INTERNATIONAL STANDARD

ISO 9292

First edition
1988-12-01

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

Manganese ores and concentrates — Determination of total iron content — 1,10-Phenanthroline spectrometric method

Minerais et concentrés de manganèse — Dosage du fer total — Méthode spectrométrique à la phénanthroline-1,10

Reference number
ISO 9292 : 1988 (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 approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 9292 was prepared by Technical Committee ISO/TC 65, *Manganese and chromium ores*.

Printed in Switzerland

Manganese ores and concentrates — Determination of total iron content — 1,10-Phenanthroline spectrometric method

1 Scope

This International Standard specifies a spectrometric method for the determination of total iron content in manganese ores and concentrates using 1,10-phenanthroline. The method is applicable to products having a total iron content from 0,1 % (m/m) to 15 % (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 listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

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

Decomposition of a test portion by treatment with hydrochloric acid. Filtration of the insoluble residue and reservation of the filtrate as the main solution. Ignition of the filter containing the residue and treatment with sulfuric and hydrofluoric acids.

Fusion of the ignited residue with potassium pyrosulfate. Dissolution of the melt in the main solution. Formation of the complex of iron with 1,10-phenanthroline and spectrometric measurement.

4 Reactions

The method is based on the formation of the coloured complex of iron(II) with 1,10-phenanthroline (pH 4 to 5) after reduction of the iron(III) to iron(II) with hydroxylammonium chloride.

5 Reagents

5.1 Hydrochloric acid, ϱ 1,19 g/ml.

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

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

5.4 Hydrofluoric acid, ϱ 1,14 g/ml, 40 % (m/m) solution.

5.5 Potassium pyrosulfate ($K_2S_2O_7$) or potassium hydrogen sulfate ($KHSO_4$).

5.6 Hydroxylammonium chloride, 10 % (m/m) solution.

5.7 Buffer solution.

Dissolve 450 g of sodium acetate in 500 ml of water, add 240 ml of glacial acetic acid, dilute to 1 000 ml with water and mix.

5.8 1,10-phenanthroline, solution $c(C_{12}H_8N_2 \cdot H_2O)$ = 5 g/l.

Dissolve 5 g of 1,10-phenanthroline ($C_{12}H_8N_2 \cdot H_2O$) in 100 ml of ethanol, and dilute to 1 000 ml with water. Alternatively dissolve 6 g of 1,10-phenanthroline hydrochloride ($C_{12}H_8N_2 \cdot HCl \cdot H_2O$) in 1 000 ml of water.

5.9 Iron, standard solution.

Solution A.

Place 0,500 0 g of metallic iron (purity 99,95 %) in a 300 ml beaker, add 100 ml of sulfuric acid (ϱ 1,84 g/ml diluted 1 + 4), cover the beaker with a watch glass and heat gently to dissolve completely. Cool the solution, add 10 ml of hydrogen peroxide solution [30 % (m/m) diluted 1 + 9] drop by drop and heat gently to boil and decompose the excess of hydrogen peroxide. After cooling, transfer the solution to a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of solution A contains 0,5 mg of iron.

Solution B.

Transfer 50 ml of solution A to a 500 ml one-mark volumetric flask, add 25 ml of hydrochloric acid (5.1), cool, dilute to the mark with water and mix.

1 ml of solution B contains 0,05 mg of iron.

6 Apparatus

Ordinary laboratory equipment, and

6.1 Crucible, of platinum or platinum alloy.

6.2 Spectrometer or **photoelectrocolorimeter.**

7 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.

8 Procedure

8.1 Test portion

Taking several increments, weigh the mass of test portion chosen from table 1 in accordance with the expected total iron content.

Table 1

Expected total Fe content % (m/m)	Mass of test portion g
Form 0,1 to 2	1,0
From 2 to 5	0,4

8.2 Blank test

Carry out a blank test in parallel with the analysis of the ore sample(s) under the same conditions.

8.3 Determination

8.3.1 Decomposition of test portion

Place the test portion (8.1) in a 300 ml beaker, moisten with water, and add 30 ml of hydrochloric acid (5.1). Cover the beaker with a watch glass and heat gently to decompose the sample. Rinse the watch glass and dilute the solution with warm water to about 50 ml.

