



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

GB 31604.57-2023

**National food safety standard - Food contact materials and
products - Determination of migration of benzophenone
substances**

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1 Scope

This Standard specifies the methods for the determination of migration of eight benzophenone substances in food contact materials and products.

Method 1 - liquid chromatography is applicable to the determination of migration of (2-hydroxy-4-methoxyphenyl) (2-hydroxyphenyl) ketone, 2,4-dihydroxybenzophenone, (2-hydroxy-4-methoxyphenyl) phenyl ketone, 4-4'-dihydroxybenzophenone, 2-hydroxy-4-n-octyloxy-benzophenone, 2-hydroxy-4-n-hexyloxy-benzophenone, benzophenone and 4-4'-difluoro-benzophenone in plastics, paints and coatings, rubber, and food contact materials and products using inks in water, 4% (volume fraction) acetic acid, 10% (volume fraction) ethanol, 20% (volume fraction) ethanol, 50% (volume fraction) ethanol food simulants, as well as chemical alternative solvents - 95% (volume fraction)

ethanol and isooctane.

Method 2 - liquid chromatography - tandem mass spectrometry is applicable to the determination of migration of (2-hydroxy-4-methoxyphenyl) (2-hydroxyphenyl) ketone, 2,4-dihydroxybenzophenone, (2-hydroxy-4-methoxyphenyl) phenyl ketone, 4-4'-dihydroxybenzophenone, 2-hydroxy-4-n-octyloxy-benzophenone, 2-hydroxy-4-n-hexyloxy-benzophenone, benzophenone and 4-4'-difluoro-benzophenone in plastics, paints and coatings, rubber, and food contact materials and products using inks in water, 4% (volume fraction) acetic acid, 10% (volume fraction) ethanol, 20% (volume fraction) ethanol, 50% (volume fraction)

ethanol and olive oil food simulants, as well as chemical alternative solvents - 95% (volume fraction) ethanol and isooctane.

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. Water, 4% (volume fraction) acetic acid, 10% (volume fraction) ethanol, 20% (volume fraction) ethanol and 50% (volume fraction) ethanol food simulants are directly injected; chemical alternative solvent -95% (volume fraction) ethanol is diluted by water, filtered, then, injected. Use nitrogen to blow the chemical alternative solvent - isooctane, then, use methanol-water to re-dissolve it, filter, then, inject the sample. Adopt the external standard method of peak area for quantitative determination.

3 Reagents and Materials

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

3.2 Preparation of Reagents

3.2.1 4% (volume fraction) acetic acid solution, 10% (volume fraction) ethanol solution, 20% (volume fraction) ethanol solution and 50% (volume fraction) ethanol solution are prepared in accordance with GB 5009.156.

3.2.2 95% (volume fraction) ethanol solution: evenly mix absolute ethanol and water in a volume ratio of 95 : 5.

3.2.3 Methanol-water solution (7 + 3): evenly mix methanol and water in a volume ratio of 7 :

3.

3.3.1 (2-hydroxy-4-methoxyphenyl) (2-hydroxyphenyl) ketone (C₁₄H₁₂O₄, CAS: 131-53-3):

purity ? 98%, or a standard substance certified by the state and awarded a reference material certificate.

3.3.2 2,4-dihydroxybenzophenone (C₁₃H₁₀O₃, CAS: 131-56-6): purity ? 98%, or a standard substance certified by the state and awarded a reference material certificate.

3.3.3 (2-hydroxy-4-methoxyphenyl) phenyl ketone (C₁₄H₁₂O₃, CAS: 131-57-7): purity ? 98%, or a standard substance certified by the state and awarded a reference material certificate.

3.3.4 4-4'-dihydroxybenzophenone (C₁₃H₁₀O₃, CAS: 611-99-4): purity ? 98%, or a standard substance certified by the state and awarded a reference material certificate.

3.3.5 2-hydroxy-4-n-octyloxy-benzophenone (C₂₁H₂₆O₃, CAS: 1843-05-6): purity ? 98%, or a standard substance certified by the state and awarded a reference material certificate.

