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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

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Steel slag - Determination of total iron content - Titanium(III) chloride-potassium dichromate titration method

钢渣 全铁含量的测定 三氯化钛-重铬酸钾滴定法

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0 Note on the printed English title

The English title is printed on the front page as Steel slag - Determination of total iron content - Titanium(III) chloride-potassium dichromate titration methods, with a plural on the last word. The document describes one method, and the Chinese title is singular, so the plural is treated here as a printing slip and the title is given with method in the singular. The Roman numeral three in the oxidation state is printed as a single ideographic character and is written here with three capital letters.

No other change has been made to the wording of the printed English title.

1 Scope

A warning printed before Clause 1 states that users of the document are to have practical experience of regular laboratory work; that the document does not set out all the safety problems that may arise; and that it is for the user to take suitable safety and health measures and to see that the relevant national regulations are met.

The document describes the method for determining total iron content by reduction with

titanium(III) chloride and titration with potassium dichromate.

It applies to the determination of total iron content in steel slag, including converter slag, electric furnace slag and refining slag. The measuring range, as a mass fraction, is 1.0 % to 35.0 %.

3 Terms and definitions

The document states that there is no term needing definition in it.

4 Principle

The test portion is decomposed on heating in a sulfuric-phosphoric acid mixture. Most of the iron(III) in the test solution is reduced with stannous chloride; sodium tungstate then serves as the indicator and titanium(III) chloride reduces all the remaining iron(III) to iron(II), until tungsten blue is formed. The excess reducing agent is oxidised with dilute potassium dichromate solution, or left to be oxidised naturally by the oxygen of the air. In the sulfuric-phosphoric medium, with sodium diphenylamine sulfonate as the indicator, the iron(II) is titrated with standard potassium dichromate titration solution and the mass fraction of total iron is calculated.

5 Reagents

Unless otherwise stated, only reagents of analytical grade or better are used, and the water is distilled or deionised water meeting at least grade three of GB/T 6682.

Sulfuric acid of density 1.84 g/mL; phosphoric acid of density 1.69 g/mL; sulfuric-phosphoric mixed acid, 3+3+4, that is 3 parts sulfuric acid plus 3 parts phosphoric acid plus 4 parts water; nitric acid of density 1.42 g/mL; hydrofluoric acid of density 1.15 g/mL; hydrochloric acid of density 1.19 g/mL; hydrochloric acid 1+4.

Stannous chloride solution, 60 g/L: 6 g of stannous chloride is dissolved in 20 mL of hydrochloric acid of density 1.19 g/mL, diluted to 100 mL with water and mixed. Potassium permanganate solution, 4 g/L. Sodium tungstate solution, 250 g/L: 25 g of sodium tungstate is dissolved in a suitable amount of water, 5 mL of phosphoric acid is added, the solution is diluted to 100 mL with water and mixed. Titanium(III) chloride solution, 1+14: 2 mL of titanium(III) chloride solution of about 15 % mass concentration by volume is diluted to 30 mL with hydrochloric acid 1+5, and is prepared fresh at the time of use. Potassium dichromate solution, 1 g/L. Sodium diphenylamine sulfonate indicator solution, 2 g/L.

Ammonium iron(II) sulfate solution of concentration 0.050 mol/L in iron(II): 19.7 g of ammonium iron(II) sulfate hexahydrate is dissolved in sulfuric acid 5+95, transferred to a 1 000 mL volumetric flask, made up to the mark with sulfuric acid 5+95 and mixed.

Standard potassium dichromate titration solution of concentration 0.050 00 mol/L,

expressed as one sixth of potassium dichromate: 2.451 5 g of potassium dichromate of primary reagent grade, dried beforehand for 2 h at 140 °C to 150 °C and cooled to room temperature in a desiccator, is weighed into a 250 mL beaker, dissolved in water, transferred to a 1 000 mL volumetric flask, made up to the mark with water and mixed thoroughly; the temperature at which the solution was prepared is noted.

