



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

GB 31604.64-2025

**National food safety standard - Food contact materials and
products - Determination of migration of citrate and sebacate
compounds**

食品安全国家标准 食品接触材料及制品 柠檬酸酯和癸二酸酯类化合物迁移量的
测定

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Commission of the People's Republic of China**

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0.1 g) of olive oil into 6 glass test tubes. Accurately pipette 0 mL,

0.015 mL,

0.030 mL,

0.060 mL,

0.12 mL, and

0.15 mL of the mixed intermediate standard solution B; mix well; and then add

0.25 mL of 10% (mass fraction) ammonia water and

3.00 mL of acetonitrile; vortex for extraction for 5 min; allow to stand for separation; and transfer the supernatant acetonitrile solution to a clean volumetric flask. Add

3.00 mL of acetonitrile and repeat the extraction once more. Combine the two extracts into a 15 mL centrifuge tube containing 900 mg of magnesium sulfate, 150 mg of PSA, and 150 mg of C18; and centrifuge at 4000 r/min for 5 min. Take

4.00 mL of the supernatant acetonitrile solution; blow it to near dryness with nitrogen; and then make constant volume to

1.00 mL with acetonitrile to obtain mixed standard working solutions with mass concentrations of 0 mg/kg,

0.50 mg/kg,

5.0 mg/kg. Prepare immediately before use.

3.5.7 Standard working solutions for chemical alternative solvents (95% ethanol and isooctane) Respectively and accurately pipette 0 mL,

0.020 mL,

1 Scope

GB 31604.64 is the Chinese national food safety method for the migration of citrate and sebacate esters from food contact materials and articles. These are the plasticisers the industry has moved to as phthalates have been restricted: acetyl tributyl citrate and the sebacates are now widely used in cling film, gaskets, tubing and closures. Being newer, they are less studied, and the migration limits set for them in GB 9685 need a method that can prove compliance; this is that method. The document describes the determination by gas chromatography-mass spectrometry, and covers the principle, the reagents and materials, the apparatus, the food simulants and migration test conditions, the analytical procedure, the calculation of results, the precision and the limit of quantification. It is part of the GB 31604 series of test methods that supports the GB 4806 framework for food contact materials. Effective 2 March 2026.

This Standard specifies the methods for determining the migration amounts of triethyl citrate, dibutyl sebacate, acetyl tributyl citrate, di(2-ethylhexyl) sebacate, diisooctyl sebacate, and bis(2,2,6,6-tetramethyl-4-piperidyl) sebacate in food contact materials and products. This Standard applies to the determination of the migration amounts of triethyl citrate, dibutyl sebacate, acetyl tributyl citrate, di(2-ethylhexyl) sebacate, diisooctyl sebacate, and bis(2,2,6,6- tetramethyl-4-piperidyl) sebacate in food contact materials and products such as plastics, rubber, adhesives, coatings and paints, and paper, etc.

2 Principle

Migration tests were conducted on food contact materials and products using food simulants or chemical substitute solvents. Aqueous, acidic, and ethanol-containing food simulant soaking solutions were extracted with n-hexane before determination on the instrument. Fat- and oil- containing food simulant soaking solutions were extracted with acetonitrile; and the extracts were purified by matrix dispersion extraction and nitrogen blowing before determination on the instrument. Soaking solutions using chemical

substitute solvents (95% ethanol and isooctane) were directly determined on the instrument. The test solutions were separated by gas chromatography column, detected by mass spectrometry; and quantified using the external standard method based on peak area.

3 Reagents and Materials

Unless otherwise specified, all reagents used in this method are of analytical reagent; and the water is Grade I water as specified in GB/T 6682. During the experiment, any plastic equipment that may contaminate the analysis shall be avoided. Glassware and equipment should be thoroughly cleaned before use.

