USDA United States Department of Agriculture
Foreign Agricultural Service

GAIN Global Agricultural Information Network

Voluntary Report – Voluntary - Public Distribution **Date:** April 12, 2023

Report Number: CH2023-0056

Report Name: National Food Safety Standard of Food Nutritional Fortifier L-Lysine L-Aspartate

Country: China - People's Republic of

Post: Beijing

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

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Report Highlights:

On March 6, 2023, China notified a new National Food Safety Standard of Food Nutritional Fortifier L-Lysine L-Aspartate to the World Trade Organization (WTO) under G/SPS/N/CHN/1273. The deadline for comment submission is May 5, 2023. The proposed date of entry into force is to be determined. Comments may be submitted by email to China's SPS Enquiry Point at sps@customs.gov.cn. This report provides an unofficial translation of the draft standard.

Summary:
On March 6, 2023, China notified a new National Food Safety Standard of Food Nutritional Fortifier L-Lysine L-Aspartate to the WTO under G/SPS/N/CHN/1273. This standard applies to related food nutritional fortifier L-Lysine L-Aspartate which is produced by neutralization, concentration, and crystallization with L-Lysine and L-Aspartic acid or with L-Lysine by ion exchange of L-Lysine hydrochloride and L-Aspartic acid.

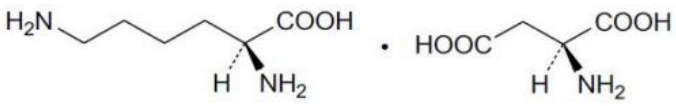
This report provides an unofficial translation of the draft standard.

BEGIN TRANSLATION

National Food Safety Standard
Food Nutritional Fortifier L-Lysine L-aspartate
(Draft for comments)

1. Scope
This standard is applicable to the preparation of food nutritional fortifier L-Lysine L-Aspartate which is produced by neutralization, concentration, and crystallization with L-Lysine and L-Aspartic acid, or with L-Lysine by ion exchange of L-Lysine hydrochloride and L-Aspartic acid.

2. Chemical name, molecular formula, structural formula, and relative molecular weight
2.1 Chemical Name
L-Lysine L-Aspartate
2.2 Molecular formula
$C_{10}H_{21}N_3O_6$
2.3 Structural formula



2.4 Relative molecular mass
279. 29 (according to 2018 international relative atomic mass)

3. Technical Requirements
3.1 Sensory Requirements
Sensory indicators should comply with provisions in the Table 1.

Table 1: Sensory Requirements

Item	Requirements	Testing Methods
Color	White	Take an appropriate amount of sample and place it in a clean, dry white porcelain dish, and observe its color and condition under natural light.
Status	Crystalline or crystalline powder	

3.2 Physical and Chemical Indicators

Physical and chemical indicators shall comply with provisions in Table 2.

Table 2: Physical and Chemical Indicators

Item		Indicator	Testing Methods
L-Lysine L-Aspartate content (on a dry basis), w/%≥		98.0	Appendix A.4
Specific rotation (on a dry basis), αm (20℃, D)/ [(°)·dm^2 ·kg^{-1}]		+24.0 ~+26.5	Appendix A.5
pH(50g/L)		5.0 ~7.0	Appendix A.6
Drying loss, w/%	≤	0.2	Appendix A.7
Ignition residue, w/%	≤	0.1	Appendix A.8
Chloride/ (calculated as Cl)/ (%)	≤	0.041	Appendix A.9
Lead (Pb)/(mg/kg)	≤	0.3	GB 5009.12 or GB 5009.75
Total arsenic (in As)/(mg/kg)	≤	0.2	GB 5009.11 or GB 5009.76

Appendix A

Testing Method

A.1 Safety warnings

Some of the reagents used in this testing method are toxic and corrosive, so the operation should be carried out carefully. If the reagent splashes onto the skin, it should be immediately rinsed with water, and in severe cases, it should be treated in hospital immediately. When using flammable materials, it is strictly prohibited to use fire for heating.

A.2 General provisions

Unless otherwise specified, the purity of the reagents used should be above analytical purity. The standard titration solution, the standard solution for impurity determination, preparations and products should be prepared in accordance with the provisions of GB/T601, GB/T602, and GB/T 603, respectively. Level III water should be used in experiments, that comply with the provisions in GB/T 6682. When the solvent used in the test is not specified, it refers to aqueous solution.

A.3 Identification tests

A.3.1 Reagents and materials

A.3.1.1 Ninhydrin

A.3.1.2 n-butanol

A.3.1.3 Acetic acid

A.3.1.4 Acetone

A.3.1.5 Methanol

A.3.1.6 L-Lysine Monohydrochloride, CAS: 657-27-2, purity is not less than 99.5%.

