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National food safety standards

Food nutrition enhancer inositol (cyclohexanol)

(draft for comments)

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National Health and Wellness Committee of the People's Republic

Release

State Market Supervisory Administration

National food safety standards

Food nutrition enhancer inositol (cyclohexanol)

1 Scope

This standard applies to the food nutrition enhancer inositol (cyclohexanol) produced by hydrolysis of calcium phytate (phenantine).

2 Molecular formula, structural formula and relative molecular mass

2.1 Molecular formula

$C_6H_{12}O_6$

2.2 Structure

2.3 Relative molecular mass

180.16 (according to 2016 international relative atomic mass)

3 Technical requirements

3.1 Sensory requirements

Sensory requirements should be in accordance with Table 1.

Table 1 Sensory requirements

item	Head	Want begging	Testing method
Color	White or white		Take an appropriate amount of the sample in a clean, dry white porcelain dish and observe it under na
status	Crystalline powder		Color and state

3.2 Physical and chemical indicators

Physical and chemical indicators should meet the requirements of Table 2.

Table 2 Physical and chemical indicators

item	Head		Means	Standard	Testing method
Inositol ($C_6H_{12}O_6$) content (on dry basis), *w* /%			97.0~101.0		Appendix A, A.3
Calcium (Ca)			Pass the test		Appendix A, A.4
Chloride (calculated as Cl^-), *w* /%		≤	0.005		Appendix A, A.5
Sulfate (with SO_4^{2-} meter), *w* /%		≤	0.006		Appendix A, A.6
Dry reduction, *w* /%		≤	0.5		GB 5009.3 direct drying method [a]
Melting point, °C			224~227		GB/T 617
Burning residue, *w* /%		≤	0.1		Appendix A, A.7
Lead (Pb)/mg/kg		≤	1.0		GB 5009.12 First Act
Total arsenic (as As) / mg / kg		≤	1.0		GB 5009.11 The first second law

a drying temperature is 105 ° C, drying time is 4 h.

Appendix A

Testing method

A.1 General provisions

The reagents and water used in this standard refer to the analytical reagents and the tertiary water specified in GB/T 6682 when no other requireme
Used in the test.
Standard solutions, standard solutions for determination of impurities, preparations and products are not included in other requirements, according to G
Preparation of the provisions. The solution used in the test refers to an aqueous solution when it is not indicated which solvent is used.

A.2 Identification test

A.2.1 Reagents and materials

A.2.1.1 Ethanol.

A.2.1.2 Ether.

A.2.1.3 Chloroform.

A.2.1.4 Nitric acid.

A.2.1.5 Barium acetate solution: 100 mg/mL. Weigh 1 g of cesium acetate, dissolve in water, and dilute to 10 mL.

A.2.1.6 litmus paper.

A.2.2 Instruments and equipment

A.2.2.1 Electronic balance: Sensing amount 0. 01 g.

A.2.2.2 polarimeter.

A.2.2.3 Melting point apparatus.

A.2.2.4 Water bath: The maximum display temperature is 100 °C.

A.2.3 Identification method

A.2.3.1 Acidity and alkalinity

Weigh about 1 g of sample, accurate to 0.01 g, dissolve in water, and dilute to 50 mL to obtain a sample solution, put the litmus paper into the solu
The color of the test paper should be unchanged.

A.2.3.2 Optical rotation

Weigh about 1 g of sample to the nearest 0.01 g, dissolve in water, and dilute to 100 mL. Determined by polarimeter at 25 °C.

The degree should be -0.10 ° ~ +0.10 ° .

A.2.3.3 Solubility

Weigh about 1 g of sample to the nearest 0.01 g, place in a beaker, add water, ethanol, ether and chloroform reagent separately, shake time
Not less than 30 seconds, and observe the dissolution of the sample within 5 minutes.
The sample is soluble in water, very slightly soluble in ethanol, and insoluble in ether and chloroform.

A.2.3.4 Identification test of barium acetate

Approximately 1 g of the sample was weighed to the nearest 0.01 g and dissolved in 50 mL of water to obtain a sample solution.

Take 1 mL of sample solution to the porcelain evaporating dish,
Add 0 mL of nitric acid, evaporate to a 100 °C water bath, dissolve the residue in 1 mL of water, add 0.5 mL of cesium acetate solution, and evaporate
Violet color.

A.2.3.5 hexaacetylinositol residue melting point or liquid chromatography

This identification experiment was selected according to the method selected by the content test.

When the content test is carried out by weight method, the residue of hexaacetylinositol obtained in the process is baked at 105 ° C for 0.5 h, and tl
Determination, its melting point should be: 212 ~ 216 °C.

When the content test is performed by high performance liquid chromatography, the retention time of the main peak of the sample solution should
See Appendix B for the spectrum.

A.3 Inositol content

A.3.1 Gravimetric method

A.3.1.1 Principle of the method

Inositol produces hexaacetylinositol dissolved in chloroform and insoluble in water under acidic conditions with acetic anhydride, depending on th
The amount is converted to the mass of inositol.

