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NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA

GB 31604.15-2016

**National food safety standard –
Food contact materials and articles - Determination of
migration of 2,4,6-triamino-1,3,5-triazine (melamine)**

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Foreword

This Standard replaces GB/T 23296.15, *Food contact materials - Polymer - Determination of 2,4,6-triamino-1,3,5-triazine (melamine) in food simulants - High performance liquid chromatography*.

Compared with GB/T 23296.15-2009, the major changes of this Standard are as follows:

- the standard name is changed into “*National food safety standard - Food contact materials and articles - Determination of migration of 2,4,6-triamino-1,3,5-triazine (melamine)*”.

National food safety standard –

Food contact materials and articles - Determination of migration of 2,4,6-triamino-1,3,5-triazine (melamine)

1 Scope

This Standard specifies the method for the determination of migration of melamine in food contact materials and articles (melamine-formaldehyde resins and their moldings).

This Standard applies to the high performance liquid chromatography for the determination of migration of melamine in food contact materials and articles (melamine-formaldehyde resins and their moldings).

2 Principle

Make food contact materials and articles (melamine-formaldehyde resins and their moldings) in contact with food simulants; carry out migration test under certain soaking conditions; use the high performance liquid chromatography to test the test solution obtained from soaking treatment; and then convert into the specific migration of melamine. Based on food categories, select the corresponding food simulants and migration test conditions (time and temperature) in accordance with GB 31604.1. When the food simulant includes water, acetic acid of 4% (volume fraction), ethyl alcohol of 10%, 20% or 50%~95% (volume fraction), use the soak solution directly as test solution. When the food simulant is vegetable oil, use the test solution obtained by the extraction treatment of soak solution directly as sample; use the high performance liquid chromatography to determine the content of melamine in test solution; determine the soak solution volume and sample contact area for migration test in accordance with GB 5009.156; and convert into the specific migration of melamine (release amount of migrated substance).

3 Reagents and materials

Unless indicated otherwise, all reagents used for this method are analytically pure and the water is grade one water specified in GB/T 6682. The use of plastic material shall be avoided in test containers and transfer instruments.

3.1 Reagents

3.4.3 Preparation of food simulant standard working solution

3.4.3.1 Standard working solutions of water-based, acidic and alcoholic food simulants

Accurately absorb 0 mL, 0.125 mL, 0.250 mL, 1.25 mL and 2.50 mL of melamine standard intermediate solution (3.4.2) respectively to pour into a 25 mL volumetric flask; and use water-based food simulant to add to scale. The concentrations of melamine are respectively 0 mg/L, 0.500 mg/L, 1.00 mg/L, 5.00 mg/L and 10.0 mg/L. Use the same method to prepare melamine standard working solutions of the same concentration series using acidic and alcoholic food simulants respectively.

3.4.3.2 Standard working solutions of oil-based food simulants

Accurately weigh 5 g (accurate to 0.01 g) of oil-based food simulant (refined corn oil or olive oil) to pour respectively into five 25 mL glass test tubes with a stopper; use an adjustable pipette to transfer 0 μ L, 25.0 μ L, 50.0 μ L, 250 μ L and 500 μ L of melamine standard intermediate solution (3.4.2) respectively to a test tube; and obtain the standard working solutions of oil-based food simulants. The concentrations of melamine are respectively 0 mg/kg, 0.500 mg/kg, 1.00 mg/kg, 5.00 mg/kg and 10.00 mg/kg. Add 5.0 mL of isooctane to each test tube and mix up; add 5.0 mL of isopropyl alcohol solution (3.2.4); place test tubes in a thermostatic water bath at 70°C for ultrasonic extraction for 30 min; and then carry out centrifugal separation. Absorb 1 mL~2 mL of the lower layer of the aqueous solution; and pass through an organic phase microporous membrane 0.22 μ m to obtain test solutions, which are used for the high performance liquid chromatograph to carry out determination.

3.5 Material

Organic phase microporous membrane: 0.22 μ m.

4 Apparatus

4.1 High performance liquid chromatograph: equipped with an ultraviolet absorption detector or luminous diode array detector.

4.2 Vortex oscillator.

4.3 Ultrasonic thermostatic water bath.

4.4 Centrifuge: rotational speed greater than 4 000 r/min.

4.5 Analytical balance: of sensitivities 0.000 1 g and 0.01 g.