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

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FOREWORD

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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.

3.11 Aluminium, standard solution corresponding to 1 g of Al per litre.

Weigh, to the nearest 0,001 g, 1 g of aluminium, purity 99,99 %. Transfer to a 600 ml beaker and dissolve in 30 ml of the hydrochloric acid solution (3.5). Transfer the solution quantitatively into a 1 000 ml volumetric flask. Make up to the mark with water and mix.

4 APPARATUS

Usual laboratory equipment, and in particular :

4.1 Platinum crucibles, capacity approximately 40 ml.

4.2 Polytetrafluorethylene (PTFE) beakers or platinum dishes, of suitable capacity.

4.3 Atomic absorption spectrophotometer, equipped with a burner fed by dinitrogen monoxide and acetylene.

4.4 Aluminium hollow-cathode lamp.

5 SAMPLE

Sampling and preparation of samples of ferro-alloys will form the subject of a subsequent International Standard.

6 PROCEDURE

WARNING – Perchloric acid fumes may produce explosions in the presence of ammonia or organic substances in general.

6.1 Test portion

Take a test portion of 1 ± 0,001 g.

6.2 Blank test

Carry out a blank test in parallel with the determination, following the same procedure and using the same quantities of all the reagents, but use in all instances 50 ml of the iron solution No. 1 (3.8).

6.3 Determination

6.3.1 *Preparation of the test solution*

Transfer the test portion (6.1) to a 150 ml PTFE beaker or a platinum dish (4.2) of capacity approximately 100 ml and add 10 ml of the nitric acid solution (3.2).

Then add, carefully and gradually, shaking after each addition to enable the reaction to develop in a gradual manner, 10 ml of the hydrofluoric acid solution (3.3).

Let the reaction continue at room temperature until effervescence ceases.

If the silicon content of the sample is equal to or greater than 65 %, add to the solution so obtained 30 ml of the iron solution No. 1 (3.8).

In all cases add 5 ml of the perchloric acid solution (3.4). Place the beaker on a hot-plate, at a temperature not exceeding 350 °C, and heat until copious white perchloric fumes are emitted.

Cool.

Add to the beaker 30 ml of the hydrochloric acid solution (3.6) and heat until all soluble salts have dissolved. Filter the residue, using a fine texture filter, and collect the filtrate in a 250 ml beaker.

Wash the residue and the filter with about 100 ml of warm water in order to remove the last trace of perchloric acid.

Place the filter in a platinum crucible (4.1).

Incinerate in a muffle furnace, set first of all at a low temperature, then calcine at 1 000 °C for about 15 min.

Allow to cool in a desiccator.

Add to the crucible 1 g of the sodium carbonate (3.1) and 0,500 g of the boric acid (3.7). Heat on a hot-plate, set at 250 °C, for 15 min. Place the crucible in a muffle furnace set at 1 000 °C and leave for 15 min.

Allow the crucible to cool, then place it in the beaker containing the filtrate from the residue of the acid attack. Add 15 ml of the hydrochloric acid solution (3.5) and heat gently until the melt residue completely dissolves.

Withdraw the crucible from the beaker, rinse it carefully into the beaker, and concentrate the solution by moderate heating until a volume of about 60 ml is attained.

Cool.

Transfer the contents of the beaker quantitatively into a 100 ml volumetric flask. Make up to the mark with water and mix.

The preparation of solution A is thus complete.

6.3.1.1 ALUMINIUM CONTENTS OF LESS THAN 0,50 % (*m/m*)

Carry out the measurement specified in 6.3.2 using solution A.

6.3.1.2 ALUMINIUM CONTENTS BETWEEN 0,50 AND 1,25 % (*m/m*)

Take 20 ml of solution A and transfer to a 50 ml volumetric flask. Add, from a burette, 24 ml of the iron solution No. 2 (3.9).

Make up to the mark with water and mix. The preparation of solution B is thus complete.

Carry out the measurement specified in 6.3.2 using solution B.

6.3.1.3 ALUMINIUM CONTENTS BETWEEN 1,25 AND 5 % (*m/m*)

Take 5 ml of solution A and transfer to a 50 ml volumetric flask. Add, from a burette, 36 ml of the iron solution No. 2 (3.9).

Make up to the mark with water and mix. The preparation of solution C is thus complete.

Carry out the measurement specified in 6.3.2 using solution C.

NOTE — Depending upon the sensitivity of the equipment used, greater dilutions can be accommodated.

6.3.2 *Spectrometric measurement*

Switch on the flame atomic absorption spectrometer (4.3) and the aluminium hollow-cathode lamp (4.4) a sufficient time in advance to ensure their stability.

Adjust the wavelength to 309,3 nm. Adjust the pressure of the dinitrogen monoxide and the acetylene according to the burner characteristics. Obtain the optimum signal using an aluminium solution of known concentration, which may vary according to the apparatus, to obtain maximum sensitivity and stability.

Measure the absorbance of the test solution (6.3.1.1 or 6.3.1.2 or 6.3.1.3) after having adjusted the instrument zero reading with respect to the solution for the blank test on the reagents for the calibration curve (6.4).

Prepare a calibration curve (see 6.4) for each series of measurements.

NOTE — Aluminium is partly ionized in the dinitrogen monoxide-acetylene flame. Iron acts as an ionization buffer and thus enhances the signal from aluminium. Trials have shown that under the conditions used, the enhancement is constant for a range of 3 to 10 g of Fe per litre.

6.4 Establishment of the calibration curve

To each of a series of eight 100 ml volumetric flasks transfer :

– 50 ml of the iron solution No. 2 (3.9), and

– 20 ml of the solution for restoration of the operating conditions of the analysis (3.10).

Then add respectively the volumes of the standard aluminium solution (3.11) given in the table below :

Make up to volume with water and mix.

Carry out the absorbance measurements on each reference solution under the conditions specified in 6.3.2.

Plot the calibration curve, indicating as abscissae the percentages by mass of aluminium in the ferrosilicon, and as ordinates the corresponding absorbance values.

7 EXPRESSION OF RESULTS

Calculate, using the calibration curve, the percentage by mass of aluminium in the ferrosilicon corresponding to the absorbance measured.

8 TEST REPORT

The test report shall include the following particulars :

a) identification of the sample;

b) the reference of the method used;

c) the results and the method of expression used;

d) any unusual features noted during the determination;

e) any operation not included in this International Standard or regarded as optional.

Standard aluminium solution (3.11)	**Corresponding mass of aluminium**	**Corresponding percentage by mass of aluminium in the test portion**		
ml	mg	**Solution A** 1 g/100 ml	**Solution B** 1 g/250 ml	**Solution C** 1 g/1 000 ml
0*	0	0	0	0
0,50	0,5	0,05	0,125	0,50
1,00	1,0	0,10	0,25	1,00
2,00	2,0	0,20	0,50	2,00
2,50	2,5	0,25	0,625	2,50
3,00	3,0	0,30	0,75	3,00
4,00	4,0	0,40	1,00	4,00
5,00	5,0	0,50	1,25	5,00

* Blank test on the reagents for the calibration curve.