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Determination of Total Arsenic in Feeds

饲料中总砷的测定

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Table of Contents

Determination of Total Arsenic in Feeds

1 Scope

This document describes the silver salt method, hydride generation-atomic fluorescence spectrometry and inductively coupled plasma mass spectrometry for the determination of total arsenic in feeds.

This document is applicable to the determination of total arsenic in compound feeds, concentrated feeds, concentrate supplements, additive premixed feeds, feedstuffs and feed additives.

When the sampling size is 5 g and the constant volume is 50 mL, the detection limit of the silver salt method is 0.25 mg/kg and the quantitation limit is 0.50 mg/kg; when the sampling size is 5 g, the constant volume is 50 mL and the dilution factor is 20, the detection limit of the hydride generation-atomic fluorescence spectrometry and inductively coupled plasma mass spectrometry is 0.02 mg/kg, and the quantitation limit is 0.05 mg/kg.

2 Normative References

The contents of the following documents constitute indispensable clauses of this document through the normative references in the text. In terms of references with a specified date, only versions with a specified date are applicable to this document. In terms of references without a specified date, the latest version (including all the modifications) is applicable to this document.

GB/T 6682 Water for Analytical Laboratory Use - Specification and Test Methods

GB/T 20195 Animal Feed - Preparation of Test Samples

3 Terms and Definitions

This document does not have terms or definitions that need to be defined.

4 Silver Salt Method (arbitration method)

4.1 Principle

After a specimen is treated by acid digestion, direct acid dissolution or dry ashing method, use potassium iodide and stannous chloride to reduce high-valent arsenic to trivalent arsenic, then, generate arsine with the new ecological hydrogen produced by zinc particles and acid, which is absorbed by the silver diethyldithiocarbamate (Ag-DDTC)-triethylamine-chloroform solution to form a red jelly, whose color shade is proportional to the arsenic content. Use a spectrophotometer to determine the absorbance and quantitatively compare it with standard

series of solutions.

4.2 Reagents or Materials

WARNING---handle all kinds of strong acids with caution. Dilute and use them in a fume hood. When using perchloric acid, be careful not to burn it to dryness, and be careful of explosion.

Unless otherwise specified, use only analytically pure reagents.

4.2.1 Water: GB/T 6682, Grade-2.

4.2.2 Nitric acid.

4.2.3 Hydrochloric acid.

4.2.4 Chloroform.

4.2.5 Magnesium oxide.

4.2.6 Magnesium nitrate.

4.2.7 L-ascorbic acid.

4.2.8 Arsenic-free zinc particles: with a particle size of 3.0 mm ± 0.2 mm.

4.2.9 Mixed acid solution: nitric acid + perchloric acid + sulfuric acid = 230 + 50 + 30. Mix it well and store it in a brown reagent bottle.

4.2.10 Hydrochloric acid solution (6 mol/L): measure-take 250 mL of hydrochloric acid (4.2.3), add 250 mL of water and mix it well.

4.2.11 Magnesium nitrate solution: weigh-take 180 g of magnesium nitrate [$Mg(NO_3)_2 \bullet 6H_2O$], add water to dissolve it, dilute and reach a constant volume of 1,000 mL, and mix it well.

4.2.12 Potassium iodide solution (150 g/L): weigh-take 75 g of potassium iodide, add water to dissolve it, dilute to 500 mL, mix it well, and store in a brown bottle.

4.2.13 Acidic stannous chloride solution: weigh-take 40 g of stannous chloride ($SnCl_2 \bullet 2H_2O$), use 100 mL of hydrochloric acid (4.2.3) to dissolve it, then, add a few tin particles, and store in a brown reagent bottle. The shelf life is 1 week.

4.2.14 Silver diethyldithiocarbamate (Ag-DDTC)-triethylamine-chloroform solution (2.5 g/L): weigh-take 2.5 g (accurate to 0.0001 g) of Ag-DDTC in a dry beaker, add an appropriate amount of chloroform. After it is completely dissolved, add 20 mL of triethylamine, use chloroform to transfer it to a 1,000 mL volumetric flask, reach a constant volume and mix it well; store it in a brown reagent bottle and in the dark at 2 °C ~ 8 °C. If there is precipitation, it shall be filtered before use.

4.2.15 Lead acetate solution (200 g/L): weigh-take 40 g of lead acetate, add water to dissolve it, dilute to 200 mL and mix it well.

