
**Iron ores — Determination of
aluminium —**

Part 1:
**Flame atomic absorption spectrometric
method**

iTeh STANDARD PREVIEW
Minerais de fer — Dosage de l'aluminium —

(standards.iteh.ai)
*Partie 1: Méthode par spectrométrie d'absorption atomique dans la
flamme*

ISO 4688-1:2006

<https://standards.iteh.ai/catalog/standards/sist/27f84a64-de16-43af-9908-10126e278056/iso-4688-1-2006>



PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 4688-1:2006

<https://standards.iteh.ai/catalog/standards/sist/27f84a64-de16-43af-9908-10126e278056/iso-4688-1-2006>

© ISO 2006

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

Foreword.....	iv
1 Scope	1
2 Normative references	1
3 Principle	1
4 Reagents	2
5 Apparatus	2
6 Sampling and samples	3
6.1 Laboratory sample	3
6.2 Preparation of predried test samples	3
7 Procedure	3
7.1 Number of determinations	3
7.2 Test portion	4
7.3 Blank test and check test	4
7.4 Determination	4
8 Expression of results	6
8.1 Calculation of mass fraction of aluminium	6
8.2 General treatment of results	6
8.3 Oxide factor	8
9 Test report	8
Annex A (normative) Flowsheet of the procedure for the acceptance of analytical values for test samples.....	9
Annex B (informative) Derivation of repeatability and permissible tolerance equations	10
Annex C (informative) Precision data obtained by international analytical trials	11

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 4688-1 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 4688-1:1992), which has been technically revised. It has been updated to alter the manner in which precision data are presented.

ISO 4688 consists of the following parts, under the general title *Iron ores — Determination of aluminium* :

- *Part 1: Flame atomic absorption spectrometric method*

Iron ores — Determination of aluminium —

Part 1: Flame atomic absorption spectrometric method

WARNING — This part of ISO 4688 may involve hazardous materials, operations and equipment. This part of ISO 4688 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 4688 to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This part of ISO 4688 specifies a flame atomic absorption spectrometric method for the determination of the mass fraction of aluminium in iron ores.

This method is applicable to mass fractions of aluminium between 0,1 % and 5,0 % in natural iron ores, iron ore concentrates and agglomerates, including sinter products.

(standards.iteh.ai)

2 Normative references

ISO 4688-1:2006

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

3 Principle

The test portion is decomposed by treatment with hydrochloric acid and a small amount of nitric acid.

The mixture is evaporated to dehydrate silica, followed by dilution and filtration.

The residue is ignited and silica is removed by evaporation with hydrofluoric and sulfuric acids. The residue is then fused with sodium carbonate and the cooled melt is dissolved in the filtrate.

The solution obtained is aspirated into the flame of an atomic absorption spectrometer using a dinitrogen oxide/acetylene burner.

The absorbance values obtained for aluminium are compared with those obtained from the calibration solutions.

4 Reagents

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

- 4.1 **Sodium carbonate** (Na_2CO_3), anhydrous.
- 4.2 **Hydrochloric acid**, ρ 1,19 g/ml.
- 4.3 **Nitric acid**, ρ 1,4 g/ml.
- 4.4 **Hydrochloric acid**, ρ 1,19 g/ml, diluted 1 + 9.
- 4.5 **Hydrofluoric acid**, ρ 1,13 g/ml, 40 % (m/m), or ρ 1,185 g/ml, 48 % (m/m).
- 4.6 **Sulfuric acid**, ρ 1,84 g/ml, diluted 1 + 1.
- 4.7 **Background solution**

Dissolve 10 g of high purity iron [minimum purity 99,9 % (mass fraction)] of mass fraction of aluminium less than 0,002 %, in 50 ml of hydrochloric acid (4.2) and oxidize by adding nitric acid (4.3) drop by drop.

Evaporate until a syrupy consistence is obtained. Add 20 ml of hydrochloric acid (4.2) and dilute to 200 ml with water. Dissolve 17 g of sodium carbonate (4.1) in water and add it to the iron solution. Transfer the solution to a 1 000 ml one-mark volumetric flask and dilute to volume with water.

- 4.8 **Aluminium standard solution**, 500 $\mu\text{g Al/ml}$.

