
**Iron ores — Determination of manganese
content —**

**Part 2:
Periodate spectrophotometric method**

Minerais de fer — Dosage du manganèse

Partie 2: Méthode spectrophotométrique au periodate



Reference number
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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 9682-2 was prepared by Technical Committee ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 2, *Chemical analysis*.

This first edition of ISO 9682-2 cancels and replaces ISO 3886:1986, which has been technically revised. It has been updated to alter the manner in which precision data are presented.

ISO 9682 consists of the following parts, under the general title *Iron ores — Determination of manganese content*:

- *Part 1: Flame atomic absorption spectrometric method*
- *Part 2: Periodate spectrophotometric method*

Iron ores — Determination of manganese content —

Part 2: Periodate spectrophotometric method

WARNING — This part of ISO 9682 may involve hazardous materials, operations and equipment. This part of ISO 9682 does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this part of ISO 9682 to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This part of ISO 9682 specifies a spectrophotometric method using sodium periodate for the determination of the mass fraction of manganese in iron ores.

This method is applicable to a mass-fraction range of 0,02 % to 8 % of manganese in natural iron ores, and iron ore concentrates and agglomerates, including sinter products.

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

A test portion is decomposed by one of the following methods:

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

The mixture is filtered and the residue is ignited, followed by treatment with hydrofluoric and sulfuric acids, and fusion with sodium carbonate. The cooled melt is dissolved in the main solution.

Manganese in an aliquot is oxidized to permanganate ion, using sodium periodate in sulfuric acid/phosphoric acid medium.

The absorbance due to the permanganate ion is measured spectrophotometrically at a wavelength of about 535 nm.

4 Reagents

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

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

4.3 Sodium carbonate (Na_2CO_3), anhydrous.

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

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

4.6 Nitric acid, ρ 1,4 g/ml.

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

4.8 Hydrogen peroxide, 3 % (by volume).

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 % (by mass) (ρ 1,13 g/ml).

4.12 Sulfuric acid/phosphoric acid mixture.

Carefully pour 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.