A.3.1.7 Sodium L (+) - aspartate monohydrate, CAS: 323194-76-9, purity is 99%.

A.3.1.8 L-Lysine L-aspartate, CAS: 27348-32-9, purity is not less than 98%.

A.3.1.9 Ninhydrin solution: take 0.1g of ninhydrin, dissolve with water, and bring to a constant volume of 100mL.

A.3.1.10 Developing agent: n-butanol: acetic acid: water=2:1:1.

A.3.1.11 Acetic acid - methanol solution of ninhydrin: weigh 1 g of ninhydrin, add 3.0 mL of acetic acid, then dissolve in methanol and dilute to 100 mL.

A.3.2 Instruments and equipment

A.3.2.1 Electronic balance, with a sensitivity of 0.001 g

A.3.2.2 Electric furnace

A.3.2.3 Drying oven

A.3.2.4 Chromatography cylinder

A.3.2.5 Silicone rubber plate GF_{254}

A.3.2.6 Infrared spectrum analyzer

A.3.3 Identification method

A.3.3.1 Identification of L-Lysine L-Aspartate salt

Weigh 0.1g (accurate to 0.001 g) of sample, add water to dissolve and bring to a constant volume of 100 mL. Take 5 mL of sample solution into a test tube, add 1 mL of ninhydrin solution, and heat in a boiling water bath for 3 minutes. The solution should be purple.

A.3.3.2 Identification of L-Lysine and L-Aspartic Acid

Weigh 0.1 g (accurate to 0.001 g) of L-Lysine hydrochloride standard product and 0.1 g (accurate to 0.001 g) of L-Aspartate sodium monohydrate standard product, add water to dissolve and bring to a constant volume of 100 mL. Use this mixed standard solution as a control

solution. Weigh 0.2 g (accurate to 0.001 g) of sample, add water to dissolve and bring to a constant volume of 100 mL, as the sample solution.
Absorb 5 µl of control solution and 5 µl of sample solution respectively, and place onto the same silica gel plate GF_{254}, and develop them in a chromatography cylinder with a developing agent for at least 15 cm. Take out and dry, and heat in a drying oven at 80℃ for 30 minutes. Spray the acetic acid methanol solution of ninhydrin and heat for 10 minutes in a drying oven at 80℃. Check immediately when spots appear.
Observe the silicone panel under natural light. Two main spots should be observed at the corresponding positions of the sample solution and the control solution.
A.3.3.3 Infrared identification
The determination of infrared spectral analysis shall be carried out in accordance with 6.2 in GB/T 32199. The spectrogram of the sample shall be compared with the standard spectrogram (see Figure B.1 in Appendix B), and the two spectrograms shall be basically consistent.

A.4 Determination of L-Lysine L-Aspartate content (on a dry basis)
A.4.1 Method summary
The sample is titrated with standard perchloric acid titration solution using formic acid and acetic acid as solvents and crystal violet as indicator. The content of L-Lysine L-Aspartate on $C_{10}H_{21}N_3O_6$ basis is calculated according to the consumed volume of the perchloric acid standard titration solution.
A.4.2 Reagents and materials
A.4.2.1 Formic acid
A.4.2.2 Acetic acid
A.4.2.3 Perchloric acid
A.4.2.4 Crystal violet
A.4.2.5 Standard perchloric acid titration solution: c ($HClO_4$) = 0.1mol/L.
A.4.2.6 Crystal violet - acetic acid indicator (5g/L): Weigh 0.5 g of crystal violet, dissolve in acetic acid, and dilute to 100 mL with acetic acid.
A.4.3 Instruments and equipment
A.4.3.1Electronic balance: the reciprocal sensibility is 0.0001 g.
A.4.4 Analysis steps
Weigh 0.2g (accurate to 0.0001g) of the sample into a conical flask, add 3 mL of formic acid and 50 mL of acetic acid, and dissolve and mix well.
Add 3 drops of crystal violet - acetic acid indicator and titrate with 0.1mol/L standard perchloric acid titration solution. The color of the solution changes from purple to blue finally to green. A blank test should be conducted at the same time.
A.4.5 Calculation of Results
The mass fraction W_1 of L-Lysine L-Aspartate (calculated on a dry basis) is calculated according to formula (A.1).

$$w_1 = \frac{(V_1 - V_0) \times c \times 9.310}{m \times 100} \times \frac{100}{100 - w_2} \times 100\% \qquad \text{(A.1)}$$

Where:
V_1 - Consumed volume of standard perchloric acid titration solution by the sample solution, in milliliters (mL).

