BRITISH STANDARD

BS ISO 9682-1:2009

Iron ores — Determination of manganese content

Part 1: Flame atomic absorption spectrometric method

ICS 73.060.10

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Full document details: BS ISO 9682-1:2009

National foreword

This British Standard is the UK implementation of ISO 9682-1:2009. It supersedes BS 7020-9.2:1993 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee ISE/58, Iron ores.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 July 2009

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ISBN 978 0 580 62727 9

Amendments/corrigenda issued since publication

Date	Comments

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INTERNATIONAL STANDARD

ISO 9682-1

Second edition
2009-06-15

Iron ores — Determination of manganese content —

Part 1: Flame atomic absorption spectrometric method

Minerais de fer — Dosage du manganèse —

Partie 1: Méthode par spectrométrie d'absorption atomique dans la flamme

Reference number
ISO 9682-1:2009(E)

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Published in Switzerland

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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 9682-1 was prepared by Technical Committee ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 2, *Chemical analysis*.

This second edition cancels and replaces the first edition (ISO 9682-1:1991), which has been technically revised.

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*

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Iron ores — Determination of manganese content —

Part 1:
Flame atomic absorption spectrometric 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 flame atomic absorption spectrometric method for the determination of the mass fraction of manganese in iron ores.

This method is applicable to a mass-fraction range of 0,01 % to 2,5 % of manganese in natural iron ores, 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 — Single-volume pipettes*

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

ISO 3082, *Iron ores — Sampling and sample preparation procedures*

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

3 Principle

The test portion is decomposed by one of the following methods.

a) Fusion of the test portion in sodium carbonate/sodium tetraborate flux and dissolution of the cooled melt in hydrochloric acid.

b) Treatment with hydrochloric acid, with the addition of a small amount of nitric acid. Evaporation to dehydrate silica, addition of hydrochloric acid, dilution and filtration. Ignition of the residue and removal of silica by evaporation with hydrofluoric and sulfuric acids. Fusion with sodium carbonate and sodium tetraborate and dissolution of the cooled melt in the filtrate.

The solution obtained, in both cases, is aspirated into the flame of an atomic absorption spectrometer using a dinitrogen oxide/acetylene flame.

The absorbance values obtained are compared with those obtained from the calibration solutions.

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4 Reagents

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

4.1 Sodium carbonate (Na_2CO_3), anhydrous.

4.2 Sodium tetraborate ($Na_2B_4O_7$), anhydrous.

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 1 + 1.

4.5 Nitric acid, ρ 1,4 g/ml.

4.6 Hydrofluoric acid, ρ 1,13 g/ml, 40 % (by mass), or 1,19 g/ml, 48 % (by mass).

4.7 Sulfuric acid, ρ 1,84 g/ml.

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

4.9 Background solution.

Dissolve 12 g of metallic iron (wire or powder) or 17 g of iron oxide (in both cases with a mass fraction of manganese less than 0,003 %) in 100 ml of hydrochloric acid (4.3) by heating in a covered 1 000 ml beaker. If metallic iron is used, oxidize by adding nitric acid (4.5) drop by drop. Cool and add 300 ml of hydrochloric acid (4.3). Dissolve 32 g of sodium carbonate (4.1) and 16 g of sodium tetraborate (4.2) in 300 ml of water, add carefully to the iron solution and heat to remove carbon dioxide. Cool, transfer to a 1 000 ml one-mark volumetric flask, dilute to volume with water and mix.

4.10 Manganese standard solutions.

4.10.1 Manganese stock solution, 500 µg Mn/ml.

Dissolve 0,500 g of high-purity manganese metal [minimum purity 99,9 % (by mass)] in 25 ml of hydrochloric acid (4.4) in a covered tall-form beaker, while heating. When dissolution is complete, cool, transfer the solution to a 1 000 ml one-mark volumetric flask, dilute to volume with water and mix.

4.10.2 Manganese standard solution, 50 µg Mn/ml.

Transfer 50 ml of manganese stock solution (4.10.1) to a 500 ml one-mark volumetric flask, dilute to volume with water and mix.

4.11 Manganese calibration solutions.

To a series of 100 ml one-mark volumetric flasks, transfer 0 ml; 5,0 ml; 10,0 ml; 20,0 ml; 30,0 ml, respectively, of manganese standard solution (4.10.2). Add 25,0 ml of background solution (4.9) to each flask, dilute to volume with water and mix.

The range of manganese concentrations that can be covered may vary from one instrument to another. Attention should be paid to the minimum performance criteria described in 5.5. For instruments with high sensitivity, the solution of highest concentration shown above may be deleted from the series and an additional solution included at the lower end, e.g. 2,0 ml of manganese standard solution (4.10.2).

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 the following.

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