NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB 5009.306-2025

National food safety standard - Determination of diphenyl ether in foods

食品安全国家标准 食品中二苯醚的测定

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Standardization Administration of the PRC

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1 Scope

GB 5009.306-2025 is the Chinese national food safety standard for determining diphenyl ether in food. Diphenyl ether is used as a post-harvest fungicide on citrus and as a fragrance carrier, and it is both persistent and strongly odorous, so residues are a quality problem as well as a safety one. The standard gives two methods, gas chromatography and

gas chromatography-mass spectrometry, each set out with its principle, reagents and materials, instruments and equipment, analytical procedure, expression of results, precision and additional notes, with an annex giving the chromatogram of the standard solution. It applies to fruits, fruit products, fruit and vegetable juices and soft candies, and takes effect on 16 September 2025.

This Standard specifies the determination method of diphenyl ether in foods by gas chromatography and gas chromatography-mass spectrometry. This Standard is applicable to the determination of diphenyl ether in fruits, fruit products, fruit and vegetable juices, and soft candies. Method-I Gas Chromatography

2 Principle

Diphenyl ether in the specimen is extracted with n-hexane; leached by salting out; and the extract liquor is purified by N-propylethylenediamine (PSA) solid phase adsorbent and graphitized carbon black (GCB) solid phase adsorbent; detected by gas chromatograph equipped with hydrogen flame ionization detector; and quantified by external standard method.

3 Reagents and Materials

Unless otherwise specified, all the reagents used in this method are analytically pure; and water is Grade-1 water specified in GB/T 6682.

3.1 Reagents

3.1.1 n-hexane (C6H14). Chromatographically pure.

3.1.2 Sodium chloride (NaCl).

3.1.3 Anhydrous magnesium sulfate (MgSO4).

3.1.4 Sodium citrate (C6H5Na3O7).

3.1.5 Disodium hydrogen citrate (C6H6Na2O7).

3.2 Standard sample Diphenyl ether (Phenyl ether, C12H10O, CAS No.. 101-84-8). Standard substance purity >= 99%; or standard sample certified by the state and awarded with a standard substance certificate.

3.3 Preparation of standard solutions

3.3.1 Diphenyl ether standard stock solution (1,000 mg/L). Accurately weigh 10 mg (accurate to

0.1 mg) of diphenyl ether standard sample; dissolve it in n-hexane and make constant volume to 10 mL; store at 4 °C in the dark; the validity period is 3 months; or directly

purchase a standard solution that meets the traceability requirements.

3.3.2 Diphenyl ether standard intermediate solution (

10.0 mg/L). Accurately pipette

0.25 mL of diphenyl ether standard stock solution into a 25 mL volumetric flask; and make constant volume with n-hexane. Store at 4 °C away from light; with a validity period of 1 week.

3.3.3 Standard working solution. Accurately pipette

0.50 mL,

10.00 mL of diphenyl ether standard intermediate solution; make constant volume to 10 mL with n-hexane; and prepare standard working solutions with mass concentrations of 0.5 μg/mL, 1.0 μg/mL, 2.0 μg/mL, 5.0 μg/mL and 10.0 μg/mL, respectively. Prepare immediately before use.

3.4 Materials

3.4.1 N-propylethylenediamine (PSA) solid phase adsorbent. Particle size 40 μm~60 μm.

3.4.2 Graphitized carbon black (GCB) solid phase adsorbent. Particle size 120 μm~400 μm.

4 Instruments and Equipment

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

4.2 Analytical balance. Sensitivity is

0.001 g and 0.000 1 g, respectively.

4.3 Centrifuge. speed >= 5,000 r/min.

4.4 Vortex mixer.

5 Analysis Procedures

5.1 Preparation of specimen Fruit samples refer to GB 2763 Appendix A Food Categories and Test Parts for sampling. Fruit products take the edible part, and soft candy takes the whole; and fully mash them with a masher. Fruit and vegetable juice samples are directly sampled after being fully mixed. The sampling volume shall be no less than 500 g. The prepared specimens shall be placed in a clean container; sealed; labeled with sample information; and tested as soon as possible.

5.2.1 Fruits and fruit and vegetable juices. Weigh 5 g of the specimen (accurate to

0.001

g) in a 50 mL centrifuge tube; add 10 mL of n-hexane; and vortex and oscillate for 1 min.

5.2.2 Fruit products, soft candy. Weigh 5 g of the specimen (accurate to

0.001

g) into a 50 mL centrifuge tube; add 10 mL of warm water at about 50°C; dissolve by ultrasonic for 30 min; accurately add 10 mL of n-hexane; and vortex for 1 min.

5.3 Salting out Respectively weigh 1 g of sodium chloride, 4 g of anhydrous magnesium sulfate, 1 g of sodium citrate, and

0.5 g of disodium hydrogen citrate in a n-hexane-water mixed solution; vortex and mix for 1 min; stand and cool to room temperature. Centrifuge at 5,000 r/min for 5 min; and wait for purification. NOTE. Commercial salt packs with the same adsorbent can also be used directly.

5.4 Purification Transfer 5 mL of the upper-layer n-hexane extract to a centrifuge tube containing 885 mg of anhydrous magnesium sulfate, 150 mg of N-propylethylenediamine (PSA) solid phase adsorbent, and 15 mg of graphitized carbon black (GCB) solid phase adsorbent; vortex and mix for 1 min; centrifuge at 5,000 r/min for 5 min; and take about 1 mL of the upper-layer clear liquid for determination. NOTE. Commercial salt packs with the same adsorbent can also be used directly.

5.5 Instrument reference conditions

5.5.1 Gas chromatography reference conditions The gas chromatography reference conditions are as follows:

a) Chromatographic column. 5% phenyl-methyl polysiloxane non-polar column [30 mx

0.32 mm (inner diameter), 0.25 μm (film thickness)], or equivalent;

b) Chromatographic column temperature. Maintain 80 °C for 1 min; increase to 150 °C at a heating rate of 10 °C/min, maintain for 10 min; increase to 280 °C at a heating rate of 30 °C/min, maintain for 1 min;

c) Inlet temperature. 285 °C;

d) Carrier gas flow rate.

e) Combustion gas. hydrogen, flow rate of about 30 mL/min;

f) Combustion-supporting gas. air, flow rate of about 300 mL/min; Method-II Gas Chromatography-Mass Spectrometry