



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

GB 31604.9-2016

Replacing GB/T 5009.67-2003

**National food safety standard - Food contact materials and
articles - Determination of heavy metals in food simulants**

食品安全国家标准 食品接触材料及制品 食品模拟物中重金属的测定

(English Translation)

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Foreword

This document was issued on 31 August 2016 by the National Health and Family Planning Commission of the People's Republic of China, and has applied since 20 September 2016.

It replaces GB/T 5009.67-2003.

It is a GB national food safety standard: compliance is mandatory in China. It belongs to the GB 31604 series of test methods for food contact materials and articles.

1 Scope

This standard specifies the determination method of heavy metals in food simulants (4% acetic acid).

Method I of this standard is applicable to the determination of heavy metals in food simulants (4% acetic acid) of food contact materials and articles.

Method II of this standard is applicable to the determination of heavy metals in food simulants (4% acetic acid) of food contact rubbers.

Method I Direct Colorimetry

2 Principle

React the heavy metal (calculated by lead) in test solution of food simulant obtained from migration test with sodium sulfide, form the yellowish-brown sulfide in acid solution, then compare its color with the color of lead standard solution.

3 Reagents and materials

Note: unless otherwise specified, all reagents adopted for this method are analytically pure and water is Grade 2 water as specified in GB/T 6682.

3.1 Reagents

3.1.1

Sodium sulfide 9-hydrate ($\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$).

3.1.2

Glycerol ($\text{C}_3\text{H}_8\text{O}_3$).

3.1.3

Glacial acetic acid (CH_3COOH).

3.1.4

Nitric acid (HNO_3 with mass fraction of 60%).

3.2 Preparation of reagents

3.2.1

Nitric acid solution (with mass fraction of 10%)

Pipette 16.7mL of nitric acid (3.1.4) and dilute with water to 100mL.

3.2.2

Sodium sulfide solution

Weigh 15.4g of sodium sulfide 9-hydrate (3.1.1), add 10mL of water, stir sufficiently with glass rod until it is dissolved, then add 30mL of glycerol (3.1.2), mix uniformly and keep sealed for preservation. The stability of this solution can be ensured for a month at room temperature.

3.2.3

Food simulant

4% acetic acid solution (volume fraction). Put 40mL of glacial acetic acid (3.1.3) into a 1L volumetric flask, and dilute it with water to the scale.

3.3 Standards

Lead nitrate ($\text{Pb}(\text{NO}_3)_2$, >98%) or lead standard solution (with 1mg lead per milliliter) approved and awarded with reference material certificate by the nation.

3.4 Preparation of standard solutions

3.4.1

Lead standard stock solution

Accurately weigh 0.1598g of lead nitrate (3.3.1, to the nearest 0.0001g), dissolve it in 10mL of nitric acid solution (3.2.1), transfer the solution into a 1,000mL volumetric flask, dilute it with water to the scale. 1mL of this solution is equivalent to 100 μg of lead.

3.4.2

Lead standard application solution

Pipette 10.0mL of lead standard stock solution (3.4.1) into a 100mL volumetric flask, and dilute it with water to the scale, then shake well; or pipette 1.0mL of lead standard solution (3.3) approved and awarded with reference material certificate by the nation into a 100mL volumetric flask, and dilute it with water to the scale, then shake well.

1mL of this solution is equivalent to 10 μg of lead.

4 Apparatus

4.1

Constant-temperature oven.

4.2

Volumetric flask

100mL and 1,000mL.

4.3

Pipette

10mL and 20mL.

4.4

Colorimetric tube

50mL.

4.5

Measuring cylinder

50mL.

4.6

Analytical balance

With a sensitivity of 0.0001g and 0.1g.

5.1 Preparation of specimen

According to the requirements of "General Principle of Migration Test Pretreatment of Food Contact Materials and Their Products" (GB/T 5009.156) and national standards of corresponding products, take 4% acetic acid solution (3.2.3) as the specimen of food simulant to conduct migration test, and obtain the test solution of food simulant. Protect it from light in a refrigerator with 0°C~4°C.

5.2 Determination of specimen solution

Pipette 20mL of test solution (5.1) of food simulant obtained through migration test into a 50mL colorimetric tube, and dilute it with water to the scale; pipette 2mL of standard lead application solution (3.4.2) into a 50mL colorimetric tube, add 20mL of 4% acetic acid solution (3.2.3), and add water to the scale, then mix uniformly. Respectively add 2 drops of sodium sulfide solution (3.2.2) into these two solutions, mix uniformly and keep them still for 5min; take white color as the background, observe these two solutions from top or side face, and compare the color of test solution of simulant and standard solution.

6 Expression of analysis results

In the case that the color of specimen is darker than that of the standard solution, the migration volume (calculated by lead) of heavy metals in food contact materials and articles is larger than 1mg/L;

In the case that the color of specimen is lighter than that of the standard solution, the migration volume (calculated by lead) of heavy metals in food contact materials and articles is less than 1mg/L.

Method II Masking Interference Colorimetry

7 Principle

React the heavy metal (calculated by lead) in test solution of food simulant obtained from migration test with sodium sulfide, form the yellowish-brown sulfide in acid solution, then compare its color with that of lead standard solution.

8 Reagents and materials

Unless otherwise specified, all reagents adopted for this method are analytically pure and water is Grade 2 water as specified in GB/T 6682.