3.3.6 2-hydroxy-4-n-hexyloxy-benzophenone (C₁₉H₂₂O₃, CAS: 3293-97-8): purity ? 98%, or a standard substance certified by the state and awarded a reference material certificate.

3.3.7 Benzophenone (C₁₃H₁₀O, CAS: 119-61-9): purity ? 98%, or a standard substance certified

by the state and awarded a reference material certificate.

3.3.8 4-4'-difluoro-benzophenone (C₁₃H₈F₂O, CAS: 345-92-6): purity ? 98%, or a standard substance certified by the state and awarded a reference material certificate.

3.4.1 Standard stock solutions (1,000 mg/L)

Accurately weigh-take 50 mg (accurate to 0.1 mg) of each of the eight benzophenone reference materials, respectively use methanol to dissolve them, then, transfer them to eight 50 mL volumetric flasks. Use methanol to reach a constant volume to the scale and evenly mix them.

Transfer the solutions to brown glass containers and store them airtight at 4 ?C away from light.

They shall remain valid for 6 months.

3.4.2 Standard mixed intermediate solution

Respectively draw-take 0.5 mL of 4-4'-dihydroxybenzophenone, benzophenone and 4-4'-difluoro-benzophenone standard stock solutions (1,000 mg/L) and 2.0 mL of the standard stock solutions (1,000 mg/L) of the other five benzophenone substances into a

100 mL volumetric flask. Use methanol to reach a constant volume to the scale, evenly mix it to obtain a Standard mixed intermediate solution, in which, the concentration of 4,4'-dihydroxybenzophenone, benzophenone and 4,4'-difluoro-benzophenone is 5.0 mg/L, and the concentration of the other five benzophenone substances is 20.0 mg/L. Transfer the solution to a brown glass container and store it airtight at 4 °C away from light. It shall remain valid for 3 months.

3.4.3.1 Standard mixed series of working solutions of water, 4% (volume fraction) acetic

acid, 10% (volume fraction) ethanol, 20% (volume fraction) ethanol and 50% (volume fraction) ethanol. Respectively draw-take 40 µL, 100 µL, 200 µL, 400 µL and 600 µL of the standard mixed intermediate solution into five 10 mL volumetric flasks. Use the corresponding food simulants to reach a constant volume to the scale, evenly mix them to obtain standard mixed series of working solutions, in which, the concentration of 4,4'-dihydroxybenzophenone, benzophenone and 4,4'-difluoro-benzophenone is 0.020 mg/L, 0.050 mg/L, 0.10 mg/L, 0.20 mg/L and 0.30 mg/L, and the concentration of the other five benzophenone substances is 0.080 mg/L, 0.20 mg/L, 0.40 mg/L, 0.80 mg/L and 1.2 mg/L. Prepare them right before use.

3.4.3.2 Standard mixed series of working solutions of chemical alternative solvent - 95%

(volume fraction) ethanol

5 Analytical Procedures

5.1 Preparation of test solutions

5.1.1 Migration test In accordance with the requirements of GB 31604.1 and GB 5009.156, conduct a migration test.

After the soaking solution returns to room temperature, handle it in accordance with 5.1.2. If the soaking solution obtained from the migration test cannot be immediately tested, it shall be stored in a refrigerator at 4 °C away from light for no more than 72 hours. After returning to room temperature, it shall be handled in accordance with 5.1.2.

5.1.2 Treatment of soaking solutions

5.1.2.1 Food simulant test solutions of water, 4% (volume fraction) acetic acid, 10% (volume fraction) ethanol, 20% (volume fraction) ethanol and 50% (volume fraction)

ethanol Transfer-take an appropriate amount of water, 4% (volume fraction) acetic acid, 10% (volume fraction) ethanol, 20% (volume fraction) ethanol and 50% (volume fraction) ethanol soaking solutions obtained after the migration test; centrifuge at 5,000 r/min for 5 minutes. Reserve the supernatant for determination.