

Translated English of Chinese Standard: GB31604.54-2023

www.ChineseStandard.net → Buy True-PDF → Auto-delivery.

Sales@ChineseStandard.net

GB

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**

食品安全国家标准 食品接触材料及制品 双酚 F 和双酚 S 迁移量
的测定

Issued on: September 6, 2023

Implemented on: March 6, 2024

**Issued by: National Health Commission of the People's Republic of China;
State Administration for Market Regulation.**

Table of Contents

1 Scope.....	3
Method 1 - Liquid Chromatography.....	3
2 Principle.....	3
3 Reagents and Materials.....	3
4 Instruments and Equipment.....	6
5 Analytical Procedures.....	6
6 Expression of Analysis Results.....	9
7 Precision.....	10
8 Others.....	10
Method 2 - Liquid Chromatography - Tandem Mass Spectrometry.....	11
9 Principle.....	11
10 Reagents and Materials.....	11
11 Instruments and Equipment.....	14
12 Analytical Procedures.....	14
13 Expression of Analysis Results.....	17
14 Precision.....	17
15 Others.....	17
Appendix A Liquid Chromatograms of Bisphenol F and Bisphenol S.....	18
Appendix B Mass Spectrometry Reference Conditions of Liquid Chromatography - Tandem Mass Spectrometry.....	23
Appendix C Total Ions Chromatograms of Bisphenol F and Bisphenol S.....	24

National Food Safety Standard - Food Contact Materials and Products - Determination of Migration of Bisphenol F and S

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 Reagents

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.

3.1.2 n-Hexane (C_6H_{14}).

3.1.3 Methanol (CH_4O).

3.1.4 Acetonitrile ($\text{C}_2\text{H}_3\text{N}$).

3.1.5 Isooctane (C_8H_{18}).

3.1.6 Acetone ($\text{C}_3\text{H}_6\text{O}$).

3.1.7 Ethanol ($\text{C}_2\text{H}_6\text{O}$).

3.1.8 Corn oil: chemically pure, complies with the requirements of GB 5009.156.

3.2 Preparation of Reagents

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 Reference Materials

3.3.1 Bisphenol F ($\text{C}_{13}\text{H}_{12}\text{O}_2$, CAS: 620-92-8): purity $\geq 99\%$, or a standard substance certified by the state and awarded a reference material certificate.

3.3.2 Bisphenol S ($\text{C}_{12}\text{H}_{10}\text{O}_4\text{S}$, CAS: 80-09-1): purity $\geq 99\%$, or a standard substance certified by the state and awarded a reference material certificate.

3.4 Preparation of Standard Solutions

3.4.1 Standard stock solutions

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

Respectively and accurately add 2 g (accurate to 0.01 g) of corn oil into five 10 mL test tubes with stoppers, add 200 μ L, 300 μ L, 400 μ L, 1.00 mL and 2.00 mL of bisphenol F standard intermediate solution C (3.4.2.3); add 600 μ L of bisphenol S standard intermediate solution D (3.4.2.4), and 100 μ L, 200 μ L, 400 μ L 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 μ g/kg, 150 μ g/kg, 200 μ g/kg, 500 μ g/kg and 1.00 mg/kg, and bisphenol S concentration of 30.0 μ g/kg, 50.0 μ g/kg, 100 μ g/kg, 200 μ g/kg and 500 μ g/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 μ m 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 μ g/L, 50.0 μ g/L, 100 μ g/L, 500 μ g/L and 1.00 mg/L. Before injecting into the instrument, it must be handled simultaneously with the food simulatant soaking solution in 5.1.2.4.

4 Instruments and Equipment

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

4.2 Vortex oscillator.

4.3 Pipettes: 20 μ L, 50 μ L, 250 μ L, 1,000 μ L and 5,000 μ L.

4.4 Analytical balance: with a division value of 0.0001 g and 0.01 g, respectively.

4.5 Constant-temperature water bath.

4.6 Nitrogen blower.

4.7 Polytetrafluoroethylene microporous filter membrane: 0.45 μ m.

5 Analytical Procedures

5.1 Preparation of Test Solutions

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 Treatment of food simulants

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:

- a) Chromatographic column: C₁₈ column, column length 150 mm, inner diameter 4.6 mm, particle size 5 µm; or one with equivalent performance.

oily food simulants, the detection limit of bisphenol F in food contact materials and products is 0.03 mg/kg and the quantification limit is 0.1 mg/kg; the detection limit of bisphenol S is 0.01 mg/kg and the quantification limit is 0.03 mg/kg.

