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National Food Additive Standard- DL-malic acid

Report Categories:

FAIRS Subject Report

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Report Highlights:

On May 5, 2010, China notified the WTO of "National Food Safety Standard: Food Additives - DL-Malic Acid" as SPS/N/CHN/282. This measure "applies to the technical requirements and testing methods for sucralose." The date for submission of final comments to the WTO is May 20, 2010. The proposed date of entry is May 30, 2010. This report is an INFORMAL translation of this document.

Executive Summary:

On May 5, 2010, China notified the WTO of "National Food Safety Standard: Food Additives - DL-Malic Acid" as SPS/N/CHN/282. This measure "applies to the technical requirements and testing methods for sucralose." The date for submission of final comments to the WTO is May 20, 20, 2010. The proposed date of entry is May 30, 2010.

Thanks go to the Keller and Heckman LLP Shanghai Representative Office for their assistance in translating this document.

This report contains an UNOFFICIAL translation of National Food Safety Standard: Food Additives - Polydextrose.

General Information:

BEGIN TRANSLATION

GB National Food Safety Standard
GB/T 5009.11—xxxx
To replace GB/T 5009.11—2003

National Food Safety Standard
DL- Malic Acid

(Draft for discussion)

Issued on xx-xx-xxxx Implemented on xx-xx-xxxx

Issued by the Ministry of Health

of the People's Republic of China

Foreword

The Standard was drafted with reference to the "DL-malic acid" standard in the US Food Chemicals Codex Version 6 (FCC 6).

Annexes A, B and C of the Standard are mandatory annexes.

National Food Safety Standard
Food Additive - DL-malic acid

1.**Scope**

The Standard is applicable to the food additive DL-malic acid prepared with malic anhydride and malic acid/fumaric acid as the raw materials through hydration, concentration and crystallization, dehydration and drying.

2. **Normative reference**

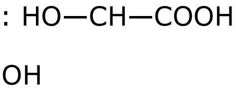
The documents cited in the Standard are mandatory portions of the Standard. For any dated reference, only the dated version applies; and for any undated reference, its latest version (including all revisions) applies.

3. **Chemical name, molecular formula, structural formula and relative molecular mass**

Chemical name: DL - malic acid

Molecular formula: $C_4H_6O_5$

Structural formula:



$$\begin{array}{l} HO—CH—COOH \\ \quad\;\; CH_2—COOH \end{array}$$

Relative molecular mass: 134.09 (to 2007 International Relative Atomic Mass)

4. **Sensory**

White or near white crystal powder or grains, with special acidic taste.

5. **Technical requirements**

Technical requirements are set forth in Table 1 below.

Table 1 Technical Requirements

Water insoluble, *w* / % ≤	0.1	A.10, Annex A

Annex A

Item	Index	Test method
DL-malic acid (per $C_4H_6O_5$) , *w* /%	99.0～100.5	A.4, Annex A
Specific rotation α_m(25℃,D)/〔(º)·dm^2· kg^{-1}〕	-0.10 ～ +0.10	A.5, Annex A
Arsenic (As) / (mg/kg) ≤	2	A.6, Annex A
Lead (Pb) / (mg/kg) ≤	2	A.7, Annex A
Residue on ignition, *w* /% ≤	0.10	A.8, Annex A
Fumaric acid , *w* / % ≤	1.0	A.9, Annex A
Malic acid, *w* / % ≤	0.05	A.9, Annex A

(Normative)

Test method

A.1 **Warning**

Danger may be caused in some processes specified in the test methods, so the operator shall take the proper safety and protection measures.

A.2 **General requirements**

Unless otherwise specified, only the reagents of analytic purity and Level 3 water specified in GB/T6682 are used for analysis.

When no other requirements are indicated, the standard titration solution, and the standard solution, preparation or products for measuring the impurity in the test methods shall be prepared according to GB/T 601, GB/T 602 and GB/T 603.

