
**Iron ores — Determination of nickel —
Flame atomic absorption spectrometric
method**

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

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 15633 was prepared by Technical Committee ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 2, *Chemical analysis*.

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Introduction

The objective of a proposed revision of ISO 9685:1991 was to extend the lower limit for a flame atomic absorption spectrometric method determination of both chromium and nickel in iron ores down to 0,001 %. However, due to bias, the method for nickel could not be approved for referee purposes.

The 22nd meeting of ISO/TC 102/SC 2 decided to progress the document as a non-referee method.

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Iron ores — Determination of nickel — Flame atomic absorption spectrometric method

WARNING — This International Standard may involve hazardous materials, operations and equipment. This International Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this International Standard to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies a flame atomic absorption spectrometric method for the determination of the nickel mass fraction of iron ores.

This method is applicable to mass fractions of nickel between 0,001 % and 0,1 % in natural iron ores, iron ore concentrates and agglomerates, including sinter products.

This method is not appropriate for referee purposes.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 648, *Laboratory glassware — One-mark pipettes*

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

ISO 3082, *Iron ores — Sampling and sample preparation procedures*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

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

3 Principle

The test portion of iron ore is decomposed by treatment with hydrochloric and nitric acids.

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

The insoluble residue is ignited and silicon dioxide is removed by evaporation with hydrofluoric and sulfuric acids. The residue is fused with a mixture of sodium carbonate and sodium tetraborate, and then dissolved with hydrochloric acid and combined with the main solution.

The solution is aspirated into the flame of an atomic absorption spectrometer using an air-acetylene burner.

The absorbance values obtained are compared with those obtained from the calibration solutions.

4 Reagents

During the analysis, use only reagents of recognized analytical grade, and only water that conforms to grade 2 of ISO 3696.

- 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**, $\rho = 1,16$ g/ml to 1,19 g/ml.
- 4.5 **Hydrochloric acid**, $\rho = 1,16$ g/ml to 1,19 g/ml, diluted 2 + 1.
- 4.6 **Hydrochloric acid**, $\rho = 1,16$ g/ml to 1,19 g/ml, diluted 1 + 1.
- 4.7 **Hydrochloric acid**, $\rho = 1,16$ g/ml to 1,19 g/ml, diluted 2 + 100.
- 4.8 **Nitric acid**, $\rho = 1,4$ g/ml.
- 4.9 **Nitric acid**, $\rho = 1,4$ g/ml, diluted 1 + 1.
- 4.10 **Hydrofluoric acid**, $\rho = 1,13$ g/ml, 40 % (mass fraction), or $\rho = 1,19$ g/ml, 48 % (mass fraction).
- 4.11 **Sulfuric acid**, $\rho = 1,84$ g/ml.
- 4.12 **Sulfuric acid**, $\rho = 1,84$ 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 0,1 000 g of nickel metal [purity > 99,9 % (mass fraction)] (see Note to 4.15) in 30 ml of nitric acid (4.9). After cooling, transfer quantitatively to a 1 000 ml one-mark volumetric flask, 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 1 000 ml one-mark volumetric flask. Dilute to volume with water and mix.

NOTE The purity of the metals stated on the certificates does not generally take into account the presence of absorbed gases such as 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 the following.

- 5.1 **Platinum crucible**, of minimum capacity 25 ml.
- 5.2 **Muffle furnace**.
- 5.3 **Atomic absorption spectrometer**, equipped with an air-acetylene burner.

WARNING — Follow the manufacturer's instructions for igniting and extinguishing the air-acetylene flame to avoid possible explosion hazards. Wear tinted safety glasses whenever the burner is in operation.

The atomic absorption spectrometer used in this method shall meet the following criteria.

- a) *Minimum sensitivity*: the absorbance of the most concentrated calibration solution (see 7.4.4) is at least 0,3.
- b) *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.
- c) *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.

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

NOTE Instrument parameters vary with each instrument. The following parameters were successfully used in several laboratories and they can be used as guidelines. An air-acetylene flame was used.

Hollow-cathode lamp, mA 10

Wavelength, nm 232,0

Air flow rate, l/min 10

Acetylene flow rate, l/min 2,5

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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 < 100 µm particle size which has been taken and prepared in accordance with ISO 3082. In the case of ores containing significant contents of combined water or oxidizable compounds, use a particle size of < 160 µm.

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

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 entire contents of the container. Dry the test sample at 105 °C ± 2 °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 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 be carried out either by the same operator at a different time or by a different operator including, in either case, appropriate recalibration.

7.2 Test portion

Taking several increments, weigh, to the nearest 0,000 2 g, approximately 1 g of the predried test sample obtained in accordance with 6.2.

