

Voluntary Report – Voluntary - Public Distribution

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Report Name: 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, WTO Notifications

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Report Highlights:

On October 25, 2023, China notified the National Food Safety Standard for Food Additive L-Malic Acid to the World Trade Organization (WTO) under G/SPS/N/CHN/1289. The proposed date of entry into force is to be determined. Comments may be submitted to China's SPS National Notification and Enquiry Center at sps@customs.gov.cn until December 24, 2023. The report provides an unofficial translation of the draft standard.

Report Summary:

On October 25, 2023, China notified the National Food Safety Standard for Food Additive L-Malic Acid to the World Trade Organization (WTO) under [G/SPS/N/CHN/1289](#). The proposed date of entry into force is to be determined. Comments may be submitted to China's SPS National Notification and Enquiry Center at sps@customs.gov.cn until December 24, 2023.

This standard is applicable to food additive L-malic acid produced by enzyme engineering method and fermentation method. The technical requirements and testing methods for L-malic acid are specified. This notified draft standard will replace the current implementing national standard [GB 1886.40-2015](#) (link in Chinese). This report provides an unofficial translation of the draft standard notified to WTO.

BEGIN TRANSLATION

**National Food Safety Standard
Food Additive L-Malic Acid**

Foreword

This standard replaces GB 1886.40-2015 National Food Safety Standard for Food Additive L-Malic Acid.

Comparing with GB 1886.40-2015, the following major changes are made to this draft standard:

- Physical and chemical indicators for water content and succinic acid are added,
- The content of physical and chemical indicators and the description of total arsenic are revised and heavy metal content is deleted,
- The testing methods for identification, determination of contents and clarity, fumaric acid, maleic acid, lead, and total arsenic are updated,
- The testing method for succinic acid is added,
- The appendices A and B are revised, and the appendix C is added.

1. Scope

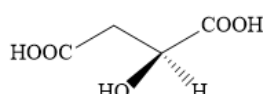
This standard applies to food additive L-malic acid made by enzyme engineering and fermentation.

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 International Relative Atomic Mass in 2022)

3. Technical Requirements

3.1 Sensory requirements

Sensory requirements shall comply with requirements in Table 1.

Table 1: Sensory Requirements

Items	Requirements	Testing Methods
Color	White	Take a reasonable amount of the sample, place it in a clean dry white porcelain plate, observe its color and status under the natural light, and smell it.
Status	Crystal or crystal powder	
Smell	Special sour smell	

3.2 Physical and Chemical Indicators

Physical and chemical indicators shall comply with Table 2.

Table 2: Physical and Chemical Indicators

Items	Indicators	Testing Methods
Content (as C ₄ H ₆ O ₅), w/%	≥ 99.0	A.4 in Appendix A
Specific rotation α _m (20°C, D) / [(°) · dm ² · kg ⁻¹]	-1.6~-2.6	A.5 in Appendix A
Clarity	Tests passed	A.6 in Appendix A
Water content, w/%	≤ 1.0	GB 5009.3 Karl Fischer Method
Ignition residue, w/%	≤ 0.10	A.7 in Appendix A
Chloride (as Cl), w/%	≤ 0.004	A.8 in Appendix A
Sulfate (as SO ₄), w/%	≤ 0.02	A.9 in Appendix A
Fumaric acid, w/%	≤ 0.5	A.10 in Appendix A
Maleic acid, w/%	≤ 0.05	A.10 in Appendix A
Succinic acid, w/%	≤ 2.0	A.10 in Appendix A
Lead (Pb)/(mg/kg)	≤ 2.0	GB 5009.12 or GB 5009.75
Total arsenic (as As)/(mg/kg)	≤ 2.0	GB 5009.11 or GB 5009.76

Appendix A

Testing Methods

A.1 Warnings

Some procedures of the tests may cause hazardous situations, so operators should take appropriate preventive safety and health measures.

A.2 General Rules

Any reagent used in this standard is analytically pure if no other requirement is indicated. The standard titration solution, the standard solution, preparations, and products used in impurity determination tests, shall be prepared according to GB/T 601, GB/T 602, and GB/T 603, if no other requirement is indicated. If it is not indicated which solvent is used for the preparation of solutions in the tests, level three water specified in GB/T 6682 should be used.

A.3 Identification Tests

A.3.1 Reagents and materials

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

A.3.1.2 Sulfanilic 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 Analytic steps

A.3.2.1 Identifying the color of amine salts

Measure 0.5 g of the sample, accurate to 0.01 g, put it into a 50 mL of test tube, add in 10 mL of aqueous solution. Use ammonia solution to neutralize the substance until it becomes neutral, add in 1 mL of sulfanilic acid solution, put the tube in boiling water bath, and heat it for 5 minutes. Add in 5 mL of sodium nitrite solution, put the tube in the boiling water bath again for 3 minutes, add in 5 mL of sodium hydroxide solution, and observe the solution from top to bottom, which should turn red instantly.

A.3.2.2 Identifying the infrared absorption spectrum

Using the method of potassium bromide pellet in accordance with GB/T 6040 to determine the infrared absorption spectrum, the infrared absorption spectrum of the sample should conform to Appendix B.

A.4 Content Determination (calculated as $C_4H_6O_5$)

A.4.1 Summary of the method

Taking phenolphthalein as indicator, use the standard titration solution of sodium hydroxide to titrate the sample solution, and calculate the total acidity concentration in the sample solution (calculated as $C_4H_6O_5$) according to the consumption of the standard titration solution of sodium hydroxide.

A.4.2 Reagents and materials

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

A.4.2.2 Indicator solution of phenolphthalein: 10 g/L.

A.4.3 Instruments and equipment

Analytic balance: with a sensitivity of 0.0001 g.

A.4.4 Analytic steps

Measure about 1.0 g of sample, accurate to 0.0001 g, add in 20 mL of carbon dioxide free aqueous solution, add in 2 drops of the indicator solution of phenolphthalein, use the standard titration solution of sodium hydroxide for titration until the color becomes reddish, and end the process if the color hasn't faded for 30 seconds. Carry out the blank tests by following the same steps as in sample determination tests.

A.4.5 Results calculation

The mass fraction ω_1 for the content (calculated as $C_4H_6O_5$) is calculated according to the formula (A.1).

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

Where:

- V_1 Volume of the standard titration solution of sodium hydroxide consumed in the sample solution, expressed in (mL),
- V_0 Volume of the standard titration solution of sodium hydroxide consumed in the blank solution, expressed in (mL),
- c Concentration of sodium hydroxide in the standard titration solution, expressed in mol/L,
- M Molar mass of 1/2 L-malic acid, expressed in (g/mol) ($M = 67.04$),
- m Mass of the sample, expressed in (g),
- 1000 Conversion factor.

The test result is expressed as arithmetic mean of parallel testing results, accurate to two decimals. The absolute value of the differences between two independent testing results obtained under repetitive conditions should be no more than 0.2%.

A.5 Specific Rotation

A.5.1 Instruments and equipment

A.5.1.1 Polarimeter: with a sodium lamp (Sodium spectrum D-line is 589.3 nm), accurate to $\pm 0.01^\circ$.

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

A.5.2 Analytic steps

Measure 4.25 g of the sample, accurate to 0.01 g, add in 20 mL of aqueous solution to dissolve the substance, make the volume at 50 mL, and follow instructions given in GB/T 613.

A.5.3 Results calculation

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

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

Where:

- Value of the specific rotation of the sample solution measured at 20 °C, expressed in ($^\circ$);
- Value of the length of polarimeter tube, expressed in (dm);
- Mass concentration of effective components of the sample in the solution, expressed in (g/mL).

A.6 Clarity Determination

A.6.1 Reagents and materials

A.6.1.1 Nitric acid

A.6.1.2 Nitric acid solution (1+2): mix nitric acid and water in a volume ratio of 1:2.

A.6.1.3 Silver nitrate solution: 20 g/L.

A.6.1.4 Dextrin aqueous solution: 20 g/L.

A.6.1.5 Standard hydrochloric acid solution: $c(\text{HCl}) = 0.1 \text{ mol/L}$.

