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Test Method for Analysis of Coal Ash

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Standardization Administration (SAC) of the People's Republic of China.**

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Foreword

This standard replaces GB/T 1574-1995 *Analysis of Coal Ash*, GB/T 4634-1996 *Determination of Potassium, Sodium, Iron, Calcium, Magnesium and Manganese in Coal Ash by Atomic Absorption Spectrometric Method,* and GB/T 18856.13-2002 *Test Methods for Quality of Coal Water Mixture - Part 13: Determination for the Ash of Coal Water Mixture*. This standard integrates the 3 standards - GB/T 1574-1995, GB/T 4634-1996 and GB/T 18856.13-2002.

Compared with GB/T 1574-1995, GB/T 4634-1996 and GB/T 18856.13-2002, the main changes of this standard are as follows:

— The volume of standard working solution of titanium dioxide was modified (6.3.3.1 in GB/T 1574-1995; 6.3.3.1.1 in this edition);

— A method to separately determine titanium dioxide was added (see 6.4);

— Wrong units were modified (9.1.2.10 and 9.1.2.11 in GB/T 1574-1995; 9.2.2.10 and 9.2.2.11 in this edition);

— Errors in formula were modified (9.2.7 in GB/T 1574-1995; Formula 26 in 9.2.4 of this edition);

— Content of "note" was added (see 11.2);

— Appendix A in the former standard (GB/T 4634-1996) was deleted.

This standard was proposed by the China National Coal Association.

This standard shall be under the jurisdiction of National Technical Committee on Coal of Standardization Administration of China.

Drafting organization of this standard: the Coal Analysis Laboratory of Coal Research Institute.

Chief drafting staffs of this standard: Zhang Kerui, Xia Huili, Chen Suize, Deng Xiumin, Zhou Guoyue, Wang Zhiqian, and Sui Yan.

The previous editions replaced by this standard are as follows:

- GB 1574-1979, GB/T 1574-1995;

- GB/T 4634-1984, GB/T 4634-1996;

- GB/T 18856.13-2002.

Test Method for Analysis of Coal Ash

1 Scope

This standard specifies the reagents and materials, instruments and equipment, analysis procedure, result calculation and method precision for the determination of ferrum, calcium, magnesium, potassium, sodium, manganese, phosphorus, silicon, aluminum, titanium and sulphur in coal ash.

This standard is applicable to coal, coke, coal water slurry and coal gangue.

2 Normative References

The following documents contain provisions which, through reference in this text, constitute provisions of this standard. For dated references, subsequent amendments (excluding correction) or revisions of these publications do not apply. However, the parties who enter into agreement according to these specifications are encouraged to research whether the latest editions of these labels are applicable. For any undated references, the latest edition of the document referred to applies.

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3 Reagents

Water mentioned in this standard is deionized water or distilled water with equivalent purity.

4 Instruments and Equipment

4.1 Muffle furnace: with temperature regulating device, thermocouple, and pyrometer; it can maintain at (815 ± 10) °C and shall have corresponding constant-temperature area; upper part of its back wall is chimney with diameter of 25mm~30mm, lower part has a hole (for insertion of thermocouple) whose position shall be such that the heat contact-point of the thermocouple can be 20mm~30mm away from the furnace bottom; a ventilation hole (diameter: about 20mm) shall be on the furnace door.

4.2 High-temperature muffle furnace: with temperature control device; it can maintain at (1000±10)°C.

6.3 Continuous Determination of Ferric Oxide and Titanium Dioxide (Spectrophotometric Method with Ferrotitanium Reagent)

6.3.1 Method summary

Under the condition of pH= 4.7~4.9, ferric ion reacts with ferrotitanium reagent to generate purple complex; use spectrophotometric method to determine ferric oxide. Add adequate amount of ascorbic acid, so that purple of the solution disappears; titanic ion reacts with ferrotitanium reagent to generate yellow complex, use spectrophotometric method to determine titanium dioxide.

6.3.2 Reagents and materials

6.3.2.1 Ascorbic acid.

6.3.2.2 Ferrotitanium reagent solution: 20 g/L. Weigh 2 g of ferrotitanium reagent ($C_6H_4O_8S_2Na_2$); dissolve it into water; dilute to 100 mL with water.

