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GB 7658-2005

Replacing GB 7658-1987

Food additive - Sorbitol solution

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Foreword

Some indicators in Table 1 of this Standard are mandatory, and the rest are recommendatory.

This Standard modifies and adopts "Sorbitol solution" (issued in 1991) of Codex Alimentarius Commission (CAC) (hereinafter referred to as CAC Standard) (English version).

This Standard is redrafted based on CAC Standard.

Considering China's national conditions, this Standard makes some modifications when adopting CAC Standard. The main differences between this Standard and CAC Standard are as follows:

- Added pH value, total sugar amount, and relative density (3.2 of this Standard), so as to meet user's needs;
- Modified indicators of solid content, reducing sugar content, arsenic content, heavy metal content, chloride content, and sulfate content (3.2 of this Standard), so as to improve product quality;
- Added weighting method for determination of solid content, taking Karl Fischer method as arbitration law (4.4 of this Standard) for easy use;
- Determinations of arsenic and heavy metal content shall use China national standards of product testing methods (4.10 and 4.11 of this Standard);
- Determination of reducing sugar content uses permanganate titration to replace cuprous oxide gravimetric method (4.8 of this Standard), so as to improve the accuracy of test results.

This Standard replaces GB 7568-1987 *Food additive - Sorbitol solution*.

Compared with GB 7568-1987, the main changes of this Standard are as follows:

- Added solid content, lead content, chloride content and sulfate content and corresponding test methods (see 3.2, 4.4, 4.11, 4.13 and 4.14 of this edition);
- Modified reducing content indicator from " $\leq 0.63\%$ " to " $\leq 0.21\%$ ", total sugar content indicator from " $\leq 2.0\%$ " to " $\leq 8.0\%$ ", nickel content indicator from " $\leq 0.0003\%$ " to "2 mg/kg" (1.3 of 1987 edition, 3.2 of this edition);

- Determination of sorbitol content uses high performance liquid chromatography to replace iodimetry; determinations of reducing sugar content and total sugar content use potassium permanganate titration to replace cuprous oxide gravimetric method (2.1, 2.7 and 2.8 of 1987 edition, 4.5, 4.8 and 4.9 of this edition);
- Modified “all items are factory inspection items” to “all items are type inspection items of which solid content, sorbitol content, reducing sugar content, total sugar content, nickel content, pH value are factory inspection items” (see 5.1 of this edition).

Appendix A of this Standard is normative.

This Standard was proposed by China Petroleum and Chemical Industry Federation.

This Standard shall be under the jurisdiction of Subcommittee of Organic Chemicals of the National Technical Committee on Chemical Standards (SAC/TC 63 / SC 2), and National Institute for Nutrition and Health of Chinese Center for Disease Control and Prevention.

The drafting organizations of this Standard: Roquette Lianyungang Co., Ltd., and Lida (Liuzhou) Chemistry Co., Ltd.

Main drafters of this Standard: Zhuang Defeng, Lao Ming, Li Yan, and Wu Yanhua.

This Standard was issued in April, 1987 for the first time.

Food additive - Sorbitol solution

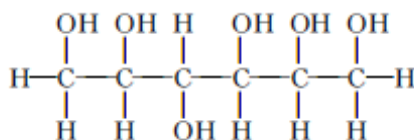
1 Scope

This Standard specifies requirements, test method, inspection rules as well as marks, package, transport and storage of food additive - sorbitol solution.

This Standard is applicable to food additive - sorbitol solution which takes glucose as raw material and is refined by hydrogenation in the presence of catalyst. This product shall be used as humectant and sweetener during food processing.

Molecular formula: $C_6H_{14}O_6$

Structural formula:



Relative molecular mass: 182.17 (according to international relative atomic mass in 2007)

2 Normative references

The following standards contain the provisions which, through reference in this Standard, constitute the provisions of this Standard. For dated references, the subsequent amendments (excluding corrections) or revisions do not apply to this Standard. However, the parties who enter into agreement based on this Standard are encouraged to investigate whether the latest versions of these documents are applicable. For undated reference documents, the latest versions apply to this Standard.

