GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

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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

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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.

3.1 Reagents

3.1.1 Anhydrous ethanol (C_2H_5OH).

3.1.2 Sea sand.

3.1.3 Acetonitrile (CH_3CN).

3.1.4 Hexane [$CH_3(CH_2)_4CH_3$].

3.2 Preparation of reagent

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.

5.1.2 Wet noddle products

The specimen is crushed by the mincer. Accurately weigh 2 g of well-mixed specimen (to the nearest of 0.01g) into the mortar. Add an appropriate amount of sea sand (the mass ratio of sea sand to sample is about 3:1 to 4:1) to grind into dry powder. Transfer all to the 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. 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.

5.1.4 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

Inject the specimen solution into the gas chromatograph. Obtain the peak area of corresponding 1,2-propylene glycol. According to the standard curve, obtain the mass concentration of 1,2-propylene glycol in the testing liquid.

See Figure A.1 for standard gas chromatograph of 1,2-propylene glycol.

6 Expression of analysis results

The content of 1,2-propylene glycol in specimen is calculated according to formula (1):

$$X = c \times \frac{V}{m \times 1\,000} \times \frac{1\,000}{1\,000} \qquad \cdots\cdots(1)$$

where,

X - the content of 1,2-propylene glycol in specimen, in grams per kilogram (g/kg);

c - the concentration of 1,2-propylene glycol in specimen solution found in the standard working curve, in microgram per milliliter (μg/mL);

V - the set volume of specimen, in milliliters (mL);

m - the specimen mass, in grams (g);

1000 - the conversion coefficient.

The calculated results are expressed as the arithmetic mean of two independent determinations obtained under repetitive conditions. The result remains three digits after the decimal point.

7 Precision

The absolute difference between two independent determinations obtained under repeatability conditions shall not exceed 10% of the arithmetic mean.

8 Other

When the sampling amount is 5 g, the detection limit of this Standard is 0.01 g/kg; the limit of quantification is 0.03 g/kg.

Method Two -- Gas chromatography-mass

11 Apparatus

11.1 Gas chromatography-mass spectrometry (GC-MS): EI source.

11.2 Analytical balances: resolutions of 0.01g and 0.0001g.

11.3 Mincer.

11.4 Vortex mixer.

11.5 Cyclotron oscillator.

12 Analysis steps

12.1 Preparation of specimen

12.1.1 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 mL of absolute ethanol. Vortex and mix for 2min, then perform oscillation extraction for 40min. After standing for 1h, use anhydrous sodium sulfate (about 2 g) to filter. The filtrate obtained is put into gas chromatography-mass spectrometry for analysis.

12.1.2 Wet noodle products

The specimen is crushed by the mincer. Accurately weigh 2 g of well-mixed specimen (to the nearest of 0.01g) into the mortar. Add an appropriate amount of sea sand (the mass ratio of sea sand to sample is about 3:1 to 4:1) to grind into dry powder. Transfer all to the 100mL stoppered conical flask. Add into 50 mL of absolute ethanol. Vortex and mix for 2min, then perform oscillation extraction for 40min. After standing for 1h, use anhydrous sodium sulfate (about 2 g) to filter. The filtrate obtained is put into gas chromatography-mass spectrometry for analysis.

12.2 Instrument reference conditions

12.2.1 Chromatographic column: bonded/crosslinked polyethylene glycol stationary phase quartz capillary column, 60m × 0.25mm, 0.25μm, or equivalent column.

12.2.2 Carrier gas: high purity helium; constant current mode, column flow rate of 1.0 mL/min.

12.2.3 Use program temperature rise: Column initial temperature is 80°C.

13 Expression of analysis results

The content of 1,2-propylene glycol in specimen is calculated according to formula (2):

$$X = c \times \frac{V}{m \times 1\,000} \times \frac{1\,000}{1\,000} \qquad \cdots\cdots(2)$$

where,

X - the content of 1,2-propylene glycol in specimen, in grams per kilogram (g/kg);

c - the concentration of 1,2-propylene glycol in specimen solution found in the standard working curve, in microgram per milliliter (μg/mL);

V - the set volume of specimen, in milliliters (mL);

m - the specimen mass, in grams (g);

1000 - the conversion coefficient.

The calculated results are expressed as the arithmetic mean of two independent determinations obtained under repetitive conditions. The result remains three digits after the decimal point.

14 Precision

The absolute difference between two independent determinations obtained under repeatability conditions shall not exceed 10% of the arithmetic mean.

15 Other

When the sampling amount is 5 g, the detection limit of this Standard is 0.002 g/kg; the limit of quantification is 0.006 g/kg.

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