GB

NATIONAL STANDARD OF

THE PEOPLE'S REPUBLIC OF CHINA

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National Food Safety Standard –

Determination of Zinc in Foods

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Issued by: National Health and Family Planning Commission of the People 's Republic of China;

State Administration of Food and Drug Administration.

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Foreword

This Standard replaces GB/T 2009.14-2003 *Determination of Zinc in Foods*, 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 9695.20-2008 *Meat and meat products - Method for determination of zinc*, 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, zine, iron, copper, manganese, chromium, lead, cadmium contents in honey - Atomic absorption spectrometry*, NY/T 1201-2006 *Determination of copper iron and zinc content in vegetables and derived products*.

Compared with GB/T 2009.14-2003, the main modifications in this Standard are as follows:

- modified the name of the standard to “National Food Safety Standard - Determination of Zinc in Foods”;
- in the pretreatment method, maintained the dry ashing method, added the wet digestion, pressure tank digestion and microwave digestion;
- preserved flame atomic absorption spectrometry as method one, dithizone colorimetric method as method four;
- added inductively coupled plasma emission spectroscopy as method two;
- added inductively coupled plasma mass spectrometry as method three;
- added microwave digestion procedures and flame atomic absorption spectrometer for instrument reference conditions as appendix.

National Food Safety Standard – Determination of Zinc in Foods

1 Scope

This Standard specifies the flame atomic absorption spectrometry method, the inductively coupled plasma emission spectroscopy method, the inductively coupled plasma mass spectrometry method and the dithizone colorimetric method in the determination of zinc in foods.

This Standard is applicable to the determination of zinc in foods, irrespective of materials.

Method One - Flame atomic absorption spectrometry

2 Principle

After digestion of the sample, the absorbance shall be measured at 213.9 nm by flame atomization. The absorbance of zinc in a certain concentration range shall be proportional to the zinc content, quantitatively compared with the standard series.

3 Reagents and materials

Unless otherwise noted, the reagents used in this method shall be guaranteed reagents, and water shall be Grade Two water specified in GB/T 6682.

3.1 Reagents

3.1.1 Nitric acid (HNO_3)

3.1.2 Perchloric acid ($HClO_4$)

3.2 Reagent preparation

3.2.1 Nitric acid solution (5+95): measure 50 mL of nitric acid; slowly add to 950 mL of water; well mix.

3.2.2 Nitric acid solution (1+1): measure 250 mL of nitric acid; slowly add to

250 mL of water; well mix.

3.3 Standard product

Zinc oxide (ZnO, CAS number: 1314-13-2): zinc standard solution with a purity >99.99%, or certified by the state and given a certain concentration of the standard substance certificate.

3.4 Standard solution preparation

3.4.1 Zinc standard stock solution (1000 mg/L): accurately weigh 1.2447 g (nearest to 0.0001g) of zinc oxide; add a small amount of nitric acid solution (1+1); heat to dissolve; after cooling, move into a 1000 mL volumetric flask; add water to the scale; well mix.

3.4.2 Zinc standard intermediate solution (10.0 mg/L): accurately pipet 1.00 mL of zinc standard stock solution (1000 mg/L) in a 100 mL volumetric flask; add nitric acid solution (5+95) to the scale; well mix.

3.4.3 Zinc standard series solutions: respectively and accurately pipet 0 mL, 1.00 mL, 2.00 mL, 4.00 mL, 8.00 mL and 10.0 mL of zinc standard intermediate solutions into 100 mL volumetric flasks; add nitric acid solution (5+95) to the scale; well mix. The concentrations of the zinc standard series solutions shall be 0 mg/L, 0.100 mg/L, 0.200 mg/L, 0.400 mg/L, 0.800 mg/L and 1.00 mg/L.

NOTE Determine the mass concentration of zinc in standard series solutions according to the sensitivity of the instrument and the actual content of zinc in the sample.

4 Apparatus

NOTE All glassware and polytetrafluoroethylene digestion tanks shall be soaked by nitric acid (1+5) overnight, repeatedly rinsed with tap water, and finally washed clean with water.

