
**Iron ores — Determination of phosphorus
content — Titrimetric method**

Minerais de fer — Dosage du phosphore — Méthode titrimétrique

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 2599 was prepared by Technical Committee ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 2, *Chemical analysis*.

This third edition cancels and replaces the second edition (ISO 2599:1983), which has been editorially revised.

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Iron ores — Determination of phosphorus content — Titrimetric method

WARNING — This International Standard may involve hazardous materials, operations and equipment. This International Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this International Standard to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies a titrimetric method for the determination of the phosphorus content of iron ores, using hexaammonium heptamolybdate (ammonium molybdate).

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

This International Standard provides a quality control method for the determination of phosphorus by titration, however, the method cannot be used for referee purposes.

2 Normative references

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The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 1042:1998, *Laboratory glassware — One-mark volumetric flasks*

ISO 3082:2000, *Iron ores — Sampling and sample preparation procedures*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*

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

3 Principle

The test portion is decomposed by treatment with hydrochloric, nitric and perchloric acids. The insoluble residue is filtered and the filtrate is reserved as the main solution.

The residue is ignited, treated with sulfuric and hydrofluoric acids to remove silicon dioxide, fused with sodium carbonate and the melt is leached with water. The alkaline solution is filtered and the residue is discarded.

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

The filtrate from residue treatment is acidified using hydrochloric acid, iron(III) chloride is added and the phosphorus is collected by precipitation with ammonia solution. The precipitate is dissolved in hydrochloric acid and combined with the main solution. Hydrobromic acid is added and evaporated to fumes of perchloric acid to expel arsenic. The salts are dissolved in water and the solution, or an aliquot, is neutralized with ammonia solution and the acidity is adjusted using nitric acid. The solution is cooled to 20 °C and vanadium is reduced by iron(II) sulfate. Ammonium molybdate is added to the cold solution and the yellow precipitate of ammonium molybdophosphate is collected by filtration.

The precipitate is washed free of acid and dissolved 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 7.4.1.2).

4 Reagents

During the analysis, use only reagents of recognized analytical grade, and only water that conforms to grade 2 of ISO 3696:1987.

4.1 Ammonium nitrate (NH_4NO_3).

4.2 Sodium carbonate (Na_2CO_3), anhydrous.

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

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

4.5 Nitric acid, ρ 1,42 g/ml.

4.6 Nitric acid, ρ 1,42 g/ml diluted 1 + 50.

4.7 Nitric acid, ρ 1,42 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 $[(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}]$ in 300 ml of warm water and 80 ml of 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 d. Filter before use.

4.17 Iron(III) chloride solution, containing 3 g of Fe/l.

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.

4.18 Iron(III) sulfate solution, 100 g/l.

Dissolve 100 g of iron(II) sulfate heptahydrate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) in 1 l of sulfuric acid (1 + 19).

4.19 Water, free of CO_2 .

Heat distilled or deionized water to boiling point in a conical flask for 5 min to expel carbon dioxide. Cool, protecting the water in an appropriate way against any pickup of carbon dioxide.

4.20 Sodium hydroxide, standard volumetric solution, $c(\text{NaOH}) = 0,2 \text{ mol/l}$ approximately.

Dissolve 8 g of sodium hydroxide in 1 l of freshly boiled and cooled water (4.19). Standardize this solution using either amidosulfuric acid (sulfamic acid) or potassium hydrogen phthalate.

Dry either potassium hydrogen phthalate or sulfamic acid at 105°C for 1 h and weigh, to the nearest 0,001 g, either approximately 1 g of potassium hydrogen phthalate or approximately 0,5 g of sulfamic acid. Dissolve either material in 50 ml of water free of CO_2 (4.19) and titrate with sodium hydroxide solution (4.20) using phenolphthalein solution (4.22) as indicator.

4.21 Nitric acid, standard volumetric solution $c(\text{HNO}_3) = 0,2 \text{ mol/l}$ approximately.

Dilute 13 ml of nitric acid (4.5) with water, to 1 l.

Standardize the solution as follows: accurately measure 25 ml of sodium hydroxide standard volumetric solution (4.20) and titrate with nitric acid solution, using phenolphthalein solution (4.22) as indicator.

4.22 3,3-Bis(4-hydroxyphenyl)phthalide (phenolphthalein) solution, 0,1 g/100 ml.

Dissolve 0,10 g of powdered phenolphthalein in 90 ml of ethanol, 95 % (V/V), and dilute with water, to 100 ml.

5 Apparatus

Ordinary laboratory apparatus, including one-mark pipettes and one-mark volumetric flasks in accordance with ISO 648 and ISO 1042.

6 Sampling and samples**6.1 Laboratory sample**

For analysis, use a laboratory sample of – 100 μm particle size which has been taken and prepared in accordance with ISO 3082. In the case of ores having significant contents of combined water or oxidizable compounds, use a particle size of – 160 μm .

NOTE A guideline on significant contents of combined water and oxidizable compounds is incorporated in ISO 7764.

6.2 Preparation of predried test samples

Thoroughly mix the laboratory sample and, taking multiple increments, extract a test sample in such a manner that it is representative of the whole contents of the container. Dry the test sample at $105\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ as specified in ISO 7764. (This is the predried test sample.)

