

Analysis of iron ores —

Part 22: Method for the determination of acid-soluble iron (II) content: titrimetric method

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Committees responsible for this British Standard

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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.

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National foreword

This Part of BS 7020 has been prepared under the direction of the Iron and Steel Standards Policy Committee. It is identical with ISO 9035:1989 “*Iron ores — Determination of acid-soluble iron (II) content — Titrimetric method*” published by the International Organization for Standardization (ISO).

Cross-references

International Standard	Corresponding British Standard
ISO 4695:1984	BS 6598:1985 <i>Method for determination of reducibility of iron ores</i> (Identical)
ISO 4791-1:1985	BS 6711 <i>Vocabulary relating to laboratory apparatus made essentially from glass, porcelain or vitreous silica</i> Part 1:1986 <i>Names for items of apparatus</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 7215:1985, 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 385-1:1984 is BS 846:1985 “*Specification for burettes*”.

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

This International Standard specifies a method for the determination of iron(II) content in natural and processed iron ores containing from 1 % to 25 % acid-soluble iron(II) and only traces of metallic iron. This method is specifically limited for use in conjunction with the reducibility test methods specified in ISO 4695 or ISO 7215.

This method is not applicable to ores containing more than 0,3 % (m/m) sulfur (as sulfide) and/or 5 % (m/m) free carbon. Experiments have shown that the acid-insoluble residue can be neglected, especially as regards the needs of the reducibility test methods concerned and the precision obtainable.

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 listed 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 4695:1984, *Iron ores — Determination of reducibility*.

ISO 4791-1:1985, *Laboratory apparatus — Vocabulary relating to apparatus made essentially from glass, porcelain or vitreous silica — Part 1: Names for items of apparatus*.

ISO 7215:1985, *Iron ores — Determination of relative reducibility*.

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

3 Principle

Dissolution with hydrochloric acid in an inert atmosphere, addition of a mixture of sulfuric and phosphoric acid and dilution with water. Determination of the iron(II) content by titration with potassium dichromate standard solution using sodium diphenylaminesulfonate as indicator.

4 Reagents

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

NOTE The water used for the procedure should be de-oxygenated by boiling.

4.1 Sodium carbonate (Na_2CO_3) or *sodium hydrogen carbonate* (NaHCO_3).

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

NOTE This reagent should be de-oxygenated by purging with either nitrogen or argon, or by heating for a short time.

4.3 Hydrofluoric acid, 40 % (m/m), ρ 1,13 g/ml or 48 % (m/m), ρ 1,16 g/ml.

4.4 Sodium carbonate or *sodium hydrogen carbonate*, saturated solution, at ambient temperature.

4.5 Sulfuric acid — phosphoric acid mixture.

To 300 ml of water, add cautiously 150 ml of sulfuric acid (ρ 1,84 g/ml), with constant stirring. After cooling, add 150 ml of phosphoric acid (ρ 1,71 g/ml). Cool and dilute with water to 1 000 ml.

4.6 Nitrogen (99,99 % N_2) or *argon* (optional).

4.7 Potassium dichromate, standard volumetric solution, $c(\text{K}_2\text{Cr}_2\text{O}_7) = 0,016\,67\text{ mol/l}$.

Dissolve 4,904 g of pulverized potassium dichromate (dried at 140 °C to 150 °C for 2 h and cooled to room temperature in a desiccator) in water and dilute to a volume of 1 000 ml after cooling to 20 °C.

4.8 Sodium diphenylaminesulfonate solution

Dissolve 0,2 g of powdered sodium diphenylaminesulfonate ($\text{C}_6\text{H}_5\text{NHC}_6\text{H}_4\text{SO}_3\text{Na}$) in 100 ml of water. Store the solution in a brown glass bottle.

NOTE 1 Alternatively, an equivalent quantity of indicator in pellet form may be used.

NOTE 2 Barium diphenylaminesulfonate may be used instead of sodium diphenylaminesulfonate.

5 Apparatus

Ordinary laboratory apparatus, and

5.1 Conical flask, of capacity 500 ml.

5.2 Göckel safety trap (see ISO 4791-1) or an apparatus with a feed tube for inert gas.

NOTE Alternatively, a slit rubber valve (Bunsen valve) may be used, but its efficiency depends on the length of the slit and the quality and flexibility of the rubber tubing.

5.3 Burette, class B, conforming with ISO 385-1.

6 Sampling and samples

6.1 Laboratory sample

Crush and pulverize the whole of one of the test portions for the reducibility test, which has been reserved for chemical analysis, down to minus 100 μm particle size. In the case of ores with 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.