4.2.16 Lead acetate cotton: soak the medical absorbent cotton in the lead acetate solution (4.2.15) for 1 hour, squeeze out the excess lead acetate solution, make it loose, and naturally dry it, or dry it at 90 °C ~ 100 °C, and store in an airtight bottle.

4.2.17 Sodium hydroxide solution (200 g/L): weigh-take 200 g of sodium hydroxide, add 500 mL of water to dissolve it, then, use water to dilute to 1,000 mL and mix it well.

4.2.18 Sulfuric acid solution (6%): measure-take 60 mL of sulfuric acid, slowly add 500 mL of water. After cooling, use water to dilute to 1,000 mL, and mix it well.

4.2.19 Arsenic standard stock solution (0.1 mg/mL, calculated by arsenic): accurately weigh-take 0.1320 g (accurate to 0.0001 g) of arsenic trioxide (CAS: 1327-53-3, purity ≥ 99.5%) that has been dried at 100 °C for 2 hours, and add 5 mL of sodium hydroxide solution (4.2.17) to dissolve it, then, add 25 mL of sulfuric acid solution (4.2.18) and transfer to a 1,000 mL volumetric flask. Use newly boiled and cooled water to reach a constant volume and mix it well. Store it in a plastic bottle and in the dark at 2 °C ~ 8 °C. The shelf life is 12 months. Alternatively, purchase certified reference material of arsenic.

4.2.20 Arsenic standard intermediate solution (1 μg/mL): accurately transfer-take 1 mL of arsenic standard stock solution (4.2.19) into a 100 mL volumetric flask, add 1 mL of hydrochloric acid solution (4.2.10), use water to reach a constant volume and mix it well. Store it in a plastic bottle and in the dark at 2 °C ~ 8 °C. The shelf life is 1 month.

4.3 Instruments and Equipment

4.3.1 Spectrophotometer: with a wavelength accuracy of ± 2 nm.

4.3.2 Analytical balance: with an accuracy of 0.0001 g.

4.3.3 Electric drying oven: with a temperature control accuracy of ± 5 °C.

4.3.4 Temperature controllable electric heating plate: with a temperature control accuracy of ± 5 °C.

4.3.5 Muffle furnace: with a temperature control accuracy of ± 15 °C.

4.3.6 Graphite digestion instrument: equipped with matching polytetrafluoroethylene digestion tube.

4.3.7 Arsine generation and absorption device (see Figure 1): consists of a 125 mL arsenic reaction bottle with graduations, a 5 mL arsenic absorption tube, and an air guide tube connecting the arsenic reaction bottle and the arsenic absorption tube. The arsenic reaction bottle, air guide tube and arsenic absorption tube are connected with ground joints to ensure no air leakage.

4.5.1 Preparation of specimen solution

4.5.1.1 Mixed acid digestion method

Carry out two tests in parallel. Respectively weigh-take 3 g ~ 5 g of compound feed, concentrate supplement, and animal and vegetable feedstuff; respectively weigh-take 2 g ~ 3 g of concentrated feed, additive premixed feed containing organic matter, feed additive and attapulgite powder specimen, accurate to 0.001 g. Place it in a 250 mL conical flask, add a small amount of water to moisten the specimen, add 25 mL of mixed acid solution (4.2.9), mix it well, then, cover the funnel and let it stand overnight. Remove the funnel, transfer it to an adjustable electric furnace and heat it for digestion at 150 °C. After the brown gas disappears and the solution becomes clear, raise the temperature to 200 °C and continue digestion, until the smoke of perchloric acid dissipates and the white smoke of sulfuric acid beings to emerge. Immediately take it off and cool it. If there are still undecomposed substances or if the color is relatively dark, add 5 mL ~ 10 mL of nitric acid (4.2.2), continue heating and digestion, and repeat 2 ~ 3 times. Be careful to avoid carbonization, until the specimen is completely digested. Add 25 mL of water, then evaporate it, until the sulfuric acid emits white smoke. Remove it, cool it to room temperature, and use water to transfer to a 50 mL volumetric flask. Use a small amount of water to rinse the conical flask for 3 ~ 5 times, merge the washing liquid into the volumetric flask, reach a constant volume, mix it well and filter it. The filtrate is the specimen solution. Meanwhile, carry out a blank test.

