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NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA

GB 31064.51-2021

**National food safety standard - Food contact material
and products - Determination of migration of 1,4-
butanediol**

食品安全国家标准 食品接触材料及制品 1, 4-丁二醇迁移量的测定

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State Administration for Market Regulation.**

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National food safety standard - Food contact material and products - Determination of migration of 1,4-butanediol

1 Scope

This standard specifies the method for determining the migration of 1,4-butanediol in food contact materials and products.

This standard applies to the determination of migration of 1,4-butanediol, in plastics, coatings, food contact materials and products, which use adhesives and inks.

2 Principle

Food contact materials and products are immersed in food simulants OR alternative solvents. The 1,4-butanediol migrates into the food simulants. The migration amount of 1,4-butanediol in the food simulants is determined. For the water-based, acidic and alcoholic food simulants, it is tested after being diluted by methanol. For the oily food simulants, it is tested after being extracted by methanol. The test solution is separated by a gas chromatographic column; detected by a gas chromatograph, which is equipped with a flame ionization detector (FID). The retention time is used for qualitative, whilst the external standard method is used for quantification.

3 Reagents and materials

Unless otherwise stated, the reagents used in this method are all analytically pure; the water is the grade 1 water, as specified in GB/T 6682.

3.1 Reagents

3.1.1 Olive oil: It must meet the requirements of Appendix A in GB 5009.156.

3.1.2 Acetic acid (C₂H₄O₂).

3.1.3 Ethanol (C₂H₆O).

standard intermediate solution 1 (3.4.2), into six 10 mL volumetric flasks. Respectively, accurately add 2.0 mL of corresponding food simulants. Use methanol to make its volume accurately reach to the mark. Shake well. Let stand. It gets the standard working solutions, which have mass concentrations of 0.20 mg/L, 0.40 mg/L, 0.60 mg/L, 0.80 mg/L, 1.00 mg/L, 2.00 mg/L. Prepare it before use.

3.4.4.2 Standard working solution (oily food simulant)

Respectively, accurately weigh 2.0 g (accurate to 0.001 g) of oily food simulant (olive oil) into six centrifuge tubes with stoppers. Respectively add 50 µL of 6 standard intermediate solution 2 (3.4.3) of oily food simulant, at different mass concentrations. Shake well. Let it stand, to obtain standard working solutions, which have mass concentrations of 1.0 mg/kg, 2.0 mg/kg, 3.0 mg/kg, 4.0 mg/kg, 5.0 mg/kg, 10.0 mg/kg. Respectively add 2.00 mL of methanol solution for extraction. Mix well. Centrifuge for layering. Take the upper layer solution. Make it pass through a 0.45 µm filter membrane. Prepare for testing.

3.4.4.3 Standard working solution [Alternative solvent (95% ethanol)]

Accurately pipette 0.20 mL, 0.40 mL, 0.60 mL, 0.80 mL, 1.00 mL, 2.00 mL of standard intermediate solution 1 (3.4.2), into six 10 mL volumetric flasks. Respectively, accurately add 2.0 mL of 95% ethanol food simulant. Use methanol to make its volume reach accurately to the mark. Shake it well. Let it stand, to obtain the standard working solution, which has mass concentrations of 0.20 mg/L, 0.40 mg/L, 0.60 mg/L, 0.80 mg/L, 1.00 mg/L, 2.00 mg/L, respectively. Prepare it before use.

4 Instrument and equipment

4.1 Gas chromatograph: It is equipped with hydrogen flame ionization detector (FID).

4.2 Analytical balance: The sensitivity is 0.1 mg and 1 mg.

4.3 Organic phase microporous filter membrane: 0.45 µm.

4.4 Centrifuge: The maximum speed is > 3000 r/min.

5 Analytical procedures

5.1 Preparation of test solution

5.1.1 Migration test

5.2.5 Flow rate: 1 mL/min.

5.2.6 Detector (FID) temperature: 260 °C; hydrogen flow rate: 30 mL/min; air flow rate: 400 mL/min.

5.3 Drawing of standard curve

Determine the standard working solution, in accordance with the instrument reference conditions, which are listed in 5.2. Use the mass concentration of 1,4-butanediol in the standard working solution, as the abscissa, AND the response value of the corresponding peak area, as the ordinate, to draw the standard curve. Refer to Appendix A for the reference chromatogram of 1,4-butanediol, in the food simulant standard solution.

5.4 Determination of test solution

According to the instrument reference conditions listed in 5.3, the sample solution (5.1.2) and the blank solution (5.1.3) are respectively determined, to obtain the peak area of the target object. From the standard curve, calculate the content of the 1,4-butanediol, c and c_0 , in the sample solution and the blank solution. The content of the 1,4-butanediol, c_0 , in the blank solution shall not be higher than the mass concentration of the method detection limit.

Note: As the specimen contains a small amount of water, it is recommended to maintain the equipment regularly, based on actual testing conditions.

6 Expression of analysis results

6.1 Calculation of specific migration of 1,4-butanediol in food contact materials and products (expressed in mg/kg)

When the specific migration of 1,4-butanediol in food contact materials and products is expressed in mg/kg, it is calculated according to formula (1).

$$X = \frac{(c - c_0) \times V_1}{S_1} \times \frac{S_2}{V_2} \dots\dots\dots (1)$$

Where:

X - The specific migration of 1,4-butanediol, in milligrams per kilogram (mg/kg);

c - The content of 1,4-butanediol in the specimen soaking solution, in milligrams per liter (mg/L) or milligrams per kilogram (mg/kg);

c_0 - The content of 1,4-butanediol in the blank soaking solution, in milligrams per liter (mg/L) or milligrams per kilogram (mg/kg);

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