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

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Food additives -- Indigo

食品添加剂 靛蓝

(English Translation)

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 - 2.1 Chemical name
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1 Scope

This standard applies to indigo as raw material, the sulfonation, refined and made of food additives indigo.

2.1 Chemical name

5,5'-indocyanine disulfonic acid disodium salt

2.2 Molecular formula

C₁₆H₈N₂Na₂O₈S₂

2.4 Relative molecular mass

466.36 (according to the 2007 International Relative Atomic Quality)

3 Technical requirements

3.1

sensory requirements. should be consistent with the provisions of Table 1.

Table 1 sensory requirements

The project requires a test method Color dark purple to dark purple brown State powder or particles Take the appropriate amount of sample placed in a clean, dry white porcelain dish, in the natural light, Observe its color and state

3.2

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

Table 2 Physical and chemical indicators

Item Index Test Method Indigo content, w /% ≥ 85.0 Appendix A, A.3 Dry reduction, chloride (in terms of NaCl) and sulfate (In terms of Na₂SO₄), w /% ≤ 15.0 Appendix A A.4 Water insoluble matter, w /% ≤ 0.20 Appendix A, A.5 Sub-dye, w /% ≤ 1.0 Appendix A A.6 Total arsenic (in As)/(mg/kg) ≤ 1 Appendix A A.7 Lead (Pb)/(mg/kg) ≤ 10 Appendix A A.8 Appendix A Testing method A.1 General provisions the reagents and water used in this standard, when not specified in other requirements, refer to the analysis of pure reagents and GB/T 6682-2008 in the provisions of the three Water level. Standard titration solution used in the test, the standard solution for the determination of impurities, preparations and products, without any other requirements, GB/T 601, GB/T 602, GB/T 603. The solution used in the test refers to water when it is not specified with the formulation of the solvent Solution.

A.2 Identification test A.2.1 Reagents and materials A.2.1.1 Sulfuric acid solution. 1 20.

A.2.1.2 Sodium hydroxide solution. 2.5 g/L.

A.2.1.3 Ammonium acetate solution. 1.5 g/L.

A.2.2 Instruments and equipment A.2.2.1 Spectrophotometer.

A.2.2.2 Cuvette. 10mm.

A.2.3 Analysis steps A.2.3.1 Color response A.2.3.1.1 Weigh about 0.1g sample, add 10mL sulfuric acid solution, shake from time to time, the solution was dark purple, after cooling, take 2 to 3 drops, add 5mL water, the solution was blue and purple.

A.2.3.1.2 measure the blue-purple indigo-sulfuric acid solution 5mL, add 1mL sodium hydroxide solution, slightly shaking, was yellow-green.

A.2.3.2 Maximum absorption wavelength Weigh about 0.1g sample, add 100mL water, shake from time to time, dissolved into purple blue solution. Measure the solution 1mL, add ammonium acetate solution with to 100 mL. The maximum absorption wavelength of this solution is $612 \text{ nm} \pm 2 \text{ nm}$.

A.3 Determination of indigo content A.3.1 Titanium trichloride titration (Arbitration) A.3.1.1 Method Summary in the acidic medium, the amino group in the dye structure is reduced to the amino compound by titanium trichloride, and the solution is titrated according to the standard of titanium trichloride Consumption, calculate its content.

A.3.1.2 Reagents and materials A.3.1.2.1 Sodium hydrogen tartrate.

A.3.1.2.2 Titanium trichloride standard titration solution. $c(\text{TiCl}_3) = 0.1 \text{ mol/L}$ (now with the use of the preparation method see Appendix B).

A.3.1.2.3 Carbon dioxide.

A.3.1.3 Instruments and equipment See Figure A.1.

A - conical flask (500mL);

B - brown burette (50mL);

C --- black paper with black paper bottle (2000mL);

D - a mixture of ammonium carbonate and ferrous sulfate (5000 mL);

E --- piston

F - empty bottle;

G --- water washing gas bottle.

