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METHODS OF CHEMICAL ANALYSIS OF MANGANESE ORES

DETERMINATION OF COMBINED WATER CONTENT

1st EDITION

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BRIEF HISTORY

The ISO Recommendation R 549, *Methods of Chemical Analysis of Manganese Ores—Determination of Combined Water Content*, 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 (GOST).

Work on this question by the Technical Committee began in 1957 and led, in 1959, to the adoption of a Draft ISO Recommendation.

In November 1962, this Draft ISO Recommendation (No. 538) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies:

Australia
Austria
Burma
Chile
Czechoslovakia
France
Germany
Hungary
India
Iran
Ireland
Italy
Japan
Poland
Romania
Spain
U.A.R.
United Kingdom
U.S.S.R.
Yugoslavia

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 December 1966, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 549 December 1966

METHODS OF CHEMICAL ANALYSIS OF MANGANESE ORES

DETERMINATION OF COMBINED WATER CONTENT

(Molecular mass 18.02)

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 combined water, take three more test samples for the determination of hygroscopic moisture.

Calculate the results of the determination of combined water content in ore which is absolutely dry by multiplying the numerical results of the determination of combined water 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 combined water in manganese ore is carried out by analysing simultaneously 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 combined water 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 of the determination of combined water content in the ore analysed should 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 under clause 6.2, " Accuracy of method ".

The average result of the simultaneous check analysis of the standard sample of manganese ore for combined water 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 combined water content), shown in the table under clause 6.2, " 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 test samples and absorption tubes should be weighed to an accuracy of ± 0.0002 g.

1.4 The following symbols and abbreviations are used:

d	relative density
g	gramme
mm	millimetre
PFA	pure for analysis

2. PRINCIPLE OF METHOD

The combined water is separated by heating ore in a jet of dry air and is collected into absorption tubes, filled with magnesium perchlorate.

3. REAGENTS REQUIRED

3.1 *Magnesium perchlorate* ($Mg(ClO_4)_2$), PFA.

3.2 *Lead oxide* (PbO), PFA.

3.3 *Lead dioxide* (PbO_2), PFA.

3.4 *Hydrochloric acid*, PFA, diluted (1 : 1).

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

4. APPARATUS AND PREPARATION OF APPARATUS

4.1 Silver spiral.

4.2 The apparatus for the determination of combined water content (see diagram, page 7) consists of a quartz (or porcelain) tube, 500 to 600 mm in length with an inner diameter of 18 to 20 mm; the taper end of the tube is 50 mm long and has an inner diameter of 1 to 2 mm.

At some distance from this end the tube is filled with the mixture, consisting of equal parts of lead oxide, lead dioxide and calcinated pumice, or a silver spiral is placed into this tube. The quartz (or porcelain) tube is placed in a tubular electric furnace. Two absorption glass U-tubes filled with magnesium perchlorate and previously saturated with carbon dioxide gas (the total mass of each filled tube should not exceed 40 g) and a potash-apparatus with sulphuric acid (*d* 1.84) are connected with the taper end of the tube, the potash-apparatus being connected with an aspirator or a special vacuum installation.

4.3 The air passing through the apparatus should previously be dried. For this purpose the drying system, consisting of a rinsing vessel with sulphuric acid (*d* 1.84) and a column with magnesium perchlorate, is connected with the left end of the quartz (or porcelain) tube.

5. PROCEDURE

5.1 Check the assembled apparatus for air-tightness. Before the determination is started, disconnect the absorption tubes and pass the air at a rate of two or three bubbles per second. Simultaneously, switch on the tube furnace and heat it up to 800 to 900 °C. Heat a part of the tube containing a layer of lead and pumice oxides up to 200 to 250 °C by means of the electric furnace. 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 into a scale of the balance and 30 to 35 min later weigh each of the absorption tubes, the tube cocks having been previously opened for a moment to equilibrate 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 under the same conditions. If the results of the two successive weighings agree (within the limits of the permissible error of weighing), place temporarily the absorption tubes into the scale of the balance and proceed to the determination of combined water. For this purpose introduce a platinum or porcelain boat containing the test sample of manganese ore of 0.5 to 1.0 g into the quartz (or porcelain) tube, which has previously been allowed to cool, connect the absorption tubes with the apparatus and pass the dry air at a rate of two bubbles per second.

5.2 Simultaneously heat the part of the quartz (or porcelain) tube with the mixture of lead oxides in the furnace up to 250 to 300 °C. At the same time switch on the tube furnace (3), raise gradually the temperature up to 800 to 900 °C and pass the air for 30 min. Then, with the air flow passing, allow the quartz (or porcelain) tube to cool, disconnect the absorption tubes, close the cocks and place the absorption tubes into the scale of the balance for 30 to 35 min to equilibrate the temperature; open for a moment the tube cocks to equilibrate the pressure in the tubes with that of the atmosphere, and then weigh the tubes.

6. EXPRESSION OF RESULTS

6.1 Method of calculation

The percentage content of combined water is calculated from the following formula:

$$H_2O = \frac{(B-D) \times 100}{G} - A, \text{ per cent}$$

where B = mass of the absorption tubes after the absorption of water, in grammes;

D = mass of the absorption tubes before the absorption of water, in grammes;

G = mass of test sample of ore, in grammes;

A = percentage content of hygroscopic water.

6.2 Accuracy of method

The permissible tolerances, per cent (absolute value), are given 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%