NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB 5009.293-2023

National food safety standard - Determination of monocaprylin in foods

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1 Scope

This standard specifies the gas chromatography and gas chromatography-mass spectrometry methods for the determination of capryl monoglyceride in food.

This standard is applicable to the determination of capryl monoglyceride in wet flour products, pastries, baked food fillings and surface paste (only bean paste), meat sausages, and spicy strips.

Method A - Gas chromatography

2 Principles

Capryl monoglyceride in wet flour products is extracted with n-hexane saturated acetonitrile, degreased with acetonitrile-saturated n-hexane, measured by gas chromatography, qualitatively analyzed according to retention time, and quantitatively analyzed with the external standard method.

Capryl monoglyceride in bean paste, pastries, meat sausages and spicy strips is extracted with n-hexane saturated acetonitrile, purified by gel permeation chromatography, and measured by gas chromatography, qualitatively analyzed according to retention time, and quantitatively analyzed with external standard method.

3 Reagents and materials

Unless otherwise stated, the reagents used in this method are all chromatographically pure, and the water is first-grade water specified in GB/T 6682.

3.2.1 n-hexane saturated acetonitrile, acetonitrile-saturated n-hexane: Measure 100 mL

of acetonitrile (3.1.2) into a 250 mL separatory funnel, add 100 mL of n-hexane, shake vigorously for several minutes, and let it stand; after it separates into layers, take the lower layer, then the n-hexane saturated acetonitrile solution is obtained; take the upper layer, then the acetonitrile-saturated n-hexane solution is obtained.

3.2.2 Ethyl acetate-cyclohexane (1+1): Mix ethyl acetate and cyclohexane in a volume

ratio of 1:1.

3.3 Standards

Capryl monoglyceride standard (capryl monoglyceride, C11H22O4, CAS number:

26402-26-6) with a purity of >=98%, or a standard with national authentication and a Reference Material Certificate.

3.4.1 Capryl monoglyceride standard stock solution (10.0 mg/mL): Accurately weigh

0.1 g (accurate to 0.0001 g) of standard capryl monoglyceride in a 10 mL volumetric flask, dissolve it in absolute ethanol, dilute to the mark, and mix well. Transfer the solution to a brown glass bottle and store it at -20 °C. It will be valid for 6 months.

3.4.2 Capryl monoglyceride standard intermediate solution (1.0 mg/mL): Take 1 mL of

capryl monoglyceride standard stock solution (10.0 mg/mL) into a 10 mL volumetric flask, dilute to volume with absolute ethanol, and mix well. Prepare it fresh just before use.

3.4.3 Capryl monoglyceride standard series working solution: Take 20 uL, 50 uL of

capryl monoglyceride standard intermediate solution (1.0 mg/mL), and 10 uL, 20 uL, 50 uL and 100 uL of capryl monoglyceride standard stock solution (10.0 mg/mL)

respectively; use absolute ethanol to prepare capryl monoglyceride standard series working solutions with concentrations of 0.0200 mg/mL, 0.0500 mg/mL, 0.100 mg/mL, 0.200 mg/mL, 0.500 mg/mL, and 1.00 mg/mL, respectively. Prepare the solutions fresh just before use.

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

at 30 °C to 40 °C until nearly dry; dilute to 2 mL with absolute ethanol, and pass through a 0.22 um organic filter membrane, for gas chromatography determination later.

5.1.3.2 Bean paste, pastries, meat sausages and spicy strips

Transfer bean paste, pastry, meat sausage, and spicy strip concentrate processed in 5.1.2 to a 10 mL volumetric flask, wash the eggplant-shaped evaporation flask twice with 6 mL of ethyl acetate-cyclohexane (3 mL each time), and combine the washing liquid into the same 10 mL volumetric flask; dilute to the mark with ethyl acetate-cyclohexane (1+1), and filter with a 0.22 um organic filter membrane. After the filtrate is purified by gel permeation chromatography, collect the eluate (for gel permeation chromatography conditions, please refer to Appendix A), rotary evaporate at 30 °C~40 °C until almost dry, dilute to 1 mL with absolute ethanol, and pass through a 0.22 um organic filter membrane, for gas chromatography determination later.

5.2 Instrument reference conditions

The reference conditions for gas chromatography are as follows:

a) Chromatographic column: (35%-phenyl)-methylpolysiloxane capillary column, 30 mx0.25 mm (inner diameter) x0.25 um (film thickness), or equivalent.

b) Temperature programming: The initial temperature is 60 °C, and hold for 1 minute; increase temperature to 340 °C at 20 °C/min, and hold for 5 minutes.

c) Inlet temperature: 340 °C.

d) Detector temperature: 340 degrees C, with a hydrogen flow of 30 mL/min, an air flow of 400 mL/min, and a makeup flow of 30 mL/min.

e) Carrier gas: nitrogen with a purity of >=99.999%, constant flow mode, and flow rate of

1.0 mL/min.

f) Injection method: Splitless injection; open the valve after 0.5 minutes.

g) Injection volume: 1.0 uL.

5.3 Preparation of standard curve

Inject the capryl monoglyceride standard series working solutions into the gas chromatograph respectively, and measure the corresponding peak area of capryl monoglyceride. Take the concentration of capryl monoglyceride in the standard working solution as the abscissa, the peak area of capryl monoglyceride as ordinate, and draw the standard curve. For the chromatogram of the capryl monoglyceride standard solution, see Figure B.1 in Appendix B.

8 Others The detection limit of this method is 0.050 g/kg, and the quantitation limit is 0.10 g/kg.

Method B - Gas chromatography-mass spectrometry

9 Principles Capryl monoglyceride in wet flour products is extracted with n-hexane saturated acetonitrile, degreased with acetonitrile-saturated n-hexane, and measured with gas chromatography-mass spectrometry; the selected ion monitoring mode (SIM) is used, the qualitative analysis is carried out according to the retention time and the abundance ratio of qualitative ion fragments, and external standard method is used for quantitative analysis.

Capryl monoglyceride in bean paste, pastries, meat sausages and spicy strips is extracted with n-hexane saturated acetonitrile, purified with gel permeation chromatography, and measured with gas chromatography-mass spectrometry; the selected ion monitoring mode (SIM) is used, the qualitative analysis is carried out according to the retention time and the abundance ratio of qualitative ion fragments, and external standard method is used for quantitative analysis.

10 Reagents and materials

10.1 Reagents Same as 3.1.

10.2 Reagent preparation Same as 3.2.

10.3 Standards Same as 3.3.

10.4 Preparation of standard solution

10.4.1 Capryl monoglyceride standard stock solution (10.0 mg/mL): Same as 3.4.1.

10.4.2 Capryl monoglyceride standard intermediate solution (1.0 mg/mL): Same as 3.4.2.

10.4.3 Capryl monoglyceride standard series working solution: Take 20 uL, 50 uL of capryl