6.3.2.3 Ammonia water (GB/T 631): volume ratio is (1+6).

6.3.2.4 Hydrochloric acid (GB/T 622) solution: volume ratio is (1 + 19).

6.3.2.5 Sulfuric acid solution: volume fraction is 50mL/L. Measure 5 mL of sulfuric acid (GB/T 625); add water slowly; dilute to 100 mL with water.

6.3.2.6 Buffer solution: pH = 4.7. Weigh 68 g of sodium acetate trihydrate ($CH_3COONa•3H_2O$) (GB/T 693) or 41 g of anhydrous sodium acetate (CH_3COONa) (GB/T 694); place into a 400 mL beaker; add water to dissolve; add 29 mL of glacial acetic acid (GB/T 676); dilute to 1 L with water.

6.3.2.7 Standard stock solution of ferric oxide: 1 mg/mL.

Accurately weigh 1.0000g (accurate to 0.0002g) of ferric oxide GR which has been dried at 105°C~110°C for 1 h; place it in a 400 mL beaker; add 50mL of concentrated hydrochloric acid (GB/T 622, GR); put on watch-glass; heat it until they are dissolved; cool down to room temperature; transfer into a 1 L volumetric flask; dilute to the mark with water; shake well.

6.3.2.8 Standard working solution of ferric oxide: 0.1 mg/mL.

Accurately pipet 10mL of standard stock solution of ferric oxide; place into a 100mL volumetric flask; dilute to the mark with the hydrochloric acid solution (6.3.2.4); shake well.

6.3.2.9 Standard stock solution of titanium dioxide: 1mg/mL.

Accurately weigh 0.5000g (accurate to 0.0002g) of titanium dioxide GR; burn at 1000°C for 30min; put it in a 30 mL porcelain crucible; add 8g of potassium

6.5 Determination of Aluminum Oxide (Complexometric Titration with Villiaumite Replacing EDTA)

6.5.1 Method summary

Add excessive EDTA solution into weak-acidic solution, so as to let it to complex with ferrum ion, aluminum ion and titanium ion, etc.; under the condition of pH = 5.9, use xylenol orange as indicator; use zinc salt to drop-add the remaining EDTA solution; then add villiaumite to replace the EDTA that complexes with aluminum and titanium; use zinc acetate standard solution to titrate; deduct the amount of titanium, so as to obtain the amount of aluminum.

6.5.2 Reagents

6.5.2.1 EDTA solution: 11 g/L. Weigh 1.1 g of EDTA ($C_{10}H_{14}N_2O_8Na_2 \cdot 2H_2O$) (GB/T 1401); dissolve it into water; and dilute to 100 mL with water.

6.5.2.2 Buffer solution: pH = 5.9. Weigh 200 g of sodium acetate trihydrate ($CH_3COONa \cdot 3H_2O$) (GB/T 693) or 120.6 g of anhydrous sodium acetate (CH_3COONa); dissolve it into water; add 6.0 mL of glacial acetic acid (GB/T 676); dilute to 1000 mL with water.

6.5.2.3 Zinc acetate: 20 g/L. Weigh 2 g of zinc acetate [$Zn(CH_3COO)_2 \cdot 2H_2O$] (HG 3-1098); dissolve it into water; dilute to 100 mL with water.

6.5.2.4 Potassium fluoride solution: 100 g/L. Weigh 10 g of potassium fluoride ($KF \cdot 2H_2O$)(GB/T 1271); dissolve it into water; dilute to 100 mL with water; store it into a polyethylene bottle.

6.5.2.5 Glacial acetic acid (GB/T 676) solution: volume ratio is (1+3).

6.5.2.6 Ammonia water (GB/T 631) solution: volume ratio is (1+1).

6.5.2.7 Standard working solution of aluminum oxide: 1 mg/mL.

Place spectral-pure aluminum sheet into a beaker; use (1+9) hydrochloric acid (GB/T 622) solution to soak and dissolve for few minutes, so that the surface oxidation layer is dissolved; use tilting method to pour out the hydrochloric acid solution; use water to clean for several times; then use anhydrous ethanol (GB/T 678) to clean for several times; put it into a dryer to dry for 4 h. Anyone of the following methods may be selected for treatment. **Method 1** (acid-dissolution method): accurately weigh 0.5293 g (accurate to 0.0002 g) of aluminum sheet after treatment; place it in a 150 mL beaker; add 50mL (1+1) of hydrochloric acid (GB/T 622) solution; heat it on electric furnace at low temperature until it dissolves; transfer the solution into a 1000 mL volumetric flask; dilute to the mark with water; shake well. **Method 2** (alkali-dissolution method): accurately weigh 0.5293 g (accurate to 0.0002 g) of aluminum sheet after treatment; place it in a 150 mL beaker; add 2g of potassium hydroxide (HGB 3006)

Add sodium hydroxide into ash sample for melting; place it into boiling water for leaching; acidify it with hydrochloric acid; evaporate it to dryness; in the medium of hydrochloric acid, use animal glue to condense silicic acid; precipitate and filter it; burn and weigh it.