GB/T 601 Chemical Reagent - Preparations of Standard Volumetric Solutions

GB/T 602 Chemical Reagent - Preparations of Standard Solutions for Impurity (GB/T 602-2002, ISO 6353-1:1982, NEQ)

GB/T 603 Chemical Reagent - Preparations of Reagent Solution for Use in Test (GB/T 603-2002, ISO 6353-1:1982, NEQ)

Relative density (d_{20}^{20})	1.285~1.315
Reducing sugar (in glucose) mass fraction / % $\leq$	0.21
Total sugar (in glucose) mass fraction / % $\leq$	8.0
Arsenic (As) mass fraction / % $\leq$	0.0002
Lead (Pb) mass fraction / % $\leq$	0.0001
Heavy metal (in Pb) mass fraction / % $\leq$	0.0005
Chloride (in Cl) mass fraction / % $\leq$	0.001
Sulfate (in SO ₄) mass fraction / % $\leq$	0.005
Nickel (Ni) mass fraction / % $\leq$	0.0002
Residue on ignition mass fraction / % $\leq$	0.10
NOTE: Arsenic (As) mass fraction, lead (Pb) mass fraction and heavy metal (in Pb) mass fraction are mandatory.	

4 Test method

4.1 Warning

Some test procedures prescribed in test method may lead to dangerous situations. The operator shall take appropriate safety and health measures.

4.2 General provisions

Unless other requirements specified, reagents and water used in this Standard all refer to analytical-pure reagents and Grade 3 water stipulated in GB/T 6682-2008.

Unless other requirements specified, standard solution, impurity standard solution, preparations and products used for test shall be prepared based on the provisions of GB/T 601, GB/T 602 and GB/T 603.

4.3 Identification test

4.3.1 Reagent

4.3.1.1 Sulfuric acid;

4.3.1.2 Ferrous sulfate solution: 80 g/L;

4.3.1.3 Sodium hydroxide solution: 200 g/L;

sample solution through chromatographic column. Use refractive index detector to detect. Adopt external standard method to quantify. Calculate sorbitol content in sample.

4.5.2 Reagent

4.5.2.1 Water, GB/T 6682, Grade 2: filtered by 0.45 μm membrane filter, ultra pure and degasified;

4.5.2.2 Sorbitol standard sample: sorbitol mass fraction $\geq 98\%$;

4.5.2.3 Mannitol

4.5.3 Apparatus

4.5.3.1 High performance liquid chromatography (HPLC)

4.5.3.1.1 High-pressure pump: no pulse, it can maintain flow rate between 0.1 mL/min ~ 10.0 mL/min;

4.5.3.1.2 Dosing ring: 20 μL ;

4.5.3.1.3 Chromatographic column: see Table 2;

4.5.3.1.4 Refractive index detector: 500×10^{-6} Δ RIU or refractive index detector with corresponding sensitivity

4.5.3.1.5 Data processing system: analysis software with Millennium 32, or chromatography workstation with corresponding functions, or chromatography data processor.

4.5.3.2 Suction filtration system

Suction filtration system uses cellulose ester membrane filter of which aperture is 0.45 μm (used for preprocessing of mobile phase water).

4.5.3.3 Filtration system

Filtration system uses cellulose ester membrane filter of which aperture is 0.45 μm (used for preprocessing of sample).

4.5.3.4 Micro syringe

Special for HPLC, 100 μL .

4.5.4 Chromatography conditions

Recommended chromatography conditions are shown in Table 2. Typical HPLC

4.8 Determination of reducing sugar content

4.8.1 Method summary

Heat at a certain temperature, time and concentration. Reducing sugar in sample shall be oxidized by an excess of Fehling solution, react and produce cuprous oxide precipitate. Sulfate is reduced to ferrous sulfate by cuprous oxide. Use potassium permanganate standard solution to generate ferrous sulfate. Based on the consumption of potassium permanganate standard solution, check Potassium permanganate cuprous oxide - glucose conversion table, and glucose mass shall be obtained. By calculation, reducing sugar (in glucose) content shall be calculated.

