

Analysis of iron ores —

Part 6: Methods for the determination of phosphorus content —

Section 6.1 Titrimetric method

[ISO title: Iron ores — Determination of phosphorus 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 Section of BS 7020 has been prepared under the direction of the Iron and Steel Standards Committee. It is identical with ISO 2599:1983 “*Iron ores — Determination of phosphorus content — 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.

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
	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 reference to ISO 5725 is for information only.

Additional information. In clause 2 the footnote to ISO 3082 and ISO 7764 no longer applies as these standards have 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 for the determination of phosphorus content in iron ores, using hexaammonium heptamolybdate (ammonium molybdate).

This method is applicable to a concentration range of 0,10 to 5,0 % (m/m)¹ of phosphorus in natural iron ores, and iron ore concentrates and agglomerates including sinter products.

2 References

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

ISO 3082, *Iron ores — Increment sampling and sample preparation — Mechanical method*².

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

ISO 7764, *Iron ores — Preparation of predried test samples for chemical analysis*².

3 Principle

Decomposition of the test portion by treatment with hydrochloric, nitric and perchloric acids. Filtration of the insoluble residue, the filtrate being reserved as the main solution.

Ignition of the residue, treatment with sulfuric and hydrofluoric acids to remove silicon dioxide, fusion with sodium carbonate and leaching of the melt with water. Filtration of the alkaline solution, the residue being discarded.

Acidification of the filtrate from residue treatment with hydrochloric acid, addition of iron(III) chloride and collection of the phosphorus by precipitation with ammonia solution. Dissolution of the precipitate in hydrochloric acid and combination with the main solution. Addition of hydrobromic acid and evaporation to fumes of perchloric acid to expel arsenic.

Dissolution of the salts in water, neutralization of the solution, or an aliquot, with ammonia solution and adjustment of the acidity with nitric acid. Cooling of the solution to 20 °C and reduction of vanadium by iron(II) sulfate. Addition of ammonium molybdate to the cold solution and collection by filtration of the yellow precipitate of ammonium molybdophosphate.

Washing free of acid and dissolution in a slight excess of sodium hydroxide, the excess being titrated with nitric acid.

For test portions containing more than 18 mg of titanium, a modified procedure is used (see note 3 to 7.3.1).

4 Reagents

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

4.1 Ammonium nitrate (NH₄NO₃).

4.2 Sodium carbonate (Na₂CO₃), anhydrous.

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

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

4.5 Nitric acid, ρ 1,42 g/ml.

4.6 Nitric acid, ρ 1,4 g/ml diluted 1 + 50.

4.7 Nitric acid, ρ 1,4 g/ml diluted 1 + 5 000.

4.8 Sulfuric acid, ρ 1,84 g/ml.

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

4.10 Perchloric acid, ρ 1,54 g/ml, 60 % (m/m) solution, or ρ 1,67 g/ml, 70 % (m/m) solution.

4.11 Hydrofluoric acid, ρ 1,13 g/ml, 40 % (m/m) solution.

4.12 Hydrobromic acid, ρ 1,38 g/ml, 40 % (m/m) solution or ρ 1,48 g/ml, 47 % (m/m) solution.

4.13 Ammonia solution, ρ 0,9 g/ml.

4.14 Hydrogen peroxide, 30 % (m/m) solution, free from phosphate stabilizer.

4.15 Potassium nitrate, 10 g/l solution.

4.16 Ammonium molybdate, solution.

Dissolve 40 g of finely pulverized crystalline ammonium molybdate tetrahydrate [(NH₄)₆Mo₇O₂₄·4H₂O] in 300 ml of warm water and 80 ml of the ammonia solution (4.13), allow the solution to cool, and add this solution in small portions to 600 ml of nitric acid (1 + 1), while stirring. Add a few milligrams of sodium hydrogen phosphate or ammonium hydrogen phosphate and allow to stand for at least 2 days. Filter before use.

4.17 Iron(III) chloride solution, containing 3 g of Fe per litre.

Dissolve 0,3 g of pure iron, with as small a content of phosphorus as possible, by treatment with hydrochloric acid (4.3), add a few drops of nitric acid (4.5) to oxidize the iron, and boil the mixture to remove chlorine.

Allow the solution to cool and dilute with water to 100 ml.

¹ This method has been tested internationally on samples with phosphorus contents ranging from 0,03 to 1,50 % (m/m). See Annex B.

² At present at the stage of draft.