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NATIONAL STANDARD OF THE

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GB 5009.264-2016

National food safety standard -

Determination of benzyl acetate in food

食品安全国家标准

食品中乙酸苄酯的测定

Issued on: December 23, 2016 **Implemented on: June 23, 2017**

Issued by: National Health and Family Planning Commission of the PRC;
China Food and Drug Administration

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National food safety standard - Determination of benzyl acetate in food

1 Scope

This standard specifies the gas chromatographic method for the determination of benzyl acetate in tea-drink.

This standard applies to the determination of benzyl acetate in tea-drink.

2 Principle

After the benzyl acetate in the tea-drink is extracted and made to certain volume, it is separated from the internal standard n-dodecane and other components in a gas chromatographic column, detected by a hydrogen flame ionization detector, quantified by an internal standard method.

3 Reagents and materials

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

3.1 Reagents

3.1.1 Dichloromethane (CH_2Cl_2).

3.1.2 Anhydrous ethanol (C_2H_6O).

3.2 Standard substance

3.2.1 Benzyl acetate ($C_9H_{10}O_2$): Content ≥ 98%.

3.2.2 n-Dodecane [$CH_3(CH_2)_{10}CH_3$]: Content ≥ 98%.

3.3 Preparation of standard solution

3.3.1 Benzyl acetate standard stock solution (1000 μg/mL): Accurately WEIGH 100 mg (accurate to 0.1 mg) of benzyl acetate; USE absolute ethanol to dissolve and dilute it to 100 mL.

3.3.2 Benzyl acetate use solution (80 μg/mL): Accurately TAKE 8.00 mL of 1000 μg/mL benzyl acetate solution; USE dichloromethane to make its volume reach

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to 100 mL. PREPARE it before use.

3.3.3 n-Dodecane internal standard solution (200 μg/mL): Accurately WEIGH 20 mg (accurate to 0.1 mg) of n-dodecane; USE dichloromethane to dilute it to 100 mL.

3.3.4 Preparation of mixed standard solution: ADD 4 mL of 80 μg/mL benzyl acetate and 1 mL of 200 μg/mL n-dodecane solution into a 5 mL colorimetric tube; MIX it uniformly; it is used to calculate the relative correction factor.

4 Instruments and equipment

4.1 Gas Chromatograph: It is equipped with a hydrogen flame ionization detector.

4.2 Nitrogen blow dryer.

4.3 Analytical balance: The sensitivities are 0.1 mg and 0.01 g.

5 Analytical procedures

5.1 Pretreatment of sample

Accurately WEIGH 40 g (accurate to 0.01 g) of tea-drink in a 125 mL separatory funnel; ADD 20 mL of dichloromethane; SHAKE it for 1 min; LET it be standing for layering; DROP the dichloromethane layer slowly into the funnel (a little cotton wool at the bottom, 10 g anhydrous sodium sulfate above it) to perform dehydration and filtration; COLLECT it into a 50 mL graduated test tube; then ADD 20 mL of dichloromethane into the separatory funnel; SHAKE it for 1 min; LET it be standing for layering; PERFORM filtration and collection as above; then USE a small amount of dichloromethane to rinse the funnel for 3 times; COLLECT it. USE nitrogen blow dryer (30 °C) to concentrate the collected solution to about 3 mL; USE a small amount of dichloromethane to completely transfer it into a 5 mL colorimetric tube. After adding 1 mL of 200 μg/mL n-dodecane, USE the dichloromethane to make its volume reach to 5 mL; MIX it uniformly to prepare for determination.

5.2 Reference conditions for gas chromatography

5.2.1 Chromatography column: SE-30 column, the column length is 30 m, the inner diameter is 0.53 mm, the film thickness is 1.0 μm, or others of equivalent performance.

5.2.2 Column temperature: The initial temperature is 90 °C. It is maintained at constant temperature 1 min, then subjected to programmed temperature-rise at the rate of 10 °C/min, the final temperature is 180 °C, which is kept for 3 min.

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