
International Standard



4688

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

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

First edition — 1980-02-01

UDC 553.31 : 543.422 : 546.621

Ref. No. ISO 4688-1980 (E)

Descriptors : metalliferous minerals, iron ores, chemical analysis, determination of content, aluminium, atomic absorption method, composition tolerances.



Documents

Document Sharing Hub

Price based on 7 pages

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (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 set up 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 4688 was developed by Technical Committee ISO/TC 102, *Iron ores*, and was circulated to the member bodies in February 1978.

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

Australia	Iran	Portugal
Austria	Italy	Romania
Bulgaria	Japan	South Africa, Rep. of
Canada	Korea, Dem. P. Rep. of	Sweden
Czechoslovakia	Korea, Rep. of	Turkey
Egypt, Arab Rep. of	Mexico	United Kingdom
France	Netherlands	USA
Germany, F. R.	New Zealand	USSR
India	Poland	

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

4.6 Sulphuric acid, (ρ 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.)

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 µm particle size which has been taken in accordance with ISO 3081¹⁾ and prepared in accordance with ISO 3083¹⁾ shall be used. In the

case of ores with high contents of combined water and/or oxidizable compounds, a sample of 160 µm 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.