INTERNATIONAL STANDARD

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Manganese ores and concentrates — Determination of iron content — Flame atomic absorption spectrometric method

Minerais et concentrés de manganèse — Dosage du fer — Méthode par spectrométrie d'absorption atomique dans la flamme

Reference number
ISO 9681:1990(E)

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 9681 was prepared by Technical Committee ISO/TC 65, *Manganese and chromium ores*.

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Manganese ores and concentrates — Determination of iron content — Flame atomic absorption spectrometric method

1 Scope

This International Standard specifies a flame atomic absorption spectrometric method for the determination of iron content in manganese ores and concentrates. The method is applicable to products having an iron content from 0,2 % (*m/m*) to 10 % (*m/m*).

This International Standard should be read in conjunction with ISO 4297.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard 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 310:1981, *Manganese ores — Determination of hygroscopic moisture content in analytical samples — Gravimetric method.*

ISO 4296-1:1984, *Manganese ores — Sampling — Part 1: Increment sampling.*

ISO 4296-2:1983, *Manganese ores — Sampling — Part 2: Preparation of samples.*

ISO 4297:1978, *Manganese ores and concentrates — Methods of chemical analysis — General instructions.*

3 Principle

Method 1: Decomposition of the test portion by treatment with hydrochloric, perchloric and hydrofluoric acids. Separation of the insoluble residue, fusion of the residue with a fusion mixture and dissolution of the cooled melt in the test solution. Aspiration of the test solution into an air-acetylene flame in an atomic absorption spectrometer and measurement of the absorbance at a wavelength of 248,3 nm or 344,06 nm.

Method 2: Decomposition of the test portion by treatment with hydrochloric, nitric and perchloric acids and filtration of the insoluble residue. Removal of silica by volatilization with sulfuric and hydrofluoric acids, fusion of the residue with a fusion mixture and dissolution of the cooled melt in the test solution. Aspiration of the test solution into an air-acetylene flame in an atomic absorption spectrometer and measurement of the absorbance at a wavelength of 248,3 nm or 344,06 nm.

4 Reagents

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

4.1 Metallic manganese, 99,9 % purity.

4.2 Hydrochloric acid, ρ 1,19 g/ml.

4.3 Hydrochloric acid, ρ 1,19 g/ml, diluted 1 + 50.

4.4 Hydrofluoric acid, ρ 1,14 g/ml.

4.5 Perchloric acid, ρ 1,61 g/ml.

4.6 Nitric acid, ρ 1,40 g/ml.

4.7 Nitric acid, ρ 1,40 g/ml, diluted 1 + 1.

4.8 Sulfuric acid, ρ 1,84 g/ml, diluted 1 + 1.

Slowly and with great care, pour 1 volume of concentrated sulfuric acid into an equal volume of water.

4.9 Hydrogen peroxide, 30 % (*m*/*m*) solution.

4.10 Fusion mixture.

Mix anhydrous sodium carbonate and anhydrous sodium tetraborate, in the proportion 3 + 1.

4.11 Background solution A, for ores having CaO content less than 5 % (*m*/*m*).

Dissolve 5 g of manganese (4.1) in 40 ml of nitric acid (4.7). Add 30 ml of perchloric acid (4.5). Heat until fumes of perchloric acid appear. Cool the solution and dilute with water. Add 7,5 g of anhydrous sodium carbonate, 2,5 g of anhydrous sodium tetraborate and 30 ml of hydrochloric acid (4.2). After dissolution, transfer the solution to a 500 ml one-mark volumetric flask, dilute to the mark with water and mix.

4.12 Background solution B, for ores having CaO content greater than 5 % (*m*/*m*).

Dissolve 4 g of manganese (4.1) and 1,8 g of calcium carbonate in 40 ml of nitric acid (4.7) and proceed as in 4.11.

4.13 Iron, standard solutions.

4.13.1 Iron, standard solution A corresponding to 4 g of Fe per litre.

Dissolve 4,000 g of carbonyl iron (purity 99,99 %) in 40 ml of nitric acid (4.6). Add 40 ml of perchloric acid (4.5) and evaporate until fumes of perchloric acid appear. Cool the solution and dilute with water. Transfer the solution to a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution contains 4 mg of Fe.

4.13.2 Iron, standard solution B corresponding to 0,1 g of Fe per litre.

Transfer 5 ml of iron standard solution A (4.13.1) to a 200 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution contains 0,1 mg of Fe.

5 Apparatus

Ordinary laboratory equipment, and

5.1 Platinum crucibles.

5.2 Polytetrafluoroethylene (PTFE) beakers.

5.3 Atomic absorption spectrometer, equipped with an air-acetylene burner.

The atomic absorption spectrometer used will be satisfactory if it meets the following criteria.

a) *minimum sensitivity*: the absorbance of the calibration solution of highest iron content (7.4) shall be at least 0,25;

b) *curve linearity*: the slope of the calibration graph covering the top 20 % of the concentration range (expressed as a change in absorbance) shall be not 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 calibration solution of highest concentration 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 calibration solution of highest concentration.

NOTES

1 The use of a strip chart recorder and/or digital readout device is recommended to evaluate the criteria 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.

Iron hollow cathode lamp: 20 mA
Wavelength: 248,3 nm or 344,06 nm
Air flow rate: 13,3 l/min
Acetylene flow rate: 1,7 l/min.

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

6 Sampling and samples

For analysis, use a laboratory sample of minus 100 µm particle size which has been taken in accordance with ISO 4296-1 and prepared in accordance with ISO 4296-2.

7 Procedure

7.1 Test portion

Weigh 1 g of an air-dried sample or the sample dried at 105 °C to 110 °C.

NOTE 3 When using the sample dried at 105 °C to 110 °C, the test portion should be taken and weighed quickly in order to avoid reabsorption of moisture.