



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

GB 5009.245-2016

**National food safety standard - Determination of Polydextrose
in Food**

食品安全国家标准 食品中聚葡萄糖的测定

(English Translation)

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1 Application Scope

This Standard specifies the method for determination of polydextrose in food.

This Standard applies to the determination of polydextrose added in food.

2 Terms and Definitions

For the purposes of this document, the following term and definition apply.

2.1

polydextrose [(C₆H₁₀O₅)_n]

A polymer which is made by mixing glucose, sorbitol and citric acid (or phosphoric acid) in accordance with a certain proportion and heating polymerization, refining and dying at high temperature, with an average polymerization degree 12. It is a soluble dietary fiber.

3 Principle

Carry out hot water extraction and ultrafiltration centrifugation of polydextrose in food;
remove interfering substances in the filtrate such as starch and fructan;

4 Reagents and Materials

Unless specified otherwise, all reagents used for this method are guaranteed reagents and the water is water grade II specified in GB/T 6682.

4.1.1 Sodium hydroxide solution (50%). chromatographically pure, exclusive use for

ion chromatography.

4.1.6 Amyloglucosidase. $\geq 3\,260$ U/mL (soluble starch); ≥ 200 U/mL (p-NP-beta maltoside).

4.1.7 Isoamylase. $\geq 1\,000$ U/mL.

4.2 Preparation of reagents

4.2.1

Acetic acid solution (0.2 mol/L). accurately absorb 1.2 mL of glacial acetic acid; use water to dilute to 100 mL.

4.3.1 Mobile phase A (containing 0.15 mol/L sodium hydroxide). accurately absorb

15.7 mL of sodium hydroxide solution (50%); use pre-degassed water to dilute to 2 L; use an inert gas for protection.

4.3.2 Mobile B (containing 0.15 mol/L sodium hydroxide, 0.50 mol/L sodium acetate).

accurately weigh 41 g of sodium acetate anhydrous (or 68 g of sodium acetate trihydrate); use mobile phase A to dissolve before adding dropwise to 1 L; mix up; pass through 0.45 μm membrane; conduct degassing.

4.4 Reference substance

Polydextrose $[(\text{C}_6\text{H}_{10}\text{O}_5)_n]$. purity greater than 90%; CAS 68424-04-4.

4.5.1 Reference substance stock solution (2.00 mg/g). accurately weigh 0.20 g of polydextrose reference substance (accurate to 0.000 1 g) to place into a pre-weighed 100 mL sample bottle with a screw stopper (accurate to 0.001 g).

4.5.2 Reference substance intermediate solution. accurately weigh 10.0 g, 7.50 g, 5.00 g, 3.75 g, 2.50 g and 1.50 g (accurate to 0.000 1 g) of reference substance stock solution apiece; add water to 20.0 g (accurate to 0.000 1 g); then obtain reference substance intermediate solution of concentrations 1.000 mg/g, 0.750 mg/g, 0.500 mg/g, 0.375 mg/g, 0.250 mg/g and 0.150 mg/g.

5 Apparatus

5.1

Analytical balance. sensitivity 1 mg and 0.1 mg.

5.2

Constant temperature water bath. temperature control accuracy $\pm 1^{\circ}\text{C}$.

5.6

Syringe filter. pore diameter 0.22 μm .

5.7 Ultrafiltration centrifugal equipment. 100 000 Da molecular retention; PES

membrane; 0.5 mL volume.

5.8 Magnetic stirrer. equipped with stirrer.

5.9 Ion chromatograph. equipped with pulse ampere detector and gradient pump.

6.1.1 Solid sample. ground and crushed and sealed for protection before analysis to

prevent water content changing.

6.1.2 Liquid sample. mixing up and shaking up before determination.

6.2 Extraction of polydextrose

6.2.1

Solid sample. take 100 mL sample bottle with stopper; add 1 magnetic stirrer; put screw stopper on; weigh (accurate to 0.001 g) and record as m1. Accurately weigh a certain amount of sample (containing about 0.05 g of polydextrose, accurate to 0.000 1 g, recorded as m2) to place into sample bottle; add 100 mL of water pre-heated to 80°C; conduct magnetic stirring for 30 s; maintain in water bath at 80°C for 10 min; conduct magnetic stirring for 30 s every other 5 min.

6.3 Enzymolysis

6.3.1

Take 2 mL centrifugal tube, weigh (accurate to 0.000 1g) and record as m4.

6.3.2 Absorb 0.2 mL of ultrafiltration centrifugate to add to centrifugal tube, weigh

(accurate to 0.000 1 g) and record as m5; add 0.8 mL of enzyme mixture, weigh (accurate to 0.000 1 g) and record as m6; vibrate to mix up evenly, maintain in water bath at 50°C for 60 min, maintain in boiling water bath for 10 min, take out, maintain in ice bath for 5 min and conduct centrifugation at 10 000 r/min for 10 min. Pass liquid supernatant through 0.2 µm filter membrane, conduct analysis. Liquid supernatant shall be tested within 72 h.

6.4 Chromatographic reference conditions

See Annex B for testing conditions.

7 Accuracy

The absolute difference between the results obtained from two independent determination results under repeatable conditions shall not exceed 10% of the arithmetic mean value.

8 Others

When the minimum sample weight is 5 g, detection limit of solid sample is 0.2 mg/100 g and quantification limit 0.5 mg/100 g;

Annex A

Enzyme Activity Determination

A.1.1 Principle

Under the standard conditions of pH 4.5, temperature 40°C and substrate (kestose) concentration 10 mmol/L, the amount of enzyme required for release of 1 µmol of fructose for 1 min is 1 U.

A.1.2 Reagents and solvents

A.1.2.1 Acetic acid (0.2 mol/L). accurately absorb 12 mL of glacial acetic acid; use water to dilute to 1 000 mL.

A.1.2.6 Fructose standard solution (1 mg/mL). accurately weigh 100 mg of analytical

pure fructose dried to a constant weight at 80°C and place into small beaker; transfer to 100 mL volumetric flask; use acetate buffer solution to add dropwise to 100 mL; mix up evenly; store in refrigerator at 4°C as standby.