



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

GB 31604.54-2023

**National food safety standard - Food contact materials and
products - Determination of migration of bisphenol F and S**

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1 Scope

This Standard specifies the methods for the determination of migration of bisphenol F and S in food contact materials and products by liquid chromatography and liquid chromatography-tandem mass spectrometry.

Method 1 - liquid chromatography is applicable to the determination of migration of bisphenol F and S in plastic materials and products for food contact, paints and coatings for food contact, paper and cardboard materials and products for food contact.

Method 2 - liquid chromatography - tandem mass spectrometry is applicable to the determination of migration of bisphenol F and S in food contact materials and products.

Method 1 - Liquid Chromatography

2 Principle

After food contact materials and products are subject to migration test in accordance with GB 31604.1 and GB 5009.156, adopt liquid chromatography for detection. Specifically speaking, after filtration, aqueous, acidic and alcoholic food simulants are directly injected; use nitrogen to blow-dry 95% (volume fraction) ethanol, then, use ethanol-water to reach a constant volume, filter and inject the sample; use methanol-water mixture to extract oily food simulants, filter and inject the sample; use nitrogen to blow-dry the chemical alternative solvent - isooctane, use methanol-water to reach a constant volume, filter and inject the sample. Adopt the external standard method for quantitative determination.

3 Reagents and Materials

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

3.1.1 Aqueous, acidic, alcoholic and oily food simulants and chemical alternative solvents: the

reagents used shall comply with the stipulations of GB 5009.156.

3.2.1 Aqueous, acidic, alcoholic, corn oil food simulants and chemical alternative solvents:

operate in accordance with GB 5009.156.

3.2.2 Methanol-water mixture (1 + 1): measure-take 100 mL of methanol and 100 mL of water, and evenly mix it.

3.2.3 Ethanol-water mixture (1 + 1): measure-take 100 mL of ethanol and 100 mL of water, and

evenly mix it.

3.3.1 Bisphenol F (C₁₃H₁₂O₂, CAS: 620-92-8): purity ? 99%, or a standard substance certified

by the state and awarded a reference material certificate.

3.3.2 Bisphenol S (C₁₂H₁₀O₄S, CAS: 80-09-1): purity ? 99%, or a standard substance certified

by the state and awarded a reference material certificate.

3.4.1.1 Bisphenol F (bisphenol S) standard stock solution A (100 mg/L, methanol)

Accurately weigh-take 10 mg (accurate to 0.1 mg) of bisphenol F (bisphenol S) reference material, use methanol to dissolve it and reach a constant volume in a 100 mL volumetric flask, then, evenly mix it. Transfer the solution to a brown standard solution stock bottle and store it in a refrigerator at 4 °C and away from light. It shall remain valid for 3 months.

3.4.1.2 Bisphenol F (bisphenol S) standard stock solution B (100 mg/L, isooctane)

Accurately weigh-take 10 mg (accurate to 0.1 mg) of bisphenol F (bisphenol S) reference material, use 10 mL of acetone to dissolve it, then, use isooctane to reach a constant volume in a 100 mL volumetric flask, then, evenly mix it. Transfer the solution to a brown standard solution stock bottle and accurately add 2 g (accurate to 0.01 g) of corn oil into five 10 mL test tubes with stoppers, add 200 µL, 300 µL, 400 µL, 1.00 mL and 2.00 mL of bisphenol F standard intermediate solution C (3.4.2.3); add 600 µL of bisphenol S standard intermediate solution D (3.4.2.4), and 100 µL, 200 µL, 400 µL and 1.00 mL of bisphenol S standard intermediate solution C (3.4.2.3) to obtain mixed standard solutions with bisphenol F concentration of 100 µg/kg, 150 µg/kg, 200 µg/kg, 500 µg/kg and 1.00 mg/kg, and bisphenol S concentration of 30.0 µg/kg, 50.0 µg/kg, 100 µg/kg, 200 µg/kg and 500 µg/kg. Prepare them right before use.

Respectively add 3 mL of n-hexane to each test tube and evenly mix it. Respectively add 2.20 mL, 2.60 mL, 2.40 mL, 1.60 mL and 0 mL of methanol-water mixture into five 10 mL test tubes with stoppers; conduct vortex oscillation for 2 min and let it stand for stratification. Draw-take the lower-layer solution, filter it through a 0.45 µm polytetrafluoroethylene microporous filter membrane, and reserve it for determination.

3.4.3.3 Standard working solutions of chemical alternative solvent - isooctane

Accurately draw-take 0.150 mL, 0.250 mL, 0.500 mL, 2.50 mL and 5.00 mL of bisphenol F and bisphenol S standard intermediate solution B (3.4.2.2) into five 50 mL volumetric flasks;

use isooctane to reach a constant volume to obtain bisphenol F and bisphenol S mixed standard working solutions with a concentration of 30.0 µg/L, 50.0 µg/L, 100 µg/L, 500 µg/L and 1.00 mg/L. Before injecting into the instrument, it must be handled simultaneously with the food simulant soaking solution in 5.1.2.4.

4.1 Liquid chromatograph, equipped with ultraviolet detector (UV) or diode array detector

(DAD).

5.1.1 Migration test

In accordance with the requirements of GB 5009.156 and GB 31604.1, conduct a migration test on the sample to obtain a food simulant test solution. If the test solution obtained from the migration test cannot be immediately tested, it shall be stored in a refrigerator at 4 °C and away from light. The storage period is 7 days. Before proceeding to the next step of test, the food simulant test solution shall be returned to room temperature.

5.1.2.1 Aqueous, acidic and alcoholic [except for 95% (volume fraction) ethanol] food

simulants Accurately transfer-take about 2 mL of the aqueous, acidic and alcoholic food simulants obtained from the migration test, use a 0.45 µm polytetrafluoroethylene microporous filter membrane to filter it, and reserve it for determination.

5.1.2.2 95% (volume fraction) ethanol Accurately draw-take 1 mL of 95% (volume fraction) ethanol obtained from the migration test into a test tube and use nitrogen to blow-dry it. Accurately add 1 mL of ethanol-water mixture into the test tube and conduct vortex mixing for 2 min. Use a 0.45 µm polytetrafluoroethylene microporous filter membrane to filter the solution, and reserve it for determination.

5.1.2.3 Oily food simulants

Accurately weigh-take 2 g (accurate to 0.01 g) of corn oil obtained from the migration test into a 10 mL test tube, add 3 mL of n-hexane and evenly mix it. Add 3 mL methanol-water mixture, conduct vortex oscillation for 2 min, and let it stand for stratification. Draw-take the lower-layer solution, filter it through a 0.45 µm polytetrafluoroethylene microporous filter membrane, and reserve it for determination.

5.1.2.4 Chemical alternative solvent - isooctane

Accurately draw-take 5 mL of chemical alternative solvent - isooctane obtained from the migration test into a test tube and use nitrogen to blow-dry it. Accurately add 5 mL of methanol-water mixture into the test tube and conduct vortex mixing for 2 min. Use a 0.45 µm polytetrafluoroethylene microporous filter membrane to filter the solution, and reserve it for determination.

5.1.3 Preparation of blank test solution

In accordance with 5.1.1 and 5.1.2, handle the food simulants and chemical alternative solvents not in contact with food contact materials and products.

5.2 Reference Conditions of Instrument

The reference conditions of the instrument are as follows: