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INTERNATIONAL STANDARD ISO 548

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Manganese ores — Determination of barium oxide content — Barium sulphate gravimetric method

Minerais de manganèse — Dosage de l'oxyde de baryum — Méthode gravimétrique à l'état de sulfate de baryum

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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 548 and found it technically suitable for transformation. International Standard ISO 548 therefore replaces ISO Recommendation R 548-1966 to which it is technically identical.

ISO Recommendation R 548 was approved by the Member Bodies of the following countries :

Australia
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Hungary
India
Iran
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Italy
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Spain
United Kingdom
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Yugoslavia

No Member Body expressed disapproval of the Recommendation.

No Member Body disapproved the transformation of ISO/R 548 into an International Standard.

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Manganese ores – Determination of barium oxide content – Barium sulphate gravimetric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a barium sulphate gravimetric method for the determination of the barium oxide 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

Fusion of a test portion with sodium potassium carbonate; extraction of the fusion in water, filtering and dissolution of the barium carbonate in hydrochloric acid. Separation of the silicic acid, and precipitation of barium as barium sulphate, with a small quantity of sulphuric acid in weak hydrochloric acid solution. Filtration, ignition and weighing of the residue of barium sulphate.

4 REAGENTS

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

4.1 Sodium potassium carbonate, anhydrous.

4.2 Sodium carbonate (Na_2CO_3).

4.3 Hydrochloric acid, ρ 1,19 g/ml.

4.4 Hydrochloric acid, diluted 1 : 3.

4.5 Hydrochloric acid, diluted 1 : 100.

4.6 Nitric acid, ρ 1,40 g/ml.

4.7 Sulphuric acid, ρ 1,84 g/ml.

4.8 Sulphuric acid, diluted 1 : 1.

4.9 Sulphuric acid, diluted 1 : 4.

4.10 Sulphuric acid, diluted 1 : 1 000.

4.11 Hydrofluoric acid, 40 % (*m/m*).

4.12 Hydrogen peroxide, 30 % (perhydrol).

4.13 Ammonium acetate, 600 g/l solution.

4.14 Sodium carbonate, 10 g/l solution.

4.15 Silver nitrate, 2 g/l solution.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Platinum crucibles.

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.

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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.*

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 barium oxide 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, 1 to 2 g of the test sample into a platinum crucible.

7.5 Determination

7.5.1 Mix with the test portion (7.4) in the platinum crucible 6 to 9 g of anhydrous sodium potassium carbonate (4.1), cover with a lid and fuse at a temperature of 900 to 1 000 °C for 20 to 30 min.

After cooling, place the crucible with the fusion in a 300 to 400 ml beaker containing 80 to 100 ml of hot water (60 to 70 °C), add 2 to 3 ml of hydrogen peroxide (4.12), heat to boiling and boil for 10 to 15 min. Remove the crucible and wash off with hot water (60 to 70 °C), into the same beaker, the residue of the fusion adhering to the walls of the crucible and the lid; allow the residue to settle, filter off on a filter of average density and wash with hot (60 to 70 °C) sodium carbonate solution (4.14).

Wash off the residue from the filter with a jet of hot water into a 300 to 400 ml beaker. For a complete extraction of barium carbonate residue, wash the filter four or five times with hot (60 to 70 °C) hydrochloric acid solution (4.4), containing a small quantity of hydrogen peroxide, and add it to the main solution. Add 30 ml of hydrochloric acid (4.3) to the contents of the beaker and heat the solution until the residue is completely dissolved.

7.5.2 Evaporate the solution obtained until dry; add 10 to 15 ml of hydrochloric acid (4.3) to the dry residue and evaporate until dry once again, keeping it at a temperature of 120 to 130 °C for 40 to 60 min.

7.5.3 Add 10 to 15 ml of hydrochloric acid (4.3) to the dry residue and heat for 3 to 5 min, add 30 to 40 ml of hot water, heat to boiling once again and filter off the residue of silicic acid. Wash the residue on the filter three or four times with hot hydrochloric acid (4.5) and then six to eight times with hot water.

7.5.4 Place the filter with the residue in a platinum crucible, ignite at a temperature of 500 to 600 °C, moisten after cooling with 2 or 3 drops of water, add 2 or 3 drops of sulphuric acid (4.7), 5 to 6 ml of hydrofluoric acid (4.11) and evaporate until dry. Dissolve the dry residue in 2 to 3 ml of hydrochloric acid (4.3) while heating and add it to the main solution.

7.5.5 Evaporate the combined solution until dry. Moisten the dry residue with 1 to 2 ml of hydrochloric acid (4.3) and dissolve in 100 to 150 ml of water. Heat the solution obtained to boiling, add 5 ml of sulphuric acid (4.9), heat to boiling once again and boil for 15 to 20 min; then allow the residue to settle for 12 h.

7.5.6 Filter the residue on a dense filter containing a small quantity of paper pulp, and wash several times with cold sulphuric acid (4.10), until the reaction for chloride ion ceases (test reaction with silver nitrate solution (4.15)). If there is lead in the ore, wash the residue of barium sulphate six to eight times with hot ammonium acetate solution (4.13), then three or four times with hot water. Place the filter with the residue in a weighed platinum crucible and ignite at a temperature of 600 to 700 °C for 20 to 25 min. If the ignited residue is somewhat coloured, fuse it with 0,3 to 0,4 g of sodium carbonate (4.2). Extract the cooled fusion in hydrochloric acid (4.4) and repeat the process of precipitation of barium sulphate. Filter off the residue, ignite and weigh to the nearest 0,000 2 g.

8 EXPRESSION OF RESULTS

8.1 Method of calculation

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

$$\frac{m_1 \times 0{,}657\,0 \times 100}{m_0} \times \frac{100}{100 - A}$$

where

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

m_1 is the mass, in grams, of the residue of barium sulphate;

0,657 0 is the factor for converting barium sulphate to barium oxide;

A is the hygroscopic moisture 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 barium oxide content) shown in the table below.

Barium oxide content, %		**Permissible tolerance,** % (in absolute value)
from	to	
0,1	0,3	± 0,02
(over) 0,3	0,5	± 0,03
" 0,5	1,0	± 0,06
" 1,0	5,0	± 0,10
" 5,0	10,0	± 0,20
" 10,0	20,0	± 0,30

The average result of the simultaneous check analysis of the standard sample of manganese ore for barium oxide 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 barium oxide 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.