4.8.2 Reagent

4.8.2.1 Fehling solution;

4.8.2.2 Ferric sulfate solution: 50 g/L. Weigh 50 g of ferric sulfate. Add 200 mL of water to dissolve. Gradually add 100 mL of sulfuric acid. After mixing and cooling, add water to dilute to 1000 mL;

4.8.2.3 Potassium permanganate standard solution: $c(1/5 \text{ KMnO}_4)=0.1 \text{ mol/L}$.

4.8.3 Apparatus

Sand core crucible: filter plate aperture $5 \mu\text{m} \sim 15 \mu\text{m}$ (G4).

4.8.4 Analysis procedures

4.8.4.1 Weigh 25 g ~ 50 g of laboratory sample (sample weighing amount is determined by amount of reducing sugar), accurate to 0.0002 g. Place it in a 250 mL Erlenmeyer flask filled with about 5 mL of water. Well mix it.

4.8.4.2 Add 40 mL of Fehling solution and some glass balls. Completely mix it. Heat it on electric furnace and make it boil in 4 min. Continue boiling it for 3 min. Rapidly cool it to room temperature. Immediately use sand core crucible to decompress and conduct suction filtration. Use warm water to repeatedly wash the beaker and precipitate, so as to make filtrate clear till filtrate is not alkaline. Abandon the filtrate and wash the suction bottle. Add 60 mL of ferric sulphate solution in three portions into sand core crucible, so as to make cuprous oxide precipitate completely dissolved. Conduct suction filtration. Use water to wash sand core crucible for several times. Collect the filtrate. Use potassium permanganate standard solution to titrate the filtrate till it is slight red. Record the volume V_0 of consumed potassium permanganate standard solution.

4.8.5 Result calculation

4.10 Determination of arsenic content

Conduct according to “arsenic stain method” stipulated in GB/T 5009.76. During determination, weigh 1.0 g of laboratory sample, accurate to 0.01 g. Add 10 mL of water to dissolve. Weigh 2.00 mL of arsenic (As) standard solution (equivalent to 0.002 mg of As) to prepare limited standard solution.

4.11 Determination of lead content

Conduct according to “limited test” stipulated in GB/T 5009.75. During determination, weigh 10.0 g of laboratory sample, accurate to 0.01 g. Add 10 mL of water to dissolve. Weigh 1.00 mL of lead standard solution (equivalent to 0.01 mg of Pb) to prepare lead limited standard solution.

4.12 Determination of heavy metal content

Conduct according to the provisions in GB/T 5009.74. During determination, weigh 2.0 g of laboratory sample, accurate to 0.01 g. Add 10 mL of water to dissolve. Weigh 1.00 mL of lead (Pb) standard solution (equivalent to 0.01 mg of Pb) to prepare limited standard solution.

4.13 Determination of chloride content

Conduct according to the provisions in GB/T 9729. During determination, weigh 10.0 g of laboratory sample, accurate to 0.01 g. Weigh 1.00 mL of chloride (Cl) standard solution (equivalent to 0.1 mg of Cl) to prepare limited standard solution. Sample turbidity shall not be greater than the standard one.

4.14 Determination of sulfate

Conduct according to the provisions in GB/T 9728. During determination, weigh 2.0 g of laboratory sample, accurate to 0.01 g. Weigh 1.00 mL of sulfate (SO₄) standard solution (equivalent to 0.1 mg of SO₄) to prepare limited standard solution. Sample turbidity shall not be greater than the standard one.