A.3.1.2 Reagents and materials

A.3.1.2.1 Acetic anhydride sulfuric acid solution: Take 2 mL of 1 mol/L sulfuric acid, slowly add dropwise to 100 mL of acetic anhydride, and let cool.

A.3.1.2.2 Trichloromethane.

A.3.1.3 Instruments and equipment

A.3.1.3.1 Electronic balance: Sensitivity 0. 0001 g.

A.3.1.3.2 Water bath: The maximum display temperature is 100 °C.

A.3.1.3.3 Beaker: 250 mL.

A.3.1.3.4 Surface dish.

A.3.1.3.5 Separating funnel

A.3.1.3.6 Filtering device.

A.3.1.3.7 Constant temperature drying oven.

A.3.1.4 Determination method

Weigh approximately 0.2 g of dry sample to the nearest 0.0001 g, place in a 250 mL beaker, and add 5 mL of acetic anhydride sulfuric acid solutio
Cover the watch glass, water bath at 100 °C for 20 min, remove the ice bath and cool, add 100 mL water slowly, boil for 20 min, remove the ice bath ar
The solution was transferred to a 250 mL separatory funnel, the beaker was washed with a little chloroform and the washings were combined in a sep. f
Extraction and extraction with chloroform
6 times, each time the amount of chloroform is 30 mL, 25 mL, 20 mL, 15 mL, 10 mL and 10 mL, combined with the chloroform layer in another
In a 250 mL separatory funnel, 10 mL of water was added to shake and stand, and the chloroform layer was collected by filtration through absorbent co
Add 10 mL to the separatory funnel
Trichloromethane was shaken and allowed to stand. The chloroform layer was filtered with a filter and a cotton wool, and the chloroform layer was con
Placed in a weighed conical flask.
The chloroform was evaporated and dried at 105 ° C to constant weight.
The weight of the obtained residue was multiplied by 0.4167 to obtain the inositol content of the sample. This residue can be used in A2.3.5
Identification test.

A.3.1.5 Calculation of results

Inositol content w_1 , calculated as mass fraction, calculated according to formula (1):

$$= ( ) \times . \times 100\% \quad \text{.....(1)}$$

In the formula:

m_1 - the mass of the conical flask and hexaacetylinositol after drying constant weight, in grams (g);

m_2 - the constant weight mass of the conical flask, in grams (g);

0.4167 - the coefficient of conversion of hexacyanoinositol to inositol;

m - the mass of the sample in grams (g).

The experimental results are based on the arithmetic mean of the parallel determination results.

The absolute difference between two independent determinations obtained under repetitive conditions is not
More than 2.0%.

A.3.2 High performance liquid chromatography

A.3.2.1 Method summary

The sample was dissolved in water and detected by high performance liquid chromatography. The external standard was quantified by a single

A.3.2.2 Reagents and materials

A.3.2.2.1 Distilled water: Level 1 water in accordance with GB/T 6682.

A.3.2.2.2 Acetonitrile: chromatographically pure.

A.3.2.2.3 Inositol standard: purity ≥ 98.5%.

A.3.2.3 Instruments and equipment

A.3.2.3.1 Electronic balance: The sensitivity is 0. 0001 g.

A.3.2.3.2 High performance liquid chromatograph with differential detector.

A.3.2.4 Chromatographic conditions

A.3.2.4.1 Column: Calcium-type strong cation exchange column (350 mm × 7.8 mm, particle size 9 μm), or equivalent analytical column.

A.3.2.4.2 Mobile phase: water.

A.3.2.4.3 Flow rate: 0.5 mL/min.

A.3.2.4.4 Injection volume: 10 μL.

A.3.2.4.5 Column temperature: 85 °C.

A.3.2.4.6 Differential detector temperature: 35 °C.

A.3.2.5 Analysis steps

A.3.2.5.1 Preparation of standard solution

Accurately weigh 0.1 g of inositol standard to 0.0001 g, place in a 100 mL volumetric flask, dissolve in water and dilute to volume.

A.3.2.5.2 Preparation of sample solution

Accurately weigh 0.1 g of dry sample to the nearest 0.0001 g, place in a 100 mL volumetric flask, dissolve in water and dilute to volume.

A.3.2.6 Determination

Under the reference chromatographic conditions of A.3.2.4, separate the sample solution and the standard solution, repeat the injection twice, and t

The peak area value of inositol in the figure.

Calculate the inositol content of the sample according to the formula in the calculation of the results of A.3.2.7.

A.3.2.7 Calculation of results

Inositol content w_2, calculated as mass fraction, calculated according to formula (2):

$$= \frac{\times \times}{\times} \times 100\% \quad \text{.....(2)}$$

In the formula:

A_1 - the average peak area value of inositol in the chromatogram of the sample solution;

m_4 - the mass of inositol in the standard solution, in grams (g);

w_3 - the purity of the standard, %;

A_2 - the average peak area value of inositol in the chromatogram of the standard solution;

m_3 - the mass of the sample in grams (g);

The experimental results are based on the arithmetic mean of the parallel determination results.

