; ADD 1 mL of ammonium oxalate solution and 1 mL of acetic acid solution; SHAKE, PLACE it for 15 min. TAKE 10 mL of standard solution of calcium in a 25 mL colorimetric tube; ADD 5 mL of water; and starting from “ADD 1 mL of ammonium oxalate solution”, TREAT it the same way as the test piece. The turbidity of the test piece tube

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