

Analysis of iron ores —

Part 4: Method for the determination of total iron content —

Section 4.2 Titanium (III) chloride reduction methods

Committees responsible for this British Standard

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

- British Steel Industry
- Institution of Mining and Metallurgy
- Coopted members

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 Policy Committee, was published under the authority of the Board of BSI and comes into effect on 31 October 1990

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National foreword

This Section of BS 7020 has been prepared under the direction of the Iron and Steel Standards Policy Committee. It is identical with ISO 9507:1990 “*Iron ores — determination of total iron content — Titanium (III) chloride reduction methods*” published by the International Organization for Standardization (ISO).

Cross-references

International Standard	Corresponding British Standard
ISO 1042:1981	BS 1792:1982 <i>Specification for one-mark volumetric flasks</i> (Identical) BS 7020 <i>Analysis of iron ores</i>
ISO 2596:1984	Part 2:1988 <i>Method for the determination of hygroscopic moisture in analytical samples</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) BS 7020 <i>Analysis of iron ores</i>
ISO 7764:1985	Part 1:1988 <i>Method for the preparation of pre-dried test samples for chemical analysis</i>

The Technical Committee has reviewed the provisions of ISO 385-1 and 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 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 10, 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

This International Standard specifies two titrimetric methods, free from mercury pollution, for the determination of total iron content in iron ores, using potassium dichromate as titrant after reduction of the iron(III) by tin(II) chloride and titanium(III) chloride. The excess reductant is then oxidized by either dilute potassium dichromate (method 1) or perchloric acid (method 2).

Both methods are applicable to a concentration range of 30 % (m/m) to 72 % (m/m) of iron in natural iron ores, iron ore concentrates and agglomerates, including sinter products.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 385-1:1984, *Laboratory glassware — Burettes — Part 1: General requirements*.

ISO 648:1977, *Laboratory glassware — One-mark pipettes*.

ISO 1042:1983, *Laboratory glassware — One-mark volumetric flasks*.

ISO 2596:1984, *Iron ores — Determination of hygroscopic moisture in analytical samples — Gravimetric and Karl Fischer methods*.

ISO 3081:1986, *Iron ores — Increment sampling — Manual method*.

ISO 3082:1987, *Iron ores — Increment sampling and sample preparation — Mechanical method*.

ISO 3083:1986, *Iron ores — Preparation of samples — Manual method*.

ISO 7764:1985, *Iron ores — Preparation of predried test samples for chemical analysis*.

3 Principle

3.1 Decomposition of the test portion

3.1.1 Acid decomposition

For samples containing not more than 0,05 % (m/m) of vanadium, 0,1 % (m/m) of molybdenum or 0,1 % (m/m) of copper: by treatment with hydrochloric acid, filtration of the residue, ignition, treatment with hydrofluoric and sulfuric acids, fusion with potassium pyrosulfate, leaching of the melt and combination with the main iron solution.

3.1.2 Fusion-acidification

For samples containing not more than 0,05 % (m/m) of vanadium, 0,1 % (m/m) of molybdenum or 0,1 % (m/m) of copper: by fusion with alkali, leaching of the cold melt with water and acidification with hydrochloric acid.

3.1.3 Fusion-filtration

For samples containing more than 0,05 % (m/m) of vanadium and/or 0,1 % (m/m) of molybdenum but not more than 0,1 % (m/m) of copper: by fusion with alkali, leaching of the cold melt with water followed by filtration. Dissolution of the precipitate in hydrochloric acid.

3.2 Titration of iron

Reduction of the major portion of the iron(III) by tin(II) chloride and reduction of the remainder of the iron(III) by titanium(III) chloride. Oxidation of the excess reductant with either dilute potassium dichromate solution (method 1) or dilute perchloric acid (method 2). Titration of the reduced iron with potassium dichromate solution using sodium diphenylaminesulfonate indicator.

4 Reagents

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

	Method
4.1 Potassium pyrosulfate ($K_2S_2O_7$), fine powder.	1 and 2
4.2 Sodium carbonate (Na_2CO_3), anhydrous, or pre-ignited at 500 °C.	1 and 2
4.3 Sodium peroxide (Na_2O_2), dry powder.	1 and 2
NOTE Sodium peroxide should be stored as dry as possible and should not be used once it has begun to agglomerate.	
4.4 Hydrochloric acid , ρ 1,16 g/ml to 1,19 g/ml.	1 and 2
4.5 Hydrochloric acid , ρ 1,16 g/ml to 1,19 g/ml, diluted 1 + 1.	1 and 2
4.6 Hydrochloric acid , ρ 1,16 g/ml to 1,19 g/ml, diluted 1 + 10.	2
4.7 Hydrochloric acid , ρ 1,16 g/ml to 1,19 g/ml, diluted 1 + 50.	1 and 2
4.8 Hydrofluoric acid , 40 % (m/m) (ρ 1,13 g/ml) or 48 % (m/m) (ρ 1,19 g/ml).	1 and 2
4.9 Sulfuric acid , ρ 1,84 g/ml.	1 and 2
4.10 Sulfuric acid , ρ 1,84 g/ml, diluted 1 + 1.	1 and 2
Carefully pour 1 volume of reagent 4.9 into 1 volume of cold water.	1 and 2
4.11 Orthophosphoric acid , ρ 1,7 g/ml.	1 and 2