Nederlandse norm

NEN-ISO 5418-1

(en)

Iron ores - Determination of copper content - Part 1: 2,2'-Biquinolyl spectrophotometric method (ISO 5418-1:2006,IDT)

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INTERNATIONAL STANDARD

ISO 5418-1

Second edition
2006-12-01

Iron ores — Determination of copper —

Part 1: 2,2′-Biquinolyl spectrophotometric method

Minerais de fer — Dosage du cuivre —

Partie 1: Méthode spectrophotométrique à la biquinoléine-2,2′

Reference number
ISO 5418-1:2006(E)

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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 5418-1 was prepared by Technical Committee ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 2, *Chemical analysis*.

This second edition cancels and replaces the first edition (ISO 5418-1:1994), which has been technically revised. It has been updated to alter the manner in which precision data are presented.

ISO 5418 consists of the following parts, under the general title *Iron ores — Determination of copper*:

— *Part 1: 2,2'-Biquinolyl spectrophotometric method*

— *Part 2: Flame atomic absorption spectrometric method*

INTERNATIONAL STANDARD **ISO 5418-1:2006(E)**

Iron ores — Determination of copper —

Part 1:
2,2′-Biquinolyl spectrophotometric method

WARNING — This part of ISO 5418 may involve hazardous materials, operations and equipment. This part of ISO 5418 does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this part of ISO 5418 to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This part of ISO 5418 specifies a 2,2′-biquinolyl spectrophotometric method for the determination of copper in iron ores.

This method is applicable to mass fractions of copper between 0,005 % and 0,77 % in natural iron ores, iron ore concentrates and agglomerates, including sinter products.

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, *Laboratory glassware — One-mark pipettes*

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

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

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

ISO 7764, *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.

Silica is dehydrated and the solution is diluted and filtered. The residue is ignited, treated with hydrofluoric and sulfuric acids, and fused with sodium carbonate. The cooled melt is dissolved in the filtrate.

Copper(II) is reduced with ascorbic acid. 2,2′-biquinolyl is added in the presence of *N,N*-dimethylformamide to form the red-violet complex of copper(I).

The absorbance of the coloured complex is measured spectrophotometrically at a wavelength of approximately 545 nm.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and water that complies with grade 3 of ISO 3696.

The distillation apparatus used should not contain any copper, and deionized water should not come into contact with copper tubing or taps.

4.1 Sodium carbonate (Na_2CO_3), anhydrous powder.

4.2 Iron(III) oxide, minimum purity: 99,9 % (mass fraction), mass fraction of copper less than 0,000 2 %.

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 + 2.

4.5 Hydrochloric acid, ρ 1,16 g/ml to 1,19 g/ml, diluted 1 + 10.

4.6 Nitric acid, ρ 1,4 g/ml.

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

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

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

4.10 Hydrofluoric acid, ρ 1,13 g/ml, 40 % (*m/m*), or ρ 1,185 g/ml, 48 % (*m/m*).

4.11 Ascorbic acid ($C_6H_8O_6$), solution, 200 g/l.

Prepare this solution at the time of use.

4.12 *N,N*-Dimethylformamide [$HCON(CH_3)_2$].

WARNING — Take care not to inhale toxic fumes.

4.13 2,2′-Biquinolyl ($C_{18}H_{12}N_2$), solution.

Dissolve 0,15 g of 2,2′-biquinolyl in 250 ml of *N,N*-dimethylformamide. Protect the solution from light and store in a brown bottle.

4.14 Copper standard solutions.

4.14.1 Standard solution A, 1 000 µg Cu/ml.

Dissolve 0,500 g of copper metal [of minimum purity 99,9°% (mass fraction)] in 20 ml of dilute nitric acid (4.7) in a 250 ml tall-form beaker. After elimination of the nitrous fumes by boiling, cool, transfer to a 500 ml one-mark volumetric flask, dilute to volume with water and mix.

4.14.2 Standard solution B, 50 µg Cu/ml.

Transfer 25,0 ml of standard solution A (4.14.1) to a 500 ml one-mark volumetric flask and dilute to volume with water.

5 Apparatus

Ordinary laboratory equipment, including one-mark pipettes and one-mark volumetric flasks complying with the specifications of ISO 648 and ISO 1042 respectively (unless otherwise indicated), and the following.

5.1 Platinum crucible, of capacity 25 ml to 30 ml.

5.2 Muffle furnace, suitable for heating at 1 000 °C.

5.3 Spectrophotometer, suitable for measurement of an absorbance of approximately 545 nm.

6 Sampling and samples

6.1 General

For analysis, use a laboratory sample of minus 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 minus 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 °C ± 2 °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 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 is carried out either by the same operator at a different time or by a different operator, including appropriate recalibration in either case.

7.2 Test portion

Taking several increments, weigh, to the nearest 0,000 2 g, approximately 0,5 g or 1 g of the test sample (see Table 1) obtained in accordance with 6.2.

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

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