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# International Standard



# 4688

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## Iron ores — Determination of aluminium content — Flame atomic absorption spectrometric method

*Minerais de fer — Dosage de l'aluminium — Méthode par spectrométrie d'absorption atomique dans la flamme*

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## Foreword

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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 4688 was developed by Technical Committee ISO/TC 102, *Iron ores*, and was circulated to the member bodies in February 1978.

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

Australia	Iran	<u>ISO 4688:1980</u>
Austria	Italy	Portugal
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Canada	Korea, Dem. P. Rep. of	South Africa, Rep. of
Czechoslovakia	Korea, Rep. of	Sweden
Egypt, Arab Rep. of	Mexico	Turkey
France	Netherlands	United Kingdom
Germany, F. R.	New Zealand	USA
India	Poland	USSR

No member body expressed disapproval of the document.

# Iron ores — Determination of aluminium content — Flame atomic absorption spectrometric method

## 1 Scope and field of application

This International Standard specifies a method for the determination of aluminium in iron ores by flame atomic absorption spectrometry.

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

## 2 References

ISO 2596, *Iron ores — Determination of hygroscopic moisture in analytical samples.*

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

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

## 3 Principle

Decomposition of the test portion by treatment with hydrochloric acid with the addition of 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 in the flame of an atomic absorption apparatus using a dinitrogen oxide burner.

Measurement of the absorbance value obtained for aluminium in comparison with those obtained from the calibration solutions.

## 4 Reagents

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

**4.1 Sodium carbonate** ( $\text{Na}_2\text{CO}_3$ ), anhydrous.

**4.2 Hydrochloric acid**, ( $\rho$  1,19 g/ml).

**4.3 Nitric acid**, ( $\rho$  1,4 g/ml).

**4.4 Hydrochloric acid**, ( $\rho$  1,19 g/ml), diluted 1 + 9.

**4.5 Hydrofluoric acid**, 40 % (*m/m*) ( $\rho$  1,13 g/ml), or 48 % (*m/m*) ( $\rho$  1,185 g/ml).

**4.6 Sulphuric acid**, ( $\rho$  1,84 g/ml), diluted 1 + 1.

**4.7 Background solution.**

Dissolve 10 g of iron of aluminium content less than 0,01 %, in 50 ml of hydrochloric acid (4.2) and oxidize by dropwise addition of nitric acid (4.3).

Evaporate to syrupy consistency. Add 20 ml of hydrochloric acid (4.2) and dilute to 200 ml with water. Add 17 g of sodium carbonate (4.1) dissolved in water and dilute in a volumetric flask to 1 000 ml with water.

**4.8 Aluminium**, standard solution.

Dissolve 0,500 0 g of high-purity aluminium (99,9 %) in 25 ml of hydrochloric acid (4.2). Cool, transfer to a 1 000 ml volumetric flask, dilute to volume with water and mix.

1 ml of this solution contains 0,50 mg of aluminium.

**4.9 Aluminium**, calibration solutions.

Transfer 2,0 — 5,0 — 10,0 — 20,0 — 40,0 and 50,0 ml portions of standard aluminium 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). 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 volumes to 200 ml with water and mix. (For an instrument with high sensitivity smaller portions of the standard solution can be used.)

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## 5 Apparatus

Ordinary laboratory equipment, and

### 5.1 Platinum crucible

### 5.2 Atomic absorption spectrometer

The atomic absorption spectrometer used in this method will be satisfactory if it meets the following criteria :

- a) Minimum sensitivity — the absorbance of the highest calibration solution (see 4.9) shall be at least 0,3.
- b) Curve linearity — the slope of the calibration curve 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 the standard deviation of the absorbance 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 solution.

#### NOTES

- 1 A strip chart recorder and/or digital readout device is advisable to measure the above criteria and for all subsequent measurements.
- 2 Instrument parameters will 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.

— Al 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

For analysis, a laboratory sample of minus 100  $\mu\text{m}$  particle size which has been taken in accordance with ISO 3081<sup>1)</sup> and prepared in accordance with ISO 3083<sup>1)</sup> shall be used. In the

case of ores with high contents of combined water and/or oxidizable compounds, a sample of 160  $\mu\text{m}$  particle size shall be used.

## 7 Procedure

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

NOTE — The expression "independently" implies the change of the person carrying out the analysis. If the same person must carry out the analysis, the procedure shall be carried out at different times.

Simultaneously with the analysis, determine the hygroscopic moisture in accordance with ISO 2596.

### 7.1 Safety precaution

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.

### 7.2 Test portion

Weigh, to the nearest 0,000 2 g, approximately 1 g of the test sample obtained in accordance with clause 6.

### 7.3 Blank test and check test

In each run, one analysis of a certified reference material of the same type of ore and one blank test shall be carried out in parallel with the analysis of the ore sample(s) under the same conditions.

NOTE — The certified reference material should be of the same type as the sample to be analysed. Such certified reference material cannot, however, be considered as being of the same type if the properties of the sample to be analysed differ from those of the certified reference material to such an extent that the analytical procedure must be changed substantially.

