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# INTERNATIONAL STANDARD



# 314

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## Manganese ores — Determination of carbon dioxide content — Gravimetric method

*Minerais de manganèse — Dosage du dioxyde de carbone — Méthode gravimétrique*

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# Manganese ores – Determination of carbon dioxide content – Gravimetric method

## 1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a gravimetric method for the determination of the carbon dioxide content 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*.<sup>1)</sup>

## 3 PRINCIPLE

Decomposition of a test portion with acid, and absorption of the carbon dioxide evolved into soda asbestos (askarite). The increase in mass of the soda asbestos corresponds to the content of the carbon dioxide in the test portion.

## 4 REAGENTS

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

### 4.1 Chromium(III) oxide

**4.2 Copper(II) sulphate**, anhydrous, prepared by heating crystalline copper(II) sulphate at a temperature of 180 to 200 °C.

### 4.3 Magnesium perchlorate.

### 4.4 Soda asbestos (askarite).

### 4.5 Sulphuric acid, $\rho$ 1,84 g/ml.

### 4.6 Phosphoric acid, $\rho$ 1,7 g/ml.

### 4.7 Potassium hydroxide, 300 g/l solution.

## 5 APPARATUS

An example of the apparatus is given for guidance only, in the figure.

## 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, to enable a corresponding correction in the result of the determination to be made.

### 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 carbon 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, 0,5 to 1 g of the test sample into a 150 ml conical flask.

### 7.5 Determination

**7.5.1** 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, K) placed at the outlet end of the system and filled with sulphuric acid (4.5).

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*.

Every 5 to 10 min, disconnect and weigh the absorption tubes (I) and (J) 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.

**7.5.2** Add a little water to the test portion (7.4) in the conical flask, close the flask tightly with a bung provided with a dropping funnel (C) and a condenser (D), 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.

Pour into the dropping funnel 15 ml of the phosphoric acid (4.6), 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 portion, carefully opening for that purpose the tap of the dropping funnel. Add the acid in small portions so as to make the carbon dioxide pass through the drying tube containing sulphuric acid (E) 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 pass a current of oxygen 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 to an accuracy of  $\pm 0,0002$  g.

**8 EXPRESSION OF RESULTS**

**8.1 Method of calculation**

The carbon dioxide content of the absolutely dry ore is given, as a percentage by mass, by the formula

$$\frac{(m_2 - m_1) \times 100}{m_0} \times \frac{100}{100 - A}$$

where

- $m_0$  is the mass, in grams, of the test portion;
- $m_1$  is the mass, in grams, of tubes (I) and (J) before the absorption;

$m_2$  is the mass, in grams, of tubes (I) and (J) after the absorption;

A is the hygroscopic moisture content, expressed 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 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 carbon dioxide content) shown in the table below.

Carbon dioxide content, %		Permissible tolerance, % (in absolute value)
from	to	
0,5	1,0	$\pm 0,02$
(over) 1,0	2,0	$\pm 0,03$
2,0	5,0	$\pm 0,05$
" 5,0		$\pm 0,10$

The average result of the simultaneous check analysis of the standard sample of manganese ore for carbon dioxide content shall not differ from the result shown in the certificate by more than the  $\pm$  value of the permissible tolerance (for the corresponding interval of carbon 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.