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National Standard of the People's Republic of China

GB/T 235282—2009

Fructooligosaccharide

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Foreword

This standard is based on QB 2581-2003 “Fructooligosaccharide”.

Annex A of this standard is normative, and Annex B. Annex C is an informative appendix.

This standard was proposed by the China National Light Industry Council.

This standard is under the jurisdiction of the Industrial Fermentation Subcommittee of the National Food Industry Standardization Technical Committee.

GB/T 235282—2009

Fructooligosaccharide

1 Scope

This standard specifies the terms and definitions of oligofructose, product classification, requirements, test methods, inspection rules, signs, packaging, transportation and storage.

This standard applies to the production, inspection and sales of fructo-oligosaccharides made from sucrose, or from the roots of Jerusalem artichoke and chicory.

2 Normative references

The clauses in the following documents become clauses of this standard after being quoted. For dated reference documents, all subsequent amendments (excluding errata content) or revisions do not apply to this standard. However, all parties that have reached an agreement based on this standard are encouraged to study whether the latest versions of these documents can be used . For undated references, the latest version is applicable to this standard.

GB/T 191 packaging, storage and transportation pictorial signs

GB317 white granulated sugar

GB/T 6682 Analysis of laboratory water specifications and test methods (GB/T 6682- -2008, ISO 3696:1987, MOD)

GB 7718 General Rules for Labeling of Prepackaged Food

GB 15203 Starch Sugar Hygienic Standard

GB 16740 General Standard for Health (Function) Food

GB/T 20884 Maltodextrin

GB/T 20885

Glucose syrup

3 Terms and Definitions

The following terms and definitions apply to this standard.

3.1

Fructooligosaccharide, FOS

A functional oligosaccharide with a degree of polymerization of 2-9, which is formed by the (β 2→1) glycosidic linkage of fructosyl groups, is a food ingredient.functional oligosaccharide with a degree of polymerization of 2-9, which is a functional oligosaccharide with a degree of polymerization of 2-9, is a food ingredient.

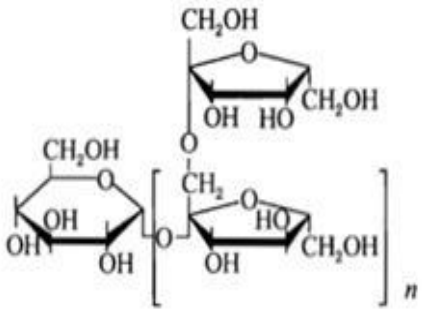
3.2

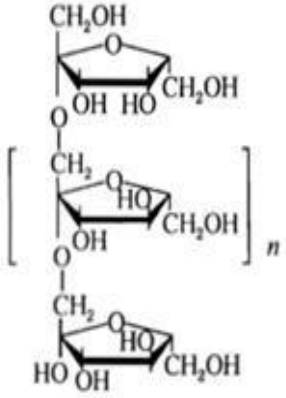
The total content of fructooligosaccharides.

The total content of fructooligosaccharides is fructotriose (GF1), fructotriose (F), fructotetraose (GF), fructotetraose (F,), fructopentaose (GF,). The sum of pentasaccharide (F,), fructose (GF). Fructose (Fn) is the percentage of a thousand substances.

4 Product Classification

4.1 According to the structure, it is divided into cane-fruit type fructooligosaccharide and fruit-fruit type fructooligosaccharide. The molecular structures are as follows:





Schematic diagram of the molecular structure of cane-fruit fructooligosaccharides **Schematic diagram of the molecular structure of fruit-fruit-type fructooligosaccharides**

4.2 According to the form, it is divided into liquid (L) and solid (S).

5 Claim

5.1 Sensory Requirements

Liquid oligofructose is a colorless or light yellow, transparent and viscous liquid, with a unique aroma of this crystal, soft and refreshing sweetness, no peculiar smell, and no visible impurities for normal vision.

The solid oligofructose is white or slightly yellow, with a unique aroma of this product, with a soft and refreshing sweetness, no peculiar smell, and no visible impurities.

