NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB 31604.53-2022

National food safety standard - Food contact materials and articles - Determination of migration of 5-Ethylidene-2-norbornene, mixt. of endo- and exo-isomers

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1 Scope

This document specifies the method for the determination of the migration of 5-Ethylidene-2-norbornene in food contact materials and products.

This document applies to the determination of the migration of 5-Ethylidene-2-norbornene in plastics, rubber, and coated food contact materials and products.

2 Principle

After the food contact materials and products are subjected to migration test in accordance with the provisions of GB 31604.1 and GB 5009.156, conduct headspace injection for 5-Ethylidene-2-norbornene in 4% (volume fraction) acetic acid, 10% (volume fraction) ethanol, 20% (volume fraction) ethanol, 50% (volume fraction)

ethanol and olive oil food simulants. Conduct liquid injection for chemical alternative solvent 95% (volume fraction) ethanol and isooctane. Use gas chromatography-mass spectrometry for determination. Use peak area external standard method for quantification.

3 Reagents and materials

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

3.2 Reagent preparation

Acidic and ethanol-containing food simulants. be prepared in accordance with the provisions of GB 5009.156.

3.3 Standard product

5-Ethylidene-2-norbornene (C9H12, CAS No.. 16219-75-3). purity is >=98%, or the standard product certified by the state and granted the certificate of reference material.

3.4 Preparation of standard solution

3.4.1 5-Ethylidene-2-norbornene standard stock solution (1000mg/L)

Accurately weigh 25mg of 5-Ethylidene-2-norbornene standard product (accurate to 0.1mg). Use n-hexane to dissolve. Transfer to a 25mL brown volumetric flask. Use n-hexane to set volume to the scale. Mix well. Transfer the solution to a brown glass container. Store in the dark at 0°C~4°C. Shelf life is 3 months.

3.4.2 5-Ethylidene-2-norbornene standard intermediate solution (100mg/L)

Pipette 2.50mL of 5-Ethylidene-2-norbornene standard stock solution (1000mg/L) in a 25mL brown volumetric flask. Add n-hexane to set volume to the scale. Mix well.

Transfer the solution to a brown glass container. Store in the dark at 0°C~4°C. Shelf life is 3 months.

3.4.3 5-Ethylidene-2-norbornene standard intermediate solution A Pipette 0.15mL, 0.30mL, 0.50mL, 1.50mL and 3.00mL of 5-Ethylidene-2-norbornene standard intermediate solution (100mg/L) respectively into 5mL volumetric flasks. Use tetrahydrofuran to set volume and shake well. At this time, the concentrations of the series of standard intermediate solutions are respectively equivalent to 3.0mg/L, 6.0mg/L, 10mg/L, 30mg/L and 60mg/L. Transfer the solutions to brown glass containers. Store in the dark at 0°C~4°C. Shelf life is 1 month.

3.4.4 5-Ethylidene-2-norbornene standard intermediate solution B Pipette 0.15mL, 0.30mL, 0.50mL, 1.50mL and 3.00mL of 5-Ethylidene-2-norbornene standard intermediate solution (100mg/L) respectively into 5mL volumetric flasks. Use n-hexane to set volume and shake well. At this time, the concentration of the series of standard intermediate solutions is equivalent to 3.0mg/L, 6.0mg/L, 10mg/L, 30mg/L and 60mg/L, respectively. Transfer the solutions to brown glass containers. Store in the dark at 0°C~4°C. Shelf life is 1 month.

3.4.5 Standard working solution of acidic and ethanol-containing food simulants

test is carried out, the soaking solution shall be returned to room temperature before use.

5.1.2 Pretreatment of soaking solution obtained from migration test

5.1.2.1 Soaking solution of acidic and ethanol-containing food simulants Accurately pipette 5mL of soaking solution of acid and ethanol-containing food simulants obtained after the migration test in a 20mL headspace vial. Immediately cap and seal for testing.

5.1.2.2 Soaking solution of oil-containing food simulants Accurately weigh 5g of soaking

solution of oil-containing food simulant obtained after the migration test in a 20mL headspace vial. Immediately cap and seal for testing.

