
International Standard



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Manganese ores — Determination of active oxygen content, expressed as manganese dioxide — Titrimetric method

Minerais de manganèse — Dosage de l'oxygène actif, exprimé en dioxyde de manganèse — Méthode titrimétrique

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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.

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

This third edition cancels and replaces the second edition (ISO 312-1980), of which it constitutes a minor revision.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

Manganese ores — Determination of active oxygen content, expressed as manganese dioxide — Titrimetric method

1 Scope and field of application

This International Standard specifies a titrimetric method, by reduction with ammonium iron(II) sulfate, for the determination of the active oxygen content (conventionally expressed as manganese dioxide) of manganese ores.

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

Dissolution of a test portion in an excess of a standard solution of ammonium iron(II) sulfate in sulfuric acid, to reduce the manganese dioxide present in the test portion. Back-titration of the excess of ammonium iron(II) sulfate with standard volumetric potassium dichromate solution in the presence of sodium diphenylamine sulfonate as indicator.

4 Reagents

4.1 Phosphoric acid, ρ 1,7 g/ml.

4.2 Ammonium iron(II) sulfate, 60 g/l solution.

Dissolve 60 g of ammonium iron(II) sulfate $[(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}]$ in sulfuric acid, diluted (1 + 7), and dilute to 1 litre with the same acid.

4.3 Potassium dichromate, 8,780 g/l standard volumetric solution.

4.3.1 Preparation of the solution

Dissolve 8,780 g of potassium dichromate, recrystallized and dried at 180 to 200 °C, in 100 ml of water. Transfer the solution quantitatively to a 1 litre one-mark volumetric flask, dilute to the mark and mix.

4.3.2 Standardization of the solution

Take three test portions from a standard sample of manganese ore having a known manganese dioxide content approximately the same as that of the sample to be analysed and pass them through all stages of the analysis (7.4).

The titre of the potassium dichromate solution is given by the formula

$$T = \frac{B \times m}{V \times 100}$$

where

T is the titre of the potassium dichromate solution, expressed as grams of manganese dioxide corresponding to 1 ml of the solution;

B is the manganese dioxide content, as a percentage by mass, of the standard sample of manganese ore;

m is the mass, in grams, of the test portion from the standard sample;

V is the volume, in millilitres, of potassium dichromate solution used.

Take as the titre the average of the three results.

4.4 Sodium diphenylamine sulfonate, 0,8 g/l solution.

Dissolve 0,8 g of powdered sodium diphenylamine sulfonate ($\text{C}_6\text{H}_5\text{NHC}_6\text{H}_4\text{SO}_3\text{Na}$) in a small volume of water and dilute with water to 1 litre.

Store the solution in a brown glass bottle.

5 Apparatus

Ordinary laboratory apparatus and

5.1 Conical flask, 300 ml capacity, fitted with a stopper with two outflow pipes (see figure).

5.2 Source of carbon dioxide.

6 Sample

For the sampling of manganese ores, see ISO 4296/1. For the preparation of samples, see ISO 4296/2.

Use a test sample which has been crushed to a size not exceeding 100 µm (checked on a sieve of appropriate aperture size) and air dried under laboratory conditions.

7 Procedure

7.1 Test portion

Weigh 0,25 g of the test sample into the conical flask (5.1).

7.2 Number of analyses

Carry out the determination simultaneously on three test portions taken from the same test sample.

7.3 Blank test

Carry out a blank test following all stages of the analysis.

7.4 Determination

Add to the conical flask containing the test portion 7.1 50 ml of the ammonium iron(II) sulfate solution (4.2). Close the flask with its stopper and allow a current of carbon dioxide to pass through it; thoroughly mix and, without stopping the current of carbon dioxide, heat the contents of the flask moderately until the ore is dissolved (until the dark-coloured particles have disappeared).

Cool the contents of the flask (without stopping the current of carbon dioxide). Open the flask; add 10 ml of the phosphoric acid (4.1) and 2 ml of the sodium diphenylamine sulfonate solution (4.4). Dilute with cold water (from which air has been

removed by boiling) to 150 ml and titrate the excess of ammonium iron(II) sulfate with the potassium dichromate solution (4.3) until the solution becomes permanently violet-blue.

8 Expression of results

8.1 Calculation

The active oxygen content is given, as a percentage by mass of manganese dioxide, by the formula

$$\frac{T(V_1 - V_2) \times 100}{m_0} \times K$$

where

T is the titre of the standard volumetric potassium dichromate solution (see 4.3.2);

*V*₁ is the volume, in millilitres, of the standard volumetric potassium dichromate solution used in the blank test;

*V*₂ is the volume, in millilitres, of the standard volumetric potassium dichromate solution used in the determination;

*m*₀ is the mass, in grams, of the test portion;

K is the conversion factor for the expression of the active oxygen content on the dry basis.

8.2 Permissible tolerance on results of three parallel determinations

Active oxygen content ¹⁾ % (m/m)		Permissible tolerance % (m/m)
from	to	
	50,00	0,30
50,00	70,00	0,40
70,00	90,00	0,50

1) Conventionally expressed as manganese dioxide.

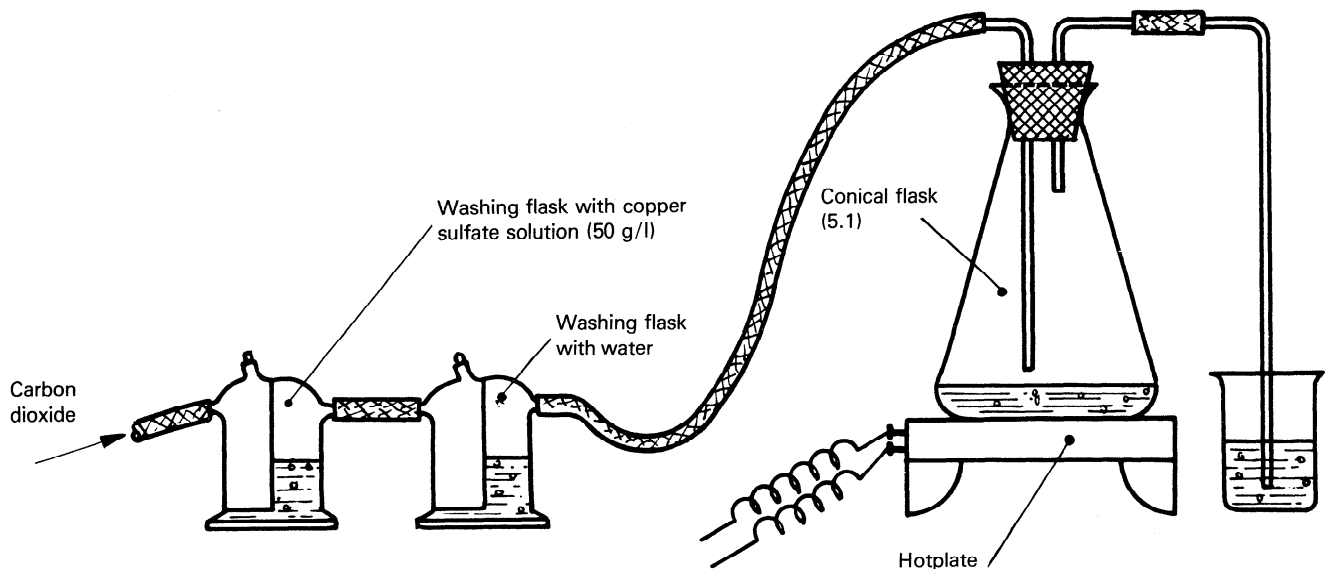


Figure — Example of apparatus