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NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA

GB 31604.52-2021

**National food safety standard - Food contact material
and products - Determination of migration of primary
aromatic amine**

食品安全国家标准 食品接触材料及制品 芳香族伯胺迁移量的测定

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National food safety standard - Food contact material and products - Determination of migration of primary aromatic amine

1 Scope

This standard specifies the method for determining the migration of 29 aromatic primary amines, in food contact materials and products.

This standard applies to the determination of the migration of aromatic primary amines, in food contact materials and products.

2 Principles

After the migration test of food contact materials and products, according to GB 31604.1 and GB 5009.156, they are detected by liquid chromatography-mass spectrometry/mass spectrometry. Among them, the food simulants, including 4% acetic acid, 10% ethanol, 20% ethanol, 50% ethanol, AND the alternative chemical solvents, that is, 95% ethanol, are filtered and injected directly. The alternative chemical solvent isooctane is extracted by methanol-water solution; then the water layer is filtered and injected. Use the peak area external standard method for quantification.

3 Reagents and materials

The water is the grade 1 water, which is specified in GB/T 6682.

3.1 Reagents

3.1.1 Acidic and alcoholic food simulants and alternative chemical solvents: The reagents used are in accordance with the provisions of GB 5009.156.

3.1.2 Methanol (CH₃OH): Chromatographically pure.

3.1.3 Formic acid (HCOOH): Chromatographically pure.

3.1.4 Acetonitrile (CH₃CN): Chromatographically pure.

3.1.5 Glacial acetic acid (CH₃COOH): Excellent grade pure.

3.1.6 Isooctane (C₈H₁₈): Chromatographically pure.

of isooctane standard working solution into a test tube. Add 2 mL of methanol-water extract (3.2.3). Shake it for 30 s. Let it stand for stratification. Take the lower layer of aqueous solution. Use a nylon filter to filter it, to prepare for determination.

3.4.4.2 Standard working solutions for other food simulants and alternative chemical solvents

Accurately pipette 30 μL , 50 μL , 100 μL , 300 μL , 500 μL of the standard intermediate solution A (3.4.2) of aromatic primary amine, in a 10 mL volumetric flask. Use 10% ethanol to make its volume reach to the mark, to obtain the standard working solution, which has an aromatic primary amine concentration of 3.0 $\mu\text{g/L}$, 5.0 $\mu\text{g/L}$, 10.0 $\mu\text{g/L}$, 30.0 $\mu\text{g/L}$, 50.0 $\mu\text{g/L}$, respectively. In the same way, use the 4% acetic acid, 20% ethanol, 50% ethanol food simulant, alternative chemical solvent (95% ethanol), to prepare the standard working solutions of the aromatic primary amine, in the same concentration series.

4 Instruments and equipment

4.1 Liquid chromatography tandem mass spectrometer: Electrospray ion source (ESI)

4.2 Analytical balance: Sensitivity is 0.0001 g.

4.3 Micro syringe: 10 μL , 100 μL , 1000 μL .

4.4 Constant temperature drying oven or constant temperature water bath device.

4.5 Vortex mixer.

4.6 Needle type nylon filter: Pore size 0.22 μm .

5 Analytical procedures

5.1 Preparation of food simulants

5.1.1 General

The migration test of this standard uses 4% acetic acid, 10% ethanol, 20% ethanol, 50% ethanol food simulants, alternative chemical solvent (95% ethanol), isooctane. Vegetable oil is not suitable for the determination of the migration of aromatic primary amines.

5.1.2 Migration test

test, in liters (L);

S - The area of contact between the sample and the soaking solution in the migration test, in square decimeters (dm²);

S₀ - The area of the sample in contact with the food in actual use, in square decimeters (dm²);

m₁ - The mass of the sample, which is actually contacted with solid food, OR the mass of the food corresponding to the volume in the actual contact with liquid food, in kilograms (kg); the volume of various liquid foods is converted to the corresponding mass, at a density of 1 kg/L.

The result retains at least 2 significant digits.

6.2 Calculation of specific migration of single aromatic primary amine in food contact materials and products of sealed products (expressed in mg/kg)

When the intended use is known, AND the specific migration of a single aromatic primary amine is expressed in mg/kg, for food contact materials and products of sealed products, it is calculated according to formula (2).

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

Where:

X₂ - The specific migration of a single aromatic primary amine, in milligrams per kilogram (mg/kg);

S₀ - The area in contact with food in actual use of the sealed product, in square decimeters (dm²);

m₂ - The mass of the solid food in the container, which is actually used by the sealed product, OR the mass of the food corresponding to the volume in actual contact with the liquid food, in kilograms (kg); the volume of various liquid foods is converted to the corresponding mass, according at the density of 1 kg/L.

The result retains at least 2 significant digits.

6.3 Calculation of specific migration of single aromatic primary amine in food contact materials and products of sealed products (expressed in mg/piece)

When the intended use is unknown, AND the specific migration of a single aromatic primary amine in the food contact material and product of the sealed

product is expressed in mg/piece, it is calculated according to formula (3). It needs indicating the migration test method, the contact area of a single sealed product with the food simulant in the migration test.

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

Where:

X_3 - Specific migration of a single aromatic primary amine, in milligrams per piece (mg/piece);

n - The number of sealed products for immersion, in pieces.

The result retains at least 2 significant digits.

6.4 Calculation of the total migration of aromatic primary amines

The total migration of aromatic primary amines is the sum of the migration of each primary aromatic amine. If a certain aromatic primary amine has no detection signal (that is, less than 3 times the background noise), it shall be recorded as "not detected" or "ND"; its value is treated as zero.

7 Precision

The absolute difference between two independent determination results, which are obtained under repeatability conditions, shall not exceed 10% of their arithmetic mean.

8 Others

This method has a detection limit of 1.0 µg/kg AND a quantification limit of 3.0 µg/kg, for a single aromatic primary amine in food simulants, including 4% acetic acid, 10% ethanol, 20% ethanol, 50% ethanol, alternative chemical solvents (95% ethanol), isooctane.

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