National Standard of the People's Republic of China

GB 25541—2010

National Standards For Food Safety
Food additive - Polydextrose

2010-12-21 Issue 2011-02-21 Implementation

Ministry of Health of the PRC Issue

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Food additive - Polydextrose

1 Scope

This standard applies to polydextrose products that are mixed with glucose, sorbitol, citric acid or phosphoric acid in a certain proportion, polymerized and refined under high temperature, as well as food additives such as polydextrose that have been neutralized and decolorized.

2 Normative References

The documents referenced in this standard are essential for the application of this standard. For dated references, only the version with the date indicated applies to this standard. For undated references, the latest version (including all amendments) applies to this standard.

3 Technical Requirements

3.1 Sensory requirements: Shall comply with the provisions of Table 1.

Table 1

Item	Requirement	Test Method
Color	White to yellowish	Take an appropriate amount of sample and place it in a clean and dry white porcelain plate. Under natural light, observe its color and tissue state, and smell its taste.
Smell	No peculiar smell	
Texture	Granular or powder	

3.2 Physical and chemical index: They should comply with the provisions of

Table 2.

Table2

Item	Index	Test Method
Polydextrose (based on dry and ash free products), w/% ≥	90.0	AppendixA- A.3
Drying reduction, w/% ≤	4.0	GB 5009.3-2010 Direct drying
pH	2.5-7.0	Appendix A -A.4
Ash, w/% ≤	0.3	GB 5009.4
1,6-Dehydrated D-glucose (calculated as dry and ash free), w/% ≤	4.0	Appendix A-A.5
Glucose and sorbitol (calculated as dry and ash free products), w/% ≤	6.0	Appendix A-A.5
5-hydroxymethylfurfural (calculated as dry basis and ash free product), w/% ≤	0.1	Appendix A-A.6
Lead（Pb）/（mg/kg） ≤	0.5	GB 5009.12

Appendix A
(Normative Appendix)
Inspection Method

A.1 General regulations

Unless otherwise specified, only reagents confirmed as analytically pure and water specified in GB/T 6682-2008 shall be used in the analysis. The standard titration solution used in the analysis, the standard solution for impurity determination, the formulation, and the product are prepared in accordance with the provisions of GB/T 601, GB/T 602, and GB/T 603 unless other requirements are specified. The solution used in this experiment refers to aqueous solution when no specific solvent is specified for preparation.

A.2 Identification test

A. 2.1 Reagents and Materials

a) Sulfuric acid.

b) Acetone.

c) Phenol solution: 50g/L.

d)Copper citrate alkaline test solution: Weigh 173g of sodium citrate ($C_6H_5Na_3O_7 \cdot 2H_2O$) and 117g of sodium carbonate ($Na_2CO_3 \cdot H_2O$), dissolve them in approximately 700mL of water under heating, and filter them with filter paper if necessary. In another container, weigh 17.3g of copper sulfate ($CuSO_4 \cdot 5H_2O$) and dissolve it in approximately 100mL of water. Then, slowly add this solution under stable stirring. After cooling, dilute with water to 1000mL and mix well.

A. 2.2 Analysis steps

A. **2.2.1** Take 1 drop of 100 g/L sample solution, add 4 drops of phenol solution, and then quickly add 15 drops of sulfuric acid to produce a deep yellow to orange color.

A. **2.2.2** Take 1mL of 100 g/L sample solution and add 1mL of acetone under strong stirring. The solution should be transparent. Add 2mL of acetone to the transparent solution, stir vigorously, and the solution immediately turns milky white and cloudy.

A. **2.2.3** Take 1mL of 20g/L sample solution, add 4mL of copper citrate alkaline test solution, heat to vigorous boiling for 2-4 minutes, remove the heat source, let it settle and clarify, and the upper clear liquid should be blue or blue-green.

A.3 Determination of polydextrose

A. 3.1 Reagents and Materials

A) α- D-glucose standard: mass fraction ≥ 98.0%.

b) Phenol.

c) Sulfuric acid.

d) Phenol solution: 4g/mL; Accurately weigh 80g of phenol, dissolve in 20mL of water, and mix well.

