



**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
(English Translation)**

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People's Republic of China**

Table of Contents

Foreword...	3
1 Application Scope...	4
2 Principle...	4
3 Reagents and Materials...	4
4 Apparatus...	5
5 Specimen Treatment...	6
6 Determination...	7

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.

3.3 Standard substance

High-purity lead nitrate $[\text{Pb}(\text{NO}_3)_2]$, a standard substance whose purity is 99.99% or which passes the national certification and is granted of the certificate of standard substance.

3.4.1 Lead standard stock solution (1 mg/mL). weigh 0.159 8 g of high-purity lead nitrate $[\text{Pb}(\text{NO}_3)_2]$ to dissolve in 10 mL of nitric acid solution (1%),

3.4.2 Lead standard working solution (10 $\mu\text{g/mL}$). absorb 1.0 mL of lead standard stock solution (1 mg/mL) to pour into a 100 mL volumetric flask, add water to the scale.

4 Apparatus

NOTE All glass apparatus used needs to be soaked in nitric acid (1 + 4) for more than 24 h, rinsed with water repeatedly and finally rinsed clean with deionized water.

4.1 50 mL Nessler tubes.

5.1 Preparation of inorganic specimens

The "specimen treatment" of inorganic specimens may be the pretreatment as required by the specifications of all products including dissolution or digestion.

5.2 Preparation of organic specimens

The "specimen treatment" of organic specimens may generally be conducted in accordance with the following methods, in addition to the specification requirements of all products.

a) wet digestion. weigh 5.00 g of specimen, place in a 25 mL conical flask, add 10 mL ~ 15 mL of nitric acid to moisten the specimen, heat on an electric hot plate after placing aside for a moment (or overnight), take down for cooling after the reaction becomes gentle, add 5 mL of sulphuric acid along the flask wall,

continue to heat until the solution in the flask becomes brown, add nitric acid continuously (add some perchloric acid if necessary) until the organic materials are fully decomposed, continue to heat until lots of sulfur dioxide white smoke is generated and finally the solution shall be colourless or yellowish.

b) dry digestion. weigh 5.00 g of specimen, place in a hard glass evaporating dish or quartz crucible, add an appropriate quantity of sulphuric acid to moisten the specimen, use a soft fire on an electric furnace for carbonization before adding

2 mL of nitric acid and 5 drops of sulphuric acid, heat carefully until white smoke

disperses completely, transfer to a muffle furnace, conduct full ashing at 500°C, 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.

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 Teflon, and add 2 mL ~ 4 mL of nitric acid to soak overnight.

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.

6.4 Add 10 mL of freshly-prepared hydrogen sulphide saturated solution to all tubes,

add water to the 50 mL scale, mix up, observe on the white background after placing in a dark place for 5 min.

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