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Quality requirements for starch sugar –

Part 4: High fructose syrup

淀粉糖质量要求 第 4 部分: 果葡糖浆

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Quality requirements for starch sugar – Part 4: High fructose syrup

1 Scope

This document specifies the quality requirements for high fructose syrup, including terms and definitions, product classification, requirements, test methods, inspection rules, labeling, packaging, transportation, and storage.

This document is applicable to the production, inspection, and sale of high fructose syrup.

2 Normative references

The following documents contain the provisions which, through normative reference in this document, constitute the essential provisions of this document. For the dated referenced documents, only the versions with the indicated dates are applicable to this document; for the undated referenced documents, only the latest version (including all the amendments) is applicable to this document.

GB/T 191 Packaging - Pictorial marking for handling of goods

GB/T 601 Chemical reagent - Preparations of reference titration solutions

GB/T 602 Chemical reagent - Preparations of standard solutions for impurity

GB/T 603 Chemical reagent - Preparations of reagent solutions for use in test methods

GB/T 6682 Water for analytical laboratory use - Specification and test methods

3 Terms and definitions

The following terms and definitions apply to this document.

3.1 high fructose syrup

fructose syrup

Starch sugar products whose main components are fructose and glucose that are made

The water used in this method shall meet the water specifications in GB/T6682 unless other requirements are specified, and the reagents used shall be analytical reagents unless other specifications are specified. The standard titration solution used in the analysis, the standard solution for the determination of impurities, preparations, and products, unless otherwise specified, shall be prepared in accordance with the provisions of GB/T 601, GB/T 602, and GB/T 603.

Note: Liquid products with crystals can be detected after heating and dissolving in an appropriate way.

6.2 Sensory requirements

Take an appropriate amount of sample, observe the state and color of the sample under natural light, smell the odor, add pure water at room temperature to prepare a solution with 10% ~ 15% dry matter, and taste it.

6.3 Fructose and glucose content (calculated as dry matter)

6.3.1 Principle

The components entering the chromatographic column at the same time, due to the different effects of distribution, size exclusion, or ligand exchange between the mobile phase and the stationary phase, move at different speeds in the chromatographic column. After flowing past a certain length of the chromatographic column, they are separated from each other, and enter the detector in a certain order to generate response signals; the signals are collected by computer software and processed, and then the chromatogram and the retention value and peak area or peak height of the components contained in the sample are obtained. According to the retention time, the qualitative analysis is carried out by the control method; according to the peak area, the content of each component is calculated quantitatively.

6.3.2 Reagents and solutions

6.3.2.1 Water: Grade I water in GB/T 6682.

6.3.2.2 Glucose standard (CAS No.: 492-62-6, purity ≥ 98%).

6.3.2.3 Fructose standard (CAS No.: 7660-25-5, purity ≥ 98%).

6.3.2.4 Serial standard solutions of glucose and fructose: Use each sugar standard to prepare 5 series of standard solutions with different concentrations in the range of 0.5 mg/mL~10.0 mg/mL.

6.3.3 Instruments and equipment

6.3.3.1 High-performance liquid chromatography: it shall be equipped with a differential refraction detector and a column constant temperature system.

6.3.3.2 Mobile phase vacuum filtration degassing device and 0.2 μm or 0.45 μm microporous membrane.

6.3.3.3 Electronic balance: The sense quantity shall be 0.0001 g.

6.3.3.4 Micro sampling syringe.

6.3.4 Reference chromatographic conditions

6.3.4.1 Chromatographic column: a cation exchange resin column (with a column length of 300 mm, an inner diameter of 7.8 mm, and a filler particle size of 5 μm or 9 μm), or a chromatographic column with an equivalent analytical effect.

6.3.4.2 Mobile phase: water.

6.3.4.3 Detector temperature: 45 °C.

6.3.4.4 Column temperature: 85 °C.

6.3.4.5 Flow rate: 0.5 mL/min.

6.3.4.6 Injection volume: 10 μL.

6.3.5 Analysis steps

6.3.5.1 Preparation of the sample solution

Weigh 0.5 g of the sample (calculated as dry matter; the content of various sugar components shall be within the range of the standard solution series, otherwise the sampling amount can be added or reduced appropriately), and the weight shall be accurate to 0.0001 g; dissolve it in water, transfer it to a 50 mL volumetric flask, and dilute it to the mark with water; shake well, filter it with 0.2 μm or 0.45 μm aqueous microporous membrane, and leave the filtrate for later use.

