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NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB 31604.55-2023

**National Food Safety Standards -- Food Contact Materials
and Products -- Determination of Migration Amount of
Isothiazolinone Compounds**

食品安全国家标准 食品接触材料及制品 异噻唑啉酮类化合物迁
移量的测定

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National Food Safety Standards -- Food Contact Materials and Products -- Determination of Migration Amount of Isothiazolinone Compounds

1 Scope

This Standard specifies the methods to determine 1,2-benzisothiazolyl-3(2H)-one, 2-methyl-3(2H)-isothiazolinone and 5-chloro-2-methyl-2H-isothiazol-3-one migration amounts in food contact materials and products.

This Standard applies to the determination of 1,2-benzisothiazolyl-3(2H)-one, 2-methyl-3(2H)-isothiazolinone and 5-chloro-2-methyl-2H-isothiazol-3-one migration amounts in food contact plastics, paints and coatings, rubber, paper and cardboard, and food contact materials and articles using inks or adhesives.

Method One -- High-performance Liquid Chromatography

2 Principle

Food contact materials and products are tested using high-performance liquid chromatography after migration tests in accordance with GB 31604.1 and GB 5009.156. Among them, the samples of water, 4% (volume fraction) acetic acid, 10% (volume fraction) ethanol, 20% (volume fraction) ethanol, 50% (volume fraction) ethanol food simulant and chemical alternative solvent 95% (volume fraction) ethanol soaking solution are injected directly after filtration. After the oil-containing food simulants and the chemical alternative solvent isooctane soaking solution are extracted by methanol-water solution, the lower layer is filtered and injected. Use UV detector or diode array detector for detection, and peak area external standard method for quantification.

3 Reagents and materials

Unless otherwise stated, the reagents used in this method are all chromatographically pure, and the water is grade one water specified in GB/T 6682.

3.1 Reagents

3.1.1 Acidic, ethanol-containing, grease-containing food simulants and chemical alternative solvents: the reagents used must meet the requirements of GB 5009.156.

3.1.2 Methanol (CH₄O).

3.1.3 Tetrahydrofuran (C₄H₈O).

3.1.4 n-Hexane (C₆H₁₄).

3.2 Reagent preparation

3.2.1 Acidic, ethanol-containing food simulants and chemical alternative solvents: Prepared in accordance with the provisions of GB 5009.156.

3.2.2 Methanol solution: Measure 50 mL of methanol and 50 mL of water respectively, and mix well.

3.2 Standard product

3.3.1 1,2-benzisothiazolyl-3(2H)-one (C₇H₅NOS, CAS number: 2634-33-5): Purity ≥95%, or standard products certified by the country and awarded a reference material certificate.

3.3.2 2-methyl-3(2H)-isothiazolinone (C₄H₅NOS, CAS number: 2682-20-4): Purity ≥95%, or standard products certified by the country and awarded a reference material certificate.

3.3.3 5-chloro-2-methyl-2H-isothiazol-3-one (C₄H₄ClNOS, CAS number: 26172-55-4): Purity ≥95%, or standard products certified by the country and awarded a reference material certificate.

3.4 Preparation of standard solution

3.4.1 Isothiazolinone standard stock solution (1000 mg/L, solvent is methanol): Accurately weigh 25 mg of each of the three isothiazolinone standard products (accurate to 0.1 mg). Dissolve in methanol and transfer to the same 25 mL brown volumetric flask. Dilute to volume with methanol and shake well. Transfer the solution to a brown glass bottle. Store in a 4°C refrigerator, protected from light, and sealed. Shelf life is 6 months.

3.4.2 Isothiazolinone standard intermediate solution A (100 mg/L, solvent is methanol): Accurately transfer 1 mL of isothiazolinone standard stock solution into a 10 mL brown volumetric flask, dilute to volume with methanol and shake well. Transfer the solution to a brown glass container and store it airtight in a refrigerator at 4°C away from light. The storage period is 3 months.

3.4.3 Isothiazolinone standard intermediate solution B (10 mg/L, solvent is methanol): Accurately transfer 1 mL of isothiazolinone standard intermediate solution A into a 10 mL brown volumetric flask, dilute to volume with methanol and shake well. Transfer the solution to a brown glass container and store it in an airtight container away from light in a refrigerator at 4°C. The storage period is 3 months.

3.4.4 Isothiazolinone standard intermediate solution C (100 mg/L, solvent is tetrahydrofuran): Accurately pipette 1 mL of each of the three isothiazolinone standard stock solutions into a 10 mL brown volumetric flask. Dilute to volume with tetrahydrofuran and shake well. Transfer the solution to a brown glass container. Store in a 4°C refrigerator, protected from light, and sealed. The shelf life is 3 months.

