



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

Second edition — 1981-11-15

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UDC 669.74 : 543.21 : 546.431-31

Ref. No. ISO 548-1981 (E)

Descriptors : manganese ores, chemical analysis, determination of content, barium oxides, gravimetric analysis.

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 548 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 548:1975), 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.

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.

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

2 References

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

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

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 Sulphuric acid, ρ 1,84 g/ml.

4.7 Sulphuric acid, diluted 1 + 4.

4.8 Sulphuric acid, diluted 1 + 1 000.

4.9 Hydrofluoric acid, 40 % (m/m).

4.10 Hydrogen peroxide, 30 % (perhydrol).

4.11 Ammonium acetate, 600 g/l solution.

4.12 Sodium carbonate, 10 g/l solution.

4.13 Silver nitrate, 2 g/l solution.

5 Apparatus

Ordinary laboratory apparatus and

5.1 Platinum crucibles.

6 Sample

For increment sampling of manganese ores, see ISO 4296/1. For the preparation of the 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 1 to 2 g of the test sample into a platinum crucible (5.1).

¹⁾ At present at the stage of draft.

7.2 Determination

7.2.1 Mix with the test portion (7.1) in the platinum crucible 6 to 9 g of the 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 the hydrogen peroxide (4.10), heat to boiling and boil for 10 to 15 min. Remove the crucible and wash 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 on a filter paper of average density and wash with hot (60 to 70 °C) sodium carbonate solution (4.12).

Wash 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 the hydrochloric acid (4.3) to the contents of the beaker and heat the solution until the residue is completely dissolved.

7.2.2 Evaporate the solution obtained until dry; add 10 to 15 ml of the 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.

7.2.3 Add 10 to 15 ml of the hydrochloric acid (4.3) to the dry residue and heat for 3 to 5 min, add 30 to 40 ml of hot water (60 to 70 °C), heat to boiling once again and filter the residue of silicic acid. Wash the residue on the filter paper three or four times with hot (60 to 70 °C) hydrochloric acid solution (4.5) and then six to eight times with hot water (60 to 70 °C).

7.2.4 Place the filter paper with the residue in a platinum crucible (5.1), 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 the sulphuric acid (4.6), 5 to 6 ml of the hydrofluoric acid (4.9) and evaporate until dry. Dissolve the dry residue in 2 to 3 ml of the hydrochloric acid (4.3) while heating and add it to the main solution.

7.2.5 Evaporate the combined solution until dry. Moisten the dry residue with 1 to 2 ml of the hydrochloric acid (4.3) and dissolve in 100 to 150 ml of water. Heat the solution obtained to boiling, add 5 ml of the sulphuric acid solution (4.7), heat to boiling once again and boil for 15 to 20 min; then allow the residue to settle for 12 h.

7.2.6 Filter the residue on a dense filter paper containing a small quantity of paper pulp, and wash several times with cold sulphuric acid solution (4.8), until the reaction for chloride ion ceases [test reaction with the silver nitrate solution (4.13)]. If there is lead in the ore, wash the residue of barium sulphate six to eight times with hot (60 to 70 °C) ammonium acetate solution (4.11), then three or four times with hot water (60 to 70°C). 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 the sodium carbonate (4.2). Extract the cooled fusion in the hydrochloric acid solution (4.4) and repeat the process of precipitation of barium sulphate. Filter the residue, ignite and weigh.

8 Expression of results

8.1 Calculation

The barium oxide (BaO) content, expressed as a percentage by mass, is given by the formula

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

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;

K is the conversion factor for the expression of the barium oxide content on the dry basis;

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

8.2 Permissible tolerances on results of duplicate determinations

Barium oxide content, % (m/m)		Permissible tolerance, % (m/m)
from	to	
0,1	0,3	0,02
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