International Standard 549

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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.

International Standard ISO 549 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 549-1975), which had been approved by the member bodies of the following countries :

Australia	Hungary	Romania
Austria	India	South Africa, Rep. of
Chile	Iran	Spain
Czechoslovakia	Ireland	United Kingdom
Egypt, Arab Rep. of	Italy	USSR
France	Japan	Yugoslavia
Germany, F. R.	Poland	

No member body had expressed disapproval of the document.

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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.

This International Standard should be read in conjunction with ISO 4297.

2 References

ISO 310, *Manganese ores — Determination of hygroscopic moisture content in analytical samples — Gravimetric method.*

ISO 4296/1, *Manganese ores — Sampling — Part 1 : Increment sampling.*[1)]

ISO 4296/2, *Manganese ores — Sampling — Part 2 : Preparation of samples.*[1)]

ISO 4297, *Manganese ores and concentrates — Methods of chemical analysis — General instructions.*

3 Principle

Separation of the combined water by heating a test portion in a current of dry air and collection in absorption tubes filled with magnesium perchlorate.

4 Reagents

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

4.2 Lead oxide (PbO).

4.3 Lead dioxide (PbO_2).

4.4 Sulphuric acid, ϱ 1,84 g/ml.

5 Apparatus

An example of the apparatus is shown, 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 the lead oxide (4.2), the lead dioxide (4.3) and calcined pumice, or a silver spiral is placed in the tube. The tube passes through two tubular electric furnaces (C) and (E). Two glass absorption 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 the sulphuric acid (4.4) are connected to the taper end of the tube, the potash-apparatus being connected to 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 the sulphuric acid (4.4) and a column (B) containing the magnesium perchlorate (4.1), is connected to the inlet end of the tube (D).

6 Sample

For increment sampling of manganese ores, see ISO 4296/1; for the preparation of samples, see ISO 4296/2.

Use a test sample which has been crushed to a size not exceeding 100 µm (checked on a sieve of appropriate size) and air-dried under laboratory conditions.

7 Procedure

7.1 Test portion

Weigh 0,5 to 1 g of the test sample into a platinum or porcelain boat.

1) At present at the stage of draft.

7.2 Determination

7.2.1 Check the assembled apparatus for air-tightness. Before starting the determination, disconnect the absorption tubes and pass air at a rate of 2 or 3 bubbles per second. Simultaneously, switch on the tube furnace (C) and heat to 800 to 900 °C. Heat the part of the tube containing the lead oxides (4.2 and 4.3) and pumice or the silver spiral to 200 to 250 °C by means of the electric furnace (E). Connect the absorption tubes to the apparatus for 30 min and pass air for a further 30 min. Then disconnect the absorption tubes, place them on the pan of a balance and 30 to 35 min later weigh each of them, the tube cocks having been previously opened for a moment to equalize the pressure in the tubes with that of the atmosphere. After weighing, again connect the absorption tubes to the apparatus and, opening the cocks, pass air for a further 30 to 60 min. Reweigh 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 the absorption tubes temporarily on the pan of the balance and proceed with the determination of combined water as follows.

7.2.2 Having allowed the tube (D) to cool, introduce the platinum or porcelain boat containing the test portion (7.1) into the part of the tube which passes through the furnace (C), connect the absorption tubes to the apparatus and pass dry air at a rate of 2 bubbles per second.

7.2.3 Simultaneously heat the part of the tube (D) containing the lead oxides (4.2 and 4.3) and pumice or the silver spiral to 250 to 300 °C by means of the electric furnace (E). At the same time, switch on the tube furnace (C), raise its temperature gradually to 800 to 900 °C and pass air for 30 min. Then, with the air flowing, 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.

8 Expression of results

8.1 Calculation

The combined water content, expressed as a percentage by mass, is given by the formula

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

where

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

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

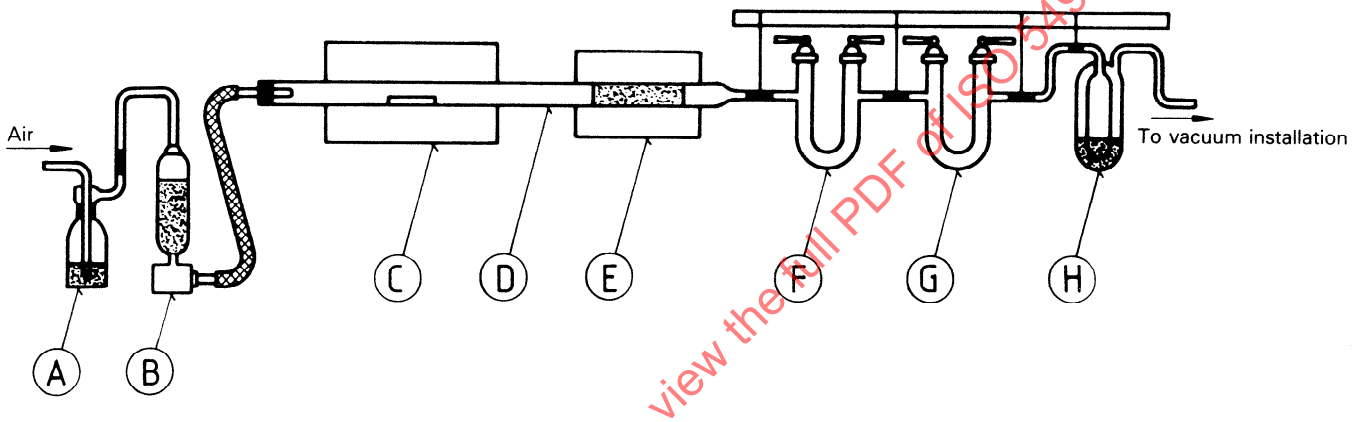
m_2 is the mass, in grams, of the absorption tubes after absorption of water;

A is the hygroscopic moisture content, expressed as a percentage by mass, determined in accordance with ISO 310;

K is the conversion factor for the expression of the combined water content on the dry basis.

8.2 Permissible tolerances on results of duplicate determinations

Combined water content, % (m/m)		Permissible tolerance, % (m/m)
from	to	
	1,0	0,050
1,0	3,0	0,075
3,0	5,0	0,100
5,0		0,150



A Rinsing vessel containing sulphuric acid (4.4)

B Column containing magnesium perchlorate (4.1)

C Electric tube furnace

D Quartz (or porcelain) tube in which either a mixture of lead oxide (4.2), lead dioxide (4.3) and calcinated pumice or a silver spiral is placed

E Electric tube furnace

F and G Two absorption tubes containing magnesium perchlorate (4.1) and first saturated with carbon dioxide gas

H Potash-apparatus containing sulphuric acid (4.4).

Figure — Example of apparatus

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