NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB 31604.50-2020

National food safety standard - Food contact materials and articles - Determination of nonylphenol migration

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1 Scope

This Standard specifies the liquid chromatography-mass spectrometry/mass spectrometry method for nonylphenol migration of food contact materials and articles.

This Standard is applicable to the determination of nonylphenol of food contact materials and products with water, 4% (volume fraction) acetic acid solution, 10% (volume fraction) ethanol solution, 20% (volume fraction) ethanol solution, 50% (volume fraction) ethanol solution, olive oil , 95% (volume fraction) ethanol solution as food simulant OR the determination of nonylphenol in the soaking solution obtained by using 95% (volume fraction) ethanol solution, isooctane chemical substitute solvent in a migration test.

2 Principle

Take 4% (volume fraction) acetic acid solution as food simulant. In the soaking solution obtained by migration test, add ammonia to neutralize then determine the injected sample. Use olive oil as a food simulant. The soaking solution obtained by migration test is extracted by acetonitrile and purified by n-hexane.

Then determine the injected sample. Use isooctane as a chemical alternative solvent. The soaking solution obtained by migration test is steamed to dry. Use methanol to reconstitute. Determine the injected sample. For soaking solutions obtained from other migration tests, soaking liquids for chemical substitute solvents, directly determine the injected samples. Nonylphenol in the soaking solution is determined by liquid chromatography-mass spectrometry/mass spectrometry. Quantitatively determine by external standard method.

3 Reagents and materials

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

3.1.1 Glacial acetic acid (C2H4O2).

(volume fraction) acetic acid solution to set volume to the graduation of scale.

Obtain standard working solutions of which the mass concentrations are 0.01mg/L, 0.02mg/L, 0.03mg/L, 0.05mg/L, 0.10mg/L, 0.20mg/L. Before getting on the machine, it needs to be processed synchronously with the food simulant soaking solution in accordance with 5.1.2.1.

3.4.3.2 Oil-based food simulant standard working solution

Respectively and accurately weigh 5.00g (to the nearest of 0.1mg) of olive oil into 6 stoppered glass centrifuge tubes. Respectively add 0.01mL, 0.02mL, 0.03mL, 0.05mL, 0.10mL, 0.20mL of standard intermediate solution (5mg/L).

Vortex to mix well. Obtain 0.01mg/kg, 0.02mg/kg, 0.03mg/kg, 0.05mg/kg, 0.10mg/kg, 0.20mg/kg standard working solutions. Before getting on the machine, it needs to be processed synchronously with the food simulant soaking solution in accordance with 5.1.2.2.

3.4.3.3 Isooctane standard working solution

Respectively and accurately pipette 0.02mL, 0.04mL, 0.06mL, 0.10mL, 0.20mL, 0.40mL of standard intermediate solution (5mg/L) into six 10mL volumetric flasks. Use isooctane to set volume to the graduation of scale. Mix well. Obtain standard working solutions of which the mass concentrations are 0.01mg/L, 0.02mg/L, 0.03mg/L, 0.05mg/L, 0.10mg/L, 0.20mg/L. Before getting on the machine, it needs to be processed synchronously with the food simulant soaking solution in accordance with 5.1.2.3.

3.4.3.4 Standard working solutions of other food simulants, chemical

alternative solvents Respectively pipette 0.02mL, 0.04mL, 0.06mL, 0.10mL, 0.20mL, 0.40mL of standard intermediate solution (5mg/L) into six 10mL volumetric flasks. Use corresponding food simulants or chemical alternative solvents to set volume to the graduation of scale. Obtain other water-based food simulants or chemical alternative solvent standard working solutions of which the mass concentrations are 0.01mg/L, 0.02mg/L, 0.03mg/L, 0.05mg/L, 0.10mg/L, 0.20mg/L.

4.1 Liquid chromatography tandem triple quadrupole mass spectrometer:

equipped with electrospray ion source (ESI).

5.2.1 Liquid chromatography conditions

The reference conditions of liquid chromatography are as follows:

a) Chromatographic column: C18 column, 2.6um, 2.1mmx150mm (or equivalent chromatographic column).

b) Mobile phase: methanol-water (90+10, volume ratio).

c) Flow rate: 0.3mL/min.

d) Column temperature: 40°C.

e) Injection volume: 2uL.

5.2.2 Mass spectrometry conditions

The mass spectrometry reference conditions are as follows:

a) Ionization mode: electrospray ionization negative ion mode (ESI-).

b) Mass spectrometry scan mode: multiple reaction monitoring (MRM).

c) For other mass spectrometer parameters, refer to Table A.1 in Annex A.

5.3 Drawing of standard curve

According to the instrument reference conditions listed in 5.2, determine the standard working solution. Take the concentration of nonylphenol in the standard working solution as the abscissa, the corresponding quantitative ion peak area as the ordinate to draw the standard curve and obtain linear equation.

Refer to Annex B for the chromatogram of the nonylphenol standard working solution.

5.4.1 qualitative determination

According to the instrument reference conditions listed in 5.2, determine the food simulant test solution and standard working solution. If the deviation of the retention time of the mass chromatographic peak of the analyte in the test solution and the standard solution is within +/-2.5%, the signal-to-noise ratio of all qualifier ions exceeds 3:1; the relative abundance of the qualitative ion pair is consistent with the relative abundance of the standard solution of equivalent concentration. When the relative abundance deviation does not exceed the provisions of Table 1, it can be judged that the corresponding analyte exists in and articles to the volume (V) of food or food simulants; the density of various liquid foods is usually calculated as 1kg/L, in square decimeters per kilogram (dm2/kg). When S/V is known in actual use, F is the actual S/V. When S/V cannot be estimated, F uses 6dm2/kg, that is,

6dm2 of food contact materials and articles contact 1kg of food or food simulants.

The result retains at least 2 significant digits.

6.2 Calculation of specific nonylphenol migration for food contact

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

When the intended use is unknown, when the specific nonylphenol migration for food contact materials and articels of sealed products is expressed in mg/piece, it is calculated according to formula (2). The migration test method used, the contact area of a single sealed product and the food simulant in the migration test shall be indicated.

Where, X2 - Specific nonylphenol migration, in milligrams per piece (mg/piece);

n - Quantity of sealing products for immersion, in pieces.

The result retains at least 2 significant digits.

7 Precision

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

8 Others

When the S/V in the migration test is the same as the S/V in the actual use situation, the detection limit of this method for nonylphenol in various food simulants and chemical alternative solvents is 0.005mg/kg; the limit of quantification is 0.01mg/kg.