Standard potassium dichromate titration solution of concentration 0.010 00 mol/L, expressed as one sixth of potassium dichromate: prepared in the same way from 0.490 3 g of the dried salt. The volumetric flasks used are to meet the class A requirements of GB/T 12806 and are to be calibrated beforehand where necessary. Two notes add that where a potassium dichromate reference material of certified content is used the mass to be weighed may be calculated from that content, and that the temperature at titration is to be kept as close as possible to the temperature at which the standard solution was prepared.

6 Instruments and equipment

Unless otherwise specified, all burettes, volumetric flasks and pipettes are to meet GB/T 12805, GB/T 12806 and GB/T 12808.

A drying oven able to hold 105 °C +/- 2 °C, and a weighing scoop made of non-magnetic material or of demagnetised stainless steel.

7 Sample preparation

The test sample is prepared by the method of GB/T 2007.2, metallic iron being separated with a magnet. The particle size is to be below 0.125 mm. The screened sample is dried for 1 h in the oven at 105 °C +/- 2 °C, then taken out, cooled to room temperature in a desiccator and held for use.

8 Analytical procedure

8.1 A test portion of 0.20 g is weighed to 0.000 1 g.

8.2 At least two independent determinations are made on the same test portion material.

8.3 A blank test is run with the test portion.

8.4.1 The test portion is placed in a 300 mL conical flask and moistened with a little water; 15 mL of the sulfuric-phosphoric mixed acid, 2 mL of nitric acid and 3 mL of hydrofluoric acid are added and the portion is dissolved on gentle heating, the flask being swirled two or three times; the temperature is then raised until sulfuric fumes are given off, and the flask is taken off when the fumes stand 50 mm to 60 mm above the surface of the liquid. A note adds that for high-silicon steel slag samples rather more hydrofluoric acid may be added according to the silicon content, and that where there is no hydrofluoric acid 0.5 g to 1 g of a fluoride may be added as a flux.

8.4.2 After slight cooling, 20 mL of hydrochloric acid 1+4 is added down the wall of the flask and stannous chloride solution is added dropwise while hot until the solution turns pale yellow; the solution is cooled to room temperature, about 50 mL of water and 1 mL of sodium tungstate solution are added, and titanium(III) chloride solution is added dropwise until the solution turns blue. A note adds that if the stannous chloride solution has been added in excess the solution becomes colourless, and that potassium permanganate solution may then be added dropwise until it is pale yellow, after which this step is repeated.

8.4.3 Potassium dichromate solution is added dropwise until the blue colour goes, or the solution is left until the oxygen of the air discharges it; 5 drops of sodium diphenylamine sulfonate indicator solution are added, and the solution is titrated with the 0.050 00 mol/L standard potassium dichromate titration solution from green through blue-green to the end point, at which the last drop turns it purplish red. A note adds that where the iron content is below 5 % the 0.010 00 mol/L standard solution is used.

8.4.4 The blank value is determined at the same time as the test portion, by the same steps and with the same amounts of reagent. Before the sulfuric-phosphoric mixed acid is added, 5.00 mL of the ammonium iron(II) sulfate solution and 5 drops of the indicator solution are added and the solution is titrated with the standard potassium dichromate solution to the end point, consuming V1; a further 5.00 mL of the ammonium iron(II) sulfate solution is added and the solution is titrated again to the end point, consuming V2. The difference between the two volumes consumed is the blank value V0, the document giving V0 as V1 minus V2.

9 Calculation of the analytical result

The mass fraction of total iron, in percent, is calculated by formula (1). The formula comes out of the extracted text with its fraction bar lost and is not reconstructed here. Its symbols are: c, concentration of the standard potassium dichromate titration solution, in moles per litre; V, volume of standard potassium dichromate titration solution consumed in titrating the test solution, in millilitres; V0, volume of standard potassium dichromate titration solution consumed in titrating the blank test solution, in millilitres; m, mass of the test portion, in grams; and 55.85, the molar mass of iron, in grams per mole.

Where the sample contains vanadium, the following correction is applied: 0.1 % of vanadium corresponds to 0.11 % of iron.

The analytical result is rounded as laid down in GB/T 8170 and kept to two decimal places.

10 Precision

Precision was established in an interlaboratory trial in which 9 laboratories tested 6 samples at different levels, each laboratory making 3 independent determinations at each level.