A.3 **Identification test**

A.3.1 Ammonia salt color test for malic acid

A.3.1.1 Reagent

A.3.1.1.1 Ammonia solution: 2+3.

A.3.1.1.2 Sulfanilic acid: 10g/L.

A.3.1.1.3 Sodium nitrite solution: 200g/L.

A.3.1.1.4 Sodium hydroxide solution: 40g/L.

A.3.1.2 Analytic procedures

Weigh 0.5g laboratory samples (to the accuracy of 0.01g) to a 50mL test tube, dissolve with 10mL water; neutralize with ammonia solution to neutral, add in 1mL sulfanilic acid, and heat in the boiled water bath for 5min; add in 5mL sodium nitrite solution, heat again in the water bath for 3min, add in 5mL sodium hydroxide solution, and the test solution shall become red immediately.

A.3.2 Comparison of infrared spectra

Weigh about 1mg laboratory samples and 100mg potassium bromide, grind to uniform powder, place in the shaper and press to a sheet, measure the absorption spectrum with the infra-red spectrometer, and the spectrum shall be basically consistent with the standard infrared spectrum of DL-malic acid shown in Figure B.1, Annex B.

A.4 **Measurement of DL-malic acid content**

A.4.1 Method

Adopt phenolphthalein as the indicator and the sodium hydroxide standard solution for titrating the sample water solution, and calculate and obtain the total acid amount as per $C_4H_6O_5$, i.e. the DL-malic acid content, according to the amount of sodium hydroxide standard titration solution used.

A.4.2 Reagent

A.4.2.1 Sodium hydroxide standard titration solution: C(NaOH) = 1.0mol/L.

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

A.4.3 Analytic procedures

A.4.3.1 Weigh 2.0g laboratory samples (to the accuracy of 0.0002g), dissolve with 40mL water containing no carbon dioxide, add in 2 drops of phenolphthalein indicator, titrate with sodium hydroxide standard solution to pale red, and hold 30s till no fading occurs.

A.4.3.2 During the measurement, conduct the blank test with the reagent solution of the same amount instead of the specimen, according to the same procedures of the measurement.

A.4.4 Calculation

The DL-malic acid mass fraction *w₁* (per $C_4H_6O_5$, in %) is calculated as per the following Equation A.1:

$$w_1 = \frac{[(V - V_0)/1000]cM}{m} \times 100 \qquad \text{.............................. (A.1)}$$

where:

· *V*-volume of sodium hydroxide standard titration solution (A.4.2.1) consumed in the specimen, mL;

· *V0*-olume of sodium hydroxide standard titration solution (A.4.2.1) consumed in the blank test, mL;

C-accurate concentration of sodium hydroxide standard titration solution, mol/L;

m-specimen mass, g;

M-molar mass of malic acid (1/2 $C_4H_6O_5$) , g/ mol (*M* =67.04).

Take the arithmetic average of the results of 2 replicate measurements as the result for reporting.

The absolute difference between 2 replicate measurements shall not be more than 0.2%.

A.5 **Measurement of specific rotation**

A.5.1 Weigh 4.25g laboratory samples (to the accuracy of 0.001g), dissolve with 20mL water, transfer to a 50mL volumetric flask, dilute with water to scale, and shake well. The temperature for measurement shall be 25℃±0.5℃.

$$\alpha_m(25\,^{\circ}C, D) = \frac{\alpha}{l\rho_{\alpha}}$$

The specific rotation α_m (25℃, D) is expressed as “($^{\circ}$)·dm^2· kg^{-1}” and calculated according to the following Equation (A.2):

……………………… (A.2)

where:

α —angle of rotation measured, (°);

l — length of optical rotation tube, dm;

ρ_{α} —— mass concentration of active components in the solution, g/mL.

A.5.2 Conduct other procedures as per GB/T 613.

A.6 **Measurement of arsenic**

A.6.1 Weigh 1.0g laboratory samples (to the accuracy of 0.01g) to a conical flask for measuring arsenic, dissolve with 5mL water, add in 1 drop of bromophenol blue indicator（0.4g/L）, neutralize with ammonia solution (1+4) till the solution becomes purple; add water to make about 35mL, add in 20mL sulphuric acid solution（1+5）and shake well, as the specimen solution. Conduct the limit test with absorption solution B, and weigh (2±0.02) mL arsenic (As) standard solution (equivalent to 2.0μg As) to prepare the limit standard.

A.6.2 Conduct other procedures as per the silver diethyldithiocarbamate (Ag-DDC) colorimetric method in GB/T 5009.76.

A.7 **Measurement of lead**

A.7.1 Colorimetric method (referee method)

Conduct as per GB/T 5009.75, and process the specimen as per the dry digestion method. Dilute 1mg/mL lead (Pb) standard solution to 5μg/mL immediately before use. For measurement, weigh (25±0.02) mL specimen solution (equivalent to 2.5g laboratory sample) and (1±0.02) mL lead

(Pb) standard solution (equivalent to 5μg Pb) to 125mL separating funnel respectively, and add 1% nitric acid solution in the lead standard solution to make 25mL.

A.7.2 Atomic absorption spectrometry

Process the specimen as per the dry digestion method in GB/T 5009.75, and others as per GB/T 5009.12.

A.8 **Measurement of residue on ignition**

Weigh about 2g laboratory samples (to the accuracy of 0.0001g). The ignition temperature shall be (800±25) ℃, others as per GB/T 9741.

Take the arithmetic average of the results of 2 replicate measurements as the result for reporting. The absolute difference between 2 replicate measurements shall not be more than 0.01%.

A.9 **Measurement of fumaric acid and malic acid**

A.9.1 Method

Separate the components in the sample solution with the chromatographic column in the selected working conditions with the high performance liquid chromatography (HPLC), detect with ultraviolet absorption detector, quantify with the external standard method and calculate the contents of fumaric acid and malic acid in the sample.

A.9.2 Reagent

A.9.2.1 Fumaric acid: mass fraction ≥99.0%.

A.9.2.2 Malic acid: mass fraction ≥99.0%.

A.9.2.3 Sodium hydroxide solution: 20g/L.

A.9.2.4 Phosphoric acid solution: Weigh (1±0.02)mL G.R. phosphoric acid to a 1000mL volumetric flask, add in 100mL methanol (HPLC grade)（It is allowed to adjust the amount added according to the column efficiency）, dilute with water to scale, and filter with 0.45μm filter membrane.

A.9.3 Instruments

A.9.3.1 High performance liquid chromatography (HPLC)

A.9.3.1.1 High pressure pump: no pulse, to maintain the flow rate of 0.1mL/min～10.0 mL/min.

A.9.3.1.2 Dosing ring: 5mL.

A.9.3.1.3 Ultraviolet detector: variable wavelength.

A.9.3.1.4 Data processing system: chromatogram workstation or data processer.

A.9.3.2 Suction filter system

Adopting 0.45μm cellulose ester membrane filter paper（for pretreatment of mobile phase）.

A.9.3.3 Filter system

Adopting 0.45μm cellulose ester membrane filter paper（for sample pretreatment）.

A.9.3.4 Micro sample injector

50mL and 100mL (or automatic injector) , dedicated for HPLC.

A.9.4 Conditions of chromatographic analysis

The recommended chromatographic column and the typical operation conditions are shown in Table A.1; the typical HPLC measured in fumaric acid and malic acid is shown in C.1, Annex C; and the relative retention time of components is shown in C.1, Annex C. Other chromatographic columns and operation conditions meeting the same degree of separation can also be used.