4.1 Atomic absorption spectrometer: with flame atomizer, with zinc hollow cathode lamp.

4.2 Analysis balance: resolutions of 0.1 mg and 1 mg.

4.3 Adjustable electric furnace.

4.4 Adjustable heating plate.

4.5 Microwave digestion system: with polytetrafluoroethylene digestion tank.

procedures of microwave digestion. Refer to Annex A for digestion conditions. After cooling, take out the digestion tank. Acid on the heating plate at 140°C ~ 160°C to about 1 mL. After cooling the digestion tank, move the digestion solution to a 25 mL or 50 mL volumetric flask. Wash the digestion tank with a small amount of water for 2 times to 3 times. Combine the washing solution into the volumetric flask. Use water to set the volume to the scale. Well mix for use. Carry out the reagent blank test at the same time.

5.2.3 Pressure tank digestion

Accurately weigh 0.2 g ~ 1 g (nearest to 0.001 g) of solid sample OR accurately remove 0.500 mL ~ 5.00 mL of liquid sample in the digestion tank. Add 5 mL of nitric acid. Cover the inner cover. Tighten the stainless steel jacket. Put into the constant temperature drying oven, keeping at 140°C ~ 160°C for 4h ~ 5h. After cooling, slowly spin out the outer tank and remove the digestion tank. Acid on the adjustable heating plate at 140°C ~ 160°C to about 1 mL. After cooling, transfer the digestion solution to a 25 mL ~ 50 mL volumetric flask. Wash the inner tank and inner lid with a small amount of water for 2 times to 3 times. Combine the washing solution in the volumetric flask. Use water to set the volume to the scale. Well mix for use. Carry out the reagent blank test at the same time.

5.2.4 Dry ashing

Accurately weigh 0.5 g ~ 5 g (nearest to 0.001 g) of solid sample OR accurately remove 0.500 mL ~ 10.0 mL of liquid sample in the crucible. Heat it with small fire. Carbonize to smokeless. Transfer to the muffle furnace for 3h ~ 4h ashing at 550°C. Cool and take it out. For sample with incomplete ashing, add a few drops of nitric acid. Heat it with small fire. Carefully evaporate to dry. Then transfer it to a 550°C muffle furnace. Continue ashing for 1h ~ 2h till the sample is grayish. Cool and take it out. Use an appropriate amount of nitric acid solution (1+1) to dissolve. And use water to set volume to 25 mL or 50 mL. Carry out the reagent blank test at the same time.

5.3 Determination

5.3.1 Instrument reference conditions

Adjust to the best conditions according to the performance of each instrument. Refer to Annex B for the conditions for reference.

5.3.2 Standard curve making

Introduce the zinc standard series of solutions into the flame atomizer according to the mass concentration from low to high. After atomization, measure the absorbance value. Take the mass concentration as abscissa, absorbance value as vertical axis to make the standard curve.

carbon tetrachloride. Combine the carbon tetrachloride layer to dithizone-carbon tetrachloride solution.

10.2.11 Acetic acid-acetate buffer: mix sodium acetate solution (2 mol/L) and acetic acid solution (2 mol/L) in same volume; the pH of this solution shall be around 4.7. Use dithizone-carbon tetrachloride solution (0.1 g/L) to extract several times, 10 mL for each time. Remove the zinc till the carbon tetrachloride layer remains green. Discard the carbon tetrachloride layer. And use carbon tetrachloride to extract the excess dithizone in acetic acid-acetate buffer till carbon tetrachloride is colorless. Discard the carbon tetrachloride layer.

10.2.12 Hydroxylamine Hydrochloride Solution (200 g/L): weigh 20 g of hydroxylamine hydrochloride; add 60 mL of water; add aqueous solution of ammonia (1+1) in drops; adjust pH to 4.0 ~ 5.5; add water to 100 mL. Use dithizone-carbon tetrachloride solution (0.1 g/L) to extract several times, 10 mL for each time. Remove the zinc till the carbon tetrachloride layer remains green. Discard the carbon tetrachloride layer. And use carbon tetrachloride to extract the excess dithizone in acetic acid-acetate buffer till carbon tetrachloride is colorless. Discard the carbon tetrachloride layer.