When the analysis is carried out on several samples at the same time, the blank value may be represented by one test, providing the procedure is the same and 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.

1) A further International Standard now in preparation (ISO 3082) will specify mechanical methods of increment sampling as well as methods for preparation of samples.

## 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 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 cover 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 paper is free of iron. Transfer the paper and precipitate to a platinum crucible. Evaporate the filtrate to about 100 ml and reserve.

### 7.4.2 Treatment of the residue

Ignite the paper and residue in the platinum crucible at a low temperature (500 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 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 (4.1) and hydrochloric acid (4.2).

Dissolve the cool melt in the reserved filtrate from 7.4.1, remove and wash the crucible and cover.

NOTE — 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 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 and 2,5 % (*m/m*). For aluminium contents greater than 2,5 %, transfer a 40 ml aliquot to a 200 ml volumetric flask (see note below), 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 — For instruments with high sensitivity, smaller portions of the test solution may be preferable. In this 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.)

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

### 7.4.3 Adjustment of atomic absorption spectrometer

Fit the correct burner for nitrogen dioxide and, in accordance with manufacturer's instructions, light the flame. Set the wavelength for aluminium (396,2 nm) to obtain minimum absorbance. After 10 min preheating of the burner, adjust fuel and burner to obtain maximum absorbance while aspirating the highest concentration calibration solution (4.9), then evaluate the criteria in 5.2.

Aspirate water and the highest concentration calibration solution to establish that the reading is not drifting and then set the reading for water to zero absorbance.

### 7.4.4 Atomic absorption measurements

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

Repeat the measurements at least two more times.

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 the calibration curve by plotting the net absorbance values of the calibration solutions against micrograms of aluminium 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 curve.

## 8 Expression of results

### 8.1 Calculation of aluminium content

The aluminium content, as a percentage by mass, is calculated from the following formula :

$$\text{Al (\%)} = \frac{c \times 200}{m \times 10\,000} \times K$$

$$= \frac{c}{m \times 50} \times K$$

where

$c$  is the concentration, in micrograms per millilitre, of aluminium of the final test solution;

$m$  is the mass, in grams, of sample represented in 200 ml volume of the final test solution;

$K$  is the conversion factor calculated from the following formula :

$$K = \frac{100}{100 - A}$$

where  $A$  is the hygroscopic moisture content, as a percentage by mass, determined in accordance with ISO 2596.

## 8.2 General treatment of results

### 8.2.1 Acceptance of analytical values

The analytical values for the test sample shall be accepted when the respective analytical values obtained for the corresponding certified reference material agree with the certified value within the limit of permissible tolerance for certified reference materials, and when the difference between the two values for the test sample does not exceed the permissible tolerance for test samples (8.3).

When the analytical value obtained for the certified reference material is outside the limit of the permissible tolerance (second column in the table in 8.3), an analysis shall be carried out simultaneously on one test sample and one certified reference material, with one blank test. The analytical value obtained for the certified reference material shall be examined for the acceptance of the value for the test sample, as above. If the value obtained for the certified reference material is again outside the limit, the procedure shall be repeated with a different reference material of the same type of ore, until two acceptable values for the test sample are obtained.

When the range (absolute difference) of the two values for the test sample is outside the limit of the permissible tolerance (third column in the table in 8.3), additional analyses shall be carried out on one test sample with one blank test, as required by the procedure specified in annex A, simultaneously with an analysis of a certified reference material of the same type of

ore. Acceptance of such additional values for the test sample shall be subject, in each case, to the acceptability of the value obtained for the certified reference material.

### 8.2.2 Calculation of final result

The arithmetical mean of the acceptable values of the test sample is the final result.

The arithmetical mean of the acceptable analytical values, calculated to the fourth decimal place, is to be 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 there is no figure other than 0 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 Permissible tolerances

The permissible tolerances described in 8.2.1 are shown in the table 1.

Table — Permissible tolerances within laboratories

Aluminium content Al [% (m/m)]	Certified reference material Al [% (m/m)] 1,96 $s_w$	Test sample Al [% (m/m)]	
		2,77 $s_w$	3,31 $s_w$
0,1 to 0,25	± 0,018	0,025	0,030
0,25 to 1,0	± 0,024	0,033	0,040
1,0 to 2,5	± 0,080	0,111	0,134
2,5 to 5,0	± 0,100	0,139	0,167

1) The derivation of the figures in the table is given in annex B.

#### 8.4 Oxide factor

$\text{Al}_2\text{O}_3$  [% (m/m)] = 1,889 5 × Al [% (m/m)].

#### 9 Test report

The test report shall include the following information :

a) reference to this International Standard;

- b) details necessary for the identification of the sample;
- c) results of the analysis;
- d) reference number of the results;
- e) any characteristics noticed during the determination, and any operations not specified in this International Standard which may have had an influence on the results.

