
INTERNATIONAL STANDARD



312

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

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

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

1 SCOPE AND FIELD OF APPLICATION

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

2 REFERENCES

ISO 310, *Manganese ores — Determination of hygroscopic moisture content in analytical samples — Gravimetric method*.

ISO . . ., *Manganese ores and concentrates — Sampling and sample preparation for chemical analysis and determination of moisture content*.¹⁾

3 PRINCIPLE

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

4 REAGENTS

During the analysis, use only reagents of recognized analytical reagent grade and only distilled water or water of equivalent purity.

4.1 Phosphoric acid, ρ 1,7 g/ml.

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

Dissolve 60 g of ammonium iron(II) sulphate $[(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}]$ in sulphuric acid, diluted (1 + 7), and dilute to 1 l 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 290 °C, in 100 ml of water. Transfer

the solution quantitatively to a 1 l 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 (7.5).

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 sulphonate, 0,8 g/l solution.

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

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.

1) This document, at present at the stage of draft proposal, is intended to complete and replace ISO/R 309, *Methods of sampling manganese ores — Part I — Ore loaded in freight wagons*.

6 SAMPLE

Use a test sample which has been crushed to a size not exceeding 0,10 mm (checked on a sieve of appropriate size) and air-dried under laboratory conditions (see ISO . . .).

7 PROCEDURE

7.1 Number of analyses

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

7.2 Blank test

In parallel with the determination and under the same conditions, carry out a blank test, in duplicate.

7.3 Check test

In parallel with the determination and under the same conditions, carry out a check analysis of a standard sample of manganese ore of known manganese dioxide content and of the type of ore to which the sample being analysed belongs.

7.4 Test portion

Weigh, to the nearest 0,000 2 g, 0,25 g of the test sample into the conical flask (5.1).

7.5 Determination

Add to the conical flask containing the test portion (7.4) 50 ml of the ammonium iron(II) sulphate 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 sulphonate solution (4.4). Dilute with cold water (from which the air has been removed by boiling) to 150 ml and titrate the excess of ammonium iron(II) sulphate with the potassium dichromate solution (4.3) until the solution becomes permanently violet-blue.

8 EXPRESSION OF RESULTS

8.1 Method of calculation

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

$$\frac{T (V_1 - V_2) \times 100}{m_o} \times \frac{100}{100 - A}$$

where

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

V₁ is the volume, in millilitres, of the standard volumetric potassium dichromate solution used in the blank test;

V₂ is the volume, in millilitres, of the standard volumetric potassium dichromate solution used in the determination;

m_o is the mass, in grams, of the test portion;

A is the hygroscopic moisture content of the test sample, as a percentage by mass, determined in accordance with ISO 310.

Take as the result the arithmetic mean of the three determinations, provided that the requirement of repeatability (see 8.2) is satisfied.

8.2 Repeatability

The maximum difference between the highest and the lowest results shall not exceed double the absolute value of the permissible tolerance on the result of the analysis (for the corresponding interval of manganese dioxide content) shown in the table below.

Active oxygen content ¹⁾ , %		Permissible tolerance, % (in absolute value)
from (over)	to	
	50,00	± 0,15
50,00	70,00	± 0,20
70,00	90,00	± 0,25

1) Conventionally expressed as manganese dioxide.

The average result of the simultaneous check analysis of the standard sample of manganese ore for active oxygen (conventionally expressed as manganese dioxide) content shall not differ from the result shown in the certificate by more than the ± value of the permissible tolerance (for the corresponding interval of manganese dioxide content) shown in the table.

9 TEST REPORT

The test report shall include the following information :

- a) indications necessary for the identification of the sample;
- b) reference to this International Standard;
- c) results of the analysis;
- d) the reference number of the results;
- e) any characteristics noticed during the determination, and any operations not specified in this International Standard which may have had an influence on the results.

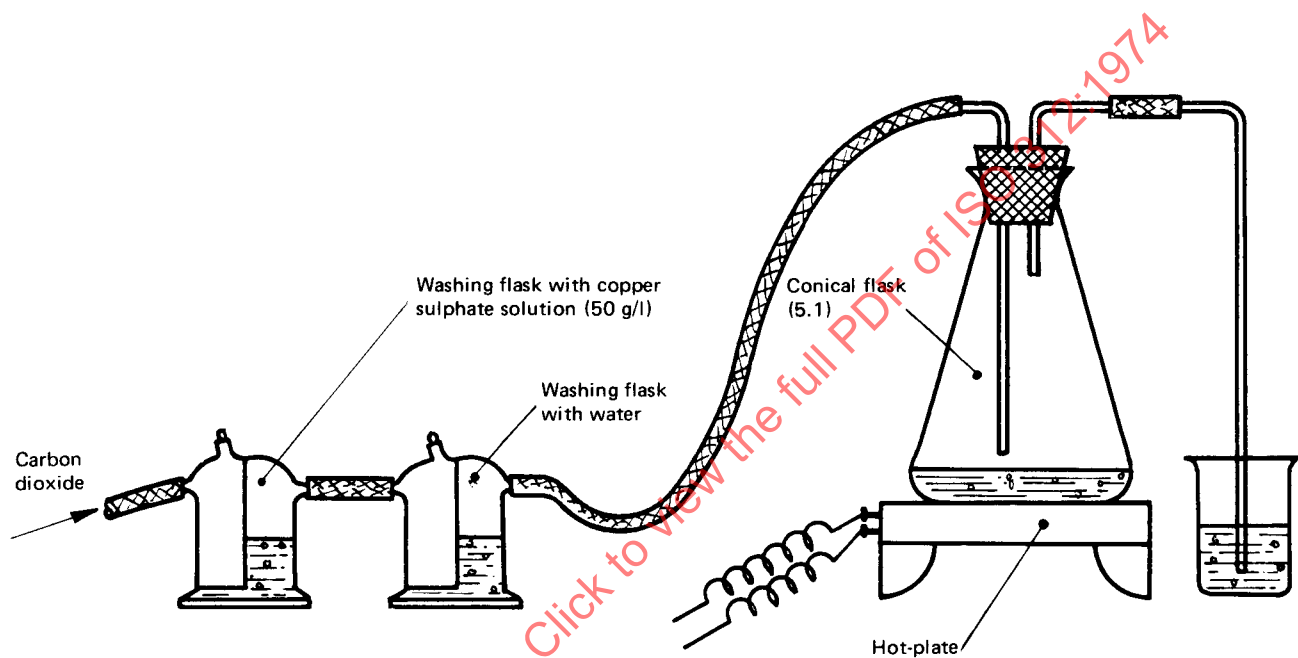


FIGURE — Example of apparatus