7.1.2 Reagents and materials

7.1.2.1 Sodium hydroxide (GB/T 629): granular.

7.1.2.2 Hydrochloric acid (GB/T 622).

7.1.2.3 95% ethanol (GB/T 679) or absolute ethanol (GB/T 678).

7.1.2.4 Hydrochloric acid solution: volume ratio is (1+1).

7.1.2.5 Hydrochloric acid solution: volume ratio is (1+3).

7.1.2.6 Hydrochloric acid solution: volume ratio is (1+50).

7.1.2.7 Animal glue solution: 10 g/L. Weigh 1 g of animal glue; place into 100mL of water at 70°C ~ 80°C. It shall be prepared immediately before use.

7.1.3 Test procedure

7.1.3.1 Weigh 0.48~0.52 g (accurate to 0.0002 g) of ash sample; place into a silver crucible; wet it with a few drops of ethanol (7.1.2.3); add 4 g of sodium hydroxide; put on crucible cover; put it into muffle furnace (4.1); rise furnace temperature slowly from room temperature to 650 °C~700 °C in 1~1.5 h; melt for 15 ~20 min. Take it out cool down with water; wipe up outer wall of the crucible; place it into a 250mL beaker; add 1 mL of ethanol (7.1.2.3) and adequate amount of boiling water; immediately put on watch-glass. After severe reaction stops, use hydrochloric acid solution (7.1.2.4) and hot water to alternately clean the crucible and its cover; add 20 mL of hydrochloric acid solution (7.1.2.2); shake well.

7.1.3.2 Place the beaker on the hot plate; slowly evaporate it to dryness (with yellow salt particle); take it down. After cooling; add 20 mL of hydrochloric acid solution (7.1.2.2); put on watch-glass; heat it to about 80°C; add 10mL of animal glue solution at 70°C ~ 80°C; stir it sharply for 1 min; keep it warm for 10 min; take it out; cool down; add about 50mL of hot water; stir it so that the salt is fully dissolved; filter it with quantitative filter paper into a 250mL volumetric flask; wash the sediment with hydrochloric acid (7.1.2.5) for 4~5 times; wipe up the cup wall and glass rod (with rubber head) with hot hydrochloric acid solution (7.1.2.6); wash the sediment for 3~5 times; then wash it with hot water for about 10 times.

7.1.3.3 Transfer the sediment and filter paper into a porcelain crucible that has been constant weight; incinerate the filter paper at low temperature; then burn in high-temperature muffle furnace (4.2) of 1000°C± 20°C for 1h; take it out and cool titrate the resultant of calcium and magnesium.

7.4.2 Reagent

7.4.2.1 Triethanolamine solution: volume ratio is (1+4).

7.4.2.2 Ammonia (GB/T 631) solution: volume ratio is (1+1).

7.4.2.3 Sodium diethyldithiocarbamate (referred to as cupron) solution: 50 g/L. Weigh 2.5g of cupron (HG 3-962); dissolve it in water; add five drops of ammonia solution (7.4.2.2); dilute with water to 50mL; store in a brown bottle after being filtered with rapid filter paper.

7.4.2.4 Sodium potassium tartrate tetrahydrate solution: 100g/L. Weigh 10g of sodium potassium tartrate tetrahydrate (GB/T 1288); dissolve it in water; dilute to 100mL with water.

7.4.2.5 EDTA standard solution: as specified in 7.3.2.4. Titer $T(\mathrm{MgO})$ of the solution to magnesium oxide is converted according to Formula (18):

$$T(\mathrm{MgO})=0.7187\times T(\mathrm{CaO}) \qquad (18)$$

Where,

$T(\mathrm{CaO})$ — the titer of EDTA standard solution to calcium oxide, in mg/mL;

0.7187 — the factor by which calcium oxide is converted to magnesium oxide.

