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Iron ores — Determination of nickel and/or chromium contents — Flame atomic absorption spectrometric method

*Minerais de fer — Dosage du nickel et/ou du chrome — Méthode par
spectrométrie d'absorption atomique dans la flamme*



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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 9685 was prepared by Technical Committee ISO/TC 102, *Iron ores*, Sub-Committee SC 2, *Chemical analysis*.

Annex A forms an integral part of this International Standard. Annexes B and C are for information only.

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Iron ores — Determination of nickel and/or chromium contents — Flame atomic absorption spectrometric method

1 Scope

This International Standard specifies a flame atomic absorption spectrometric method for the determination of nickel and/or chromium contents of iron ores.

This method is applicable to nickel and/or chromium contents between 0,003 % (m/m) and 0,1 % (m/m)¹⁾ in natural iron ores, iron ore concentrates and agglomerates, including sinter products.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 648:1977, *Laboratory glassware — One-mark pipettes*.

ISO 1042:1983, *Laboratory glassware — One-mark volumetric flasks*.

ISO 3081:1986, *Iron ores — Increment sampling — Manual method*.

ISO 3082:1987, *Iron ores — Increment sampling and sample preparation — Mechanical method*.

ISO 3083:1986, *Iron ores — Preparation of samples — Manual method*.

ISO 7764:1985, *Iron ores — Preparation of predried test samples for chemical analysis*.

3 Principle

Decomposition of the test portion by treatment with hydrochloric and nitric acids.

Removal of the major portion of iron in the filtrate by extraction with 4-methylpentan-2-one.

Ignition of the insoluble residue, removal of silicon dioxide by evaporation with hydrofluoric and sulfuric acids. Fusion of the residue with a mixture of sodium carbonate and sodium tetraborate, followed by dissolution with hydrochloric acid. Combination with the main solution.

Aspiration into the flame of an atomic absorption spectrometer using an air-acetylene burner for nickel and a dinitrogen oxide-acetylene burner for chromium.

Comparison of the absorbance values obtained with those obtained from the calibration solutions.

4 Reagents

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

4.1 Sodium carbonate (Na_2CO_3), anhydrous powder.

4.2 Sodium tetraborate ($\text{Na}_2\text{B}_4\text{O}_7$), anhydrous powder.

4.3 Lithium tetraborate ($\text{Li}_2\text{B}_4\text{O}_7$), anhydrous powder.

4.4 Hydrochloric acid, ρ 1,16 g/ml to 1,19 g/ml.

1) This method has not been tested on ores containing more than 0,056 % (m/m) of chromium. (See annex B.)

4.5 Hydrochloric acid, ρ 1,16 g/ml to 1,19 g/ml, diluted 2 + 1.

4.6 Hydrochloric acid, ρ 1,16 g/ml to 1,19 g/ml, diluted 1 + 1.

4.7 Hydrochloric acid, ρ 1,16 g/ml to 1,19 g/ml, diluted 2 + 100.

4.8 Nitric acid, ρ 1,4 g/ml.

4.9 Nitric acid, ρ 1,4 g/ml, diluted 1 + 1.

4.10 Hydrofluoric acid, ρ 1,13 g/ml, 40 % (m/m), or ρ 1,19 g/ml, 48 % (m/m).

4.11 Sulfuric acid, ρ 1,84 g/ml.

4.12 Sulfuric acid, ρ 1,84 g/ml, diluted 1 + 1.

4.13 4-Methylpentan-2-one, (methyl isobutyl ketone, MIBK).

4.14 Nickel standard solution A, 100 μg Ni/ml.

Dissolve 0,1000 g of nickel metal [purity > 99,9 % (m/m)] (see note 1, 4.18) in 30 ml of nitric acid (4.9). After cooling, transfer to a 1000 ml one-mark volumetric flask quantitatively, dilute to volume with water and mix.

4.15 Nickel standard solution B, 10 μg Ni/ml.

Transfer 100,0 ml of nickel standard solution A (4.14) to a 1000 ml one-mark volumetric flask. Dilute to volume with water and mix.

4.16 Chromium standard solution A, 100 μg Cr/ml.

Dissolve 0,1000 g of chromium metal [purity > 99,9 % (m/m)] (see note 1, 4.18) in 20 ml of hydrochloric acid (4.6). After cooling, transfer to a 1000 ml one-mark volumetric flask quantitatively, dilute to volume with water and mix.

4.17 Chromium standard solution B, 10 μg Cr/ml.

Transfer 100,0 ml of chromium standard solution A (4.16) to a 1000 ml one-mark volumetric flask. Dilute to volume with water and mix.

4.18 Nickel and chromium mixture solution.

Introduce into a 200 ml beaker, 30 ml of hydrochloric acid (4.6), 0,2 ml of sulfuric acid (4.12) and a mixture of 0,8 g of sodium carbonate (4.1) and 0,4 g of sodium tetraborate (4.2). Heat to remove carbon di-

oxide and cool. Using pipettes or burettes, add 10,0 ml of nickel standard solution A (4.14) and 10,0 ml of chromium standard solution A (4.16). Transfer to a 100 ml one-mark volumetric flask, dilute to the mark with water and mix.

