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

GB 28301-2012

Food additives -- Riboflavin 5-sodium phosphate

食品添加剂 核黄素5'-磷酸钠

(English Translation)

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1 Scope

This standard applies to riboflavin as raw material, phosphorylation, and purification, refining and other steps to produce food additives riboflavin 5'-phosphate.

2.1 Molecular formula

$C_{17}H_{20}N_4NaO_9P \cdot 2H_2O$

2.2 Relative molecular mass

514.36 (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 orange yellow State crystalline powder Take appropriate sample in a clean, dry white porcelain dish Natural light, observe the 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 Riboflavin ($C_{17}H_{20}N_4O_6$) content, w /% 73.0 ~ 79.0 Appendix A, A.3 Specific rotation $\alpha_D(25^\circ C)$ [(°) · dm² · kg⁻¹] 37.0 ~ 42.0 Appendix A A.4 pH 5.0 to 6.5 Appendix A, A.5 Dry reduction, w /% ≤ 7.5 Appendix A, A.6 Residue on ignition, w /% ≤ 25.0 Appendix A A.7 Free phosphoric acid, w /% ≤ 1.0 Appendix A A.8 Free riboflavin, w /% ≤ 6.0 Appendix A A.9 Riboflavin diphosphate, w /% ≤ 6.0 Appendix A A.9

Lead (Pb)/(mg/kg) $\leq$ 2.0 GB 5009.12 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 hydrochloric acid solution. take hydrochloric acid 1mL, diluted with water to 3mL.

A.2.1.2 sodium hydroxide solution. take sodium hydroxide 1g, dissolved in water and diluted to 25mL.

A.2.2 Identification method Weigh about 1.5mg sample, add 100mL water dissolved, this is the sample solution. The sample solution was light yellowish green under transmission light and was strong Yellow-green fluorescent. Take the sample solution 25mL, add 0.5mL hydrochloric acid solution, fluorescence disappeared; another sample solution 25mL, add 0.1mL hydrogen Sodium oxide solution, fluorescence disappeared.

A.3 Determination of riboflavin (C₁₇H₂₀N₄O₆) content A.3.1 Reagents and materials

A.3.1.1 Glacial acetic acid.

A.3.1.2 Sodium acetate solution. 14 g/L.

A.3.2 Instruments and equipment Spectrophotometer.

A.3.3 Analysis steps Avoid light operation. Weigh about 100mg sample, accurate to 0.0001g, placed in 500mL volumetric flask, add 1mL glacial acetic acid and 75mL water. After the sample is dissolved, dilute to the mark with water and shake it. Accurately measure 10mL, placed in 100mL volumetric flask, add 7mL sodium acetate solution, diluted with water to the mark, shake. To the water as a blank, with a 1cm caliber cup, measured at 444nm wavelength the absorbance of the sample solution. The absorption coefficient (E₁ m) of riboflavin is calculated as 328.

A.3.4 Calculation of results Riboflavin content of riboflavin to the mass fraction w₀, the value expressed in%, according to formula (A.1) calculation.

$w_0 = 50 \times A_{328} \times m \times (1 - w_1) \times 100\%$ (A.1) where.

50 --- dilution factor;

A - the absorbance of the sample solution;

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

w₁ --- Measured sample dry reduction value,%.

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 ratio of the value to the arithmetic mean is not more than 1.0%.

A.4 Determination of specific rotation A.4.1 Reagents and materials Hydrochloric acid solution. take hydrochloric acid 27.0mL, diluted with water, constant volume to 50mL.

A.4.2 Instruments and equipment Polarimeter, accuracy $\pm 0.01^\circ$.

A.4.3 Analysis steps Avoid light operation. Weigh 0.75g (in anhydrous terms) sample, accurate to 0.0001g, placed in 50mL volumetric flask, add hydrochloric acid solution Solution and dilution to the scale, in 15min, $25^\circ\text{C} \pm 1^\circ\text{C}$ conditions with a polarimeter to determine the specific rotation.

A.4.4 Calculation of results the value of the specific rotation $\alpha_m(25^\circ\text{C}, D)$ is expressed by $(^\circ) \cdot \text{dm}^2 \cdot \text{kg}^{-1}$, calculated according to the formula (A.2) $\alpha_m(25^\circ\text{C}, D) = \alpha / l \rho$ where.

α - the measured angle of rotation, in degrees ($^\circ$);

l - the length of the rotating tube in dm (dm);

ρ --- The concentration of the sample solution in grams per milliliter (g/mL).

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 ratio of the value to the arithmetic mean is not more than 1.0%.

A.5 Determination of pH A.5.1 Instruments and equipment Acidity meter, accuracy ± 0.01 .

A.5.2 Analysis steps Weigh 1.0g sample, accurate to 0.0001g, placed in 200mL clean and dry beaker, add 100mL of water dissolved at 20°C the pH was measured with an acidity meter.

A.5.3 Calculation of results 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 ratio of the value to the arithmetic mean is not more than 1.0%.

A.6 Determination of dry reduction A.6.1 Instruments and equipment Constant temperature vacuum oven.

A.6.2 Analysis steps Weigh about 2g sample, accurate to 0.0001g, placed in the constant weight to the weighing bottle, at $100^\circ\text{C} \pm 1^\circ\text{C}$, 0.01MPa Dried for 5 h, then placed in a desiccator to cool to room temperature and weighed.

A.6.3 Calculation of results the drying loss is expressed in terms of mass fraction w_1 and the value in%, calculated according to formula (A.3) $w_1 = (m_1 - m_2) / m_3 \times 100\%$ (A.3) where.

m1 --- the value of the sample before the dry and the quality of the weighing bottle, in grams (g);

m2 --- the value of the sample after drying and the mass of the weighing bottle, in grams (g);

m3 --- the value of the sample quality before drying, in grams (g).

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 ratio of the value to the arithmetic mean is not more than 1.0%.

A.7 Determination of burning residue A.7.1 Instruments and equipment High temperature furnace.

A.7.2 Analysis steps Weigh about 1g sample, accurate to 0.001g, placed in the 600 °C ~ 700 °C was burning to constant weight of the crucible, slowly heated to the same the product is fully carbonized. The carbonized sample was cooled, the residue was wetted with 0.5 mL of sulfuric acid, heated to sulfuric acid to escape, 600 °C ~ 700 °C high temperature furnace burning to constant weight.

A.7.3 Calculation of results the ignition residue is expressed in terms of mass fraction w_2 and the value in%, calculated according to formula (A.4) $w_2 = \frac{m_4 - m_5}{m_6} \times 100\%$ (A.4) where.

m4 - the value of the mass of the residue and the empty crucible, in grams (g);

m5 --- the value of the quality of the empty crucible, in grams (g);

m6 --- the value of the sample quality, in grams (g).

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 ratio of the value to the arithmetic mean is not more than 1.0%.

A.8 Determination of free phosphoric acid A.8.1 Reagents and materials A.8.1.1 Standard Phosphate Solution. 0.22 g of potassium dihydrogen phosphate was dried at 105 °C for 2 h, placed in a 1000 mL volumetric flask, Add the appropriate amount of water to dissolve, and diluted to the mark, shake, temporary use and then diluted 5 times.

A.8.1.2 Ammonium molybdate $[(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}]$ solution. 70 g/L.

A.8.1.3 sulfuric acid solution. 3.75 mol/L.

A.8.1.4 Acid Ammonium Molybdate Solution. 25 mL of ammonium molybdate solution was diluted with water to 200 mL and then slowly added 25 mL of sulfuric acid solution Liquid, shake.