7.4.2.6 Acid chrome blue K-naphthol green B mixed indicator:

Weigh 0.50g of acid chrome blue K (HG 10-1282) and 1.25g of naphthol green B; uniformly grind them with potassium chloride (GB/T 646) that has been pre-dried at 110°C; put into a flask with ground mouth; store in a dryer. Or respectively prepare then into water solutions, namely, weigh 0.04g of acid chrome blue K and 0.08g of naphthol green B; respectively dissolve them into 20mL of water; prior to use, proper mixture ratio shall be confirmed through experiment. Since acid chrome blue K solution is unstable, it shall be prepared immediately before use.

7.4.3 Analysis procedure

Accurately pipet 10 mL of solution C and 10mL of solution D; respectively pour them into 250 mL beakers; dilute to about 100mL with water; add 10 mL of triethanolamine solution (if the content of titanium dioxide is greater than 4.00%, 5mL of sodium potassium tartrate tetrahydrate solution can be added in advance), 10 mL of ammonia solution, and 1 drop of sodium potassium tartrate tetrahydrate; uniformly mix each reagent added; add in the amount of EDTA standard solution (7.3.2.4) that is slightly less than that consumed in the process of calcium titration; then add a small amount of acid chrome blue K-naphthol green B mixed indicator, or several drops of liquid

8.1.2.2 Ammonia (GB/T 631) solution: volume ratio is (1+1).

8.1.2.3 Hydrochloric acid (GB/T 622) solution: volume ratio is (1+1).

8.1.2.4 Barium chloride solution: 100 g/L. Weigh 10g of barium chloride (GB/T 652); dissolve it in water; dilute to 100 mL with water.

8.1.2.5 Silver nitrate solution: 10g/L. Weigh 1g of silver nitrate (GB/T 670); dissolve it in water; dilute to 100 mL with water; add several drops of nitric acid (GB/T 626); store in a brown bottle.

8.1.2.6 Methyl orange indicator: 2g/L. Weigh 0.2 g of methyl orange; dissolve in water; dilute to 100 mL with water.

8.1.2.7 Filter paper: medium-speed qualitative filter paper.

8.1.2.8 Filter paper: slow-speed qualitative filter paper.

8.1.3 Analysis procedure

8.1.3.1 Weigh 0.2g~0.5g of ash sample (accurate to 0.0002g); place it in a 250mL beaker; add 50 mL of hydrochloric acid solution (8.1.2.1); put watch-glass on; heat up for 20min; then take it down; add two drops of methyl orange indicator while the solution is still warm; neutralize the solution by injecting ammonia water until the solution just changes color; then add excessive three to six drops. After ferric hydroxide precipitates, filter the solution in a 300 mL beaker with medium-speed qualitative filter paper (8.1.2.7); use nearly-boiled hot water to clean and precipitate for 10 to 12 times; drop-add hydrochloric acid solution (8.1.2.3) into the filtered solution until the solution just changes color; then add excessive 2mL; dilute the solution to about 250mL with water.

8.1.3.2 Heat the solution to boil; drop-add 10 mL of barium chloride solution with continuous stirring; slightly boil it on electric hot plate or sand bath for 5min; maintain for 2h. The final volume of the solution shall be maintained at about 150mL.

8.1.3.3 Filter the solution with slow-speed qualitative filter paper (8.1.2.8); use hot water to clean until there is no chlorine ion (use silver nitrate solution to inspect).

8.1.3.4 Transfer the sediment and filter paper into a constant porcelain crucible; incinerate the filter paper at low temperature; then burn in muffle furnace (3.1) of 800°C~850°C for 40min; take out the crucible and cool down; put it into a dryer; weigh it after cooling to room temperature.

8.1.3.5 Blank test shall be carried out prior to preparing each batch of reagents or changing for any other reagents (except for ash sample, the remaining is as specified in 8.1.3).

8.2.2.5 Standard solution of sodium hydroxide : c(NaOH)=0.025 mol/L.

Preparation: weigh 5g of sodium hydroxide (GB/T 629); dissolve it in 5L of boiled and cooled water; mix up uniformly; preserve it in a polyethylene bottle to isolate from carbon dioxide.

Calibration: accurately weigh 0.1000g (accurate to 0.0002g) of reference reagent of potassium hydrogen phthalate that has been pre-dried at 120°C for 1 hour; place it in a 300 mL beaker; pour 150mL of water that has been boiled, neutralized and cooled into the beaker; add two to three drops of phenolphthalein indicator (8.2.2.8); titrate the reagent with standard solution of sodium hydroxide until it becomes slightly red. The titer $T(SO_3)$ of standard solution of sodium hydroxide to sulfur trioxide is calculated according to Formula (22).