Table A.1 Chromatographic column and typical chromatographic operation conditions

Chromatographic column	Nonpolar packing chromatographic column, 250mm in length, 4.6mm in inner diameter, with silica gel matrix, and C8 functional group linked on the surface
Column temperature	15～60◦C, control accuracy ±1◦C
Mobile phase	Phosphoric acid solution
Flow rate, mL/min	1.0
Detection wavelength of detector, nm	214
Injected amount, μL	5

A.9.5 Analytic procedures

A.9.5.1 Preparation of standard sample solution

A.9.5.1.1 Preparation of standard sample solution of fumaric acid

Weigh 50mg fumaric acid (to the accuracy of 0.0002g), dissolve in appropriate amount of water (add in small amount of sodium hydroxide solution if necessary) , transfer to a 50mL volumetric flask, and dilute to scale with phosphoric acid solution.

Transfer (1±0.02) mL of the above solution to a 50mL volumetric flask, and dilute to scale with phosphoric acid solution, filter with 0.45μm filter membrane, and conduct ultrasonic degassing.

A.9.5.1.2 Preparation of standard sample solution of malic acid

Weigh 50mg malic acid (to the accuracy of 0.0002g), dissolve in appropriate amount of water (add in small amount of sodium hydroxide solution if necessary) , transfer to a 250mL volumetric flask, and dilute to scale with phosphoric acid solution.

Transfer (1±0.02) mL of the above solution to a 100mL volumetric flask, and dilute to scale with phosphoric acid solution, filter with 0.45μm filter membrane, and conduct ultrasonic degassing.

A.9.5.2 Preparation of sample solution

Weigh 0.2g laboratory samples (to the accuracy of 0.0002g) to a 50mL volumetric flask, and dilute to scale with the mobile phase, shake well, filter with 0.45μm filter membrane, and conduct ultrasonic degassing.

A.9.5.3 Measurement

According to the HPLC operation procedures, start the instrument for preheating, adjust the temperature and the flow rate to reach the analytic conditions and the stable baseline, and add the standard sample solution.

Take 5mL standard sample solution with the micro sample injector (dedicated for HPLC), add the sample (or by automatic injection), and record the peak area A_2 of fumaric acid or malic acid.

Take 5mL sample solution with the micro sample injector (dedicated for HPLC), add the sample (or by automatic injection), and record the peak area A_1 of the substance to be measured.

A.9.6 Calculation

The fumaric acid or malic acid mass fraction w_2 (in %) is calculated as per the following Equation A.3:

$$w_2 = \frac{A_1 \times m_2}{A_2 \times m} \times 100$$

.... (A.3)

where:

A_1 —— peak area of the substance in the sample solution to be measured;

A_2 —— peak area of fumaric acid or malic acid in the standard sample solution;

I m2 -- ejected amount of fumaric acid or malic acid in the standard sample solution. μg;

m — injected amount of the sample, μg.

A.10 **Measurement of water insoluble**

A.10.1 Procedures

Weigh 25.0g laboratory samples (to the accuracy of 0.1g), resolve in 100mL water and filter with crucible acid proof funnel dried to constant weight; rinse the funnel with hot water, place and dry in 100℃ oven to constant weight, cool and weigh it.

A.10.2 Calculation

$$w_3 = \frac{m_1}{m} \times 100$$

The water insoluble mass fraction w_3 (in %) is calculated as per the following Equation A.4:

…………………… (A.4)

Where:

m_1 ——Filter residue, g;

m —— Reagent mass, g.

Annex B

(Normative)

Standard infrared spectrum of DL-malic acid

Figure B.1 shows the standard infrared spectrum of DL-malic acid.

