



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

GB 5009.75-2014

**National food safety standard - Determination of Lead in Food
Additives**

食品安全国家标准 食品添加剂中铅的测定

(English Translation)

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1 Scope

This Standard specifies the limit test and quantitative test methods for lead in food additives.

This Standard applies to the limit test and quantitative test of lead in food additives.

Method One -- Diphenylthiocarbazone (dithizone) colorimetric method

2 Principle

The specimen is treated by adding diammonium hydrogen citrate, potassium cyanide and hydroxylamine hydrochloride to eliminate the interference of iron, copper, zinc and other ions. At pH 8.5 ~ 9.0, lead ions and diphenylthiocarbazone (dithizone) form a red complex. Extract with chloroform and compare with the standard series to do limit test or quantitative test.

3 Reagents and materials

NOTE: Unless otherwise specified, all reagents used in this method are analytically pure and water is grade one water as specified in GB/T 6682.

3.1 Reagents

3.1.1

Nitric acid (HNO_3).

3.1.2

Sulfuric acid (H_2SO_4).

3.1.3

Ammonia ($\text{NH}_3 \cdot \text{H}_2\text{O}$).

3.1.4

Hydrochloric acid (HCl).

3.1.5

Phenol red ($\text{C}_{19}\text{H}_{14}\text{O}_5\text{S}$).

3.1.6

Chloroform (CHCl_3).

3.1.7

Diammonium hydrogen citrate ($\text{C}_6\text{H}_{14}\text{N}_2\text{O}_7$).

3.1.8

Hydroxylamine hydrochloride ($\text{NH}_2\text{OH} \cdot \text{HCl}$).

3.1.9

Potassium cyanide (KCN).

3.1.10

Diphenylthiocarbazone (dithizone) ($\text{C}_{13}\text{H}_{12}\text{N}_4\text{S}$).

3.1.11

Ethanol ($\text{C}_2\text{H}_6\text{O}$).

3.1.12

Perchloric acid (HClO_4).

3.2 Reagent preparation

3.2.1

Nitric acid solution (1+1)

Take 50 mL of nitric acid and slowly add it into 50 mL of water.

3.2.2

Ammonia solution (1+1)

Mix 1 part of ammonia water with 1 part of water. If it contains lead, re-distillate it using an all-glass distiller.

3.2.3

Phenol red indicator solution (1 g/L ethanol solution)

Weigh 100 mg of phenol red. Dissolve it in 100 mL of ethanol (filter if necessary).

3.2.4 Diammonium hydrogen citrate solution (500 g/L): Weigh 100 g of diammonium

hydrogen citrate and dissolve it in 100 mL of water. Add two drops of phenol red indicator solution. Add ammonia solution (1+1) to adjust the pH to 8.5~9.0 (from yellow to red, add two more drops). Extract with dithizone solution several times. 10 mL~20 mL each time, until the chloroform layer turns green and does not change.

Discard the chloroform layer. Wash twice with chloroform, 5 mL each time. Discard the chloroform layer. Add water to dilute to 200 mL.

3.2.5 Ammonia solution (1+99): Mix 1 part chlorine water with 99 parts water.

3.2.6 Hydroxylamine hydrochloride solution (200 g/L): Weigh 20 g of hydroxylamine

hydrochloride. Dissolve in 40 mL of water. Add two drops of phenol red indicator solution. Add ammonia solution (1+1) to adjust the pH to 8.5~9.0 (add two more drops when the color changes from yellow to red). Extract with dithizone chloroform solution several times, 10 mL~20 mL each time until the chloroform layer remains green. Wash twice with chloroform, 5 mL each time. Discard the chloroform layer. Add hydrochloric acid (1+1) to the water layer to make it acidic. Add water to 100 mL.

3.2.7 Potassium cyanide solution (100 g/L): Weigh 10 g of potassium cyanide. Dissolve

it in water and make up to 100 mL.

WARNING: Potassium cyanide (KCN) is a hazardous chemical and appropriate protective measures shall be taken.

3.2.8 Dithizone stock solution (0.05% chloroform solution): Weigh 0.5 g of finely

ground dithizone and dissolve it in 50 mL of chloroform. If there is any residue, filter it with filter paper into a 250 mL separatory funnel. Extract it with ammonia solution (1+99) three times, 100 mL each time. Filter the extract with absorbent cotton into a

500 mL separatory funnel. Adjust it to acidity with hydrochloric acid solution (1+1).

4.2 125 mL separatory funnel.

4.3 250 mL Kjeldahl flask or 250 mL conical flask.

4.4 Electronic balance: with a sensitivity of 0.1 mg and 1 mg.

5.1 Preparation of inorganic specimens

The "specimen processing" of inorganic samples is measured according to the corresponding standard methods.

5.2 Preparation of organic specimens

In addition to the corresponding standard methods for organic specimens, the

"specimen processing" is generally carried out according to the following methods:

a) Wet digestion: Weigh 5.000 g of the specimen and place it in a 250 mL conical flask. Add 10 mL of nitric acid. After leaving it for a while (or overnight), heat it.

After the reaction has subsided, remove it and cool it. Add 5 mL of sulfuric acid along the wall of the flask. Continue heating until the solution in the flask begins to turn brown. Keep adding nitric acid (if necessary, add some perchloric acid) until the organic matter is completely decomposed. Continue heating until a large amount of white sulfur dioxide smoke is generated. The solution shall be colorless or slightly yellow in the end. After cooling, add 20 mL of water and boil it.

Remove the remaining nitric acid until white smoke is generated. Do this twice.

Let it cool. Transfer the solution to a 50 mL volumetric flask. Wash the conical flask 2~3 times with a small amount of water. Transfer the washing solution to the volumetric flask. Add water to the mark. Mix well and set aside. Take the same amount of nitric acid and sulfuric acid. At the same time, do a reagent blank test.

b) Dry ashing method: This method is used for specimens that are not suitable for wet digestion. Weigh 5.000 g of specimen into a porcelain crucible. Add an appropriate amount of sulfuric acid to wet the specimen. After careful carbonization, add 2 mL of nitric acid and 5 drops of sulfuric acid. Carefully heat until the white smoke evaporates. Transfer to a high-temperature furnace. Ash completely at 500°C. Take out after cooling. Add 1 mL of nitric acid solution (1+1). Heat to dissolve the ash. Transfer the specimen solution to a 50