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GB 31604.11-2016

National Food Safety Standard - Food Contact Materials and Articles - Determination of Migration of 1,3-benzenedimethanamine

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Foreword

This Standard replaces GB/T 23296.25-2009 *Food contact materials - Polymer - Determination of 1 3-benzenedimethanamine in food simulants - High performance liquid chromatography* and SN/T 2550-2010 *Food contact materials. Polymer materials. Determination of 1,3-benzenedimethanamine in food simulants*.

Compared with GB/T 23296.25-2009, the main change in this Standard is as follow:

- modified the standard's name to "National Food Safety Standard - Food Contact Materials and Articles - Determination of Migration of 1,3-benzenedimethanamine".

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National Food Safety Standard - Food Contact Materials and Articles - Determination of Migration of 1,3-benzenedimethanamine

1 Scope

This Standard specifies the high-performance liquid chromatography method for determination of migration of 1,3-benzenedimethanamine.

This Standard is applicable to the determination of migration of 1,3-benzenedimethanamine in food contact materials and articles.

2 Principle

After the migration test of food contact materials, 1,3-benzenedimethylamine in water-based, acidic and alcohol food simulants shall be subjected to fluorescent amine derivatization, then separation by high performance liquid chromatography. Using fluorescence detector detection, external standard method to quantify.

3 Reagents and materials

Unless otherwise specified, the reagents used in this method are of analytical grade, water is of grade one specified in GB/T 6682 and saturated with nitrogen.

3.1 Reagents

3.1.1 Water-based, acidic, alcohol-based, oil-based food simulants: reagents used in accordance with the provisions of GB 31604.1

3.1.2 Methanol (CH_4O, CAS No.: 67-56-1): chromatographic pure

3.1.3 Tetrahydrofuran (C4H8O, CAS No: 109-99-9): chromatographic pure

3.1.4 Acetone (C_3H_6O, CAS No: 67-64-1)

3.1.5 Heptane (C_7H_{16}, CAS No.: 142-82-5)

3.1.6 Acetic acid ($C_2H_4O_2$, CAS No.: 64-19-7)

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Weigh 50 mg (nearest to 0.0001 g) of 1,3-benzenedimethylamine. Dissolve with water. Set volume to 50 mL. The concentration shall be 1000 mg/L. This solution shall be stored in the darkness at 5°C for three months.

3.4.2 1,3-benzenedimethylamine standard working solution used for water-based (acidic, alcoholic) food simulants

Pipette 0.20 mL of 1,3-benzenedimethanamine standard stock solution (3.4.1) in a 100mL volumetric flask with a graduated pipette. Dilute to scale with a water-based (acidic, alcoholic) food simulant to obtain a 1,3-benzenedimethanamine standard working solution for water-based (acidic, alcoholic) food simulants with a concentration of 2.0 mg/L. This solution shall be stored in the darkness at 5°C for one month.

3.4.3 1,3-benzenedimethylamine standard working solution used for oil-based simulants

Pipette 2.0 mL of 1,3-xylylenediamine standard stock solution (3.4.1) in a 100 mL volumetric flask with a graduated pipette. Dilute to scale with water to obtain a solution with a concentration of 20.0 mg/L. Pipette 10 mL of this solution in a 100 mL volumetric flask. Dilute to scale with tetrahydrofuran solution (3.2.2) to obtain 1,3-benzenedimethylamine standard working solution for oil-based simulants with a concentration of 2.0 mg/L. This solution shall be stored in the darkness at 5°C for one month.

3.4.4 1,3-benzenedimethylamine standard working solution of water-based (alcoholic) food simulants

Respectively using a graduated pipette to pipette 0.5 mL, 2.0 mL, 3.0 mL, 4.0 mL, 5.0 mL of 1,3-benzenedimethylamine standard working solution (3.4.2) in five 100 mL volumetric flasks. Use corresponding food simulants (water-based, alcohol food simulants) to set volume to scale and obtain standard working solutions with concentrations of 0.010 mg/L, 0.040 mg/L, 0.060 mg/L, 0.080 mg/L, 0.10 mg/L. Pipette 2.0 mL of the above solution into 5 5-mL tubes with a graduated pipette. Separately add 0.4 mL of boric acid buffer solution (3.2.5), mix well. Add 300 μL of fluorescein solution (3.2.3) derivatization reagent separately, oscillating 1min, placing still 10min. The derivative solution is filtered through a 0.45 μm filter for high performance liquid chromatography injection.

3.4.5 1,3-benzenedimethylamine standard working solution of acidic food simulants

Respectively using a graduated pipette to pipette 0.5 mL, 2.0 mL, 3.0 mL, 4.0 mL, 5.0 mL of 1,3-benzenedimethylamine standard working solution (3.4.2) in acidic food simulants into five 100 mL volumetric flasks. Use acidic food simulants to set volume to scale to obtain standard working solutions with concentrations of 0.010 mg/L, 0.040 mg/L, 0.060 mg/L, 0.080 mg/L, 0.10 mg/L.

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5 Analysis steps

5.1 Sample migration test

In accordance with the requirements of GB 5009.156 and GB 31604.1, carry out the sample migration test so as to obtain food simulant test solution. If the resulting food simulant test solution cannot be immediately used for the next test, the food simulant test solution shall be stored in a 4°C refrigerator from light.

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

5.2 Preparation of test solution

5.2.1 Preparations of water-based and alcohol food simulants

Pipette 2.0 mL of a water-based (alcoholic) food simulant obtained from the migration test in a 5-mL tube with a graduated pipette. Add 0.4 mL of borate buffer, mixing well. Then add 300 μL of fluorescent amine solution derivatization reagent, oscillating 1min, placing still 10min. The derivative solution is filtered through a 0.45 μm filter for high performance liquid chromatography injection.

5.2.2 Preparation of acidic food simulants

Pipette 10.0 mL of acidic food simulants from the migration test in a 25-mL beaker by using a graduated pipette. Drop 5 mol/L sodium hydroxide solution (3.2.4) with same volume as sodium hydroxide solution in 3.4.5, well mixing (solution pH shall be within 8.0~9.9). Pipette 2.0 mL of the above solution in a 5-mL tube with a graduated pipette. The rest shall follow “add 0.4 mL of borate buffer…for high performance liquid chromatography injection” in 5.2.1.

5.2.3 Preparation of oil-based food simulants

Weigh 20.0g (nearest to 0.01 g) olive oil mimics placing in a 125-mL separating funnel. Separately add 1 mL of tetrahydrofuran solution and 5.0 mL of heptane. After thoroughly mixing, add 20 mL of acetic acid solution, oscillating 5min, placing still 15min. After the two phases are completely separated, collect the aqueous phase. Pipette 10.0 mL of the above solution in a 25-mL beaker by using a graduated pipette. Drop 5 mol/L sodium hydroxide solution (3.2.4) with same volume as sodium hydroxide solution in 3.4.6, well mixing (solution pH shall be within 8.0~9.9). Pipette 2.0 mL of the above solution in a 5-mL tube with a graduated pipette. The rest shall follow “add 0.4 mL of borate buffer…for high performance liquid chromatography injection” in 5.2.1.

5.3 Preparation of blank solution