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

7.3 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.

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 either case no significant changes in the analytical procedure become necessary. Where a certified reference material is not available, a reference material may be used (see 8.2.4).

Where 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.

Where 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.

7.4 Determination

7.4.1 Decomposition of the test portion

Transfer the test portion (7.2) 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 °C in a test beaker containing a similar volume and depth of sulfuric acid (4.11).

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

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.

If the test sample contains significant barium content, the addition of sulfuric acid (4.12) should be omitted.

Be sure to cover with a watch-glass, in order to prevent sublimation of chlorides. During evaporation, shift the glass slightly.

Add 20 ml of hydrochloric acid (4.6), and heat to dissolve the salts. Cool, wash the watch-glass and the walls of the beaker, and filter the solution through a close-texture filter paper containing filter pulp (0,3 g to 0,4 g dry mass) into a 200 ml beaker. Carefully remove all adhering particles with a rubber-tipped glass rod or piece of moistened filter paper, and transfer to the filter, washing the paper with warm hydrochloric acid (4.7) until it is visibly free from iron, and again wash the paper with three or four portions of warm water. Reserve the filtrate and washings in the 200 ml beaker as the main solution. Transfer the filter paper and residue to a platinum crucible (5.1).

7.4.2 Removal of iron

Heat the main solution and evaporate almost to dryness. Dissolve the salts in 15 ml of hydrochloric acid (4.5) and transfer to a 200 ml separating funnel. Rinse the beaker with 20 ml of hydrochloric acid (4.5) and transfer the washings to the funnel.

Add 50 ml of 4-methylpentan-2-one (4.13) to the funnel and shake it for 1 min. Allow the two layers to separate and drain the lower aqueous layer into the original 200 ml beaker. Wash the organic layer by adding 10 ml of hydrochloric acid (4.5) to the funnel and shaking it for 30 s. Allow the two layers to separate and drain the lower aqueous layer into the 200 ml beaker to combine with the first aqueous layer. Discard the organic layer.

Heat the solution gently and expel almost all of the 4-methylpentan-2-one in the solution. Then add 5 ml of nitric acid (4.8) and evaporate to dryness. Dissolve the salts in 20 ml of hydrochloric acid (4.6).

7.4.3 Treatment of the residue

Dry and burn off the filter paper in the platinum crucible (5.1) at a low temperature (500 °C to 800 °C), and ignite the residue. Cool, moisten with 1 to 2 drops of water and add 3 drops of sulfuric acid (4.11) and 5 ml of hydrofluoric acid (4.10). Evaporate slowly to expel silicon dioxide and continue heating to remove the sulfuric acid. Ignite at 800 °C for several minutes and then cool. Add 1,2 g of a mixture of 0,8 g of sodium carbonate (4.1) and 0,4 g of sodium tetraborate (4.2) to the residue and mix. Heat gently for several minutes, then heat at 1 000 °C for 15 min in a muffle furnace (5.2) or over a pressurized air burner for a time sufficient to produce a clear melt.

Lithium tetraborate (4.3) may be used instead of sodium tetraborate (4.2). In this case, sodium tetraborate should be replaced with lithium tetraborate in the calibration solutions (7.4.4) and the flame-setting solutions (7.4.5).

Allow the crucible to cool, add 10 ml of hydrochloric acid (4.6) and heat gently to dissolve the melt. Combine this solution with the main solution from 7.4.2, heat to remove carbon dioxide and cool.

For samples containing a mass fraction of more than or equal to 0,01 % nickel, transfer to a 100 ml one-mark volumetric flask quantitatively, dilute to volume with water and mix. (This is the test solution.)

For samples containing a mass fraction of less than 0,01 % nickel, transfer to a 50 ml one-mark volumetric flask quantitatively, dilute to volume with water and mix. (This is the test solution.)

7.4.4 Preparation of the calibration solutions

Introduce into a series of six 200 ml beakers, 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 dioxide and cool.

Using pipettes or burettes, add 0 ml, 1,0 ml, 3,0 ml, 5,0 ml, 7,0 ml and 10,0 ml of nickel standard solution A (4.14) for samples containing 0,01 % to 0,1 % nickel, or nickel standard solution B (4.15) for samples containing 0,001 % to 0,01 % nickel.

Transfer to six 100 ml one-mark volumetric flasks for samples containing 0,01 % to 0,1 % nickel or 50 ml one-mark volumetric flasks for samples containing 0,001 % to 0,01 % nickel, respectively, dilute to volume with water and mix. These calibration solutions cover the nickel concentration ranges 0 µg/ml to 10 µg/ml in the case of standard solution A, or 0 µg/ml to 2 µg/ml in the case of standard solution B.