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Replacing GB/T 6730.73-2016

Iron ores - Determination of total iron content - EDTA photometric titration method

铁矿石 全铁含量的测定 EDTA光度滴定法

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1 Scope

Warning - persons using this document need practical experience of work in a proper laboratory. This document does not set out every possible safety problem. It is the responsibility of the user to take appropriate safety and health measures and to ensure compliance with the conditions laid down in the relevant national regulations.

This document describes a method for the determination of total iron content in iron ores by EDTA photometric titration.

This document applies to the determination of total iron content in natural iron ore, iron ore concentrate and man-made lump ore, including sinter and pellets. Determination range, as a mass fraction: 30.0 percent to 72.0 percent.

2 Normative references

The contents of the following documents constitute indispensable provisions of this document through normative reference in the text. For dated references, only the edition corresponding to that date applies to this document; for undated references, the latest edition, including all amendments, applies to this document.

GB/T 6730.1 Iron ores - Preparation of predried test samples for chemical analysis

GB/T 6730.3 Iron ores - Determination of hygroscopic moisture in analytical samples - Gravimetric, Karl Fischer and mass loss methods

GB/T 8170 Rules of rounding off for numerical values and expression and judgement of limiting values

GB/T 10322.1 Iron ores - Sampling and sample preparation procedures

GB/T 12805 Laboratory glassware - Burettes

GB/T 12806 Laboratory glassware - One-mark volumetric flasks

GB/T 12808 Laboratory glassware - One-mark pipettes

3 Terms and definitions

There are no terms and definitions that need to be defined in this document.

4 Principle

4.1 Method 1 - microwave digestion. The test portion is decomposed by microwave digestion in a mixed solution of hydrochloric acid, nitric acid and hydrofluoric acid. The solution is diluted with water and adjusted with sodium hydroxide to pH 1. Sulfuric acid is used to remove the interference of titanium and boric acid to reduce the effect of fluoride. The solution is titrated with standard disodium ethylenediaminetetraacetate solution and the total iron content is calculated.

4.2 Method 2 - alkali fusion. The test portion is fused with sodium peroxide and sodium hydroxide. The fused mass is dissolved in hydrochloric acid and nitric acid. Sulfuric acid is used to remove the interference of titanium, ammonium fluoride to mask aluminium, and boric acid to remove excess fluoride. The solution is diluted with water and adjusted with sodium hydroxide to pH 1, then titrated with standard disodium ethylenediaminetetraacetate solution and the total iron content is calculated.

5 Reagents

Unless otherwise stated, only reagents confirmed to be of analytical grade and distilled water, deionized water or water of equivalent purity are used in the analysis.

5.1 High purity iron powder, of purity (mass fraction) not less than 99.98 percent. 5.2 Sodium hydroxide. 5.3 Disodium ethylenediaminetetraacetate ($C_{10}H_{14}N_2Na_2O_8$, EDTA). 5.4 Sodium peroxide. 5.5 Ammonium fluoride. 5.6 Boric acid. 5.7 Hydrochloric acid, of density 1.19 g/mL. 5.8 Nitric acid, of density 1.42 g/mL. 5.9 Hydrofluoric acid solution, made up 1 to 9 by volume. 5.10 Sulfuric acid solution, made up 1 to 1 by volume.

5.11 Boric acid solution: weigh 100 g of boric acid (5.6) into a 1 L beaker, add 25 g of sodium hydroxide (5.2), make up to 1 L with water and mix. Store in a plastic bottle. The

mass concentration of this solution is 100 g/L.

5.12 Sodium hydroxide solution: weigh 100 g of sodium hydroxide (5.2) into a 1 L beaker, add water to 500 mL and mix. The mass concentration of this solution is 200 g/L. Store in a plastic bottle.

5.13 Iron standard solution: weigh 2.792 g of high purity iron powder (5.1) into a 250 mL beaker and add 5 mL of hydrochloric acid (5.7) and 5 mL of nitric acid (5.8). Cover with a watch glass and heat on a hot plate at 80 degrees Celsius until dissolution is complete. After cooling, transfer to a 500 mL volumetric flask and dilute to the mark with water. The concentration of this solution is 0.100 0 mol/L.

5.14.1 Preparation of the standard EDTA titration solution: weigh 40 g of disodium ethylenediaminetetraacetate (5.3), add 800 mL of water, heat to dissolve, cool, dilute to 1 000 mL and mix. Store in a plastic bottle. The concentration of this solution is 0.1 mol/L.

