GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB 31604.48-2016

National standard for food safety – Food contact materials and products - Determination of formaldehyde migration

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Table of Contents

Foreword

This Standard replaces GB/T 5009.178-2003 “Determination of formaldehyde for food packaging material”, Clause 8 “Formaldehyde” in GB/T 5009.61-2003 “Method for analysis of hygienic standard of products of tripolycyanamide for food packaging”, Clause 7.2 “Free formaldehyde” in GB/T 5009.69-2008 “Method for analysis of hygienic standard of epoxy phenolic coatings for inner wall of food cans” and determination of formaldehyde in water-based simulants in GB/T 23296.26-2009 “Food contact materials - Polymer - Determination of formaldehyde and hexamethylenetetramine in food simulants - Spectrophotometry”.

Compared with GB/T 5009.178-2003, the main changes of this Standard are as follows:

- MODIFY the standard name TO “National standard for food safety - Food contact materials and products - Determination of formaldehyde migration”;

- DELETE the oscillopolarographic method; ADD acetylacetone spectrophotometric method and chromotropic acid spectrophotometric method.

National standard for food safety – Food contact materials and products – Determination of formaldehyde migration

1 Scope

This Standard specifies the spectrophotometric method for the determination of formaldehyde migration in food contact materials and products.

This Standard applies to the determination of formaldehyde migration in food contact materials and products.

Method I Acetylacetone spectrophotometric method

2 Principle

After the food simulant is in contact with the sample, the formaldehyde in the sample migrates to the food simulant. In the presence of ammonium acetate, gormaldehyde reacts with acetylacetone and generate yellow 3,5-diacetyl-1,4 ˇ dihydrodimethylpyridine; USE a spectrophotometer to measure the absorbance of the test solution at 410 nm; COMPARE it with that of the reference series to obtain the content of formaldehyde in the food simulant, and then obtain the formaldehyde migration in the sample.

3 Reagents and materials

Unless otherwise stated, the reagents used in this method are of analytical reagent and water is the tertiary water specified in GB/T 6682.

3.1 Reagents

3.1.1 Anhydrous ethanol (CH_3CH_2OH).

3.1.2 Anhydrous ammonium acetate (CH_3COONH_4).

3.1.3 Acetylacetone ($C_5H_8O_2$).

3.1.4 Glacial acetic acid (CH_3COOH): guarantee reagent.

3.1.5 Sodium hydroxide (NaOH).

3.2 Preparation of reagents

3.2.1 Water-based food simulants: PREPARE according to the specifications of GB 5009.156.

3.2.2 Acetylacetone solution: WEIGH 15.0 g of anhydrous ammonium acetate and dissolve in the appropriate amount of water; TRANSFER into a 100 mL volumetric flask; ADD 40 μL of acetylacetone and 0.5 mL of glacial acetic acid; DILUTE with water to the scale; MIX well. This solution is prepared when it is in need.

3.3 Preparation of standard solutions

3.3.1 Formaldehyde solution (37 % ~ 40 %, mass fraction): preserve at 0 °C ~ 4 °C.

3.3.2 Formaldehyde standard stock solution: PIPETTE 5.0 mL of formaldehyde solution to a 1000 mL volumetric flask; DILUTE with water to the scale; PRESERVE at 0 °C ~ 4 °C; it is valid for 12 months; CALIBRATE before use (see Annex A); or use formaldehyde solution standard products to prepare directly.

3.3.3 Formaldehyde standard use solution: according to the calibrated formaldehyde concentration, accurately transfer a certain volume of formaldehyde standard stock solution to dilute with the corresponding simulants, respectively, to the equivalent of 10 mg formaldehyde per liter. This solution is prepared when it is in need.

4 Instruments and equipment

4.1 UV-visible spectrophotometer.

4.2 Constant-temperature water bath: the precision is controlled at ± 1 °C.

4.3 Colorimetric tube with stopper: 10 mL (with scale).

5 Analysis steps

5.1 Migration test

According to the intended use and the use conditions of the sample to be tested, in accordance with the requirements of GB 5009.156 and GB 31604.1, carry out the migration test to the sample. During the migration test to prior to the determination, it shall pay attention to sealing, in order to avoid the loss of formaldehyde volatile. At the same time carry out the blank test.