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(22)

Where,

m — the mass of potassium hydrogen phthalate, in g;

V_1 — the volume of standard solution of sodium hydroxide for calibration, in mL;

0.2042 — the molar mass of potassium hydrogen phthalate, in g/m mol;

0.04003 — the molar mass of sulfur trioxide, in g/m mol.

8.2.2.6 Methyl red - bromocresol green mixed indicator: weigh 1g of bromocresol green; dissolve it in 14 mL of sodium hydroxide (GB/T 629) [c(NaOH)=0.1mol/L] solution; dilute to 1L with water. Then weigh 1g of methyl red (HG 3-958); dissolve it in 37 mL of sodium hydroxide (GB/T 629) [c(NaOH)=0.1mol/L] solution; dilute to 1L with water, mix these two types of solutions in equal-volume.

8.2.2.7 Phenolphthalein indicator: 10 g/L. Weigh 1g of phenolphthalein; dissolve it in 95% ethanol (GB/T 679); dilute to 100mL with ethanol.

8.2.3 Instruments and equipment

8.2.3.1 Combustion furnace: tubular; heated by silicon carbon bar or silicon-carbon tube; furnace temperature can be maintained at 1300°C±20°C; the length of constant temperature zone is 80 mm~100mm; equipped with platinum-rhodium thermocouple and millivoltmeter.

8.2.3.2 Reducing combustion tube: made of corundum; tube length is about 750mm; at one end, the outer diameter is 22 mm, the inner diameter is 19 mm, the length is about 690 mm; at the other end, the outer diameter is 10 mm, the inner diameter is 7 mm, the length is about 60 mm.

8.2.3.3 Combustion boat: made of corundum or porcelain with refractability greater than 1400°C, 77mm long, 22mm wide and 8mm high.

8.2.3.4 Nichrome wire push rod: about 2mm in diameter, and about 650 mm long; its one end is rolled into a ring with a diameter of about 10mm for boosting combustion boat.

8.2.3.5 T-shaped glass tube: used for installation of push rod and air inlet.

8.2.3.6 Gas flowmeter: the maximum measuring range is 1 L/min.

8.2.3.7 Gas filter: glass melting plate made from the sintering of glass sand; model of melting plate: G_2; pore size: 4.9 μm~9 μm; used for gas dispersion.

8.2.3.8 Conical flask: 250mL.

8.2.3.9 Gas bottle: 250 mL, 2 pieces; one with potassium hydroxide solution (8.2.2.4) and the other with concentrated sulfuric acid (8.2.2.2).

8.2.3.10 Drying tower: 250 mL, with allochroic silicagel inside.

8.2.3.11 A water suction pump or vacuum pump.

8.2.3.12 Silicone rubber tube: its outer diameter and inner diameter are 11mm and 6mm respectively.

8.2.3.13 Fix-sulfur absorber: 60 mL.

8.2.4 Analysis procedures

8.2.4.1 Install the apparatuses as shown in Figure 1; heat it up to 1300°C±20°C after being energized; add 50 mL of hydrogen peroxide solution into fixed sulfur absorber and conical flask.

9.1.2.10 Standard stock solution of phosphorus pentoxide: 0.2292 g/L.

Accurately weigh 0.4392g (accurate to 0.0002g) of potassium dihydrogen phosphate GR that has been pre-dried at 110°C for 1h; dissolve it in water; move it into a 1L volumetric flask; dilute to the mark with water; shake well.

9.1.2.11 Standard working solution of phosphorus pentoxide: 0.02292 g/L.

Accurately pipet 10 mL of standard stock solution of phosphorus pentoxide; pour it into a 100 mL volumetric flask; dilute to the mark with water; shake it well. It shall be immediately prepared before use.

9.1.3 Analysis procedure

9.1.3.1 Preparation of to-be-tested sample solution

Weigh 0.1000g of ash sample (accurate to 0.0002g); put it into a 30 mL PTFE crucible; wet it with water; add 2 mL of perchloric acid and 10 mL of hydrofluoric acid; place the crucible on electric hot plate to slowly heat it up at low temperature (not higher than 250°C) until nearly dry it; then raise the temperature until white smoke is basically exhausted and the solution is dried but not burned black. Take down the crucible and cool down; add 10 mL of hydrochloric acid solution (9.1.2.4) and 10 mL of water; place the crucible on electric hot plate to heat it up for boiling; maintain for 2min. Take down the crucible again; use hot water to move the sample solution in it to a 100mL volumetric flask; cool down to room temperature; dilute to the mark with water; shake well.

