
International Standard



3886

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Iron ores — Determination of manganese content — Periodate spectrophotometric method

Minerais de fer — Dosage du manganèse — Méthode spectrophotométrique au periodate

Second edition — 1986-06-15

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 3886 was prepared by Technical Committee ISO/TC 102, *Iron ores*.

This second edition cancels and replaces the first edition (ISO 3886-1977), clauses 6, 7 and 8 and annex A of which have been technically revised.

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 manganese content — Periodate spectrophotometric method

1 Scope and field of application

This International Standard specifies a spectrophotometric method using sodium periodate for the determination of the manganese content of iron ores.

This method is applicable to a concentration range of 0,02 to 8 % (m/m) of manganese 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 by one of the following methods:

- treatment with hydrochloric, nitric and perchloric acids;
- sintering with sodium peroxide, followed by treatment with hydrochloric and perchloric acids.

Filtration and ignition of the residue, followed by treatment with hydrofluoric and sulfuric acids and fusion with sodium carbonate. Dissolution of the cooled melt in the main solution.

Oxidation of manganese in an aliquot to permanganate ion, using sodium periodate in sulfuric acid - phosphoric acid medium.

Spectrophotometric measurement of the absorbance due to the permanganate ion at a wavelength of about 535 nm.

4 Reagents

During the analysis, use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity, except that water free from organic matter (4.1) shall be used for the spectrophotometric measurement.

4.1 Water, free from organic matter.

Add 20 ml of sulfuric acid (4.9) to 1 litre of water, bring to the boil, add several crystals of sodium periodate, continue boiling for 10 min, then cool.

4.2 Sodium peroxide (Na_2O_2), powder.

NOTE — Sodium peroxide should be kept away from humidity and should be not used once it has begun to agglomerate.

4.3 Sodium carbonate (Na_2CO_3), anhydrous.

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

4.5 Hydrochloric acid, ρ 1,16 to 1,19 g/ml, diluted 1 + 9.

4.6 Nitric acid, ρ 1,4 g/ml.

4.7 Perchloric acid, 60 % (m/m) (ρ 1,54 g/ml) or 70 % (m/m) (ρ 1,67 g/ml).

4.8 Hydrogen peroxide, 3 % (V/V).

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

4.10 Sulfuric acid, ρ 1,84 g/ml, diluted 1 + 100.

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

4.12 Sulfuric acid - phosphoric acid mixture.

Pour carefully 100 ml of sulfuric acid (ρ 1,84 g/ml) into about 600 ml of water while stirring, cool, add 150 ml of phosphoric acid (ρ 1,70 g/ml) and dilute to 1 000 ml with water.

4.13 Sodium periodate (NaIO_4) solution, 50 g/l.

4.14 Sodium nitrite (NaNO_2) solution, 10 g/100 ml.

4.15 Manganese, standard solutions.

4.15.1 Stock solution

Dissolve 0,500 g of pure metallic manganese in 20 ml of nitric acid (4.6), add 20 ml of sulfuric acid (4.9) and heat to dense white fumes for about 10 min to expel all oxides of nitrogen. Cool, add about 100 ml of water to dissolve the salt, transfer into a 500 ml volumetric flask, dilute to the mark with water and mix.

1 ml of stock solution contains 1,00 mg of manganese.

4.15.2 Standard solution A

Take 100 ml of stock solution (4.15.1) and dilute to 1 000 ml in a volumetric flask.

1 ml of this standard solution contains 0,100 mg of manganese.

4.15.3 Standard solution B

Take 250 ml of standard solution A (4.15.2) and dilute to 1 000 ml in a volumetric flask.

1 ml of this standard solution contains 0,025 mg of manganese.

5 Apparatus

5.1 Ordinary laboratory equipment.

Any pipettes and volumetric flasks shall be one-mark pipettes and volumetric flasks complying with the specifications of ISO 648 and ISO 1042.

5.2 Spectrophotometer, suitable for the measurement of absorbance at approximately 535 nm.

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

WARNING — Perchloric acid vapour may cause explosions in the presence of ammonia, nitrous fumes or organic matter in general.

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 shall 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, carry out one blank test and one analysis of a certified reference material of the same type of ore in parallel with the analysis of the ore sample(s) under the same conditions. Prepare a predried test sample of the certified reference material 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 would 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.