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Ferroniobium — Determination of niobium content — Gravimetric method

Ferro-niobium — Dosage du niobium — Méthode gravimétrique

0 Introduction

Studies of the methods specified in this Technical Report have revealed that it is not possible, using wet chemical methods, to obtain results of sufficient accuracy to satisfy the criteria for the publication of an International Standard. For this reason, it has been decided to publish the methods in the form of a Technical report.

1 Scope and field of application

This Technical Report specifies a gravimetric method for the determination of the niobium content of ferroniobium. It also specifies a photometric method for the determination of the titanium content (annex A), a gravimetric method for the determination of tantalum contents greater than 1 % (m/m) (annex B) and a photometric method for the determination of tantalum contens less than 1 % (m/m) (annex C).

The methods are applicable to ferroniobium having niobium contents between 40 and 75 % (m/m), tantalum contents between 0,25 and 8,0 % (m/m) and titanium contents between 0,05 and 5,0 % (m/m).

Interference may arise if bismuth is present. This will appear in the tantalum fraction, but the element is seldom present in concentrations greater than 0,005 % (m/m) in ferroniobium. Trivalent antimony, if present, is eluted with titanium and precipitated with cupferron, but it does not interfere in the photometric method for the determination of titanium.

2 Reference

ISO 3713, *Ferroalloys — Sampling and preparation of samples — General rules.* [1]

3 Principle

Dissolution of a test portion in a mixture of hydrochloric acid hydrofluoric acid and passing the solution through an anion-exchange column. Elution of titanium, iron and other elements with a mixture of ammonium chloride, hydrochloric acid and hydrofluoric acid.

Treatment of this eluate with boric acid and cupferron, ignition of the precipitate, containing the titanium, fusion with potassium hydrogen sulphate, and leaching with sulphuric acid solution. Oxidation of titanium with hydrogen peroxide to the yellow pertitanate and photometric measurement at a wavelength of approximately 410 nm.

1) At present at the stage of draft.

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Elution of niobium with a mixture of ammonium chloride and hydrofluoric acid and of tantalum with an ammonium chloride/ammonium fluoride solution adjusted to a pH of 5 to 6.

Treatment of the eluates with boric acid to complex the fluorides, precipitation of niobium and tantalum with cupferron ignition, and weighing as the pentoxide.

For tantalum contents of less than 1 % (m/m), addition of zirconium as a gatherer in the cupferron separation, conversion of the tantalum to the pyrogallol complex, and photometric measurement at a wavelength of approximately 420 nm.

4 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1 Nitric acid (ϱ = 1,42 g/ml).

4.2 Hydrochloric acid (ϱ = 1,19 g/ml).

4.3 Hydrofluoric acid (ϱ = 1,14 g/ml).

4.4 Ammonium chloride, 240 g/l solution.

Dissolve, by warming, 480 g of ammonium chloride (NH_4Cl) in 1 600 ml of water, cool, dilute to 2 000 ml and mix. Filter, if necessary.

Use this stock solution the preparation of the solutions described in 4.5, 4.6 and 4.7.

4.5 Ammonium chloride/ammonium fluoride, neutral solution.

Transfer 600 ml of the ammonium chloride solution (4.4) and 40 ml of the hydrofluoric acid (4.3) to a plastic beaker. Adjust the pH to 5 to 6 with ammonium hydroxide solution [c (NH_4OH) $\simeq$ 13 mol/l; approximately 80 to 85 ml will be required], dilute to 1 000 ml with water, and mix.

NOTE — Prepare this solution with care. If the pH is too low, the volume specified will not completely elute the tantalum; if the pH is too high, tantalum will precipitate in the column, thus leading to errors in this and subsequent determinations.

4.6 Ammonium chloride/hydrochloric acid/hydrofluoric acid mixture.

Transfer 240 ml of the ammonium chloride solution (4.4), 200 ml of the hydrofluoric acid (4.3) and 150 ml of the hydrochloric acid (4.2) to a plastic bottle, dilute to 1 000 ml with water, and mix.

4.7 Ammonium chloride/hydrofluoric acid mixture.

Transfer 600 ml of the ammonium chloride solution (4.4) and 40 ml of the hydrofluoric acid (4.3) to a plastic bottle, dilute to 1 000 ml with water, and mix.

4.8 Ammonium nitrate, 20 g/l wash solution.

Dissolve 20 g of ammonium nitrate (NH_4NO_3) in water, and dilute to 1 000 ml.

4.9 Boric acid (H_3BO_3).

4.10 Cupferron, 60 g/l solution.

Dissolve 60 g of cupferron in 800 ml of cold water, dilute to 1 000 ml and filter.

This solution should be freshly prepared as required and cooled to 5 °C before use.