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**Methods for chemical analysis of granulated
electric furnace phosphorus slag**

粒化电炉磷渣化学分析方法

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Methods for chemical analysis of granulated electric furnace phosphorus slag

1 Scope

This standard specifies the benchmark method and substitution method for the method of chemical analysis of granulated electric furnace phosphorus slag. In case of dispute, the benchmark method shall prevail.

This standard applies to the granulated electric furnace phosphorus slag used in the building materials industry and other materials designated to adopt this standard.

2 Normative references

The provisions in following documents become the provisions of this standard through reference in this standard. For the dated references, the subsequent amendments (excluding corrections) or revisions do not apply to this standard; however, parties who reach an agreement based on this standard are encouraged to study if the latest versions of these documents are applicable. For undated references, the latest edition of the referenced document applies.

GB/T 176-2008 Methods for chemical analysis of cement

GB/T 2007.1 General rules for the sampling and sample preparation of minerals in bulk - Sampling by manual method

GB/T 6682 Water for analytical laboratory use - Specification and test methods

3 Terms and definitions

The terms and definitions as established in the GB/T 15000 series as well as the following terms and definitions apply to this standard.

3.1

Repeatability conditions

The conditions for independent test of the same tested object in the same laboratory, by the same operator using the equipment, according to the same

of other standard titration solutions retain four significant digits after the rounding off.

Unless otherwise stated, the results of each analysis are based on mass fraction. The analytical results of the chloride ion are expressed in % to three decimal places. The analytical results of other items are expressed in % to two decimal places.

4.3 Blank test

Use the same amount of reagent. Do not add specimen. Use the same determination procedures to carry out test, to correct the determination results as obtained.

4.4 Ignition

Place the filter paper and the precipitate in a crucible which has previously been burned to a constant weight. To avoid producing flame, in an oxidizing atmosphere, slowly dry and ash it. After ashing it to the absence of black carbon particle, then place it in a high-temperature furnace (6.4), to burn it at the specified temperature. In a desiccator (6.3), cool it to room temperature. Weigh it.

4.5 Constant weight

After the first burning, cooling, weighing, use the method of applying continuous burning of 15 min, cooling, weighing, to check the constant mass. When the difference between two consecutive weighing results is less than 0.0005 g, it reaches constant weight.

5 Reagents and materials

Unless otherwise stated, the reagents used shall be not less than analytical pure. The water used shall meet the requirements for grade-3 water as specified in GB/T 6682.

The density of commercially available concentrated liquid reagents as listed in this standard refers to a density (ρ) at 20 °C, in grams per cubic centimeter (g/cm^3). In chemical analysis, the acid or ammonia used, unless otherwise the concentration is not indicated, refers to commercially available concentrated acid or concentrated ammonia. Use the volume ratio to indicate the degree of dilution of the reagent. For example, hydrochloric acid (1 + 2) means that 1 part by volume of concentrated hydrochloric acid is mixed with 2 parts by volume of water.

5.1 Hydrochloric acid (HCl)

5.14 Ascorbic acid solution (50 g/L)

Dissolve 5 g of ascorbic acid (V.C) in 100 mL of water. If necessary, filter it before use. Prepare it before use.

5.15 pH10 buffer solution

Dissolve 67.5 g of ammonium chloride (NH_4Cl) in water. Add 570 mL of ammonia water ($\text{NH}_3 \cdot \text{H}_2\text{O}$). Add water to dilute it to 1 L.

5.16 Sodium potassium tartrate solution (100 g/L)

Dissolve 10 g of sodium potassium tartrate ($\text{C}_4\text{H}_4\text{KNaO}_6 \cdot 4\text{H}_2\text{O}$) in water. Add water to dilute it to 100 mL.

5.17 Potassium fluoride dissolution (20 g/L)

Dissolve 20 g of potassium fluoride ($\text{KF} \cdot 2\text{H}_2\text{O}$) in water. Add water to dilute it to 1 L. Store it in a plastic bottle.

5.18 Preparation of phosphorus pentoxide (P_2O_5) standard solution

5.18.1 Preparation of phosphorus pentoxide standard solution

Weigh 0.1917 g of potassium dihydrogen phosphate (KH_2PO_4 , reference reagent) which has been baked at $105^\circ\text{C} \sim 110^\circ\text{C}$ for 2 h, accurate to 0.0001 g. Place it in a 300 mL beaker. Add water to dissolve it. Transfer it in a 1000 mL volumetric flask. Use water to dilute it to the mark. Shake it uniformly. Each milliliter of this standard solution contains 0.1 mg of phosphorus pentoxide.

Pipette 50.00 mL of the above standard solution into a 500 mL volumetric flask. Use water to dilute it to the mark. Shake it uniformly. Each milliliter of this standard solution contains 0.01 mg of phosphorus pentoxide.

