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Limit and Test Method of Ammonium Ion Content in Fly Ash

粉煤灰中铵离子含量的限量及检验方法

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Limit and Test Method of Ammonium Ion Content in Fly Ash

1 Scope

This Standard specifies the limit and test method of ammonium ion content in fly ash.

This Standard is applicable to fly ash that is used as cement admixtures, mortar and concrete admixtures and other materials designated to adopt this Standard.

2 Normative References

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) is applicable to this document.

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

GB/T 8170 Rules of Rounding off for Numerical Values & Expression and Judgement of Limiting Values

GB/T 12573 Sampling Method for Cement

3 Terms and Definitions

For the purposes of this Document, the following terms and definitions apply.

3.1 Ammonium ion in fly ash

Ammonium ions in the form of NH_4^+ attached to the surface of fly ash.

3.2 Series standard specifications for determining ammonium content of fly ash

Standard samples with different concentrations of ammonium ion content.

NOTE: It is used for comparative analysis to reduce the test error of ammonium ion content in fly ash.

Appendix A

(Normative)

Test Method for Ammonium Ion Content in Fly Ash -Distillation Titration Method

A.1 Principle

Mix the fly ash with water; adjust the pH to alkaline; perform distillation; absorb the distilled NH_3 by a dilute sulfuric acid solution; and transfer the ammonium ions in the fly ash to the distillate. Take methyl red-methylene blue mixed indicator as indicator; titrate the excessive sulfuric acid by sodium hydroxide standard titration solution; and calculate the ammonium ion content in fly ash based on the NH_4^+ content in the distillate.

A.2 Basic requirements of the test

A.2.1 Test requirements

The number of times for each test is specified as twice; and the difference between the two test results shall be within the listed tolerance (see Table A.1), and the average of the two test results is used to indicate the test result.

The blank test specified in this Standard can be carried out every 3 months when the quality of distilled water remains unchanged.

It is recommended to use series standard specifications for determining ammonium content of fly ash to reduce test errors.

In all titration processes, the needle-tube weighing method can be used instead of the burette method of measuring the solution.

A.2.2 Expression of mass, volume and result

The mass is expressed in "grams (g)" and is accurate to 0.001g. The volume of the burette is expressed in "milliliters (mL)", and the reading is accurate to 0.01mL. Refer to the burette for the indication of the needle tube.

The ratio of concentration and volume of the standard titration solution retain three significant figures. Unless otherwise stated, all analysis results are based on mass fraction. The unit of ammonium ion content in fly ash is mg/kg, and it is retained to one digit after the decimal point.

The rounding-off of the value is carried out according to GB/T 8170.

A.2.3 Blank test

Do not add fly ash, use the same amount of reagent, and perform the test in accordance with the same measurement procedures.

A.2.4 General rules of reagents

Unless otherwise specified, the used reagents shall be no lower than the analytical reagent, and the guaranteed reagent is preferred. The reagent used for calibration shall be the reference reagent. The used water shall be no lower than the requirements for Class-III water specified in GB/T 6682; and distilled water is generally used.

The density of commercially available concentrated liquid reagents listed in this Standard refers to the density (ρ) at 20°C, and the unit is grams per cubic centimetre (g/cm^3).

In chemical analysis, any acid whose concentration is not marked refers to a commercially available concentrated acid.

Unless otherwise stated, the validity period of the standard titration solution is 3 months. If it exceeds 3 months, re-calibrate it.

A.3 Reagents and materials

A.3.1 Sulfuric acid (H_2SO_4)

$1.84g/cm^3$, the mass fraction is 95%~98%.

A.3.2 Sodium hydroxide (NaOH)

Chemically pure.

A.3.3 Potassium hydrogen phthalate ($C_8H_5KO_4$)

Reference reagent.

A.3.4 Ethanol (C_2H_5OH)

The volume fraction of ethanol is 95%.

A.3.5 Phenolphthalein indicator solution (10g/L)

Dissolve 1g of phenolphthalein in 100 mL of ethanol (A.3.4).

A.3.6 Sulfuric acid solution $c(1/2H_2SO_4) = 0.1mol/L$

Pipette 3mL of sulfuric acid; slowly pour into 1000mL of water; cool and shake well.

A.3.7 Sodium hydroxide standard titration solution $c(NaOH)=0.1mol/L$

A.4.1.1 Analytical balance: the division value is no more than 0.0001g.

A.4.1.2 Electronic balance: the division value is no more than 0.01g.

A.4.2 Glass volumetric utensils

A.4.2.1 500mL distillation flask.

A.4.2.2 50mL alkali burette.

A.4.2.3 250mL, 300mL beaker.

A.4.2.4 20mL pipette.

NOTE: It is recommended to use a pipette gun.

A.4.2.5 1000mL volumetric flask.

A.4.2.6 250mL graduated cylinder.

A.4.3 1000 W electric jacket

The capacity is 500mL.

A.4.4 Medical syringe

The capacity is no less than 30mL.

A.5 Test procedures

A.5.1 Preparation of distillate

Weigh 10g of fly ash sample (m_2), accurate to 0.01g; and put it into a 250mL beaker. In another graduated cylinder (A.4.2.6), pipette about 210mL of distilled water; mix the distilled water and fly ash evenly and transfer it into a 500mL distillation flask (A.4.2.1). In order to transfer all the fly ash into the distillation flask, the beaker shall be rinsed twice.

Add about 0.2g of sodium hydroxide (A.3.2) in the distillation flask; quickly connect the distillation device and ensure that it is sealed to prevent ammonia from escaping; and wait for distillation.

