GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

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National food safety standard -

Determination of ethyl maltol in food

食品安全国家标准

食品中乙基麦芽酚的测定

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National food safety standard - Determination of ethyl maltol in food

1 Scope

This standard specifies the method for the determination of ethyl maltol in food by high-performance liquid chromatography.

This standard applies to the determination of content of ethyl maltol in beverages, candies, jellies, meat products, biscuits, bread, cakes, milk powder foods.

2 Principle

After the specimen is extracted and purified, it is detected by a high-performance liquid chromatograph which is equipped with a diode array detector or a UV detector, quantified by an external standard method. The positive sample needs to be qualitatively confirmed by mass spectrometry.

3 Reagents and materials

Unless otherwise stated, the reagents used in this method are of analytical grade; the water is the grade II water as specified in GB/T 6682.

3.1 Reagents

3.1.1 Methanol (CH_3OH): Chromatographically pure.

3.1.2 Acetonitrile (CH_3CN): Chromatographically pure.

3.1.3 Sodium dihydrogen phosphate ($NaH_2PO_4 \cdot 2H_2O$).

3.2 Preparation of sodium dihydrogen phosphate solution

WEIGH 3.90 g of sodium dihydrogen phosphate; ADD water to dissolve and dilute it to about 1000 mL; USE phosphoric acid to adjust the pH to 4.0 ± 0.1; MAKE its volume reach to 1000 mL; USE a microporous membrane to filter it to prepare for use.

3.3 Ethyl maltol ($C_7H_8O_3$) standard substance

The purity is not less than 99%.

3.4 Preparation of standard solution

3.4.1 Standard stock solution: WEIGH 0.1 g of ethyl maltol (accurate to 0.0001 g); USE methanol to dissolve it and make its volume reach to the mark in a 100 mL volumetric flask. The concentration of this solution is 1 mg/mL.

3.4.2 Standard series working solution: Respectively and accurately TAKE different volume of standard stock solution; USE methanol to dilute it to the standard working solution which has a content of ethyl maltol of 0.0 μg/mL, 0.5 μg/mL, 2.0 μg/mL, 5.0 μg/mL, 25.0 μg/mL, 100.0 μg/mL.

4 Instruments and equipment

4.1 High-performance liquid chromatograph (HPLC): It is equipped with a diode array detector (DAD) or UV detector (UVD).

4.2 Ultrasonic cleaner.

4.3 Vortex mixer.

4.4 Water bath.

4.5 Centrifuge: The speed is not less than 6000 r/min.

4.6 Analytical balance: Sensitivity is 0.01 g, 0.001 g, 0.0001 g, respectively.

5 Analytical procedures

5.1 Preparation of specimen

5.1.1 Specimens of carbonated drinks, fruit drinks, milk drinks, vegetable protein drinks

Accurately WEIGH 10 g of specimen (accurate to 0.01 g) (carbonated beverage needs to be ultrasonic for 2 min ~ 3 min to remove carbon dioxide before being sampled) in a 25 mL stoppered test tube; USE acetonitrile to make its volume reach to the mark; MIX it uniformly; ULTRASONIC it for 10 min (if sample solution is turbid, CENTRIFUGE it at 6000 r/min for 10 min); TAKE the supernatant; USE the microporous membrane to filter it; LOAD the filtrate into the liquid chromatography for analysis.

5.1.2 Candies and jelly specimens

Accurately WEIGH 2 g of specimen (accurate to 0.001 g) in a 25 mL stoppered test tube; ADD 20 mL of water; PUT it in a water bath at 60 °C ~ 70 °C to heat

5.2.4 Column's temperature: 30 °C.

5.2.5 Detection wavelength: 276 nm.

5.2.6 Injection volume: 10 μL.

5.3 Production of standard curve

Respectively, INJECT the standard series working solution into the high-performance liquid chromatograph, to determine the chromatographic peak area of the corresponding ethyl maltol. USE the concentration of the standard working solution as the abscissa and the peak area of the chromatographic peak as the ordinate, to draw a standard curve.

5.4 Determination of sample solution

INJECT the sample solution into a high-performance liquid chromatograph, to obtain the chromatographic peak area of the corresponding ethyl maltol. According to the standard curve, OBTAIN the concentration of ethyl maltol in the test solution.

The standard liquid chromatogram of ethyl maltol is as shown in Figure A.1.

5.5 Qualitative confirmation

When the specimen is determined, if the retention time of the chromatographic peak of ethyl maltol is consistent with that of the standard substance, meanwhile the ultraviolet absorption spectrum of this substance is consistent with the ultraviolet absorption spectrum of the standard substance, it may initially confirm the presence of the ethyl maltol to be determined in the specimen. Positive specimen is subjected to a confirmation test by the use of a mass spectrometer (see Appendix B).

6 Expression of analytical results

The content of ethyl maltol in the specimen is calculated according to formula (1):

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

Where:

X - The content of ethyl maltol in the specimen, in milligrams per kilogram (mg/kg);

c - The concentration of ethyl maltol in the sample solution which is obtained

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