4.5.1.2 Dry ashing method

4.5.1.2.1 Compound feeds, concentrated feeds, concentrate supplements, animal and vegetable feedstuffs (except marine feedstuff), additive premixed feeds containing organic matter and feed additives

Carry out two tests in parallel. Respectively weigh-take 3 g ~ 5 g of compound feed, concentrate supplement, and animal and vegetable feedstuff (except marine feedstuff); respectively weigh-take 2 g ~ 3 g of concentrated feed, additive premixed feed containing organic matter and feed additive specimen, accurate to 0.001 g. Place it in a 50 mL porcelain crucible, add 10 mL of magnesium nitrate solution (4.2.11) and 1 g of magnesium oxide (4.2.5), mix it well and soak for 4 hours. After evaporating it to dryness at low temperature on an electric heating plate at 100 °C (or drying in an electric drying oven at 105 °C), transfer it to an electric furnace to carbonize, until it becomes smokeless, then, transfer to a muffle furnace at 550 °C and ash for 4 hours. Take it out, cool to room temperature, slowly add 10 mL of hydrochloric acid solution (4.2.10), and heat and dissolve it, until the solution becomes clear. Then, transfer it to a 50 mL volumetric flask, use a small amount of water to rinse the crucible 3 ~ 5 times, and merge the washing liquid into the volumetric flask. Reach a constant volume, shake it well, and filter it. The filtrate is the specimen solution. Meanwhile, carry out a blank test.

4.5.1.2.2 Animal and vegetable oil feedstuffs

Carry out two tests in parallel. Weigh-take 5 g of animal and vegetable oil feedstuff specimen (accurate to 0.001 g), place it in a 50 mL porcelain crucible, add 10 g of magnesium nitrate

(4.2.6), 2 g of magnesium oxide (4.2.5), mix it well, and transfer onto an electric furnace, and heat it over low heat, until it emits smoke. Immediately remove the crucible to prevent the contents from overflowing. After the oily fume dissipates, then carbonize it, until it becomes smokeless. Then, transfer to a 550 °C muffle furnace for ashing for 4 hours. Take it out and cool, and slowly add 10 mL of hydrochloric acid solution (4.2.10). Heat and dissolve it, until the solution becomes clear. Then, transfer to a 50 mL volumetric flask, use a small amount of water to rinse the crucible 3 ~ 5 times, merge the washing liquid into the volumetric flask. Reach a constant volume, mix it well and filter it. The filtrate is the specimen solution. Meanwhile, carry out a blank test.

4.5.1.3 Direct acid dissolution method

Carry out two tests in parallel. Respectively weigh-take 1 g ~ 3 g of mineral feedstuff, trace element premixed feed without organic matter, and mineral element feed additive specimen without complexes (chelates), accurate to 0.0001 g, place into 150 mL conical flask, and slowly add 10 mL of hydrochloric acid solution (4.2.10) and 1 mL of nitric acid (4.2.2) to dissolve it. After the reaction, transfer it to an electric stove or electric heating plate for heating and digestion. When the solution becomes clear and has a volume of about 5 mL, remove it, cool to room temperature, transfer to a 50 mL volumetric flask, and use a small amount of water to rinse the conical flask for 3 ~ 5 times. Merge the washing liquid into the volumetric flask, reach a constant volume, shake it well and filter it. The filtrate is the specimen solution. Meanwhile, carry out a blank test.

4.5.2 Determination

4.5.2.1 Preparation of standard series of solutions

Accurately transfer-take 0.00 mL, 1.00 mL, 2.00 mL, 4.00 mL, 6.00 mL, 8.00 mL and 10.00 mL of the arsenic standard intermediate solution (4.2.20), respectively place them in arsenic reaction bottle, and use water to dilute to 30 mL. Add 10 mL of hydrochloric acid (4.2.3), mix it well, and follow the steps specified in 4.5.2.2, starting from the addition of 2 mL of potassium iodide solution (4.2.12).

4.5.2.2 Reduction reaction

In accordance with the total arsenic content in the specimen, accurately transfer-take 2 mL ~ 20 mL of specimen solution (4.5.1) into the arsenic reaction bottle, add hydrochloric acid (4.2.3) to a total volume of 10 mL, and use water to dilute to 40 mL, so that the hydrochloric acid concentration of the solution is 3 mol/L, then, respectively add 2 mL of potassium iodide solution (4.2.12) to the specimen solution, blank solution and the standard series of solutions, mix well. Add 1 mL of acidic stannous chloride solution (4.2.13), 0.2 g of L-ascorbic acid (4.2.7) and mix it well. Let it stand for 15 minutes of the reduction reaction, and reserve it for testing.

