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# GAIN Report

Global Agricultural Information Network

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## China - Peoples Republic of

**Post:** Beijing

### National Food Additive Standard-Fumaric 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 - Fumaric Acid" as SPS/N/CHN/284. This measure "applies to the production, circulation, supervision and management of the food additive fumaric acid. It specifies the scope, requirements and testing methods." 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 - Fumaric Acid" as SPS/N/CHN/284. This measure "applies to the production, circulation, supervision and management of the food additive fumaric acid. It specifies the scope, requirements and testing methods." The date for submission of final comments to the WTO is May 20, 2010. The proposed date of entry is May 30, 2010.

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

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

**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  
Food additive Fumaric 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 "Fumaric Acid" standards 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 - Fumaric Acid****1. Scope**

The Standard is applicable to food additive fumaric acid prepared with maleic acid as a raw material, through isomerization, crystallization and drying.

**2. Mandatory Reference**

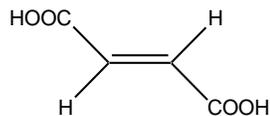
The documents cited in the Standard are mandatory portions of the Standard. For any dated reference, only the dated version applies; and for any undated reference, its latest version (including all revisions) applies.

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

Chemical name: fumaric acid

Molecular formula: C<sub>4</sub>H<sub>4</sub>O<sub>4</sub>

Structural formula:



Relative molecular mass: 116.07 (to 2007 International Relative Atomic Mass)

#### 4. Sensory

White crystal powder or grains, with acidic taste.

#### 5. Technical requirements

Technical requirements are set forth in Table 1 below.

Table 1 Technical Requirements

Annex A		
Item	Index	Test method
Fumaric acid ( per C <sub>4</sub> H <sub>4</sub> O <sub>4</sub> ) ( dry basis ) , w /%	99.5 ~ 100.5	A.4, Annex A
Arsenic ( As ) /(mg/kg) ≤	2	A.5, Annex A
Lead ( Pb ) /(mg/kg) ≤	2	A.6, Annex A
Residue on ignition , w /% ≤	0.10	A.7, Annex A
Maleic acid , w /% ≤	0.10	A.8, Annex A
Water content, w /% ≤	0.5	A.9, Annex A

( Normative )

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#### 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

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 fumaric acid shown in Figure B.1, Annex B.

#### A.4 Measurement of fumaric 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<sub>4</sub>H<sub>4</sub>O<sub>4</sub> on dry basis, i.e. the fumaric 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) =  
0.5mol/L.

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

A.4.3 Analytic procedures

A.4.3.1 Weigh 1.0g laboratory samples (to the accuracy of 0.0002g) to a 250mL conical flask, heat and dissolve with 100mL water, allow it to cool, add in 3 drops of phenolphthalein indicator, titrate with sodium hydroxide standard solution to pale red, and hold 30s till no fading occurs.

A.4.3.1 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 fumaric acid mass fraction  $w_1$  (per C<sub>4</sub>H<sub>4</sub>O<sub>4</sub> on dry basis, in %) is calculated as per the following Equation A.1:

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

where:

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

V<sub>0</sub> —volume 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;

W<sub>3</sub> —water content measured in A.9, %;

M —molar mass of fumaric acid ( 1/2 C<sub>4</sub>H<sub>4</sub>O<sub>4</sub> ) , g/ mol (M =58.04).

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 arsenic**

A.5.1 Weigh 1.0g laboratory samples (to the accuracy of 0.01g), and process the specimen as per 5.2.1 Wet digestion method in GB/T 5009.76. 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.5.2 Conduct other procedures as per the silver diethyldithiocarbamate (Ag-DDC) colorimetric method in GB/T 5009.76.

**A.6 Measurement of lead**

A.6.1 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.6.2 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.7 Measurement of ignition residue

Weigh about 2g laboratory samples (to the accuracy of 0.0001g). The ignition temperature shall be (800±25) °C, 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.8 Measurement of maleic acid

#### A.8.1 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 maleic acid in the sample.

#### A.8.2 Reagent

A.8.2.1 Maleic acid: mass fraction ≥99.0%.

A.8.2.2 Sodium hydroxide solution: 20g/L.

A.8.2.3 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.8.3 Instruments

##### A.8.3.1 High performance liquid chromatography (HPLC)

A.8.3.1.1 High pressure pump: no pulse, to maintain the flow speed of 0.1mL/min ~ 10.0 mL/min.

A.8.3.1.2 Dosing ring: 5µL.

A.8.3.1.3 Ultraviolet detector: variable wavelength

A.8.3.1.4 Data processing system: chromatogram workstation or data processor with Millennium 32 analysis and process software or equivalent functions.