NOTE 1 The purity of the metals stated on the certificates does not generally take into account the presence of adsorbed gases like oxygen, carbon monoxide, etc.

5 Apparatus

Ordinary laboratory apparatus, including one-mark pipettes and one-mark volumetric flasks complying with the specifications of ISO 648 and ISO 1042, and

5.1 Platinum crucible, of minimum capacity 25 ml.

5.2 Muffle furnace.

5.3 Atomic absorption spectrometer, equipped with an air-acetylene and a dinitrogen oxide-acetylene burner.

The atomic absorption spectrometer used in this method will be satisfactory if it meets the following criteria:

- Minimum sensitivity — the absorbance of the most concentrated calibration solution (see 7.5.4) is at least 0,3.
- Graph linearity — the slope of the calibration graph covering the top 20 % of the concentration range (expressed as a change in absorbance) is not less than 0,7 of the value of the slope for the bottom 20 % of the concentration range determined in the same way.
- Minimum stability — the standard deviation of the absorbance of the most concentrated calibration solution and that of the zero calibration solution, each being calculated from a sufficient number of repetitive measurements, are less than 1,5 % and 0,5 % respectively of the mean value of the absorbance of the most concentrated solution.

NOTES

2 The use of a strip-chart recorder and/or digital read-out device is recommended to evaluate criteria a), b) and c) and for all subsequent measurements.

3 Instrument parameters will vary with each instrument. The following parameters were successfully used in several laboratories and they can be used as guidelines. Solutions were aspirated into an air-acetylene or a dinitrogen oxide-acetylene flame of a premix burner:

	Ni	Cr
Hollow-cathode lamp, mA	10	10
Wavelength, nm	232,0	357,9
Air flow rate, l/min	10	—
Acetylene flow rate, l/min	2,5	5
Dinitrogen oxide flow rate, l/min	—	≈ 10

In systems where the values shown above for gas flow rates do not apply, the ratio of the gas flow rates may still be a useful guideline.

6 Sampling and samples

6.1 Laboratory sample

For analysis, use a laboratory sample of minus 100 µm particle size which has been taken in accordance with ISO 3081 or ISO 3082 and prepared in accordance with ISO 3082 or ISO 3083. In the case of ores with significant contents of combined water or oxidizable compounds, use a particle size of minus 160 µm.

NOTES

4 A guideline on significant contents of combined water and oxidizable compounds is incorporated in ISO 7764.

5 Ensure that the sample has not been pulverized in a nickel/chromium pot.

6.2 Preparation of predried test samples

Thoroughly mix the laboratory sample and, taking multiple increments, extract a test sample in such a way that it is representative of the whole contents of the container.

Dry the test sample at $105\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ as specified in ISO 7764. (This is the predried test sample.)

7 Procedure

7.1 Number of determinations

Carry out the analysis at least in duplicate in accordance with annex A, independently, on one predried test sample.

NOTE 6 The expression "independently" means that the second and any subsequent result is not affected by the previous result(s). For this particular analytical method, this condition implies that the repetition of the procedure shall be carried out either by the same operator at a different time or by a different operator including, in both cases, appropriate recalibration.

7.2 Safety precautions

WARNING — Follow the manufacturer's instructions for igniting and extinguishing the air-acetylene flame

or the dinitrogen oxide-acetylene flame to avoid possible explosion hazards. Wear tinted safety glasses whenever the burner is in operation.

7.3 Test portion

Taking several increments, weigh, to the nearest 0,0002 g, approximately 1 g of the predried test sample obtained in accordance with 6.2.

NOTE 7 The test portion should be taken and weighed quickly, in order to avoid reabsorption of moisture.

7.4 Blank test and check test

In each run, one blank test and one analysis of a certified reference material of the same type of ore shall be carried out in parallel with the analysis of the ore sample(s) under the same conditions. A predried test sample of the certified reference material shall be prepared as specified in 6.2.

NOTE 8 The certified reference material should be of the same type as the sample to be analysed and the properties of the two materials should be sufficiently similar to ensure that in both cases no significant changes in the analytical procedure will become necessary.

When the analysis is carried out on several samples at the same time, the blank value may be represented by one test, provided that the procedure is the same and the reagents used are from the same reagent bottles.

When the analysis is carried out on several samples of the same type of ore at the same time, the analytical value of one certified reference material may be used.

7.5 Determination

7.5.1 Decomposition of the test portion

Transfer the test portion (7.3) to a 250 ml tall-form beaker. Moisten with a few millilitres of water. Add 25 ml of hydrochloric acid (4.4), cover with a watch-glass, and heat for about 1 h on a hotplate adjusted to give a temperature of $100\text{ }^{\circ}\text{C}$ in a test beaker containing a similar volume and depth of sulfuric acid (4.11).

NOTES

9 When the amount of insoluble residue is high, continue heating on a higher temperature zone of the hotplate without boiling the solution.

10 After heating, the volume of the solution should have decreased to about 10 ml.

Add 5 ml of nitric acid (4.8) and 0,2 ml of sulfuric acid (4.12), digest for about 15 min and evaporate the solution almost to dryness.