



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

GB 5009.279-2016

**National food safety standard - Determination of Xylitol,
Sorbitol, Maltitol and Erythritol in Foods**

**食品安全国家标准 食品中木糖醇、山梨醇、麦芽糖醇、赤藓糖醇的测定
(English Translation)**

Issued on: December 23, 2016

Implemented on: June 23, 2017

Issued by: National Health and Family Planning Commission of the PRC

Table of Contents

Foreword...	3
1 Application Scope...	4
2 Principle...	4
3 Reagents and Materials...	4
4 Instruments and Apparatuses...	5
5 Analysis Steps...	6
6 Description of the Analysis Result...	8
7 Precision...	9
8 Others...	9
9 Principle...	9
10 Reagents and Materials...	9
11 Instruments and Apparatuses...	10
12 Analysis Steps...	11
13 Description of the Analysis Result...	13
14 Precision...	13
15 Others...	13

1 Application Scope

This Standard specifies the method to use high performance liquid chromatography - differential refractive index detection and evaporative light scattering detection for the test of xylitol, sorbitol, maltitol and erythritol in chewing gum, biscuit, pastry, bread and beverage.

This Standard applies to the determination of the content of xylitol, sorbitol, maltitol and erythritol in chewing gum, biscuit, pastry, bread and beverage.

Method 1 -- High Performance Liquid Chromatography -

Differential Refractive Index Detection.

2 Principle

After the protein is precipitated, filter the sample; put the supernatant into to the high performance liquid chromatography, use amino chromatographic column or cation exchange chromatographic column to separate; use differential refractive index detector to test; use external standard method to fix-volume.

3 Reagents and Materials

Unless otherwise specified, all the reagents in this method are analytical reagents, the water is grade-1 water specified by GB/T 6682.

3.1 Reagents

3.1.1

Acetonitrile (CH₃CH₃)

Chromatographic purity.

3.1.2

Trichloroacetic acid (CCl₃COOH).

3.1.3

Anhydrous sodium carbonate (Na₂CO₃).

3.2 Preparation of reagents

4.6 Ultrasonic cleaning machine: with a working frequency of 40 kHz and the power of 500 W.

5.1.1 Chewing gum

Take at least 20 g of the chewing gum sample; use a blade to cut into small pieces; put into a closed container to mix. Accurately weigh 2 g of chopped sample; place the sample in a 50 mL centrifuge tube; add 40 mL of water; mix and heat in a water bath at 80°C for 20 min; mix and shake every 6 min; take out and centrifuge at 9 000 r/min for 10 min. Take 8 mL of supernatant in a 10 mL volumetric flask; add water to fix- volume; shake well; filter through a 0.22 μm filter membrane; test on the machine.

5.1.2 Beverage

For non-protein beverages, take at least 200 mL of sample; mix well; place in a closed container. Take 10 mL of beverage in a 50 mL volumetric flask; add water to fix-volume to 50 mL; shake well; filter through a 0.22 μm filter membrane; test on the machine.

For protein beverages, take at least 200 mL of sample; place in a closed container to mix well. Weigh 5 g of sample; place in a 50 mL volumetric flask; add 35 mL of water; shake well; then sonicate for 30 min; shake it every 6 min; take out and centrifuge at 9 000 r/min for 10 min.

Precipitate protein: add 5 mL of trichloroacetic acid solution (100 g/L) into the supernatant;

shake it; then place it at room temperature for 30 min; centrifuge at 9 500 r/min for 10 min. Take 8 mL of the supernatant in a 10 mL volumetric flask; add water to fix-volume. After shaking, take 850 μ L of filtrate; add 150 μ L of sodium carbonate solution (21.2 g/L); shake and neutralize; or take 10 mL of supernatant; add 20 mL of acetonitrile; shake well and place it at room temperature for 30 min; centrifuge at 9 500 r/min for 10 min; fix-volume the supernatant to 50 mL; shake well; filter through 0.22 μ m microporous membrane; test on the machine.

