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Report Name: Draft National Food Safety Standard for Food additive L-malic acid Notified to WTO

Country: China - People's Republic of

Post: Beijing

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

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Report Highlights:

On July 11, 2024, China notified a new National Food Safety Standard for Food additive L-malic acid to the World Trade Organization (WTO) under G/SPS/N/CHN/1305. China's SPS Enquiry Point at sps@customs.gov.cn will accept comments until September 9, 2024. This report provides an unofficial translation of the draft standard. Stakeholders should conduct their own review of the standard and provide comments as necessary.

Report Summary:

On July 11, 2024, China notified draft National Food Safety Standard Code of Practice for Food additive L-malic acid to the World Trade Organization (WTO) under [G/SPS/N/CHN/1305](#). China's SPS Enquiry Point at sps@customs.gov.cn will accept comments until September 9, 2024.

This standard replaces GB 1886.40-2015 "National Food Safety Standard - Food Additives - L-Malic Acid". It is applicable to L-malic acid, a food additive prepared by enzymatic engineering using fumaric acid or fumarate salts as raw materials, or by fermentation using starch or sugar as raw materials. It added physical and chemical indicators such as moisture and succinic acid; revised the description of physical and chemical indicators content and total arsenic, and removed heavy metals; updated the testing methods for identification, content, clarity, fumaric acid, maleic acid, lead, and total arsenic; and added testing method for succinic acid.

This report provides an unofficial translation of the draft notified standard. Stakeholders should conduct their own review of the standard.

BEGIN TRANSLATION

Preface

This standard replaces GB 1886.40-2015 "National Food Safety Standard - Food Additives - L-Malic Acid".

Compared with GB 1886.40-2015, the main changes in this standard are as follows:

- Modified the scope;

- Added physical and chemical indicators such as moisture and succinic acid;

- Revised the description of physical and chemical indicators content and total arsenic, and removed heavy metals;

- Updated the testing methods for identification, content, clarity, fumaric acid, maleic acid, lead, and total arsenic;

- Added testing method for succinic acid;

- Modified Appendix A and Appendix B, and added Appendix C.

National Food Safety Standard

Food Additives L-malic acid

1 Scope

This standard is applicable to L-malic acid, a food additive prepared by enzymatic engineering using fumaric acid or fumarate salts as raw materials, or by fermentation using starch or sugar as raw materials.

2 Chemical name, molecular formula, structural formula and relative molecular mass

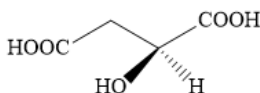
2.1 Chemical name

L-Hydroxysuccinic acid

2.2 Molecular formula

$C_4H_6O_5$

2.3 Structural formula



2.4 Relative molecular mass

134.09 (according to 2022 international relative atomic mass)

3 Technical Requirements

3.1 Sensory Requirements

Sensory requirements shall conform to the provisions in Table 1.

Table 1 Sensory Requirements

Items	Requirement	Analysis method
Color	White	Take an appropriate amount of the sample and place it on a clean, dry white porcelain plate. Under natural light, observe its color and state, and smell its odor.
State	Crystalline or crystalline powder	
Smell	Special sour taste	

3.2 Physical and Chemical Indices

It shall conform to the provisions in Table 2.

Table 2 Physical and Chemical Indicators

Items	Index	Analytical method
Content (calculated as $C_4H_6O_5$), w/%	\geq 99.0	A.4 in Appendix A
Specific rotation angle $\alpha_m(20)$	-1.6~-2.6	A.5 in Appendix A

Items		Index	Analytical method
$^{\circ}\text{C}, \text{D})/[(^{\circ})\cdot\text{dm}^2\cdot\text{kg}^{-1}]$			
Clarity		By experimentation	A.6 in Appendix A
Moisture, w/%	\leq	1.0	GB 5009.3 Karl Fischer Method
Ignition residue, w/%	\leq	0.10	A.7 in Appendix A
Chloride (in Cl), w /%	\leq	0.004	A.8 in Appendix A
Sulfate (in SO_4), w /%	\leq	0.02	A.9 in Appendix A
Fumaric acid, w/%	\leq	0.5	A.10 in Appendix A
Maleic acid, w/%	\leq	0.05	A.10 in Appendix A
Succinic acid, w/%	\leq	2.0	A.10 in Appendix A
Lead (Pb)/(mg/kg)	\leq	2.0	GB 5009.12 or GB 5009.75
Total arsenic (in As)/(mg/kg)	\leq	1.0	GB 5009.11 or GB 5009.76

Appendix A

Inspection method

A.1 Warning

Some of the testing procedures specified in the testing method may lead to dangerous situations. The operator should take appropriate safety and health measures.

