

ICS 67.020
CCS C 54



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

GB 31629-2014

**National Food Safety Standard -- Food Additives --
Polyacrylamide**

食品安全国家标准 食品添加剂 聚丙烯酰胺

(English Translation)

Issued on: December 24, 2014

Implemented on: May 24, 2015

**Issued by: National Health and Family Planning Commission of the
People's Republic of China**

Table of Contents

- 1 Scope
- 2 Technical Requirements
 - 2.1 Sensory requirements
 - 2.2 Physical and Chemical Indicators

1 Scope

This standard applies to food additives polyacrylamide acrylamide as main raw materials.

2.1 Sensory requirements

Shall comply with the requirements in Table 1.

Table 1 Sensory requirements

Project requires test methods Color status White or yellow Granules or powder Proper amount of sample is placed in a 50mL beaker, under natural light Concept Color and state police

2.2 Physical and Chemical Indicators

Shall be in accordance with Table 2.

Table 2. Physical and chemical indicators

Item Index Test Method Solids content (w) /% ≥ 90.0 Appendix A A.4 Acrylamide monomer (dry basis, w) /% ≤ 0.025 A.5 Lead (Pb)/(mg/kg) ≤ 3 GB 5009.12 Total arsenic (As)/(mg/kg) ≤ 3 GB/T 5009.11 or GB/T 5009.76 Dissolution times/min ≤ 90 A.6 Insolubles (w) /% ≤ 0.3 A.7 Appendix A Testing method A.1 Warning Reagents of this standard test method for use in corrosive or toxic, the operation should take appropriate safety and protective measures.

A.2 General Provisions this standard reagents and water, in the absence of other requirements specified, refer to three predetermined water analytical reagents and GB/T 6682 in.

Standard solution used in the test, the formulations and products, in the absence of other requirements specified, are by use GB/T 602 and GB/T 603 Determination of impurities Preparation of regulations. When used in a solution prepared with a solvent which is not specified, refer to an aqueous solution.

A.3 Identification Test Infrared absorption spectrometry, the sample spectrum and the standard spectrum (see Figure B.1) comparison, the two should be consistent, and the wave number 3380cm⁻¹, 1660cm⁻¹ (amide), 1610cm⁻¹, 1460cm⁻¹ and 1130cm⁻¹ absorption peak near.

Determination of solid content A.4 A.4.1 Method summary at a certain temperature, the specimen was placed into the electric oven dry mass constant.

A.4.2 Instruments and Equipment A.4.2.1 electric oven. temperature can be controlled to 120 °C ± 2 °C.

A.4.2.2 weighing bottle (phi40mm x 30mm) or aluminum pan.

A.4.3 Analysis step

Previously used at 120 °C ± 2 °C dried to constant mass in the weighing bottle was weighed sample of about 1g, accurate to 0.0002g, placed in an electric dry Dry box at 120 °C ± 2 °C dried to constant mass.

A.4.4 calculation results W1 solids mass fraction basis, calculated according to formula (A.1).

$m_1 - m_0$

$m \times 100\%$

Where.

M1 sample values and dried weighing bottle --- mass in grams (G);

Quality value M0 --- weighing bottles in grams (G);

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

The test result to the arithmetic mean of replicates results. Obtained in two independent determination results under repeatability conditions of absolute difference Value is not more than 0.3%.

Determination A.5 acrylamide monomer content (dry basis) of A.5.1 Method summary Acrylamide sample extracted by the solvent was purified by high performance liquid chromatography (UV detector), external standard.

A.5.2 Reagents and materials A.5.2.1 Water. in line with a water GB/T 6682 provisions.

A.5.2.2 acrylamide standard sample. purity ≥99.5%.

A.5.2.3 ethanol. chromatography.

A.5.2.4 Methanol. HPLC grade.

Potassium dihydrogen phosphate solution A.5.2.5. 20mmol/L. 2.72g potassium dihydrogen

phosphate was weighed, dissolved in water, after complete dissolution, adjusted with phosphoric acid pH to 3.8. All transferred to 1000mL volumetric flask, dilute to the mark.

