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Iron ores — Determination of trace elements — Plasma spectrometric method

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National foreword

This British Standard is the UK implementation of ISO 22682:2017.

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A list of organizations represented on this committee can be obtained on request to its secretary.

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see the following URL: www.iso.org/iso/foreword.html.

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

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Iron ores — Determination of trace elements — Plasma spectrometric method

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

1 Scope

This document specifies a method for the determination of phosphorus, vanadium, titanium, copper, nickel, chromium, barium and cobalt in iron ores, by inductively coupled plasma atomic emission spectrometry (ICP-AES).

This method is applicable to the concentration ranges (mass fraction) given in Table 1, in natural iron ores, iron ore concentrates and agglomerates, including sinter products.

Table 1 — Concentration ranges

Element	Concentration range for referee purpose % (mass fraction)	Concentration range for non-referee purpose % (mass fraction)
P	0,000 2 to 0,150	0,000 2 to 0,150
V	0,003 0 to 0,024	0,003 0 to 0,024
Ti	0,015 0 to 0,120	0,015 0 to 0,120
Cu	0,0014 to 0,250	0,001 4 to 0,250
Ni	0,005 0 to 0,090	0,005 0 to 0,090
Cr		0,004 0 to 0,015
Ba		0,002 8 to 0,035
Co		0,002 0 to 0,100

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 — Single-volume pipettes*

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

ISO 2596, *Iron ores — Determination of hygroscopic moisture in analytical samples — Gravimetric, Karl Fischer and mass-loss methods*

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*

ISO 11323, *Iron ore and direct reduced iron — Vocabulary*

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3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 11323 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at https://www.iso.org/obp

4 Principle

The test portion is decomposed with L-ascorbic acid, hydrofluoric acid, concentrated hydrochloric acid, concentrated nitric acid and then evaporated to dryness. The dry mass is dissolved in hydrochloric acid (1 + 1) and the solution is made up to 100 ml.

The solution is diluted to volume and measured on an ICP spectrometer. Final results are read from a calibration graph prepared using standard calibration solutions.

5 Reagents

During the analysis, use only highly pure reagents of recognized analytical grade and only water that complies with grade 2 of ISO 3696.

5.1 High-purity iron, metal, of minimum purity grade 99,99 % (mass fraction).

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

5.3 Nitric acid, ρ 1,38 g/ml to 1,4 g/ml

5.4 Hydrofluoric acid, ρ 1,13 g/ml to ρ 1,16 g/ml.

5.5 Sulfuric acid, p.a. grade.

5.6 L-ascorbic acid, $C_6H_8O_6$.

5.7 Stock solutions.

Stock solutions shall be prepared by convenient handling moisture-free high-purity salts, dried until a constant mass and cooled in a desiccator. High-purity metals of minimum purity grade of 99,9 % (mass fraction) can also be used to prepare vanadium, titanium, copper, nickel and chromium stock solutions. Stock solutions may also be prepared by independent laboratories or reagent suppliers.

5.7.1 Phosphorus, 1 000 µg/ml.

Dissolve 4,393 6 g of potassium dihydrogen orthophosphate (KH_2PO_4) in about 200 ml of water in a 1 000 ml one-mark volumetric flask. When the dissolution is complete, dilute to volume with water and mix.

5.7.2 Vanadium, 1 000 µg/ml.

Dissolve 1,000 0 g of high-purity vanadium metal or 2,296 3 g of ammonium metavanadate (NH_4VO_3) in 20 ml of nitric acid (5.3) in a covered tall-form beaker with heating. When dissolution is complete, cool and transfer to a 1 000 ml one mark-volumetric flask, dilute to volume with water and mix.

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5.7.3 Titanium, 1 000 µg/ml.

Dissolve 1,000 0 g of high-purity titanium metal in 100 ml of hydrochloric acid (5.2) in a covered tall-form beaker with heating or 4,135 1 g of high-purity ammonium hexafluorotitanate [$(NH_4)_2TiF_6$] in warm distilled water containing drops of hydrofluoridic acid (5.4) in a covered tall-form PTFE beaker. When dissolution is complete, cool and transfer to a 1 000 ml one-mark volumetric flask, dilute to volume with water and mix.

5.7.4 Copper, 1 000 µg/ml.

Dissolve 1,000 0 g of high-purity copper metal in 20 ml of nitric acid (5.3) in a covered tall-form beaker. When dissolution is complete, add about 20 ml of water and heat to liberate oxides of nitrogen. Cool and transfer to a 1 000 ml one-mark volumetric flask, dilute to volume with water and mix.

5.7.5 Nickel, 1 000 µg/ml.

Dissolve 1,000 0 g of high-purity nickel metal in 20 ml of nitric acid (5.3) in a covered tall-form beaker with heating. When dissolution is complete, cool and transfer to a 1 000 ml one-mark volumetric flask, dilute to volume with water and mix.