A.2 General provisions

The reagents used in this standard refer to analytical grade reagents unless otherwise specified. The standard titration solution, impurity determination standard solution, formulation, and product used in the experiment shall be prepared in accordance with the provisions of GB/T 601, GB/T 602, and GB/T 603 unless otherwise specified. The solution used in the experiment refers to Grade III water as specified in GB/T 6682, unless otherwise specified.

A.3 Identification test

A.3.1 Reagents and materials

A. 3.1.1 Ammonia solution (2+3): Take 30 mL of water and slowly pour it into 20 mL of ammonia solution, mix well.

A. 3.1.2 Aminobenzenesulfonic acid solution: 10 g/L

A. 3.1.3 Sodium nitrite solution: 200 g/L

A. 3.1.4 Sodium hydroxide solution: 40 g/L

A.3.2 Analysis steps

A. 3.2.1 Color identification of ammonium salts

Weigh 0.5 g of the sample to the nearest 0.01 g, place it in a 50 mL test tube, and add 10 mL of aqueous solution. Neutralize with ammonia solution until neutral, add 1 mL of p-aminobenzenesulfonic acid solution, and heat in a boiling water bath for 5 minutes. Add 5 mL of sodium nitrite solution and heat it in a boiling water bath for 3 min. Then add 5 mL of sodium hydroxide solution and observe from top to bottom. The sample solution should immediately turn red.

A. 3.2.2 Infrared absorption spectroscopy identification

According to GB/T 6040, the potassium bromide tablet method is used to determine the infrared absorption spectrum. The infrared absorption spectrum of the sample should be basically consistent with Appendix B.

A.4 Determination of content (calculated as C₄H₆O₅)

A.4.1 Method summary

Using phenolphthalein as an indicator, titrate the sample solution with sodium hydroxide standard titration solution, and calculate the total acid concentration in the sample solution (calculated as C₄H₆O₅) based on the consumption of sodium hydroxide standard titration solution.

A.4.2 Reagents and materials

A. 4.2.1 Sodium hydroxide standard titration solution: $c(\text{NaOH})=0.5 \text{ mol/L}$.

A. 4.2.2 Phenolphthalein indicator solution: 10 g/L

A.4.3 Instruments and equipment

Analytical balance: with a sensitivity of 0.0001 g .

A.4.4 Analysis steps

Weigh about 1.0g of the sample, accurate to 0.0001g , and dissolve it in 20 mL water that is free of carbon dioxide; add 2 drops of phenolphthalein indicator solution, titrate with sodium hydroxide standard titration solution until it turns slightly red, and when it keeps for 30s without fading, it will take as the endpoint. Perform a blank test using the same measurement steps as the sample.

A.4.5 Calculation of results

The mass fraction ω_1 of the content (calculated as $\text{C}_4\text{H}_6\text{O}_5$) is calculated according to equation (A.1).

$$\omega_1 = \frac{(V_1 - V_0) \times c \times M}{m \times 1000} \times 100\% \dots\dots\dots (A.1)$$

In which:

V_1 - the volume of sodium hydroxide standard titration solution consumed by the sample solution, in milliliters (mL);

V_0 - the volume of sodium hydroxide standard titration solution consumed by the blank solution, in milliliters (mL);

c - the concentration of standard sodium hydroxide titration solution, in mole per liter (mol/L);

M - the molar mass of 1/2 L-malic acid, in grams per mole (g/mol) ($M=67.04$);

m - the mass of the sample, in grams (g);

1000- Conversion factor.

The experimental results are expressed as the arithmetic mean of parallel measurement results, and the calculated results are kept to two decimal places. The absolute difference between two independent measurement results obtained under repetitive conditions shall not exceed 0.2%.

