**International Standard** 



INTERNATIONAL ORGANIZATION FOR STANDARDIZATION MEXAYHAPOAHAR OPFAHUSALUR IIO CTAHAAPTUSALUNOORGANISATION INTERNATIONALE DE NORMALISATION

# Manganese ores and concentrates – Determination of manganese content – Potentiometric method

Minerais et concentrés de manganèse – Dosage du manganèse – Méthode potentiométrique

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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. 0

International Standard ISO 4298 was prepared by Technical Committee ISO/TC 65,

Anganese and chromium ores. ISO 4298 was first published in 1978. This second edition cancels and replaces the first international of which it constitutes a minor revision. 2 Jenerated by FLS

# Manganese ores and concentrates – Determination of manganese content – Potentiometric method

## 1 Scope and field of application

This International Standard specifies a potentiometric method for the determination of the manganese content of manganese ores and concentrates having a manganese content equal to or greater than 15 % (m/m).

It should be read in conjunction with ISO 4297.

### 2 References

ISO 4296/1, Manganese ores — Sampling — Part 1: Increment sampling.

ISO 4296/2, Manganese ores — Sampling — Part 2: Preparation of samples.

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

### **3** Principle

Decomposition of a test portion by treatment with hydrochloric, nitric, perchloric and hydrofluoric acids. Separation of the insoluble residue, and reservation of the filtrate as the main solution. Ignition of the residue, fusion with sodium carbonate, leaching of the melt with hydrochloric acid and combination with the main solution. Addition of an aliquot portion of the resulting solution to sodium pyrophosphate solution, adjustment of the pH to 7,0, and potentiometric titration with potassium permanganate standard volumetric solution.

#### 4 Reaction

The method is based on the oxidation of manganese(II) ions to manganese (III) ions by potassium permanganate in a neutral medium in the presence of sodium pyrophosphate:

 $4Mn^{2+} + MnO_4^{-} + 8H^{+} - - - 5Mn^{3+} + 4H_2O$ 

Iron and other interfering elements are eliminated by binding them into soluble pyrophosphate complexes.

#### 5 Reagents

5.1 Sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>), anhydrous.

5.2 Sodium carbonate, 50 g/l solution.

**5.3** Sodium pyrophosphate decahydrate  $(Na_4P_2O_7, 10H_2O)$ , 120 g/l solution.

This solution shall be prepared 24 h before use.

- Hydrochloric acid,  $\varrho$  1,19 g/ml.
- 5.5 Hydrochloric acid, diluted 1 + 4.
- **5.6 Hydrofluoric acid**,  $\varrho$  1,14 g/ml.
- **5.7** Perchloric acid,  $\rho$  1,51 g/ml.
- **5.8** Nitric acid, *ρ* 1,40 g/ml.
- **5.9** Potassium permanganate (KMnO<sub>4</sub>), degree of purity not less than 99,5 %, recrystalized

Dissolve 250 g of potassium permanganate (purity not less than 99,5 %) with 800 ml of hot water (90 °C) in a 1 000 ml beaker. Filter the solution under vacuum through a filter crucible with a sintered glass plate No. 3. Cool the filtered solution in an icebath to 10 °C, while stirring vigorously. Allow the fine-grained precipitate to settle. Then pour out the solution, transfer the crystalline mass to the crucible with sintered glass plate No. 3 and place under suction. Repeat the recrystallization.

After thorough suction, transfer the crystalline mass thus obtained to a glass or porcelain dish and dry in air in the dark, protecting from dust. When the crystalline mass no longer sticks together when crushed with a glass rod, dry it at 80 to 100  $^{\circ}$ C