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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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.

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

This second edition was submitted directly to the ISO Council, in accordance with clause 5.10.1 of part 1 of the Directives for the technical work of ISO. It cancels and replaces the first edition (i.e. ISO 312-1974), which had been approved by the member bodies of the following countries :

Austria	India	Romania
Bulgaria	Ireland	South Africa, Rep. of
Chile	Italy	Spain
Czechoslovakia	Japan	United Kingdom
France	Netherlands	USSR
Germany, F.R.	Poland	
Hungary	Portugal	

No member body had expressed disapproval of the document.

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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.

It should be read in conjunction with ISO 4297.

2 Reference

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) 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

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 [$(NH_4)_2Fe(SO_4.6H_2O$] 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 ($C_6H_5NHC_6H_4.SO_3Na$) 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.

6 Procedure

6.1 Number of analyses

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

6.2 Test portion

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

6.3 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.

7 Expression of results

7.1 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.

7.2 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.

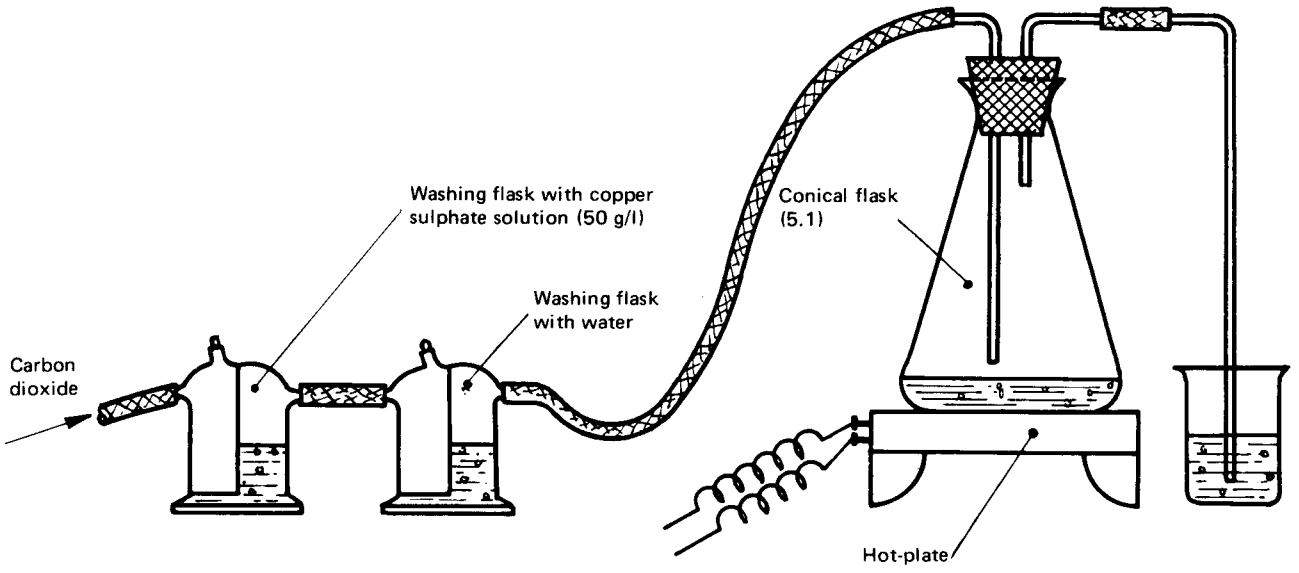

Figure — Example of apparatus