



**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB 5009.271-2016

**National food safety standard - Determination of phthalates in
food**

食品安全国家标准 食品中邻苯二甲酸酯的测定

(English Translation)

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Foreword

This standard replaces GB/T 21911-2008 "Determination of phthalate esters in foods" and SN/T 3147-2012 "Determination of phthalates in exported foods".

As compared with GB/T 21911-2008, the main changes of this standard are as follows.

- CHANGE the standard name to "National food safety standard - Determination of phthalates in food";
- ADD two target compounds. diallyl phthalate and diisononyl phthalate;
- ADD the isotope internal standard method for quantification as the method I;
- MODIFY the pre-processing method;

- MODIFY the method detection limit.

National food safety standard -

Determination of phthalates in food

1 Scope

The first method of this standard specifies the method for the determination of the contents of 16 phthalate esters in food by gas chromatography-mass spectrometry (GC-MS); the second method specifies the method for the determination of the contents of 18 phthalates in foods gas chromatography-mass spectrometry (GC-MS).

The first method of this standard applies to the determination and confirmation, by the internal standard method, of the contents of Dimethyl phthalate (DMP), Diethyl phthalate (DEP), Diisobutyl phthalate (DIBP), Dibutyl phthalate (DBP), Bis (2-methoxyethyl) phthalate (DMEP), Bis(4-methyl-2-pentyl) phthalate (BMPP), Bis(2-ethoxyethyl) phthalate (DEEP), Dipentyl phthalate (DPP), Dihexyl phthalate (DHXP), Benzylbutyl phthalate (BBP), Bis (2-n-butoxyethyl) phthalate (DBEP), Dicyclohexyl phthalate (DCHP), Bis (2-ethylhexyl) phthalate (DEHP), Diphenylphthalate (DPhP), Di-n-octylphthalate (DNOP) and Dinonyl phthalate (DNP) in foods; the second method is applicable to the determination and confirmation, by the external standard method, of the contents of Dimethyl phthalate (DMP), Diethyl phthalate (DEP), Diallyl phthalate (DAP), Diisobutyl phthalate (DIBP), Dibutyl phthalate (DBP), Bis (2-methoxyethyl) phthalate (DMEP), Bis(4-methyl-2-pentyl) phthalate (BMPP), Bis(2-ethoxyethyl) phthalate (DEEP), Dipentyl phthalate (DPP), Dihexyl phthalate (DHXP), Benzylbutyl phthalate (BBP), Bis (2-n-butoxyethyl) phthalate (DBEP), Dicyclohexyl phthalate (DCHP), Bis (2-ethylhexyl) phthalate (DEHP), Diphenylphthalate (DPhP), Di-n-octylphthalate (DNOP), Diisononyl ortho-phthalate (DINP), Dinonyl phthalate (DNP) in foods.

Method I -- Gas chromatography - Mass spectrometry

- Isotope internal standard method

2 Principle

The deuterated phthalate is added to the specimen as an internal standard, various foods are extracted and purified, and then determined by gas chromatography-mass spectrometry. The characteristic selective ion monitoring scan mode (SIM) is used, the ratio of the retention time to the abundance of the qualitative ion fragments is used for qualitative, the isotope internal standard method is used for quantification.

3 Reagents and materials

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

3.2.1 The 16 phthalate standard substances

The mixed liquid standard substance of Dimethyl phthalate (DMP), Diethyl phthalate (DEP), Diisobutyl phthalate (DIBP), Dibutyl phthalate (DBP), Bis (2-methoxyethyl) phthalate (DMEP), Bis(4-methyl-2-pentyl) phthalate (BMPP), Bis(2-ethoxyethyl) phthalate (DEEP), Dipentyl phthalate (DPP), Dihexyl phthalate (DHXP), Benzylbutyl phthalate (BBP), Bis (2-n-butoxyethyl) phthalate (DBEP), Dicyclohexyl phthalate (DCHP),

3.3.1 The 16 phthalate standard intermediate solutions (10 µg/mL). accurately

PIPETTE 1 mL of phthalate standard substance (1000 µg/mL) into a 100 mL volumetric flask, USE the n-hexane to make its volume reach to the mark.

3.3.2 Phthalate internal standard solution of 16 deuterated isotopes (100 µg/mL).

4 Instruments and equipment

Note: After washing the glassware, it is rinsed 3 times with distilled water, it is soaked in acetone for 1 hour, baked at 200 °C for 2 hours, and cooled to room temperature to prepare for use.

4.9 Solid phase extraction column. PSA/Silica composite packed glass column
(1000 mg, 6 mL).

5.2.1.1 Liquid specimen A. liquid milk, beverage, soy sauce, vinegar, liquor, honey, etc.

5.2.1.2 Liquid specimen B. vegetable oil, etc.

MIX the liquid fat uniformly, accurately WEIGH 0.5 g (accurate to 0.0001 g) in a 10 mL centrifuge tube, ADD 25 µL of isotope internal standard solution, ADD

100 µL of n-hexane and 2 mL of acetonitrile in order, VORTEX it for 1 min,

PERFORM ultrasonic extraction for 20 min, CENTRIFUGE it at 4000 r/min for

5.2.2.1 Semi-solid specimen A. jelly, sweet sauce, etc.

Accurately WEIGH 0.5 g (accurate to 0.0001 g) of the uniformly mixed specimen in a 25 mL stoppered centrifuge tube, ADD 125 µL of isotope internal standard solution, ADD 2 mL ~ 5 mL of distilled water, VORTEX it uniformly, then accurately ADD 10 mL of n-hexane, VORTEX it for 1 min, vigorously SHAKE it for 1 min, PERFORM ultrasonic extraction for 30 min, CENTRIFUGE it at 1000 r/min for 5 min, TAKE the supernatant to prepare for GC-MS analysis.

5.3 SPE purification

ADD 5 mL of dichloromethane and 5 mL of acetonitrile for activation, DISCARD the effluent; ADD the liquid to be purified to the SPE column, COLLECT the effluent; then ADD 5 mL of acetonitrile, COLLECT the effluent, COMBINE the two collected effluent, ADD 1mL of acetone, USE 40 °C nitrogen to blow it