The absolute difference between two independent determinations obtained under repetitive conditions is not

More than 2.0%.

A.4 Calcium

A.4.1 Reagents and materials

Ammonium oxalate solution: Weigh 3.5 g of ammonium oxalate, dissolve in water, and dilute to 100 mL.

A.4.2 Analysis steps

Weigh 1.0 g of the sample to the nearest 0.01 g, add 10 mL of water to dissolve, shake well, add 1 mL of ammonium oxalate solution, and should r

A.5 Chloride

A.5.1 Method summary

Under acidic conditions, the chloride ion and the silver nitrate solution in the inositol solution form a white silver chloride precipitate, which is turl

degree.

A.5.2 Reagents and materials

A.5.2.1 Nitric acid solution: 1.7 mol/L. Take 105 mL of nitric acid and dilute to 1000 mL with water.

A.5.2.2 Silver nitrate solution: 0.1 mol/L. 17.5 g of silver nitrate was weighed, dissolved in water and made up to 1000 mL.

A.5.2.3 Sodium chloride standard solution: Weigh accurately 165 mg of sodium chloride, dissolve in water and dilute to 100 mL. Precisely measure 10.0 mL of the above chlorine Sodium solution, diluted with water and made up to 1000 mL. 10 μg of Cl^- per 1 mL .

A.5.3 Analysis steps

Weigh the sample about 0.4 g to the nearest 0.0001 g, place it in a 50 mL Nessler colorimetric tube, dissolve it with 20 mL to 30 mL of water, add Nitric acid solution and 1 mL silver nitrate solution, make up to 50 mL with water. Shake slowly and leave it in the dark for 5 min.

At the same time, take 2 mL of sodium chloride standard solution, place it in a 50 mL Nessler colorimetric tube, add 10 mL of nitric acid solution Add water to a volume of 50 mL. Shake slowly and leave it in the dark for 5 min.

Place the two on the same black background, and observe the turbidity of the sample solution from the top of the colorimetric tube. The turbidity o The chloride is less than 0.005%.

A.6 Sulfate

A.6.1 Principle of the method

Under acidic conditions, the sulfate ion in the inositol solution and the barium chloride solution form barium sulfate precipitate, which is turbid by degree.

A.6.2 Reagents and materials

A.6.2.1 Hydrochloric acid solution: 2.7 mol/L. Pipette 226 mL of hydrochloric acid and dilute to 1000 mL with water.

A.6.2.2 Barium chloride solution: Weigh 12.0 g of barium chloride, dissolve it in water and dilute to 100 mL.

A.6.2.3 Standard sulfate solution: Weigh accurately 148 mg of anhydrous sodium sulfate, dissolve in water and dilute to 100 mL. Precise measurement on 10.0 mL The solution was diluted with water and made up to 1000 mL. 10 μg SO_4^{2-} per 1 mL

A.6.3 Analysis steps

Weigh 5 g of the sample to the nearest 0.01 g, place it in a 50 mL Nessler colorimetric tube, dissolve it with 20 mL to 30 mL of water, add 1 mL o Solution and 3 mL cesium chloride solution, make up to 50 mL with water. Shake slowly and let stand for 10 min.

At the same time, take 30 mL of the sulfate standard solution, place it in a 50 mL Nessler colorimetric tube, add 1 mL of hydrochloric acid solutio Add water to a volume of 50 mL. Shake slowly and let stand for 10 min.

Place the two on the same black background, and observe the sample solution from the top of the colorimetric tube. The turbidity should not be deeper than the control solution, ie the sulfate in the sample is less than 0.006%.

A.7 Burning residue

A.7.1 Reagents and materials

sulfuric acid.

A.7.2 Instruments and equipment

A.7.2.1 Muffle furnace.

A.7.2.2 Electric furnace.

A.7.2.3 坩埚.

A.7.3 Analysis steps

Weigh 2 g of the sample to the nearest 0.0001 g, place it in a crucible that has been ignited to constant weight, and slowly heat it in an electric furn Soak the smoke and cool it.

The residue is immersed in an appropriate amount of sulfuric acid and heating is continued until the sulfuric acid vapor has escaped. Burn for 15 min and keep to constant weight and weigh. Place the crucible in a muffle furnace and burn at 800 °C ± 25 °C

A.7.4 Calculation

The mass fraction $w_{4\ of\ the}$ burning residue is calculated according to formula (3):

$$= \times 100\% \quad \text{.....(3)}$$

In the formula:

m_5 - the mass of the sputum and residue, in grams (g);

m_6 ——the mass of 坩埚, in grams (g);

m_7 - the mass of the sample in grams (g).

The test results are based on the arithmetic mean of the parallel determination results.

The absolute difference between two independent determinations obtained under repetitive conditions is not Greater than 2% of the arithmetic mean.

Appendix B

Inositol liquid chromatogram

The liquid chromatogram of the inositol standard is shown in Figure B.1.

Figure B.1 Inositol standard liquid chromatogram

The liquid chromatogram of the inositol sample is shown in Figure B.2.

Figure B.2 Liquid chromatogram of inositol samples