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NATIONAL STANDARD

OF THE PEOPLE'S REPUBLIC OF CHINA

GB 5009.13-2017

National food safety standard -

Determination of copper in foods

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Issued on: April 06, 2017 **Implemented on: October 06, 2017**

Issued by: National Health and Family Planning Commission of the PRC;
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Table of Contents

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Foreword

This standard replaces GB/T 5009.13-2003 “Determination of copper in food”, GB 5413.21-2010 “National food safety standard - Determination of calcium, iron, zinc, sodium, potassium, magnesium, copper and manganese in foods for infants and young children, milk and milk products”, GB/T 23375-2009 “Determination of copper, iron, zinc, calcium, magnesium and phosphorus content in vegetables and derived products”, GB/T 9695.22-2009 “Meat and meat products - Determination of copper content”, GB/T 14609-2008 “Inspection of grain and oils—Determination of copper, iron, manganese, zinc, calcium, magnesium in cereals and derived products by atomic absorption and flame spectrophotometry”, GB/T 18932.12-2002 “Method for the determination of potassium, sodium, calcium, magnesium, zinc, iron, copper, manganese, chromium, lead, cadmium contents in honey - Atomic absorption spectrometry”, and NY/T 1201-2006 “Determination of copper iron and zinc content in vegetables and derived products”.

As compared with GB/T 5009.13-2003, the main changes of this standard are as follows:

- CHANGE the standard name into “National food safety standard - Determination of copper in foods”;

- In the pretreatment method, ADD the wet digestion, pressure tank digestion, and microwave digestion;

- RETAIN the graphite furnace atomic absorption spectrometry method as the first method, using the ammonium dihydrogen phosphate-palladium nitrate solution as a matrix modifier; RETAIN the flame atomic absorption spectrometry method as the second method; and DELETE the diethyldithiocarbamate colorimetric method

- ADD the inductively coupled plasma mass spectrometry method as the third method;

- ADD the inductively coupled plasma emission spectrometry method as the fourth method;

- ADD the microwave digestion temperature rising program, graphite furnace atomic absorption spectrometry and flame atomic absorption spectrometry instrument reference conditions as the appendixes.

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National food safety standard -

Determination of copper in foods

1 Scope

This standard specifies the determination of copper content in food by graphite furnace and flame atomic absorption spectrometry, inductively coupled plasma mass spectrometry and inductively coupled plasma-atomic emission spectrometry.

This standard applies to the determination of copper in all types of foods.

Method 1: Graphite furnace atomic absorption spectrometry

2 Principle

After digestion, the sample is atomized in graphite furnace AND the absorbance is measured at 324.8 nm. In a certain concentration range, the copper absorbance value is proportional to the copper content, AND it is compared with the standard series for quantitative.

3 Reagents and materials

Unless otherwise indicated, the reagents used in this method are excellent pure AND the water is the level II water as specified in GB/T 6682.

3.1 Reagents

3.1.1 Nitric acid (HNO_3).

3.1.2 Perchloric acid ($HClO_4$).

3.1.3 Ammonium dihydrogen phosphate ($NH_4H_2PO_4$).

3.1.4 Palladium nitrate [$Pd(NO_3)_2$].

3.2 Reagent preparation

3.2.1 Nitric acid solution (5 + 95): MEASURE 50 mL of nitric acid; slowly ADD it into 950 mL of water; MIX it uniformly.

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digest it until white smoke is produced and the digestion solution is in colorless transparent or slightly yellow; TAKE the digestion tube out; COOL it down, USE water to make its volume reach to 10 mL; MIX it uniformly to prepare for use. Meanwhile MAKE the reagent blank test. It may also use the conical flask; and PERFORM wet digestion on the adjustable heating plate in accordance with the aforementioned operation procedures.

5.2.2 Microwave digestion

WEIGH 0.2 g ~ 0.8 g (accurate to 0.001 g) of solid samples or accurately PIPETTE 0.500 mL ~ 3.00 mL of liquid sample into the microwave digestion tank; ADD 5 mL of nitric acid; DIGEST the sample in accordance with the operation procedures of microwave digestion, AND the digestion conditions are as shown in A.1. TAKE out the digestion tank after cooling; REMOVE acid on the electrical heating plate at 140 °C ~ 160 °C to about 1 mL. After cooling the digestion tank, TRANSFER the digestion solution into a 10 mL volumetric flask; USE a small amount of water to rinse the digestion tank for 2 ~ 3 times; COLLECT the rinsing solution into the volumetric flask; USE water to make the volume reach to the mark; MIX it uniformly and PREPARE for use. And meanwhile MAKE the reagent blank test.

