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

GB/T 21703-2008

Replacing GB/T 21703-2008

**National food safety standard -- Determination of benzoic acid
and sorbic acid in milk and milk products**

乳与乳制品中苯甲酸和山梨酸的测定

(English Translation)

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Table of Contents

Foreword
1 Scope
2 Normative references
4 Reagents and materials
4.8 Standard solution
5 Apparatus
6 Step, Analysis
6.1 Sample Preparation
6.2 Extraction and purification
6.3 Reference conditions chromatography
6.4 Determination
7 Expression analysis
8 Precision
9 Other

Foreword

This revised standard adopts the International Dairy Federation standard IDF 139. 1987 Milk, dried milk, yogurt and other fermented milks-Determination of benzoic and sorbic acid.

Appendix A of this standard is an informative annex.

This standard replaces the standards previously issued as follows.

--GB/T 21703-2008.

National Food Safety Standard Milk and milk products Determination of benzoic acid and sorbic acid

1 Scope

This standard specifies the method for determination of milk and dairy products benzoic acid and sorbic acid content.

This standard applies to milk and dairy products Determination of benzoic acid and sorbic acid content.

2 Normative references

The standard file referenced in the application of this standard is essential. For dated references, only the edition date of the note Apply to this standard. For undated references, the latest edition (including any amendments) applies to this standard.

Principle 3

Removal of fat and protein in the sample, diluted with methanol, filtered by reverse-phase liquid chromatography separation and determination.

4 Reagents and materials

Unless otherwise specified, the reagents used in this method are analytically pure water GB/T 6682 provided a water use.

4.1

methanol (CH_3OH). chromatography.

4.2

potassium ferrocyanide solution (92 g/L). Weigh potassium ferrocyanide $[\text{K}_4\text{Fe}(\text{CN})_6 \cdot 3\text{H}_2\text{O}]$ 106 g, was dissolved in 1000 mL of water capacity Flask, dilute to the mark and mix.

4.3

zinc acetate solution (183 g/L). Weigh zinc acetate $[\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}]$ 219 g, was added 32 mL of acetic acid was dissolved in water 1000 mL volumetric flask, dilute to the mark and mix.

4.4

phosphate buffer (pH = 6.7). Weigh 2.5 g of potassium dihydrogen phosphate (KH_2PO_4) and 2.5 g potassium phosphate dibasic ($\text{K}_2\text{HPO}_4 \cdot 3\text{H}_2\text{O}$) in 1000 mL volumetric flask, dilute to the mark with water and mix, filtered through a membrane filter (4.9) alternate.

4.5

sodium hydroxide solution (0.1 mol/L). Weigh 4 g of sodium hydroxide (NaOH), was dissolved in 1000 mL volumetric flask with water, the volume to the mark and mix.

4.6

sulfuric acid solution (0.5 mol/L). Pipette 30 mL of concentrated sulfuric acid (H_2SO_4) to 500 mL of water was added slowly with stirring, cooled After cooling to room temperature, transferred to a 1000 mL volumetric flask, dilute to the mark and mix.

Aqueous methanol 4.7. volume fraction of 50%.

4.8 Standard solution

4.8.1

benzoic acid and sorbic acid standard stock solution. Each ml of benzoic acid, sorbic 500 µg.

Weigh accurately benzoic acid, sorbic acid standard each 50.0 mg, were placed in 100 mL volumetric flask, dissolved in methanol (4.1) and dilute Explanation to the mark. Shake, stored in a refrigerator, valid for two months.

Working standard solutions 4.8.2 benzoic acid and sorbic acid. Each milliliter contains benzoic acid, sorbic acid, each 10 µg.

Pipette stock standard solution of benzoic acid and sorbic acid (4.8.1) each 5 mL, to 250 mL volumetric flask with aqueous methanol (4.7) After the volume up to the mark and mix. Stored in a refrigerator, valid for 5 d.

4.9 filter. 0.45 µm.

5 Apparatus

5.1

high performance liquid chromatography with UV detection.

5.2

Balance. a sense of the amount of 0.1 mg, 0.01 g.

6.1.1 Liquid Sample

Stored in a refrigerator for milk and dairy products should be removed before the test in advance and up to room temperature, and weighing 20 g (accurate to 0.01g) sample was 100 mL volumetric flask.

6.1.2 Solid samples

Weighing 3 g (accurate to 0.01g) sample was 100 mL volumetric flask, add 10 mL of water, with a glass rod and stir until completely dissolved.

6.2 Extraction and purification

To the flask containing the sample (6.1) was added 25 mL of sodium hydroxide solution (4.5), mixed and placed in an ultrasonic bath water or 70 °C Bath treatment 15 min. After cooling, a solution of sulfuric acid (4.6) and the pH was adjusted to 8 (pH paper or pH meter may be), followed by addition of 2 mL Asia Iron potassium cyanide solution (4.2) and 2 mL of zinc acetate solution (4.3). Vigorously shaken, allowed to stand for 15 min, cooled

to room temperature after mixing, then A Alcohol (4.1) constant volume, standing 15 min, the supernatant through a membrane filter (4.9) and filtered. The filtrate was collected as a sample solution for high performance liquid chromatography Spectrometer (5.1) measured.

6.3 Reference conditions chromatography

Column. C18, 250 mm x 4.6mm, 5 μ m.

Mobile phase. Methanol (4.1) - phosphate buffer solution (4.4) + 1 = 9.

Flow rate. 1.2 mL/min.

Detection wavelength. 227 nm.

Column temperature. room temperature.

Injection volume. 10 μ L.

6.4 Determination

Imbibe 10 μ L sample solution (6.2) and benzoic acid and sorbic acid working standard solutions (4.8.2) of not less than 2 parts each, in order to Chromatographic peak area. Under the above chromatographic conditions, the peak order of benzoic acid, sorbic acid, liquid chromatogram of the standard solution See attached Appendix A in Figure A.1.

7 Expression analysis

Sample benzoic acid, sorbic acid content in accordance with the formula (1) is calculated.

VcA

Where.

X - a sample of benzoic acid, sorbic acid content in milligrams per kilogram (mg/kg);

A-- sample solution of benzoic acid, sorbic acid peak area;

As - standard solution of benzoic acid, sorbic acid peak area;

cs - the concentration of standard solution, in micrograms per milliliter (μ g/mL);

V - the final volume of the sample, in milliliters (mL);

m - sample mass, in grams (g).

The arithmetic mean of two under the same condition of independent determination results indicated that the results to three significant figures.