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Manganese ores — Determination of combined water content — Gravimetric method

Minerais de manganèse — Dosage de l'eau de constitution — Méthode gravimétrique

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 65 has reviewed ISO Recommendation R 549 and found it technically suitable for transformation. International Standard ISO 549 therefore replaces ISO Recommendation R 549-1968 to which it is technically identical.

ISO Recommendation R 549 was approved by the Member Bodies of the following countries :

Australia
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Hungary
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Iran
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Italy
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Romania
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Spain
United Kingdom
U.S.S.R.
Yugoslavia

No Member Body expressed disapproval of the Recommendation.

No Member Body disapproved the transformation of ISO/R 549 into an International Standard.

Printed in Switzerland

Manganese ores — Determination of combined water content — Gravimetric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a gravimetric method for the determination of the combined water 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.* [1]

3 PRINCIPLE

Separation of the combined water by heating ore in a jet of dry air and collection into absorption tubes filled with magnesium perchlorate.

4 REAGENTS

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

4.1 **Magnesium perchlorate** [$Mg(ClO_4)_2$].

4.2 **Lead oxide** (PbO).

4.3 **Lead dioxide** (PbO_2).

4.4 **Hydrochloric acid,** diluted 1 : 1.

4.5 **Sulphuric acid,** ρ 1,84 g/ml.

5 APPARATUS

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

It consists of a quartz (or porcelain) tube (D), 500 to 600 mm in length with an inner diameter of 18 to 20 mm; the tapered end of the tube is 50 mm long and has an inner diameter of 1 to 2 mm.

At some distance from this end, either the tube is filled with a mixture consisting of equal parts of lead oxide (4.2), lead dioxide (4.3) and calcinated pumice, or a silver spiral is placed in the tube. The tube passes through two tubular electric furnaces (C) and (E). Two absorption glass U-tubes (F) and (G) filled with magnesium perchlorate (4.1) and previously saturated with carbon dioxide gas (the total mass of each filled tube should not exceed 40 g) and a potash-apparatus (H) containing sulphuric acid (4.5) are connected with the taper end of the tube, the potash-apparatus being connected with an aspirator or a special vacuum installation.

The air passing through the apparatus should previously have been dried. For this purpose the drying system, consisting of a rinsing vessel (A) containing sulphuric acid (4.5) and a column (B) containing magnesium perchlorate (4.1), is connected with the inlet end of the tube (D).

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[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, 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 combined water 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 platinum or porcelain boat.

7.5 Determination

7.5.1 Check the assembled apparatus for air-tightness. Before starting the determination, disconnect the absorption tubes and pass the air at a rate of two or three bubbles per second. Simultaneously, switch on the tube furnace (C) and heat it up to 800 to 900 °C. Heat the part of the tube containing the lead oxides and pumice or the silver spiral up to 200 to 250 °C by means of the electric furnace (E). Connect the absorption tubes with the apparatus for 30 min and pass the air for 30 min more. Then disconnect the absorption tubes, place them on the pan of a balance and 30 to 35 min later weigh each of the absorption tubes to the nearest 0,000 2 g, the tube cocks having been previously opened for a moment to equalize the pressure in the tubes with that of the atmosphere. After weighing, connect the absorption tubes with the apparatus again and, opening the cocks, pass the air for 30 to 60 min more. Then weigh the absorption tubes to the nearest 0,000 2 g under the same conditions. If the results of the two successive weighings agree (within the limits of the permissible error of weighing), place the absorption tubes temporarily on the pan of the balance and proceed to the determination of combined water as follows.

7.5.2 Having allowed the tube (D) to cool, introduce the platinum or porcelain boat containing the test portion (7.4) into the part of the tube which passes through the furnace (C), connect the absorption tubes with the apparatus and pass the dry air at a rate of two bubbles per second.

7.5.3 Simultaneously heat the part of the tube (D) containing the lead oxides and pumice or the silver spiral in the furnace (E) up to 250 to 300 °C. At the same time switch on the tube furnace (C), raise its temperature gradually up to 800 to 900 °C and pass the air for 30 min. Then, with the air flow passing, allow the tube (D) to cool, disconnect the absorption tubes, close the cocks and place the absorption tubes on the pan of the balance for 30 to 35 min to equalize the temperature; open the tube cocks for a moment to equalize the pressure in the tubes with that of the atmosphere, and then weigh the tubes to the nearest 0,000 2 g.

8 EXPRESSION OF RESULTS

8.1 Method of calculation

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

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

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the absorption tubes before the absorption of water;

m_2 is the mass, in grams, of the absorption tubes after the absorption of water;

A is the hygroscopic water 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 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 combined water content) shown in the table below.

Combined water content, %		Permissible tolerance, % (in absolute value)
from (over)	to	
	1,0	± 0,050
1,0	3,0	± 0,075
3,0	5,0	± 0,100
5,0		± 0,150

The average result of the simultaneous check analysis of the standard sample of manganese ore for combined water 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 combined water content) shown in the table.

9 TEST REPORT

The test report shall include the following information :

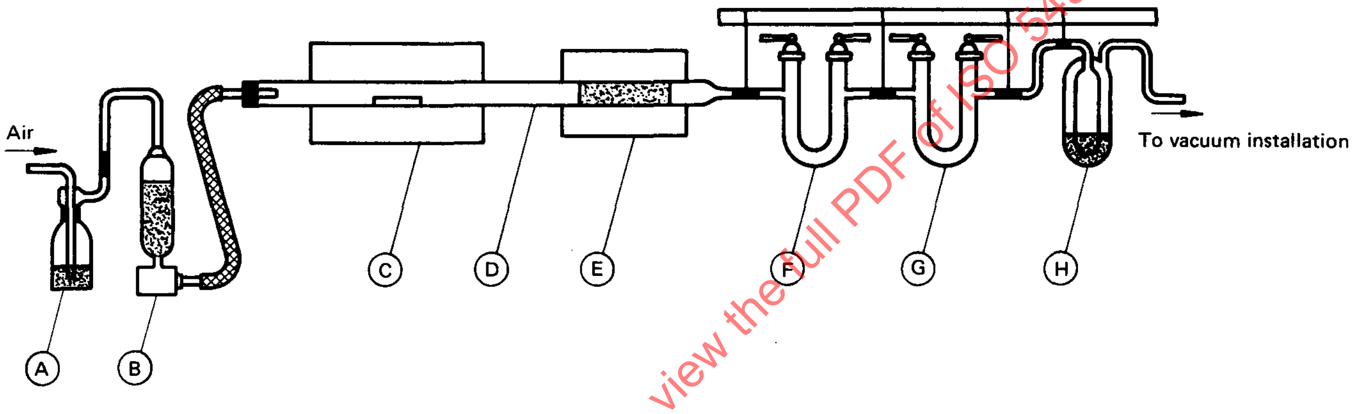
a) indications necessary for the identification of the sample;

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.



A Rinsing vessel containing sulphuric acid (4.5)

B Column containing magnesium perchlorate (4.1)

C Electric tube furnace

D Quartz (or porcelain) tube in which either a mixture of lead oxide, lead dioxide and calcinated pumice or a silver spiral is placed

E Electric tube furnace

F, G Two absorption tubes containing magnesium perchlorate and first saturated with carbon dioxide gas

H Potash-apparatus containing sulphuric acid (4.5).

FIGURE – **Example of apparatus**