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

**Part 2:
Periodate spectrophotometric method**

Minerais de fer — Dosage du manganèse
Partie 2: Méthode spectrophotométrique au periodate
iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 9682-2:2006

<https://standards.iteh.ai/catalog/standards/sist/e0bddfd2-1cee-4eeb-977f-32196545545a/iso-9682-2-2006>



PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 9682-2:2006

<https://standards.iteh.ai/catalog/standards/sist/e0bddfd2-1cee-4eeb-977f-32196545545a/iso-9682-2-2006>

© ISO 2006

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

Foreword.....	iv
1 Scope	1
2 Normative references	1
3 Principle	1
4 Reagents	2
5 Apparatus	3
6 Sampling and samples	3
6.1 Laboratory sample	3
6.2 Preparation of predried test samples	3
7 Procedure	3
7.1 Number of determinations	3
7.2 Test portion	4
7.3 Blank test and check test	4
7.4 Calibration	4
7.4.1 Samples containing more than 0,1 % (by mass) of manganese	4
7.4.2 Samples containing less than 0,1 % manganese	5
7.5 Determination	5
7.5.1 Decomposition of the test portion	5
7.5.2 Treatment of the residue	6
7.5.3 Treatment of the test solution	6
7.5.4 Spectrophotometric measurements	6
8 Expression of results	7
8.1 Calculation of mass fraction of manganese	7
8.2 General treatment of results	8
8.2.1 Repeatability and permissible tolerance	8
8.2.2 Determination of analytical result	8
8.2.3 Between-laboratories precision	8
8.2.4 Check for trueness	9
8.2.5 Calculation of final results	9
8.3 Oxide factor	10
9 Test report	10
Annex A (normative) Flowsheet of the procedure for the acceptance of analytical values for test samples	11
Annex B (informative) Derivation of repeatability and permissible tolerance	12
Annex C (informative) Precision data obtained by international analytical trials	13

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

ISO 9682-2:2006

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.

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

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

- 5.1 **Spectrophotometer**, suitable for the measurement of absorbance at approximately 535 nm.
- 5.2 **Nickel crucible**, free of manganese.
- 5.3 **Platinum crucible**, of capacity 25 ml to 30 ml.

6 Sampling and samples

6.1 Laboratory sample

For analysis, use a laboratory sample of minus 100 μm particle size which has been taken and prepared in accordance with ISO 3082. 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 sample at $105 \pm 2^\circ\text{C}$ 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 is carried out either by the same operator at a different time or by a different operator, including, in either case, appropriate recalibration.

7.2 Test portion

Taking several increments, weigh, to the nearest 0,000 2 g, approximately 1 g of the predried test sample obtained in accordance with 6.2.

The test portion should be taken and weighed quickly in order to avoid reabsorption of moisture.

7.3 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.

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. Where a certified reference material is not available, a reference material may be used (see 8.2.4).

Where 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.

Where 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.

7.4 Calibration

iTeh STANDARD PREVIEW

7.4.1 Samples containing more than 0,1% (by mass) of manganese

7.4.1.1 Set of calibration solutions and formation of the absorbing compound

<https://standards.iteh.ai/catalog/standards/sist/e0bddfd2-1cee-4eeb-977f-929231580220>

Into a series of five 300 ml beakers, introduce 2,0 ml; 5,0 ml; 10,0 ml; 15,0 ml and 20,0 ml of manganese standard solution A (4.15.2). Add 30 ml of sulfuric acid/phosphoric acid mixture (4.12), and dilute to about 60 ml with water (4.1).

Add 10 ml of sodium periodate solution (4.13) to each solution, cover each beaker with a watch-glass, heat to boiling, and maintain just below boiling point for 10 min after colour development of the permanganate ion. Cool each solution, transfer to a series of five 100 ml volumetric flasks, make up to the mark with water (4.1) and mix.

7.4.1.2 Calibration compensation solution

Prepare a calibration compensation solution according to 7.4.1.1, but omitting the manganese standard solution A.

7.4.1.3 Spectrophotometric measurements

Transfer a part of each solution (7.4.1.1) to a spectrophotometric cell of suitable thickness, and measure the absorbance of each, A_{i1} , where i is 1 to 5 cm optical path length, with the spectrophotometer (5.1), at the wavelength of maximum absorbance near 535 nm, after having adjusted the instrument to zero absorbance against the calibration compensation solution (7.4.1.2).

Add sodium nitrite solution (4.14), drop by drop, to the solutions in the volumetric flasks, while mixing, until 1 drop in excess decolorizes the pink colour of the permanganate ion; transfer a part of each solution to the corresponding spectrophotometric cell, and measure the absorbances, A_{i2} , as described in the first paragraph.

NOTE An increase in the volume of the solution by addition of sodium nitrite solution is disregarded.

The measurement of absorbance should not be postponed unduly after decoloration of the pink colour of the permanganate ion by sodium nitrite solution, because reoxidation of the manganese ion occurs on standing.

7.4.1.4 Plotting the calibration graph

Plot a calibration graph showing the differences between the absorbances, $A_{i1} - A_{i2}$, of the set of calibration solutions as a function of the quantities of manganese contained in these solutions.

7.4.2 Samples containing less than 0,1 % manganese

7.4.2.1 Set of calibration solutions and formation of the absorbing compound

As in 7.4.1.1, but using manganese standard solution B (4.15.3) instead of manganese standard solution A.

For mass fractions of manganese below 0,1 %, the heating time should be approximately 30 min and, if necessary, water (4.1) should be added to maintain the volume.

7.4.2.2 Calibration compensation solution

Prepare a calibration compensation solution according to 7.4.2.1, but omitting the manganese standard solution B.

7.4.2.3 Spectrophotometric measurements

Proceed as in 7.4.1.3, but using the solutions from 7.4.2.1.

7.4.2.4 Plotting the calibration graph

See 7.4.1.4.

<https://standards.iteh.ai/catalog/standards/sist/e0bddfd2-1cee-4eeb-977f-32196545545a/iso-9682-2-2006>
 ISO 9682-2:2006

7.5 Determination

7.5.1 Decomposition of the test portion

If the decomposition is to be based on acid attack, proceed as instructed in 7.5.1.1. If the decomposition is to be based on alkali sintering, proceed as instructed in 7.5.1.2.

7.5.1.1 Acid attack

Place the test portion (7.2) in a 300 ml beaker, add 30 ml of hydrochloric acid (4.4), cover the beaker with a watch-glass and heat the solution gently without boiling.

For decomposition of the test portion, place the beaker for about 1 h on the low temperature zone (60 °C to 100 °C) of the hotplate, then transfer to a higher temperature zone and heat for about 10 min at just under boiling point.

Add 5 ml of nitric acid (4.6), 20 ml of perchloric acid (4.7), and 0,2 ml of sulfuric acid (4.9), cover the beaker again with the watch-glass, heat until dense white fumes of perchloric acid appear, and maintain a steady refluxing of acid on the walls of the beaker for about 10 min.

Allow the beaker to cool, add about 50 ml of warm water and a few drops of hydrogen peroxide (4.8), heat the mixture to dissolve the soluble salts, and boil to decompose the excess of hydrogen peroxide.

Filter the solution through a close-texture paper. Wash the beaker with water, scrubbing the wall with a rubber-tipped glass rod. Wash the residue first with three or four portions of sulfuric acid (4.10) and then with warm water. Collect the filtrate and washings in a 300 ml beaker and reserve as the main solution. Keep the filter paper with the residue and continue according to 7.5.2.