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GB 1987-2007

Replacing GB 1987-1986

Food Additive – Citric Acid

食品添加剂　柠檬酸

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Foreword

Physiochemical indices in this standard are not equivalent to those specified in "*Compendium of Food Additive Specifications, Volume 1, Joint FAO/WHO Expert Committee on Food Additive (JBCFA)".* They refer to those specified in "*British Pharmacopeia"* BP-2003 and "*United Stated Pharmacopeia"* USP-27. In which, oxalate and sulfated ash indices are stricter than those specified in BP-2003; and arsenic indices are stricter than those specified in USP-27.

Test methods in this standard refer to those specified in BP-2003, USP-27 and "*Pharmacopoeia of the People's Republic of China" (2000 edition)*.

This standard replaces GB 1987-1986 "*Food Additive - Citric Acid*".

Compared with GB 1987-1986, the main changes of this standard are as follows:

— ADD the physiochemical indices of anhydrous citric acid;

— ADJUST the citric acid monohydrate content range;

— DELETE the barium salt indices;

— ADD the indices for moisture content, light transmittance and water insoluble;

— Test methods are increased or decreased and modified correspondingly with the change of indices.

This standard was proposed by China National Light Industry Council.

This standard shall be under the jurisdiction of Food Additives of the National Standardization Technical Committee.

This standard is drafted by China National Research Institute of Food & Fermentation Industries.

Chief drafting staffs of this standard: Zhang Wei, Guo Xinguang, and Kang Yongpu.

The previous editions replaced by this standard are as follows:

— GB 1987-1980, GB 1987-1986.

Food Additive – Citric Acid

1 Scope

This standard specifies classification, requirements, analysis methods, inspection rules, and marking, packaging, transportation, and storage of citric acid for food processing.

This standard is applicable to citric acid products made of starchy materials or sugar-containing materials, and such citric acid is mainly used as sour agent, anti-oxidation synergist and flavouring agent for food processing.

2 Normative References

The following standards contain provisions which, through reference in this standard, constitute provisions of this standard. For dated references, subsequent amendments (excluding corrigendum) or revisions of these publications do not apply. However, all parties who have entered into an agreement based on this standard are encouraged to investigate whether the most recent editions of these standards apply. For undated references, the latest editions of the normative documents referred to apply.

GB/T 191	Packaging - Pictorial Marking for Handling of Goods (GB/T 191-2000, eqv ISO 780: 1997)
GB/T 601	Chemical Reagent - Preparations of Standard Volumetric 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 603-2002, ISO 6353-1: 1982, NEQ)
GB/T 606	Chemical Reagent - General Method for the Determination of Water - Karl Fischer Method (GB/T 606-2003, ISO 6353-1: 1982, NEQ)
GB/T 5009.11-2003	Determination of Total Arsenic and Abio-arsenic in Food
GB/T 5009.12	Determination of Lead in Foods
GB/T 6682	Water for Analytical Laboratory Use - Specification and Test

6.6.2.5 Hydrogen peroxide solution (3%): pipette 10mL of 30% hydrogen peroxide and dilute with water to 100mL.

6.6.2.6 Sodium hydroxide solution (300g/L): weigh 30g of sodium hydroxides, dissolve with water and dilute with water to 100mL.

6.6.2.7 Starch indicating solution (10g/L): prepare according to GB/T 603.

6.6.2.8 Ferric chloride.

6.6.2.9 Cobalt chloride.

6.6.2.10 Yellow stock solution

Weigh 46g of ferric trichloride (6.6.2.8); dissolve in about 900mL of hydrochloric acid solution (6.6.2.1); dilute with such hydrochloric acid solution to 1000mL. In calibration, adjust the yellow stock solution with hydrochloric acid solution (6.6.2.1), so as to make that 1mL of such yellow stock solution contains 46mg of $FeCl_3 \cdot 6H_2O$. Solution shall be protected from light and calibrated for immediate use.

Calibration: pipette 10mL of the newly-prepared ferric trichloride solution; add 15mL of water, 4g of potassium iodide (6.6.2.2), and 5mL of hydrochloric acid solution (6.6.2.1); plug the bottle cap immediately and stand still from light for 15min; add 100mL of water; titrate the iodine precipitated out with standard volumetric solution of sodium thiosulfate (6.6.2.4) until to pale yellow; add 0.5mL of starch indicating solution (6.6.2.7); titrate continuously until to endpoint.

Note: 1mL of 0.1mol/L standard volumetric solution of sodium thiosulfate is equivalent to 27.03mg of $FeCl_3 \cdot 6H_2O$.

6.6.2.11 Red stock solution

Weigh 60g of cobalt chloride (6.6.2.9); dissolve in about 900mL of hydrochloric acid solution (6.6.2.1); dilute with such hydrochloric acid solution to 1000mL. In calibration, adjust the red stock solution with hydrochloric acid solution (6.6.2.1), so as to make that 1mL of such red stock solution contains 59.5mg of $CoCl_2 \cdot 6H_2O$. Solution shall be protected from light and calibrated for immediate use.