A.5.2.6 extract free monomer. amount of 540mL of isopropanol, water and 450mL of ethanol were mixed 10mL, reagent bottle placed 1000mL Save. This solution is a solution A. Measure 740mL of isopropanol, water and 250mL of ethanol were mixed 10mL, reagent bottle placed 1000mL Save. This solution is a solution B.

A.5.2.7 microporous membrane. 0.45 μ m.

A.5.3 Instruments and Equipment A.5.3.1 HPLC. with UV detector.

A.5.3.2 mechanical oscillator.

A.5.4 Reference chromatographic conditions A.5.4.1 mobile phase. methanol = 85 15 potassium dihydrogen phosphate solution (volume ratio).

A.5.4.2 column. reverse phase C18 column, 150mm x 4.6mm, filler particle diameter 3 μ m.

A.5.4.3 flow rate. 1.0mL/min.

A.5.4.4 Column temperature. 20 °C.

A.5.4.5 Injection volume. 10 μ L.

A.5.4.6 wavelength. 205nm.

A.5.5 Analysis step

Preparation of standard solution A.5.5.1 Weigh 1g acrylamide standard sample, accurate to 0.0002g, was placed in a 10mL beaker, water was added and completely dissolved, transfer quantitatively Move 100mL flask, diluted with water to the mark. The solution was 10mg/mL solution of acrylamide.

Acrylamide solution 0.1mL tube Pipette 10mg/mL by pipetting, 0.2mL, 0.4mL, 0.5mL, 1.0mL, were placed 100mL volumetric flask and dilute to volume with water, and the solution were 0.01 mg/mL, 0.02 mg/mL, 0.04 mg/mL, 0.05mg/mL, acrylamide standard sample solution 0.1mg/mL of. Chromatography microporous membrane filter prior to analysis.

Preparation of sample solution A.5.5.2 Weigh about 2g sample, accurate to 0.0002g, was placed on the dried ground-50mL Erlenmeyer flask. Pipette with a pipette 10mL of A sample was soaked and shaken 40min, 10mL of solution B was then added, shaking continued 40min. Microporous membrane prior to chromatographic analysis filter.

A.5.5.3 Determination Under the chromatographic conditions specified, taking each standard solutions and sample solutions were injected into the liquid chromatograph 10 μ L, retention time given acrylamide of quantitative peak area external standard method. Standard liquid chromatography shown in Figure B.2.

A.5.5.4 calculation results Acrylamide mass fraction w_2 , calculated according to equation (A.2).

$$m \times w_1 \times 1000 \times$$

$$100\% \text{ (A.2)}$$

Where.

C --- external standard sample solution resulting acrylamide actual concentration, in milligrams per milliliter (mg/mL);

V --- sample constant volume in milliliters (mL);

Sample mass m ---, in grams (G);

W_1 --- mass fraction solids,%;

1000 --- conversion factor.

The test result to the arithmetic mean of replicates results. Obtained in two independent determination results under repeatability conditions of absolute difference Value is not more than 5%.

A.6 dissolution measurement time A.6.1 Method summary with the dissolution of the sample, the conductance of the solution is increasing. After complete dissolution, the conductance value is constant. An amount of a certain amount of sample when dissolved in water, the time constant conductance value reaches the desired dissolution time of the sample.

A.6.2 Instruments and Equipment A.6.2.1 conductivity meter. measuring range $0.01\mu\text{S}/\text{cm} \sim 106\mu\text{S}/\text{cm}$.

A.6.2.2 thermostatic chamber. the temperature can be controlled to $30\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$.

A.6.2.3 electromagnetic stirrer. a heating means and temperature control, a stirrer with a length of 3cm.

A.6.3 Analysis step

It will be filled with 100mL water and 200mL beaker with a stir thermostatic bath on a magnetic stirrer. The conductivity meter electrode is inserted Beaker, with the beaker wall distance 5mm ~ 10mm, and a stirring distance of about 5mm. Start electromagnetic stirring, adjusting the liquid level vortex depth of about 20mm. Turn on the heating means, and the bath temperature was raised to $30\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$, thermostat 10min ~ 15min.

Weigh $0.040\text{g} \pm 0.002\text{g}$ sample was added to the upper portion of the vortex in the beaker. When no change in 3min solution conductance stopped test.

A.6.4 calculation results Sample was added to the conductance value from the start time is