5.2 Physical and Chemical Requirements

Should meet the requirements of Table 1.

Table 1

Project	Liquid (L)						Solid (S)					
	50	55	70	75	90	95	50	55	70	75	90	95
Moisture (mass fraction)/% ≤	–	–					5.0					
Dry matter (solid matter, mass fraction) ≥	70	75					–					
PH	4.5~7.0											
Total content of oligofructose (accounting for dry matter, mass fraction)/% ≥	50.0	55.0	70.0	75.0	90.0	95.0	50.0	55.0	70.0	75.0	90.0	95.0
Conductivity ash (mass fraction)/% ≤	0.4											
Chroma ≤	0.2						–					

Transmittance/%	≥	85	–

5.3 Hygiene requirements

5.3.1 As a food ingredient, the hygienic index should meet the requirements of GB 15203.

5.3.2 As the sanitary index of the end product, it shall meet the requirements of GB 16740.

6 Test method

The water used in this standard, when other requirements are not specified, should meet the specifications of Grade 3 or higher (including Grade 3) water in GB 6682. All reagents used refer to analytic-al grade (AR) when other specifications are not indicated.

6.1 Appearance

6.1.1 Liquid

Take about 30mL of the sample in a colorless, clean and dry sample cup (or 50mL small beaker), place it in a bright place, observe its color and clarity with the naked eye, and check whether there are visible impurities with normal vision; take a suitable amount of sample and put it in the mouth Taste its taste in the middle (before tasting each sample, apply water to rinse your mouth), and make a sensory record.

6.1.2 Solid

Take an appropriate amount of sample, observe the color and shape of the sample with the naked eye under suitable natural light, and check whether it has visible impurities with normal vision; take an appropriate amount of sample and put it in the population to taste its taste (before tasting each sample, apply water to rinse your mouth), Make sensory records.

6.2 Moisture

Carry out according to the method specified in GB/T 20884.

6.3 Dry matter (Solid matter)

6.3.1 Apparatus

6.3.1.1 Abbe refractometer: the accuracy is 0.0001 units.

6.3.1.2 Constant temperature water bath: accuracy ±0.1 C.

6.3.1.3 Glass rod: the end is bent and flat.

6.3.2 Instrument calibration

At 20 C, the refractive index of the refractometer calibrated with redistilled distilled water is 1.333 0, which is equivalent to zero dry matter (solids) content. The instrument is calibrated at least once a day.

6.3.3 Analysis steps

Place the refractometer in a well-lit position, connect it to a constant temperature water bath, adjust the temperature of the refractometer prism to 20 C. Separate the two prisms, and use a glass rod to add a small amount of sample (1 drop to 2 drops) on the fixed prism surface (The glass rod must not touch the prism surface, and the sample application time should be less than 2 s), immediately close the prism and stay for a few minutes to make the sample reach the temperature of the calibration lens. Adjust the spiral of the prism until the field of view is divided into light and dark parts. Turn the compensator knob to eliminate the rainbow and make the light and dark

boundary clear. Continue to adjust the spiral to align the light and dark dividing line on the cross line, read the refractive index (reading accurately to 0.00 1) and the percentage of dry matter from the scale, and then reread it immediately--times, and take the average value as- -The measured value. Clean and dry the two prisms. Perform the second measurement on the same sample as described above. Take the arithmetic average of the two measurements and report the result, which is the dry matter content of the sample (if the measurement temperature is not 20 C, the temperature should be corrected according to Appendix A).

6.4 pH

Carry out according to the method specified in GB/T 20885.

6.5 Determination of total oligofructose content (high performance liquid chromatography)

6.5.1 Principle

Each component entering the chromatographic column at the same time, due to the different effects of dissolution, adsorption, permeation or ion exchange between the mobile phase and the stationary phase, the mobile phase is repeatedly distributed between the two phases of the chromatographic column. Due to the different moving speeds of each component in the chromatographic column, after a certain length of the chromatographic column, they are separated from each other, flow out of the chromatographic column in order, enter the signal detector, and display each component on the recorder or data processing device. The peak value of the component is quantified by the external standard method or the peak area normalization method according to the retention time, and the external standard method is the arbitration method.

6.5.2 Apparatus

6.5.2.1 High-performance liquid chromatograph (equipped with differential refractive index detector or evaporative light scattering detector and column thermostat system).

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

6.5.2.3 Chromatographic column: amino column.

6.5.2.4 Analytical balance: sensitivity 0.000 1 g.

6.5.2.5 Micro sampler: 10 μL.

6.5.3 Reagents

6.5.3.1 Water: secondary distilled water or ultra-pure water (through 0.45 μm water system microporous membrane).

6.5.3.2 Acetonitrile: Chromatographically pure.

6.5.3.3 Standard solutions: glucose, fructose, sucrose, sucrose, sucrose tetraose, sucrose pentaose, sucrose hexaose standard products, respectively with ultrapure water to prepare 40 mg/mL aqueous solution.

6.5.3.4 Mobile phase: acetonitrile: water (volume ratio) = 75: 25 (the ratio can be adjusted according to the actual situation).

6.5.4 Analysis steps

6.5.4.1 Preparation of sample solution

Weigh an appropriate amount of liquid or solid samples (so that the content of various components should be within the linear range of 6. 5. 4. 2.2 standard solution), dilute to 100 ml with ultrapure water, shake well, and filter with a 0.45 μm membrane (12000r/min, Centrifuge for 5 mins), and collect the filtrate as the sample solution to be tested.

6.5.4.2 Determination

6.5.4.2.1 Install the chromatographic column on the day before the measurement, the column temperature is indoor temperature, turn on the power of the differential refractive index detector (or evaporative light scattering detector), preheat and stabilize, and flow into the phase equilibrates at a flow rate of 0.1 mL/min overnight. Before the formal sample injection and analysis, if a differential refractive index detector is used, input the mobile phase used into the reference cell at a flow rate of 0.1 mL/min for more than 20 minutes, and then restore the normal flow path so that the mobile phase passes through the sample cell (the evaporative light scattering detector does not need this operation), adjust the flow rate to 1.0 mL/min to take the baseline, and inject the sample after the baseline stabilizes. The injection volume is 5 μL~10 μL.

6.5.4.2.2 In the range of 0.4 mg/mL~40 mg/mL, prepare 6 standard solution series with different concentrations of the standard solution, and then draw a standard curve based on the concentration of the standard sample versus the peak area after sample injection. The linear correlation coefficient should be above 0.9990, otherwise the concentration range needs to be adjusted.

6.5.4.2.3 Inject the standard solution and the prepared sample separately. According to the retention time of the standard sample, the chromatographic peaks of various sugar components in the sample are qualitatively determined. According to the peak area of the sample, the percentage content of various sugars is calculated by the external standard method or the peak area normalization method.

Note 1: The active ingredients of fructo-oligosaccharides based on normal sugar only include fructotriose (GF;), fructotetraose (GF) , fructopentaose (GF,) and fructohexaose (GF;) .

Note 2: Fructose oligosaccharides using Jerusalem artichoke and chicory as raw materials, the chromatogram of fructotriose (F), fructotetraose (F,), fructo-fructose (F,), fructohexaose (F) The peaks are respectively contained in the color peaks of sucrose (GF), sucrose (GF,), ricinose (GF,), and fructohexaose (GF,).

Note 3: As there is no standard sample for fructotriose (F,), fructotetraose (F,), fructose (F,), fructohexaose (F,), using Jerusalem artichoke and Bangying as raw materials The peak area normalization method should be used when calculating the content of fructooligosaccharides.

6.5.4.3 Calculation

6.5.4.3.1 External standard method

The percentage of each component in the sample is calculated according to formula (1):

$$X_i = \frac{A_i \times \frac{m_s}{V_s}}{A_s \times \frac{m}{V}} \times 100 \qquad \cdots\cdots(1)$$

In the formula:

Xi: Component i (Glucose, Fructose, Sucrose, Sucrose triose, Sucrose tetraose, Sucrose pentaose, Sucrose hexaose) in the sample accounted for the percentage of dry matter (mass fraction), %;

Ai: The peak area of component i in the sample;

m_s: the mass of a component sugar standard in the standard sample, in grams (g);.

