GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB 31604.20-2016

National food safety standard - Food contact materials and products - Determination of vinyl acetate migration quantity

Issued on: October19, 2016 **Implemented on: April 19, 2017**

Issued by: National Health and Family Planning Commission of the PRC

Table of Contents

National food safety standard - Food contact materials and products - Determination of vinyl acetate migration quantity

1 Scope

This standard specifies determination method of vinyl acetate migration quantity in food contact materials and products.

This standard applies to determination of vinyl acetate migration quantity in food contact materials and products.

2 Principles

In the sample, after the vinyl acetate is leached from the food simulation soaking solution, use acetone to dilute it, and finally use gas chromatography to determine and external standard method to quantify. Once the sample is detected, it must be confirmed by gas chromatography - mass spectrometry.

3 Reagents and materials

Unless otherwise indicated, the reagents used in this method are analytical grade, the water is the grade-1 water specified in GB/T 6682. The containers and transfer apparatuses in the test shall avoid using plastic materials.

3.1 Reagents

3.1.1 Acetone (C_3H_6O): It does not contain vinyl acetate.

3.1.2 Methanol (C_2H_4O).

3.1.3 Water-based, acidic, alcoholic, oil-based food simulants: The reagents used are in accordance with the provisions of GB 31604.1.

3.2 Reagent preparation

3.2.1 Water-based, acidic, alcoholic, oil-based food simulants: Operate according to GB 5009.156.

5.2 Preparation of standard working solution of food simulants that are water-based, acidic food, alcoholic, and oil-based

Accurately PIPETTE 0 mL, 0.01 mL, 0.05 mL, 0.1 mL, 0.5 mL, 1.0 mL of vinyl acetate standard intermediate solution into 10 mL volumetric flask; USE acetone to dilute it; OBTAIN standard working solution with vinyl acetate concentration of 0.00 mg/L, 0.1 mg/L, L, 0.5 mg/L, 1.0 mg/L, 5.0 mg/L, 10.0 mg/L, respectively. In the same way, OBTAIN standard working solution of food simulants that are water-based, acidic food, alcoholic, and oil-based.

5.3 Gas chromatograph (GC) reference conditions

The GC reference conditions are listed below:

a) Chromatographic column: DB-5 quartz capillary column, column length 30 m, inner diameter 0.32 mm, film thickness 0.25 μm, or equivalent;

b) Temperature programming: 35 °C (keep for 5 min), rising from 5 °C/min to 50 °C, rising from 40 °C/min to 250 °C (keep for 2 min);

c) Inlet temperature: 250 °C;

d) Injection mode: split injection (10:1), injection volume is 1 μL;

e) Detector temperature: 300 °C;

f) Carrier gas (N_2) flow rate: 0.5 mL/min;

g) Hydrogen flow rate: 30 mL/min;

h) Air flow rate: 400 mL/min.

5.4 Determination

5.4.1 Sample determination

TAKE a clean and dry 25 mL volumetric flask; ADD 1 g (accurate to 0.0001 g) of simulated food soaking solution after migration experiment; USE acetone to dilute it to the mark; MIX it uniformly for spare-use. USE micro-syringe to pipette respectively a certain amount of the above test solution; INJECT it into gas chromatograph. According to the above gas chromatographic conditions, REPEAT the determination twice. The gas chromatogram of vinyl acetate is shown in Figure A.1.

5.4.2 Blank Test

Blank test refers to, except for not adding sample, use exactly the same analytical procedure, reagents and dosage, carry out parallel operation.

Appendix B

Gas chromatography-mass spectrometry confirmation reference conditions

B.1 Gas chromatographic conditions

The gas chromatographic conditions are listed below:

a) Chromatographic column: Capillary column: DB-5 ms 30 m × 0.25 mm (inner diameter) × 0.25 μm, or equivalent;

b) Temperature programming: 30 °C (keep for 4 min), rising from 5 °C/min to 50 °C, rising from 40 °C/min to 250 °C (keep for 2 min);

c) Inlet temperature: 250 °C;

d) GC-MS interface temperature: 280 °C;

e) Carrier gas (He): 0.55 mL/min;

f) Injection mode: split injection, split ratio is 10:1.

B.2 Mass Spectrometry Conditions

The mass spectrometry conditions are listed below:

a) Ion source temperature: 230 °C;

b) Quadrupole rod temperature: 150 °C;

c) Ion source: EI source, ionization energy 70 eV;

d) Quantitative ion: 43 m/z;

e) Qualitative ion: 86,44 m/z.

Note: For different mass spectrometers, the instrument parameters may be different. Before the determination, the mass spectrometry parameters shall be optimized to the best.

B.3 Qualitative determination

When determining the sample, dilute the sample solution appropriately, and determine the sample solution and standard working solution according to gas chromatography-mass spectrometry conditions. If it is confirmed that the

This is an excerpt of the PDF (Some pages are marked off intentionally)

------ The End ------