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Raw sugar

原 糖

(Codex Stan 212-1999, Codex standard for sugars, NEQ)

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Raw sugar

1 Scope

This Standard specifies requirements, test methods, inspection rules and marks, packaging, transportation, storage for raw sugar.

This Standard is applicable to cane sugar crystals with molasses made from sugarcane juice after purification, smelting, and honey separation.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 191, *Packaging and storage marks*

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

GB 7718, *National Food Safety Standard - Standard for nutrition labelling of prepackaged foods*

GB/T 10498, *Sugarcane*

GB 13104, *National Food Safety Standard - Sugars*

JJF 1070, *Rules of Metrological Testing for Net Quantity of Products in Prepackages with Fixed Content*

Measures for the Supervision and Administration of Quantitative Packaging Commodities Measurement (Order No. 75 of General Administration of Quality Supervision, Inspection and Quarantine)

3 Requirements

For the purposes of this document, the following terms and definitions apply.

3.1 Raw material requirements

4.2.1.1 Balance: Resolution is 0.1mg.

4.2.1.2 Volumetric flask: (100.00±0.02) mL.

4.2.1.3 Filtration equipment: Glass rod-less funnel, beaker, medium-speed quantitative filter paper.

4.2.1.4 Sugar tester: It shall have an international sugar content scale. According to the sugar content °Z scale, the accuracy of the automatic sugar tester is 0.05°Z. The accuracy of the visual inspection of the sugar meter is 0.1°Z.

NOTE: If using a sugar tester with the old sugar content °S scale, the reading °S needs to be multiplied by a factor of 0.99971 to convert to °Z.

4.2.1.5 Observation tube: Length is (200.00±0.02) mm.

4.2.2 Reagents

4.2.2.1 Basic lead acetate solution: Weigh 340g of basic lead acetate powder. Dissolve in about 1000mL of distilled water that has just been boiled. Adjust the brix to 54.3°Bx. The prepared solution shall prevent contact with carbon dioxide in the air.

4.2.2.2 Distilled water: Without optically active substances.

4.2.3 Determination steps

4.2.3.1 Calibration of sugar tester

The reading of the sugar tester shall be calibrated with a standard quartz tube.

For sugar tester without quartz wedge compensator, the temperature shall be measured when reading the optical rotation of the quartz tube, to the nearest of 0.2°C. If this temperature differs from 20°C by more than ±0.2°C, then use formula (1) to perform temperature correction of the optical rotation of the quartz tube. Then use the correction value to calibrate the reading of the sugar tester.

$$\alpha_t = \alpha_{20}[1 + 0.000\ 144 \times (t_1 - 20)] \qquad \cdots\cdots(1)$$

Where,

α_t - Optical rotation value of the quartz tube at t°C, in international sugar content (°Z);

α_{20} - Optical rotation value of the quartz tube at 20°C, in international sugar content (°Z);

P - Sugar content, %;

P_t - Observed sugar content of raw sugar sample, in international sugar content (°Z);

t_2 - Temperature of the sugar solution when measuring P_t, in Celsius (°C).

4.2.3.4 Allowable error

The difference between the two measured values shall not exceed 0.05% of the average value.

4.3 Determination of safety factor (SF)

4.3.1 Instruments, equipment

4.3.1.1 Electric heating thermostat: During the measurement, the temperature at (2.5±0.5) cm above the drying dish shall be maintained at (105±1)°C.

4.3.1.2 Desiccator with thermometer.

4.3.1.3 Weighing bottle: 6cm in diameter and 3cm in height.

4.3.1.4 Balance: Resolution is 0.1mg.

4.3.2 Determination steps

4.3.2.1 Drying

Preheat the drying oven to 105°C. Put the empty weighing bottle together with the open lid into the drying oven. Dry for at least 30min. Then put the lid on the weighing bottle and take it out of the drying oven. Put it in a desiccator to cool to room temperature. Weigh as soon as possible, to the nearest of 0.1mg.

Put (10.0±0.5)g of sample in the weighing bottle as soon as possible (the thickness of the sugar layer in the weighing bottle shall not exceed 1cm). Cover with the lid. Weigh, to the nearest of 0.1mg.

Remove the lid and put with the weighing bottle into the drying oven. Dry accurately at 105°C for 3h. Cover with the lid. Move to the desiccator to cool to room temperature. Weigh as soon as possible, to the nearest of 0.1mg.

4.3.2.2 Calculation and result presentation

Loss on drying D is calculated according to formula (4). The value is expressed in%. The calculation result retains two significant digits.

1.300~1.700. The minimum index value of refractive index: 0.0005. The sucrose mass fraction brix (°Bx) is 0~95. The minimum graduation value: 0.2.

4.5.1.4 pH meter: The graduation value or minimum display value is 0.02.

4.5.1.5 Membrane filter: Use a microporous membrane with a pore size of 0.45μm.

4.5.2 Reagents

4.5.2.1 0.05mol/L sodium hydroxide solution.

4.5.2.2 0.05mol/L hydrochloric acid solution.

4.5.3 Determination steps

Weigh a certain amount of sugar sample. Use distilled water to dissolve (so that the sugar solution concentration is about 10°Bx). Adjust the pH of the sugar solution to 7.00±0.02 with sodium hydroxide or hydrochloric acid solution. Pour into the pre-laid microporous membrane filter. Filter under vacuum. Discard the first part of the filtrate. Collect not less than 50mL of remaining filtrate. Use Abbe refractometer to determine the refractive brix of the filtrate. Then use a cuvette to hold the filtrate. Use filtered distilled water as a reference standard for zero color value. On a spectrophotometer, use 420nm wavelength to determine its absorbance.

4.5.4 Calculation and result presentation

The color value C_v is calculated according to formula (7). The calculation result is kept as an integer.

$$C_v = \frac{A}{b \times c} \times 1\ 000 \qquad \cdots\cdots (7)$$

Where,

C_v - Color value, in international sugar color unit (IU);

A - Absorbance of the sample solution measured at 420nm wavelength;

b - Cuvette thickness, in centimeters (cm);

c - Concentration of sample solution (obtained from table 2 of refractive hammer corrected to 20°C), in grams per milliliter (g/mL).

4.6.1.1 Crucible glass filter: Pore size is 40μm~80μm.

4.6.1.2 Electric heating thermostat: 125°C~130°C.

4.6.1.3 Desiccator: With thermometer.

4.6.1.4 Balance: Resolution is 1mg.

4.6.2 Reagents

4.6.2.1 1% α-Naphthol in ethanol.

4.6.2.2 Concentrated sulfuric acid: Density is 1.84g/mL.

4.6.3 Determination steps

On the glass filter plate, spread a layer of about 5mm thick glass fiber that has been washed by dilute hydrochloric acid solution and rinsed by distilled water. The glass filter is filtered and cleaned with distilled water under reduced pressure. Then dry it to constant weight.

Weigh 250.0g of the sample in a 1000mL beaker (if it is mixed with packaging fiber, fluff and so on, it shall be removed and then weighed). Add about 700mL of distilled water. Stir until they are completely dissolved. Pour into the glass filter prepared above for decompression filtration. Use distilled water to fully wash the filter residue. Use alpha-naphthol ethanol solution to check until the washing solution contains no sugar. Dry the glass filter together with the filter residue at 125°C~130°C. Move to a desiccator and cool to room temperature. Weigh. Continue drying for about 30min. Cool and weigh until the difference between two successive weighings does not exceed 1mg.

Microsugar test method: Take 2mL of washing solution in a test tube. Add a few drops of 1% α-naphthol ethanol solution. Then slowly add 2mL of concentrated sulfuric acid along the wall of the tube. Sucrose reacts strongly with phenols in the presence of concentrated sulfuric acid. When a purple ring appears at the interface of water and acid, it indicates the presence of sucrose. If it is a yellow-green ring, it means that there is no sucrose.

4.6.4 Calculation and result presentation

The mass F of water-insoluble impurities per kilogram of raw sugar sample is calculated according to formula (8). The calculation result is kept as an integer.

$$F = (m_2 - m_1) \times 4\ 000 \qquad \cdots\cdots (8)$$

Where,

78mL of trichloroacetic acid (TCA) solution. Add water to the graduation of scale. Shake well. This solution shall be prepared fresh when needed.

4.7.2.6 Amylase: heat stable alpha-amylase.

4.7.2.7 Pickling diatomaceous earth: Add (50±5) g of diatomaceous earth to 1L of distilled water. After stirring well, add (50±5) mL of 1.19g/mL concentrated hydrochloric acid. Stir for 5min. Use Buchner funnel to filter. Use distilled water to rinse until the filtrate is not acidic (test with litmus paper). The washed diatomaceous earth shall be dried in an oven at 96°C~100°C for 6h. Store in a closed container.

4.7.3 Determination steps

4.7.3.1 Preparation of standard solution and drawing of standard curve

4.7.3.1.1 Determination of standard dextran moisture: Weigh about 2g of dextran (4.7.2.1) in a weighing bottle that has been dried to a constant weight (weigh to the nearest of 0.1mg). Put it in a drying oven and dry at 105°C for 3h. Take it out. Put it in the desiccator. Cool to room temperature. Weigh (to the nearest of 0.1mg).