5.14.2.1 Dissolution of the high purity iron powder for standardisation: weigh 0.20 g of high purity iron powder (5.1), to the nearest 0.000 2 g, into a 250 mL beaker and add 5 mL of hydrochloric acid (5.7) and 5 mL of nitric acid (5.8). Cover with a watch glass and heat on a hot plate at 80 degrees Celsius until dissolution is complete. While stirring with a glass rod, add 2 mL of sulfuric acid solution (5.10), 15 mL of sodium hydroxide solution (5.12) and 5 mL of sulfosalicylic acid solution (5.15), rinse off the glass rod and dilute to 150 mL with water.

5.14.2.2 Titration: cover with a watch glass and heat to boiling on a hot plate at 210 degrees Celsius. Take the beaker off and rinse the watch glass and the inner wall of the beaker. While the solution is still hot, above 80 degrees Celsius, place the beaker on the titration stand of the automatic potentiometric titrator and insert the combination glass pH electrode (6.3). Stir with a magnetic stirrer or with the rod stirrer supplied with the instrument and, with continuous stirring, add sodium hydroxide solution (5.12) to adjust the acidity of the solution to pH 1 plus or minus 0.05. Insert the photometric electrode (6.4) and the titration tip, check that both are immersed in the solution, and make sure that there are no bubbles in the light path of the photometric electrode. Run the titration programme and titrate with the standard EDTA titration solution (5.14.1) under continuous stirring. Repeat the determination four times and record the four end points and the volumes of standard EDTA titration solution consumed. Examples of titration curves are given in Annex B. If bubbles appear during the titration, the titration may be paused and the photometric electrode withdrawn from the solution and reinserted to clear them. Note: temperature compensation is not used when adjusting the acidity; at the end of the titration the temperature of the solution shall be kept above 50 degrees Celsius.

5.14.2.3 The EDTA concentration without blank correction is calculated by formula (1) and expressed in mol/L, to four decimal places. In the formula the symbols have the following meanings: the number of the determination, one to four; the mass of high purity iron

powder, in grams; the volume of standard EDTA titration solution consumed at the end point, in millilitres; and 0.055 847, the conversion factor for iron. If the range of the four results, maximum minus minimum, is equal to or less than 0.000 2 mol/L, the mean of the four results is taken as the final result; if the range is greater than 0.000 2 mol/L, the median is taken as the final result.

5.14.3 Reagent blank: pipette 1.00 mL of the iron standard solution (5.13) into a 250 mL beaker and add 5 mL of hydrochloric acid (5.7), 5 mL of nitric acid (5.8) and 2 mL of sulfuric acid solution (5.10). While stirring with a glass rod, add 15 mL of sodium hydroxide solution (5.12) and 5 mL of sulfosalicylic acid solution (5.15), rinse off the glass rod and dilute to 150 mL with water. Proceed as in 5.14.2.2, recording the four end points and the volumes of standard EDTA titration solution consumed. If the range of the four volumes is equal to or less than 0.02 mL, the mean is taken as the final result; if the range is greater than 0.02 mL, the median is taken. The reagent blank value is then calculated by formula (2) as the final volume result minus the volume of standard EDTA titration solution equivalent to 1.00 mL of the iron standard solution, the result being calculated to two decimal places; that equivalent volume is itself obtained by formula (3) from the volume and concentration of the iron standard solution and the uncorrected concentration of the EDTA solution.

If there is no iron in the solution, the iron sulfosalicylate complex cannot form and photometric titration is not possible. Iron is therefore added when the blank test is carried out, and the added iron is converted into the equivalent volume of standard EDTA titration solution. A 1 mL single-mark pipette shall first be calibrated by weighing the water it delivers and converting that mass to a volume.

5.14.4 Calculation of the concentration of the standard EDTA titration solution: the concentration is calculated by formula (4), expressed in mol/L to four decimal places, from the mass of pure iron powder, the volume of standard EDTA titration solution consumed at the end point, the reagent blank value obtained in 5.14.3, and the conversion factor for iron. If the range of the four results is equal to or less than 0.000 2 mol/L, the mean of the four is taken as the final result; if the range is greater than 0.000 2 mol/L, the median is taken.

5.15 Sulfosalicylic acid solution: weigh 10 g of sulfosalicylic acid, dissolve it in 100 mL of water and keep it in a 125 mL reagent bottle. The mass concentration of this solution is 100 g/L.

6 Apparatus

Unless otherwise specified, ordinary laboratory apparatus is used. Burettes, one-mark volumetric flasks and one-mark pipettes shall comply with GB/T 12805, GB/T 12806 and GB/T 12808 respectively.

6.1 Analytical balance, with a scale interval of 0.1 mg.

6.2 Automatic potentiometric titrator, able to deliver both at fixed intervals and dynamically.