Note: For samples with sugar alcohol content lower than the detection-limit after being precipitated and diluted by acetonitrile, trichloroacetic acid shall be used for precipitation. For samples with low erythritol content ($\leq 1\%$), acetonitrile shall be used for precipitation. In other cases, both methods are available.

5.1.3 Biscuit, pastry, bread

Take at least 200 g of sample; use a grinder to grind; put it into a closed container to mix. Weigh 1 g ~ 5 g of pulverized sample; place it in a 50 mL centrifuge tube; add 40 mL of water; shake it; then sonicate for 30 min; shake it every 6 min; take out and

10.3 Standard

10.3.1

Xylitol (C₅H₁₂O₅, CAS No.: 87-99-0), with a purity $\geq 99\%$, or a standard substance certified by the state and awarded with a standard substance certificate.

10.3.2

Sorbitol (C₆H₁₄O₆, CAS No.: 50-70-4), with a purity $\geq 99\%$, or a standard substance certified by the state and awarded with a standard substance certificate.

10.3.3

Maltitol (C₁₂H₂₄O₁₁, CAS No.: 585-88-6), with a purity $\geq 99\%$, or a standard substance certified by the state and awarded with a standard substance certificate.

10.3.4

Erythritol (C₄H₁₀O₄, CAS No.: 149-32-6), with a purity $\geq 99\%$, or a standard substance certified by the state and awarded with a standard substance certificate.

10.4.1 Standard stock solution: respectively and accurately weigh 25 mg of xylitol, 25

mg of sorbitol, 25 mg of maltitol and 35 mg of erythritol, (accurate to 0.1 mg); add water to fix-volume to 10 mL; every milliliter of solution is equivalent to 3.5 mg of erythritol, 2.5 mg of xylitol, 2.5 mg of sorbitol and 2.5 mg of maltitol; it can be stored for 1 month at 4°C if sealed

well.

10.4.2 Standard working solution: respectively and accurately transfer 40 μL , 60 μL , 80 μL , 100 μL , 120 μL and 140 μL of various sugar alcohol standard stock solutions; add water to fix-volume to 1 mL; prepare erythritol into mixed series standard working solution with the concentration of 0.14mg/mL, 0.21 mg/mL, 0.28 mg/mL, 0.35 mg/mL, 0.42 mg/mL and 0.49 mg/mL; and prepare xylitol, sorbitol and maltitol into mixed series standard working solutions with the concentration of 0.10 mg/mL, 0.15 mg/mL, 0.20 mg/mL, 0.25 mg/mL, 0.30 mg/mL and 0.35 mg/mL respectively.

11.1 High performance liquid chromatography: with evaporative light scattering detector.

11.2 Chromatographic column: amino column with a column length of 250 mm, an

inner diameter of 4.6 mm and a particle size of 5 μm , or equivalent columns.

11.3 Food grinder.

11.4 Analytical balance: with the sensitivity of 0.1 mg and 0.01 g.

11.5 High speed centrifuge: speed $\geq 9\ 500$ r/min.

11.6 Ultrasonic cleaning machine: with the working frequency of 40 kHz, the power of 500 W.

Precipitate protein: add 5 mL of trichloroacetic acid solution into the supernatant; shake it; then place it at room temperature for 30 min; centrifuge at 9 500 r/min for 10 min. Take 8 mL of the supernatant in a 10 mL volumetric flask; add water to fix-volume.

After shaking, take 850 μL of filtrate; add 150 μL of sodium carbonate solution; shake and neutralize; or take 10 mL of supernatant; add 20 mL of acetonitrile; shake well and place it at room temperature for 30 min; centrifuge at 9 500 r/min for 10 min; fix-volume the supernatant to 50 mL; shake well. After filtering through a 0.22 μm filter, test it on the machine after dilution.

Note: For samples with sugar alcohol content lower than the detection-limit after being precipitated and diluted by acetonitrile, trichloroacetic acid shall be used for precipitation. For samples with low erythritol content ($\leq 1\%$), acetonitrile shall be used for precipitation. In other cases, both methods are available.