



SLOVENSKI STANDARD
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Železne rude -- Določitev vsebnosti aluminija -- Del 1: Metodna postopek absorpcijske atomske absorpcije v plamni

Iron ores -- Determination of aluminium content -- Part 1: Flame atomic absorption spectrometric method

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Minerais de fer -- Dosage de l'aluminium -- Partie 1: Méthode par spectrométrie d'absorption atomique dans la flamme

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INTERNATIONAL STANDARD

ISO
4688-1

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Iron ores — Determination of aluminium content —

Part 1:

**Flame atomic absorption spectrometric method
(standards.iteh.ai)**

Minerais de fer — Dosage de l'aluminium —

*Partie 1: Méthode par spectrométrie d'absorption atomique dans la
flamme*



Reference number
ISO 4688-1:1992(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.

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.

International Standard ISO 4688-1 was prepared by Technical Committee ISO/TC 102, *Iron ores*, Sub-Committee SC 2, *Chemical analysis*.

This part of ISO 4688 cancels and replaces ISO 4688:1990, of which it constitutes a technical revision.

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

— *Part 1: Flame atomic absorption spectrometric method*

Additional parts will be published later.

Annex A forms an integral part of this part of ISO 4688. Annexes B and C are for information only.

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Iron ores — Determination of aluminium content —

Part 1:

Flame atomic absorption spectrometric method

1 Scope

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

This method is applicable to aluminium contents between 0,1 % (*m/m*) and 5,0 % (*m/m*) in natural iron ores, iron ore concentrates and agglomerates, including sinter products.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 4688. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 4688 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 648:1977, *Laboratory glassware — One-mark pipettes*.

ISO 1042:1983, *Laboratory glassware — One-mark volumetric flasks*.

ISO 3081:1986, *Iron ores — Increment sampling — Manual method*.

ISO 3082:1987, *Iron ores — Increment sampling and sample preparation — Mechanical method*.

ISO 3083:1986, *Iron ores — Preparation of samples — Manual method*.

ISO 7764:1985, *Iron ores — Preparation of predried test samples for chemical analysis*.

3 Principle

Decomposition of the test portion by treatment with hydrochloric acid and a small amount of nitric acid.

Evaporation to dehydrate silica, followed by dilution and filtration.

Ignition of the residue and removal of silica by evaporation with hydrofluoric and sulfuric acids. Fusion with sodium carbonate and dissolution of the cooled melt in the filtrate.

Aspiration of the solution obtained into the flame of an atomic absorption spectrometer using a dinitrogen oxide-acetylene burner.

Comparison of the absorbance values obtained for aluminium 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.

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4.7 Background solution.

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

Evaporate until a syrupy consistency 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 µg Al/ml.

Dissolve 0,500 0 g of high purity aluminium [minimum purity 99,9 % (*m/m*)] 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

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.

NOTES

1 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.

2 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.

6 Sampling and samples**6.1 Laboratory sample**

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 having significant contents of combined water or oxidizable compounds, use a particle size of minus 160 µm.

NOTE 3 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 4 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.

NOTE 5 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 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 6 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.

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 that the reagents used 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.

7.4 Determination

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 7), 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 7 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 the crucible and cover (see note 8).

NOTE 8 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 aluminium content of the test sample is between 0,1 % (m/m) and 2,5 % (m/m). For aluminium contents greater than 2,5 % (m/m), transfer a 40 ml aliquot to a 200 ml one-mark volumetric flask (see note 9), 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.)

NOTE 9 For instruments having high sensitivity, smaller portions of the test solution may be preferable. In this

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case, the amounts of background solution (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 note 10.)

NOTE 10 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 aluminium content (4.9). Then evaluate the criteria in 5.3.

Aspirate water and the calibration solution of highest aluminium content 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 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 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.

8 Expression of results

8.1 Calculation of aluminium content

The aluminium content, w_{Al} , as a percentage by mass, is calculated to four decimal places using the equation

$$w_{\text{Al}} = \frac{\rho_{\text{Al}} \times 200}{m \times 10\,000} = \frac{\rho_{\text{Al}}}{m \times 50} \quad \dots (1)$$

where

ρ_{Al} is the concentration, in micrograms per millilitre, of aluminium of the final test solution;

m is the mass, in grams, of sample contained in 200 ml of the final test solution.