##### A.8.3.2 Suction filter system

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

##### A.8.3.3 Filter system

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

##### A.8.3.4 Micro sample injector

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

#### A.8.4 Conditions of chromatographic analysis

The recommended chromatographic column and the typical operation conditions are shown in Table A.1; the typical HPLC in measuring maleic acid content is shown in B.1, Annex B; and the relative retention time of components is shown in B.1, Annex B. 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.8.5 Analytic procedures

##### A.8.5.1 Preparation of standard sample solution

Weigh 50mg maleic 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, shake well, filter with 0.45µm filter membrane, and conduct ultrasonic degassing.

##### A.8.5.2 Preparation of sample solution

Weigh 0.1g 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.8.5.3 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 5µL standard sample solution with the micro sample injector (dedicated for HPLC), add the sample (or by automatic injection), and record the peak area A2 of maleic acid.

Take 5µL sample solution with the micro sample injector (dedicated for HPLC), add the sample (or by automatic injection), and record the peak area A1 of the substance to be measured.

##### A.8.6 Calculation

The maleic acid mass fraction w<sub>2</sub> (in %) is calculated as per the following Equation A.2:

$$w_2 = \frac{A_1 \times m_2}{A_2 \times m} \times 100 \dots\dots\dots ( A.2 )$$

where:

A1 — peak area of the substance in the sample solution to be measured;

A2 — peak area of maleic acid in the standard sample solution;

m<sub>2</sub> — injected amount of maleic acid in the standard sample solution. µg;

m — injected amount of the sample, µg.

#### A.9 Measurement of water content

##### A.9.1 Loss on drying method

Weigh about 5g laboratory sample (to the accuracy of 0.0002g) and others as per GB/T 6284.

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 0.05%.

##### A.9.2 Karl Fischer method ( referee method )

Weigh about 0.5 ~ 1.0g laboratory sample (to the accuracy of 0.0002g) and others as per GB/T 6283.

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 0.05%.

**Annex B**  
( Normative )

**Standard infrared spectrum of fumaric acid**

Figure B.1 shows the standard infrared spectrum of fumaric acid.

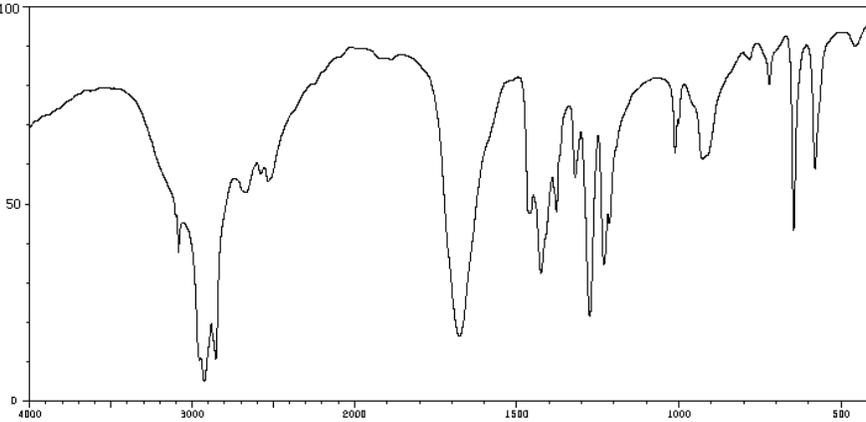
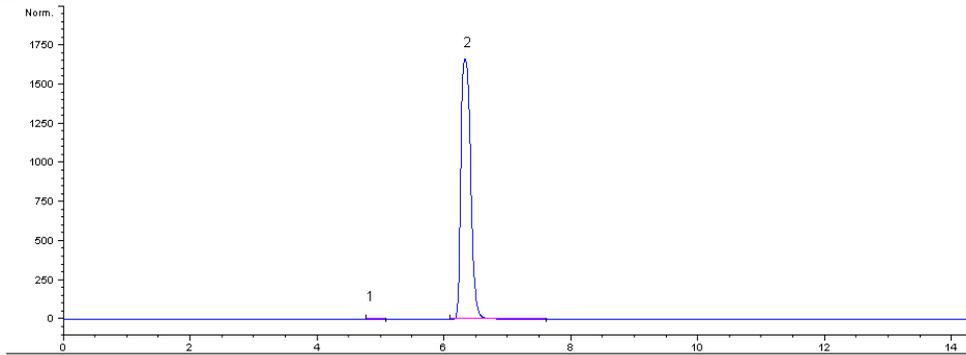


Figure B.1 Standard infrared spectrum of fumaric acid

**Annex C**  
( Informative )

**Typical HPLC measured in maleic acid and relative retention time**

Figure C.1 shows the typical HPLC measured in maleic acid.



- 1 — Maleic acid
- 2 — Fumaric acid

Figure C.1 Typical HPLC measured in maleic acid  
Table C.1 shows the relative retention time of components.  
C.1 Relative retention time of components

Peak No.	Component	Relative retention time
1	Maleic acid	0.77

2	Fumaric acid	1
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END TRANSLATION