A.5 Determination of specific rotation

A.5.1 Instruments and equipment

A. 5.1.1 Polarimeter: Equipped with a sodium lamp (sodium spectrum D line 589.3 nm), with an accuracy of $\pm 0.01^\circ$.

A. 5.1.2 Analytical balance: with a sensitivity of 0.01 g .

A.5.2 Analysis steps

Weigh 4.25 g of the sample to the nearest 0.01 g, dissolve it in 20mL of water and make up to 50mL, and follow the procedures specified in GB/T 613 for the remaining steps.

A.5.3 Calculation of results

The specific rotation is measured as $\alpha_m(20\text{ }^\circ\text{C, D})$ and expressed as $(^\circ)\cdot\text{dm}^2\cdot\text{kg}^{-1}$, calculated according to equation (A.2).

$$\alpha_m = \frac{\alpha}{l \times \rho} \dots\dots\dots (A.2)$$

In which:

α - the value of the optical rotation of the sample solution measured at 20 °C, in degrees (°);

L - the numerical value of the length of the polarizing tube, in decimeters (dm);

ρ - the mass concentration of effective components in the sample in the solution, in grams per milliliter (g/mL).

A. 6 Determination of clarity

A.6.1 Reagents and materials

A. 6.1.1 Nitric acid solution (1+2): Mix nitric acid and water evenly in a volume ratio of 1:2.

A.6.1.2 Silver nitrate solution: 20 g/L.

A. 6.1.3 Dextrin solution: 20 g/L.

A. 6.1.4 Hydrochloric acid standard solution: $c(\text{HCl})=0.1\text{ mol/L}$.

A. 6.1.5 Standard working solution (containing 0.01 mg/mL chlorine): Accurately transfer 14.1 mL of hydrochloric acid standard solution, dilute with water, and make up to 50 mL. Accurately measure 10.0 mL of the solution, dilute with water, and bring to a volume of 1000 mL.

A.6.2 Instruments and equipment

A. 6.2.1 Nessler's colorimetric tube.

A. 6.2.2 Analytical balance: with a sensitivity of 0.01 g

A.6.3 Analysis steps

A.6.3.1 Preparation of sample solution

Weigh 1.0 g sample with accuracy to 0.01g, put it in a Nessler colorimetric tube, and add 20mL water to dissolve and shake well.

A.6.3.2 Preparation of standard reference solution

Accurately measure 0.20 mL of standard working solution, add water to 20 mL, and add 1 mL of nitric acid solution, 1 mL of silver nitrate solution, and 0.2 mL of dextrin solution; shake well, and place in the dark for 15min.

A.6.4 Result determination

Observe axially and laterally in the absence of direct sunlight. If the turbidity of the sample solution is not greater than that of the standard control solution, it is considered to have passed the test.

A.7 Burning residue

Weigh 2.5g of the sample to the nearest 0.0001g, and follow the provisions of GB/T 9741 for the rest. The experimental results shall be based on the arithmetic mean of the parallel measurement results, and the absolute difference between the two independent measurement results obtained under repeatability conditions shall not exceed 0.02%.

A.8 Determination of chloride

According to GB/T 9729, weigh 1.0 g of the sample to the nearest 0.01 g, and accurately transfer 0.4 mL of chloride standard solution. The turbidity of the sample solution should not exceed that of the standard solution, that is, the chloride content should be $\leq 0.004\%$.

A.9 Determination of sulfate

A.9.1 Reagents and materials

A. 9.1.1 Hydrochloric acid solution (1+4): Mix Hydrochloric acid and water evenly in a volume ratio of 1:4.

A.9.1.2 Barium chloride solution: 250 g/L

A. 9.1.3 Sulfate (SO₄) standard solution: 0.1 mg/mL.

A.9.2 Instruments and equipment

A. 9.2.1 Nessler's colorimetric tube.

A. 9.2.2 Analytical balance: with a sensitivity of 0.01 g.

A.9.3 Analysis steps

A.9.3.1 Preparation of sample solution

Weigh 1.0 g of the sample, accurate to 0.01 g, and place it in a 50 mL Nessler's colorimetric tube. Dissolve in water to make about 40 mL and shake well.

