NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB 5009.31-2025

National food safety standard - Determination of p-hydroxybenzoic acid esters in foods

食品安全国家标准 食品中对羟基苯甲酸酯类化合物的测定

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Foreword

This document was issued on 16 March 2025 by the State Administration for Market Regulation; Standardization Administration of the PRC and takes effect on 16 September 2025.

It is a GB standard without the /T suffix: compliance is mandatory in China.

1 Scope

GB 5009.31-2025 is the Chinese national food safety standard for determining parabens in food. The p-hydroxybenzoic acid esters are permitted preservatives with maximum use levels, and the reason they are watched more closely than most is that the longer-chain members have shown weak oestrogenic activity, so the several esters have to be determined separately rather than as a group. The standard gives the methods in sequence - gas chromatography, liquid chromatography and liquid chromatography-tandem mass spectrometry - each with its principle, reagents and materials, instruments and equipment, analytical steps, expression of results, precision and notes, with annexes giving the

chromatograms of the standard solutions and the multiple reaction monitoring conditions. It takes effect on 16 September 2025.

This Standard specifies the determination method for p-hydroxybenzoic acid esters (methyl paraben, ethyl paraben, propyl paraben, isopropyl paraben, butyl paraben, isobutyl paraben and heptyl 4-hydroxybenzoate) in foods.

The first method of this Standard, gas chromatography, is applicable to the determination of p-hydroxybenzoic acid esters in foods (except heat-coagulated egg products, brewed sauce and seasoning sauce).

The second method of this Standard, high performance liquid chromatography, and the third method, liquid chromatography-tandem mass spectrometry, are applicable to the determination of p-hydroxybenzoic acid esters in foods.

Method I - Gas Chromatography

2 Principle

The specimen is extracted with methanol-water, purified and enriched by a mixed strong anion exchange solid phase extraction column, and concentrated and then, determined by a gas chromatograph equipped with a hydrogen flame ionization detector, and quantified by the external standard method.

3 Reagents and Materials

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

3.1 Reagents

3.1.1 Methanol (CH3OH).

3.1.2 Formic acid (HCOOH).

3.1.3 Concentrated ammonia water (NH3 ? H2O). 25% ~ 28%.

3.2 Preparation of Reagents

3.2.1 Methanol-water solution (2 + 8). measure-take 100 mL of methanol and 400 mL of water,

solutions is 2.0 ?g/mL, 5.0 ?g/mL, 10 ?g/mL, 20 ?g/mL, 50 ?g/mL and 100 ?g/mL.

3.5 Materials

3.5.1 Organic phase microporous filter membrane. with a pore size of 0.22 ?m.

3.5.2 Mixed strong anion exchange solid phase extraction column. 500 mg/6 mL, or other columns with equivalent performance.

4 Instruments and Equipment

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

4.2 Electronic balance. respectively with a division value of 0.001 g and 0.0001 g.

4.3 Vortex mixer.

4.4 High-speed centrifuge. maximum speed is not less than 6,000 r/min.

4.5 Ultrasonic oscillator.

4.6 Nitrogen blowing instrument.

4.7 Homogenizer.

4.8 Grinder.

5 Analytical Steps

5.1 Preparation of Specimens

Liquid samples are mixed, semi-solid samples are homogenized with a homogenizer, and solid

samples are thoroughly grinded with a grinder and evenly stirred. The prepared specimens are

placed in a clean specimen container, sealed and labeled. Liquid specimens are stored at 4 ?C,

and other specimens are stored at ?18 ?C.

5.2 Processing of Specimens

5.2.1 Extraction

5.2.1.1 Fresh fruits, fresh vegetables, baked food fillings and surface batter, vinegar, soy sauce, liquid compound seasoning, etc.

Weigh-take 5 g (accurate to 0.01 g) of sample into a 50 mL graduated centrifuge tube, add 5 mL of water, conduct vortex for 3 min, add 10 mL of methanol, conduct vortex for 3 min, conduct ultrasound for 20 min, and at 6,000 r/min, centrifuge it for 3 min. Take all the supernatant into another 50 mL graduated centrifuge tube, add 10 mL of methanol-water solution (2 + 8) to the residue, repeat the extraction once, combine the two extracting solutions, add water to 40 mL, at 6,000 r/min, centrifuge it for 3 min, take all the supernatant for purification.

5.2.1.2 Fruit and vegetable juice, carbonated beverages, flavored beverages, etc.

Weigh-take 5 g (accurate to 0.01 g) of specimen into a 50 mL graduated centrifuge tube (carbonated beverages need to be ultrasonically degassed in an ultrasonic cleaner for 10 minutes before weighing), add 30 mL of methanol-water solution (3 + 7), conduct vortex for 3 minutes, and conduct ultrasound for 20 minutes. Then, add methanol-water solution (3 + 7) and reach 40 mL, at 6,000 r/min, centrifuge it for 3 minutes, filter the supernatant, take the filtrate for purification.

5.2.1.3 Jams, etc.

Weigh-take 5 g (accurate to 0.01 g) of sample into a 50 mL graduated centrifuge tube, add 5 mL of concentrated ammonia water, conduct vortex for 3 min, let it stand for 1 h; add 10 mL of methanol, conduct vortex for 3 min, conduct ultrasound for 20 min, and at 6,000 r/min, centrifuge it for 3 min. Take all the supernatant into another 50 mL graduated centrifuge tube.

Add 10 mL of methanol-water solution (2 + 8) to the residue, conduct vortex for 3 min, conduct