5.2.3 Pressure tank digestion

WEIGH 0.2 g ~ 1 g (accurate to 0.001 g) of solid samples or accurately PIPETTE 0.500 mL ~ 5.00 mL of liquid sample into the digestion inner tank; ADD 5 mL of nitric acid. Cover the inner cap, TIGHTEN the stainless steel jacket; PLACE it into the constant temperature drying oven; LET it stand at 140 °C ~ 160 °C for 4 h ~ 5 h. Slowly LOOSEN the outer tank after cooling; TAKE out the digestion inner tank. REMOVE acid on the electrical heating plate at 140 °C ~ 160 °C to about 1 mL. After cooling it down, TRANSFER the digestion solution into a 10 mL volumetric flask; USE a small amount of water to rinse the inner tank and inner cap for 2 ~ 3 times; COLLECT the rinsing solution into the volumetric flask; USE water to make the volume reach to the mark; MIX it uniformly and PREPARE for use. And meanwhile MAKE the reagent blank test.

5.2.4 Dry ashing

Weigh 0.5 g ~ 5 g (accurate to 0.001 g) of solid sample or accurately PIPETTE 0.500 mL ~ 10.0 mL of liquid sample into a crucible; USE small fire to heat it to carbonize it to smokeless; TRANSFER it into a muffle furnace; MAKE it subject to ashing at 550 °C for 3 h ~ 4 h. COOL it down; TAKE it out; ADD several drops of nitric acid into the sample which is not thoroughly ashed; USE small fire to heat it and EVAPORATE it dry carefully; TRANSFER it into a muffle furnace at 550 °C; CONTINUE ashing for 1 h ~ 2 h, until the sample is in white

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10 Reagents and materials

Unless otherwise specified, the reagents used in this method are excellent pure, AND the water is the level II water as specified in GB/T 6682.

10.1 Reagents

10.1.1 Nitric acid (HNO_3).

10.1.2 Perchloric acid ($HClO_4$).

10.2 Reagent preparation

10.2.1 Nitric acid solution (5 + 95): MEASURE 50 mL of nitric acid and slowly ADD it into 950 mL of water; MIX it uniformly.

10.2.2 Nitric acid solution (1 + 1): MEASURE 250 mL of nitric acid and slowly ADD it into 250 mL of water; MIX it uniformly.

10.3 Standard substance

Copper sulfate pentahydrate ($CuSO4 \cdot 5H_2O$, CAS No.: 7758-99-8): purity > 99.99%, OR the copper standard solution of a certain concentration certified and awarded with the standard substance certificate by the state.

10.4 Standard solution preparation

10.4.1 Standard copper stock solution (1000 mg/L): Accurately WEIGH 3.9289 g (accurate to 0.0001 g) of copper sulfate pentahydrate; USE a small amount of nitric acid (1 + 1) to dissolve it; TRANSFER t into a 1000 mL volumetric flask; ADD water to the mark; MIX it uniformly.

10.4.2 Copper standard intermediate solution (10.0 mg/L): Accurately PIPETTE 1.00 mL of copper standard stock solution (1000 mg/L) into a 100 mL volumetric flask; ADD nitric acid solution (5 + 95) to the mark; MIX it uniformly.

10.4.3 Copper standard series solution: respectively PIPETTE 0 mL, 1.00 mL, 2.00 mL, 4.00 mL, 8.00 mL, and 10.00 mL of copper standard intermediate solution (10.0 mg/L) into a 100 mL volumetric flask; ADD nitric acid solution (5 + 95) to the mark; MIX it uniformly. The concentration of this copper standard series solution is 0 mg/L, 0.100 mg/L, 0.200 mg/L, 0.400 mg/L, 0.800 mg/L and 1.00 mg/L, respectively.

Note: It may be based on the sensitivity of the instrument and the actual content of copper in the sample to determine the mass concentration of the copper element in the standard series solution.

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MEASURE the absorbance value after atomization; USE the mass concentration as the abscissa and the absorbance value as the ordinate, to draw the standard curve.

12.3.3 Sample determination

Under the same experimental conditions as the standard solution for measurement, the blank solution and the sample solution were respectively introduced into a flame atomizer, and the absorbance value after atomization was measured, compared with the standard series.

Under the same test conditions as the determination of standard solutions, INJECT the blank solution and sample solution, respectively, into the flame atomizer; after atomization, MEASURE its absorbance value; COMPARE it with the standard series for quantitative.

13 Presentation of analytical results

The content of copper in the sample is calculated in accordance with the formula (2).

$$X = \frac{(\rho - \rho_0) \times V}{m} \qquad \cdots\cdots (2)$$

Where:

X - The copper content in the sample, in the unit of milligrams per kilogram or milligram per liter (mg/kg or mg/L);

ρ - The mass concentration of copper in the sample solution, in micrograms per liter (μg/L);

ρ_0 - The mass concentration of copper in the blank solution, in micrograms per liter (μg/L);

V - The constant volume of the sample digestion solution, in milliliters (mL);

m - Weighed volume or pipetted volume of sample, in grams or milliliters (g or mL);

When the copper content is more than 10.0 mg/kg (or mg/L), the calculation result is retained with three significant figures; when the copper content is less than 10.0 mg/kg (or mg/L), the calculation result is retained with two significant figures.

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