9.1.3.2 Preparation of blank solution

It is as specified in 9.1.3.1, but ash sample is not included.

9.1.3.3 Drawing of working curve

9.1.3.3.1 Accurately pipet 0mL, 1mL, 2mL, and 3mL of standard working solution of phosphorus pentoxide; respectively pour them into 50mL volumetric flasks; add 5 mL of reagent solution (9.1.2.9); maintain for 1 min ~2 min; then dilute to the mark with water; shake well. After being placed at 20°C~30°C for 1h, use a 1cm~3 cm cuvette to determine its absorbance at wave length of 650 nm on spectrophotometer.

9.1.3.3.2 Draw a working curve by taking the mass (mg) of phosphorus pentoxide as x abscissa and the absorbance as *y* coordinate.

9.1.3.4 Determination

Accurately pipet 10 mL of to-be-tested sample solution (9.1.3.1) and 10 mL of blank solution (9.1.3.2); respectively pour them into 50mL volumetric flasks; then repeat the procedures as specified in 9.1.3.3.1 (if the absorbance determined is out of working

9.2.2.4 Sulfuric acid (GB/T 625) solution: $c(1/2H_2SO_4)$=4 mol/L.

9.2.2.5 Ascorbic acid solution: 50 g/L. Weigh 5g of ascorbic acid; dissolve it in water; dilute to 100 mL with water. The solution shall be prepared immediately before use.

9.2.2.6 Sulfuric acid (GB/T 625) solution: $c(1/2H_2SO_4)$=7.2 mol/L.

9.2.2.7 Ammonium molybdate - sulfuric acid solution: weigh 17.2g of ammonium molybdate (GB/T 657); dissolve in sulfuric acid solution (9.2.2.6); dilute to 1L with this solution.

9.2.2.8 Antimony potassium tartrate solution: weigh 0.34g of antimony potassium tartrate; dissolve it in 250mL water.

9.2.2.9 Reagent solution: add 10 mL of ascorbic acid solution (9.2.2.5) and 5 mL of antimony potassium tartrate solution (9.2.2.8) into 35 mL of ammonium molybdate-sulfuric acid solution (9.2.2.7); mix them up uniformly. The solution shall be prepared immediately before use.

9.2.2.10 Standard stock solution of phosphorus pentoxide: 229.2 g/L.

Accurately weigh 0.4392g of potassium dihydrogen phosphate GR that has been pre-dried at 110°C for 1h; dissolve it in water. Move it into a 1L volumetric flask; dilute to the mark with water; shake well.

9.2.2.11 Standard working solution of phosphorus pentoxide: 22.9 g/L.

Accurately pipet 10 mL of standard stock solution of phosphorus pentoxide; put it into a 100 mL volumetric flask; dilute to the mark with water; shake it well. The solution shall be prepared immediately before use.

9.2.3 Analysis procedure

9.2.3.1 Preparation of to-be-tested sample solution

Weigh 0.2000g of ash sample (accurate to 0.0002g); put it into a 30 mL of PTFE crucible; add 10mL of hydrofluoric acid and 10 mL of sulfuric acid (9.2.2.2). In fuming cupboard, place the crucible on electric hot plate to slowly heat it up at low temperature until nearly dry it; then raise the temperature until white smoke is basically exhausted and the solution is dried but not burned black. Take down the crucible and cool down; use hot water to move the melt in it to a 100mL beaker; add 20 mL of acid solution (9.2.2.3) and an appropriate amount of water; heat up until the salt is dissolved; then cool down to room temperature; move the solution into a 200 mL volumetric flask; dilute to the mark with water; shake well; preserve it after being clarified.

9.2.3.2 Preparation of blank solution

It is same as specified in 9.2.3.1, but ash sample is not included.

9.2.3.3 Drawing of working curve

9.2.3.3.1 Accurately pipet 0mL, 1mL, 2mL, and 3mL of standard working solution of phosphorus pentoxide; respectively pour them into 50mL volumetric flasks; add 5 mL of reagent solution (9.2.2.9); maintain for 1 min ~2 min; then dilute to the mark with water; shake well. After being placed at 20°C~30°C for 1h, use a 1cm~3 cm cuvette to determine its absorbance at wave length of 650 nm on spectrophotometer.

