GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

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National food safety standard - Food additive - Cellulose

食品安全国家标准 食品添加剂 纤维素

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1 Scope

This standard applies to food additive cellulose, which is obtained by purifying and mechanically crushing fibrous plant pulp containing α-cellulose as raw material.

2 Chemical name, molecular formula, structural formula, relative molecular mass

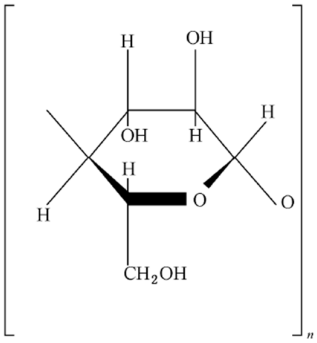
2.1 Chemical name

Cellulose

2.2 Molecular formula

$(C_6H_{10}O_5)_n$

2.3 Structural formula



2.4 Relative molecular mass

$(162.14)_n$ (according to the 2018 international relative atomic mass)

3 Technical requirements

3.1 Sensory requirements

Sensory requirements shall comply with the provisions of Table 1.

Appendix A
Test method

A.1 Safety tips

Some reagents used in the test method of this standard are toxic or corrosive. Appropriate safety and health measures shall be taken during operation. If splashed on the skin, rinse with water immediately. In severe cases, treatment shall be given immediately. When using flammable materials, it is strictly forbidden to use open flames for heating.

A.2 General provisions

The reagents and water used in this standard refer to analytical pure reagents and grade 3 water specified in GB/T 6682, unless otherwise specified. The standard titration solution, standard solution for impurity determination, preparations and products used in the test shall be prepared, in accordance with the provisions of GB/T 601, GB/T 602, GB/T 603, unless otherwise specified. The solutions used in the test refer to aqueous solutions unless otherwise specified.

A.3 Identification test

A.3.1 Solubility test

Insoluble in water, ethanol, ether, diluted inorganic acid. Slightly soluble in 1 mol/L sodium hydroxide solution.

A.3.2 Suspension test

Mix 30 g of specimen with 270 mL of water. Stir the mixture with a high-speed stirrer (about 12000 r/min) for 5 min. The final solution is a suspension with poor fluidity, thick, lumpy matter. If a free-flowing suspension is obtained, take 100 mL of the solution; place it in a 100 mL measuring cylinder; let it stand for 1 h; then a precipitate will appear at the bottom of the measuring cylinder.

A.3.3 Infrared spectrum identification

The test is carried out according to GB/T 6040 using the potassium bromide tablet method. The infrared spectrum of the specimen shall be consistent with the standard reference spectrum of cellulose (see Appendix B).

A.3.4 Polymerization test

A.3.4.1 Reagents and materials

Ethylenediamine copper dihydroxide solution: 1.0 mol/L.

A.3.4.2 Instruments and equipment

A.3.4.2.1 Balance: Sensitivity is 0.1 mg.

A.3.4.2.2 Constant temperature water bath (tank).

A.3.4.2.3 Ubbelohde viscometer: Capillary inner diameters are 0.5 mm ~ 0.6 mm and 0.7 mm ~ 0.8 mm, respectively.

A.3.4.3 Analytical procedures

Weigh about 0.25 g (m_1) of specimen. Place it in a stoppered conical flask. Add 25 mL of water and 25 mL of 1.0 mol/L copper dihydroxide solution, respectively. Add the stopper and shake to completely dissolve. Transfer an appropriate amount of the solution to an Ubbelohde viscometer (capillary inner diameter 0.7 mm ~ 0.8 mm; select an appropriate viscometer constant K_1). Balance it in a 25 °C ± 0.1 °C water bath for at least 5 min. Record the time the solution flows through the upper and lower scales of the Ubbelohde viscometer (do not reload the specimen; repeat the measurement twice; the difference in the flow time of the two measurements shall not exceed ±0.5% of the average value). Take the average of the two measurements as the outflow time t_1 of the solution. Calculate the kinematic viscosity ν_1 of the solution according to formula (A.1):

$$\nu_1 = t_1 \times K_1 \qquad \cdots\cdots (A.1)$$

Where:

t_1 - The time the solution flows through the upper and lower scales of the Ubbelohde viscometer, in seconds (s);

K_1 - Viscometer constant.

Take an appropriate amount of 0.1 mol/L copper dihydroxide ethylenediamine solution. Mix it with an equal amount of water. Use an Ubbelohde viscometer (capillary inner diameter 0.5 ~ 0.6 mm; select an appropriate viscometer constant K_2), to determine the outflow time t_2 in the same way. The kinematic viscosity ν_2 of the blank solution is calculated according to formula (A.2):

$$\nu_2 = t_2 \times K_2 \qquad \cdots\cdots (A.2)$$

Where:

t_2 - The time for the solution to flow through the upper and lower scales of the Ubbelohde viscometer, in seconds (s);

K_2 - Viscometer constant.