Figure A.1 Titanium trichloride titration device

A.3.1.4 Analysis steps Approximately 0.5 g of the sample was weighed to 0.0001 g, placed in a 500 mL Erlenmeyer flask, dissolved in 50 mL of freshly boiled and cooled to room temperature Water, add 15 g of sodium hydrogen tartrate and 150 mL of freshly boiled and cooled to room temperature, dissolve and dissolve, install the instrument as shown in Figure A.1, Liquid under the surface of carbon dioxide at the same time, heated to boiling, and titration with titanium titanate titration to its inherent color disappeared as the end.

A.3.1.5 Calculation of results the indigo content is expressed in terms of mass fraction w_1 and the value in%, calculated according to formula (A.1) $w_1 = (V/1000) \times c (M/2) m_1 \times 100\%$ (A.1) where.

V --- titration sample consumption of titanium trichloride standard titration solution volume of the exact value in milliliters (mL);

1000 --- volume conversion factor;

c - Titanium trichloride standard titration solution concentration of the exact value in moles per liter (mol/L);

M - the molar mass of indigo in grams per mole (g/mol) [M (C₁₆H₈N₂Na₂O₈S₂) = 466.36];

2 --- concentration conversion factor;

m_1 --- the value of the sample quality, in grams (g).

The result of the calculation indicates one bit after the decimal point.

The experimental results are based on the arithmetic mean of the parallel measurement results. The absolute difference between the two independent determinations obtained under repeatability conditions the value should not exceed 1.0% of the arithmetic mean.

A.3.2 Spectrophotometric colorimetric method A.3.2.1 Methodological Summary the sample and the known content of indigo standard samples were dissolved in water, respectively, at the maximum absorption wavelength, respectively, measured its absorbance value, and then Calculate its content.

A.3.2.2 Reagents and materials A.3.2.2.1 Ammonium acetate solution. 1.5 g/L.

A.3.2.2.2 Indigo standard sample. content $\geq 85.0\%$ (mass fraction, measured in A.3.1).

A.3.2.3 Instruments and equipment A.3.2.3.1 Spectrophotometer.

A.3.2.3.2 Cuvette. 10mm.

A.3.2.4 Analysis steps A.3.2.4.1 Preparation of indigo standard sample solution Weigh about 0.25g indigo standard sample, accurate to 0.0001g, dissolved in water, into the 1000mL volumetric flask, diluted with water to the scale, shake. Accurately absorb 10mL, into the 500mL volumetric flask, add ammonium acetate solution diluted to the mark, shake, spare.

A.3.2.4.2 Preparation of indigo sample solution Weighing and handling methods are prepared with indigo standard sample solutions.

A.3.2.4.3 Determination the indigo standard sample solution and indigo sample solution were placed in 10mm cuvette, respectively, with the maximum absorption wavelength ($612\text{nm} \pm 2\text{nm}$) with a spectrophotometer to determine the respective absorbance values to ammonium acetate solution as the reference solution.

A.3.2.5 Calculation of results the indigo content is expressed in terms of mass fraction w_2 and the value in%, calculated according to formula (A.2) $w_2 = A_1 \times m_0 A_0 \times m \times w_0$ (A.2) where.

A_1 --- Indigo sample solution absorbance value;

m_0 --- Indigo standard sample quality value in grams (g);

A_0 --- Indigo standard sample solution absorbance value;

m - the mass of the sample, in grams (g);

w_0 --- indigo standard sample content of the value,%.

The result of the calculation indicates one bit after the decimal point.

The experimental results are based on the arithmetic mean of the parallel measurement results. The absolute difference between the two independent determinations obtained under repeatability conditions the value should not exceed 1.0% of the arithmetic mean.

A.4 Determination of dry reduction, total amount of chloride (in terms of NaCl) and sulfate (calculated as Na_2SO_4) A.4.1 Determination of dry reduction A.4.1.1 Analysis steps Weigh about 2g sample, accurate to 0.0001g, placed in $135^\circ\text{C} \pm 2^\circ\text{C}$ constant temperature oven constant volume of the bottle, the $135^\circ\text{C} \pm 2^\circ\text{C}$ constant temperature oven bake to constant volume.

A.4.1.2 Calculation of results the drying loss is expressed in terms of mass fraction w_3 and the value in%, calculated according to formula (A.3) $w_3 = \frac{m_2 - m_3}{m_2} \times 100\%$ (A.3) where.

m_2 --- the value of the sample before drying, in grams (g);