4.15 Determination of nickel content

4.15.1 Reagent

4.15.1.1 Ammonia;

4.15.1.2 Dimethyl glyoxal oxime (dimethylglyoxime) absolute ethanol: 1→100;

4.15.1.3 Nickel (Ni) standard solution: 0.01 mg/mL.

4.15.2 Analysis procedures

Weigh 10.0 g of laboratory sample, accurate to 0.01 g. Place it in a 50 mL

- a) Renewal of key product techniques;
- b) Change on main raw material;
- c) Production resumed after it is stopped;
- d) Great differences between factory inspection results and previous type inspection;
- e) Contract stipulation.

5.1.2 In Table 1 Technical requirements of this Standard, solid mass fraction, sorbitol mass fraction, reducing sugar mass fraction, total sugar mass fraction, nickel mass fraction, and pH value are exit-factory inspection items.

5.2 Food additive - sorbitol solution shall be accepted in batch. Each inspection batch shall be consisted of a production batch, or several product batches which comply with the following requirements.

5.2.1 These production batches are produced by basically same raw material, in basically same process and under basically same equipment conditions.

5.2.2 The time for several production batches to form an inspection batch shall not exceed one week.

5.3 When inspecting the lot of bottle-packaging of food additive - sorbitol solution, the sampling unit shall be determined according to 7.6 of GB/T 6678-2003. During tanker-packaging, conduct sampling from each tanker. When bottle-packaging, use sampler to conduct sampling from upper, middle and lower layers in proportion of 1:3:1. When tanker-packaging, user sampler to conduct sampling from tanker top inlet, following upper, middle and lower parts in proportion of 1:3:1. If it is unable to conduct sampling at the top, it may conduct sampling at discharge port of tanker. Sampling process shall also comply with the provisions of GB/T 6680. The taken sample amount shall not be less than 500 mL. Well mix it and respectively put into two clean and dry grinding mouth glass jars. Label it and indicate the product name, batch number, date of manufacture, sampling date and sampling staff's name. One copy is for inspection as laboratory sample and the other is for future reference as a sample.

5.4 Food additive - sorbitol solution shall be inspected by quality supervision and inspection department of the manufacturer according to the provisions of this Standard. The manufacturer shall ensure that each batch of products comply with requirements of this Standard. If one indicator in inspection results fails to meet requirements of this Standard, for bottle-packaging products, it shall re-conduct sampling from packaging unit of twice amounts; for canned

products, it shall re-conduct multi-sampling to inspect. Even one indicator in re-inspection results fails to meet requirements of this Standard, the whole batch of products shall be regarded as unqualified.

6 Marks, package, transport and storage

6.1 Marks

6.1.1 There shall be firm and distinct marks on packaging container, including product name, manufacturer's name, address, trademark, words of "food additive", this Standard number, hygienic license number, production batch or production date, and net mass.

6.1.2 Each batch of food additive - sorbitol solution shall be attached with quality certificate, including: manufacturer's name, address, product's name, trademark, words of "food additive", production batch number or production date, net mass, shelf life, certificate that product complies with this Standard requirements and this Standard number.

6.2 Package

Food additive - sorbitol solution uses metal drum and plastic drum of which inner coating is epoxy resin for package. It shall also use stainless steel tank packaging for food additive, or according to user requirements.

6.3 Transport and storage

Food additive - sorbitol solution shall be stored in a dry and ventilated warehouse. Pay attention to light unloading during transport, avoiding drum rupture. During transport and storage, it must not put it together, transport or stack it together with harmful toxic substances or other pollutants.

6.4 Shelf life

Under conditions that comply with provisions on package, transport and storage of this Standard, the shelf life of food additive - sorbitol solution shall be 12 months since the date of production. Conduct re-inspection when it is overdue and it may be used when inspection results meet requirements of this Standard.

Appendix A

(Normative)

Potassium permanganate cuprous oxide - reducing sugar (in glucose) conversion

Table A.1 lists out potassium permanganate cuprous oxide - glucose conversion.

Table A.1 Potassium permanganate cuprous oxide - glucose conversion

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