9.2.3.3.2 Draw a working curve by taking the mass (mg) of phosphorus pentoxide as x abscissa and the absorbance as *y* coordinate.

9.2.3.4 Determination

Accurately pipet 10 mL of to-be-tested sample solution (9.2.3.1) and 10mL of blank solution (9.2.3.2); respectively pour them into 50 mL volumetric flasks; add 0.4 mL of sulfuric acid solution (9.2.2.4); then repeat the procedures as specified in 9.2.3.3.1 (if the absorbance determined is out of the working curve, the amount of solution to be pipetted shall be appropriately reduced). The mass (mg) of phosphorus pentoxide can be obtained from the working curve.

9.2.4 Result calculation

The mass fraction $w(P_2O_5)$(%) of phosphorus pentoxide is calculated according to Formula (26):

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(26)

Where,

$m(P_2O_5)$ — the mass of phosphorus pentoxide, which is obtained from working curve, in mg;

V_2 — the volume of solution pipetted from the total volume of ash sample solution, in mL;

m — the mass of ash sample, in g.

Calculated result shall be rounded to two decimal places in accordance with Rules for Rounding off of Numerical Values specified in GB/T 483.

9.2.5 Method precision

Same as specified in 9.1.5.

10.3.2 Preparation of blank solution: as specified in 9.2.3.2.

10.3.3 Drawing of working curve

10.3.3.1 Accurately pipet 0mL, 2mL, 4mL, 6mL, 8mL and 10mL of mixed standard solution of sodium oxide; respectively pour them into 100mL volumetric flasks; add 10mL of sulfuric acid solution (10.2.1) to synthetize ash solution; dilute to the mark with water; shake well.

10.3.3.2 Pre-heat flame photometer for 15min; adjust the best air pressure and gas pressure; place in the potassium filter; respectively use zero adjusting solution and full-scale adjusting solution to adjust the grating, so as to make the pointer of galvanometer to be at "zero" and full scale positions respectively; repeat the procedure until it is stable. Then determine in sequence; record the readings of potassium.

10.3.3.3 Change to sodium filter; respectively use zero adjusting solution and full-scale adjusting solution to re-adjust the grating, so as to make the pointer of galvanometer to be at "zero" and "1/2" full scale positions respectively; repeat the procedure until it is stable. Then determine in sequence; record the readings of sodium.

10.3.3.4 Draw working curves by respectively taking the mass (mg) of potassium oxide and that of sodium oxide as x abscissa, and relevant readings as *y* coordinate.

10.3.4 Determination

Determine sample solution (10.3.1) and blank solution (10.3.2) according to the procedures as specified in 10.3.3.2 and 10.3.3.3; record readings of potassium and sodium; obtain the mass of potassium oxide and mass of sodium oxide from the working curves.

10.4 Result calculation

The mass fraction $w(K_2O)(\%)$ of potassium oxide and that [$w(Na_2O)(\%)$] of sodium oxide are calculated according to Formula (27) and Formula (28):

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(27)

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(28)

Where,

$m(K_2O)$, $m(Na_2O)$ — the mass of potassium oxide and that of sodium oxide, which are obtained from the working curve, in mg;

move the dissolved solution into a 500 mL volumetric flask after being cooled down; dilute to the mark with water; shake well. Store the solution in a plastic bottle.

11.2.6 Strontium solution: 50 mg/mL. Weigh 152 g of strontium chloride ($SrCl_2•6H_2O$) that has been purified by recrystallization; put it into a 400 mL beaker; dissolve with water; move the dissolved solution into a 1 000 mL volumetric flask; dilute to the mark with water; shake well. Store it in a plastic bottle.

Note: purification method for strontium chloride: dissolve 1 000 g of strontium chloride ($SrCl_2•6H_2O$) with 400 mL of water; heat it up at 70°C; add 400 mL of ethanol while it is still warm; perform suction filtration after it is recrystallized at low temperature; dry it at 40°C~50°C.

11.2.7 Aluminum solution: containing 1mg/mL Al_2O_3. Weigh 4.736 g ($AlCl_3•6H_2O$); put it into a 400 mL beaker; dissolve with water; move the dissolved solution into a 1000 mL volumetric flask; dilute to the mark with water; shake well. Store the solution in a plastic bottle.