A. 3.2 Instruments and Equipment

Spectrophotometer.

A. 3.3 Analysis steps

A. 3.3.1 Preparation of glucose standard solution

Weigh an appropriate amount α- D-glucose standard solution, dissolved in water, is prepared into a 0.2 mg/mL standard stock solution. Prepare a series of concentration standard solutions using standard stock solution: 5μg/mL,10μg/mL,20μg/mL,30μg/mL,40μg/mL, 50μg/mL.

A. 3.3.2 Preparation of sample solution

Weigh approximately 0.25 g of the sample (accurate to 0.000 1g), dissolve it in water and bring to a constant volume of 250mL, mix well, pipette 10.0mL, dilute with water and bring to a constant volume of 250mL, this is the sample solution.

A.3.3.3 Drawing of glucose standard curve and determination of sample solution

Use a pipette to pipette 2.0mL of standard solution, sample solution, and distilled water (as blank) at a series of concentrations, and place them in 15mL spiral necked vials without acetone. Add 0.12mL of phenol solution, cap the bottle, and gently mix. Open the bottle stopper and quickly add 5.0mL of sulfuric acid. Cover the small bottle tightly and shake vigorously. Please wear rubber gloves and other safety equipment when adding sulfuric acid.

Keep the vials at room temperature for 45 minutes, then select a suitable spectrophotometer to measure the absorbance value of the solution at 490nm in each vial. Use a phenol sulfuric acid mixture with distilled water as a blank reference during the measurement. Repeat the experiment three times to obtain the average absorbance value of the standard solution with a series of concentrations and the average absorbance value of the sample solution. Draw a standard curve using the average absorbance value of a series of standard solutions as the vertical axis and the concentration of the standard solution（μg /mL） as the horizontal axis.

A.3.4 Result calculation

Calculate the polydextrose content X1 according to equation (A.1):

$$X_1 = 1.05 \times \frac{100(A-Y)}{S \times c} - P_G - 1.11P_L \quad \cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots \text{（A.1）}$$

In the formula:

X1- The content of polydextrose in the sample (calculated as dry basis and ash free sample),%.

1.05- Derive correction factor.

A - Absorbance value of the sample solution.

Y - Y-axis intercept of standard curve.

S - The slope of the absorbance value to the glucose concentration（μg/mL）

standard curve, approximately 0.02

C - Concentration of the sample solution (converted to concentration based on dry basis and ash content of the sample), in micrograms per milliliter（μg/mL）.

PG, PL - The content of glucose and 1,6-dehydrated D-glucose measured separately in monomer experiments,%.

1.11- Conversion coefficient of 1,6-dehydrated D-glucose

A. 4 Determination of pH

Weigh approximately 10g of the sample (accurate to 0.001g), dissolve it in water and bring to a constant volume of 100mL, shake well, and measure with a pH meter.

A.5 Determination of 1-6-Dehydrated D-Glucose, Glucose, and Sorbitol

A. 5.1 Reagents and Materials

a) 1,6-Dehydration D-glucose standard: mass fraction ≥ 98.0%.

b) Glucose(α- D-glucose)standard: mass fraction ≥ 98.0%.

c) Sorbitol standard: mass fraction ≥ 98.0%.

d) Sulfuric acid.

A. 5.2 Instruments and Equipment

High performance liquid chromatography with differential refractive detector.

A. 5.3 Reference chromatographic conditions

a) Chromatographic column: Sulfonic acid type styrene divinylbenzene copolymer

resin column (styrene as monomer, divinylbenzene as crosslinking agent, size exclusion+ion exchange; separation mode: exclusion limit greater than 1000, theoretical number of plates n ≥ 17000), column length 300mm, column inner diameter 8mm; Or other equivalent chromatographic columns.

b) Mobile phase: Suck 0.42mL of sulfuric acid, dilute with water to 1000mL, and use 0.45 μm membrane filtration, degassing with ultrasound for 15 minutes.

c) Column temperature: 60 ℃.

d) Flow rate: 0.5 mL/min.

e) Injection volume: 20 μL.