5.1.2.3 Soaking solution of chemical alternative solvent Pipette about 1mL of soaking solution of chemical alternative solvent obtained after the migration test. Use 0.45um filter to filter into a small injection bottle. Immediately cap and seal for testing.

5.1.3 Blank test Food simulants and chemical alternative solvents not in contact with food contact materials and products are respectively processes according to 5.1.2.1~5.1.2.3 to obtain the blank test solutions.

5.2 Instrument reference conditions

5.2.1 Instrumental reference conditions for acidic, ethanol-containing and oil-containing food simulants

5.2.1.1 Headspace injector reference conditions (for acidic, ethanol-containing food simulants)

a) Equilibrium temperature. 90°C.

b) Equilibration time. 30min.

c) Quantitative ring temperature. 110°C.

d) Transfer line temperature. 120°C.

5.2.1.2 Headspace injector reference conditions (for oil-containing food simulants)

a) Equilibrium temperature. 145°C.

b) Equilibration time. 60min.

c) Quantitative ring temperature. 150°C.

d) Transfer line temperature. 160°C.

5.2.1.3 Reference conditions for gas chromatography mass spectrometer determination a) Chromatographic column. 5% phenyl-methyl polysiloxane quartz capillary column. Column length is 30m. Internal diameter is 0.25mm. Film thickness is 0.25μm. Or the equivalent.

b) Program temperature. Initial temperature is 35°C. Maintain for 5min. Raise to 280°C at 30°C/min. Maintain for 3min.

c) Inlet temperature. 280°C.

d) Chromatography-MS interface temperature. 280°C.

e) Ion source temperature. 230°C.

f) Quadrupole temperature. 150°C.

g) Carrier gas. He (purity >=99.999%), 1mL/min, constant flow mode.

h) Injection volume. 1mL.

i) Injection method. Split injection. Split ratio 5.1 [for 4% (volume fraction) acetic acid, 10% (volume fraction) ethanol and 20% (volume fraction) ethanol] or 2.1 [for 50% (volume fraction) ethanol and olive oil].

j) Ionization method. Electron bombardment ionization source (EI).

k) Solvent delay. 4min.

l) Mass spectrometer scanning mode. Selected ion scanning (SIM). Quantitative ions m/z 66.Qualitative ions m/z 91, m/z 105, m/z 120.

5.2.2 Instrument reference conditions for chemical alternative solvents 95% (volume fraction) ethanol and isooctane a) Chromatographic column. 5% phenyl-methyl polysiloxane quartz capillary column. Column length is 30m. Internal diameter is 0.25mm. Film thickness is 0.25μm. Or the equivalent.

b) Program temperature. Initial temperature is 35°C. Maintain for 5min. Raise to 280°C at 30°C/min. Maintain for 3min.

c) Inlet temperature. 280°C.

oil-containing simulants. it is the mass of the specimen soaking solution in the migration test, in kilograms (kg);

S1 - The contact area between the specimen and the soaking solution in the migration test, in square decimeters (dm2);

S2 - The area of the sample in contact with food in actual use, in square decimeters (dm2);

m - The mass of solid food contacted by the sample in actual use or the mass of food corresponding to the volume actually in contact with the liquid food, in kilograms (kg).

The volume of various liquid foods is usually converted into the corresponding mass according to the density of 1 kg/L.

NOTE. In the actual use of the sample, when the area/volume ratio contacting with food is unknown, "S2/m" in formula (1) is taken as 6dm2/kg.

The calculation result must have at least two significant figures.

7 Precision The absolute difference between the results of two independent determinations obtained under repeatability conditions shall not exceed 20% of their arithmetic mean.

8 Others When the area/volume ratio of the sample in the migration test is consistent with the area/volume ratio of the sample in contact with food in actual use, for aqueous, acidic, ethanol-containing food simulants, the detection limit of this method is 0.001mg/kg, and the quantification limit is 0.003mg/kg. For oil-containing food simulants and chemical alternative solvents, the detection limit of this method is 0.01mg/kg, and the quantification limit is