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Ferroniobium - Determination of aluminum content - EDTA titrimetric method

铌铁 铝含量的测定 EDTA滴定法

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0 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 aluminum content of ferroniobium by EDTA titration.

It applies to the determination of aluminum in ferroniobium over a mass fraction range of 1.00 % to 9.00 %.

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 fused with sodium peroxide and sodium hydroxide, leached with sodium chloride solution and manganese is reduced with ethanol; the bulk of the niobium, iron, titanium and the manganese are separated by dry filtration. Part of the filtrate is acidified, hydrogen peroxide is added to test solutions containing tungsten, the acidity of the solution is adjusted to pH 3 to 3.5 and aluminum is precipitated with ammonium benzoate. After separation, an excess of EDTA solution is added to the aluminum in acid solution. At pH 5.5 to 6.2, in the presence of xylenol orange indicator, the excess EDTA is titrated with zinc standard solution; the EDTA complexed with aluminum is then displaced with sodium fluoride and titrated again with zinc standard solution.

5 Reagents

Unless otherwise stated, only approved analytical grade reagents are used in the analysis, and the water used is distilled water of grade three or better as specified in GB/T 6682, or water of equivalent purity.

The list comprises: sodium peroxide, solid; sodium hydroxide, solid; sodium fluoride, solid; hydrochloric acid of density 1.19 g/mL; hydrochloric acid 1+1; nitric acid of density 1.42 g/mL; perchloric acid of density 1.67 g/mL; hydrogen peroxide 30 %; anhydrous ethanol; ammonia solution of density 1.15 g/mL; ammonia solution 1+1; sodium chloride solution 200 g/L; ammonium acetate solution 200 g/L; ammonium benzoate solution 100 g/L; wash solution, prepared by adding 1 mL of the ammonium benzoate solution to 88 mL of water; 4-nitrophenol solution 1 g/L; acetic acid and sodium acetate buffer solution, prepared by dissolving 20 g of sodium acetate trihydrate in water, adding 0.9 mL of glacial acetic acid, transferring to a 100 mL volumetric flask, diluting to the mark with water and mixing; xylenol

orange solution 2 g/L; methyl orange solution 1 g/L.

EDTA standard solution, c(EDTA) = 0.01000 mol/L: 3.7226 g of disodium ethylenediaminetetraacetate dihydrate, primary reference reagent, is dissolved in water, transferred to a 1000 mL volumetric flask, diluted to the mark with water and mixed.

Aluminum standard solution, 0.5 mg/mL: 0.5000 g of high purity aluminum is placed in a plastic beaker, 20 mL of 10 % sodium hydroxide solution is added and the beaker is heated in a hot water bath until dissolution is complete. After cooling, the solution is acidified by pouring in 30 mL of the hydrochloric acid of density 1.19 g/mL in one operation, mixed, cooled to room temperature, transferred to a 1000 mL volumetric flask, diluted to the mark with water and mixed; 1 mL of this solution contains 0.50 mg of aluminum.

Zinc standard solution, c(Zn) = 0.01 mol/L: it is prepared either by dissolving 0.6538 g of zinc of purity above 99.9 % in a small volume of hydrochloric acid 1+1, diluting with water to about 200 mL, adjusting with the two ammonia solutions until Congo red paper turns purplish grey, transferring to a 1000 mL volumetric flask, diluting to the mark and mixing; or by weighing 0.8138 g of zinc oxide, primary reference reagent, previously ignited to constant mass at 800 °C, wetting it with water in a beaker, adding 20 mL of hydrochloric acid 1+1, dissolving with heating, evaporating to 3 mL to 5 mL, transferring to a 1000 mL volumetric flask, adding 2 to 3 drops of methyl orange solution, neutralising with the ammonia solution 1+1 until the colour is yellow, then adding hydrochloric acid 1+1 until the colour turns red plus an excess of 5 to 6 drops, diluting to the mark with water and mixing.

Standardisation of the zinc standard solution: a volume of aluminum standard solution corresponding to the aluminum content of the test portion is transferred to a pure iron crucible and evaporated to dryness, 0.20 g of niobium metal is added, and the procedure of 8.4 is followed, omitting 8.4.3; a blank test is carried out by the same procedure, with no aluminum standard solution added to the iron crucible. The titre of the zinc standard solution for aluminum is obtained from equation (1). The equation as printed could not be reconstructed from the digitised text and is therefore not reproduced; its symbols are T, titre of the zinc standard solution for aluminum, in mg/mL; V, volume of aluminum standard solution taken for the standardisation, in mL; C, concentration of the aluminum standard solution, in mg/mL; K, aliquot ratio of the test solution, equal to 0.25; V1, volume of zinc standard solution consumed in the standardisation, in mL; V0, volume of zinc standard solution consumed by the reagent blank in the standardisation, in mL.

6 Instruments and equipment

Ordinary laboratory instruments and equipment are used; unless otherwise specified, volumetric flasks and pipettes conform to GB/T 12806 and GB/T 12808 respectively.

Analytical balance with a readability of 0.1 mg.

7 Sampling and sample preparation

Sampling and sample preparation are carried out as specified in GB/T 4010; the test sample passes a 0.125 mm sieve.

8 Analytical procedure

Test portion: 0.25 g of sample is weighed to the nearest 0.0001 g.

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

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

Determination, first step: the test portion is placed in a pure iron crucible with 5 g of sodium peroxide and 5 g of sodium hydroxide, stirred and covered, and fused for 10 min to 15 min in a furnace at 700 °C; it is then removed, cooled to room temperature and placed in a 250 mL polytetrafluoroethylene beaker containing 50 mL of sodium chloride solution, and the melt is leached with heating at low temperature, 100 mL of hot water being added for the leaching. The cover and the crucible are removed and washed, 5 mL of anhydrous ethanol is added and the solution is mixed; heating is continued to boiling, gentle boiling is maintained for 5 min and the solution is cooled to room temperature under running water.

Determination, second step: the solution is transferred to a 200 mL volumetric flask, diluted to the mark with water and mixed, then poured quickly into a dry plastic beaker and left to stand for a moment; it is dry filtered through two rapid quantitative filter papers in a plastic funnel into a plastic beaker and the precipitate is discarded. Ferroniobium containing tungsten is treated by the third step, ferroniobium free from tungsten by the fourth step.

Determination, third step, for material containing tungsten: 50.00 mL of the filtrate is transferred to a 250 mL beaker, 15 mL of hydrochloric acid of density 1.19 g/mL and 5 mL of hydrogen peroxide are added quickly and the solution is mixed. It is heated to boiling until large bubbles form, the hydrogen peroxide being completely decomposed. After removal and slight cooling, 10 mL of ammonium acetate solution is added, the solution is adjusted to pH 3 to 3.5 with the two ammonia solutions and heated to near boiling, and 10 mL of ammonium benzoate solution is added dropwise, followed by mixing. The solution is held for 30 min, filtered hot through a medium speed filter paper, and the beaker and the precipitate are washed 2 to 3 times with warm wash solution. The precipitate and the filter paper are transferred to the original beaker, 5 mL of nitric acid and 5 mL of perchloric acid are added, the filter paper is destroyed on a high temperature hot plate and the solution is fumed with perchloric acid to near dryness; it is then removed, cooled to room temperature, 50 mL of water is added and the salts are dissolved with slight heating, after which the fifth step is followed.

Determination, fourth step, for material free from tungsten: 50.00 mL of the filtrate is taken