6.3.5.2 Drawing of the standard curve

After the series of standard solutions of glucose and fructose are injected respectively, draw the standard curve according to the concentration of the series of standard solutions versus the peak area. The linear correlation coefficient shall be above 0.9990.

6.3.5.3 Determination of samples

Inject the prepared sample solution. Qualitatively analyze the chromatographic peaks of various sugar components in the sample according to the retention time of the standard. According to the peak area of the sample, calculate the percentage content of various sugar components by the external standard method or the peak area normalization method; the external standard method is the arbitration method.

6.4 Dry matter (solid matter)

6.4.1 Instruments and equipment

6.4.1.1 Abbe refractometer: The accuracy shall be 0.0001.

6.4.1.2 Glass rod: The end shall be bent and flat.

6.4.2 Instrument calibration

At 20 °C, the refractive index of the refractometer calibrated with pure water is 1.3330, which is equivalent to zero content of dry matter (solid matter). The instrument shall be calibrated at least once a day.

6.4.3 Analysis steps

6.4.3.1 Determination method

Put the refractometer in a place with sufficient light, and adjust the temperature of the refractometer prism to 20 °C; separate the two prisms, add a small amount of sample (one drop to two drops) to the fixed prism surface with a glass rod (the glass rod shall not touch the prism surface, and the time for dropping sample shall be less than 2 s), immediately close the prism and stay for a few minutes, so that the sample reaches the temperature of the prism. Adjust the knob of the prism until the field of view is divided into light and dark parts, turn the compensator knob to eliminate the iridescence and make the dividing line between light and dark clear; continue to adjust the knob to align the dividing line between light and dark to the cross line, read the refractive index (the reading shall be accurate to 0.0001) or content value from the scale, re-read it immediately, and take the average value as the measured value for one time. Clean and dry the two prisms, and carry out the second measurement with the same sample according to the above operation; or, according to Appendix A, obtain the content of dry matter (solid matter) in the sample by using the refractive index to look-up the table, and use the looking-up table method as the arbitration method.

6.4.3.2 Result presentation

Take the arithmetic mean of two direct readings as the result for reporting.

6.4.4 Precision

The absolute difference between two independent determination results obtained under repeatability conditions shall not exceed 1% of their arithmetic mean.

6.5 pH

6.5.1 Principle

Insert the glass electrode (indicating electrode) and the calomel electrode (reference electrode) into the solution to be measured together to form a battery, the electromotive force of the battery is related to the pH of the solution, measure the electromotive force of the battery, and then obtain the pH of the solution.

6.5.2 Instruments

6.5.2.1 pH meter: The accuracy shall be ±0.01 and equipped with an electromagnetic stirrer.

6.5.2.2 Beaker: 250 mL.

6.5.2.3 Electronic balance: The sense quantity shall be 0.1 g.

6.5.3 Analysis steps

6.5.3.1 Determination method

Debug and calibrate the pH meter according to the instrument manual. Weigh 100 g of the sample and place it in a clean 250 mL beaker, and put a stir bar in it. Then, rinse the electrode head with water, and dry it gently with filter paper; then, insert the electrode into the sample to be tested, turn on the electromagnetic stirrer, and adjust the temperature compensation; measure the pH of the sample solution, and take the reading after it is stabilized for 1 min, which is the pH of the sample.

6.5.3.2 Result presentation

The result obtained is rounded to one decimal place.

6.5.4 Precision

The absolute difference between two independent determination results obtained under repeatability conditions shall not exceed 3% of their arithmetic mean.

6.6 Chroma

6.6.1 Principle

When a beam of parallel monochromatic light passes through a colored solution, the darker the solution, the greater the absorbance.

6.6.2 Instruments

6.6.2.1 Spectrophotometer: The wavelength range shall be 420 nm~720 nm.

6.6.2.2 Cuvettes.

6.6.2.3 Electronic balance: The sensitivity shall be 0.1 g.

6.8.2 Instruments and equipment

6.8.2.1 Porcelain crucible: 50 mL.

6.8.2.2 High-temperature furnace: The control range of the temperature shall be 525 °C±25 °C.

6.8.2.3 Desiccator: The desiccant shall be silica gel.

6.8.2.4 Analytical balance: The accuracy shall be 0.0001 mg.

6.8.3 Analysis steps

6.8.3.1 The crucible shall be boiled and washed with hydrochloric acid first, then rinsed with tap water, and then rinsed with distilled water. Put the cleaned crucible in a high-temperature furnace, and burn it at 525 °C±25 °C for 0.5 h; then open the furnace door, move the crucible to the furnace mouth, and take it out after it cools to below 200 °C; put it in a desiccator, cool it to room temperature, and weigh it accurately; repeat burning until its weight becomes constant (the difference between two weighings is not more than 0.3 mg).