Filter off the insoluble residue on a medium texture filter paper with a small quantity of paper pulp. Wash the filter with the residue 4 to 5 times with hot hydrochloric acid (5.2) and 4 to 5 times with hot water. Collect the filtrate and washings in a 300 ml beaker and reserve as the main solution.

8.3.2 Treatment of residue

Transfer the filter containing the residue to a platinum crucible (6.1), dry the paper and residue and ignite at 500 °C to 600 °C. Allow the crucible to cool, moisten the residue with a few drops of water, add 3 to 4 drops of sulfuric acid (5.3), then add 5 ml to 6 ml of hydrofluoric acid (5.4) and evaporate to dryness. Cool the crucible, add 2 g of potassium pyrosulfate or potassium hydrogen sulfate (5.5) to the residue and fuse at 600 °C to 650 °C until a clear melt is obtained.

Allow the crucible to cool, leach the melt in the main solution (8.3.1) while heating, then remove the crucible, rinsing it with water. Cool the solution and transfer to a 250 ml one-mark volumetric flask, dilute to the mark with water and mix.

8.3.3 Preparation of solution for spectrometric measurement

For ores with an iron content up to 0,8 % (m/m) take a 10 ml aliquot from the solution (8.3.2); for ores with an iron content greater than 0,8 % (m/m) take a 5 ml aliquot of solution (8.3.2). Transfer the aliquot to a 100 ml one-mark volumetric flask and add 50 ml of water.

Add 5 ml of hydroxylammonium chloride solution (5.6), mix, allow to stand for 5 min, then add 10 ml of buffer solution (5.7) and 10 ml of 1,10-phenanthroline solution (5.8). Mix and allow to stand for 1 h at room temperature or for 15 min at 30 °C. Cool the solution, dilute to the mark with water and mix.

8.3.4 Spectrometric measurement

Measure the absorbance of the test solution (8.3.3) using the spectrometer (6.2) set to maximum absorbance (at the wavelength 508 nm to 512 nm) or the photoelectrocolorimeter (6.2) fitted with a light filter (at the wavelength 480 nm to 520 nm) against water as reference.

NOTE — In the case of measurement with a mercury vapour lamp, measure the absorbance at 546 nm.

8.3.5 Preparation of calibration graph

Transfer into six 100 ml one-mark volumetric flasks, using a burette, 0,0; 1,0; 2,0; 4,0; 6,0 and 8,0 ml of standard iron solution B (5.9) corresponding to 0,0; 0,05; 0,10; 0,20; 0,30 and 0,40 mg of iron. Add to each flask 50 ml of water and 5 ml of hydroxylammonium chloride solution (5.6), mix, allow to stand for 5 min, then add 10 ml of buffer solution (5.7) and 10 ml of 1,10-phenanthroline solution (5.8). Mix and allow to stand for 1 h at room temperature or for 15 min at 30 °C. Cool the solution, dilute to the mark with water and mix. Carry out the spectrometric measurement as specified in 8.3.4.

Prepare a calibration graph by plotting absorbance values (subtracting the absorbance value of zero calibration solution) against the nominal iron contents of the solutions.

9 Expression of results

9.1 Calculation

Convert the absorbance reading for the test solution to total iron content by means of the calibration graph (8.3.5), subtracting the absorbance reading for the blank test.

The total iron content, w_{Fe}, expressed as a percentage by mass, is given by the formula

$$w_{Fe} = \frac{m_1 \times 100}{m_0} \times K$$

where

m_0 is the mass, in grams, of the test portion, corresponding to the aliquot portion of the test solution;

m_1 is the mass, in grams, of iron in the aliquot portion of the test solution, obtained from the calibration graph;

K is the conversion factor for the expression of the total iron content on the dry basis.

9.2 Permissible tolerances on results of parallel determinations

The permissible tolerances on results of parallel determinations are given in table 2.

Table 2

Total iron content % (*m*/*m*)	Permissible tolerance	
	Two parallel determinations % (*m*/*m*)	Three parallel determinations % (*m*/*m*)
From 0,1 to 0,2	0,02	0,03
From 0,2 to 0,4	0,03	0,04
From 0,4 to 1,0	0,06	0,07
From 1,0 to 2,5	0,09	0,10
From 2,5 to 5,0	0,12	0,15

This page intentionally left blank