A.6.1.6 Standard working solution (with chlorine content of 0.01 mol/L): accurately measure 14.1 mL of standard hydrochloric acid solution, dilute it with water until the volume reaches 50 mL of the solution. Accurately measure 10.0 mL of the solution and dilute it with water until the volume of the solution reaches to 1,000 mL.

A.6.2 Instruments and equipment

A.6.2.1 Nessler tube

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

A.6.3 Analytic steps

A.6.3.1 Preparing the sample solution

Measure about 1.0 g of the sample, accurate to 0.01 g, put it into the Nessler tube, add in 20 mL of water to dissolve, and shake the tube well.

A.6.3.2 Preparing the standard solution

Accurately measure 0.20 mL of standard solution, add in water until the volume reaches 20 mL, add in 1 mL of nitric acid solution, 1 mL of silver nitrate solution, and 0.2 mL of dextrin aqueous solution, shake well, and keep it away from light for 15 minutes.

A.6.4 Results determination

When observed axially and laterally without exposure to direct sunlight, the turbidity level of the sample solution is no more than that of the standard solution, the sample is qualified.

A.7 Ignition Residues

Measure 2.5 g of the sample, accurate to 0.01 g, and follow instructions specified in GB/T 9741. The testing result is calculated as the arithmetic mean of parallel testing. The absolute value of the differences between two independent testing results obtained under repetitive conditions should be no more than 0.02%.

A.8 Chloride Determination

Measure 1.0 g of the sample according to GB/T 9729, accurate to 0.01 g. Measure 0.4 mL of standard chloride solution. When turbidity of the sample solution is no more than that of the standard solution, the chloride content is no more than 0.004%.

A.9 Sulfate Determination

A.9.1 Reagents and materials

A.9.1.1 Hydrochloric acid

A.9.1.2 Hydrochloric solution (1+4): mix hydrochloric acid and water in a volume ratio of 1:4.

A.9.1.3 Barium chloride solution: 250 g/L.

A.9.1.4 Standard sulfate (SO₄) solution: 0.1 mol/L.

A.9.2 Instruments and equipment

A.9.2.1 Nessler tube

A.9.2.2 Analytic balance: with a sensitivity of 0.01 g and 0.0001 g.

A.9.3 Analytic steps

A.9.3.1 Preparing the sample solution

Measure 1.0 g of the sample, accurate to 0.01 g, put it into a 50 mL Nessler tube, add water to dissolve until the volume reaches about 40 mL, and shake well.

A.9.3.2 Preparing the reference solution

Accurately measure 2.0 mL of standard sulfate solution, put it into a 50 mL Nessler tube, add water until the volume reaches about 40 mL, and shake well.

A.9.3.3 Determination

Add 0.5 mL of hydrochloric solution and 1 mL of barium chloride solution into the sample solution and the reference solution respectively, add water to dilute the solutions until the volumes reach 50 mL, keep them still for 10 minutes after shaking well.

A.9.4 Results determination

Place both tubes against the same black background and observe turbidity from the top to the bottom of the Nessler tube. If the turbidity of the sample solution is no more than that of the reference solution, the sulfate content is $\leq 0.02\%$.

A.10 Determination of Fumaric Acid, Maleic Acid, and Succinic Acid

A.10.1 Principle

Use high performance liquid chromatography with an ultraviolet detector to conduct the quantitative analysis by external standard methods, after the sample has been processed.

A.10.2 Reagents and materials

A.10.2.1 Water: level one water

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

A.10.2.3 The standard substance of fumaric acid (C₄H₄O₄, CAS No.: 110-17-8): purity $\geq 99.0\%$, or the standard substance certified and granted with a Reference Material Certificate at the national level.

A.10.2.4 The standard substance of maleic acid (C₄H₄O₄, CAS No.: 110-16-7): purity $\geq 99.0\%$, or the standard substance nationally certified and granted with a Reference Material Certificate at the national level.

A.10.2.5 The standard substance of succinic acid (C₄H₆O₄, CAS No.: 110-15-6): purity $\geq 99.0\%$, or the standard substance certified and granted with a Reference Material Certificate at the national level.