7 Procedure

7.1 Number of determinations

Carry out the analysis at least in duplicate in accordance with the flowsheet in Annex A, independently, on one predried test sample.

NOTE The expression “independently” means that the second and any subsequent result is not affected by the previous result(s). For this particular analytical method, this condition implies that the repetition of the procedure be carried out either by the same operator at a different time or by a different operator including, in either case, appropriate recalibration.

7.2 Test portion

Taking several increments, weigh, to the nearest 0,000 2 g, the amount of the predried test sample (6.2) specified in Table 1.

NOTE The test portion should be taken and weighed quickly, in order to avoid reabsorption of moisture.

Table 1 — Mass of test portion

Phosphorus content %	Mass of test portion g	Volume of aliquot portion ml	Volume of ammonium molybdate solution (4.16) ml
0,1 to 0,5	1,0	total	50
0,5 to 2,5	0,5	total	100
2,5 to 5,0	0,5	100/250	100

7.3 Blank test and check test

In each run, one blank test and one analysis of a certified material of the same type of ore shall be carried out in parallel with the analysis of the ore sample(s) under the same conditions. A predried test sample of the certified reference material shall be prepared as specified in 6.2.

NOTE The certified reference material should be of the same type as the sample to be analysed and the properties of the two materials should be sufficiently similar to ensure that in either case no significant changes in the analytical procedure would become necessary. Where a certified reference material is not available, a reference material may be used (see 8.2.4).

Where the analysis is carried out on several samples at the same time, the blank value may be represented by one test, provided that the procedure is the same and the reagents used are from the same reagent bottles.

Where the analysis is carried out on several samples of the same type of ore at the same time, the analytical value of one certified reference material may be used.

7.4 Determination

7.4.1 Decomposition of the test portion

7.4.1.1 Test portions containing less than 18 mg titanium

7.4.1.1.1 Initial decomposition

Place the test portion (7.2) in a 300 ml beaker, add 25 ml of hydrochloric acid (4.3), cover the beaker with a watch-glass and heat gently to decompose the ore.

For decomposition of the test portion, place the beaker in a low temperature zone (60 °C to 100 °C) of the hot-plate to digest the sample for about 1 h, then transfer to a higher temperature zone and heat for about 10 min just below boiling.

Add 5 ml of nitric acid (4.5) and 15 ml of perchloric acid (4.10), cover the beaker and heat to dense white fumes of perchloric acid. Maintain a steady refluxing of the acid on the walls of the beaker for about 10 min. Allow the beaker to cool, add about 50 ml of warm water and heat to dissolve soluble salts.

Filter the solution through a close-texture paper and wash the residue three to five times with nitric acid (4.6) and finally with hot water until free from acid, receiving the filtrate and washings in a 300 ml beaker. Evaporate the filtrate by gently heating and reserve as the main solution.

NOTE If a sample obviously contains organic matter, carbonates or sulfides, and decomposition becomes easier by roasting, place the test portion, accurately weighed, in a porcelain crucible. Cover the crucible and roast at 700 °C to 750 °C for about 30 min. Allow the crucible to cool to room temperature in a desiccator, wash out the contents of the crucible into a 300 ml beaker, and proceed as directed in 7.4.1.1.

7.4.1.1.2 Treatment of the residue

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While the main solution is evaporating, place the filter paper and residue in a platinum crucible, dry, char the paper and finally ignite at 750 °C to 800 °C. Cool the crucible, moisten the residue with a few drops of sulfuric acid (4.9), add 5 ml of hydrofluoric acid (4.11) and evaporate to remove silicon dioxide and sulfuric acid. Cool the crucible, add 3 g of sodium carbonate (4.2) and fuse until a clear melt is obtained.

Cool the crucible, place it in a 300 ml beaker, add 100 ml of water and heat to disintegrate the melt and to dissolve all soluble salts. Remove and rinse the crucible. Filter the alkaline solution through a medium-texture paper and wash with warm water, collecting the filtrate and washings in a 300 ml beaker. Discard the residue.

Add hydrochloric acid (4.3) to the filtrate until it is acidic, boil to remove carbon dioxide, cool and add 10,0 ml of iron(III) chloride solution (4.17). Add ammonia solution (4.13) in small portions while stirring, until the solution is slightly alkaline (indicator paper), in order to precipitate iron hydroxide and iron phosphate. Boil the solution for about 2 min, then remove from the source of heat. Allow the precipitate to settle, filter through a rapid filter paper and wash with hot water. Discard the filtrate.

Place the beaker containing the main solution from 7.4.1.1.1 under the funnel. Dissolve the precipitate by pouring on to the paper about 10 ml of hydrochloric acid (4.4). Wash with warm water.

7.4.1.2 Test portions containing more than 18 mg titanium

If the test portion contains more than 18 mg of titanium, the following procedure should be applied.

Place the test portion (7.2) in a platinum crucible lined with 1 g of fused sodium carbonate. Mix the test portion with ten times its mass of fusion mixture (sodium carbonate:sodium nitrate 10:1) and heat gently at first, then at a higher temperature to complete fusion of the test portion. After cooling, place the crucible together with its contents in a 300 ml beaker, add about 100 ml of warm water and stir gently. Remove the melt from the crucible, rinse the crucible with water and remove.