Accurately transfer-take 5 mL of silver diethyldithiocarbamate (Ag-DDTC)-triethylamine-chloroform solution (4.2.14) into the arsenic absorption tube, insert the tip of the air guide tube of the reaction device [the expanded part of the tube is stuffed with fluffy lead acetate cotton (4.2.16)] into the arsenic absorption tube, and quickly add 4 g of arsenic-free zinc particles

5.2.7 Magnesium nitrate.

5.2.8 Potassium hydroxide: guaranteed reagent.

5.2.9 Potassium borohydride: guaranteed reagent.

5.2.10 Mixed acid solution: nitric acid (5.2.2) + perchloric acid + sulfuric acid (5.2.4) = 230 + 50 + 30, mix well and store in a brown reagent bottle.

5.2.11 Hydrochloric acid solution (6 mol/L): measure-take 250 mL of hydrochloric acid (5.2.3) and 250 mL of water, and evenly mix it.

5.2.12 Potassium hydroxide solution (8 g/L): weigh-take 8 g of potassium hydroxide (5.2.8), add water to dissolve it, dilute and reach a constant volume of 1,000 mL, and mix it well. Prepare it right before use.

5.2.13 Potassium borohydride solution (20 g/L): weigh-take 20 g of potassium borohydride (5.2.9), dissolve it in 1,000 mL of potassium hydroxide solution (5.2.12), and mix it well. Prepare it right before use.

5.2.14 Magnesium nitrate solution (180 g/L): weigh-take 180 g of magnesium nitrate [$Mg(NO_3)_2 \bullet 6H_2O$], add water to dissolve it, dilute and reach a constant volume of 1,000 mL, and mix it well.

5.2.15 Thiourea-ascorbic acid solution (100 g/L): weigh-take 100 g of thiourea and 100 g of L-ascorbic acid, add water to dissolve it, reach a constant volume of 1,000 mL and mix it well. Prepare it right before use.

5.2.16 Ammonium thiocyanate-hydroxylamine hydrochloride solution (100 g/L): weigh-take 100 g of ammonium thiocyanate and 100 g of hydroxylamine hydrochloride, add water to dissolve it, reach a constant volume of 1,000 mL and mix it well. Prepare it right before use.

5.2.17 Sodium hydroxide solution (200 g/L): weigh-take 200 g of sodium hydroxide, add water to dissolve it, reach a constant volume of 1,000 mL and mix it well.

5.2.18 Sulfuric acid solution (6%): measure-take 60 mL of sulfuric acid (5.2.4), slowly add it to 500 mL of water. After cooling, use water to dilute to 1,000 mL and mix it well.

5.2.19 Arsenic standard stock solution (0.1 mg/mL): accurately weigh-take 0.1320 g (accurate to 0.0001 g) of arsenic trioxide (CAS: 1327-53-3, purity ≥ 99.5%) that has been dried at 100 °C for 2 hours, and add 5 mL of sodium hydroxide solution (5.2.17) to dissolve it, then, add 25 mL of sulfuric acid solution (5.2.18) and transfer to a 1,000 mL volumetric flask. Use newly boiled and cooled water to reach a constant volume and mix it well. Store it in a plastic bottle and in the dark at 2 °C ~ 8 °C. The shelf life is 12 months. Alternatively, purchase certified reference material of arsenic.

5.2.20 Arsenic standard intermediate solution (1 μg/mL): accurately transfer-take 1 mL of arsenic standard stock solution (5.2.19) into a 100 mL volumetric flask, add 1 mL of

hydrochloric acid solution (5.2.11), use water to reach a constant volume and mix it well. Store it in a plastic bottle and in the dark at 2 °C ~ 8 °C. The shelf life is 1 month.

5.2.21 Arsenic standard working solution (100 ng/mL): accurately transfer-take 10 mL of arsenic standard intermediate solution (5.2.20) into a 100 mL volumetric flask, add 1 mL of hydrochloric acid solution (5.2.11), use water to dilute it and reach a constant volume, and mix it well. The shelf life is 1 week.

