



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

GB 31604.30-2025

Replacing GB 31604.30-2016

**National food safety standard - Food contact materials and
articles - Determination of phthalates and of their migration**

食品安全国家标准 食品接触材料及制品 邻苯二甲酸酯类化合物的测定和迁移量的测定

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1 Scope

GB 31604.30 is the Chinese national food safety method for phthalates in food contact materials: both the content in the material and the amount that migrates into the food. Phthalates are the most regulated of all food contact substances and the most frequently found: DEHP, DBP, DINP and their relatives are used to make PVC gaskets, tubing, films and closures flexible, and they move readily into fatty food. The method has to serve two different questions, what is in the material and what comes out of it, and the standard covers both, describing the determination by gas chromatography-mass spectrometry with the sample preparation and extraction for the content determination, the food simulants and the migration test conditions for the migration determination, the analytical procedure, the calculation of results, the precision and the limit of quantification. The 2025 edition replaces GB 31604.30-2016. It supports the limits set in GB 9685 and GB 4806.1. For a converter, a

brand or a testing laboratory working on Chinese food packaging, this is the prescribed method.

This Standard specifies the determination methods for 19 phthalate esters and the migration in food contact materials and products. dimethyl phthalate (DMP), diethyl phthalate (DEP), diallyl phthalate (DAP), diisobutyl phthalate (DIBP), di-n-butyl phthalate (DBP), bis(2-methoxyethyl) phthalate (DMEP), bis(4-methyl-2-pentyl) phthalate (BMPP), bis(2-ethoxyethyl) phthalate (DEEP), di-n-pentyl phthalate-D4 (DPP), di-n-hexyl phthalate (DHXP), benzyl butyl phthalate (BBP), bis(2-n-butoxyethyl) phthalate (DBEP), dicyclohexyl phthalate (DCHP), bis(2-ethylhexyl) phthalate (DEHP), diphenyl phthalate (DPhP), di-n-octyl phthalate (DNOP), dinonyl phthalate (DNP), diisononyl phthalate (DINP-1), diisononyl phthalate - C8 ~ C10 (C9 enriched) (DINP-2), diisodecyl phthalate (DIDP-1), and diisononyl phthalate - C9 ~ C11 (C10 enriched) (DIDP-2). Section I - Determination of Phthalate Esters is applicable to the determination of 19 phthalate esters in food contact materials and products, such as. plastics, rubber, paint coatings and adhesives, etc. Section II - Determination of Migration of Phthalate Esters is applicable to the determination of the migration of 19 phthalate esters in food contact materials and products. Section I - Determination of Phthalate Esters

2 Principle

Phthalate esters in food contact materials and products are extracted by ultrasonic method using tetrahydrofuran as the solvent. For soluble specimens, polymers and other substances are precipitated with n-hexane, and the extracting solution is centrifuged and concentrated, and after reaching a constant volume, it is used for gas chromatography-mass spectrometry. The insoluble specimens are repeatedly extracted using tetrahydrofuran, and the extracting solutions are concentrated, and after reaching a constant volume, they are used for gas chromatography- mass spectrometry. The retention times and characteristic ions are used for qualitative analysis, and the external standard method for quantitative analysis.

3 Reagents and Materials

Unless otherwise specified, the reagents used in this Method are chromatographically pure reagents, or analytically pure reagents, in which, no phthalate esters to be tested are detected by blank test analysis, and the water is Grade-1 water specified in GB/T 6682. During the test, avoid using plastic utensils, or use plastic utensils, in which, no phthalate esters to be tested are detected by blank test analysis.

3.1 Reagents

3.1.1 n-Hexane (C₆H₁₄).

3.1.2 Tetrahydrofuran (C₄H₈O).

3.2 Standard Substances 19 phthalate esters standard substances (see Appendix A). purity 95%, or standard substances certified by the state and awarded with standard substance certificates.