Pipette 20.00mL of sulfuric acid solution (A.3.6) in a 250mL beaker; add 3 ~ 4 drops of methyl red-methylene blue mixed indicator solution (A.3.8). Insert the glass tube at the outlet of the distillation flask into the sulfuric acid solution at the bottom of the beaker.

Heating distillation, when the volume of distillate in the beaker reaches 200mL, stop heating; remove the distillation flask; rinse the condenser by water; and collect the washing liquid in the beaker.

Appendix B

(Normative)

Test Method for Ammonium Ion Content in Fly Ash - Electrode Method

B.1 Principle

Mix the fly ash with water; transfer the ammonium ions in the fly ash to the filtrate; and filter the mixture by filter paper. Then by adding a certain amount of ionic strength adjuster to the filtrate to make the value of pH >12 of the test solution; the NH_4^+ in the filtrate is converted into NH_3; and the content of NH_3 in the filtrate is analyzed and determined by a high-performance ammonia gas sensing electrode, and calculate the ammonium ion content in the fly ash.

B.2 Basic requirements of the test

The same as A.2.

B.3 Reagents and materials

B.3.1 Ammonium chloride

Dry ammonium chloride (NH_4Cl, guarantee reagent) at 110°C for 2h; cool to room temperature in a desiccator; and store in a sealed and dark place.

B.3.2 Ammonia stock solution (1mg/mL, NH_3)

Accurately weigh 0.3142g of ammonium chloride dried at 105°C for 1h. Dissolve by a small amount of water; transfer to a 100mL volumetric flask; dilute to the mark with water; shake well; 1mL of this solution contains 1.00mg of ammonia.

B.3.3 Preparation of ammonia standard solution

Before the start of the test, the ammonia stock solution (B.3.2) is diluted step by step into an ammonia standard solution with a concentration difference of 10 times according to the following requirements.

a) Ammonia standard solution 1 (0.1 mg/mL, NH_3): Before the test, use a pipette to accurately pipette 10 mL of ammonia stock solution (B.3.2) in a 100 mL volumetric flask; dilute to the mark with water; and shake well.

b) Ammonia standard solution 2 (0.01mg/mL, NH_3): Before the test, use a pipette to accurately pipette 10mL of ammonia standard solution 1 in a 100mL volumetric flask; dilute to the mark with water; and shake well.

Appendix C

(Normative)

Test Method for Ammonium Ion Content in Fly Ash - Nessler's Reagent Spectrophotometry

C.1 Principle

Mix the fly ash with water; adjust the pH value to alkaline for distillation; then absorb the distilled NH_3 by the dilute sulfuric acid solution; and transfer the ammonium ions in the fly ash to the distillate. The ammonia nitrogen in the form of ammonium ion (NH_4^+) reacts with the alkaline solution of mercury iodide and potassium iodide to form a reddish-brown complex whose absorbance is proportional to the content of ammonia nitrogen. The absorbance is measured at a wavelength of 420nm. Use the content of ammonia nitrogen in the distillate to calculate the content of ammonium ions in fly ash.

C.2 Basic requirements of the test

The same as A.2.

C.3 Reagents and materials

C.3.1 Potassium sodium tartrate

Weigh 50.0g of potassium sodium tartrate and dissolve it in 100mL of water; heat it to boil and drive off ammonia; dilute to 100 mL after cooling; and prepare it immediately before use.

C.3.2 Nessler's reagent [mercury iodide-potassium iodide-sodium hydroxide (HgI_2-KI-NaOH) solution]

Weigh 16.0g of sodium hydroxide; dissolve in 50mL of water; and cool to room temperature.

Weigh 7.0g of potassium iodide and 10.0g of mercury iodide; dissolve in water; then slowly add this solution to the above 50mL of sodium hydroxide solution as stirring; and dilute with water to 100mL. Store in a polyethylene bottle; cover tightly with a rubber stopper or polyethylene cap; and store in the dark. The validity period is 1 year.

C.3.3 Sodium hydroxide solution (200g/L)

Weigh 20g of sodium hydroxide to dissolve in water; add water to dilute to 100mL.

C.3.4 Ammonia nitrogen standard solution

Appendix D

(Normative)

Test Method for Ammonium Ion Content in Fly Ash - Ion Chromatography

D.1 Principle

Mix the fly ash with water; adjust the pH value to alkaline for distillation. Absorb the distilled NH_3 by the dilute sulfuric acid solution; and the ammonium ions in the fly ash are transferred to the distillate, which is quantified by ion chromatography. The cation analysis column is used for separation; the retention time of ammonium ions is qualitative; the peak area or peak height is quantitative. The concentration of ammonium ions (NH_4^+) in the absorption solution is determined, and then calculate the content of ammonium ions in fly ash.

D.2 Basic requirements of the test

The same as A.2.

D.3 Ammonium standard solution

D.3.1 Ammonium standard stock solution (1000μg/mL)

Certified standard substance/standard sample, ammonium standard solution: containing ammonium (NH_4^+) 1000μg/mL.

D.3.2 Preparation of ammonium standard solution (100μg/mL)

Pipette 25.00mL of ammonium standard stock solution (1000μg/mL) in a 250mL volumetric flask; dilute to the mark with water; and shake well.

D.3.3 Drawing of ammonium standard working curve

Prepare the ammonium standard working solution according to actual needs; adopt the stepwise dilution method; and use water as the blank solution at the same time. The ammonium ion concentrations in the standard working solution are usually 0.00μg/mL, 0.10μg/mL, 0.50μg/mL, 1.00μg/mL, 5.00μg/mL. This solution is prepared for current use.

Turn on the power according to the requirements of the ion chromatographic instruction manual; and debug the instrument in the best working condition. Install the cation chromatographic column and mobile phase, and when the system balances to a stable baseline, inject a series of standard solutions into the ion chromatogram to obtain a

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