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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

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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 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*.

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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 6,6 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.