3.1 Reagents

3.1.1 N-hexane (C_6H_{14}). Chromatographic grade.

3.1.2 Acetonitrile (C_2H_3N). Chromatographic grade. 3.1.3 25% (mass fraction) ammonia water.

3.1.4 Magnesium sulfate ($MgSO_4$).

3.1.5 Anhydrous ethanol (C_2H_6O). Chromatographic grade.

3.1.6 Glacial acetic acid ($C_2H_4O_2$).

3.1.7 Isooctane (C_8H_{18}). Chromatographic grade.

3.1.8 Olive oil. It shall comply with the provisions of fat and oil food simulants in GB 5009.156.

3.2 Materials

3.2.1 Ethylenediamine-N-propylsilylane silica gel filler (PSA). 40 μm ~60 μm .

3.2.2 Octadecylsilane chemically bonded silica gel filler (C18). 40 μm ~60 μm .

3.3 Preparation of reagents

3.3.1 Acidic, ethanol-containing, and fat- and oil-containing food simulants and chemical substitute solvents. As specified in GB 31604.1 and GB 5009.156. 3.3.2 10% (mass fraction) ammonia water. Pipette 211 mL of 25% ammonia water and 289 mL of water; and mix well.

3.4 Reference standard

3.4.1 Triethyl citrate ($C_{12}H_{20}O_7$, CAS No.. 77-93-0). Purity $\geq 98\%$, or a reference standard certified by the state and granted a reference standard certificate.

3.4.2 Dibutyl sebacate (C₁₈H₃₄O₄, CAS No.. 109-43-3). Purity $\geq 98\%$, or a reference standard certified by the state and granted a reference standard certificate.

3.4.3 Acetyl tributyl citrate (C₂₀H₃₄O₈, CAS No.. 77-90-7). Purity $\geq 98\%$, or a reference standard certified by the state and granted a reference standard certificate.

3.4.4 Di(2-ethylhexyl) sebacate (C₂₆H₅₀O₄, CAS No.. 122-62-3). Purity $\geq 98\%$, or a reference standard certified by the state and granted a reference standard certificate.

3.4.5 Diisooctyl sebacate (C₂₆H₅₀O₄, CAS No.. 27214-90-0). Purity $\geq 98\%$, or a reference standard certified by the state and granted a reference standard certificate.

3.4.6 Bis(2,2,6,6-tetramethyl-4-piperidyl) sebacate (C₂₈H₅₂N₂O₄, CAS No.. 52829-07-9). Purity $\geq 98\%$, or a reference standard certified by the state and granted a reference standard certificate.

3.5 Preparation of standard solutions

3.5.1 Preparation of single standard stock solutions (1000 mg/L) Respectively and accurately take 10 mg (accurate to

0.1 mg) of each of the following reference standards. triethyl citrate, dibutyl sebacate, acetyl tributyl citrate, di(2-ethylhexyl) sebacate, diisooctyl sebacate, and bis(2,2,6,6-tetramethyl-4-piperidyl) sebacate. Dissolve each reference standard in n-hexane and then transfer to a 10 mL brown volumetric flask. Make constant volume to the mark with n-hexane and mix well. Transfer the solution to a brown glass bottle and store in a refrigerator at 0°C~4°C, protected from light. Shelf life is 6 months.

3.5.2 Preparation of mixed standard intermediate solution A (100 mg/L) Respectively and accurately pipette

1.00 mL of each of the above 6 single standard stock solutions into the same 10 mL brown volumetric flask. Accurately make constant volume to the mark with anhydrous ethanol and mix well. Transfer the solution to a brown glass bottle and store in a refrigerator at 0°C~4°C, protected from light. Shelf life is 3 months.

3.5.3 Preparation of mixed standard intermediate solution B (100 mg/L) Respectively and accurately pipette

1.00 mL of each of the above 6 single standard stock solutions into the same 10 mL brown volumetric flask. Accurately make constant volume to the mark with n-hexane and mix well. Transfer the solution to a brown glass bottle and store in a refrigerator at 0°C~4°C, protected from light. Shelf life is 3 months.

3.5.4 Standard working solution for acidic food simulants Respectively pipette

5.00 mL of 4% acetic acid into six graduated glass tubes. Accurately transfer 0 mL,