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Iron ores — Determination of lead and/or zinc content — Flame atomic absorption spectrometric method

*Minerais de fer — Dosage du plomb et/ou du zinc — 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 8753 was prepared by Technical Committee ISO/TC 102, *Iron ores*.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

Iron ores — Determination of lead and/or zinc content — Flame atomic absorption spectrometric method

1 Scope and field of application

This International Standard specifies a flame atomic absorption spectrometric method for the determination of the lead and/or zinc content of iron ores.

This method is applicable to a concentration range of 0,001 to 0,5 % (m/m) of lead and 0,001 to 0,5 % (m/m) of zinc in natural iron ores, and iron ore concentrates and agglomerates including sinter products.

2 References

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

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

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

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

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

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

3 Principle

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

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 MIBK. Recovery of lead and zinc extracted. Decomposition of MIBK 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 or for zinc with those obtained from the calibration solutions.

4 Reagents

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

4.1 Sodium carbonate, anhydrous.

4.2 Methyl isobutyl ketone (MIBK), high purity.

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

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

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

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

4.7 Nitric acid, ρ 1,4 g/ml.

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

4.9 Lead standard solutions.

4.9.1 Stock lead standard solution.

Dissolve 1,000 g of high purity lead metal (free from iron) in 40 ml of nitric acid (ρ 1,4 g/ml, diluted 1 + 1). Cool and dilute to 1 000 ml in a volumetric flask and mix.

1 ml of the stock solution contains 1 000 μ g of lead.

4.9.2 Standard solution A.

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

1 ml of this standard solution contains 100 μ g 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 the mark and mix.

1 ml of this standard solution contains 10 μ g of lead.

4.10 Zinc standard solutions.

4.10.1 Stock zinc standard solution.

Dissolve 1,000 g of high purity zinc metal (free from iron) in 40 ml of hydrochloric acid (4.5). Cool and dilute to 1 000 ml in a volumetric flask and mix.

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

4.10.2 Standard solution C.

Transfer 10,0 ml of stock zinc standard solution (4.10.1) to a 1 000 ml volumetric flask. Dilute to the mark and mix.

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

5 Apparatus

NOTE — Unless otherwise indicated, any pipettes and volumetric flasks shall be one-mark pipettes and volumetric flasks complying with the specifications of ISO 648 and ISO 1042.

Ordinary laboratory apparatus, 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 used will be satisfactory if it meets the following criteria :

- a) *Minimum sensitivity* — the absorbance of the calibration solution of highest concentration (7.4.4) is at least 0,25.
- b) *Curve linearity* — the slope of the calibration graph covering the top 20 % concentration range (expressed as a change in absorbance) is not less than 0,7 of the value of the slope for the bottom 20 % concentration range determined in the same way.
- c) *Minimum stability* — the standard deviation of the absorbance of the calibration solution of highest concentration and the standard deviation of the absorbance 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 calibration solution of highest concentration.

NOTES

- 1 The use of a strip chart recorder and/or digital readout device is recommended to evaluate the criteria and for all subsequent measurements.
- 2 Instrument parameters may vary with each instrument. The following parameters were successfully used in several laboratories and they can be used as guidelines.

	Pb	Zn
— Hollow cathode lamp, mA	6	8
— Wavelength, nm	283,3	213,9
— Air flow rate, l/min	14	14
— Acetylene flow rate, l/min	3	3

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.

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 sample at 105 ± 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 should 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 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 — 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.

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.