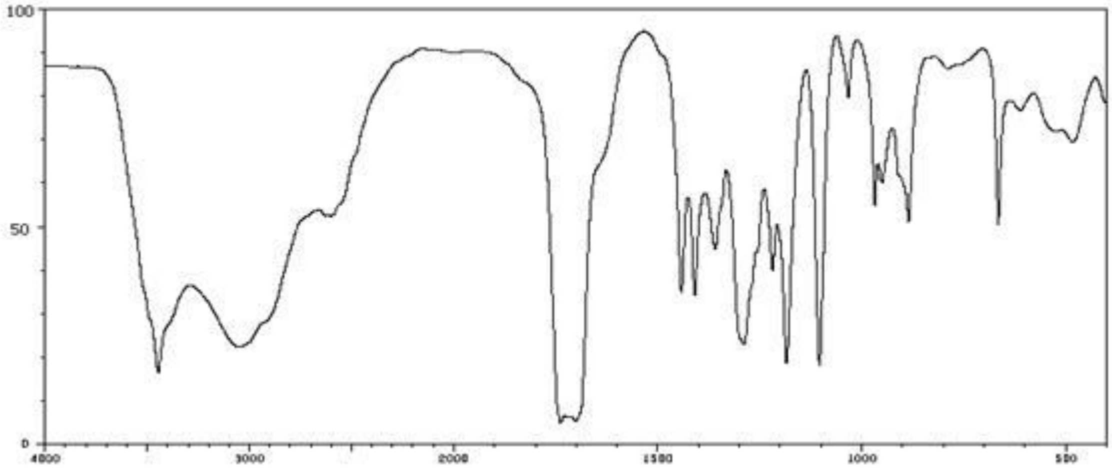


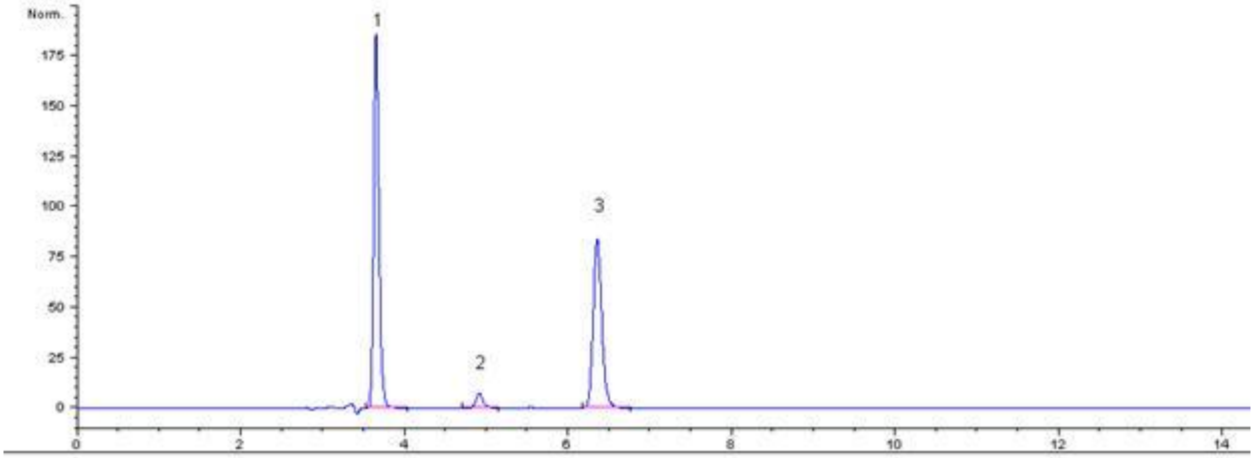
Figure B.1 Standard infrared spectrum of DL-malic acid

Annex C

(Normative)

Typical HPLC measured in fumaric acid and malic acid and relative retention time

C.1 Figure C.1 shows the typical HPLC measured in fumaric acid and malic acid.



1 -- DL-malic acid

2 -- Malic acid

3 -- Fumaric acid

Figure C.1 Typical HPLC measured in fumaric acid and malic acid

C.2 Table C.1 shows the relative retention time of components.

Table C.1 Relative retention time of components

Peak No.	Component	Relative retention time
1	DL-malic acid	1
2	Malic acid	1.34
3	Fumaric acid	1.74

END TRANSLATION