



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

GB 5009.251-2016

**National food safety standard - Determination of 1,2-propylene
glycol in food**

食品安全国家标准 食品中1, 2-丙二醇的测定

(English Translation)

Issued on: August 30, 2016

Implemented on: March 1, 2017

Issued by: National Health and Family Planning Commission of PRC

Table of Contents

Foreword...	4
1 Scope...	5
Method One -- Gas chromatography...	5
2 Principle...	5
3 Reagents and materials...	5
4 Apparatus...	6
5 Analysis steps...	6
6 Expression of analysis results...	9
7 Precision...	9
8 Other...	9
Method Two -- Gas chromatography-mass spectrometry...	9
9 Principle...	10
10 Reagents and materials...	10
11 Apparatus...	11
12 Analysis steps...	11
13 Expression of analysis results...	13
14 Precision...	13
15 Other...	13

Foreword

This Standard replaces GB/T 23813-2009 "Determination of 1,2-propanediol in foods", NY/T 1662-2008 "Determination of 1,2-propylene glycol in milk and dairy products - Gas chromatography method".

Compared with GB/T 23813-2009, the main deviations in this Standard are as follows.

- modified the standard name as "National Food Safety Standard - Determination of 1, 2-propylene glycol in food";
- added the pretreatment method for milk tablets;
- modified the pretreatment methods for milk powder, butter, cream, liquid

milk;

- added the limit of quantitation of the method.

National Food Safety Standard - Determination of 1,2-propylene glycol in food

1 Scope

This Standard specifies the methods that use gas chromatography and gas chromatography-mass spectrometry to determine 1,2-propylene glycol in food. Method One of this Standard is applicable to the determination of 1,2-propylene glycol in pastries, puffed food, cream, cheese, soy products, milk tablets, wet noodle products, frozen drinks, liquid milk, vegetable protein drinks, milk powder, butter, cream. Method Two is applicable to the determination of 1,2-propylene glycol in pastries, puffed food, cheese, soy products, milk tablets, wet noodle products.

Method One -- Gas chromatography

2 Principle

1,2-propylene glycol in the specimen is extracted by absolute ethanol. After the extract is filtered, use gas chromatography to determine. Characterize by retention time. Quantify by external standard method.

3 Reagents and materials

Unless otherwise stated, the reagents used in this method are analytically pure, the water is grade two water specified in GB/T 6682.

3.2.1 N-hexane saturated acetonitrile solution. Take the same volume of acetonitrile and n-hexane. Place in a separatory funnel. Oscillate. Perform static layering. Take the lower layer solution.

3.2.2 Acetonitrile saturated n-hexane solution. Take the same volume of acetonitrile and n-hexane. Place in a separatory funnel. Oscillate. Perform static

layering. Take the upper layer solution.

3.4 Preparation of standard solution

3.4.1 1,2-propylene glycol standard stock solution (10.0 mg/mL). Accurately weigh 1 g of 1,2-propylene glycol standard sample (to the nearest of 0.0001g). Use absolute ethanol to dissolve and transfer to a 100mL volumetric flask. Set volume to the scale. The mass concentration of 1,2-propylene glycol of this solution is 10.0 mg/mL. Store in a refrigerator at 4°C. It shall be valid for 3 months.

4.1 Gas chromatograph (GC). equipped with hydrogen flame ionization detector (FID).

5.1.1 Pastries, puffed food, cheese, soy products, milk tablets

The specimen is crushed by the mincer. Accurately weigh 5 g of well-mixed specimen (to the nearest of 0.01g) to a 100mL stoppered conical flask. Add into

50.0 mL of absolute ethanol. Vortex and mix for 2min, then perform oscillation extraction for 40min. After standing for 1h, use 0.45μm organic phase filter membrane to filter. The filtrate obtained is put into gas chromatograph for analysis.

5.1.3 Frozen drinks

A solid specimen is placed in a dry beaker to unfreeze. After melting, use a glass rod to smash and stir evenly. The liquid specimen is put at room temperature and shaken evenly.

5.1.4 Liquid milk, vegetable protein drinks

Accurately weigh 10 g of well-mixed specimen (to the nearest of 0.01g) into a 50mL stoppered colorimetric tube. Use absolute ethanol to set volume. Vortex and mix for 2min. After standing for 1h (centrifuge at 8000 r/min for 5min if necessary), use 0.45μm organic phase filter membrane to filter. The filtrate

obtained is put into gas chromatograph for analysis.

5.1.5 Milk powder

Accurately weigh 2 g of well-mixed specimen (to the nearest of 0.01g) into a 50mL stoppered colorimetric tube. Use 8 mL of 40°C water to dissolve and well mix. Use absolute ethanol to set volume. Vortex and mix for 2min. After standing for 1h (centrifuge at 8000 r/min for 5min if necessary), use 0.45µm organic phase filter membrane to filter. The filtrate obtained is put into gas chromatograph for analysis.

5.1.6 Butter, cream

Accurately weigh 2 g of well-mixed specimen (to the nearest of 0.01g) into a 50mL stoppered colorimetric tube. Use 6 mL of hexane to dissolve and well mix. Add into 20 mL of n-hexane saturated acetonitrile solution.

5.2.1 Chromatographic column. bonded/crosslinked polyethylene glycol

stationary phase quartz capillary column, 60m x 0.25mm, 0.25µm, or equivalent column.

5.2.2 Carrier gas. high purity nitrogen; constant current mode, column flow rate

of 1.0 mL/min.

5.2.3 Use program temperature rise. Column initial temperature is 80°C.

Maintain 1min. Rise temperature to 160°C at 20°C/min. Maintain 2min. Then rise temperature to 220°C at 15°C/min. Maintain 10min.

5.3 Production of standard curve

Inject the standard series working solutions into the gas chromatograph.

Determine the peak area of corresponding 1,2-propylene glycol. The standard curve is drawn by taking the mass concentration of the standard working solution as the abscissa and the peak area of the chromatographic peak as the ordinate.