6.8.3.2 Weigh about 2 g of the sample (the weight shall be accurate to 0.0001 g), and put it into a crucible that has been burned to constant weight; add 1 mL of concentrated sulfuric acid dropwise, rotate slowly to make them mix well, place the crucible on an electric furnace and heat it carefully until the mixture is completely carbonized. Then, put the crucible in a high-temperature furnace, burn it at 525 °C±25 °C, and keep this temperature until all the carbonized substances disappear (at least 2 h). Take the crucible out and cool it, add a few drops of concentrated sulfuric acid to moisten the residue, put the crucible back into the high-temperature furnace, and burn it until the residue is completely ashed; open the furnace door, move the crucible to the furnace mouth, and take it out after it cools to below 200 °C; put it in a desiccator, cool it to room temperature, and accurately weigh it; repeat burning until its weight becomes constant (the difference between two weighings is not more than 0.3 mg).

6.8.4 Calculation of the result

The content of the sulfated ash in the sample is calculated according to formula (5):

$$X_4 = \frac{m_7 - m_5}{m_6 - m_5} \times 100 \qquad \cdots\cdots (5)$$

where:

X_4 --- The content of the sulfated ash in the sample, in grams per hundred grams (g/100g);

m_7 --- The mass of the crucible and ash, in grams (g);

m_5 --- the mass of the crucible, in grams (g);

m_6 --- The mass of the crucible and the sample, in grams (g).

The calculation result is rounded to three decimal places.

6.8.5 Precision

The absolute difference between two independent determination results obtained under repeatability conditions shall not exceed 5% of their arithmetic mean.

6.9 Light transmittance

6.9.1 Instruments and equipment

Spectrophotometer: The measurable wavelength range shall be 420 nm~720 nm.

6.9.2 Analysis steps

6.9.2.1 Determination method

According to the instrument manual, adjust the zero point and light transmittance of the instrument at a wavelength of 720 nm. Weigh an appropriate amount of sample, and prepare the high fructose syrup test solution with a dry matter of 30% by boiled and cooled (the carbon dioxide is removed) water with a pH of 5.0~7.0. Then, inject the liquid to be tested into a 1 cm cuvette, and use a spectrophotometer to measure the light transmittance of the sample liquid at a wavelength of 720 nm, using the same batch of water as a reference.

6.9.2.2 Result presentation

The result obtained is rounded to one decimal place.

6.9.3 Precision

The absolute difference between two independent determination results obtained under repeatability conditions shall not exceed 2% of their arithmetic mean.

7 Inspection rules

7.1 Batching

The products of uniform quality, which are continuously produced with the same raw material, same formula, same process, and from the same production line, are grouped into a batch.

e) When the national supervisory agency implements random inspection according to the relevant regulations.

7.5 Judgment rules

7.5.1 After the sample are inspected, and all the inspection items meet the requirements, then the batch of products can be judged to conform to this document.

7.5.2 If one or two of the inspection items do not meet the requirements, twice the number of samples shall be taken from the same batch of products for re-inspection, and the results of the re-inspection shall prevail. If there is still one item that does not meet the requirements, it shall be judged that the batch of products does not conform to this document. If there are three or more indicators in the inspection results that do not meet the requirements, it shall be judged that the batch of products does not conform to this document.

8 Labeling, packaging, transportation, and storage

8.1 Labeling

8.1.1 The product category shall be indicated on the label or instructions.

8.1.2 The pictorial marking for packaging, storage, and transportation shall meet the requirements of GB/T 191.

8.2 Packaging

8.2.1 Packaging containers (bottles, barrels, bags, etc.) shall be clean and undamaged.

8.2.2 If the high fructose syrup is transported by tank cars, special tank cars shall be used.

8.3 Transportation

8.3.1 The means of transport shall be clean.

8.3.2 The high fructose syrup shall not be loaded and transported with toxic, harmful, corrosive, or odorous items, and shall be protected from moisture, pressure, and exposure to the sun. When loading and unloading, the high fructose syrup shall be handled with care, and the package shall not be hooked directly.

8.4 Storage

8.4.1 The high fructose syrup shall be stored in a ventilated, dry, and clean warehouse. Exposure to the sun and rain shall be avoided, and fire is strictly prohibited.

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