GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB 31604.61-2025

National Food Safety Standard - Food Contact Materials and Products - Determination of Migration of 2,2,4,4-Tetramethyl-1,3-Cyclobutanediol

食品安全国家标准 食品接触材料及制品 2,2,4,4-四甲基-1,3-环丁二醇迁移量的测定

Issued on: March 16, 2025 **Implemented on: September 16, 2025**

Issued by: National Health Commission of the People's Republic of China;

State Administration for Market Regulation.

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National Food Safety Standard - Food Contact Materials and Products - Determination of Migration of 2,2,4,4-Tetramethyl-1,3-Cyclobutanediol

1 Scope

This Standard specifies the method for determining the migration of 2,2,4,4-tetramethyl-1,3-cyclobutanediol in food contact materials and products.

This Standard is applicable to the determination of the migration of 2,2,4,4-tetramethyl-1,3-cyclobutanediol in plastic and coating food contact materials and products.

2 Principle

After the food contact materials and products are subjected to migration test with food simulants or alternative solvents, water, acidic and ethanol-containing food simulant soaking solutions are diluted with methanol and then tested. Oil-fat-containing food simulant soaking solutions are extracted with methanol and then tested. Chemical alternative solvent (95% ethanol and isooctane) soaking solutions are directly tested on the machine. The test solutions are separated by gas chromatography column and tested by hydrogen flame ionization detector. The retention time is used for qualitative analysis and the peak area is used for quantitative analysis.

3 Reagents and Materials

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

3.1 Reagents

3.1.1 Acetic acid ($C_2H_4O_2$).

3.1.2 Ethanol (C_2H_6O).

3.1.3 Isooctane (C_8H_{18}): Chromatographically pure.

3.1.4 Olive oil: Must meet the requirements of Appendix A in GB 5009.156.

3.1.5 Methanol (CH_4O): Chromatographically pure.

3.1.6 Methyl tert-butyl ether ($C_5H_{12}O$): Chromatographically pure.

3.2 Preparation of reagents

4% (volume fraction) acetic acid solution, 10% (volume fraction) ethanol solution, 20% (volume fraction) ethanol solution, 50% (volume fraction) ethanol solution and 95% (volume fraction) ethanol solution: Prepare according to the provisions of GB 5009.156. The used food simulants shall meet the requirements of GB 31604.1 and relevant product standards.

3.3 Standard sample

2,2,4,4-tetramethyl-1,3-cyclobutanediol ($C_8H_{16}O_2$, CAS No.: 3010-96-6): Purity ≥ 99%, or standard substances/standard samples certified by the state and awarded with standard substance certificates.

3.4 Preparation of standard solution

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

Accurately weigh 25 mg (accurate to 0.1 mg) of 2,2,4,4-tetramethyl-1,3-cyclobutanediol standard sample; dissolve it in methanol and transfer it to a 25 mL brown volumetric bottle; make constant volume to the scale with methanol; shake well and stand; then store it in a brown glass bottle. This solution is sealed and stored in a refrigerator at 0 ℃ ~4 ℃, and the validity period is 6 months.

3.4.2 Standard intermediate solution

3.4.2.1 Standard intermediate solution I (100 mg/L): Accurately transfer 1.00 mL of standard stock solution into a 10 mL brown volumetric flask; accurately make constant volume to the scale with methanol; shake well and stand; and store in a brown glass bottle. This solution is sealed and stored in a refrigerator at 0 ℃ ~4 ℃, and the validity period is 3 months.

3.4.2.2 Standard intermediate solution II: Accurately transfer 0.10 mL, 0.20 mL, 0.40 mL, 1.00 mL and 2.00 mL of standard stock solution into a 10 mL brown volumetric flask; make constant volume to the scale with methyl tert-butyl ether; shake well and stand; and obtain standard intermediate solutions with mass concentrations of 10 mg/L, 20 mg/L, 40 mg/L, 100 mg/L and 200 mg/L, respectively; and store in a brown glass bottle. This solution is sealed and stored in a refrigerator at 0 ℃ ~4 ℃, and the validity period is 3 months.

3.4.3 Standard working solution

3.4.3.1 Standard working solution A (applicable to water-based food simulants): Accurately transfer 50 μL, 100 μL, 200 μL, 300 μL and 500 μL of standard intermediate solution I with a mass concentration of 100 mg/L into 5 pieces of 10 mL volumetric flasks, respectively; accurately add 2.0 mL of the corresponding blank food simulants (water, 4% acetic acid, 10% ethanol, 20% ethanol, 50% ethanol), respectively; and then dilute and make constant volume to the scale with methanol; shake well and stand to obtain standard working solutions with mass

5.1.2.1 Water-based food simulant soaking solution

Accurately transfer 2.0 mL of the immersion solution after the migration test into a 10 mL volumetric flask; dilute with methanol and make constant volume to the scale; shake well; and filter through a 0.45 μm organic phase nylon microporous filter membrane before testing.

5.1.2.2 Chemical alternative solvent (95% ethanol and isooctane) soaking solution

Take 1 mL of soaking solution and filter it through a 0.45 μm organic phase nylon microporous filter membrane for testing.

5.1.2.3 Oil-fat-containing food simulant soaking solution

Accurately weigh 2.0 g (accurate to 0.001 g) of olive oil soaking solution obtained in the migration test into a stoppered glass test tube; add 100 μL of methyl tert-butyl ether; and then add 2 mL of methanol for extraction. After vortexing for 2 min, stand for stratification; take 1 mL of the upper-layer clear liquid; filter it through a 0.45 μm organic phase nylon microporous filter membrane for testing.