3.3 Preparation of Standard Solutions

3.3.1 Single standard stock solutions of 19 phthalate esters (5,000 mg/L). respectively and accurately weigh-take 50 mg of each of the 19 phthalate ester standard substances (in accordance with NOTE a in Table A.1, select DINP-1 or DINP-2, DIDP-1 or DIDP-2 standard substances), convert the actual weighed mass in accordance with the purity, accurate to

0.1 mg. Use n-hexane to dissolve them and accurately reach a constant volume of 10 mL, and mix them well. Transfer the solutions to brown glass containers and store them in a 20 C refrigerator away from light. The shelf life is 6 months.

3.3.2 Mixed standard intermediate solution A of 19 phthalate esters. accurately transfer-take 10 mL of DINP-1 or DINP-2 standard stock solution (5,000 mg/L), 10 mL of DIDP-1 or DIDP-2 standard stock solution (5,000 mg/L), 1 mL of DNOP standard stock solution (5,000 mg/L), 1 mL of DNP standard stock solution (5,000 mg/L) and

0.5 mL of each of the other phthalate esters standard stock solutions (5,000 mg/L) into a 100 mL volumetric flask, add n-hexane to reach a constant volume of 100 mL, and mix it well. Among them, the mass concentration of DINP-1 or DINP-2 is 500 mg/L, the mass concentration of DIDP-1 or DIDP-2 is 500 mg/L, the mass concentration of DNOP is 50 mg/L, the mass concentration of DNP is 50 mg/L, and the mass concentration of other phthalate esters is 25 mg/L. Transfer the solution to a brown glass container and store it in a 20 C refrigerator away from light. The shelf life is 6 months.

3.3.3 Phthalate esters series mixed standard working solutions. into five 50 mL volumetric flasks, respectively and accurately transfer-take

0.10 mL,

0.20 mL,

0.50 mL,

2.0 mL of 19 phthalate esters mixed standard intermediate solution A, use n-hexane to reach a constant volume and mix them well. Thus, the phthalate esters series mixed standard working solutions are obtained. Among them, the mass concentration of DINP-1 or DINP-2, DIDP-1 or DIDP-2 is

5.0 mg/L, 10 mg/L and 20 mg/L; the mass concentration of DNOP and DNP is

0.10 mg/L,

0.20 mg/L,

0.50 mg/L,

2.0 mg/L; the mass concentration of the other phthalate esters is

4 Instruments and Equipment

4.1 Gas chromatograph-mass spectrometer. equipped with electron impact ionization source (EI).

4.2 Analytical balance. with a division value of 0.0001 g.

4.3 Glassware. NOTE. after cleaning, use water to rinse it 3 times, soak in tetrahydrofuran for 1 hour and rinse 3 times, dry, and cool to room temperature for later use.

4.4 Ultrasonic generator.

4.5 Rotary evaporator.

4.6 Centrifuge. speed 4,000 r/min.

4.7 Vortex oscillator.

4.8 Cryogenic grinding instrument.

4.9 Glass filter membrane. organic phase, with a pore size of 0.45 μ m.

5.1 Sample Pre-treatment

5.1.1 Preparation of test solution Take representative specimens. For solid specimens, such as. resin, plastic and rubber, etc., cut them into fragments, or cut them into fragments and then, use liquid nitrogen to perform cryogenic grinding, so as to control the size of the fragments to

0.1 cm, and evenly mix them. For thin layer solid specimens, such as. coatings and adhesive layers, use a knife to scrape them, or use a knife to scrape them and then, cut them into fragments or use liquid nitrogen to perform cryogenic grinding, so as to control the size of the fragments to

0.3 cm, and evenly mix them. For liquid specimens, such as. coatings and adhesives, directly sample them. Accurately weigh-take

0.2 g ~

0.5 g of specimen (accurate to 0.0001

g) into a centrifuge tube, add 10 mL of tetrahydrofuran, and extract by ultrasonic method for 30 min. For specimens that are soluble or turbid during ultrasound, after extraction, slowly add 40 mL of n-hexane and perform vortex oscillation. At 4,000 r/min, centrifuge for 20 min.