A.10.2.6 Standard fumaric acid solution (500 $\mu\text{g/mL}$): accurately measure 50 mg of the standard

substance of fumaric acid, accurate to 0.0001 g, add in sulfuric solution to dissolve it until the volume reaches 100 mL, and place it in a refrigerator at 4°C for storage, valid for 6 months.

A.10.2.7 Standard maleic acid solution (50 µg/mL): accurately measure 5 mg of the standard substance of maleic acid, accurate to 0.0001 g, add in sulfuric solution to dissolve it until the volume reaches 100 mL, and place it in a refrigerator at 4 °C for storage, valid for 6 months.

A.10.2.8 Standard succinic acid solution (5 mg/mL): accurately measure 500 mg of the standard substance of succinic acid, accurate to 0.0001 g, add in sulfuric solution to dissolve it until the volume reaches 100 mL, and place it in a refrigerator at 4 °C for storage, valid for 6 months.

A.10.2.9 Microporous filter membrane: 0.22 µm, aqueous phase.

A.10.3 Instruments and equipment

A.10.3.1 High performance liquid chromatography: with a ultraviolet detector, or any other detector with the same effect.

A.10.3.2 Analytic balance: with a sensitivity of 0.01 g and 0.0001 g.

A.10.4 Reference requirements for instruments

A.10.4.1 Chromatographic column: a hydrogen cation exchange chromatography column with polystyrene-divinylbenzene resin as filling materials, 300 mm× 7.8 mm, or any other chromatographic column with the same analytic effect.

A.10.4.2 Column temperature: 45 °C

A.10.4.3 Mobile phase: sulfuric solution.

A.10.4.4 Flow rate: 0.5 mL/min.

A.10.4.5 Sample size: 10 µL.

A.10.4.6 Detection wavelength: 214 nm.

A.10.5 Analytic steps

A.10.5.1 Preparing the standard mixed solution

Take five 100mL volumetric flasks to accurately measure 1.00 mL, 2.00 mL, 4.00 mL, 5.00 mL, and 6.00 mL of standard fumaric acid solution and standard maleic acid solution respectively, and measure 0.40 mL, 1.00 mL, 2.00 mL, 3.00 mL, and 4.00 mL of standard succinic acid solution, and mix with sulfuric solution to reach the expected volumes. The standard mixed solutions are prepared with concentrations of fumaric acid of 5.00 µg/mL, 10.0 µg/mL, 20.0 µg/mL, 25.0 µg/mL, and 30.0 µg/mL respectively, the concentrations of maleic acid of 0.50 µg/mL, 1.00 µg/mL, 2.00 µg/mL, 2.50 µg/mL, and 3.00 µg/mL, and the concentrations of succinic acid of 20.0 µg/mL, 50.0 µg/mL, 100 µg/mL, 150 µg/mL, and 200 µg/mL, respectively. Place them in a refrigerator at 4 °C for storage, valid for 6 months.

A.10.5.2 Preparation of the sample solution

Measure 0.5 g of the sample, accurate to 0.01 g, use sulfuric solution to dissolve the substance to reach volume of 100 mL, shake well and filter the solution with a 0.22 µm filter membrane.

A.10.6 Determination

A.10.6.1 Drawing of the standard curve

Under conditions specified in A.8.4, put the standard mixed solutions at 5 different concentrations specified in A.8.5.1 into a liquid chromatograph separately, determine the corresponding peak areas, and use the mass concentrations in the standard mixed solutions as

abscissas and the peak areas as ordinates to draw the standard curve.

A.10.6.2 Determination of the sample solution

Under conditions specified in A.8.4, put the sample solution into a liquid chromatograph to get the peak area, and obtain the mass concentrations of fumaric acid, maleic acid, and succinic acid in the solutions according to the standard curve.

A.10.7 Results calculation

The mass fraction ω_2 for content of fumaric acid, maleic acid, and succinic acid is calculated according to formula (A.3).

$$\omega_2 = \frac{c \times V}{m \times 10^6} \times 100\% \dots\dots\dots (A.3)$$

Where:

- c Concentration of the substance to be determined in the sample solution calculated according to the standard curve, expressed as $\mu\text{g/mL}$,
- V Constant volume of the sample solution, expressed as mL,
- m Mass of the sample, expressed as g,
- 10^6 Conversion factor.

