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ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION
R 314

METHODS OF CHEMICAL ANALYSIS OF MANGANESE ORES
DETERMINATION OF CARBON DIOXIDE

1st EDITION
July 1963

For each individual country the only valid standard is the national standard of that country.

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BRIEF HISTORY

The ISO Recommendation R 314, *Methods of Chemical Analysis of Manganese Ores—Determination of Carbon Dioxide*, was drawn up by Technical Committee ISO/TC 65, *Manganese Ores*, the Secretariat of which is held by the Komitet Standartov, Mer i Izmeritel'nyh Priborov pri Sovete Ministrov SSSR.

Work on this question by the Technical Committee began in 1954 and led, in 1957, to the adoption of a Draft ISO Recommendation.

In October 1958, this Draft ISO Recommendation (No. 247) was circulated to all the ISO Member Bodies for enquiry. It was approved by the following Member Bodies:

Austria
Bulgaria
Burma
Chile
Czechoslovakia
Finland
France
Germany
Hungary
India
Ireland
Italy
Japan
Netherlands
Poland
Portugal
Republic of South Africa
Romania
Spain
United Kingdom
U.S.S.R.

No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in July 1963, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 314 July 1963

METHODS OF CHEMICAL ANALYSIS OF MANGANESE ORES

DETERMINATION OF CARBON DIOXIDE

(Atomic mass C : 12.01; molecular mass CO_2 : 44.01)

This ISO Recommendation contains two parts:

I. INTRODUCTION

1. GENERAL INSTRUCTIONS

1.1 In the following analysis, use a sample for chemical analysis of air-dried manganese ore, which has been crushed to a size not exceeding 0.10 mm and checked on a sieve of appropriate size.

Simultaneously with the collection of samples for the determination of carbon dioxide, take three more test samples for the determination of hygroscopic moisture.

Calculate the content of carbon dioxide in ore which is absolutely dry by multiplying the numerical results of the determination of carbon dioxide by the conversion factor K, as found from the following formula:

$$K = \frac{100}{100 - A}$$

where A = hygroscopic moisture content, per cent.

1.2 The determination of carbon dioxide in manganese ore is carried out by simultaneously analysing three samples of ore, with two blank determinations to enable a corresponding correction in the result of the determination to be made.

Simultaneously and under the same conditions, carry out a check analysis of a standard sample of manganese ore, for carbon dioxide content.

The arithmetical mean of the three results is accepted as the final result.

The following conditions should be observed:

The maximum difference between the highest and the lowest results should not exceed double the absolute value of the permissible tolerance on the result of the analysis (for the corresponding interval of carbon dioxide content), shown in the table under clause 5.2, " Accuracy of method ".

The average result of the simultaneous check analysis of the standard sample of manganese ore for carbon dioxide content should not differ from the result shown in the certificate by more than the ± value of the permissible tolerance (for the corresponding interval of carbon dioxide content), shown in the table under clause 5.2, " Accuracy of method ".

For the analysis, take a standard sample of the type of ore to which the sample being analysed belongs.

1.3 The samples and the residues should be weighed to an accuracy of ± 0.0002 g.

1.4 Distilled water should be used during the procedure and for the preparation of solutions.

1.5 Indications as to the concentration of solutions show the quantity of solute (in grammes) in the corresponding volume of the solvent.

1.6 The following symbols and abbreviations are used :

d	relative density
g	gramme
g/l	grammes per litre
ml	millilitre
mm	millimetre
PFA	pure for analysis

II. GRAVIMETRIC METHOD

2. PRINCIPLE OF METHOD

The sample of manganese ore is decomposed with acid, and the carbon dioxide evolved is absorbed into soda asbestos (askarite). The increase in mass of the soda asbestos tubes corresponds to the content of the carbon dioxide in the given sample of ore.

3. REAGENTS REQUIRED

3.1 *Sulphuric acid*, PFA (*d* 1.84).

3.2 *Phosphoric acid*, PFA (*d* 1.7).

3.3 *Caustic potash*, PFA, solution (300 g/l).

3.4 *Magnesium perchlorate*, PFA.

3.5 *Copper sulphate anhydrous*, PFA. (Anhydrous copper sulphate is prepared by heating crystal copper sulphate at a temperature of 180 to 200 °C).

3.6 *Soda asbestos* (askarite), PFA.

3.7 *Chromic anhydride*, PFA.

4. PROCEDURE

4.1 Before commencing the determination, pass a current of oxygen through the whole system at a rate which will permit counting the gas bubbles in the drying tube (see Figure, page 7, No. 11), placed at the outlet end of the system and filled with sulphuric acid (*d* 1.84).

4.2 Every 5 to 10 min, disconnect and weigh absorption tubes Nos. 9 and 10 until they reach constant mass. If the results of the weighing coincide (within the tolerance limits) twice in succession, place the absorption tubes temporarily in a desiccator and proceed with the determination of carbon dioxide. For this purpose, place 0.5 to 1 g of manganese ore in a 150 ml conical flask (No. 2), add a little water, close the flask tightly with a bung provided with a dropping funnel (No. 3) and a condenser (No. 4), and pass a current of oxygen free from carbon dioxide through the whole system for 5 to 10 min to drive all the carbon dioxide from the apparatus.

4.3 Pour into the dropping funnel 15 ml of phosphoric acid (*d* 1.7), and quickly connect the weighed absorption tubes to the system. Opening the taps of all the tubes and of the oxygen cylinder, add phosphoric acid to the test sample of ore, carefully opening for that purpose the tap of the dropping funnel. The acid should be added in small portions so as to make the carbon dioxide pass through the drying tube with sulphuric acid (No. 5) at a rate not exceeding 3 or 4 bubbles per second.

After all the phosphoric acid has been added and the discharge of bubbles has become slower, heat the contents of the conical flask to boiling, and boil for 5 to 10 min. Then remove the flask from the hot plate, and let a current of oxygen pass through the system for 10 min to drive off any carbon dioxide which may have been left in other parts of the apparatus.

Close the tap of the oxygen cylinder, disconnect the absorption tubes and, closing their taps, place the tubes in the balance case for 30 min in order to equalize the temperatures. Open the taps of the tubes for a moment in order to equalize the pressure within them with that of the atmosphere, and weigh them.

5. EXPRESSION OF RESULTS

5.1 Method of calculation

The percentage content of carbon dioxide is calculated from the following formula:

$$CO_2 = \frac{(B - A) \times 100}{G} \text{ per cent}$$

where

A = mass of tubes Nos. 9 and 10 before the absorption, in grammes;
B = mass of tubes Nos. 9 and 10 after the absorption, in grammes;
G = mass of sample of ore, in grammes.

5.2 Accuracy of method

The permissible tolerances, per cent (absolute value), are given in the table below:

Carbon dioxide content from	 to	Permissible tolerance (in absolute value)
0.5 per cent	1.0 per cent	± 0.02 per cent
(over) 1.0 per cent	2.0 per cent	± 0.03 per cent
„ 2.0 per cent	5.0 per cent	± 0.05 per cent
„ 5.0 per cent		± 0.10 per cent