5.18.2 Drawing of working curve

Pipette 0 mL, 2.00 mL, 4.00 mL, 6.00 mL, 8.00 mL, 10.00 mL, 15.00 mL, 20.00 mL, 25.00 mL of standard solution which contains 0.01 mg of phosphorus pentoxide per mL into a 200 mL beaker. Add water to dilute it to 50 mL. Add 10 mL of ammonium molybdate solution (5.13) and 2 mL of ascorbic acid solution (5.14). Heat to slightly boil it for (1.5 ± 0.5) min. After cooling it to room temperature, transfer it in a 100 mL volumetric flask. Use hydrochloric acid (1 + 10) to rinse the beaker. Use hydrochloric acid (1 + 10) to dilute it to the mark. Shake it uniformly. Use a spectrophotometer and 10 mm cuvette to determine the absorbance of the solution at a wavelength of 730 nm, which uses water as a reference. Use the determined absorbance as a function of the corresponding phosphorus pentoxide content, to draw the working curve.

calcium carbonate standard titration solution

Slowly release 10 mL ~ 15 mL EDTA standard titration solution (5.20) from the burette into a 400 mL beaker. Add about 200 mL of water. Add appropriate amount of CMP mixing indicator (5.22). Whilst stirring it, add potassium hydroxide solution (5.12) until a stable red color appears. Add another 2 mL ~ 3 mL. Use calcium carbonate standard titration solution (5.21.1) to titrate it until a green fluorescence appears.

The volume ratio of the EDTA standard titration solution to the calcium carbonate standard titration solution is calculated according to formula (6):

$$K_1 = \frac{V_2}{V_3} \dots\dots\dots (6)$$

Where:

K_1 - The volume ratio of EDTA standard titration solution to calcium carbonate standard titration solution;

V_2 - The volume of the EDTA standard titration solution, in milliliters (mL);

V_3 - The volume of the calcium carbonate standard titration solution as consumed in titration, in milliliters (mL).

5.22 Calcein - methyl thymol blue - phenolphthalein mixed indicator (shortly referred to as CMP mixed indicator)

Weigh 1.000 g of calcein, 1.000 g of methyl thymol blue, 0.200 g of phenolphthalein, 50 g of potassium nitrate (KNO_3) which has been dried at 105 °C ~ 110 °C. Mix and grind it fine. Store it in a mouth-grinding bottle.

5.23 Acid chrome blue K - naphthol green B mixed indicator (shortly referred to as KB mixed indicator)

Weigh 1.000 g of acid chrome blue K, 2.500 g of naphthol green B, 50 g of potassium nitrate (KNO_3) which has been dried at 105 °C ~ 110 °C. Mix and grind it fine. Store it in a mouth-grinding bottle.

5.24 P-nitrophenol indicator solution (2 g/L)

Dissolve 0.2 g of p-nitrophenol in 100 mL of water.

6 Instruments and equipment

6.1 Balance

It shall not be lower than grade-4, accurate to 0.0001 g.

6.2 Platinum, silver, porcelain crucible

It has a lid. The capacity is 15 mL ~ 30 mL.

6.3 Dryer

It is loaded with color-changing silica gel.

6.4 High-temperature furnace

For the flame-isolated heating furnace, carry out resistance heating on the periphery of the furnace. It shall use temperature controller to accurately control the furnace's temperature. The controllable temperature is $(950 \pm 25) ^\circ\text{C}$.

6.5 Glass capacity vessel

Burette, volumetric flask, pipette.

6.6 Spectrophotometer

It can, within the range of 400 nm ~ 800 nm, determine the absorbance of the solution. It is equipped with a 10 mm cuvette.

7 Preparation of specimen

Use the method of GB/T 2007.1 to take sample. The samples as sent to the lab shall be representative uniform samples. Use quartering method or a reducer to reduce the specimen to about 100 g. Use a 80 μm square-hole sieve to sieve it. Use a magnet to remove the metal iron from the materials as left on the sieve. Grind the materials as left on the sieve to make it all pass a 80 μm square-hole sieve. Thoroughly mix it uniformly. Contain it in the sample bottle. Seal it for preservation, to prepare for determination.

8 Determination of loss on ignition - Ignition reduction method

8.1 Summary of method

The specimen is burned in a high-temperature furnace at $(950 \pm 25) ^\circ\text{C}$, to drive off carbon dioxide and moisture, while oxidizing the existing oxidizable elements.

8.2 Analytical procedures

Weigh about 1 g of specimen (m_2), accurate to 0.0001 g. Place it in a porcelain crucible which has been burned to constant weight. Place the lid inclined on the

solution for calcium. Use calcium carbonate standard titration solution to back titrate the excess EDTA dropwise.

