

Analysis of iron ores —

Part 9: Methods for the determination of manganese content —

Section 9.1 Spectrophotometric method

[ISO title: Iron ores — Determination of manganese content —
Periodate spectrophotometric method]

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Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Iron and Steel Standards Committee (ISM/-) to Technical Committee ISM/58, upon which the following bodies were represented:

British Steel Industry
Institution of Mining and Metallurgy

The following body was also represented in the drafting of the standard, through subcommittees and panels:

British Ceramic Research Ltd.

This British Standard, having been prepared under the direction of the Iron and Steel Standards Committee, was published under the authority of the Board of BSI and comes into effect on 31 January 1989

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National foreword

This Section of BS 7020 has been prepared under the direction of the Iron and Steel Standards Committee. It is identical with ISO 3886:1986 “*Iron ores — Determination of manganese content — Periodate spectrophotometric method*” published by the International Organization for Standardization (ISO).

Terminology and conventions. The text of the International Standard has been approved as suitable for publication as a British Standard without deviation. Some terminology and certain conventions are not identical with those used in British Standards; attention is drawn especially to the following.

The comma has been used as a decimal marker. In British Standards it is current practice to use a full point on the baseline as the decimal marker.

In British Standards it is current practice to use the spelling “sulphur” etc., instead of “sulfur”, etc.

Wherever the words “International Standard” appear, referring to this standard, they should be read as “Section of BS 7020”.

Cross-references

International Standard Corresponding British Standard	
ISO 1042:1981	BS 1792:1982 <i>Specification for one-mark volumetric flasks</i> (Identical)
	BS 5660 <i>Methods of sampling iron ores</i>
ISO 3081:1986	Part 1:1987 <i>Manual method of increment sampling</i> (Identical)
ISO 3082:1987	Part 2:1987 <i>Mechanical method of increment sampling and sample preparation</i> (Identical)
ISO 3083:1986	BS 5661:1987 <i>Method for preparation of samples of iron ores by manual means</i> (Identical)
ISO 7764:1985	BS 7020 <i>Analysis of iron ores</i> Part 1:1988 <i>Method for the preparation of pre-dried test samples for chemical analysis</i> (Identical)

The Technical Committee has reviewed the provisions of ISO 648, to which reference is made in the text, and has decided that they are acceptable for use in conjunction with this standard.

A related British Standard to ISO 648 is BS 1583 “*Specification for one-mark pipettes*”.

The reference to ISO 5725 is for information only.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 8, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

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:

- a) treatment with hydrochloric, nitric and perchloric acids;
- b) 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.