5.7.6 Chromium, 1 000 µg/ml.

Dissolve 1,000 0 g of high-purity chromium metal in 100 ml of nitric acid (5.3) or 7,695 8 g of chromium (III) nitrate nonahydrate [$Cr(NO_3)_3 \cdot 9H_2O$] in 20 ml of nitric acid (5.3) in a covered tall-form beaker with heating. When dissolution is complete, cool and transfer to a 1 000 ml one-mark volumetric flask, dilute to volume with water and mix.

5.7.7 Barium, 1 000 µg/ml.

Dissolve 1,437 0 g of high-purity barium carbonate ($BaCO_3$) in 20 ml of nitric acid (5.3) in a covered tall-form beaker with heating. When dissolution is complete, cool and transfer to a 1 000 ml one-mark volumetric flask, dilute to volume with water and mix.

5.7.8 Cobalt, 1 000 µg/ml.

Dissolve 2,018 2 g of cobalt carbonate ($CoCO_3$) in 20 ml of nitric acid (5.3) in a covered tall-form beaker with heating. When dissolution is complete, cool and transfer to a 1 000 ml one-mark volumetric flask, dilute to volume water and mix.

5.8 Calibration and reference solutions.

Calibration solutions are defined as the solutions required for the determination of the line peak and background position(s) and for plotting the calibration graphs of the elements analysed. Their concentration ranges in solution, expressed in micrograms per millilitre, are determined with reference to the performance parameter values and the linearity response of the instrument. A minimum of five calibration solutions is necessary to cover the concentration ranges given in Table 1. For test samples having narrower concentration ranges, calibration solutions are prepared to cover the region of interest.

In the case of nonlinearity, either a less sensitive line shall be used or appropriate dilutions of sample and calibration solutions shall be carried out.

NOTE For the suggested lines shown in Table 2, the calibration solutions prepared as recommended in Annex A will be in agreement with the performance test figures.

To comply with the requirements of similarity between the test sample and the calibration solutions, iron and acids shall be added (see Note in Table A.1). For each calibration solution, the procedure described in 8.4.1 shall be followed, replacing the test sample with a suitable amount of high-purity iron (5.1).

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In addition, to comply with the requirements of similarity, calibration solutions and test samples shall be prepared from reagents taken from the same containers, to minimize purity differences between batches.

6 Apparatus

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

6.1 Analytical balance, capable of weighing to the nearest 0,000 1 g.

6.2 Platinum or suitable platinum alloy crucibles, having a minimum volume of 30 ml.

6.3 Muffle furnace, to provide a minimum temperature of 1 020 °C.

6.4 ICP spectrometer.

Any conventional ICP spectrometer, with radial or axial observation path may be used, provided that the instrument has been initially set up according to the manufacturer's recommendations and that it complies with the performance test (see 8.4.2.2), carried out prior to the measurements.

Suggested analytical lines are shown in Table 2. These lines are found to be relatively free of significant interferences from the matrix elements, but they shall be carefully checked for spectral interference, background and ionization, with appropriate corrections carried out prior to their adoption. Failure to attain the recommended performance parameters may indicate interference.

For the analysis of samples having concentrations in the background equivalent concentration (C_{BE}) region or lower, as defined in Table 3, careful assessment of the need for background correction for the particular line chosen is recommended prior to calibration and analysis.

Table 2 — Suggested analytical lines

Element	Wavelength, nm
P	178,290
V	311,071/309,311/310,230
Ti	334,941/336,120
Cu	327,396/224,700/324,75
Ni	231,604
Cr	267,716
Ba	455,405
Co	228,616/238,892

7 Sampling and samples

7.1 Laboratory sample

For analysis, use a laboratory sample of minus 100 µm 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.

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7.2 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.)

For ores having significant content of combined water or oxidizable compounds, an air-equilibrated test sample shall be prepared in accordance with ISO 2596 to determine the dry sample mass using the hygroscopic moisture.

8 Procedure

8.1 Number of determinations

Carry out the analysis at least in duplicate in accordance with Annex C, 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 the 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.

8.2 Test portion

Taking several increments, weigh, to the nearest 0,000 1 g, approximately 1,0 g of the predried test sample obtained in accordance with 7.2.

The test portion should be taken and weighed quickly to avoid re-adsorption of moisture.

8.3 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 7.2.

For the blank test, a suitable amount of high-purity iron (5.1) shall be used in place of the test sample.

The certified reference material shall be of the same type as the sample to be analysed and the properties of the two materials shall be sufficiently similar to ensure that in either case no significant changes in the analytical procedure become necessary. Where a certified reference material is not available, a reference material may be used (see 9.4.4).

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.

8.4 Determination

8.4.1 Decomposition of the test portion

Transfer the weighed test portion to a 250 ml capacity PTFE Erlenmeyer flask with cover.

Add 0,5 g of L-ascorbic acid (5.6), moisten with water, 5,0 ml of hydrofluoric acid (5.4) and 30 ml of concentrated hydrochloric acid (5.2) and take to a hotplate for 2 h. (The hotplate temperature shall be controlled between 120 °C and 140 °C; if necessary use a thermometer inserted in a beaker containing sulfuric acid.)

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