5.1.3 Preparation of blank test solution

Treat food simulants and chemical alternative solvents that are not in contact with food contact materials and products according to 5.1.1~5.1.2, respectively.

5.2 Instrument reference conditions

5.2.1 Chromatographic column: 6% cyanopropyl/phenyl 94% dimethylpolysiloxane capillary gas chromatography column; 30 m×0.25 mm×1.4 μm, or equivalent chromatographic column.

5.2.2 Inlet temperature: 250 ℃.

5.2.3 Detector temperature: 300 ℃.

5.2.4 Heating program: 90 ℃ for 0 min, increase to 250 ℃ at 15 ℃/min, and hold for 1 min.

5.2.5 Carrier gas: nitrogen, flow rate 1.5 mL/min.

5.2.6 Injection method: non-split.

5.2.7 Injection volume: 1 μL.

5.3 Drawing of standard curve

According to the instrument reference conditions listed in 5.2, the standard working solution (3.4.3) is measured from low to high. The standard curve is drawn with the concentration of 2,2,4,4-tetramethyl-1,3-cyclobutanediol in the standard working solution as the horizontal axis and the corresponding peak area (the sum of the peak areas of the cis and trans isomers of 2,2,4,4-tetramethyl-1,3-cyclobutanediol) as the vertical axis.

m_1 - mass of solid food in contact with sample during actual use; or mass of food corresponding to volume of liquid food in contact with sample, in kg. For various liquid foods, the volume is converted into corresponding mass according to the density of 1 kg/L. When the S_0/m_1 in actual use is unknown or cannot be estimated, S_0/m_1 is calculated as 6 dm^2/kg, that is, 6 dm^2 of food contact materials and products contact 1 kg of food or food simulant.

The calculation result retains 2 significant figures.

NOTE: The determination result of the migration amount of the target analyte 2,2,4,4-tetramethyl-1,3-cyclobutanediol includes the sum of the cis isomer and the trans isomer.

6.2 Calculation of the specific migration amount of 2,2,4,4-tetramethyl-1,3-cyclobutanediol in sealed food contact materials and products (expressed in mg/kg)

For sealed food contact materials and products, when the intended use is known and the specific migration amount of the target analyte 2,2,4,4-tetramethyl-1,3-cyclobutanediol is expressed in mg/kg, it is calculated according to Formula (2):

$$X_2 = \frac{(c - c_0) \times V}{S} \times \frac{S_0}{m_2} \qquad \cdots\cdots (2)$$

Where:

X_2 - specific migration amount of target analyte, in mg/kg;

c - content of target analyte in specimen soaking solution, in mg/L or mg/kg;

c_0 - content of target analyte in blank soaking solution, in mg/L or mg/kg;

V - volume or mass of specimen soaking solution, in L or kg;

S - contact area between specimen and soaking solution in migration test, in dm^2;

S_0 - area of contact between sealed product and food during actual use, in dm^2;

m_2 - mass of solid food in contact with sealed product and adapter container during actual use, or mass of food corresponding to volume of contact with liquid food, in kg. The volume of various liquid foods is converted into the corresponding mass according to density of 1 kg/L.

The calculation result retains 2 significant figures.

NOTE: The determination result of the migration amount of the target analyte 2,2,4,4-tetramethyl-1,3-cyclobutanediol includes the sum of the cis isomer and the trans isomer.

6.3 Calculation of the specific migration amount of 2,2,4,4-tetramethyl-1,3-cyclobutanediol for sealed food contact materials and products (expressed in mg/piece)

For sealed food contact materials products, when the intended use is unknown and the specific

migration amount of the target analyte 2,2,4,4-tetramethyl-1,3-cyclobutanediol is expressed in mg/piece, it is calculated according to Formula (3). At this time, the used migration test method and the contact area of a single sealed product with the food simulant in the migration test must be indicated.

$$X_3 = \frac{(c - c_0) \times V}{n} \qquad \cdots\cdots(3)$$

Where:

X_3 - specific migration amount of target analyte, in mg/piece;

c - content of target analyte in specimen soaking solution, in mg/L or mg/kg;

c_0 - content of target analyte in blank soaking solution, in mg/L or mg/kg;

V - volume or mass of specimen soaking solution, in L or kg;

n - number of sealing products used during soaking, in pieces.

The calculation result retains 2 significant figures.

NOTE: The determination result of the migration amount of target analyte 2,2,4,4-tetramethyl-1,3-cyclobutanediol includes the sum of cis-isomer and trans-isomer.

7 Precision

The absolute difference between two independent determination results obtained under repeatability conditions shall not exceed 15% of the arithmetic mean.

8 Others

The detection limit of this method for the migration of 2,2,4,4-tetramethyl-1,3-cyclobutanediol in the soaking solution of water-based food simulants (water, 4% acetic acid, 10% ethanol, 20% ethanol and 50% ethanol) is 1 mg/L; and the quantification limit is 2.5 mg/L. The detection limit for the migration of 2,2,4,4-tetramethyl-1,3-cyclobutanediol in the soaking solution of chemical alternative solvents (95% ethanol, isooctane) is 0.2 mg/L; and the quantification limit is 0.50 mg/L. The detection limit for the migration of 2,2,4,4-tetramethyl-1,3-cyclobutanediol in the soaking solution of oil-fat-containing food simulants is 0.2 mg/kg; and the quantification limit is 0.50 mg/kg. The detection limit and quantification limit of the migration amount of 2,2,4,4-tetramethyl-1,3-cyclobutanediol shall be calculated according to Clause 6.

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