# GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

# GB 5009.74-2014

## National Food Safety Standard -

## Method for Limit Test of Heavy Metals in Food Additives



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# Foreword

This Standard replaces GB/T 5009.74-2003, *Method for Limit Test of Heavy Metals in Food Additives.*

Compared with GB/T 5009.74-2003, the major changes of this Standard are as follows:

-- the standard name is changed into "*National Food Safety Standard – Method for Limit Test of Heavy Metals in Food Additives*";

-- the pressure digestion tank digestion method is added.

# National Food Safety Standard -

# Method for Limit Test of Heavy Metals in Food Additives

## 1 Application Scope

This Standard specifies the method for limit test of heavy metals in food additives.

This Standard applies to the limit test of heavy metals in food additives.

## 2 Principle

Under the weak acid (pH 3~4) conditions, the heavy metal ions in the specimen react with hydrogen sulphide to generate a brownish black colour; then it is compared with the lead standard solution which is processed using the same method.

## 3 Reagents and Materials

**NOTE** Unless stated otherwise, in this Standard, the reagents used are analytically pure and the water is grade 1 water as specified in GB/T 6682.

### 3.1 Reagents

**3.1.1** Nitric acid ($HNO_3$).

**3.1.2** Sulphuric acid ($H_2SO_4$).

**3.1.3** Hydrochloric acid (HCl).

**3.1.4** Ammonium hydroxide ($NH_3 \cdot H_2O$).

**3.1.5** Ammonium acetate ($C_2H_7NO_2$).

**3.1.6** Phenolphthalein ($C_2OH_{14}O_4$).

**3.1.7** Hydrogen peroxide ($H_2O_2$).

**3.1.8** Hydrogen sulphide ($H_2S$).

**3.1.9** Perchloric acid ($HClO_4$).

### 3.2 Preparation of reagents

take out after cooling, add 2 mL of hydrochloric acid (6 mol/L) to moisten the residual and evaporate on a water bath until dry. Use 1 drop of concentrated hydrochloric acid to moisten the residual, add 10 mL of water, heat once again on a boiling water bath for 2 min, transfer the solution to a 20 mL volumetric flask, conduct filtration if necessary, use a small quantity of water to wash the crucible and filter, transfer the filtrate together into a volumetric flask, mix up after adding dropwise to the scale. Each 10 mL of the solution is equivalent to 1.0 g of specimen. While the specimen is ashed, take another crucible and conduct reagent blank tests at the same time.

c) pressure digestion tank digestion method: weigh an appropriate quantity of specimen (accurate to 0.001 g) in accordance with the operating instructions to the pressure digestion tank to place into the inner tank of TTeflon, and add 2 mL ~ 4 mL of nitric acid to soak overnight. Then add 2 mL ~ 3 mL of hydrogen peroxide (the total volume shall not exceed 1/3 of the volume of the tank). Put on the inner cap, screw down the stainless steel outer casing, place in a thermostatic desiccator, maintain for 3 h ~ 4 h at 120°C ~ 140°C, cool to room temperature in the desiccator, use a dropper to wash or filter (depending on the salt content of the specimen after digestion) the digestive liquid to the volumetric flask, use a small quantity of water to wash the tank for multiple times, combine the washings into the volumetric flask and add dropwise to the scale, and mix up as standby; and meanwhile, conduct reagent blanks tests.

# 6 Determination

**6.1** Tube A (standard tube): absorb the lead standard working solution (containing not less than 10 μg), whose lead content is equivalent to the specified heavy metal limit, pour into a 50 mL Nessler tube (if the specimen is treated, the same quantity of reagent blank solution as the specimen solution shall be absorbed meanwhile), add water to 25 mL, mix up, add 1 drop of phenolphthalein indicator solution, use diluted hydrochloric acid (6 mol/L) or diluted ammonia hydroxide (1 mol/L) to adjust the pH to neutral (when the phenolphthalein red just fades), add 5 mL of acetate buffer solution of pH 3.5, and mix up as standby.

**6.2** Tube B (specimen tube): take one Nessler tube matched with tube A, add 10 mL ~ 20 mL (or an appropriate quantity) of specimen solution, add water to 25 mL, mix up, add 1 drop of 1% phenolphthalein indicator solution, use diluted hydrochloric acid (6 mol/L) or diluted ammonia hydroxide (1 mol/L) to adjust the pH to neutral (when the phenolphthalein red just fades), add 5 mL of acetate buffer solution of pH 3.5, and mix up as standby.

**6.3** Tube C: take a Nessler tube matched with tube A and tube B, add the same quantity of the same specimen solution as tube B, then add the same quantity of lead standard working solution (10 μg/mL) as tube A, add water to 25 mL, mix up, add 1 drop of 1% phenolphthalein indicator solution, use diluted hydrochloric acid (6 mol/L) or diluted ammonia hydroxide (1 mol/L) to adjust the pH to neutral (when the

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