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NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA

GB 31604.31-2016

**National Food Safety Standard – Food Contact
Materials and Articles – Determination of the
Vinyl Chloride and Determination of Migration**

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National Food Safety Standard – Food Contact Materials and Articles – Determination of the Vinyl Chloride and Determination of Migration

1 Scope

This Standard specifies the determination of the vinyl chloride and determination of migration in plastic and products for food contact materials.

This Standard is applicable to the determination of vinyl chloride and determination of migration in polyvinyl chloride and vinyl chloride copolymers.

This Standard is also applicable to the determination of 1,1-dichloroethane in polyvinyl chloride and polyvinyl chloride copolymers.

Determination of Vinyl Chloride

2 Principle

Place the specimen into the sealed balance bottle; use N,N-Dimethylacetamide to dissolve. At certain temperature, vinyl chloride is diffused; when the gas-liquid equilibrium is reached, take gas above the liquid to inject into the gas chromatograph; use hydrogen flame ionization detector to measure, use external standard method to quantify.

3 Reagents and Materials

Unless otherwise is specified, all reagents shall be analytically pure.

3.1 Reagents

N,N-Dimethylacetamide: purity is greater than 99%.

3.2 Standard substance

Vinyl chloride reference solution: 5000mg/L; take acetone or methanol as solvent.

3.3 Preparation of standard solution

- b) Equilibrium temperature: 70°C;
- c) Quantitative loop temperature: 90°C;
- d) Transmission line temperature: 120°C;
- e) Balance time: 30min;
- f) Pressurization time: 0.20min;
- g) Filling time of quantitative loop: 0.10min;
- h) Equilibration time of quantitative loop: 0.10min;
- i) Sample injection time: 1.50min.

NOTE: if the manual headspace sampling injection is performed, the reference method can refer to Appendix A.

5.2.1.2 Chromatographic conditions

- a) Chromatographic column: polyethylene glycol capillary chromatographic column with length of 30m, inner diameter of 0.32mm, film thickness of 1μm, or equivalent column;
- b) Column temperature program: maintain the initial temperature of 40°C for 1min; raise the temperature to 60°C at the speed rate of 2°C/min, maintain for 1min; raise the temperature to 200°C at the speed rate of 20°C [Translator note: here it should be 20°C/min], maintain for 1min;
- c) Carrier gas: nitrogen, flow rate of 1mL/min;
- d) Sample injecting mode: split, split ratio of 1:1;
- e) Sample injecting temperature: 200°C;
- f) Detector temperature: 200°C.

5.2.2 Drawing of standard working curve

The standard working solution prepared in 3.3.2 was tested under the instrumental parameters listed in 5.2; take the mass concentration of vinyl chloride standard working solution as abscissa; take the corresponding peak area as the ordinate; then draw the standard working curve; obtain the linear equation. Standard solution chromatogram is shown in Appendix B.

5.2.3 Quantitative determination

10 Reagents and Materials

Unless otherwise is specified, all reagents shall be analytically pure.

10.1 Reagents

10.1.1 Water-based, acidic, alcoholic, oil-fat food simulants: as per the provisions of GB 31604.1.

10.1.2 N,N-Dimethylacetamide: purity greater than 99%.

10.2 Preparation of reagents

Water-based, acidic, alcoholic, oil-fat food simulants: prepare as per GB 5009.156.

10.3 Standard substance

Vinyl chloride reference solution: 5000mg/L; take acetone or methanol as solvents.

10.4 Preparation of standard solution

10.4.1 Vinyl chloride stock solution: separately add 10mL of N,N-Dimethylacetamide into 6 pieces of 10mL glass bottles; successively add 5 μ L, 10 μ L, 20 μ L, 30 μ L, 40 μ L, 50 μ L of vinyl chloride into each bottle; mix evenly; then the concentrations of the obtained vinyl chloride are 2.5mg/L, 5.0mg/L, 10.0mg/L, 15.0mg/L, 20.0mg/L, 25.0mg/L. Store into 4°C refrigerator.

10.4.2 Water-based food simulant standard working solution: separately add 10mL of water into 6 headspace bottles; successively add 20mL of vinyl chloride stock solution with different concentrations into each headspace bottles; immediately use cap to seal; mix evenly; then the concentrations of the obtained vinyl chloride in water are 0.0050mg/L, 0.010mg/L, 0.020mg/L, 0.030mg/L, 0.040mg/L and 0.050mg/L. Adopting the same modes, separately use acidic, alcoholic food simulants to prepare the vinyl chloride serial standard working solution with the same concentration.

10.4.3 Oil-fat food simulant standard working solution: respectively take 10g (accurate to 0.1g) of oil-fat food simulants into 6 headspace bottles; successively add 20 μ L of vinyl chloride stock solution with different concentrations into each headspace bottle; immediately use the cap to seal; mix evenly; the concentrations of the obtained vinyl chloride in oil-fat food simulants are sequentially 0.0050mg/kg, 0.010mg/kg, 0.020mg/kg, 0.030mg/kg, 0.040mg/kg and 0.050mg/kg.

NOTE: before preparing the standard solution, food simulant needs to be fully cooled in the 4°C refrigerator; the standard solution shall be prepared quickly.

12.3 Determination

12.3.1 Instrument reference condition

The same as 5.2.

12.3.2 Drawing of standard working curve

The standard working solution is tested as per the measurement conditions listed in 12.3.1. Take the concentration of vinyl chloride in the food simulant standard working solution as the abscissa; while take the corresponding peak area as the ordinate; then draw the standard working curve.

12.3.3 Quantitative determination

Sequentially inject the samples of food simulant test solution and blank test solution; obtain the peak area of vinyl chloride chromatogram; deduct the blank value during the calculating process. According to the peak area of vinyl chloride chromatogram, calculate the concentration of vinyl chloride in sample solution by standard curve.

13 Expression of Analytical Results

The concentration of vinyl chloride in the sample solution is obtained from standard curve; perform migration calculation as per GB 5009.156; obtain the migration of vinyl chloride in the food contact materials and articles. The calculation results shall be retained 2 significant figures.

14 Precision

The absolute difference of two independent tests obtained under repeatability conditions must not exceed 10% of the arithmetic mean.

15 Others

The detection limit of vinyl chloride in the water-based, acidic and alcoholic food simulants is 0.001mg/L; quantitative limit is 0.005mg/L. The detection limit of vinyl chloride in the oil-fat food simulant is 0.001mg/kg; quantitative limit is 0.005mg/kg.

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