BS 6200 : Subsection 3.1.5 : 1985
ISO 4139-1979
UDC 669.1 : 543.422.062 : 546.62

British Standard

Sampling and analysis of iron, steel and other ferrous metals

Part 3. Methods of analysis
Section 3.1 Determination of aluminium

Subsection 3.1.5 Ferrosilicon: atomic absorption spectrometric method

[ISO title: Ferrosilicon — Determination of aluminium content — Flame atomic absorption spectrometric method]

Echantillonnage et analyse du fer, de l'acier et d'autres métaux ferreux
Partie 3. Méthodes d'analyse
Section 3.1 Dosage de l'aluminium
Sous-section 3.1.5 Ferro-silicium: méthode par spectrophotométrie d'absorption atomique

Probenahme und Analyse von Eisen, Stahl und anderen Eisenmetallen
Teil 3. Analysenverfahren
Abschnitt 3.1 Aluminiumbestimmung
Unterabschnitt 3.1.5 Ferrosilizium: atomabsorptionsspektrometrie Verfahren

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National foreword

This Subsection of BS 6200 has been prepared under the direction of the Iron and Steel Standards Committee.

It is identical with ISO 4139-1979 'Ferrosilicon — Determination of aluminium content — Flame atomic absorption spectrometric method' published by the International Organization for Standardization (ISO).

Terminology and conventions. The text of the international standard has been approved as suitable for publication as a British Standard without deviation. Some terminology and certain conventions are not identical with those used in British Standards; attention is drawn especially to the following.

The comma has been used as a decimal marker. In British Standards it is current practice to use a full point on the baseline as the decimal marker.

Wherever the words 'International Standard' appear, referring to this standard, they should be read as 'British Standard'.

Additional information. No standard is quoted to refer to for sampling. Appropriate procedures will be incorporated in BS 6200 : Part 2 'Methods of sampling and sample preparation', which will be published in due course.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

British Standards Institution

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.