Method 2 - Liquid Chromatography - Tandem Mass Spectrometry

9 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 - tandem mass spectrometry for detection. Specifically speaking, after filtration, aqueous, acidic and alcoholic food simulants are directly injected; oily food simulants are extracted through methanol-water mixture, filtered, then injected. Use nitrogen to blow-dry chemical alternative solvent - isooctane, then, use methanol-water to reach a constant volume, filter and inject the sample. Adopt the external standard method for quantitative determination.

10 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.

10.1 Reagents

10.1.1 Aqueous, acidic, alcoholic and oily food simulants and chemical alternative solvents: the reagents used shall comply with the stipulations of GB 5009.156.

10.1.2 n-Hexane (C_6H_{14}).

10.1.3 Methanol (CH_4O).

10.1.4 Isooctane (C_8H_{18}).

10.1.5 Acetone (C_3H_6O).

10.1.6 Olive oil: chemically pure, complies with the requirements of GB 5009.156.

10.2 Preparation of Reagents

10.2.1 Aqueous, acidic, alcoholic, olive oil food simulants and chemical alternative solvents: operate in accordance with GB 5009.156.

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

10.3 Reference Materials

Same as 3.3.

10.4 Preparation of Standard Solutions

10.4.1 Standard stock solutions

10.4.1.1 Bisphenol F standard stock solution A (1,000 mg/L, methanol)

Accurately weigh-take 50 mg (accurate to 0.1 mg) of bisphenol F reference material, use methanol to dissolve it and reach a constant volume in a 50 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.

10.4.1.2 Bisphenol S standard stock solution A (50 mg/L, methanol)

Accurately weigh-take 10 mg (accurate to 0.1 mg) of bisphenol S reference material, use methanol to dissolve it and reach a constant volume in a 200 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.

10.4.1.3 Bisphenol F standard stock solution B (1,000 mg/L, isooctane)

Accurately weigh-take 50 mg (accurate to 0.1 mg) of bisphenol F reference material, add 10 mL of acetone to dissolve it, then, use isooctane to reach a constant volume in a 50 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.

10.4.1.4 Bisphenol S standard stock solution B (50 mg/L, isooctane)

Accurately weigh-take 10 mg (accurate to 0.1 mg) of bisphenol S reference material, add 10 mL of acetone to dissolve it, then, use isooctane to reach a constant volume in a 200 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.

10.4.2 Standard intermediate solutions

10.4.2.1 Bisphenol F and bisphenol S standard intermediate solution A (10 mg/L, 0.5 mg/L, methanol)

Accurately draw-take 1.00 mL of bisphenol F standard stock solution A (10.4.1.1) and 1.00 mL of bisphenol S standard stock solution A (10.4.1.2) into a 100 mL volumetric flask, use methanol to reach a constant volume, 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 1 month.

10.4.2.2 Bisphenol F and bisphenol S standard intermediate solution B (10 mg/L, 0.5 mg/L,

isooctane)

Accurately 1.00 mL of bisphenol F standard stock solution B (10.4.1.3) and 1.00 mL of bisphenol S standard stock solution B (10.4.1.4) into a 100 mL volumetric flask, use isooctane to reach a constant volume, 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 1 month.

10.4.2.3 Bisphenol F and bisphenol S standard intermediate solution C (1 mg/L, 0.05 mg/L, methanol-water)

Accurately draw-take 10 mL of bisphenol F and bisphenol S standard intermediate solution A (10.4.2.1) into a 100 mL volumetric flask, use methanol-water mixture to reach a constant volume, then, evenly mix it. Prepare it right before use.

10.4.3 Preparation of standard working solutions**10.4.3.1 Standard working solutions of aqueous, acidic and alcoholic food simulants**

Accurately draw-take 50.0 µL, 250 µL, 500 µL, 750 µL and 1.00 mL of bisphenol F and bisphenol S standard intermediate solution A (10.4.2.1) into five 50 mL volumetric flasks; use water to reach a constant volume and obtain bisphenol F standard working solutions with a concentration of 10.0 µg/L, 50.0 µg/L, 100 µg/L, 150 µg/L and 200 µg/L and bisphenol S standard working solutions with a concentration of 0.500 µg/L, 2.50 µg/L, 5.00 µg/L, 7.50 µg/L and 10.0 µg/L. Adopt the same mode, and respectively use acidic and alcoholic food simulants to prepare standard working solutions of the same concentration series; prepare them right before use. Alcoholic food simulants include 10% (volume fraction) ethanol, 20% (volume fraction) ethanol, 50% (volume fraction) ethanol and 95% (volume fraction) ethanol. In accordance with GB 31604.1 and based on the actual use conditions of food contact materials, select them.