V_s: Standard sample dilution volume, in milliliter (mL);

A_s:the peak area of a certain component sugar standard crystal in a standard sample;

m: Liquid is the mass of the dry matter in the weighed sample, and solid is the mass of the sample minus the moisture content, in grams (g);

V: The dilution volume of the same product, in milliliters (mL).

The percentage of oligofructose in the sample is calculated according to formula (2):

$$FOS\% = GF_2 + GF_3 + GF_4 + GF_5 \quad \cdots\cdots(2)$$

In the formula:

FOS%: otal fructooligosaccharide content (accounting for dry matter, mass fraction), %;

GF_2 , GF_3 , GF_4 , GF_5 :-are the percentages of sucrose triose, sucrose tetraose, sucrose pentaose, and sucrose hexaose (accounting for dry matter, mass fraction), %.

The calculation result is kept to one decimal place.

6.5.4.3.2 Peak area normalization method

The peak area normalization method is used to calculate the percentage of dry matter content of each component. Because all components can produce peaks, each component is homologous, and its correction factor is the same. Calculate each group according to formula (3) The percentage of sugar in dry matter:

$$P_i = \frac{A_i}{\sum A_i} \times 100 \quad \cdots\cdots(3)$$

In the formula:

P,: the percentage of dry matter (mass fraction) of component i in the sample, %;

A,: the peak area of component i in the sample;

$\sum A_i$: The sum of the peak areas of all components in the sample.

The percentage of oligofructose in the sample is calculated according to formula (4):

$$FOS\% = GF_2 + GF_3 + GF_4 + GF_5 \quad \cdots\cdots(4)$$

In the formula:

FOS% : total fructooligosaccharide content (accounting for dry matter, mass fraction), %;

GF_2 , GF_3 , GF_4 , GF_5 :The percentage of sugar (containing fructose) (accounting for dry matter, mass fraction), %. of sucrose triose, sucrose tetraose, sucrose pentaose, and sucrose hexaose (accounting for dry matter, mass fraction)

The calculation result is kept to one decimal place.

6.5.4.4 Precision

The absolute difference between two independent determination results obtained under repeatability conditions should not exceed 5% of the average.

6.6 Conductivity Ash

According to the method specified in GB317.

6.7 Chroma

6.7.1 Principle

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

6.7.2 Apparatus

Spectrophotometer (wavelength 420 nm~850 nm).

6.7.3 Determination

Put the sample directly into a 1 cm cuvette, use distilled water as the blank zero point, and measure its absorbance at the wavelength of 420 nm and 720 nm respectively, the result is expressed to three decimal places.

6.7.4 Calculation

Calculate chromaticity according to formula (5):

$$X = A_{420} - A_{720} \qquad \cdots\cdots(5)$$

In the formula:

X: the chromaticity of the sample;

A_{420} : the absorbance of the sample at a wavelength of 420 nm;

A_{720} : The absorbance of the sample at a wavelength of 720 nm.

6.8 Light transmittance

6.8.1 Principle

When a beam of parallel monochromatic light passes through the solution, the absorbance of the solution is proportional to the concentration of the solution and the thickness of the liquid layer. The lower the absorbance of the solution, the greater the transmittance, and the clearer and transparent the solution.

6.8.2 Apparatus

Same as 6.7.2

6.8.3 Analysis steps

Put the sample directly into a 1 cm cuvette, use water as a blank zero point, measure the light transmittance at a wavelength of 720 nm, and show the result to one decimal place.

7 Inspection rules

7.1 The products shall be inspected batch by batch by the quality inspection department of the manufacturer in accordance with the provisions of this standard. The qualified products shall be accompanied by the quality certificate issued by the quality inspection department of the manufacturer before leaving the factory.

7.2 Produce with the same batch of materials. Products of the same specification and the same variety constitute a batch.

