



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

GB 1886.73-2015

**National Food Safety Standard -- Food Additives -- Insoluble
polyvinylpolypyrrolidone**

食品安全国家标准 食品添加剂 不溶性聚乙烯聚吡咯烷酮

(English Translation)

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1 Scope

This standard applies to N- vinylpyrrolidone monomer as a raw material in the presence of a basic catalyst or N, N'- divinyl - Mi Pyrazolone as a crosslinking agent to generate a food additive insoluble polyvinylpolypyrrolidone.

2 Molecular formula, structural formula

Formula 2.1

(C₆H₉NO) n

3.1 Sensory requirements

Sensory requirements shall comply with the requirements of Table 1.

Table 1 Sensory requirements

Project requires test methods Color white or similar white State hygroscopic white powder
Take appropriate amount of the sample to the transparent glass tube, in natural light black
paper backing Observed

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 Nitrogen content, w /% 11.0 ~ 12.8 Appendix A A.4 Free N-
vinylpyrrolidone/(mg/kg) <= 10.0 A.5 Appendix A pH (1% suspension) 5-8 Appendix A A.6
Water solubles, w /% <= 1.5 Appendix A A.7 Residue on ignition, w /% <= 0.4 Appendix A
A.8 Free N, N'- divinyl - imidazolidinone/(mg/kg) a <= 2.0 A.9 Appendix A Water, w /% <= 6.0 Appendix A A.10 Lead (Pb)/(mg/kg) <= 2.0 GB 5009.12 Zinc (Zn)/(mg/kg) <= 25.0 GB

5009.14 Only use a N, N'-divinyl-imidazolidinone as a crosslinking product testing the project.

Appendix A

Testing method

A.1 Safety Tips

Reagents toxic or corrosive, operator of this test method should be used with caution. As should be immediately washed with water splashed on the skin Wash, severe cases should be treated immediately. When using flammable, do not use open flame heating.

A.2 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 stipulated.

Standard Solution tests required, the determination of impurities standard solution, the test method used in preparations and products, did not indicate when additional requirements are According to GB/T 601, the provisions of the preparation of GB/T 602 and GB/T 603, the test used in the solution, in the absence of explanation, refer to the water Solution.

A.3 Identification Test A.3.1 Solubility Test 1.0g samples were taken, were added 10mL of water, 10 mL of 95% ethanol, 10mL ether, should not dissolve.

A.3.2 fading reaction A.3.2.1 Reagents and materials A.3.2.1.1 iodine solution. $c(2I_2) = 0.1\text{mol/L}$.

A.3.2.1.2 starch indicator. 10g/L.

A.3.2.2 analysis step Weigh about 1g sample was dissolved in 10mL water, shaking 30s, to prepare a suspension, was added 0.1mL iodine solution, shaken vigorously, then add 1mL starch indicator, shake vigorously, no blue produce.

A.3.3 Infrared Spectroscopy A.3.3.1 Method summary Take insoluble polyvinylpyrrolidone and reference samples, respectively, with potassium bromide tablet, infrared spectral scanning, the two infrared Absorption spectra for comparison.

A.3.3.2 Reagents and materials A.3.3.2.1 insoluble polyvinylpyrrolidone reference.

A.3.3.2.2 potassium bromide (spectroscopically pure).

A.3.3.3 instruments and equipment A.3.3.3.1 infrared spectrometer.

A.3.3.3.2 tablet device.

A.3.3.4 analysis step Were taken from about 1mg of insoluble polyvinylpyrrolidone and reference samples, and about 100mg of potassium bromide carried out in an agate mortar Grinding, until thoroughly mixed. Take a little of the mixture powder into the tablet mold, pressed into a transparent sheet (salt window) is carried out using a tablet test.

A.3.3.5 results found Insoluble polyvinylpyrrolidone infrared absorption spectrum of the sample should be the reference infrared absorption spectrum at 4000cm^{-1} ~ Absorption peaks in the range of 400cm^{-1} are similar. Insoluble polyvinylpyrrolidone infrared absorption spectrum of the reference, see Appendix B.