A.9.3.2 Preparation of control solution

Accurately transfer 2.0mL of sulfate standard solution and place it in a 50 mL Nessler's colorimetric tube. Dissolve in water to about 40 mL and shake well.

A.9.3.3 Determination

Add 0.5 mL hydrochloric acid solution and 1 mL barium chloride solution to the sample solution and control solution respectively, dilute with water to 50 mL, shake well, and set aside for 10 min.

A.9.4 Result determination

Place both on a black background and observe from above the colorimetric tube downwards to compare the resulting turbidity. The turbidity of the sample solution is not greater than that of the control solution, that is, the sulfate content is $\leq 0.02\%$.

A.10 Determination of fumaric acid, maleic acid, and succinic acid

A.10.1 Principle

After processing, the sample is separated using a hydrogen cation exchange chromatography column, detected using a UV detector or diode array detector, and quantified using an external standard method.

A.10.2 Reagents and materials

A.10.2.1 Water: GB/T 6682, Grade I water.

A.10.2.2 Sulfuric acid solution: $c(1/2 \text{H}_2\text{SO}_4)=0.01 \text{ mol/L}$.

A.10.2.3 Fumaric acid standard substance ($\text{C}_4\text{H}_4\text{O}_4$, CAS No.: 110-17-8): a standard substance with a purity of $\geq 99.0\%$, or a standard substance certified by the state and granted a standard substance certificate.

A.10.2.4 Maleic acid standard substance ($\text{C}_4\text{H}_4\text{O}_4$, CAS number: <https://www.chemsrc.com/baike/340366.html> 110-16-7): with a purity of $\geq 99.0\%$, or a standard substance certified by the state and granted a standard substance certificate.

A.10.2.5 Succinic acid standard substance ($\text{C}_4\text{H}_6\text{O}_4$, CAS number: <https://www.chemsrc.com/baike/340366.html> 110-15-6): a standard substance with a purity of $\geq 99.0\%$, or a standard substance certified by the state and granted a standard substance certificate.

A.10.2.6 Fumaric acid standard solution ($500 \mu\text{g/mL}$): Accurately weigh 50 mg of fumaric acid standard, accurate to 0.0001 g, dissolve in sulfuric acid solution and make up to 100 mL. Store in a refrigerator at 4°C with a shelf life of 6 months.

A.10.2.7 Maleic acid standard solution ($50 \mu\text{g/mL}$): Accurately weigh 5 mg of maleic acid standard, accurate to 0.0001 g, dissolve in sulfuric acid solution and make up to 100 mL. Store in a refrigerator at 4°C with a shelf life of 6 months.

A.10.2.8 Succinic acid standard solution (5 mg/mL): Accurately weigh 500 mg of succinic acid standard, accurate to 0.0001g, dissolve in sulfuric acid solution and make up to 100 mL. Store in a refrigerator at 4°C with a shelf life of 6 months.

A.10.2.9 Microporous membrane: $0.22 \mu\text{m}$, aqueous phase.

A.10.3 Instruments and equipment

A.10.2.1 High performance liquid chromatography: equipped with ultraviolet detector or diode array detector.

A.10.3.2 Analytical Balance: with a sensitivity of 0.01 g, and 0.0001 g

A.10.4 Instrument reference conditions

A.10.4.1 Chromatographic column: A hydrogen cation exchange chromatographic column packed with polystyrene divinylbenzene resin, $300 \text{ mm} \times 7.8 \text{ mm}$, or other chromatographic columns with equivalent analytical performance.

A.10.4.2 Column temperature: 45°C .

A.10.4.3 Mobile phase: sulfuric acid solution.

A.10.4.4 Flow rate: 0.5 mL/min.

A.10.4.5 Injection volume: 10 μ L.

A.10.4.6 Detection wavelength: 214 nm.