V_0 - Consumed volume of standard perchloric acid titration solution by blank solution, in milliliters (mL).
c - Concentration of standard perchloric acid titration solution, in mole per liter (mol/L).
9.310 - Every 1 mL of 0.1 mol/L standard perchloric acid titration solution is equivalent to 9.310 mg of L-Lysine L-Aspartate ($C_{10}H_{21}N_3O_6$).
m - mass of the sample, in grams (g).
100 - Conversion factor.
W_2 - Drying loss of sample, %.
The calculation result is expressed as the arithmetic mean of two independent measurement results obtained under repeatable conditions, with the result is expressed in tenths/.x. The absolute difference between two independent measurement results obtained under repeatable conditions shall not exceed 2% of the arithmetic mean.

A.5 Determination of specific rotation
A.5.1 Reagents and materials
A.5.1.1 Hydrochloric acid: 36%~38%
A.5.1.2 Hydrochloric acid solution (6 mol/L): Measure 100 mL of hydrochloric acid and dilute to 200 mL with water.
A.5.2 Instruments and equipment
A.5.2.1 Electronic balance, with a sensitivity of 0.001 g.
A.5.2.2 Polarimeter: with an accuracy of ± 0.001°, use a sodium lamp (sodium spectral D-line is 589.3 nm) as the light source.
A.5.3 Analysis steps
Weigh 4g (accurate to 0.001 g) of the sample after vacuum drying (according to the method in A.8), add 6 mol/L of hydrochloric acid solution for dissolution, and adjust the solution temperature to 20℃. Transfer into a 50 mL volumetric flask, dilute with 6 mol/L of hydrochloric acid solution, and shake well. Calculate according to the method specified in GB/T 613 and calculate the specific rotation of L-lysine L-Aspartate.
A.5.4 Calculation of Results
The specific rotation αm (20℃,D) of L-lysine L-Aspartate, expressed in "(°)·dm^2·kg^{-1}" and is calculated according to formula (A.2):

$$\alpha_m(20℃, D) = \frac{\alpha}{l \times \rho} \quad (A.2)$$

Where:
a - The optical rotation measured by the polarimeter, in degrees (°).
l - The length of the optical rotation tube, in decimeters (dm).
p - Mass concentration of L-lysine aspartate per 100 mL of solution (calculated on a dry basis), in grams per milliliter liter (g/mL).

A.6 pH measurement
A.6.1 Instruments and equipment
A.6.1.1 Electronic balance, with a sensitivity of 0.001 g.
A.6.1.2 Acidity meter
A.6.2 Analysis steps

Weigh 1.0 g of the sample (accurate to 0.001 g), add 20 mL of water to dissolve, and measure the pH value using a pH meter according to GB/T 9724.

A.7 Determination of drying loss

A.7.1 Instruments and equipment

A.7.1.1 Electronic balance: with a sensitivity of 0.0001g.

A.7.1.2 Weighing bottle.

A.7.1.3 Vacuum drying oven.

A.7.2 Analysis steps

Weigh 1~2 g of the sample (accurate to 0.0001 g) and place it in a weighing bottle that has been dried to a constant weight (105℃ ± 2℃ for 30 minutes). Spread the sample evenly in the weighing bottle, dry at temperature of 105℃ ± 2℃ and under vacuum pressure below 2.67 KPa for 3 hours. Place the weighing bottle in a dryer to cool until the temperature reaches to room temperature, then weigh when it reaches to a constant weight.

A.7.3 Calculation of Results

The mass fraction w_2 of the drying loss of L-Lysine L-Aspartate is calculated according to formula (A.3):

$$w_2 = \frac{m_1 - m_2}{m_1 - m_0} \times 100\% \qquad \text{(A.3)}$$

Where:

m_1——the mass of the weighing bottle and the sample, in grams (g).

m_2——the mass of the weighing bottle and sample after drying, in grams (g).

m_0——the mass of the weighing bottle, in grams (g).

The calculation result is expressed as the arithmetic mean of two independent measurement results obtained under repeatable conditions and the result is expressed in hundredths/.xx. The absolute difference between two independent measurement results under repeatable conditions shall not exceed 10% of the arithmetic mean.

A.8 Determination of ignition residue

A.8.1 Reagents and materials

Sulfuric acid: 98%.

A.8.2 Instruments and equipment

A.8.2.1 Electronic balance: with a sensitivity of 0.0001 g.

A.8.2.2 Muffle furnace.

A.8.2.3 Porcelain crucible.

A.8.2.4 Dryer.

A.8.3 Analysis steps

Weigh 1~2 g of the sample (accurate to 0.0001 g) into a porcelain crucible that has been heated to constant weight, add a few drops of sulfuric acid to moisten the sample, and place it on an electric furnace. Slowly heat it, carefully carbonize until the sample turns completely black then cool. Add 1 mL of sulfuric acid and heat until it is smokeless. Place the crucible in a muffle furnace at temperature of 550℃ ± 25℃ and heat it for about 3 hours to completely incinerate the sample. When the furnace temperature drops to 200℃, take it out and put it into a dryer to cool

till it reaches room temperature then weigh it. Put it into a muffle furnace and heat it at 550℃ ± 25℃ for 30 minutes, then cool it as previous cooling step and weigh it. Repeat the steps until the difference between two consecutive weighs do not exceed 0.5 mg and then record the smallest mass.