8.2 General treatment of results

8.2.1 Repeatability and permissible tolerance

The precision of this analytical method is expressed by the following regression equations¹⁾:

$$r = 0,029\,0\,X + 0,023\,0 \quad \dots (2)$$

$$P = 0,054\,1\,X + 0,070\,3 \quad \dots (3)$$

$$\sigma_r = 0,010\,2\,X + 0,008\,1 \quad \dots (4)$$

$$\sigma_L = 0,017\,7\,X + 0,024\,1 \quad \dots (5)$$

where

X is the aluminium content, expressed as a percentage by mass, of the predried test sample, calculated as follows:

- within-laboratory equations (2) and (4): the arithmetic mean of the duplicate values,
- between-laboratories equations (3) and (5): the arithmetic mean of the final results (8.2.3) of the two laboratories;

r is the permissible tolerance within a laboratory (repeatability);

1) Additional information is given in annexes B and C.

- P is the permissible tolerance between laboratories;
- σ_r is the within-laboratory standard deviation;
- σ_L is the between-laboratories standard deviation.

8.2.2 Acceptance of analytical values

The result obtained for the certified reference material shall be such that the difference between this result and the certified value of the reference material is statistically insignificant. For a reference material that has been analysed by at least 10 laboratories using method(s) that are comparable both in accuracy and in precision with this method, the following conditions shall be used to test the significance of the difference:

$$|A_c - A| \leq 2 \sqrt{\frac{s_{Lc}^2 + \frac{s_{Wc}^2}{n_{Wc}}}{N_c} + \sigma_L^2 + \frac{\sigma_r^2}{n}} \quad \dots (6)$$

$$|A_c - A| \leq 2 \sqrt{2\sigma_L^2 + \frac{\sigma_r^2}{n}} \quad \dots (7)$$

where

- A_c is the certified value;
- A is the result on the mean of results obtained for the certified reference material;
- s_{Lc} is the between-laboratories standard deviation of the certifying laboratories;
- s_{Wc} is the within-laboratory standard deviation of the certifying laboratories;
- n_{Wc} is the average number or replicate determinations in the certifying laboratories;
- N_c is the number of certifying laboratories;
- n is the number of replicate determinations on the reference material (in most cases $n = 1$);
- σ_L and σ_r are as defined in 8.2.1.

If condition (6) is satisfied, i.e. if the left-hand side is less than or equal to the right-hand side, then the difference $|A_c - A|$ is statistically insignificant; otherwise, it is statistically significant.

When the difference is significant, the analysis shall be repeated, simultaneously with an analysis of the test sample. If the difference is again significant, the procedure shall be repeated using a different certified reference material of the same type of ore.

When the range of the two values for the test sample is outside the limit for r calculated according to equation (2) in 8.2.1, one or more additional tests shall be carried out in accordance with the flowsheet presented in annex A, simultaneously with an analysis of a certified reference material of the same type of ore.

Acceptability of the results for the test sample shall in each case be subject to the acceptability of the results for the certified reference material.

NOTE 11 The following procedure should be used when the information on the reference material certificate is incomplete:

- if there are insufficient data to enable the between-laboratories standard deviation to be estimated, delete the expression s_{Wc}^2/n_{Wc} and regard s_{Lc} as the standard deviation of the laboratory means;
- if the certification has been made by only one laboratory or if the interlaboratory results are missing, it is advisable that this material not be used in the application of the standard. In case its use is unavoidable, use the condition

8.2.3 Calculation of final result

The final result is the arithmetic mean of the acceptable analytical values for the test sample, or as otherwise determined by the operations specified in annex A.

The arithmetic mean of the acceptable analytical values, calculated to the fourth decimal place, is rounded off to the second decimal place as follows:

- when the figure in the third decimal place is less than 5, it is discarded and the figure in the second decimal place is kept unchanged;
- when the figure in the third decimal place is 5 and there is a figure other than 0 in the fourth decimal place, or when the figure in the third decimal place is greater than 5, the figure in the second decimal place is increased by one;
- when the figure in the third decimal place is 5 and the figure 0 is in the fourth decimal place, the 5 is discarded and the figure in the second decimal place is kept unchanged if it is 0, 2, 4, 6 or 8 and is increased by one if it is 1, 3, 5, 7 or 9.

8.3 Oxide factor

The oxide factor is given by the following equation:

$$w_{Al_2O_3} (\%) = 1,889\ 5\ w_{Al}$$