3.4.5 Isothiazolinone standard intermediate solution D (10 mg/L, solvent is tetrahydrofuran): Accurately transfer 1 mL of isothiazolinone standard intermediate solution C into a 10 mL brown volumetric flask. Dilute to volume with tetrahydrofuran and shake well. Transfer the solution to a brown glass container. Store in a 4°C refrigerator, protected from light, and sealed. The shelf life is 3 months.

3.4.6 The standard working solution is prepared as follows:

- a) Standard working solutions for aqueous, acidic, ethanol-containing food simulants: Accurately pipette 30 μ L, 50 μ L, 100 μ L, 300 μ L, and 500 μ L of standard intermediate solution A into five 10 mL volumetric flasks. Bring to volume with water. Standard working solutions with isothiazolinone mass concentrations of 0.30 mg/L, 0.50 mg/L, 1.0 mg/L, 3.0 mg/L, and 5.0 mg/L are obtained. In the same way, 4% (volume fraction) acetic acid, 10% (volume fraction) ethanol, 20% (volume fraction) ethanol, 50% (volume fraction) ethanol food simulant and chemical alternative solvent 95% (volume fraction) are used. Use ethanol to prepare isothiazolinone standard working solutions of the same concentration series. Filter with a needle nylon filter and wait for measurement on the machine.
- b) Iso-octane standard working solution: Accurately pipette 30 μ L, 50 μ L, 100 μ L, 300 μ L, and 500 μ L of standard intermediate solution C into five 10 mL volumetric flasks. Dilute to volume with isooctane to obtain standard working solutions with isothiazolinone mass concentrations of 0.30 mg/L, 0.50 mg/L, 1.0 mg/L, 3.0 mg/L, and 5.0 mg/L, respectively. Pipette 2 mL of this series of standard working solutions into 5 stoppered glass centrifuge tubes. Add 2 mL of methanol solution (3.2.2) to each. Vortex for 1 min. After standing for separation, remove the methanol solution from the layer. Filter with a needle nylon filter and wait for measurement on the machine.
- c) Standard working solution of oil-containing food simulants: Accurately weigh 5.00 g (accurate to 0.01 g) of oil-containing food simulant olive oil into 5 stoppered glass centrifuge tubes. Then add 15 μ L, 25 μ L, 50 μ L, 150 μ L, and 250 μ L of standard intermediate solution C, respectively. Vortex and mix to obtain standard working solutions with isothiazolinone mass concentrations of 0.30 mg/kg, 0.50 mg/kg, 1.0 mg/kg, 3.0 mg/kg, and 5.0 mg/kg, respectively. Add 3 mL of n-hexane to each glass centrifuge tube. Then add 5 mL of methanol solution (3.2.2) each. Vortex for 1 min. After standing for separation, remove the methanol solution from the layer. Filter with a needle nylon filter and wait for measurement on the machine.

5.2.4 Preparation of blank test solution

Take food simulants and chemical alternative solvents that are not in contact with food contact materials and products. Follow the steps described in 5.1 and 5.2.1~5.2.3, respectively. Prepare a blank test solution and wait for measurement on the machine.

5.3 Instrument reference conditions

5.3.1 Chromatographic column: C₁₈ column that can withstand high water phase; column length 150 mm × column inner diameter 3.0 mm × stationary phase particle size 3.5 μm, or a chromatographic column with equivalent performance.

5.3.2 Mobile phase: A is water, B is methanol, A+B=8+2.

5.3.3 Flow rate: 1 mL/min.

5.3.4 Column temperature: 40°C.

5.3.5 Injection volume: 5 μL.

5.3.6 Detection wavelength: for 2-methyl-3(2H)-isothiazolinone and 5-chloro-2-methyl-2H-isothiazolin-3-one it is 275 nm; for 1,2-benzisothiazole Base-3(2H)-one it is 224 nm.

5.4 Drawing of standard working curve

According to the instrument reference conditions listed in 5.3, inject the standard series of working solutions into the high-performance liquid chromatograph in order from low to high concentration. Determine the peak area corresponding to each target substance to be measured. Draw a standard working curve with the concentration of isothiazolinone as the abscissa and the corresponding peak area as the ordinate. Obtain the linear regression equation. See Annex A for the chromatogram of the standard working solution.

5.5 Determination of specimen solution

5.5.1 Qualitative determination

According to the instrument reference conditions listed in 5.3, measure the specimen solution and standard working solution, respectively. If the retention time deviation of the corresponding chromatographic peak in the specimen solution and the standard solution is within ±2.5%, it can be judged that the corresponding analyte exists in the sample.

