NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB 31604.52-2021

National food safety standard - Food-contact materials and articles - Determination of migration of primary aromatic amine

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Table of Contents

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.

3.2.1 Acidic and alcoholic food simulants and alternative chemical solvents.

Prepared according to GB 5009.156.

3.2.2 The 0.1% formic acid. Take 1 mL of formic acid (3.1.3) in a volumetric

flask; add water to make the volume reach to 1 L.

3.2.3 Methanol-water extract. Measure 50 mL of methanol (3.1.2) AND 50 mL

of water. Pipette 0.4 mL of glacial acetic acid (3.1.5). Mix it uniformly.

3.3 Standard substances

The 29 kinds of aromatic primary amines. Standard substances (see appendix A), purity >= 98%; or standard substances that have been certified by the country and awarded a standard material certificate.

3.4.1 The 29 kinds of standard stock solutions of aromatic primary amine (1000

mg/L). Accurately weigh 25 mg (accurate to 0.1 mg) of each of 29 kinds of aromatic primary amine standard substances. Use methanol to dissolve them, respectively. Transfer it into a 25 mL brown volumetric bottle. Use methanol to dilute it to the mark. Seal and store it in a 4 °C refrigerator in the dark. The shelf life is 1 month.

3.4.4.1 Isooctane standard working solution

Accurately pipette 30 μL, 50 μL, 100 μL, 300 μL, 500 μL of the standard intermediate solution B of aromatic primary amine (3.4.3), in a 10 mL volumetric flask. Use isooctane to make the volume reach to the mark, to obtain the standard working solution, which has an aromatic primary amine concentration of 3.0 μg/L, 5.0 μg/L, 10.0 μg/L, 30.0 μg/L, 50.0 μg/L, respectively.

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.

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

The condition selection and operation steps of the migration test are in accordance with the

provisions of GB 31406.1 and GB 5009.156.

5.1.3.3 Preparation of blank test solution

Food simulants and alternative chemical solvents, which are not in contact with food contact materials and products, are treated in accordance with 5.1.3.1 and 5.1.3.2, to obtain a blank test solution.

5.2.1 Liquid chromatography conditions

The liquid chromatography conditions are as follows.

5.2.2 Mass spectrometry conditions

The mass spectrometry conditions are as follows.

5.3 Drawing of standard curve

According to the instrument reference conditions listed in 5.2, the standard working solution is determined. Use the concentration of the aromatic primary amine in the standard working solution as the abscissa, the corresponding quantitative ion peak area as the ordinate, to draw the standard curve, to obtain the linear equation.

5.4.1 Qualitative determination

Determine the food simulant sample solution (5.1.3) and standard working solution, according to the instrument reference condition (5.2). If the deviation of the retention time of the mass chromatographic peak of the food simulant sample solution and the standard solution is within +/-2.5%;

5.4.2 Quantitative determination

Inject the test solution and the blank test solution (5.1.3) into the liquid chromatography-mass spectrometer/mass spectrometer, to obtain the peak area.

6.1 Calculation of the specific migration of a single aromatic primary

amine in food contact materials and products of non-sealed products (expressed in mg/kg)

6.2 Calculation of specific migration of single aromatic primary amine in

food contact materials and products of sealed products (expressed in mg/kg)

6.3 Calculation of specific migration of single aromatic primary amine in

food contact materials and products of sealed products (expressed in mg/piece)

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.