5.2 Chromogenic reaction

and water is a tertiary water or deionized water specified in GB/T 6682. All reagents in which formaldehyde shall not be detected by this method.

10.1 Reagents

10.1.1 Anhydrous ethanol (CH_3CH_2OH).

10.1.2 Chromotropic acid ($C1_0H_8O_8S_2$).

10.1.3 Sulfuric acid: guarantee reagent.

10.1.4 Glacial acetic acid (CH_3COOH): guarantee reagent.

10.1.5 Sodium hydroxide (NaOH).

10.2 Preparation of reagents

10.2.1 Water-based food simulants: PREPARE according to the specifications of GB 5009.156.

10.2.2 Sulfuric acid solution: MEASURE 100 mL of sulfuric acid, DISSOLVE in 50 mL of water, STIR slowly, MIX well.

NOTE: Concentrated sulfuric acid will produce a lot of heat when contacting with water, and the density of concentrated sulfuric acid is greater than that of water, so adding water to concentrated sulfuric acid may lead to bruising. During preparation, the sulfuric acid shall be added slowly along the beaker wall to the water while stirring constantly.

10.2.3 Chromotropic acid solution (5 mg/mL): WEIGH 0.500 g of chromotropic acid; DISSOLVE in the appropriate amount of water; TEANSFER into a 100 mL volumetric flask; DILUTE with water to the scale; MIX and then filter with slow-speed filter paper; COLLECT the filtrate for further use. This solution is prepared when it is in need.

10.3 Preparation of standard solutions

10.3.1 Formaldehyde solution (37 % ~ 40 %, mass fraction): preserve at 0 °C ~ 4 °C.

10.3.2 Formaldehyde standard stock solution: PIPETTE 5.0 mL of formaldehyde solution to a 1000 mL volumetric flask; DILUTE with water to the scale; PRESERVE at 0 °C ~ 4 °C; it is valid for 12 months; CALIBRATE before use (see Annex A); or use formaldehyde solution standard products to prepare directly.

10.3.3 Formaldehyde standard use solution: according to the calibrated formaldehyde concentration, accurately transfer a certain volume of formaldehyde standard stock solution to dilute with the corresponding simulants, respectively, to the equivalent of 10 mg formaldehyde per liter. This solution is prepared when it is in need.

Annex A

Calibration of formaldehyde standard stock solution

A.1 Reagents

A.1.1 Sodium hydroxide (NaOH).

A.1.2 Hydrochloric acid (HCl): 36.0 % ~ 38.0 %.

A.1.3 Iodine (I_2).

A.1.4 Potassium Iodide (KI).

A.1.5 Soluble starch.

A.2 Preparation of reagents

A.2.1 Sodium hydroxide solution (40 g/L): WEIGH 40.0 g of sodium hydroxide; DISSOLVE in 1000 mL of water.

A.2.2 Hydrochloric acid solution (1 mol/L): PIPETTE 8.3 mL of hydrochloric acid; DISSOLVE in water and dilute to 100 mL.

A.2.3 Iodine solution (0.05 mol/L): WEIGH 13.0 g of iodine and 35.0 g of potassium iodide; DISSOLVE in 100 mL of water; DILUTE to 1000 mL; SHAKE well. STORE in a brown bottle.

A.2.4 Sodium thiosulfate standard titration solution (0.1 mol/L).

A.2.5 Starch solution (10 g/L): WEIGH 1.0 g of soluble starch; ADD a small amount of water to transfer to the paste; POUR into 100 mL of boiling water and mix thoroughly; BOIL for a while. This solution is prepared when it is in need.

A.3 Calibration of formaldehyde standard stock solution

PIPETTE 5.0 mL of formaldehyde standard stock solution to an iodine flask; ADD 15.0 mL of 0.05 mol/L iodine solution and 10.0 mL of 40 g/L sodium hydroxide solution; LET stand for 5 min. ADD 11.0 mL of 1 mol/L hydrochloric acid acidification; SHAKE well; USE 0.1 mol/L sodium thiosulfate standard titration solution to titrate to grass yellow; ADD 1.0 mL of starch solution to titrate until blue disappears. At the same time carry out the reagent blank test. The concentration of formaldehyde standard stock solution is calculated according to equation (A.1):

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