10.2.13 Sodium thiosulfate solution (250 g/L): weigh 25 g of sodium thiosulfate; add 60 mL of water; use acetic acid solution (2 mol/L) to adjust pH to 4.0 ~ 5.5; add water to 100 mL. Use dithizone-carbon tetrachloride solution (0.1 g/L) to extract several times, 10 mL for each time. Remove the zinc till the carbon tetrachloride layer remains green. Discard the carbon tetrachloride layer. And use carbon tetrachloride to extract the excess dithizone in acetic acid-acetate buffer till carbon tetrachloride is colorless. Discard the carbon tetrachloride layer.

10.2.14 Dithizone use solution: pipet 1.0 mL of dithizone-carbon tetrachloride solution (0.1 g/L); add carbon tetrachloride to 10.0 mL; mix well. Use a 1 cm cuvette to adjust the zero point with carbon tetrachloride. Measure the absorbance (A) at a wavelength of 530 nm. Use equation (2) to calculate the amount of dithizone-carbon tetrachloride solution (0.1 g/L) ml (V) required by the preparation of 100 mL of dithizone use solution (57% transmittance). Measure the calculated amount of dithizone-carbon tetrachloride solution (0.1 g/L). Use carbon tetrachloride to dilute it to 100 mL.

$$V = \frac{10 \times (2 - \lg 57)}{A} = \frac{2.44}{A} \qquad \cdots\cdots (2)$$

10.2.15 Phenol red indicator solution (1 g/L): weigh 0.1 g of phenol red; use ethanol to dissolve and set volume to 100 mL; mix well.

10.3 Standard product

12.3.2 Standard curve making

Accurately pipet 0 mL, 1.00 mL, 2.00 mL, 3.00 mL, 4.00 mL and 5.00 mL of zinc standard use solution (equivalent to 0 μg, 1.00 μg, 2.00 μg, 3.00 μg, 4.00 μg and 5.00 μg of zinc). Respectively place in a 125 mL separatory funnel. Add respectively hydrochloric acid solution (0.02 mol/L) to 20 mL. In each separatory funnel, respectively add 10 mL of acetic acid-acetate buffer, 1 mL of sodium thiosulfate solution (250 g/L). Mix well. Then respectively add 10 mL of dithizone use solution. Vigorously shake for 2 min. After still placement and layering, filter the carbon tetrachloride layer into a 1 cm cuvette via absorbent cotton. Adjust the zero point with carbon tetrachloride. Measure the absorbance at a wavelength of 530 nm. Take the mass as abscissa, absorbance value as vertical axis to make the standard curve.

12.3.3 Sample determination

Accurately pipet 5.00 mL ~ 10.0 mL sample digestion solution and the same volume of blank digestion solution. Respectively place in a 125 mL separatory funnel. Add 5 mL of water, 0.5 mL of hydroxylamine hydrochloride solution (200 g/L). Well mix. Then add 2 drops of phenol red indicator solution (1 g/L). Adjust with ammonia solution (1+1) to red. Add 2 drops. Add 5 mL of dithizone-carbon tetrachloride solution (0.1 g/L). Vigorously shake for 2 min. Place still for layering. Transfer the carbon tetrachloride layer into another separatory funnel. The water layer shall be extracted with a small amount of dithizone-carbon tetrachloride solution (0.1 g/L) while shaking, 2 mL ~ 3 mL for each time, till dithizone-carbon tetrachloride solution (0.1 g/L) remains green. Merge the extract. Use 5 mL of water to rinse. The carbon tetrachloride layer shall be extracted twice with hydrochloric acid solution (0.02 mol/L), 10 mL for each time. Vigorously shake for 2 min while extracting. Merge the hydrochloric acid solution (0.02 mol/L) extract. And wash the residual dithizone with a small amount of carbon tetrachloride.

Transfer the above sample extract and blank extract to a 125 mL separatory funnel. Respectively add 10 mL of acetic acid-acetate buffer, 1 mL of sodium thiosulfate solution (250 g/L). Mix well. Then respectively add 10 mL of dithizone use solution. Vigorously shake for 2 min. After still placement and layering, filter the carbon tetrachloride layer into a 1 cm cuvette via absorbent cotton. Adjust the zero point with carbon tetrachloride. Measure the absorbance at a wavelength of 530 nm. Quantitatively compare with the standard series.

13 Expression of analysis results

The zinc content in the sample is calculated according to equation (3):

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