A.10.5 Analysis steps

A.10.5.1 Preparation of mixed standard solution

Take five 100 mL volumetric flasks and accurately aspirate 1.00 mL, 2.00 mL, 4.00 mL, 5.00 mL, and 6.00 mL of fumaric acid and maleic acid standard solutions, and 0.40 mL, 1.00 mL, 2.00 mL, 3.00 mL, and 4.00 mL of succinic acid standard solution into the volumetric flasks. Dilute to the mark with sulfuric acid solution. Prepare fumaric acid with concentrations of 5.00 μ g/mL, 10.0 μ g/mL, 20.0 μ g/mL, 25.0 μ g/mL, and 30.0 μ g/mL, respectively; The concentrations of maleic acid were 0.50 μ g/mL, 1.00 μ g/mL, 2.00 μ g/mL, 2.50 μ g/mL, and 3.00 μ g/mL, respectively; Mixed standard solutions with succinic acid concentrations of 20.0 μ g/mL, 50.0 μ g/mL, 100 μ g/mL, 150 μ g/mL, and 200 μ g/mL. Store in a refrigerator at 4°C with a shelf life of 1 month.

A.10.5.2 Preparation of sample solution

Weigh 0.5 g of the sample, accurate to 0.0001 g, dissolve it in sulfuric acid solution and dilute to 100 mL; shake well, and filter through a microporous membrane.

A.10.6 Determination

A.10.6.1 Drawing of standard curve

Under the conditions of A.10.4, inject five different concentrations of mixed standard solutions from A.10.5.1 into the liquid chromatograph, measure the corresponding peak areas, and plot the standard curve with the mass concentration of the mixed standard solution as the horizontal axis and the peak area as the vertical axis.

A.10.6.2 Determination of sample solution

Under the conditions of A.10.4, inject the sample solution into the liquid chromatograph separately to obtain the peak area. Based on the standard curve, obtain the mass concentrations of fumaric acid, maleic acid, and succinic acid in the test solution.

A.10.7 Calculation of results

The mass fraction of fumaric acid, maleic acid, and succinic acid content ω_i is calculated according to formula (A.3).

$$\omega_i = \frac{c_i \times V}{m \times 10^6} \times 100\% \dots\dots\dots (A.3)$$

In which:

c_i - the concentration of the substance to be tested in the sample solution obtained from the standard curve, in micrograms per milliliter (μ g/mL);

V - the constant volume of the sample solution, in milliliters (mL);

m - the mass of the sample, in grams (g);

10^6 - Conversion factor.

The experimental results shall be based on the arithmetic mean of the parallel measurement results. The absolute difference between two independent measurement results obtained under repetitive conditions shall not exceed 10% of the arithmetic mean.

A.10.8 Detection limit and quantification limit

The detection limit of fumaric acid is 0.004%, and the quantification limit is 0.01%; The detection limit of maleic acid is 0.002%, and the quantification limit is 0.005%; The detection limit of succinic acid is 0.08%, and the quantification limit is 0.2%.

Appendix B

Infrared absorption spectrum of L-malic acid standard

The infrared absorption spectrum of L-malic acid standard is as shown in Figure B.1.