7.3 Sampling method.

7.3.1 The inspection of each batch of products shall take samples according to Table 2.

Table 2

Batch range (minimum outer packaging unit)	Number of samples taken (minimum packaging unit)	Packing number of each sample extraction unit (bottle, sample bag)
Less than 100	2	1
300	4	1
More than 500	6	1
The number of packaging units refers to the small packaging units in the large packaging.		

7.3.2 Tank truck loading products must be inspected for each truck.

7.3.3 For barreled and tanked products, samples must be taken from a place below 10cm from the

liquid level, and the sampler shall comply with food hygiene standards.

7.3.4 The crystals from tank trucks shall be drawn according to the principle of equal sampling of each sample, and the sampling volume of each batch shall not be less than 1 kg; the barreled products shall be drawn according to the principle of equal sampling of each sample, and the sampling volume of each batch shall not be less than 1 kg; Bottled products shall be sampled from each sample in an equal amount, and the total amount of sampling shall not be less than 600g.

7.3.5 Mix the samples taken, divide them into two clean, dry glass bottles and seal them. Paste the label, and indicate the product name, manufacturer's name and address, batch number, sampling date and location, and the name of the sampling person on the label. One bottle is sent to the laboratory for inspection, and the other bottle is sealed for future reference and kept until the expiry date. When microbiological testing is required, the sampler and glass sample should be sterilized in advance (the sample must not touch the bottle mouth).

7.4 Factory inspection

The factory inspection items are sensory, dry matter (solid matter), water content of powdered sugar, pH, total oligofructose content, paste color, paste light transmittance, and total number of colonies in microbial indicators.

7.5 Type inspection

All items specified in the requirements of this standard. In general, type inspection is carried out once every six months. Type inspection should also be carried out in the following situations:

a) When the original and auxiliary materials have changed greatly;

b) When changing key processes or equipment;

c) New trial-produced products or normal-produced products are resuming production after the suspension of production for 3 months;

d) When the results of the factory inspection and the last type inspection are significantly different;

e) When the national quality supervision and inspection agency requires random inspection according to relevant regulations.

7.6 Judgment rules

7.6.1 If the inspection results have sensory or 1 to 2 physical and chemical indicators that are unqualified, you can double sampling from the batch of products and re-inspect the unqualified items. As long as there is one unqualified item in the re-inspection result, the unqualified product.

7.6.2 If one of the microbiological indicators is unqualified, the batch of products shall be deemed unqualified.

8 Signs,Packaging, Transportation and storage

8.1 Logo

8.1.1 The packaging mark shall comply with the relevant regulations of GB 7718 or GB 16740.

8.1.2 The label of the package should indicate: product name, manufacturer name, factory address, registered trademark, specifications (including solid content and total oligofructose content), net content, production date, batch number, shelf life, and implementation standard number.

8.1.3 The packaging, storage and transportation diagrams on the outer packaging box shall be implemented in accordance with the provisions of GB/T 191.

8.2 Packaging

Packages and containers should be clean, hygienic, and free from damage.

8.3 Transportation and storage

8.3.1 Measures should be taken to prevent exposure and rain during transportation. Loading and unloading during transportation should comply with the requirements of the packaging, storage and transportation icons on the outer packaging.

8.3.2 The finished product should be stored in a dry, ventilated, clean warehouse without direct sunlight, and shipped out according to the principle of first in, first out.

Annex

(Normative annex)
Dry matter (solid matter) content and temperature conversion table (standard temperature: 20℃)