4.7.3.1.2 1mg/mL dextran standard solution: According to the measured moisture content of glucan, quickly weigh out the undried glucan (4.7.2.1). Make it contain about 0.2000g of moisture-free glucan in a beaker. Add about 2mL of water to dissolve into a paste. Place for about 10min. Stir from time to time. Make the particles evenly hydrated. When there is a gel, add a small amount of water several times to about 25mL. There shall be no more gel. Transfer to a 200mL volumetric flask. Use water to wash to a volume of about 80mL. Put the volumetric flask in a boiling water bath for 30min. Take out. Use cold water to cool to room temperature. Add water to the graduation of scale. Shake well. This solution contains 1mg of dextran per ml (the actual concentration is calculated based on the weighing). This solution needs to be prepared when required. It cannot be stored overnight.

4.7.3.1.3 0.2mg/mL dextran standard solution: Pipette 20mL of 1mg/mL dextran standard solution (4.7.3.1.2) into a 100mL volumetric flask. Add water to dilute to the graduation of scale. Shake well.

4.7.3.1.4 Standard curve drawing: In four 25mL volumetric flasks that are added with 8.0mL of sucrose-TCA solution (4.7.2.5), respectively add 0.50mL, 1.00mL, 2.00mL, 4.00mL of 0.2mg/mL dextran standard solution (4.7.3.1.3). Then in another four 25mL volumetric flasks that are added with 8.0mL of sucrose-TCA solution (4.7.2.5), respectively add 1.20mL, 1.60mL, 2.00mL, 2.40mL of 1mg/mL dextran standard solution. Add distilled water to each flask to a total volume of 12.5mL. Add denatured anhydrous alcohol (DAA) (4.7.2.3) to the

Where,

T – Time interval, in minutes (min);

Q - Batch size of inspection lot, in tons (t);

G - Loading-unloading volume per hour, in tons (t);

n - Number of incremental samples to be taken, in pieces.

When taking the first incremental sample, it can be randomly determined in the first interval. But it does not start at the beginning of the first interval. Incremental samples that continue to be extracted in the future are extracted at calculated intervals. The extraction interval shall not be greater than the calculated interval. If the number of incremental samples to be extracted at regular intervals has been completed, but the loading, unloading, processing or weighing of raw sugar is still in progress, it shall continue to extract incremental samples at the original interval until the entire batch of raw sugar has been extracted.

If extracting incremental samples at the conveyor belt or conveyor belt drop, it needs to intercept the full cross-section of the raw sugar stream. If in the process of loading-unloading or stacking with grabs, forklifts or other tools, sampling shall be in the process of loading-unloading or stacking. Use sampling shovel or sampling skewer, on the newly exposed sugar level, to conduct uniform sampling. Sampling points shall be evenly distributed in each part of the whole batch of raw sugar, rather than its surface or part.

5.3.4.3 Stratified sampling

A batch of raw sugar can be sampled in several layers (not less than 3 layers) in the process of loading-unloading, processing, and stacking, for example, in a cabin. According to the mass of raw sugar contained in each layer, proportionally on the newly exposed surface, distribute points evenly. First scrape about 3cm thick on the surface of this point. Take an incremental sample.

The number of incremental samples of each layer shall be calculated according to formula (11).

$$n_1 = n \times \frac{Q_1}{Q} \qquad \cdots\cdots (11)$$

Where,

n_1 - The number of incremental samples that shall be extracted for each layer, in pieces;

d) Sampling location;

e) Sampling staff, producer;

f) Sampling and sample preparation date.

5.4 Sample inspection

5.4.1 Sample inspection requirements

The analysis samples are divided into 3 portions. Inspect according to the test method specified in this Standard. Use the average of the two closest measured values as the final test result of the inspection lot. Or if the difference between two measured values and an intermediate value is equal, then use this intermediate value as the final test result of the inspection lot. This result shall be regarded as the average quality of the batch of raw sugar.

5.4.2 Result determination

If the inspection results have nonconforming indicators, double samples shall be taken. Reinspect the nonconforming indicators. The re-inspection result shall be the final result of the inspection lot. If the result of the re-inspection is nonconforming, the inspection lot shall be deemed rejected.

5.4.3 Storage of inspected sample

The analysis samples shall be kept in a safe place for inspection. The storage period may be in accordance with relevant regulations but shall not be less than 3 months.

6 Marks, packaging, transportation, storage

6.1 Marks

The label of pre-packaged raw sugar shall meet the requirements of GB 7718. The packaging, storage and transportation signs shall meet the requirements of GB/T 191. Each batch of raw sugar delivery certificate shall have the following information:

a) Product name;

b) Net content (t);

c) Producer name and address (including postal code); imported raw sugar shall indicate the country of production;

d) Production date;

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