Calibration: pipette 5.0mL of the newly-prepared cobalt chloride solution; add 5mL of hydrogen peroxide solution (6.6.2.5) and 10mL of sodium hydroxide solution (6.6.2.6); boil slowly for 10min and cool down. Add 2g of potassium iodide (6.6.2.2) additionally, and 60mL of sulfuric acid solution (6.6.2.3); plug the bottle cap immediately. Shake gently to dissolve precipitates; titrate the iodine precipitated out with standard volumetric solution of sodium thiosulfate (6.6.2.4) until to be pale yellow; add 0.5mL of starch indicating solution (6.6.2.7); titrate continuously until the solution appears in pink.

Take two 50mL colorimetric tubes with stopper; add 1 mL of barium chloride solution (6.9.2.2) respectively; add 1mL of sulfate standard solution; shake and stand still for 1min. Pipette 15mL of sample solution into one of the colorimetric tube; pipette 10mL of sulfate standard solution and 5 mL of water (standard tube) into the other colorimetric tube; then respectively add 1 mL of hydrochloric acid solution (6.9.2.1) and 0.5 mL of acetic acid solution (6.9.2.4); shake them up; after 5min, the milk scale of sample tube shall not be darker than that of standard tube.

For anhydrous citric acid. It shall use sulfate standard solution II (6.9.2.6): for citric acid monohydrate, it shall use sulfate standard solution III (6.9.2.7).

6.10 Oxalate

6.10.1 Apparatuses

6.10.1.1 Colorimetric tube with stopper: 25mL.

6.10.1.2 Test tube: 15mm×180mm.

6.10.2 Reagents and solutions

6.10.2.1 Hydrochloric acid.

6.10.2.2 Phenylhydrazine hydrochloride solution (10g/L): prepare according to GB/T 603.

6.10.2.3 Potassium ferricyanide solution (50g/L): weigh 5g of potassium ferrocyanide; dissolve it with water to 100mL.

6.10.2.4 Zinc granule.

6.10.2.5 Oxalate standard solution I (0.25 g/L): weigh 0.175 g of oxalate ($C_2H_2O_4 \cdot 2H_2O$); dissolve it with water to 500 mL.

6.10.2.6 Oxalate standard solution II (0.01g/L): pipette 4 mL of oxalate standard solution I (6.10.2.5); dilute with water to 100 mL.

6.10.3 Procedures

Weigh 0.4 g of sample (accurate to 0.01g) to a test tube; add 4ml of water, 3ml of hydrochloric acid, and 1g of zinc granule; boil for 1min and place for 2min. Transfer it to a test tube that has contained 0.25mL of phenylhydrazine hydrochloride solution (6.10.2.2); heat to boil; rapidly cool down; pour it to a 25 mL colorimetric tube with stopper; add 0.25mL of isochoric hydrochloric acid and 0.25mL of potassium ferricyanide solution (6.10.2.3). Shake it and place for 30min. Make a visual colorimetric determination with standard tube prepared by the following methods; the pink seen from the sample tube shall not be darker than that seen from the standard tube.

requirements of this standard.

7.4.2 Ex-factory inspection items: content, light transmittance, water content, readily carbonizable substance, chloride, sulfate, oxalate, calcium salt, ferrum, and water insoluble.

7.5 Type inspection

7.5.1 Type inspection items: besides the ex-factory inspection items, the type inspection items shall also cover identification test, sulfuric ash, arsenic salt, and heavy metal.

7.5.2 Generally, type inspection is carried out every three months. However, it shall also be conducted in 100% in case of any of the following conditions:

— During regular production, the inspection shall be carried out once annually;

— When there is significant change in raw materials, composition or process during normal production that may influence the product quality;

— When resumed after long shutdown;

— When the ex-factory inspection result has significant differences from the usual records;

— Where required by the national quality supervision department.

7.6 Judgment rules

7.6.1 When one inspection item among the inspection results is unqualified, the samples of double-quantity shall be collected from the same batch of products for re-inspection. The whole batch of products will be judged as unqualified if there is still one item is unqualified within the re-inspection results.

7.6.2 If the re-inspection result fails to comply with "high-grade" or "first-grade" physiochemical indices, but it meets the requirements of lower grade, then it is judged according to the lower grade.

7.6.3 When dispute occurs between the supplier and the purchaser regarding the product quality, it may be re-inspected by an arbitration organization, as agreed by both parties according to this standard.

8 Marking, Packaging, Transportation and Storage

8.1 Marking

The product shall be marked with labels on the external package, including product

name, manufacturer, address of manufacturer, net content, production date, batch number and standard number.

Marking on external package shall meet the requirements of QB/T 191.

8.2 Packaging

8.2.1 All inner packaging material shall meet the food package material sanitation requirements.

8.2.2 Packing requirements: the inner package shall be sealed tightly and free from air permeability; the outer package shall be free from pollution.

8.3 Storage and transportation

8.3.1 The products shall be handled with care and protected from pollution, rain and solar during transportation.

8.3.2 The means of transportation shall be clean, non-toxic and pollution-free. The products shall not be transported by filled and mixed with toxic, harmful or corrosive articles.

8.3.3 Products shall be stored in a shady, dry, ventilated and non-polluted place but not in open air. They are better to be stored at 30℃ and 50% relative humidity.

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