

International Standard



5418

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Iron ores — Determination of copper content — 2,2'-Biquinolyl spectrophotometric method

Minerais de fer — Dosage du cuivre — Méthode spectrophotométrique à la biquinoléine-2,2'

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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 developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been authorized 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 5418 was developed by Technical Committee ISO/TC 102, *Iron ores*, and was circulated to the member bodies in November 1981.

It has been approved by the member bodies of the following countries :

Australia	Germany, F.R.	Romania
Austria	India	South Africa, Rep. of
Brazil	Italy	Spain
Canada	Japan	Sweden
China	Korea, Dem. P. Rep. of	Thailand
Czechoslovakia	Mexico	United Kingdom
Egypt, Arab Rep. of	Netherlands	USA
France	New Zealand	Venezuela

No member body expressed disapproval of the document.

Iron ores — Determination of copper content — 2,2'-Biquinolyl spectrophotometric method

1 Scope and field of application

This International Standard specifies a 2,2'-biquinolyl spectrophotometric method for the determination of copper in iron ores.

This method is applicable to a concentration range of 0,004 to 0,8 % (m/m) of copper 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.*¹⁾

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

ISO 7764, *Iron ores — Preparation of predried test samples for chemical analysis.*¹⁾

3 Principle

Decomposition of the test portion by treatment with hydrochloric, nitric and perchloric acids.

Dehydration of silica, dilution and filtration. Ignition of the residue, treatment with hydrofluoric and sulfuric acids and fusion with sodium carbonate. Dissolution of the cooled melt in the filtrate.

Reduction of copper(II) with ascorbic acid. Addition of 2,2'-biquinolyl in the presence of *N,N*-dimethylformamide to form the red-violet complex of copper(I).

Spectrophotometric measurement of the absorbance of the coloured complex at a wavelength of about 545 nm.

4 Reagents

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

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

1) At present at the stage of draft.

4.1 Sodium carbonate (Na_2CO_3), anhydrous.

4.2 Iron(III) oxide.

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 + 2.

4.5 Hydrochloric acid, ρ 1,16 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, 60 % (m/m), ρ 1,54 g/ml, or 70 % (m/m), ρ 1,67 g/ml.

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

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

4.11 Ascorbic acid ($\text{C}_6\text{H}_8\text{O}_6$), 200 g/l solution.

Prepare this solution at the time of use.

4.12 *N,N*-Dimethylformamide [$\text{HCON}(\text{CH}_3)_2$].

Take care not to inhale toxic fumes.

4.13 2,2'-Biquinolyl ($\text{C}_{18}\text{H}_{12}\text{N}_2$) 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

Dissolve 0,500 g of copper metal (99,9 %) in 20 ml of the nitric acid solution (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 the mark with water and mix.

1 ml of this solution contains 1,00 mg of Cu.

4.14.2 Standard solution B

Take 25,0 ml of standard solution A (4.14.1) and dilute to the mark in a 500 ml one-mark volumetric flask.

1 ml of this solution contains 0,05 mg of Cu.

5 Apparatus

Ordinary laboratory apparatus and

5.1 Spectrophotometer, suitable for measurement of absorbance in the region of 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 in accordance with ISO 3081 or ISO 3082 and prepared in accordance with ISO 3082 or ISO 3083. In the case of ores with 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 $105 \pm 2^\circ\text{C}$ as specified in ISO 7764. (This is the predried test sample.)

7 Procedure

7.1 Number of determinations

Carry out the analysis generally in duplicate, independently, on one predried test sample.

NOTE — The expression "independently" implies that the repetition of the procedure shall be carried out at a different time and include appropriate recalibration.

7.2 Blank test and check test

In each run, one blank test and one analysis of a certified reference 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.

When 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 are from the same reagent bottles.

When 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.

7.3 Test portion

Weigh, to the nearest 0,000 2 g, approximately 0,5 or 1 g of the test sample (see table 1 in 7.4.4), obtained in accordance with 6.2.

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

7.4 Determination

7.4.1 Decomposition of the test portion

Place the test portion (7.3) in a 250 ml beaker (tall form) and moisten with 5 ml of water. Add 20 ml of the hydrochloric acid (4.3), cover the beaker with a watch glass and heat the solution gently without boiling until decomposition of the test portion is complete. Add 5 ml of the nitric acid (4.6) followed by 10 ml of the perchloric acid (4.8) and 0,2 ml of the sulfuric acid (4.9), cover the beaker with a watch glass and heat until perchloric acid fumes are evolved. Continue the heating for a further 3 to 5 min.

Allow the beaker to cool and add 20 ml of the hydrochloric acid solution (4.4). Boil for 1 min to remove chlorine and dilute with 10 ml of water.

Filter the solution through a medium-texture filter paper, collecting the filtrate in a 300 ml beaker. Wash the paper with the hydrochloric acid solution (4.5) (use as small a volume as possible) until the yellow colour due to iron(III) can no longer be detected. Finally, wash with hot water until the washings are free from acid. Reserve the filtrate and washings as the main solution. Transfer the filter paper containing the residue to a platinum crucible.

7.4.2 Treatment of the residue

Dry and burn off the filter paper at a low temperature and ignite the residue at about 800°C in a muffle furnace. Allow the crucible to cool, moisten the residue with a few drops of water, and add 5 drops of the sulfuric acid solution (4.9) and 5 ml of the hydrofluoric acid (4.10).

Heat gently, in a fume cupboard, to volatilize silica as the tetrafluoride, and evaporate the sulfuric acid to dryness. Finally, heat the crucible at a high temperature for several seconds to ensure complete removal of sulfuric acid. Allow to cool and add 1 g of the sodium carbonate (4.1). Heat gently for several minutes, then at 900 to $1\,000^\circ\text{C}$ until decomposition of the residue is complete.

NOTE — With a large amount of residue, additional sodium carbonate may be required. If so, the amount of sodium carbonate taken in 7.5 must be increased correspondingly.