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GB/T 3653.1-2024

Replacing GB/T 3653.1-1988

Ferroboron - Determination of boron content - Alkalimetry method

硼铁 硼含量的测定 碱量滴定法

Issued on: May 28, 2024 **Implemented on: December 1, 2024**

Issued by: State Administration for Market Regulation;
Standardization Administration of the PRC

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0 Note on the printed English title

The English title on the cover of the document is printed as Ferroboron-Determination of boron content-Alkalimelry method, with the last word spelt Alkalimelry and with the word spacing lost in the digitised text. The spacing has been restored and the misspelling has been corrected to Alkalimetry; no other word of the printed title has been altered.

0b Warning

Persons using this document should have practical experience of work in a regular laboratory. The document does not point out all the possible safety problems; it is the responsibility of the user to adopt suitable safety and health measures and to ensure compliance with the conditions laid down in the relevant national regulations.

1 Scope

The document specifies a method for determining the boron content of ferroboron by alkalimetric titration.

It applies to the determination of boron in ferroboron over a mass fraction range of 3.00 %

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to 26.00 %.

2 Normative references

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

GB/T 4010 Ferroalloys-Sampling and preparation of samples for chemical analysis; GB/T 6379.1 Accuracy (trueness and precision) of measurement methods and results-Part 1: General principles and definitions; GB/T 6379.2 Accuracy (trueness and precision) of measurement methods and results-Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method; GB/T 6682 Water for analytical laboratory use-Specification and test methods; GB/T 8170 Rules of rounding off for numerical values and expression and judgement of limiting values; GB/T 12806 Laboratory glassware-One-mark volumetric flasks; GB/T 12808 Laboratory glassware-One-mark pipettes.

3 Terms and definitions

The document lists no terms and definitions that require defining.

4 Principle

The test portion is decomposed by fusion with anhydrous sodium carbonate or with potassium sodium carbonate and sodium peroxide, leached with water, and the iron, manganese and nickel are separated by dry filtration. Part of the filtrate is taken, citric acid is added to mask aluminium, and the test solution is adjusted to pH 6.9 under the control of a pH meter; mannitol is then added and the solution is titrated with sodium hydroxide standard solution to pH 6.9, which is the end point.

5 Reagents and materials

Unless otherwise stated, only approved analytical grade or better reagents are used in the analysis, together with grade two or better distilled water complying with GB/T 6682 or water of equivalent purity.

The list comprises: anhydrous sodium carbonate, solid; potassium sodium carbonate, solid; sodium peroxide, solid; mannitol, solid; anhydrous ethanol, described in the document as solid; citric acid solution 20 g/L; hydrochloric acid 1+1; methyl red indicator 1 g/L; sodium hydroxide solution 100 g/L.

Boron standard solution: 11.4320 g of primary reference boric acid is weighed into a 500

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mL beaker, 200 mL of water is added and, after dissolution, the solution is transferred to a 500 mL volumetric flask, diluted to the mark with water and mixed; 1 mL of this solution contains 0.0040 g of boron.

Sodium hydroxide standard solution, c(NaOH) = 0.1 mol/L. Preparation: 4 g of sodium hydroxide is weighed and dissolved in 250 mL of previously boiled water, shaken, poured into a polytetrafluoroethylene container, and left closed until the solution is clear; it is diluted to 1 L with previously boiled water and mixed, and the clear upper layer is taken with a plastic tube into another bottle and used after standardisation.

Standardisation: 5.00 mL of the boron standard solution is transferred to a 250 mL beaker, 100 mL of water and 10 mL of citric acid solution are added, the electrodes are inserted and connected to the pH meter, the magnetic stirrer is started, the solution is adjusted to pH 6.9 with the sodium hydroxide solution, 20 g of mannitol is added and the solution is titrated with the sodium hydroxide standard solution to pH 6.9, which is the end point. The titre T of the sodium hydroxide standard solution for boron, in g/mL, is calculated from equation (1); the equation as printed could not be reconstructed from the digitised text and is therefore not reproduced. Its symbols are V0, volume of boron standard solution taken for the titration, in mL; c, boron content of 1 mL of the boron standard solution, in g/mL; V1, volume of sodium hydroxide standard solution used in the titration, in mL.