5.3 Instruments and Equipment

5.3.1 Atomic fluorescence spectrophotometer.

5.3.2 Analytical balance: with an accuracy of 0.0001 g.

5.3.3 Muffle furnace: with a temperature control accuracy of ± 15 °C.

5.3.4 Electric drying oven: with a temperature control accuracy of ± 5 °C.

5.3.5 Adjustable electric furnace.

5.3.6 Graphite digestion instrument: equipped with matching polytetrafluoroethylene digestion tube.

5.3.7 Microwave digestion instrument: equipped with matching polytetrafluoroethylene digestion tube.

5.3.8 High-pressure digestion tank: equipped with matching polytetrafluoroethylene digestion inner tube.

5.3.9 Acid flushing instrument: with a temperature control accuracy of ± 2 °C.

5.4 Sample

Same as 4.4.

5.5 Test Procedures

5.5.1 Preparation of specimen solution

WARNING---when using microwave digestion instrument and high-pressure digestion tube to digest samples, strong acid, high temperature and high pressure may easily cause unsafe incidents. Operators shall rigorously follow the instrument operation instructions in accordance with the type of samples to ensure personal and laboratory safety.

5.5.1.1 Mixed acid digestion method

Same as 4.5.1.1.

5.5.1.2 Dry ashing method

water to transfer it to a 50 mL volumetric flask, and use a small amount of water to rinse the conical flask for 3 times. Merge the washing liquid into the volumetric flask, reach a constant volume, shake it well and filter it. The filtrate is the specimen solution. Meanwhile, carry out a blank test.

Carry out two tests in parallel. Respectively weigh-take 2 g ~ 3 g (accurate to 0.001 g) of additive premixed feed and feed additive specimen with copper sulfate, basic copper chloride and copper content exceeding 0.8%, and place in a 150 mL conical flask. Slowly add 10 mL of hydrochloric acid solution (5.2.11) and 1 mL of nitric acid (5.2.2). After the reaction, transfer it to an electric stove or electric heating plate for heating and digestion. When the solution reaches about 5 mL, remove it and cool to room temperature. Use water to transfer it to a 50 mL volumetric flask, add 10 mL of ammonium thiocyanate-hydroxylamine hydrochloride (5.2.16), and use a small amount of water to rinse the conical flask for 3 ~ 5 times. Merge the washing liquid into the volumetric flask, reach a constant volume, mix it well, let it stand for 30 minutes and filter it. Take the filtrate as the specimen solution. Meanwhile, carry out a blank test.

5.5.2 Preparation of arsenic standard series of solutions

Respectively and accurately transfer-take an appropriate volume or arsenic standard working solution (5.2.21) into a 50 mL volumetric flask, add water to about 40 mL, add 2.5 mL of hydrochloric acid (5.2.3) and mix it well. Slowly add 5 mL of thiourea-ascorbic acid solution (5.2.15), add water to dilute to a constant volume and mix it well. Thus, the arsenic standard series of solutions respectively with a concentration of 0 ng/mL, 0.50 ng/mL, 1.00 ng/mL, 4.00 ng/mL, 8.00 ng/mL and 16.00 ng/mL are prepared.

5.5.3 Reduction reaction

In accordance with the total arsenic content in the specimen, accurately transfer-take 5 mL ~ 20 mL of specimen solution (5.5.1) into a 50 mL volumetric flask, add water to about 40 mL, then, add 2.5 mL of hydrochloric acid (5.2.3) and mix it well. Slowly add 5 mL of thiourea-ascorbic acid solution (5.2.15), add water to reach a constant volume, and mix it well. After simultaneous reduction reaction with the arsenic standard series of solutions for 30 minutes, reserve it for testing.

5.5.4 Instrument reference conditions

The instrument reference conditions are as follows:

a) Negative high voltage: 200 V ~ 400 V;

b) Arsenic hollow cathode lamp current: 15 mA ~ 100 mA;

c) Atomizer temperature: 200 °C;

d) Atomizer height: 8 mm;

e) Carrier gas flow: 600 mL/min;

6.2.8 Hydrochloric acid solution (6 mol/L): measure-take 250 mL of hydrochloric acid (6.2.3) and 250 mL of water, and evenly mix it.

6.2.9 Magnesium nitrate solution (180 g/L): weigh-take 180 g of magnesium nitrate [$Mg(NO_3)_2 \cdot 6H_2O$], add water to dissolve it, dilute and reach a constant volume of 1,000 mL, and mix it well.

