

Analysis of iron ores —

Part 8: Methods for the determination of aluminium content —

Section 8.1 Titrimetric method

[ISO title: Iron ores — Determination of aluminium content —
EDTA titrimetric 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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The following BSI references relate to the work on this standard:
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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 6830:1986 “*Iron ores — Determination of aluminium content — EDTA titrimetric 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.

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
	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 385-1, 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 is BS 846 “*Specification for burettes*”.

The reference to ISO 5725 is for information only.

Additional information. In clause 2 the footnote to ISO 3082 no longer applies as this standard has now been published.

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 titrimetric method using EDTA for the determination of the aluminium content of iron ores.

This method is applicable to a concentration range of 0,25 to 5,0 % (*m/m*) of aluminium in natural iron ores, and iron ore concentrates and agglomerates including sinter products.

2 References

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

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

ISO 3082, *Iron ores — Increment sampling and sample preparation — Mechanical method*¹⁾.

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

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

3 Principle

Fusion of a test portion, in a vitreous carbon or zirconium crucible, with a mixed flux of sodium carbonate and sodium peroxide. Dissolution of the cooled melt in hydrochloric acid, precipitation of R_2O_3 with ammonia solution, filtration and redissolution of the hydroxides in hydrochloric acid.

Treatment with cupferron and chloroform, extraction of elements such as iron and titanium and rejection of the organic phase. Treatment of the aqueous phase with nitric and per-chloric acids, evaporation, treatment with hydrochloric acid, dilution and filtration.

Addition to the filtrate of an excess of disodium dihydrogen ethylenedinitrilotetraacetate (EDTA) and titration of the excess with zinc standard volumetric solution using xylenol orange indicator. Addition of ammonium fluoride to release the EDTA bound to aluminium, and titration with zinc solution as before.

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 peroxide (Na_2O_2), dry.

4.3 Nitric acid, ρ 1,40 g/ml.

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 + 5.

4.6 Hydrochloric acid, ρ 1,16 to 1,19 g/ml diluted 1 + 1.

4.7 Ammonia, solution.

Ammonia (ρ 0,880 to 0,890 g/ml) diluted 1 + 1.

4.8 Ammonium chloride (NH_4Cl), 10 g/l solution containing 2 drops (0,1 ml) of ammonia solution per 100 ml.

4.9 Cupferron (nitrosophenylhydroxylamine ammonium salt) [$C_6H_5N(NO)ONH_4$], 60 g/l solution.

Prepare this solution in cold ($< 20^\circ C$) water on the day of use. Filter through a rapid paper and cool to $10^\circ C$.

4.10 Chloroform

4.11 Perchloric acid, ρ 1,67 g/ml approx.

4.12 Sodium hydroxide, 100 g/l solution.

4.13 Sodium hydroxide, 10 g/l solution.

4.14 Acetate buffer

Dissolve 136 g of sodium acetate tri-hydrate ($CH_3COONa \cdot 3H_2O$) in about 600 ml water. Add 7 ml of glacial acetic acid and dilute to 1 litre.

Store this solution in a polyethylene bottle.

4.15 Ammonium fluoride, 100 g/l solution.

Dissolve 10 g of ammonium fluoride (NH_4F) in 100 ml water in a polyethylene beaker.

Prepare this solution on the day of use.

4.16 Xylenol orange, indicator.

Triturate 0,1 g of xylenol orange with a little water to make a paste. Dilute to 100 ml with water. Filter. Store in an amber-coloured glass-stoppered bottle.

The solution is stable for one week.

4.17 Zinc, standard volumetric solution, $c(Zn^{2+}) = 0,01$ mol/l.

Weigh 0,653 8 g of high purity (99,99 % Zn) zinc metal (clippings or small pieces) into a 125 ml Erlenmeyer flask. Add 5 ml of water followed by 2,5 ml of nitric acid (4.1). Cover the flask and gently simmer. If the reaction subsides leaving undissolved metal, add 2 ml increments of water followed by simmering until complete solution is obtained. Add 50 ml of water and gently boil for 2 min. Cool. By dropwise addition of sodium hydroxide solution (4.13) adjust the pH to 4, measured by pH meter. Dilute to 1 000 ml with water in a volumetric flask.

4.18 Disodium dihydrogen ethylenedinitrilotetraacetate dihydrate (EDTA), solution, $c(C_{10}H_{14}N_2O_8Na_2 \cdot 2H_2O) = 0,01$ mol/l.

¹⁾ At present at the stage of draft.