10.4.3.2 Standard working solutions of oily food simulants

Respectively and accurately add 2 g (accurate to 0.01 g) of olive oil into five 10 mL test tubes with stoppers, add 100 µL, 250 µL, 500 µL, 1.00 mL and 1.50 mL of bisphenol F and bisphenol S standard intermediate solution C (10.4.2.3) to obtain bisphenol F standard working solutions with a concentration of 50.0 µg/kg, 125 µg/kg, 250 µg/kg, 500 µg/kg and 750 µg/kg and bisphenol S standard working solutions with a concentration of 2.50 µg/kg, 6.25 µg/kg, 12.5 µg/kg, 25.0 µg/kg and 37.5 µg/kg; prepare them right before use. Respectively add 3 mL of n-hexane to each test tube and evenly mix it. Respectively add 2.90 mL, 2.75 mL, 2.50 mL, 2.00 mL and 1.50 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 0.22 µm polytetrafluoroethylene microporous filter membrane, and reserve it for determination.

10.4.3.3 Standard working solutions of chemical alternative solvent - isooctane

Accurately draw-take 50.0 μL , 250 μL , 500 μL , 750 μL and 1.00 mL of bisphenol F and bisphenol S standard intermediate solution B (10.4.2.2) into five 50 mL volumetric flasks; use isooctane to reach a constant volume to obtain bisphenol F standard working solutions with a concentration of 10.0 $\mu\text{g/L}$, 50.0 $\mu\text{g/L}$, 100 $\mu\text{g/L}$, 150 $\mu\text{g/L}$ and 200 $\mu\text{g/L}$ and bisphenol S standard working solutions with a concentration of 0.500 $\mu\text{g/L}$, 2.50 $\mu\text{g/L}$, 5.00 $\mu\text{g/L}$, 7.50 $\mu\text{g/L}$ and 10.0 $\mu\text{g/L}$. Before injecting into the instrument, it must be handled simultaneously with the food simulant soaking solution in 12.1.2.3.

11 Instruments and Equipment

11.1 Liquid chromatograph - tandem mass spectrometer: equipped with electrospray ion source (ESI).

11.2 Vortex oscillator.

11.3 Pipettes: 20 μL , 50 μL , 250 μL , 1,000 μL and 5,000 μL .

11.4 Analytical balance: with a division value of 0.0001 g and 0.01 g, respectively.

11.5 Constant-temperature water bath.

11.6 Nitrogen blower.

11.7 Polytetrafluoroethylene microporous filter membrane: 0.22 μm .

12 Analytical Procedures

12.1 Preparation of Test Solutions

12.1.1 Migration test

Same as 5.1.1.

12.1.2 Treatment of food simulants

12.1.2.1 Aqueous, acidic and alcoholic food simulants

Transfer-take about 2 mL of the aqueous, acidic and alcoholic food simulants obtained from the migration test, use a 0.22 μm polytetrafluoroethylene microporous filter membrane to filter it, and reserve it for determination.

12.1.2.2 Oily food simulants

Accurately weigh-take 2 g (accurate to 0.01 g) of olive 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 0.22 μm polytetrafluoroethylene microporous filter membrane,

This is an excerpt of the PDF (Some pages are marked off intentionally)

Full-copy PDF can be purchased from 1 of 2 websites:

1. <https://www.ChineseStandard.us>

- SEARCH the standard ID, such as GB 4943.1-2022.
- Select your country (currency), for example: USA (USD); Germany (Euro).
- Full-copy of PDF (text-editable, true-PDF) can be downloaded in 9 seconds.
- Tax invoice can be downloaded in 9 seconds.
- Receiving emails in 9 seconds (with download links).

2. <https://www.ChineseStandard.net>

- SEARCH the standard ID, such as GB 4943.1-2022.
- Add to cart. Only accept USD (other currencies - <https://www.ChineseStandard.us>).
- Full-copy of PDF (text-editable, true-PDF) can be downloaded in 9 seconds.
- Receiving emails in 9 seconds (with PDFs attached, invoice and download links).

Translated by: Field Test Asia Pte. Ltd. (Incorporated & taxed in Singapore. Tax ID: 201302277C)

About Us (Goodwill, Policies, Fair Trading...): <https://www.chinesestandard.net/AboutUs.aspx>

Contact: Wayne Zheng, Sales@ChineseStandard.net

Linkin: <https://www.linkedin.com/in/waynezhengwenrui/>

----- The End -----