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NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB 1903.30-2022

National food safety standard - Food nutrition enhancer - Polyfructose

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1 Scope

This document specifies food nutrition fortifier polyfructose prepared by using chrysanthemum or chrysanthemum root as raw material, using acid/alkali treatment or other methods to remove impurities, and using drying process.

2.2 Structural formula

Polyfructose is a carbohydrate with fructose units linked by beta-2,1-bonds and terminated by glucose units with a total of 5~60 units.

Annex A Inspection method

Determination of polyfructose (>=DPS), average degree of polymerization (DP), glucose + fructose + sucrose content A.1 General provisions Unless otherwise specified, only the reagents confirmed as analytically pure, and the grade one water specified in GB/T 6682 are used in the analysis. All solutions used are aqueous solutions unless otherwise specified.

A.2 Reagents and materials A.2.1 Acetic acid: 0.2mol/L.

A.2.2 Sodium acetate: 0.2mol/L.

A.2.3 pH 4.5 acetate buffer solution: Mix 28.0mL of 0.2mol/L acetic acid and 22.0mL of 0.2mol/L sodium acetate. Use water to dilute to 100mL.

A.2.4 Hydrochloric acid solution: 0.05mol/L.

A.2.5 Potassium hydroxide solution: 0.05mol/L.

A.2.6 Amyloglucosidase: For enzymatic hydrolysis of amylase to glucose. The content of glucose, fructose and sucrose shall be less than 0.02%. Store in a 4°C refrigerator when not in use.

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A.2.7 Fructase: For enzymatic hydrolysis of polyfructose to glucose and fructose. The content of glucose, fructose and sucrose shall be less than 0.005%. Store in a 4°C refrigerator when not in use.

A.2.8 Acetonitrile.

A.2.9 Glucose: d (+) anhydrous dextrose.

A.2.10 Fructose: Dextrose.

A.2.11 Sucrose.

A.2.12 Sugar standard stock solution: Weigh 6.00g of the sugar standard product (2.00g each of glucose, fructose, and sucrose) that has been dried at 96°C +/- 2°C for 4 h. Put into a 50mL beaker. Add water to stir till it is completely dissolved. Transfer it into a 50mL volumetric flask. Set volume constant to the scale. Obtain the sugar standard stock solution (the glucose content is 40.0mg/mL, the fructose content is 40.0mg/mL, and the sucrose content is 40.0mg/mL).

A.2.13 Standard working solution: Use water to respectively dilute the sugar standard stock solution to 10.0mg/mL, 7.00mg/mL, 4.00mg/mL, 2.00mg/mL and 1.00mg/mL solutions.

A.3 Instruments and equipment A.3.1 Analytical balance: The resolution is 1mg and 0.01g.

A.3.2 pH meter: The accuracy is +/-0.05.

A.3.3 Constant temperature water bath.

A.3.4 High performance liquid chromatograph (with differential refractive index or equivalent detector).

A.3.5 0.22um microporous membrane.

A.3.6 Oven.

A.3.7 25mL, 50mL volumetric flasks.

A.3.8 Pipettes (1mL, 5mL, 10mL).

A.4 Analysis steps A.4.1 Sample weighing Weigh about 1g of sample (to the nearest of 1mg, as mass m1) that has been dried at 96°C+/-2°C for 4h. Put it into a 100mL beaker. Add 50mL of boiling water. Moderately stir. Use potassium hydroxide solution (A.2.5) or hydrochloric acid solution (A.2.4) to adjust pH to 6.5~8.0. After the sample is completely dissolved, transfer it to a 100mL volumetric flask. Incubate at 85°C +/- 2°C for 10min. Then cool it to room temperature.

Use water to set volume to constant. Shake well. After that, take care to keep the sample temperature above room temperature. Take a small amount of solution. Filter it through a 0.22um filter membrane. Then analyze it (test A0).

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A.4.2 Enzymatic hydrolysis 1 Take 60.0mL of sample solution (A.4.1). Add 30.0mL of acetate buffer solution (A.2.3).

Mix well. Use 0.05mol/L hydrochloric acid solution (A.2.4) to adjust pH = 4.5+/-0.05.

Add a sufficient amount of amyloglucosidase (A.2.6) [if the approximate content of starch and maltodextrin in the sample cannot be determined, it can be considered that 100% of the unknown part of the sample is starch, so as to calculate the amount of enzyme]. The sample liquid water bath is heated to 60°C +/- 2°C. Maintain at constant temperature for 30min. Stir moderately but it shall not foam. There shall be no air bubbles in the solution. After it is cooled to room temperature, transfer it to a 100mL volumetric flask. Use water to set volume to constant. Take a small amount of solution.

Filter it through a 0.22um filter membrane. Then analyze it (test A1).

A.4.3 Enzymatic hydrolysis 2 Take 60.0mL of sample solution (A.4.2). Add enough fructase (A.2.7) (if the polyfructose content cannot be determined, the unknown part of the sample can be considered to be 100% polyfructose; if using megazyme 2000U/mL mixed fructase, add 100uL). The sample liquid water bath is heated to 60°C +/- 2°C. Maintain at constant temperature for 30min. Stir moderately. Prevent foaming. Prevent air from entering.

After it is cooled to room temperature, transfer it to a 100mL volumetric flask. Use water to set volume to constant. Filter it through a 0.22um filter membrane. Then analyze it (test A2).

A.5 Chromatographic detection Reference chromatographic conditions: Amino column or equivalent column. The mobile phase is acetonitrile: water = 75%:25%. The flow rate is 1.0mL/min. The injection volume is 20uL. The column temperature is 30°C.

Drawing of standard curve: Respectively pipette 20uL of standard working solution (A.2.13). Inject it into the high-performance liquid chromatography. Determine the peak height or peak area of the standard solution under the above chromatographic conditions. Draw a standard curve with the concentration as the abscissa and the peak height or peak area as the ordinate.

Determination of sugar in sample: Pipette 20uL of sample solution (test A0, A1 and A2).

Inject it into the high-performance liquid chromatography. Determine the peak height or peak area of the sample under the above chromatographic conditions. Check the contents of fructose, glucose, and sucrose in the sample solution from the standard curve. The response values of fructose, glucose and sucrose in the sample solution shall be within the linear range of the standard curve. If it exceeds the linear range, the sampling amount shall be changed and re-tested.

A.6 Result calculation A.6.1 Calculation of the contents of fructose, glucose, and sucrose The sugar contents corresponding to different tests are shown in Table A.1.

Table A.1 -- Sugar contents corresponding to different tests

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Sugar Test A0 Test A1 Test A2 Glucose Gf G1 Gt Fructose Ff Ft Sucrose S A.6.3 Calculation of average degree of polymerization (DP)

The content of glucose and fructose released from polyfructose is calculated according to formula (A.8) and formula (A.9), respectively.

Where, Gi - Glucose content (mass fraction) released from polyfructose, %;

Gs - Glucose (=S/1.9) content (mass fraction) released from sucrose, %;

G1 - Glucose content (mass fraction) after the first enzymatic hydrolysis, %;

Gt - Glucose content (mass fraction) after the second enzymatic hydrolysis, %;

Ft - Fructose content (mass fraction) after the second enzymatic hydrolysis, %;

Ff - Fructose content (mass fraction) in the initial extract, %;

Fi - Fructose content (mass fraction) released from polyfructose, %;

Fs - Fructose (=S/1.9) content (mass fraction) released from sucrose, %.

The average degree of polymerization is calculated according to the formula (A.10).

Where, DP - Average degree of polymerization;

Fi - Fructose content (mass fraction) released from polyfructose, %;

Gi - Glucose content (mass fraction) released from polyfructose, %.

A.6.4 Calculation of polyfructose (>=DP5) content The content of polyfructose (>=DP5) is calculated according to formula (A.11) and formula (A.12).

Annex B Inspection method

Determination of fructooligosaccharides (DP3-DP4) content B.1 General provisions Unless otherwise specified, only the reagents confirmed as analytically pure, and the grade one water specified in GB/T 6682 are used in the analysis. All solutions used are aqueous solutions unless otherwise specified.

B.2 Reagents and materials B.2.1 Acetonitrile.

B.2.2 Kestose (content >= 99%).

B.2.3 Nystose (content >=99%).

B.2.4 Sugar standard stock solution: Respectively weigh 1.000g of sugar standard product that has been dried at 96°C+/-2°C for 4h (kestose and nystose) into a 50mL volumetric flask. Add water to stir till it is completely dissolved. Set volume constant to the scale. Obtain the 20mg/mL standard stock solution.

B.2.5 Working standard solution: Depending on the detector, the sugar standard stock

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