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

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

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

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

This third edition cancels and replaces the second edition (ISO 13313:2006), which constitutes a minor revision with the following changes:

- a new sentence has been included in [7.2](#) to make reference to ISO 2596;
- [Formula \(8\)](#) has been modified and the relevant descriptions to harmonize this subclause across all standards for which ISO/TC 102/SC 2 is responsible;
- footnotes in [5.4](#), [5.5](#), [8.4.2](#) and [9.2.1](#) have been moved to the appropriate place.

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

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

1 Scope

This document specifies a flame atomic absorption spectrometric method for the determination of the mass fraction of sodium in iron ores.

This method is applicable to mass fractions of sodium between 0,002 5 % and 0,50 % in natural iron ores, iron ore concentrates and agglomerates, including sinter products.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 — Single-volume pipettes*

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

ISO 2596, *Iron ores — Determination of hygroscopic moisture in analytical samples — Gravimetric, Karl Fischer and mass-loss methods*

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*

ISO 11323, *Iron ore and direct reduced iron — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 11323 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <http://www.iso.org/obp>

4 Principle

The test portion is decomposed by treatment with hydrochloric acid and hydrofluoric acid, followed by evaporation to dryness. The residue is wetted and the evaporation repeated with a new portion of hydrochloric acid. The residue is dissolved with hydrochloric acid and appropriately diluted. The solution is aspirated into the air/acetylene flame of the atomic absorption apparatus.

The absorbance value obtained for sodium is measured and compared with those obtained from calibration solutions.

5 Reagents

During the analysis, use only reagents of recognized analytical grade, and only water that complies with Grade 2 of ISO 3696.

Reagents are to be selected or purified for the lowest possible blank value.

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

5.2 Hydrofluoric acid, ρ 1,13 g/ml, a mass fraction of 40 %, or ρ 1,19 g/ml, a mass fraction of 48 %.

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

5.4 Background solution

Dissolve 43 g of high-purity iron oxide powder in 500 ml of hydrochloric acid (5.1). Allow to cool and dilute with water to 1 000 ml.

Instead of iron oxide, the use of metallic iron with a suitable oxidant is permitted. The alkali content of the oxidant shall be low.

5.5 Sodium, standard solution, 10 $\mu\text{g Na/ml}$.

Pulverize about 4 g of high-purity sodium chloride in an agate mortar, dry in an oven at 105 °C to 110 °C for 2 h and allow to cool to room temperature in a desiccator. Dissolve 2,542 g of it in water, dilute with water to 1 000 ml in a volumetric flask and mix.

Transfer 10,0 ml of this solution to a 1 000 ml volumetric flask, dilute with water to volume and mix.

Glass equipment may be used.

Store this standard solution in a plastic bottle.

1 ml of this standard solution contains 10 μg of sodium.

6 Apparatus

Ordinary laboratory equipment, including one-mark pipettes and one-mark volumetric flasks complying with the specifications of ISO 648 and ISO 1042, respectively, and the following.

6.1 Polytetrafluoroethylene (PTFE) beakers, of capacity 100 ml, provided with PTFE covers.

6.2 PTFE-coated magnetic stirring bars.

6.3 PTFE digestion bomb.

6.4 Plastic pipettes.

6.5 Plastic volumetric flasks and storage bottles.

6.6 Magnetic stirring hotplates.

NOTE Platinum vessels can be used instead of PTFE beakers.

Except where stated, glass equipment should be avoided, as it could contaminate the solutions.

The caps of the plastic bottles should be of a type that does not contain a separate wad insert. Such inserts usually contain a sodium compound that will contaminate the solution.

To obtain reliable values, the equipment should be cleaned and checked as follows.

- a) Rinse all volumetric ware, including the pipettes used for preparing the calibration solutions, with dilute hydrochloric acid (5.3) before use. Check calibration regularly or as needed.
- b) Clean PTFE vessels and stirring bars by stirring with 50 ml of dilute hydrochloric acid (5.3) and heating for 15 min. Reject the rinsings and conduct a blank test in each vessel in turn, exactly as specified in 8.3. If any absorbance value is above the limit specified in 8.3, the cleaning procedure should be repeated or acid reagents of a higher purity should be used. At no stage should the stirring bars be handled with the fingers.
- c) Platinum vessels, exclusively used for sodium analysis according to this document, can be cleaned by the same method as the PTFE vessels [see b)]. Otherwise, they should be pre-cleaned by fusion with lithium tetraborate or lithium borate, until the absorbance readings fall to those for the lithium salt alone.
- d) Rinse storage bottles with dilute hydrochloric acid (5.3) before use.

6.7 Atomic absorption spectrometer.

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 shall meet the following criteria.

- a) Minimum sensitivity: the absorbance of the most concentrated calibration solution (see 8.4.4) shall be at least 0,25.
- b) Graph linearity: the slope of the calibration graph covering the top 20 % of the concentration range (expressed as a change in absorbance) shall not be 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, shall be less than 1,5 % and 0,5 %, respectively, of the mean value of the absorbance of the most concentrated calibration 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 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 flame of a premix burner:

- hollow cathode lamp, 10 mA;
- wavelength, 589,0 nm;
- air flow-rate, 10 l/min;
- acetylene flow-rate, 2 l/min.

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