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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 205-2024

Replacing GB/T 205-2008

Methods for chemical analysis of aluminate cement

铝酸盐水泥化学分析方法

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Table of Contents

4 Basic requirements for the test

4.1 Each determination is made twice. Where the absolute difference between the two results falls within the permissible difference for the same laboratory given in Table 2, the average of the two is reported as the result. Unless otherwise stated, it is recommended that the loss on ignition be determined at the same time as the other work; the other determinations shall be accompanied by a blank test and the results corrected accordingly.

4.2 Mass is expressed in grams to 0.0001 g. The burette volume is expressed in millilitres and read to 0.01 mL. The titre is expressed in milligrams per millilitre; the titre and the volume ratio are kept to four significant figures after rounding. Unless otherwise stated, every analytical result is expressed as a mass fraction; the chloride ion result is kept to three decimal places and the other results to two. Rounding follows GB/T 8170.

4.3 In the blank test the same quantity of reagents is used without the sample, the same steps are followed, and the result obtained is used to correct the determination.

4.4 The filter paper and the precipitate are placed in a crucible that has already been ignited to constant mass, the lid is set on with a gap left, and the charge is dried and ashed slowly in an oxidizing atmosphere so that no flame arises; once no black carbon particles remain it is placed in the high-temperature furnace and ignited at the prescribed temperature, cooled to room temperature in a desiccator and weighed.

4.5 After the first ignition, cooling and weighing, constant mass is checked by repeating the ignition of the vessel or the test portion for 15 min at a time, then cooling and weighing; constant mass is reached when two successive weighings differ by less than 0.0005 g.

4.6 For the silver nitrate check for chloride, the precipitate is washed the prescribed number of times, the lower end of the funnel is rinsed with a few drops of water, the filter paper and the precipitate are washed with a few millilitres of water, the filtrate is collected in a test tube, a few drops of silver nitrate solution are added and the tube is examined for turbidity. If it is turbid, washing and checking continue until the silver nitrate check no longer gives

turbidity.

4.7 The methods listed in the document shall be verified against a national reference sample or certified reference material, such as GSB08-1533, or by comparison between different methods, in order to confirm their accuracy.

5 Reagents and materials

5.1 Unless otherwise stated the reagents shall be not lower than analytical grade, and the reagents used for standardization shall be primary standards. The water used shall be not lower than grade three of GB/T 6682. Carbon dioxide free water means water freshly boiled and cooled to room temperature. The reagents used for ion chromatography and for inductively coupled plasma emission spectrometry shall be not lower than guaranteed grade and the water not lower than grade two of GB/T 6682. Unless otherwise stated, the density of the commercial concentrated liquid reagents is the density at 20 °C in grams per cubic centimetre. Where no concentration is given for an acid or for ammonia, the commercial concentrated acid or concentrated ammonia is meant. The degree of dilution of a reagent is expressed as a volume ratio; hydrochloric acid (1+5), for example, means one volume of concentrated hydrochloric acid mixed with five volumes of water.

5.2 to 5.9 list the concentrated reagents with their density and content: hydrochloric acid, hydrofluoric acid, nitric acid, glacial acetic acid, hydrogen peroxide, ammonia, ethanol or absolute ethanol, and sulfuric acid.

5.10 to 5.14 list the diluted acid and ammonia solutions: hydrochloric acid (1+1), (1+2), (1+3) and (1+11); nitric acid (1+1), (1+6), (1+9) and (1+49); sulfuric acid (1+1) and (1+9); ammonia (1+1); acetic acid (1+1).

5.25 The Eschka reagent is prepared by mixing two parts by mass of light magnesium oxide with one part by mass of anhydrous sodium carbonate, grinding the mixture to a particle size below 0.2 mm and keeping it in a closed container. A blank test shall be run on every batch of Eschka reagent prepared.

5.41.1 For the silicon dioxide standard solution, 0.2000 g of spectrally pure silicon dioxide ignited for 1 h at 1000 °C to 1100 °C is weighed to 0.0001 g into a platinum crucible, 2 g of anhydrous sodium carbonate is added and stirred in, and the mixture is fused for 15 min at 1000 °C to 1100 °C. After cooling, the melt is leached with water in a 300 mL plastic beaker holding hot water; once dissolution is complete the solution is cooled to room temperature, transferred to a 1000 mL volumetric flask, diluted to the mark, shaken and kept in a plastic bottle. This solution contains 0.2 mg of silicon dioxide per millilitre. A 50.00 mL portion of it is diluted to the mark in a 500 mL volumetric flask and kept in a plastic bottle, giving a solution that contains 0.02 mg of silicon dioxide per millilitre.

5.48.1 For the EDTA standard titration solution, 5.6 g of disodium ethylenediaminetetraacetate dihydrate is placed in a beaker, about 200 mL of water is

added, the salt is dissolved by heating, and the solution is filtered, diluted to 1 L and shaken.

5.52 The calcein - methylthymol blue - phenolphthalein mixed indicator, called the CMP mixed indicator, is prepared by grinding together 1.000 g of calcein, 1.000 g of methylthymol blue, 0.200 g of phenolphthalein and 50 g of potassium nitrate dried at 105 °C to 110 °C, and is kept in a ground-glass stoppered bottle.

5.53 The acid chrome blue K - naphthol green B mixed indicator, called the KB mixed indicator, is prepared by grinding together 1.000 g of acid chrome blue K, 2.500 g of naphthol green B and 50 g of potassium nitrate dried at 105 °C to 110 °C, and is kept in a ground-glass stoppered bottle. Where the colour at the end point is not right, the ratio of acid chrome blue K to naphthol green B may be adjusted and confirmed by comparison against a national reference sample or certified reference material.

7 Preparation of the sample

7 The sample is taken according to GB/T 12573 and reduced by quartering to about 100 g. It is sieved on a 150 μm square-mesh sieve and foreign matter is removed; where necessary metallic iron is picked out of the oversize with a magnet. The oversize is ground until all of it passes the 150 μm square-mesh sieve, and the whole is mixed well and put into a clean dry sample bottle, which is sealed. Sample preparation is carried out as quickly as possible so that the material does not take up moisture. Cement and cement clinker samples need not be dried before analysis.

8 Determination of the loss on ignition - ignition difference method

8.1 The sample is ignited in a high-temperature furnace at 950 °C +/- 25 °C; the mass lost by the ignition is the loss on ignition.

8.2 1 g of the sample is weighed to 0.0001 g into a porcelain crucible already ignited to constant mass, the lid is set on with a gap left, and the crucible is placed in the high-temperature furnace, where the temperature is raised from a low value and the charge is ignited for 30 min to 40 min at 950 °C +/- 25 °C. The crucible is taken out and cooled to room temperature in a desiccator, and the ignition is repeated until constant mass is reached.

8.3 The mass fraction of the loss on ignition is calculated by formula (11) from the mass of the test portion and its mass after ignition, and is expressed as a percentage.

9 Determination of silicon dioxide - silicomolybdenum blue spectrophotometry (reference method)

9.1 In acid solution silicic acid forms a yellow complex with ammonium molybdate, which is