5.5.2 Quantitative determination

According to the instrument reference conditions listed in 5.3, inject the specimen solution and blank test solution into the high-performance liquid chromatograph,

X_3 - specific migration amount of isothiazolinone, in milligrams per piece (mg/piece);

c - the content of isothiazolinone in the specimen solution, in milligrams per liter (mg/L) or milligrams per kilogram (mg/kg);

V - the volume or mass of the sample immersion solution, in liters (L) or kilograms (kg);

c_0 - the content of isothiazolinone in the blank test solution, in milligrams per liter (mg/L) or milligrams per kilogram (mg/kg);

n - quantity of sealing products used during immersion, in pieces.

The result must contain at least 2 significant figures.

7 Precision

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

8 Other

When the S/V in the migration test is the same as the S/V of the sample in actual use, the detection limit of this method for the migration of a single isothiazolinone in food simulants and chemical alternative solvents is 0.10 mg/kg; the limit of quantification is 0.30 mg/kg. When the S/V in the migration test is different from the S/V of the sample in actual use, the detection limit and quantitation limit of the migration amount of a single isothiazolinone in food simulants and chemical alternative solvents in this method need to be converted according to the calculation formula in Chapter 6.

Method Two -- Liquid Chromatography-tandem Mass

Spectrometry

9 Principle

Food contact materials and products are tested using liquid chromatography-tandem mass spectrometry after migration tests in accordance with GB 31604.1 and GB 5009.156. Among them, the samples of water, 4% (volume fraction) acetic acid, 10% (volume fraction) ethanol, 20% (volume fraction) ethanol, 50% (volume fraction) ethanol food simulant and chemical alternative solvent 95% (volume fraction) ethanol soaking solution are injected directly after filtration. The soaking solution of oil-

10.4.4 Isothiazolinone standard intermediate solution C (100 mg/L, solvent is tetrahydrofuran): Same as 3.4.4.

10.4.5 Isothiazolinone standard intermediate solution D (10 mg/L, solvent is tetrahydrofuran): Same as 3.4.5.

10.4.6 The standard working solution is prepared as follows:

- a) Standard working solutions for aqueous, acidic, ethanol-containing food simulants: Accurately pipette 30 μL , 50 μL , 100 μL , 300 μL , and 500 μL of standard intermediate solution B into five 10 mL volumetric flasks. Bring to volume with water. Standard working solutions with isothiazolinone mass concentrations of 0.030 mg/L, 0.050 mg/L, 0.10 mg/L, 0.30 mg/L, and 0.50 mg/L are obtained. In the same way, 4% (volume fraction) acetic acid, 10% (volume fraction) ethanol, 20% (volume fraction) ethanol, 50% (volume fraction) ethanol food simulant and chemical alternative solvent 95% (volume fraction) are used. Use ethanol to prepare isothiazolinone standard working solutions of the same concentration series. Filter with a needle nylon filter and wait for measurement on the machine.
- b) Iso-octane standard working solution: Accurately pipette 30 μL , 50 μL , 100 μL , 300 μL , and 500 μL of standard intermediate solution D into five 10 mL volumetric flasks. Bring to volume with isooctane. The standard solutions with isothiazolinone mass concentration of 0.030 mg/L, 0.050 mg/L, 0.10 mg/L, 0.30 mg/L, and 0.50 mg/L are obtained, respectively. Pipette 2 mL of the standard working solution into 5 stoppered glass centrifuge tubes. Add 2 mL of methanol solution (10.2.2) to each. Vortex for 1 min. After standing for separation, remove the methanol solution from the layer. Filter with a needle nylon filter and wait for measurement on the machine.
- c) Standard working solution of oil-containing food simulants: Accurately weigh 5.00 g (accurate to 0.01 g) of oil-containing food simulant (olive oil) into 5 stoppered glass centrifuge tubes. Then add 15 μL , 25 μL , 50 μL , 150 μL , and 250 μL of standard intermediate solution D, respectively. Vortex to mix. Standard working solutions with isothiazolinone mass concentrations of 0.030 mg/kg, 0.050 mg/kg, 0.10 mg/kg, 0.30 mg/kg, and 0.50 mg/kg are obtained. Add 3 mL of n-hexane to each glass centrifuge tube. Add another 5 mL of methanol solution (10.2.2). Vortex for 1 min. After standing for separation, remove the methanol solution from the layer. Filter with a needle nylon filter and wait for measurement on the machine.

11 Instruments and equipment

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

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