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NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA

GB 31604.17-2016

**National food safety standard –
Food contact materials and articles –
Determination of the content and migration of acrylonitrile**

Issued on: October 19, 2016

Implemented on: April 19, 2017

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

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Foreword

This standard replaces GB/T 5009.152-2003 “Determination of residual acrylonitrile monomer in styrene-acrylonitrile copolymers and rubber-modified acrylonitrile-butadiene-styrene resins and their products used for foods pack” and GB/T 23296.8-2009 “Food contact materials - Polymer - Determination of acrylonitrile in food simulants - Gas chromatography” and SN/T 2197-2008 “Food contact materials - Polymer materials - Determination of acrylonitrile in food and food simulants - Gas chromatography”.

As compared with GB/T 5009.152-2003, the main changes of this standard are as follows:

- CHANGE the standard name into “National food safety standard - Food contact materials and articles - Determination of the content and migration of acrylonitrile”;
- DELETE the gas chromatography-hydrogen flame detector method, RETAIN the gas chromatography-nitrogen phosphorus detector method;
- ADD the determination of the migration of acrylonitrile in food contact materials and articles.

National food safety standard –

Food contact materials and articles –

Determination of the content and migration of acrylonitrile

1 Scope

This standard specifies the method for determination of the content and migration of acrylonitrile in food contact materials and articles.

This standard applies to the determination of the content and migration of acrylonitrile in food contact materials and articles.

Determination of acrylonitrile

2 Principle

The food contact materials and articles are dissolved or dispersed in a headspace flask via N,N-dimethylformamide, heated to allow the components to be tested to reach a gas-liquid equilibrium, then the top air is quantitatively sucked for gas chromatographic determination. The retention time is based for qualitative determination, the propionitrile is used as internal standard, the internal standard method is used for quantitative determination.

3 Reagents and materials

The water used in this method is grade I water specified in GB/T 6682. Containers and transfer apparatus used in the test shall avoid the use of plastic materials.

3.1 Reagents

N,N-dimethylformamide (DMF): analytically pure, no interference peaks at the retention times of acrylonitrile and propionitrile.

3.2 Standard substance

3.2.1 Acrylonitrile (C_3H_3N , CAS No.: 107-13-1): 1000 $\mu\text{g/mL}$, or a standard substance that is certified by the state and granted a standard substance certificate.

to add 200 μ L of propionitrile standard solution into the headspace flask, SHAKE it thoroughly, to make the specimen in the flask is completely dissolved or dispersed, PREPARE for the determination. Meanwhile MAKE blank test.

5.3 Instrument reference conditions

5.3.1 Headspace sample injector conditions

Headspace sample injector conditions are listed below:

- a) Balance time: 30 min;
- b) Furnace temperature: 80 °C;
- c) Pressure: 138 kPa;
- d) Pressurization time: 2 min;
- e) Injection time: 0.04 min;
- f) Transmission line temperature: 85 °C.

5.3.2 Gas chromatographic conditions

Gas chromatographic conditions are listed below:

- a) Column: cross-linked polyethylene glycol stationary phase capillary column, column length 30 m, inner diameter 0.32 mm, film thickness 0.25 μ m, or equivalent column;
- b) Column temperature program: 40 °C maintained for 4 min, 10 °C/min to 70 °C maintained for 1 min, 15 °C/min increased to 170 °C;
- c) Inlet temperature: 150 °C;
- d) Detector temperature: 280 °C;
- e) Carrier gas nitrogen flow rate: 50 mL/min;
- f) Hydrogen flow rate: 1.5 mL/min;
- g) Air flow rate: 145 mL/min;
- h) Sample inlet constant pressure injection: 100 kPa.

5.4 Calibration solution preparation and calibration curve making

TAKE 5 headspace flasks, respectively ADD 200 μ L of propionitrile standard solution, then ADD acrylonitrile series standard solutions in sequence. At this time, the acrylonitrile contents in the calibration solution correspond to 2.0 μ g,

obtained food simulant test solution shall be cooled or returned to room temperature before the next test.

12.1.3 Immersion solution treatment

The food simulant preparation process refers to GB/T 5009.156. USE a 5 mL pipette to pipette 5.0 mL of food simulant into a headspace flask, accurately WEIGH the transferred liquid mass. Immediately CAP to seal it, USE a 100 μ L micro-syringe to add 100 μ L of N,N-dimethylformamide and 100 μ L of propionitrile standard solution into the headspace flask, MIX it uniformly, PREPARE for determination. Meanwhile MAKE blank test.

12.2 Instrument reference conditions

12.2.1 Headspace sample injection conditions

Headspace sample injector conditions are listed below:

- a) Balance time: 30 min;
- b) Furnace temperature: 80 °C;
- c) Pressure: 138 kPa;
- d) Pressurization time: 2 min;
- e) Injection time: 0.04 min;
- f) Transmission line temperature: 85 °C.

12.2.2 Gas chromatographic conditions

Gas chromatographic conditions are listed below:

- a) Column: Cross-linked polyethylene glycol stationary phase capillary column, column length 30 m, inner diameter 0.32 mm, film thickness 0.25 μ m, or equivalent column;
- b) Column temperature program: 40 °C maintained for 4 min, 10 °C/min increased to 70 °C which is maintained for 1 min, 15 °C/min increased to 170 °C;
- c) Sample inlet temperature: 150 °C;
- d) Detector temperature: 280 °C;
- e) Carrier gas nitrogen flow rate: 50 mL/min;
- f) Hydrogen flow rate: 1.5 mL/min;

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