



**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB 31604.44-2016

**National food safety standard - Food contact materials and
articles - Determination of migration of ethylene glycol and
diethylene glycol**

食品安全国家标准 食品接触材料及制品 乙二醇和二甘醇迁移量的测定

(English Translation)

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Foreword

This Standard replaces GB/T 23296.18-2009, Food contact materials - Polymer - Determination of monoethylene glycol and diethylene glycol in food simulants - Gas chromatography.

Compared with GB/T 23296.18, 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 ethylene glycol and diethylene glycol";

-- the test types for food simulants are added.

National food safety standard - Food contact materials and articles - Determination of migration of ethylene glycol and diethylene glycol

1 Scope

This Standard specifies the gas chromatographic method for the determination of migration of ethylene glycol and diethylene glycol in food contact materials and articles.

This Standard applies to the determination of migration of ethylene glycol and diethylene glycol in food contact materials and articles.

2 Principle

Use the capillary gas chromatographic column to separate ethylene glycol and diethylene glycol in food containers and packaging materials; and use the hydrogen flame ionization detector for determination. Analyze sample directly for water-base, acidic and alcoholic food simulants; analyze sample of oil-base food simulants after water extraction; use the internal standard method for quantitation, with 1,4-butanediol as the internal standard substance.

3 Reagents and materials

Unless specified otherwise, all reagents are analytically pure reagents and all water is grade I water which is specified in GB/T 6682. Plastic material shall be avoided in the production of the containers and transferring vessels in the test.

3.1.5 Reagents required for the preparation of water-base, acidic, alcoholic and oil-

base food simulants. as specified in GB 31604.1.

5.1.1.1 Standard working solutions of water-base, acidic and alcoholic food simulants

Accurately absorb 1 mL of standard intermediate solutions of all concentrations respectively to pour into six 50 mL volumetric flasks; add water dropwise to scale; and then obtain the standard working solutions of the concentrations of ethylene glycol and diethylene glycol in water 0.0 mg/L, 3.0 mg/L, 6.0 mg/L, 15.0 mg/L, 30.0 mg/L and 60.0 mg/L. Use the same method and use the corresponding water-base, acidic and alcoholic food simulants to prepare the same concentration series of standard working solutions of ethylene glycol and diethylene glycol.

5.1.1.2 Standard working solutions of oil-base food simulants

Accurately weigh 50 g (accurate to 0.1 g) of oil-base food simulants to pour into six

250 mL separating funnels; add in succession 1 mL of the standard intermediate

solutions of all concentrations; mix up; and obtain the oil-base food simulants of the concentrations of ethylene glycol and diethylene glycol 0.0 mg/L, 3.0 mg/L, 6.0 mg/L,

15.0 mg/L, 30.0 mg/L and 60.0 mg/L. Add 50 mL of heptane to every separating funnel;

mix up; add water for oscillation extraction for 1 min; allow to stand for 5 min; and separate two phases into layers. Collect the lower water phase. And use 20 mL of water to extract oil-base food simulants once again. Combine the water for the two extractions; and add water dropwise to 50 mL. Take 1 mL of the solution to pass through a 0.2 µm filter membrane before using it for the determination.

5.1.2.1 General

In accordance with the intended use and working conditions of samples to be tested, operate in accordance with the migration test methods and test conditions which are specified in GB 5009.156 and GB 31604.1. After the migration test, mix up all the simulants which are obtained in the migration test and take part for the analysis.

The simulants obtained from the migration test may be stored in a refrigerator in a dark. Measure the standard working solutions prepared in 5.1.1 under the listed instrument parameters; plot the standard working curve, using the mass concentration of ethylene glycol or diethylene glycol as the abscissa, and using the corresponding ratio of the chromatographic peak area of ethylene glycol or diethylene glycol to that of the internal standard substance 1,4-butanediol; and obtain the linear equation. For the standard chromatogram see Annex A.

5.2.3 Quantitative determination

Inject samples of food simulants' test solutions and blank solutions in succession; and obtain respectively the corresponding ratios of the chromatographic peak area of ethylene glycol or diethylene glycol to that of the internal standard substance 1,4-

butanediol. Obtain the contents of ethylene glycol or diethylene glycol in the sample solutions in accordance with the standard curve, deducting the blank values.

6 Expression of analytical results

In accordance with the concentrations of ethylene glycol and diethylene glycol in the sample solutions which are obtained from the standard curve, calculate the migration volume as specified in GB 5009.156; and obtain the migrations of ethylene glycol and diethylene glycol in food simulants and products. The results shall be rounded off to the third decimal place.

7 Repeatability

The absolute difference between the results of two independent measurements under repeatable conditions shall not exceed 10% of the arithmetic mean value.

8 Others

The limit of quantitation of this method for ethylene glycol or diethylene glycol in water-base, acidic and alcoholic food simulants is 3 mg/L.

The limit of quantitation of this method for ethylene glycol or diethylene glycol in oil-base food simulants is 3 mg/kg.

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