INTERNATIONAL STANDARD

ISO 13311

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Iron ores — Determination of lead content — Flame atomic absorption spectrometric method

Minerais de fer — Dosage du plomb — 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.

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 13311 was prepared by Technical Committee ISO/TC 102, *Iron ores*, Subcommittee SC 2, *Chemical analysis*.

Together with ISO 13310, it cancels and replaces ISO 8753:1987, of which it constitutes a technical revision.

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

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WARNING — This International Standard may involve hazardous materials, operations and equipment. This International Standard does not purport to address all of the safety issues associated with its use. It is the responsibility of the user 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 lead content of iron ores.

This method is applicable to lead contents between 0,001 % (m/m) and 0,5 % (m/m) 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 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

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

3 Principle

Decomposition of the test portion and removal of silica by treatment with hydrochloric acid and hydrofluoric acid. Oxidation with nitric acid.

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Evaporation to dryness, followed by dilution and filtration. Ignition of the residue. Fusion with sodium carbonate. Dissolution of the cooled melt with hydrochloric acid. Reservation of the solution.

Extraction of iron in the filtrate with 4-methyl-2-pentanone. Recovery of lead extracted. Decomposition of 4-methyl-2-pentanone with nitric acid. Evaporation to dryness and dissolution of salts with the reserved solution and hydrochloric acid.

Aspiration of the solution into the flame of an atomic absorption spectrometer using an air-acetylene burner.

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

4 Reagents

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

4.1 Sodium carbonate (Na_2CO_3), anhydrous.

4.2 4-methyl-2-pentanone (MIBK), high purity.

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

4.4 Hydrochloric acid, ρ 1,16 g/ml to 1,19 g/ml, diluted 10 + 6.

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

4.6 Hydrochloric acid, ρ 1,16 g/ml to 1,19 g/ml, diluted 2 + 98.

4.7 Nitric acid, ρ 1,4 g/ml.

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

4.9 Lead standard solutions

4.9.1 Lead stock standard solution

Dissolve 1,000 g of lead metal [of purity > 99,5 % (m/m)] in 40 ml of nitric acid (ρ 1,4 g/ml, diluted 1 + 1). Cool and dilute to 1 000 ml in a one-mark volumetric flask and mix.

1 ml of the stock solution contains 1 000 µg of lead.

4.9.2 Standard solution A

Transfer 10,0 ml of lead stock standard solution (4.9.1) to a 100 ml volumetric flask. Dilute to volume and mix.

1 ml of this standard solution contains 100 µg of lead.

4.9.3 Standard solution B

Transfer 10,0 ml of standard solution A (4.9.2) to a 100 ml volumetric flask. Dilute to volume and mix.

1 ml of this standard solution contains 100 µg of lead.

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5 Apparatus

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

5.1 Polytetrafluoroethylene (PTFE) beaker, of capacity 250 ml, with a PTFE cover.

5.2 Atomic absorption spectrometer, equipped with an air-acetylene burner.

The atomic absorption spectrometer shall meet the following criteria.

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.

a) *Minimum sensitivity* — the absorbance of the most concentrated calibration solution (see 7.4.4) 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.

NOTES

1 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.

2 Instrument parameters will vary with each instrument. The following parameters were successfully used in several laboratories and they can be used as guidelines.

— Hollow cathode lamp, mA	6
— Wavelength, nm	283,3
— Air flow rate, l/min	14
— Acetylene flow rate, l/min	3

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.

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 having significant contents of combined water or oxidizable compounds, use a particle size of minus 160 µm.

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

6.2 Preparation of predried test samples

Thoroughly mix the laboratory sample and, taking multiple increments, extract a test sample in such a manner that it is representative of the whole contents of the container. Dry the test samples at 105 °C ± 2 °C as specified in ISO 7764. (This is the predried test sample.)

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