Dissolve 0,5 000 g of high purity aluminium [minimum purity 99,9 % (mass fraction)] in 25 ml of hydrochloric acid (4.2). Cool, transfer to a 1 000 ml one-mark volumetric flask, dilute to volume with water and mix.

- 4.9 **Aluminium calibration solutions**

Transfer 2,0 ml; 5,0 ml; 10,0 ml; 20,0 ml; 40,0 ml; and 50,0 ml portions of aluminium standard solution (4.8) to 200 ml volumetric flasks. Dilute to about 100 ml. Add 6 ml of hydrochloric acid (4.2) and 60 ml of background solution (4.7) to each flask. Prepare a zero aluminium calibration solution by transferring 60 ml of the background solution to a 200 ml volumetric flask, and add 6 ml of hydrochloric acid (4.2). Dilute all the solutions to 200 ml with water and mix. (For an atomic absorption spectrometer having high sensitivity, smaller portions of the standard solution may be used.)

5 Apparatus

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

- 5.1 **Platinum crucible**, of capacity 30 ml.
- 5.2 **Muffle furnace**, capable of maintaining a temperature of approximately 1 100 °C.
- 5.3 **Atomic absorption spectrometer**, equipped with a dinitrogen oxide/acetylene burner.

WARNING — Follow the manufacturer's instructions for igniting and extinguishing the dinitrogen oxide/acetylene flame to avoid possible explosion hazards. Wear tinted safety glasses whenever the flame is burning.

The atomic absorption spectrometer used in this method shall meet the following criteria.

- a) Minimum sensitivity: the absorbance of the most concentrated aluminium calibration solution (4.9) shall be at least 0,3.
- b) Graph linearity: the slope of the calibration graph covering the top 20 % of the concentration range (expressed as a change in absorbance) shall not be less than 0,7 of the value of the slope for the bottom 20 % of the concentration range determined in the same way.
- c) Minimum stability: the standard deviation of the absorbance of the most concentrated calibration solution and that of the zero calibration solution, each being calculated from a sufficient number of repetitive measurements, shall be less than 1,5 % and 0,5 %, respectively, of the mean value of the absorbance of the most concentrated calibration solution.

The use of a strip-chart recorder and/or digital readout device is recommended to evaluate criteria a), b) and c) and for all subsequent measurements.

NOTE Instrument parameters may vary with each instrument. The following parameters were successfully used in several laboratories and they can be used as guidelines. Solutions were aspirated into a dinitrogen oxide/acetylene flame of a premix burner.

Aluminium hollow cathode lamp, mA.....	25
Wavelength, nm	396,2
Dinitrogen oxide flow rate, l/min.....	13,8
Acetylene flow rate, l/min	6,6

In systems where the values shown above for gas flow rates do not apply, the ratio of the gas flow rates may still be a useful guideline.

<https://standards.iteh.ai/catalog/standards/sist/27f84a64-de16-43af-9908-10126e278056/iso-4688-1-2006>

6 Sampling and samples

6.1 Laboratory sample

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 way 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, in either case, appropriate recalibration.

7.2 Test portion

Taking several increments, weigh, to the nearest 0,000 2 g, approximately 1 g of the predried test sample obtained in accordance with 6.2.

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

7.3 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 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.

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 will become necessary.

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 that 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

ISO 4688-1:2006

<https://standards.iteh.ai/catalog/standards/sist/27f84a64-de16-43af-9908-10126e278056/iso-4688-1-2006>

7.4.1 Decomposition of the test portion

Transfer the test portion (7.2) to a 250 ml beaker. Moisten with a few millilitres of water, add 25 ml of hydrochloric acid (4.2), cover with a watch-glass and heat gently. Increase the heat and digest just below boiling, until no further attack is apparent. Add 2 ml of nitric acid (4.3) and digest for several minutes. Remove the watch-glass and evaporate the solution to dryness. Heat the salts on the hot-plate at 105 °C to 110 °C for 30 min. Add 5 ml of hydrochloric acid (4.2), cover the beaker with a watch-glass, and warm for several minutes. Add 50 ml of water, heat to boiling, wash the watch-glass and the walls of the beaker, and filter the solution through a medium-texture paper into a 250 ml beaker. Carefully remove all adhering particles with a rubber-tipped rod or moistened filter paper. Wash three times with hydrochloric acid (4.4), then with hot water until the filter paper is free of iron. Transfer the paper and residue to a platinum crucible (5.1). Evaporate the filtrate to about 100 ml and retain it.

