
International Standard



4294

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Manganese ores and concentrates — Determination of copper content — Extraction-spectrometric and spectrometric methods

Minerais et concentrés de manganèse — Dosage du cuivre — Méthodes spectrométriques avec et sans extraction

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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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 4294 was prepared by Technical Committee ISO/TC 65, *Manganese and chromium ores*.

ISO 4294 was first published in 1978. This second edition cancels and replaces the first edition, of which it constitutes a minor revision.

Manganese ores and concentrates — Determination of copper content — Extraction-spectrometric and spectrometric methods

1 Scope and field of application

This International Standard specifies the following methods for the determination of the copper content of manganese ores and concentrates

method A: extraction-spectrometric method, applicable to products having copper contents up to 0,5 % (m/m);

method B: spectrometric method, applicable to products having copper contents from 0,1 to 1 % (m/m).

It should be read in conjunction with ISO 4297.

2 References

ISO 4296, *Manganese ores — Sampling —*

Part 1: Increment sampling.

Part 2: Preparation of samples.

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

3 Method A: Extraction-spectrometric method for copper contents up to 0,5 % (m/m)

3.1 Principle

Decomposition of a test portion by treatment with hydrochloric and nitric acids, and evaporation of the solution to dryness. Dissolution of the dry residue in hydrochloric acid. Separation of the insoluble residue by filtration, ignition of the filter with the residue and treatment with sulfuric and hydrofluoric acids. Fusion of the ignited residue with sodium carbonate. Dissolution of the melt in water and combination of the solution obtained with the main solution. Addition, to an aliquot portion of the resulting solution, of ammonium citrate solution, Na₂EDTA solution, ammonium hydroxide solution to adjust the pH to 8,5, and sodium diethyldithiocarbamate solution.

Extraction of the copper-diethyldithiocarbamate complex with chloroform. Spectrometric measurement.

3.2 Reactions

The method is based on the interaction of copper(II) ions with sodium diethyldithiocarbamate, resulting in the formation of a

yellow or brown colour complex depending on the concentration of copper.

The influence of iron and other accompanying components is eliminated by binding them into soluble complexes with ammonium citrate and Na₂EDTA solution.

3.3 Reagents

3.3.1 Chloroform.

3.3.2 Sodium carbonate, anhydrous.

3.3.3 Nitric acid, ρ 1,40 g/ml.

3.3.4 Hydrofluoric acid, ρ 1,14 g/ml.

3.3.5 Hydrochloric acid, ρ 1,19 g/ml.

3.3.6 Hydrochloric acid, diluted 1 + 19.

3.3.7 Sulfuric acid, diluted 1 + 1.

3.3.8 Ammonium hydroxide, diluted 1 + 1.

3.3.9 Ammonium citrate [(NH₄)₂C₆H₅O₇], 100 g/l solution.

3.3.10 Sodium diethyldithiocarbamate trihydrate (C₅H₁₀NS₂Na·3H₂O), 1 g/l solution.

Prepare fresh as required.

3.3.11 Disodium dihydrogen ethylenedinitrilotetraacetate (Na₂EDTA), 100 g/l solution.

Dissolve 10 g of Na₂EDTA in 100 ml water while heating slightly, and filter.

3.3.12 Copper, standard solution, corresponding to 0,004 g of Cu per litre.

3.3.12.1 Copper, stock solution, 0,1 g/l.

Dissolve 0,1 g of metallic copper [purity 99,95 % (m/m)] in 10 ml of nitric acid (diluted 1 + 1), boil until free from nitrogen oxides, cool, add 10 ml of sulfuric acid (3.3.7), evaporate to