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6 Instruments and equipment

Unless specifically stated otherwise, ordinary laboratory instruments are used in the analysis; one-mark volumetric flasks and one-mark pipettes comply with GB/T 12806 and GB/T 12808 respectively.

The equipment comprises a pH meter with a smallest scale division of 0.01; an analytical balance with a readability of 0.1 mg; a magnetic stirrer with a plastic coated stirring bar and speed control; a glass electrode; and a calomel electrode.

7 Sampling and sample preparation

Sampling and sample preparation are carried out as specified in GB/T 4010; the whole of the test sample passes a 0.088 mm sieve.

8 Analytical procedure

Test portion: 0.50 g of sample is weighed to the nearest 0.0001 g.

Number of determinations: at least 2 independent determinations are carried out on the same sample.

Blank test: a blank test is carried out along with the test portion.

Determination, first step: the test portion is placed in a nickel or iron crucible containing 4 g

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of anhydrous sodium carbonate or 4 g of potassium sodium carbonate and heated in a furnace at 500 °C to 600 °C until the sample and the flux just turn red; it is then removed and cooled. 6 g of sodium peroxide is added, the crucible is placed at the mouth of the furnace and, once the sodium peroxide has become liquid, it is moved to the middle of the furnace at a furnace temperature of 800 °C to 850 °C, fused for 15 min to 20 min, removed and cooled.

Determination, second step: the crucible with the melt is placed in a 300 mL quartz beaker or a 500 mL polytetrafluoroethylene beaker containing 120 mL of water and leached with heating; the crucible is then removed and washed. If a little melt is found stuck to the bottom of the crucible, a few drops of hydrochloric acid 1+1 may be added and the residue dissolved with slight heating before the crucible is removed and washed. 2 mL of anhydrous ethanol is added, the solution is boiled for 3 min to 5 min, cooled, transferred to a 500 mL volumetric flask, diluted to the mark with water, mixed and left to stand for a moment.

Determination, third step: the solution is dry filtered through a double layer of rapid filter paper and 100.0 mL of the filtrate is transferred to a 300 mL quartz beaker; 10 mL of citric acid solution and 2 drops of methyl red indicator are added and the solution is neutralised with hydrochloric acid 1+1 until it turns red. The beaker is covered with a watch glass, the solution is heated and boiled for 10 min, cooled to room temperature, and made up with carbon dioxide free water to a volume of about 120 mL. The electrodes and the pH meter are connected and the stirrer is started. The solution is adjusted with the sodium hydroxide solution to about pH 6.0 and then with the sodium hydroxide standard solution to pH 6.90 plus or minus 0.02; 20 g of mannitol is added and, with continuous stirring, the solution is titrated with the sodium hydroxide standard solution to pH 6.90 plus or minus 0.02, which is the end point.

9 Calculation and expression of results

The boron content of the sample, expressed as a mass fraction in %, is calculated from equation (2). The equation as printed could not be reconstructed from the digitised text and is therefore not reproduced; its symbols are V2, volume of sodium hydroxide standard solution consumed in titrating the test solution, in mL; V3, volume of sodium hydroxide standard solution consumed by the blank run with the sample, in mL; T, titre of the sodium hydroxide standard solution for boron, in g/mL; m, mass of the test portion, in g; r, aliquot ratio of the test solution.

If the absolute value of the difference between two independent analytical results on the same sample is not greater than the repeatability limit r, the arithmetic mean is taken as the analytical result. If that absolute difference is greater than the repeatability limit r, further determinations are made and the result is established as specified in Annex A. The analytical result is rounded in accordance with GB/T 8170.

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