7.4.2 Treatment of the residue

Ignite the paper and residue in the platinum crucible (5.1) at a low temperature (500 °C to 800 °C). Cool, moisten with a few drops of water, add 3 or 4 drops of sulfuric acid (4.6) and 10 ml of hydrofluoric acid (4.5), Evaporate slowly to expel silica and then fume to remove the excess sulfuric acid. Ignite at about 700 °C. Add 1,0 g of sodium carbonate (4.1) to the residue (see Note), cover the crucible, and fuse over a burner or in a muffle furnace (5.2) until a clear melt is obtained (at about 1 100 °C for 15 min).

NOTE If difficulties are experienced with the fusion, 2 g of sodium carbonate (4.1) may be used, with a doubled volume of hydrochloric acid (4.2). In this case, prepare the background solution (4.7) with doubled quantities of sodium carbonate and hydrochloric acid.

7.4.3 Preparation of the test solution

Dissolve the cooled melt in the retained filtrate (see 7.4.1), then remove and wash in the crucible and cover (see next paragraph).

If the solution is cloudy at this stage, indicating the presence of substantial amounts of hydrolysed titanium, it should be filtered prior to the transfer to the 200 ml volumetric flask.

Transfer the solution to a 200 ml one-mark volumetric flask, dilute to volume with water and mix. Use this solution (the test solution) directly for the atomic absorption measurements, if the mass fraction of aluminium in the test sample is between 0,1 % and 2,5 %. For mass fractions of aluminium greater than 2,5 %, transfer a 40 ml aliquot to a 200 ml one-mark volumetric flask (see next paragraph), add 50 ml of background solution (4.7) and 4 ml of hydrochloric acid (4.2). Dilute to volume with water and mix. (This solution is the diluted test solution).

For instruments having high sensitivity, smaller portions of the test solution may be preferable. In this case, the amounts of background solutions (4.7) and hydrochloric acid (4.2) should be adjusted.

Transfer a 40 ml aliquot of blank test solution to a 200 ml volumetric flask. Add 50 ml of background solution (4.7) and 4 ml of hydrochloric acid (4.2). Dilute to volume with water and mix. (This solution is the diluted blank test solution.) (See next paragraph.)

The test solution should be measured together with the blank test solution, and the diluted test solution with the diluted blank test solution.

7.4.4 Adjustment of the atomic absorption spectrometer

Set the wavelength for aluminium (396,2 nm) to obtain minimum absorbance. Fit the correct burner for dinitrogen oxide and, in accordance with the manufacturer's instructions, light the flame. After 10 min of preheating the burner, adjust the fuel flow and burner to obtain maximum absorbance while aspirating the calibration solution of highest mass fraction of aluminium (4.9). Then evaluate the criteria in 5.3.

Aspirate water and the calibration solution of highest mass fraction of aluminium, to establish that the absorbance reading is not drifting, and then set the reading for water to zero absorbance.

7.4.5 Atomic absorption measurements

Aspirate the aluminium calibration solutions (4.9) and test solutions (see 7.4.3) in order of increasing absorption, starting with the zero calibration solution and the blank test or diluted blank test solutions. When a stable response is obtained for each solution, record the readings. Aspirate the test solutions or diluted test solutions at the appropriate points in the calibration series and record the readings. Aspirate water between each calibration solution and test solution.

Repeat the measurements at least twice.

If necessary, convert the average of the readings for each calibration solution to absorbance. Obtain the net absorbance of each calibration solution by subtracting the average absorbance of the zero calibration solution. In a similar manner, obtain the net absorbance of the test solution, or that of the diluted test solution, by subtracting the absorbance of the blank test solution, or that of the diluted blank test solution, respectively.

Prepare a calibration graph by plotting the net absorbance values of the calibration solutions against the mass concentration of aluminium, in micrograms per millilitre. (The test solution, or in the case of a dilution, the diluted test solution, is the final test solution.)

Convert the net absorbance value of the final test solution to micrograms of aluminium per millilitre by means of the calibration graph.