Calculate the relative viscosity η_{rel} according to formula (A.3):

Weigh about 125 mg of specimen (m_2), accurate to 0.1 mg. Use about 25 mL of water, to transfer it into a 300 mL conical flask. Add 50.0 mL of potassium dichromate solution (A.4.2.2). Mix well. Then slowly add 100 mL of sulfuric acid (A.4.2.1). Heat to boiling. Remove the heat source. Let it stand at room temperature for 15 minutes. Cool it in water bath. Transfer it into a 250 mL volumetric flask. Use water to dilute it to close to the mark. Cool to room temperature. Use water to dilute it to the mark. Mix well. Measure 50.0 mL of the solution. Add 2 ~ 3 drops of 1,10-phenanthroline-ferrous indicator solution (A.4.2.4). Use 0.1 mol/L ammonium ferrous sulfate standard titration solution (A.4.2.3) to titrate it, until the reddish brown color appears, which is taken as the end point. Record the consumed volume. At the same time, perform a blank test. Record the consumed volume of 0.1 mol/L ammonium ferrous sulfate standard titration solution (A.4.2.3).

A.4.5 Calculation of results

The mass fraction w_2 of cellulose content (measured in $C_6H_{10}O_5$, on a dry basis) is calculated according to formula (A.5):

$$w_2 = \frac{c \times (V_3 - V_4) \times 6.75}{m_2 \times \frac{50}{250} \times (1 - w_1)} \times 100\% \quad \cdots\cdots (A.5)$$

Wherein:

c - Concentration of standard ammonium ferrous sulfate titration solution, in mol/L;

V_3 - Volume of standard ammonium ferrous sulfate titration solution consumed in the blank test, in milliliters (mL);

V_4 - Volume of standard ammonium ferrous sulfate titration solution consumed by the specimen, in milliliters (mL);

m_2 - Mass of the specimen, in milligrams (mg);

w_1 - Loss on drying of the specimen, %;

6.75 - Number of milligrams of $C_6H_{10}O_5$ equivalent to each millimole of standard ammonium ferrous sulfate titration solution $\{c[(NH_4)_2Fe(SO_4)_2]\}$;

50 - Volume of specimen solution pipetted, in milliliters (mL);

250 - Volume of specimen solution fixed, in milliliters (mL).

The test result shall be based on the arithmetic mean of the results of parallel determinations. The absolute difference between two independent determination results, as obtained under repeatability conditions, shall not be greater than 1% of the arithmetic mean.

A.5 Determination of pH (10% solution)

Weigh 10.0 g of the specimen, which is dried at 105 °C ± 2 °C for 3 hours. Mix with 90 mL of water. Let stand for 1 hour. Stir from time to time. Centrifuge it. Then use a pH meter to determine the pH of the supernatant, according to the method specified in GB/T 9724.

A.6 Determination of water-soluble substances

A.6.1 Reagents and materials

Medium-speed qualitative filter paper.

A.6.2 Instruments and equipment

A.6.2.1 Balance: Sensitivity 0.1 mg.

A.6.2.2 Electric constant temperature drying oven: Temperature control range 105 °C ± 2 °C.

A.6.2.3 Constant temperature water bath.

A.6.3 Analytical procedures

Weigh about 6 g of the specimen (m_3) dried at 105 °C ± 2 °C for 3 h, accurate to 0.1 mg. Mix with 90 mL of water which is freshly boiled and cooled to room temperature. Let stand for 10 min. Stir from time to time. Filter it. Discard 10 mL of the initial filtrate. If necessary, re-filter the remaining filtrate to obtain a clear filtrate. Take 15 mL of the filtrate. Transfer it to an evaporating dish of known mass (m_5). Place it on a steam bath and evaporate to dryness. Then dry at 105 °C for 1 h. Cool in a desiccator and weigh it (m_4).

A.6.4 Result calculation

The mass fraction of water-soluble matter w_3 is calculated according to formula (A.6):

$$w_3 = \frac{(m_4 - m_5)}{m_3 \times \frac{15}{90}} \times 100\% \qquad \text{(A.6)}$$

Where:

m_4 - The total mass of the evaporating dish and the dried matter after drying, in grams (g);

m_5 - The mass of the evaporating dish, in grams (g);

m_3 - The mass of the specimen, in grams (g);

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