A.4 Determination of nitrogen content A.4.1 Method summary the sample with sulfuric acid and catalyst were hot, and the organic nitrogen into ammonium, alkali distillation, after absorption of free ammonia with boric acid to sulfuric acid droplets standard Fixed titration, the nitrogen content calculated based on the volume of sulfuric acid consumed.

A.4.2 Reagents and materials A.4.2.1 30% hydrogen peroxide.

A.4.2.2 sulfuric acid.

A.4.2.3 sodium hydroxide solution. 400g/L .

A.4.2.4 boric acid. 40g/L .

A.4.2.5 sulfuric acid standard titration solution. $c(2\text{H}_2\text{SO}_4) = 0.05\text{mol/L}$.

A.4.2.6 mixed indicator. 2 parts of methyl red ethanol solution (1g/L) and 1 part of methylene blue ethanol solution (1g/L), temporary use when mixing.

A.4.2.7 mixture of potassium sulfate and copper sulfate. 10 parts of the potassium persulfate, and 1 part of copper sulfate, milled into a powder form.

A.4.3 Instruments and Equipment Kjeldahl distillation apparatus.

A.4.4 Analysis step

Accurately weigh 0.1g sample, accurate to 0.1mg , placed in 100mL Kjeldahl flask, a mixture of copper sulfate and potassium sulfate 1.0g , Draw 7mL sulfuric acid was added slowly along the sides of the bottle, to adhere to the bottleneck residue into the bottom of the bottle, shake, and then along the sides of the bottle slowly adding 1mL 30% hydrogen peroxide plus side carefully shaking the flask, and then multiple times (usually 3 to 6 times) was added 30% hydrogen peroxide, per 1mL (note Italy. digestion process should not add 30% hydrogen peroxide), heating digestion until the solution became clear and bright green. Heating was continued for 4h , cooled After slowly adding 20mL water, the digestion solution into the distillation flask, plus the amount of wash water Kjeldahl flask, together with the cleaning liquid into the distillation Flask. The distillation flask connected to a distillation apparatus, and the end of the condenser has been added 15mL boric acid solution and 3 drops of mixed indicator Erlenmeyer flask Connection, should not absorbing liquid outlet through a condenser, a dropping funnel was added 30mL sodium hydroxide solution to a distillation flask, funnel spin off Plug immediately heated distillation. Distillate was collected until after the absorption liquid color green, continue to collect, until about $80\text{mL} \sim 100\text{mL}$, stop steaming Distillate. Absorbing liquid sulfuric acid standard titration solution titration to pink, recording the volume of sulfuric acid standard titration solution.

Meanwhile blank experiment.

A.4.5 Calculation Results Nitrogen content of the sample to the mass fraction w_1 meter, according to equation (A.1) Calculated.

$(V_1 - V_2) \times c \times M$

$m \times 1000 \times$

100% (A.1)

Where.

V_1 --- sample consumption volume of sulfuric acid standard titration solution, in milliliters (mL);

V_2 --- blank sample consumption of sulfuric acid standard titration solution volume in milliliters (mL);

C --- concentration of sulfuric acid standard titration solution, expressed in moles per liter (mol/L);

--- The M molar mass of nitrogen in grams per mole (g/mol), [$M(N) = 14$];

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

1000 --- conversion factor.

The results to three significant figures.

The test results of the arithmetic mean of the parallel determination results shall prevail, twice under the same condition of independent determination results obtained absolute difference not more than 10% of the arithmetic mean.

Determination of free N- vinyl pyrrolidone A.5 A.5.1 Method summary Insoluble polyvinylpyrrolidone free of N- vinylpyrrolidone extracted with water. After standing over the membrane using UV detection with High performance liquid chromatograph or diode array detector, external standard.

A.5.2 Reagents and materials A.5.2.1 Methanol. HPLC grade.

A.5.2.2 acetonitrile. HPLC grade.

A.5.2.3 water, GB/T 6682 specified in a water.

A.5.2.4 needle nylon membrane, pore size 0.22 μ m, or equivalent pore water system filters.

A.5.2.5 N- vinylpyrrolidone reference substance, the content of $\geq 99\%$.

A.5.2.6 N- vinylpyrrolidone standard stock solution. Weigh accurately 0.05g N- vinylpyrrolidone reference to the nearest 0.1mg, dissolved in methanol and diluted to 100mL, shake.