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**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

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Determination of boric acid in foods

食品中硼酸的测定

(English Translation)

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Foreword

Annex A and annex B of this Standard are informative.

This Standard was proposed by and shall be under the jurisdiction of National Food Safety Emergency Standardization Working Group.

Drafting organization of method I in this Standard. Shanghai Institute of Quality Inspection and Technical Research.

Drafting organization of method II in this Standard. Product Quality Supervision and Inspection Station of Haidian District, Beijing (National Food Quality Supervision and Inspection Center).

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Determination of Boric Acid in Foods

1 Scope

This Standard specifies the determination method of boric acid in foods.

This Standard applies to the determination of boric acid in aquatic products, meat

products (e.g. meat balls, fish balls), beans, cooked wheaten food, dried bean curd sticks, traditional Chinese rice-pudding, cakes, soy sauce, and other foods.

This Standard's detection limit. 2.50 mg/kg for method I ethohexadiol-trichloromethane extraction turmeric colorimetry; 1.00 mg/kg for method II inductively coupled plasma atomic emission spectrometry (ICP-AES); 0.20 mg/kg for inductively coupled plasma mass spectrometry (ICP-MS) method.

2 Normative references

The articles contained in the following documents have become part of this document when they are quoted herein. For the dated documents so quoted, all subsequent modifications (Including all corrections) or revisions made thereafter do not apply to this Standard. However, the parties who reach an agreement according to this Standard are encouraged to study whether the latest versions of these documents may be used. For the undated documents so quoted, the latest versions (including all modification sheets) apply to this document.

GB/T 6682 Water for laboratory use; Specifications (GB/T 6682-1992, neq ISO 3696. 1987)

Method I Ethohexadiol-trichloromethane Extraction

Turmeric Colorimetry

3 Principles

Gather and extract boric acid in samples through ethohexadiol-trichloromethane solution; remove the impact of coexisting salts; use protonated turmeric generated from the mixture of concentrated sulfuric acid and turmeric to react with boric acid to generate red products. Shade of solution color is in direct proportion to the content of boric acid in sample, which may be determined through colorimetry. In acidic conditions, borax exists in the form of

5.5 150 mL plastic beaker.

5.6 25 mL or 50 mL lidded plastic test tube.

6.1 Preparation of standard curve

Accurately measure 0.00, 1.00, 2.00, 3.00, 4.00 and 5.00 mL of standard solutions; place into 25 mL plastic tubes; add water to 5 mL respectively. Add 1 mL of sulfuric acid (1+1) solution; oscillate to mix; then add 5.00 mL of EHD-CHCl₃ solution; put the cover; put into vortex oscillator to operate for about 2 min; stand still for layering; draw under layer's EHD-CHCl₃ solution; filter through Ø7cm dried fast filter paper.

Respectively take 1.00 mL of filtrate; place into 50 mL plastic tube; successively add 1.0 mL of turmeric-glacial acetic acid solution and 5 mL of concentrated sulfuric acid; oscillate uniformly; stand still for 30 min; add 25 mL of anhydrous alcohol; after standing still for 10 min, measure absorbance at 550nm with 1 cm cuvette. Take standard series boric acid amount (µg) as abscissa, and absorbance as ordinate; draw the standard curve.

6.2.1 Sample treatment

Solid samples. Weigh 2.00 g~10.00 g (accurate to 0.01 g) of samples; use high-speed masher to mash; add 40 mL~60 mL of water and mix well; slowly add 2 mL of concentrated sulfuric acid drop-by-drop; apply ultrasound for 10 min to promote the dissolution and mixing. Add 5 mL of zinc acetate solution and 5 mL of potassium ferrocyanide solution; add water to a fixed volume 100 mL; use as sample solution after filtration. Take 1.00 mL~3.00 mL of sample solution according to the content of sample; place into a 25 mL plastic tube; add water to 5 mL. Add 1 mL of sulfuric acid (1 + 1) solution; oscillate to mix well. Then add 5.00 mL of EHD-CHCl₃ solution; put the cover; oscillator operates for 2 min; stand still for layering; draw under layer's EHD-CHCl₃ solution; filter through Ø7cm dried fast filter paper.

Liquid samples. Weigh 2.00 g~10.00 g of samples (accurate to 0.01 g); add water to a constant volume 100 mL as sample solution. For liquid samples with high protein or fat content, 5 mL of zinc acetate solution may be added to the samples; add water to a constant volume 100 mL and use it as sample solution. Take 1.00 mL~3.00 mL of sample solution according to sample content; place into a 25 mL plastic tube; add water to 5 mL; add 1 mL of sulfuric acid (1+1) solution; oscillate to mix well. Then add 5.00 mL of

EHD-CHCl₃ solution; put the cover; vertex oscillator operates for about 2 min; stand still for layering; draw under layer's EHD-CHCl₃ solution; filter through Ø7 cm dried fast filter volumetric flask; use 5% nitric acid solution to dilute and fix to constant volume; concentration of this standard stock solution is equal to 10µg of boron per milliliter. Prepared solution shall be timely transferred to a plastic bottle and be stored in refrigerator at 0°C~4°C for 3 months.

9.10 Scandium standard working solution. 1.00 mg/L.

Draw 1.00 mL of scandium standard working solution and place into a 100 mL plastic volumetric flask; use 5% nitric acid solution and fix to constant volume; each milliliter in volumetric flask is equivalent to 1.00µg of scandium.

Solution prepared shall be timely transferred to a plastic bottle and be stored in refrigerator at 0°C~4°C for 3 months.

20.0 mL of boron standard working solutions (9.9); place into 100 mL volumetric flasks;

then accurately add 1.00 mL of yttrium element standard stock solution (9.7); use 5% nitric acid solution to fix volume to scale; concentrations of solutions in volumetric flasks are equivalent to 0.00, 0.05, 0.30, 1.00 and 2.00 µg of boron per milliliter. Solution prepared shall be transferred to a plastic bottle timely.

Note: Yttrium element standard stock solution is not added to the standard solution tested through ICP-MS.

When ICP-MS is adopted for determination, adopt yttrium standard working solution (9.10) online internal standard adding.

10.5 Inductively coupled plasma atomic emission spectrometer (ICP-AES) and inductively

coupled plasma mass spectrometer (ICP-MS). All containers used have been soaked overnight with 20% nitric acid.