GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB 31604.43-2016

National food safety standard - Food contact materials and products - Determination of migration quantity of ethylenediamine and hexamethylene diamine

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Table of Contents

Foreword

This standard replaces GB/T 23296.17-2009 "Food contact materials - Polymer - Determination of 1,2-diaminoethane and 1,6-diaminohexane in food simulants - Gas chromatography"

Compared with GB/T 23296.17-2009, the main changes of this standard are as follows:

---The standard name has been changed to “National food safety standard - Food contact materials and products - Determination of migration quantity of ethylenediamine and hexamethylene diamine”.

National food safety standard - Food contact materials and products - Determination of migration quantity of ethylenediamine and hexamethylene diamine

1 Scope

This standard specifies the determination methods for gas chromatography of migration quantity of ethylenediamine and hexamethylene diamine in food contact materials and products.

This standard applies to the determination of migration quantity of ethylenediamine and ethylenediamine in food contact materials and products.

2 Principles

Use ethyl chloroformate as derivatizing reagent; derivatively convert ethylenediamine and hexamethylene diamine in food simulants to the corresponding dicarbamate derivatives; use gas chromatography column to separate; use hydrogen flame ionization detector to test. Use internal standard method for quantification; the internal standard substance is 1,3-propane diamine.

3 Reagents and materials

Unless otherwise indicated, the reagents used in this method are analytical grade, the water is the grade-1 water specified in GB/T 6682. Containers and transfer implements in the test shall avoid the use of plastic materials.

3.1 Reagents

3.1.1 Ethyl chloroformate ($C_3H_5ClO_2$): Purity is greater than 97%.

3.1.2 Glacial acetic acid ($C_2H_4O_2$).

3.1.3 Anhydrous ethanol (C_2H_6O).

3.1.4 Toluene (C_7H_8).

3.4 Standard solution preparation

3.4.1 Ethylenediamine stock solution prepared by water (1000mg/L): Accurately WEIGH 50mg (accurate to 0.1mg) of ethylenediamine; PLACE it into 50mL volumetric flask; USE water to dilute it.

3.4.2 Hexamethylene diamine stock solution prepared by water (500mg/L): Accurately WEIGH 25mg (accurate to 0.1mg) of hexamethylene diamine; PLACE it into 50mL volumetric flask; USE water to dilute it.

3.4.3 Propane diamine stock solution prepared by water (500mg/L): Accurately WEIGH 25mg (accurate to 0.1mg) of propane diamine; PLACE it into 50mL volumetric flask; USE water to dilute it.

3.4.4 Ethylenediamine stock solution prepared by toluene (1000mg/L): Accurately WEIGH 50mg (accurate to 0.1mg) of ethylenediamine; PLACE it into 50mL volumetric flask; USE toluene to dilute it.

3.4.5 Hexamethylene diamine stock solution prepared by toluene (500mg/L): Accurately WEIGH 25mg (accurate to 0.1mg) of hexamethylene diamine; PLACE it into 50mL volumetric flask; USE toluene to dilute it.

3.4.6 Propane diamine stock solution prepared by toluene (500mg/L): Accurately WEIGH 25mg (accurate to 0.1mg) of propane diamine; PLACE it into 50mL volumetric flask; USE toluene to dilute it.

The prepared stock solution can be stored in dark place at 5°C~20°C, and it is valid within 3 months.

3.5 Vessels and materials

3.5.1 Glass bottle: 2mL, 10mL, the caps are coated with Teflon-coated butyl rubber or silicone rubber gaskets.

3.5.2 Volumetric flask: 50mL.

3.5.3 Micro-syringes: 10μL and 25μL.

3.5.4 Oscillator.

4 Instrument and equipment

4.1 Gas chromatograph: equipped with hydrogen flame ionization detector and data automatic analysis software.

4.2 Analytical balance: Sensitivity is 0.0001g and 0.01g.

When food contact materials and products are contacted with olive oil at 10d, 20°C or 10d, 40°C for a long period of time, ethylenediamine and hexamethylene diamine that have migrated from food contact materials and products react with olive oil, which results in lower test results. Ethanol solution (95%, volume fraction) or isooctane shall be used to replace oil-based food simulants for testing.

5.1.2.2 Water-based, acidic food, alcoholic food simulants

Accurately MEASURE 1mL of the water-based, acidic food, and alcoholic food simulants obtained from the migration test; PLACE it into 10mL glass bottle; ADD 10μL of propane diamine stock solution prepared by water; COVER and SEAL it; MIX it uniformly.

5.1.2.3 Oil-based food simulant

Accurately WEIGH 1.0g (accurate to 0.01g) of the oil-based food simulant obtained from the migration test; PLACE it into 10mL sample bottle; ADD 10μL of propane diamine stock solution prepared by toluene; COVER and SEAL it; MIX it uniformly.

5.1.2.4 Ethanol solution (95%, volume fraction)

Accurately MEASURE 1.0mL of the ethanol solution (95%, volume fraction) obtained from the migration test; PLACE it into 10mL sample bottle; ADD 10μL of propane diamine stock solution prepared by water; COVER and SEAL it; MIX it uniformly.

5.1.2.5 Isooctane

Accurately MEASURE 1.0mL of the isooctane obtained from the migration test; PLACE it into 10mL sample bottle; ADD 10μL of propane diamine stock solution prepared by toluene; COVER and SEAL it; MIX it uniformly.

5.1.3 Preparation of blank test solution

Handle the food simulants that have not been in contact with food contact materials and products in accordance with the operation of 5.1.2.

5.2 Derivatization of food simulant test solution

5.2.1 Water-based, acidic food, alcoholic food simulant and ethanol solution (95%, volume fraction)

ADD 1mL of ammonia solution (3%, volume fraction), 3mL of sodium hydroxide solution (5mol/L), 2mL of toluene, and 200μL of ethyl chloroformate into the glass bottle which contains water-based, acidic food, and alcoholic

c) Detector temperature: 300°C;

d) Furnace temperature: keep for 1min at 100°C, rise to 270 at 25°C/min, keep for 7min;

e) Carrier gas: nitrogen, flow rate 1.8mL/min;

f) Sample injection mode: split injection, split ratio 10:1;

g) Injection volume: 1μL.

5.4.2 Drawing of standard curve

Conduct derivatization for the standard working solution prepared in 5.1.1 according to 5.2; TEST it under the instrument parameters listed in 5.4.1. With the concentration of ethylenediamine or hexamethylene diamine in the standard working solution of food simulants as abscissa, AND the ratio of the peak area between the corresponding ethylenediamine derivative or hexamethylene diamine derivative and internal standard substance propane diamine derivative as vertical axis, DRAW a standard working curve; OBTAIN a linear equation. See Appendix A for the chromatograms of standard solution.

5.4.3 Quantitative determination

After the sequential sample injection of derivatized food simulant test solution and blank test solution, OBTAIN the corresponding ratio of the peak area of ethylenediamine derivative or hexamethylene diamine derivative and internal standard substance propane diamine derivative, respectively; OBTAIN the content of ethylenediamine or hexamethylene diamine in the sample solution according to the standard curve; DEDUCT blank value.

6 Analysis results expression

OBTAIB the concentration of ethylenediamine or hexamethylene diamine in the sample solution from the standard curve; CALCULATE the migration quantity according to GB 5009.156; OBTAIN the migration quantity of ethylenediamine or hexamethylene diamine in food contact materials and products. The calculation result is retained with three significant figures.

7 Precision

The absolute difference between two independent determinations obtained under repeatability conditions shall not exceed 10% of the arithmetic mean.

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