



**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB 1886.239-2016

Replacing GB 1975-2010

National Food Safety Standard -- Food Additives -- Agar

食品安全国家标准 食品添加剂 琼脂

(English Translation)

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1 Scope

This standard applies to raw licorice (legume licorice *Glycyrrhiza uralensis* Fisch., *Glycyrrhiza inflata* *Glycyrrhiza inflata* Bat. and *Glycyrrhiza glabra* *Glycyrrhiza glabra* L.), mentioned by the flooding, sulfuric acid (or hydrochloric acid) acidified precipitation, ethanol extraction, amide, knot Crystal and other processes to prepare food additive ammonium glycyrrhizinate.

2.1 Chemical Name

Ammonium glycyrrhizinate

2.2 Formula

Formula 2.3

C₄₂H₆₁O₁₆NH₄

2.4 Relative molecular mass

839.96 (according to 2007 international relative atomic mass)

3.1 Sensory requirements

Sensory requirements shall comply with the requirements of Table 1.

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Table 1 Sensory requirements

Project requires test methods Color white, yellowish-white or pale yellow Status powdery

fine particles Take the right amount of sample is placed in a clean, dry white porcelain plate , Under natural light, observe its color and status

3.2 Physical and Chemical Indicators

Physical and chemical indicators should be consistent with the provisions of Table 2.

Table 2. Physical and chemical indicators

Item Index Test Method Licorice ammonium content (18beta- licorice ammonium meter, dry basis), w /% ≥ 65.0 Appendix A A.4 Loss on drying, w /% ≤ 7.0 Appendix A A.3 Specific rotation $[\alpha]_{20}^D$ $[(^{\circ}) \cdot \text{mL} \cdot \text{dm}^{-1} \cdot \text{g}^{-1}]$ 45 ~ 53 in Appendix A A.5 Ash, w /% ≤ 0.5 A.6 in Appendix A Lead (Pb)/(mg/kg) ≤ 2.0 Appendix A A.7 Total arsenic (As)/(mg/kg) ≤ 2.0 Appendix A A.8 GB 1886.242-2016 Appendix A Testing method A.1 General Provisions this standard reagents and water, did not indicate when the other requirements, refer to the three water analytical reagent and GB/T 6682 regulations. test Test used in the standard solution, impurity standard solution, preparations and products, did not indicate when the other requirements, according to GB/T 601, GB/T 602, GB/T 603 provisions prepared. Solution was used in the tests did not indicate what is formulated with solvent, it refers to an aqueous solution.

A.2 Identification Test A.2.1 Method summary After the case of alkali salts to produce ammonia, with ammonia and volatile basic characteristics of the top tube to make moist pH paper was a basic color, Kam the presence of other salts.

A.2.2 Reagents and materials A.2.2.1 sodium hydroxide solution. 40g/L.

A.2.2.2 pH test paper.

A.2.3 Identification method Take a sample of about 0.2g ammonium licorice in 10mL tube, add about 1mL sodium hydroxide solution (A.2.2.1), shaking, quickly has Water wet pH paper into the mouth of the tube, to produce ammonia and volatile wet pH paper was to make a basic color.

Note: If the solution is heated, the color reaction is more obvious.

A.3 Determination of loss on drying A.3.1 Instruments and Equipment A.3.1.1 vacuum drying oven.

A.3.1.2 weighing bottle.

A.3.1.3 dryer.

A.3.2 Procedure

The weighing bottle placed in a vacuum oven, $78^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$, dried under 20mm Hg 30min, taken out into the dryer the cooled to room temperature, and weighed. Weigh 1g sample, accurate to 0.0001g, stoppered and weighed. Gently shake the horizontal lateral weighing

bottle, Uniform distribution layer as a sample, is less than 5mm. The paved specimen weighing bottle placed in a vacuum oven, open the cork, in at $78\text{ }^{\circ}\text{C} \pm 0.5\text{ }^{\circ}\text{C}$, 20mmHg conditions, drying under reduced pressure 6h, out into a dryer, stoppered, allowed to cool to room temperature, and weighed.