11.2.8 Standard stock solution of potassium oxide: 1 mg/mL.

Weigh 1.5829g of high-purity (99.99%) potassium chloride that has been burned at 500°C for 30min; put it into a 400 mL beaker; dissolve with water; move the dissolved solution into a 1 000 mL volumetric flask; dilute to the mark with water; shake well. Store the solution in a plastic bottle.

11.2.9 Standard stock solution of sodium oxide: 1 mg/mL.

Weigh 1.8859g of high-purity (99.99%) sodium chloride that has been burned at 500°C for 30min; put it into a 400 mL beaker; dissolve with water; move the dissolved solution into a 1 000 mL volumetric flask; dilute to the mark with water; shake well. Store it in a plastic bottle.

11.2.10 Standard stock solution of calcium oxide: 1 mg/mL.

Weigh 1.7840g of high-purity (99.99%) calcium carbonate that has been baked at 110°C for 1h; put it into a 400mL beaker; dissolve with 50 mL of water; put on watch-glass; slowly add 20 mL of hydrochloric acid solution (11.2.3) along he wall of the beaker; heat it up to exhaust the carbon dioxide after the high-purity calcium carbonate is completely dissolved; cool down to room temperature; move into a 1 000 mL volumetric flask; dilute to the mark with water; shake well. Store the solution in a plastic bottle.

11.2.11 Standard stock solution of magnesium oxide: 1 mg/mL.

Weigh 0.6030g of high-purity (99.99%) magnesium metal; put it into a 400 mL beaker; add 40 mL of hydrochloric acid solution (11.2.3); heat up until it is dissolved; move the dissolved solution into a 1000 mL volumetric flask after being cooled down; dilute to mark with water; shake well. Store the solution in a plastic bottle.

flask; cool down to room temperature; dilute to the mark with water; shake well.

11.3.2 Preparation of sample blank solution

Decompose a batch of samples; at the same time, it shall prepare a sample blank solution. For preparation of the sample blank solution, ash sample shall not be added; the rest procedure is same as 11.3.1.

11.3.3 Preparation of to-be-tested sample solution

11.3.3.1 To-be-tested sample solutions of ferrum, calcium and magnesium: accurately pipet 5 mL of sample solution (11.3.1) and 5 mL of sample blank solution (11.3.2); respectively put them into 50 mL volumetric flasks; add 2 mL of lanthanum solution (when strontium is used as releasing agent, 2 mL of strontium solution shall be added instead) and 1 mL of hydrochloric acid solution (11.2.4); dilute to the mark with water; shake well.

11.3.3.2 To-be-tested sample solutions of potassium, sodium and manganese: accurately pipet 5 mL of sample solution (11.3.1) and 5 mL of sample blank solution (11.3.2); respectively put them into 50 mL volumetric flasks; add 1 mL of hydrochloric acid solution (11.2.4); dilute to the mark with water; shake well.

11.3.4 Preparation of a series of mixed standard solution

11.3.4.1 A series of mixed standard solution of ferrum, calcium and magnesium: respectively pipet 0mL, 1mL, 2mL, 3mL, 4mL, 5mL, 6mL, 7mL, 8mL, 9mL and 10mL of mixed standard working solutions of ferrum, calcium, magnesium; respectively put them into 100mL volumetric flasks; add 4 mL of lanthanum solution (when strontium is used as releasing agent, 4 mL of strontium solution and 3 mL of aluminum solution shall be added instead) and 4 mL of hydrochloric acid solution (11.2.4); dilute to the mark with water; shake well.

11.3.4.2 A series of mixed standard solution of potassium, sodium and manganese: respectively pipet 0mL, 1mL, 2mL, 3mL, 4mL, 5mL, 6mL, 7mL, 8mL, 9mL and 10mL of mixed standard working solution of potassium, sodium and manganese; put them into 100mL volumetric flasks; add 4mL of lanthanum solution; dilute to the mark with water; shake well.

Note: concentration interval of a series of mixed standard solution as specified in 11.3.4.1 and 11.3.4.2 can be increased or decreased according to the performance of instruments and the bending situation of working curve.

11.3.5 Determination of ferrum, calcium, magnesium, potassium, sodium and manganese

11.3.5.1 Determination of instrument working condition: except analyzing-lines of all elements as specified in Table 20 AND flame gas for use, ADJUST other parameters of instruments to the best values - such as lamp current, band width, height and

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------ The End ------