12.2 Analytical procedures

Weigh about 0.5 g of specimen (m_4), accurate to 0.0001 g. Place it in the silver crucible. Add 6 g ~ 7 g of sodium hydroxide (5.8). Cover the lid (leave a gap). Place it in a high-temperature furnace (6.4). Starting from low temperature, melt it at a high-temperature of 650 °C ~ 700 °C for 20 min, during which time it is removed and shaken once. Remove to cool it. Place the crucible into a 300 mL beaker which contains about 100 mL of boiling water. Cover the watch glass. Heat it slightly on the electric furnace. After the frit is completely leached, remove the crucible. Use water to rinse the crucible and lid. Whilst stirring it, add 25 mL ~ 30 mL of hydrochloric acid in one time. Then add 1 mL of nitric acid. Use hot hydrochloric acid (1 + 5) to clean the crucible and lid. Heat the solution to boil it. After cooling it, transfer it into a 250 mL volumetric flask. Use water to dilute it to the mark. Shake it uniformly. This solution A is used for the determination of calcium oxide (12.2) and magnesium oxide (17.2).

Pipette 25.00 mL of solution from the solution A into a 400 mL beaker. Add 7 mL ~ 10 mL of potassium fluoride (5.17) solution. Add water to dilute it to about 200 mL. Add 5 mL of triethanolamine (1 + 2) and appropriate amount of CMP mixed indicator (5.22). Whilst stirring it, add potassium hydroxide solution (5.12), until green fluorescence appears. Add another 5 mL ~ 8 mL. Use the EDTA standard titration solution [$c(\text{EDTA}) = 0.015 \text{ mol/L}$] (5.20) to titrate it, until the green fluorescence disappears and a stable red color appears. Add another 3 mL ~ 5 mL. Place it for 1 min. Then use calcium carbonate standard titration solution (5.21) to titrate it, until green fluorescence appears.

12.3 Calculation and representation of results

The mass percentage of calcium oxide w_{CaO} is calculated according to formula (8):

$$w_{\text{CaO}} = \frac{T_{\text{CaO}} \times (V_4 - K_1 \times V_5) \times 10}{m_4 \times 1000} \times 100 = \frac{T_{\text{CaO}} \times (V_4 - K_1 \times V_5)}{m_4} \dots\dots\dots (8)$$

Where:

w_{CaO} - The mass fraction of calcium oxide, %;

T_{CaO} - The titer of EDTA standard titration solution for calcium oxide, in milligrams per milliliter (mg/mL);

V_4 - The volume of EDTA standard titration solution added, in milliliters (mL);

V_5 - The volume of calcium carbonate standard titration solution as

After cooling, add 3 g of sodium carbonate-borax mixed flux (5.10) into the residue which is obtained by hydrofluoric acid treatment. Mix it uniformly. Melt it at 950 °C ~ 1000 °C for 10 min. Use the crucible tongs to hold the crucible to rotate it, to make the melt be uniformly attached to the inner wall of the crucible. After cooling, place the crucible in a 300 mL beaker which contains 10 mL of sulfuric acid (1 + 1) and 100 mL of water which had been heated to slightly boiling. Keep it in a slightly boiling state, until the melt is completely dissolved. Use water to rinse the crucible and lid. After cooling it, transfer it into a 250 mL volumetric flask. Use water to dilute it to the mark. Shake it uniformly.

Pipette 10.00 mL of the above sample solution into a 200 mL beaker (the amount of sample solution is determined by the content of phosphorus pentoxide). Add water to 50 mL. Add 1 drop of p-nitrophenol indicator solution (5.24). Add sodium hydroxide solution (5.11) dropwise to yellow. Then add hydrochloric acid (1 + 1) dropwise to colorless. Add 10 mL of ammonium molybdate solution (5.13) and 2 mL of ascorbic acid (5.14). Heat to slightly boil it for (1.5 ± 0.5) min. After cooling, transfer it into a 100 mL volumetric flask. Use hydrochloric acid (1 + 10) to rinse the beaker. Use hydrochloric acid (1 + 10) to dilute it to the mark. Shake it uniformly. Use a spectrophotometer and 10 mm cuvette to determine the absorbance of the solution at a wavelength of 730 nm, using water as a reference. From the working curve (5.18.2), find the content of phosphorus pentoxide (m_6).

16.3 Calculation and representation of results

The mass percentage of phosphorus pentoxide $w_{P_2O_5}$ is calculated according to formula (9):

$$w_{P_2O_5} = \frac{m_6 \times 5}{m_5 \times 1000} \times 100 = \frac{m_6 \times 0.5}{m_5} \dots\dots\dots (9)$$

Where:

$w_{P_2O_5}$ - The mass fraction of phosphorus pentoxide, %;

m_6 - The content of phosphorus pentoxide in 100 mL solution, in milligrams (mg);

m_5 - The mass of the sample in 16.2 (m_5), in grams (g).

17 Determination of magnesium oxide - EDTA titration reduction (substitution)

17.1 Summary of method

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