A.3.3 Calculation Results Loss on drying mass fraction w_1 , according to equation (A.1) Calculated.

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$m_1 - m_2$

$m \times 100\%$

Where.

m_1 --- vacuum dried weighing bottle before adding the sample mass, in grams (g);

m_2 --- dried under reduced pressure weighing bottle plus sample mass, in grams (g);

Numerical m --- sample mass, in grams (g).

The results parallel arithmetic mean of the measurement results shall prevail (reserved a decimal). In two independent test repeated conditions Given absolute difference is not more than 1.0%.

A.4 Determination of licorice ammonium content (18beta- licorice ammonium meter, dry basis) of A.4.1 Method summary the sample was acetonitrile - water dissolved, filtered, measured by liquid chromatography (UV detector), external standard. When the calculation results, the sample Weigh the sample by subtracting the mass of the sample water quality, the result is a dry basis ($\text{C}_{42}\text{H}_{65}\text{NO}_{16}$) Total mass fraction of ammonium glycyrrhizinate.

A.4.2 Reagents and materials A.4.2.1 acetonitrile. HPLC grade.

A.4.2.2 glacial acetic acid. pure class distinctions.

A.4.2.3 0.45 μm membrane (organic phase).

A.4.2.4 licorice ammonium Standard. Purity $\geq 90\%$ (HPLC or logo).

Note 1: The licorice ammonium standard method used was ammonium licorice 18beta- single component standard.

Note 2: The standard non-aqueous titration (NT) or other non-HPLC method for the determination of identity, its content can not be directly used to calculate the value of liquid chromatography content.

A.4.2.5 acetonitrile - water. acetonitrile and water mixed in equal volumes.

A.4.2.6 acetonitrile - acetic acid - water. glacial acetic acid aqueous acetonitrile (38 161), mix and filter through 0.45 μm (organic phase) is too Filter, fully degassed.

A.4.3 Instruments

A.4.3.1 performance liquid chromatography with UV detection.

A.4.3.2 balance, a sense of the amount of 0.01mg.

A.4.3.3 vacuum drying oven.

A.4.4 Analysis step

A.4.4.1 Standard Solution Weigh 12.5mg licorice ammonium standard, accurate to 0.01mg, placed in 25mL volumetric flask with acetonitrile - water (A.4.2.5) solution Solution, and to volume. Through 0.45μm membrane (organic phase) and filtered. Using now.

A.4.4.2 Preparation of sample solution Weigh 50.0mg licorice ammonium samples, accurate to 0.01mg, placed in 100mL flask with acetonitrile - water (A.4.2.5) solution Solution to the mark by 0.45μm membrane (organic phase) filter, tested.

A.4.4.3 reference chromatographic conditions A.4.4.3.1 Column. C18 column, length 150mm, internal diameter 4.6mm, particle size 5μm. Or equivalent separation column.

A.4.4.3.2 mobile phase. acetonitrile - glacial acetic acid - water (A.4.2.6).

A.4.4.3.3 flow rate. 1.0mL/min.

A.4.4.3.4 column temperature. room temperature.

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A.4.4.3.5 Injection volume. 10μL.

A.4.4.4 Determination Followed by injection of licorice ammonium standard solution and sample solution was measured at a wavelength of 254nm at the external standard. Licorice ammonium HPLC Chromatograms and retention time, see Appendix B.

A.4.5 Calculation Results Licorice ammonium content (18beta- licorice ammonium meter, dry basis) of the mass fraction w₂, according to equation (A.2) Calculated.

$V \times c_s \times A$

$m_3 \times (1-w_1) \times A_s \times$

100% (A.2)

Where.

V --- volume of the sample volume, in milliliters (mL);

c_s --- licorice ammonium standard solution concentration, in milligrams per milliliter (mg/mL);

A --- sample solution chromatogram peak area licorice ammonium;

Numerical m₃ --- sample mass, in milligrams (mg);