The test result is expressed as the arithmetic mean of the results from parallel tests. The absolute value of the differences between the two independently testing results obtained under repetitive conditions is no more than 10% of the arithmetic mean.

A.10.8 Detection limit and quantification limit

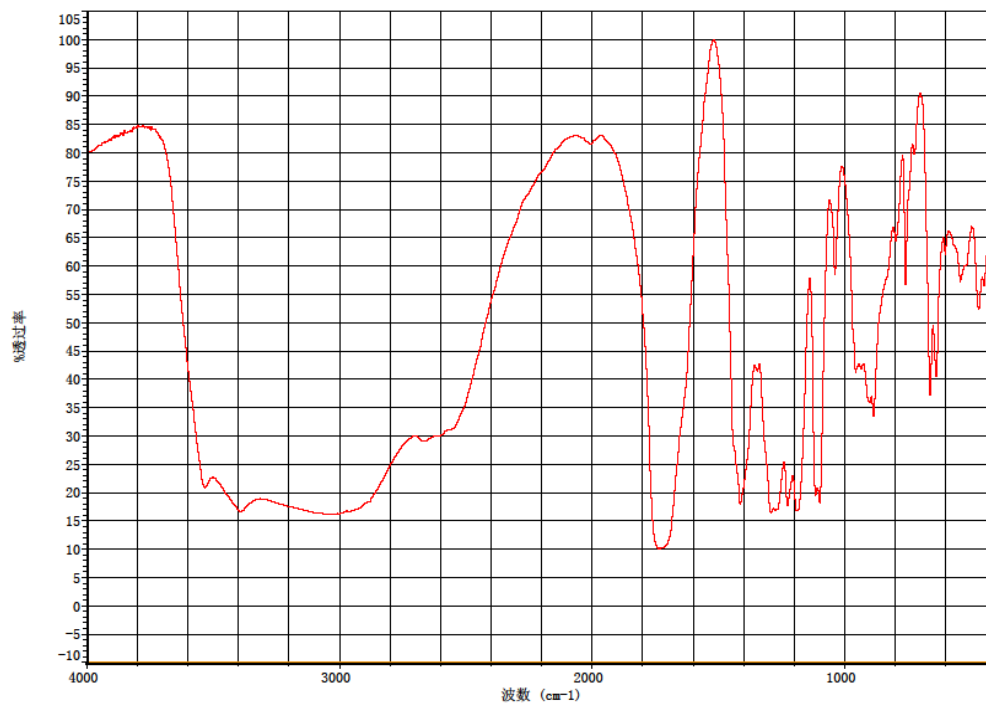
The detection limit of fumaric acid is 0.004% and the quantification limit is 0.01%. The detection limit of maleic acid is 0.002% and the quantification limit is 0.005%. The detection limit of succinic acid is 0.08 % and the quantification limit is 0.2%.

Appendix B

The infrared absorption spectrum of the standard substance of L-malic acid

See Figure B.1 for the infrared absorption spectrum of the standard substance of L-malic acid.

Figure B.1 The infrared absorption spectrum of the standard substance of L-malic acid



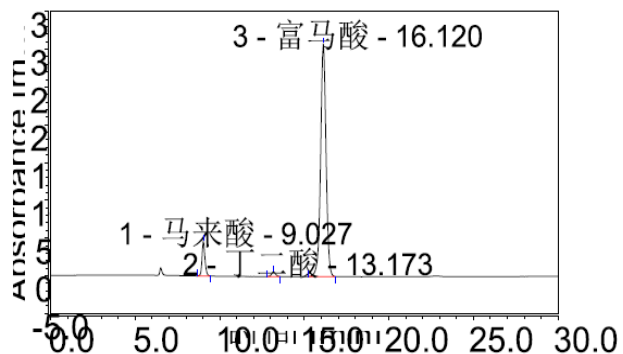
Wave number (cm-1)

Appendix C

The liquid chromatogram of fumaric acid, maleic acid, and succinic acid in standard solutions

See Figure C.1 for the liquid chromatogram of the standard substance solutions of fumaric acid, maleic acid, and succinic acid.

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



END TRANSLATION

Attachments:

No Attachments.