6.2.10 Ammonium thiocyanate-hydroxylamine hydrochloride solution (100 g/L): weigh-take 100 g of ammonium thiocyanate and 100 g of hydroxylamine hydrochloride, add water to dissolve it, reach a constant volume of 1,000 mL and mix it well. Prepare it right before use.

6.2.11 Nitric acid solution (5%): measure-take 50 mL of nitric acid (6.2.2), slowly add it to an appropriate amount of water, use water to reach a constant volume of 1,000 mL and mix it well.

6.2.12 Sodium hydroxide solution (200 g/L): weigh-take 200 g of sodium hydroxide, add water to dissolve it, reach a constant volume of 1,000 mL and mix it well.

6.2.13 Sulfuric acid solution (6%): measure-take 60 mL of sulfuric acid (6.2.4), slowly add it to 500 mL of water. After cooling, use water to dilute to 1,000 mL and mix it well.

6.2.14 Arsenic standard stock solution (0.1 mg/mL): accurately weigh-take 0.1320 g (accurate to 0.0001 g) of arsenic trioxide (CAS: 1327-53-3, purity ≥ 99.5%) that has been dried at 100 °C for 2 hours, and add 5 mL of sodium hydroxide solution (6.2.12) to dissolve it, then, add 25 mL of sulfuric acid solution (6.2.13) and transfer to a 1,000 mL volumetric flask. Use newly boiled and cooled water to reach a constant volume and mix it well. Store it in a plastic bottle and in the dark at 2 °C ~ 8 °C. The shelf life is 12 months. Alternatively, purchase certified reference material of arsenic.

6.2.15 Arsenic standard intermediate solution (1 μg/mL): accurately transfer-take 1 mL of arsenic standard stock solution (6.2.14) into a 100 mL volumetric flask, add 1 mL of hydrochloric acid solution (6.2.8), use water to reach a constant volume and mix it well. Store it in a plastic bottle and in the dark at 2 °C ~ 8 °C. The shelf life is 1 month.

6.2.16 Arsenic standard working solution (100 ng/mL): accurately transfer-take 10 mL of arsenic standard intermediate solution (6.2.15) into a 100 mL volumetric flask, add 1 mL of hydrochloric acid solution (6.2.8), use water to dilute it and reach a constant volume, and mix it well. The shelf life is 1 week.

6.2.17 Internal standard working solution: transfer-take an appropriate amount of germanium (^{72}Ge) standard solution, use nitric acid solution (6.2.11) to dilute it to 100 ng/mL. The shelf life is 3 months.

6.2.18 Arsenic standard series of solutions: accurately transfer-take an appropriate amount of arsenic standard working solution (6.2.16), use nitric acid solution (6.2.11) to respectively prepare arsenic concentrations of 0.0 ng/mL, 5.0 ng/mL, 10.0 ng/mL, 20.0 ng/mL, 25.0 ng/mL and 50.0 ng/mL, mix them well. Prepare them right before use.

6.2.19 Argon gas: with a purity ≥ 99.999%.

6.3 Instruments and Equipment

6.3.1 Inductively coupled plasma mass spectrometer.

6.3.2 Analytical balance: with an accuracy of 0.0001 g.

6.3.3 Muffle furnace: with a temperature control accuracy of ± 15 °C.

6.3.4 Electric drying oven: with a temperature control accuracy of ± 5 °C.

6.3.5 Adjustable electric furnace.

6.3.6 Graphite digestion instrument: equipped with matching polytetrafluoroethylene digestion tube.

6.3.7 Microwave digestion instrument: equipped with matching polytetrafluoroethylene digestion tube.

6.3.8 High-pressure digestion tank: equipped with matching polytetrafluoroethylene digestion inner tube.

6.3.9 Acid flushing instrument: with a temperature control accuracy of ± 2 °C.

6.3.10 Temperature controllable electric heating plate: with a temperature control accuracy of ± 5 °C.

6.4 Sample

Same as 4.4.

6.5 Test Procedures

6.5.1 Preparation of specimen solution

6.5.1.1 Dry ashing method

Same as 4.5.1.2.

6.5.1.2 Microwave digestion method

Same as 5.5.1.3.

6.5.1.3 High-pressure tank digestion method

Same as 5.5.1.4.

6.5.1.4 Direct acid dissolution method

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