SLOVENSKI STANDARD
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Ģelezove rude ï Doloļ evanje fosforja ï Titrimetrijska metoda

Iron ores -- Determination of phosphorus content -- Titrimetric method

Minerais de fer -- Dosage du phosphore -- Méthode titrimétrique

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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.

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

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.

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[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 $[(NH_4)_6Mo_7O_{24}{\cdot}4H_2O]$ 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 ($FeSO_4 \cdot 7H_2O$) 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, $c(\text{NaOH}) = 0{,}2$ 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 $c(\text{HNO}_3) = 0{,}2$ 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.