A. 5.4 Analysis steps

A. 5.4.1 Preparation of standard solution

Table A.1 Preparation Method of Standard Solution

Serial Number	Standard sample quality (g)	Liquid volume (mL)	Standard sample concentration (g/L)
1	0.005	50	0.1
2	0.01	50	0.2
3	0.02	50	0.4
4	0.03	50	0.6

Prepare standard samples of 1,6-dehydrated D-glucose, glucose, and sorbitol according to the above table. Weigh 0.005g, 0.01g, 0.02g, and 0.03g of each standard sample, dissolve them in water and make a constant volume of 50mL. Prepare standard samples of series concentrations for each of the three standards, filter them with 0.45 μm filter membranes, and use them for backup.

A. 5.4.2 Preparation of sample solution

Weigh approximately 1g of polydextrose sample (accurate to 0.000 1g), dissolve it in water and bring to a constant volume of 25mL. The sample solution is filtered with a 0.45 μm filter membrane and used for backup.

A. 5.4.3 Determination

Under the reference chromatographic conditions of A.5.3, the standard samples of a

series of concentrations of 1,6-dehydrated D-glucose, glucose, and sorbitol were measured, and the experiment was repeated twice to obtain the average peak area value of the standard samples. Draw standard curves for 1,6-dehydrated D-glucose, glucose, and sorbitol using the average peak area value of the standard sample as the ordinate and the concentration of the standard sample series (g/L) as the abscissa.

Under the above chromatographic conditions, the sample solution is determined and qualitatively determined based on the retention time of the standard. Repeat the experiment twice to obtain the average peak area value.

According to the linear relationship between the average peak area value of the standard sample and the concentration of the standard sample, the concentrations of 1,6-dehydrated D-glucose, glucose, and sorbitol in the sample solution were obtained in g/L.If the concentrations of 1,6-dehydrated D-glucose, glucose, and sorbitol in the sample solution (g/L) are not within the standard curve range, the concentration of the sample solution should be adjusted.

A. 5.5 Result calculation

Calculate the content of 1,6-dehydrated D-glucose X_2 according to formula (A.2):

$$X_2 = \frac{c_1}{c_2} \times 100\% \cdots\cdots (A.2)$$

In the formula:

X_2- The content of 1,6-dehydrated D-glucose in the sample (calculated as dry basis and ash free product),%.

C_1- The concentration of 1,6-dehydrated D-glucose obtained from the standard curve, in grams per liter (g/L).

C_2- The concentration of the sample solution (converted to concentration based on dry basis and ash content of the sample), in grams per liter (g/L).

The calculation of glucose content and sorbitol content in the sample is the same as the calculation of 1,6-dehydrated D-glucose content in the sample. The content of glucose and sorbitol in the sample is the sum of both.

The experimental results are based on the arithmetic mean of the parallel measurement results. The absolute difference between two independent measurement results obtained under repeatability conditions shall not exceed 5% of the arithmetic mean.

A. 6 Determination of 5-hydroxymethylfurfural

A. 6.1 Instruments and Equipment

Spectrophotometer.

A. 6.2 Analysis steps

A. 6.2.1 Preparation of sample solution

Weigh approximately 1g of polydextrose sample (accurate to 0.000 1g), dissolve in water and bring to a constant volume of 100 mL, mix well, and set aside.

A. 6.2.2 Determination

Select a suitable spectrophotometer, use a 1cm quartz cuvette, and use water as a blank reference to determine the absorbance value of the sample solution at a wavelength of 283 nm.

A. 6.3 Result calculation

Calculate the content of 5-hydroxymethylfurfural according to formula (A.3):

$$X_3 = \frac{0.749 \times A}{c_3} \quad \text{(A.3)}$$

In the formula:

X_3- Content of 5-hydroxymethylfurfural in the sample (calculated as dry basis and ash free product),%.

0.749- Combination ratio constant, including extinction coefficient, molecular weight, unit and volume conversion.

A - The absorbance value of the sample solution.

C_3- Concentration of the sample solution (converted to concentration based on dry basis and ash content of the sample), in milligrams per milliliter (mg/mL).