Table A.1 %

TEMP/℃	Dry matter (solid matter) content/%																	
	0	5	10	15	20	25	30	35	40	45	50	55	60	65	70	75	80	85
	Subtract from the measured																	
15	0.29	0.31	0.32	0.33	0.34	0.35	0.36	0.37	0.87	0.38	0.38	0.38	0.38	0.38	0.38	0.38	0.37	0.37
16	0.24	0.25	0.26	0.27	0.28	0.28	0.29	0.30	0.30	0.30	0.31	0.31	0.31	0.31	0.31	0.30	0.30	0.30
17	0.18	0.19	0.20	0.20	0.21	0.21	0.22	0.22	0.23	0.23	0.23	0.23	0.23	0.23	0.23	0.23	0.23	0.22
18	0.12	0.13	0.13	0.14	0.14	0.14	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15
19	0.06	0.06	0.07	0.07	0.07	0.07	0.07	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.07
	Add to the measured value																	
21	0.60	0.07	0.07	0.07	0.07	0.07	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.07
22	0.13	0.14	0.14	0.14	0.15	0.15	0.15	0.15	0.16	0.16	0.16	0.16	0.16	0.16	0.15	0.15	0.15	0.15
23	0.20	0.21	0.21	0.22	0.22	0.23	0.23	0.23	0.23	0.24	0.24	0.24	0.24	0.23	0.23	0.23	0.23	0.22
24	0.27	0.28	0.29	0.29	0.30	0.30	0.31	0.31	0.31	0.32	0.32	0.32	0.32	0.31	0.31	0.31	0.30	0.30
25	0.34	0.35	0.36	0.37	0.38	0.38	0.39	0.39	0.40	0.40	0.40	0.40	0.40	0.39	0.39	0.39	0.38	0.37
26	0.42	0.43	0.44	0.45	0.46	0.46	0.47	0.47	0.48	0.48	0.48	0.48	0.48	0.47	0.47	0.46	0.46	0.45
27	0.50	0.51	0.52	0.53	0.54	0.55	0.55	0.56	0.56	0.56	0.56	0.56	0.56	0.55	0.55	0.54	0.53	0.52
28	0.58	0.59	0.60	0.61	0.62	0.63	0.64	0.64	0.64	0.65	0.65	0.64	0.64	0.64	0.63	0.62	0.61	0.60
29	7.66	9.67	0.68	0.69	0.70	0.71	0.72	0.73	0.73	0.73	0.73	0.73	0.72	0.72	0.71	0.70	0.69	0.68
30	0.74	0.75	0.74	0.78	0.79	0.80	0.81	0.81	0.81	0.82	0.81	0.81	0.81	0.80	0.79	0.78	0.77	0.75
31	0.83	0.84	0.85	0.87	0.88	0.89	0.89	0.90	0.90	0.90	0.90	0.90	0.89	0.88	0.87	0.86	0.84	0.83
32	0.91	0.93	0.94	0.95	0.96	0.97	0.98	0.99	0.99	0.99	0.99	0.98	0.97	0.96	0.95	0.94	0.92	0.90
33	1.00	1.02	1.03	1.04	1.05	1.06	1.07	1.08	1.08	1.08	1.07	1.07	1.06	1.05	1.03	1.02	1.00	0.98
34	1.10	1.11	1.12	1.13	1.15	1.15	1.16	1.17	1.17	1.17	1.16	1.15	1.14	1.13	1.12	1.10	1.08	1.06
35	1.19	1.20	1.22	1.23	1.24	1.25	1.25	1.26	1.26	1.25	1.25	1.24	1.23	1.21	1.20	1.18	1.16	1.13
36	1.29	1.30	1.31	1.32	1.33	1.34	1.35	1.35	1.35	1.35	1.34	1.33	1.32	1.30	1.28	1.26	1.24	1.21
37	1.38	1.40	1.41	1.42	1.43	1.44	1.44	1.44	1.44	1.44	1.43	1.42	1.40	1.38	1.36	1.34	1.32	1.29
38	1.48	1.50	1.51	1.52	1.53	1.53	1.54	1.54	1.53	1.53	1.52	1.51	1.49	1.47	1.45	1.42	1.39	1.36
39	1.59	1.60	1.61	1.62	1.62	1.63	1.63	1.63	1.63	1.62	1.61	1.60	1.58	1.56	1.53	1.50	1.47	1.44
40	1.69	1.77	1.71	1.72	1.72	1.73	1.73	1.73	1.72	1.71	1.70	1.69	1.67	1.64	1.62	1.59	1.55	1.52