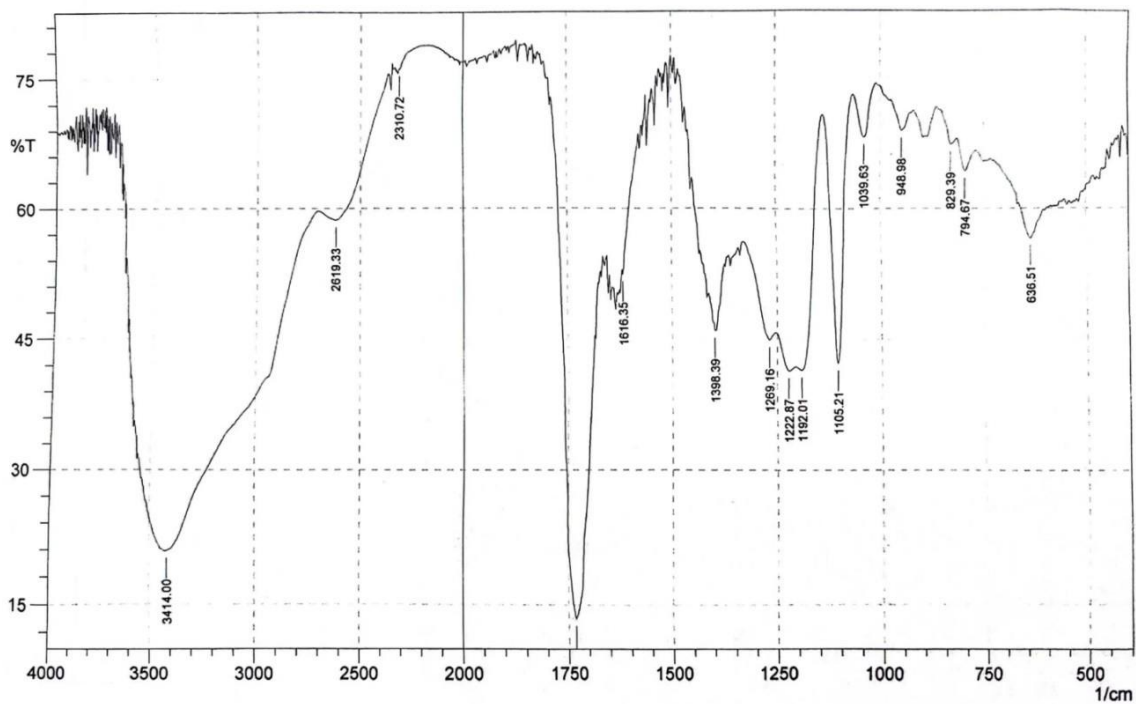


Figure B.1 Infrared absorption spectrum of L-malic acid standard

Appendix C

Liquid chromatogram of standard solutions of fumaric acid, maleic acid, and succinic acid

The liquid chromatograms of standard substance solutions of fumaric acid, maleic acid, and succinic acid are as shown in Figure C1.

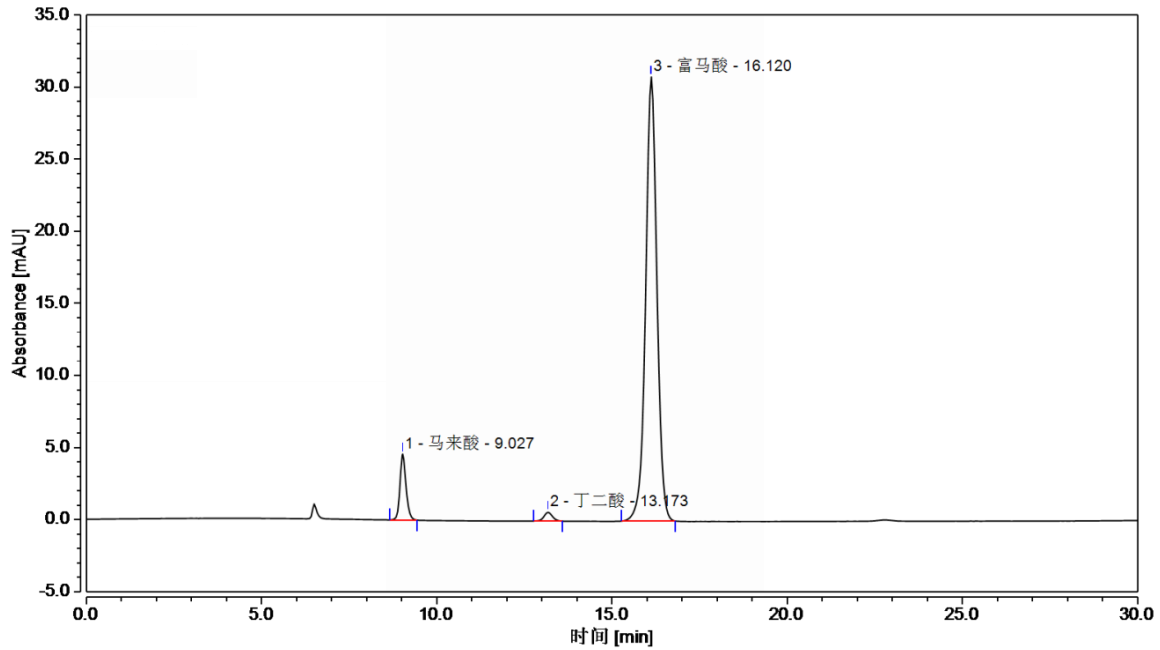


Figure C.1 Liquid chromatogram of standard solutions of fumaric acid, maleic acid, and succinic acid

END TRANSLATION

Attachments:

No Attachments.