International Standard 4139

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Ferrosilicon — Determination of aluminium content — Flame atomic absorption spectrometric method

Ferro-silicium — Dosage de l'aluminium — Méthode par spectrométrie d'absorption atomique dans la flamme

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FOREWORD

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It has been approved by the member bodies of the following countries :

Australia	India	South Africa, Rep. of
Austria	Iran	Spain
Bulgaria	Italy	Sweden
Canada	Japan	Turkey
Czechoslovakia	Mexico	United Kingdom
Egypt, Arab Rep. of	Norway	USA
France	Poland	USSR
Germany, F. R.	Romania	Yugoslavia

No member body expressed disapproval of the document.

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Ferrosilicon — Determination of aluminium content — Flame atomic absorption spectrometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the determination of the aluminium content of ferrosilicon by flame atomic absorption spectroscopy.

The method is applicable to ferrosilicon having an aluminium content between 0,05 and 5 % (*m/m*).

2 PRINCIPLE

Dissolution of a test portion in nitric, hydrofluoric and perchloric acids. Evaporation of the solution until perchloric fumes are evolved.

Separation and fusion of the residue with a mixture of sodium carbonate and boric acid; dissolution of the fused residue in the main solution.

Aspiration of the solution in a dinitrogen monoxide-acetylene flame, and direct determination of the aluminium by absorption spectroscopy of the 309,3 nm line emitted by an aluminium hollow-cathode lamp.

3 REAGENTS

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

3.1 Sodium carbonate, anhydrous.

3.2 Nitric acid, ρ 1,40 g/ml, solution approximately 68 % (*m/m*).

3.3 Hydrofluoric acid, ρ 1,16 g/ml, solution approximately 48 % (*m/m*).

3.4 Perchloric acid, ρ 1,68 g/ml, solution approximately 70 % (*m/m*).

3.5 Hydrochloric acid, ρ 1,19 g/ml, solution approximately 38 % (*m/m*).

3.6 Hydrochloric acid, solution diluted 1 + 9.

Mix 1 volume of the hydrochloric acid solution (3.5) with 9 volumes of water and stir to mix.

3.7 Boric acid, crystalline.

3.8 Iron solution No. 1, corresponding to 10 g of Fe per litre.

Weigh, to the nearest 0,001 g, 10 g of very pure, aluminium-free iron, transfer to a 600 ml beaker and dissolve in 50 ml of the hydrochloric acid solution (3.5). Heat gently until dissolution is complete. Transfer the contents of the beaker quantitatively into a 1 000 ml volumetric flask. Make up to the mark with water and mix.

3.9 Iron solution No. 2, corresponding to 10 g of Fe per litre.

Weigh, to the nearest 0,001 g, 5 g of very pure, aluminium-free iron, transfer to a 600 ml beaker and dissolve in 25 ml of the hydrochloric acid solution (3.5). Heat gently until dissolution is complete. Add 25 ml of the perchloric acid solution (3.4). Heat until white perchloric fumes are evolved. Cool, and add 50 ml of the hydrochloric acid solution (3.5). Wait until the solution becomes clear then add 50 ml of water. Plunge into this solution a platinum crucible, in which has previously been melted a mixture of 5 g of the sodium carbonate (3.1) and 2,5 g of the boric acid (3.7), using a muffle furnace set at 1 000 °C. Heat gently until complete dissolution of the melt residue. Withdraw the crucible from the beaker and rinse it carefully into the beaker. Cool. Transfer the contents of the beaker quantitatively into a 500 ml volumetric flask. Make up to mark with water and mix.

3.10 Solution used in calibration to restore the operating conditions of the analysis.

Into a 250 ml beaker place 30 ml of the hydrochloric acid solution (3.5), 15 ml of perchloric acid (3.4) and 50 ml of water. Plunge into this solution a platinum crucible in which has previously been melted a mixture of 5 g of the sodium carbonate (3.1) and 2,5 g of the boric acid (3.7), using a muffle furnace set at 1 000 °C. Heat slowly until complete dissolution of the melt residue. Withdraw the crucible from the beaker and rinse it carefully into the beaker. Cool. Transfer the contents of the beaker quantitatively into a 200 ml volumetric flask. Make up to the mark with water and mix.