A.8.4 Calculation of Results

The mass fraction w_3 of the residue from the ignition of L-Lysine L-Aspartate is calculated according to formula (A.4):

$$w_3 = \frac{m_4 - m_3}{m_5 - m_3} \times 100\% \qquad \text{(A.4)}$$

Where:

m_4 - Mass of the crucible and sample after heating, in grams (g).

m_3 - Mass of the crucible, in grams (g).

m_5 - Mass of the crucible and sample, in grams (g).

The calculation result is expressed as the arithmetic mean of two independent measurement results under repeatable conditions, with the result expressed in hundredths/.xx.. The absolute difference between two independent measurement results under repeatable conditions shall not exceed 5% of the arithmetic mean.

A.9 Determination of chloride (calculated as Cl)

A.9.1 Reagents and materials

A.9.1.1 Hydrochloric acid: 36%~38%.

A.9.1.2Nitric acid.

A.9.1.3 Silver nitrate.

A.9.1.4 Standard hydrochloric acid titration solution: c (HCl)=0.01 mol/L.

A.9.1.5 Nitric acid solution: Measure 105 mL of nitric acid and dilute to 1,000 mL with ultrapure water.

A.9.1.6 Silver nitrate solution (0.1mol/L): Weigh 1.7g of silver nitrate, dissolve it with ultrapure water until a constant volume of 100mL.

A.9.2 Instruments and equipment

A. 9. 2.1 Electronic balance: with a sensitivity of 0.001 g.

A.9.3 Analysis steps

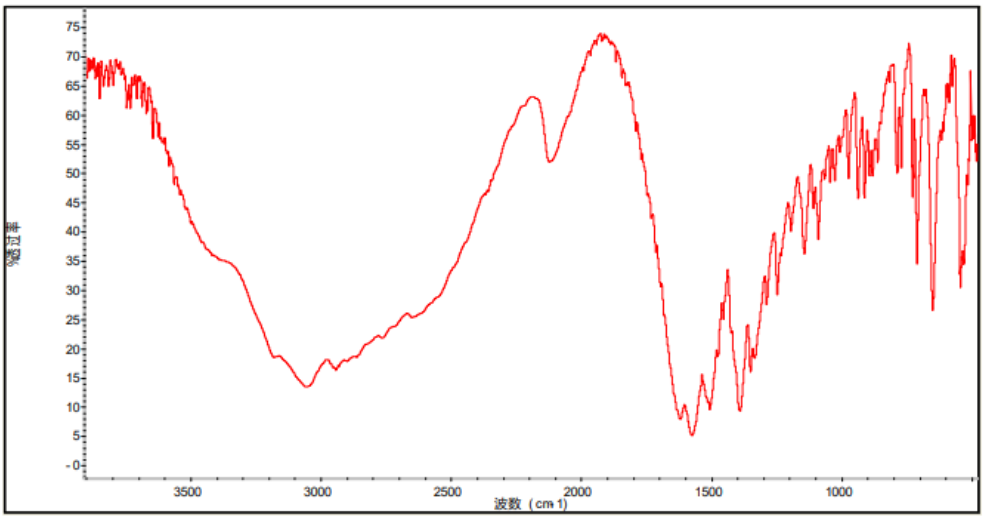
Weigh 0.3 g (accurate to 0.001 g) of the sample and place it in a 50 mL Nessler's colorimetric tube. Add 10 mL of nitric acid solution to dissolve and dilute to 50 mL with ultrapure water as the sample solution. At the same time, accurately pipette 0.35 mL of 0.01mol/L standard hydrochloric acid titration solution into another 50 mL Nessler's colorimetric tube, add 10 mL of nitric acid solution, and dilute to 50 mL with ultrapure water as a control solution. Add 1 mL of silver nitrate solution to the sample solution and the control solution, shake well, and set aside in a dark place for 5 minutes. Place the two tubes on a black background under natural light, and visually compare the turbidity of the two tubes from top to bottom. The turbidity of the sample solution tube is not deeper than that of the control tube, that is, the chloride content in the sample is less than or equal to 0.041%.

Appendix B

Reference Infrared Spectrogram of Standard L-Lysine L-Aspartate

The reference infrared spectrum of standard L-Lysine L-Aspartate is as shown in Figure B.1.

Figure B.1: Infrared spectrogram of standard L-Lysine L-Aspartate



Attachments:

No Attachments.