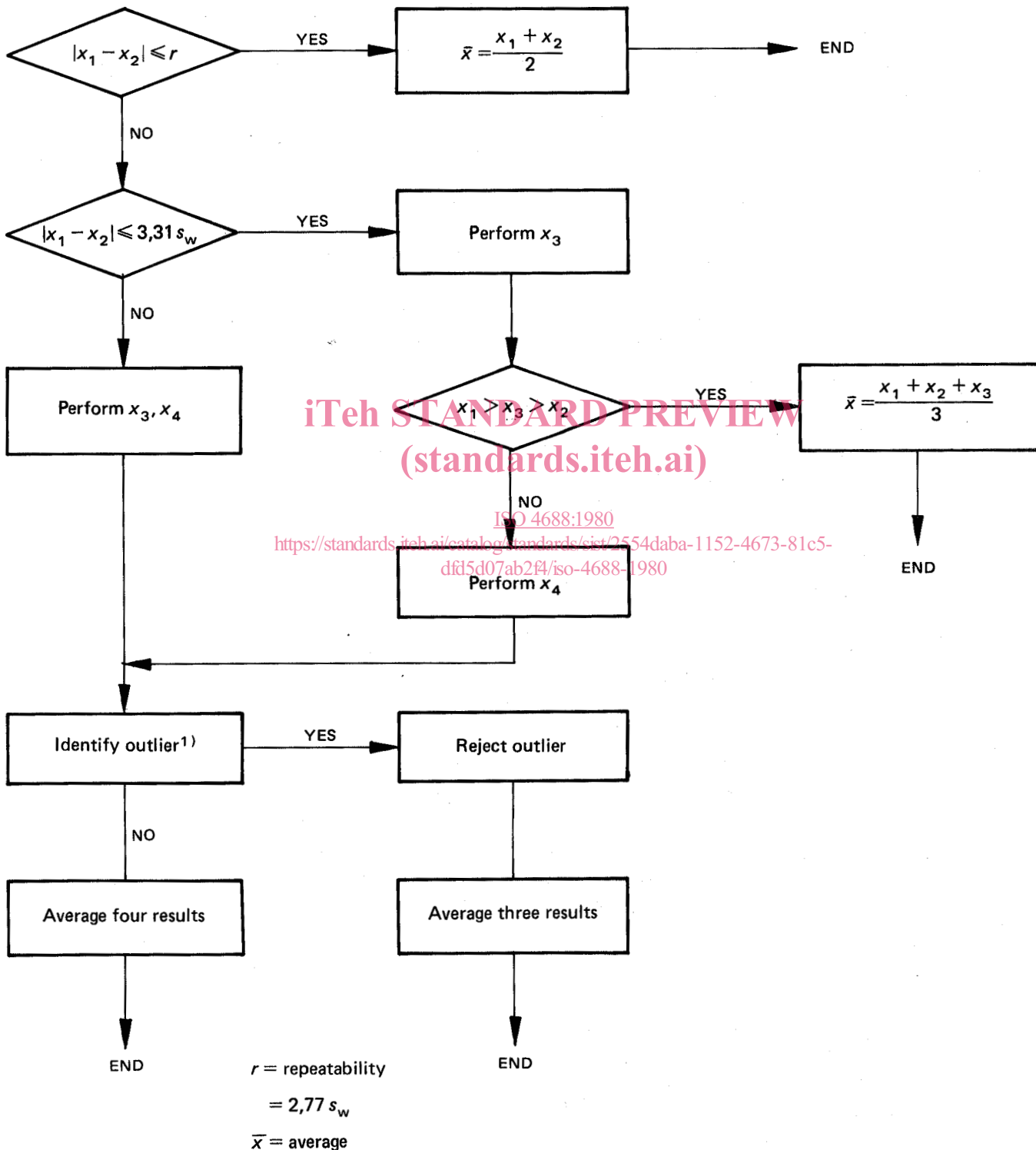
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### Annex A

## Flowsheet on the procedure for the acceptance of analytical values for test samples



1) Use Dixon's criteria for outlier.



## Annex B

### Derivation of permissible tolerances

The standard deviations within laboratories ( $s_w$ ) were derived from a comparison of the results of international analytical trials carried out in 1971-1972 and 1972-1973 on six iron ore samples : Canadian ore (0,178 % Al), Sweden-7 ore (0,284 % Al), CSR ore (Krivoj-Rog) (0,884 % Al), Philippine iron sand (1,46 % Al), Minette ore (2,14 % Al), and 72-8 ore (4,142 % Al). The results of these trials were reported in the documents 102/2N 237E, January 1972 and 102/2N 335F, January 1974.

The permissible tolerances for the certified reference material (second column in the table in 8.3) are calculated by multiplying the standard deviations by the statistical factor 1,96 and relate to the upper limit of each range of contents respectively.

The permissible tolerances for the test sample (third column in the table in 8.3) are calculated by multiplying the standard deviations by the statistical factors 2,77 and 3,31 ( $n = 2$  with 95 % confidence) and relate to the upper limit of each range of contents respectively.

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