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ISO RECOMMENDATION

R 318

METHODS OF CHEMICAL ANALYSIS OF MANGANESE ORES

DETERMINATION OF ALUMINIUM OXIDE

1st EDITION

July 1963

For each individual country the only valid standard is the national standard of that country.

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BRIEF HISTORY

The ISO Recommendation R 318, *Methods of Chemical Analysis of Manganese Ores — Determination of Aluminium Oxide*, 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.

Work on this question by the Technical Committee began in 1954 and led, in 1957, to the adoption of a Draft ISO Recommendation.

In October 1958, this Draft ISO Recommendation (No. 251) was circulated to all the ISO Member Bodies for enquiry. It was approved by the following Member Bodies:

Austria	Hungary	Portugal
Bulgaria	India	Republic of South Africa
Burma	Ireland	Romania
Chile	Italy	Spain
Czechoslovakia	Japan	United Kingdom
France	Netherlands	U.S.S.R.
Germany	Poland	

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 July 1963, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 318 July 1963

METHODS OF CHEMICAL ANALYSIS OF MANGANESE ORES

DETERMINATION OF ALUMINIUM OXIDE

(Atomic mass Al: 26.98; molecular mass Al_2O_3: 101.96)

This ISO Recommendation contains two parts:

I. INTRODUCTION

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 aluminium oxide, take three more test samples for the determination of hygroscopic moisture.

Calculate the content of aluminium oxide in ore which is absolutely dry by multiplying the numerical results of the determination of aluminium oxide 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 aluminium oxide in manganese ore is carried out by simultaneously analysing 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 aluminium oxide 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 should not exceed double the absolute value of the permissible tolerance on the result of the analysis (for the corresponding interval of aluminium oxide content), shown in the table under clause 5.2, " Accuracy of method ".

The average result of the simultaneous check analysis of the standard sample of manganese ore for aluminium oxide 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 aluminium oxide content), shown in the table under clause 5.2, " 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 the residues should be weighed to an accuracy of ±0.0002 g.

1.4 Distilled water should be used during the procedure and for the preparation of solutions.

1.5 Meanings of the following expressions:

hot water (or solution) implies a temperature of the liquid of 60 to 70 °C;

warm water (or solution) implies a temperature of the liquid of 40 to 50 °C;

diluted 1 : 1, 1 : 2, 1 : 5, etc. means that

the first figure gives the number of parts by volume of concentrated acid or some other solution, and

the second figure gives the number of parts by volume of water.

1.6 Indications as to the concentration of solutions show the quantity of solute (in grammes) in the corresponding volume of the solvent.

The following symbols and abbreviations are used:

cm	centimetre
d	relative density
g	gramme
g/l	grammes per litre
ml	millilitre
mm	millimetre
PFA	pure for analysis

II. OXYQUINOLINE METHOD OF ALUMINIUM OXIDE DETERMINATION WITH ITS PRELIMINARY ISOLATION IN THE FORM OF PHOSPHATE

2. PRINCIPLE OF METHOD

After the separation of silicic acid by dehydration, the aluminium is separated from most accompanying elements as aluminium phosphate in the presence of sodium thiosulphate. The determination is completed by the precipitation of aluminium with oxyquinoline and its subsequent weighing as aluminium oxide.

3. REAGENTS REQUIRED

3.1 *Ammonium hydroxide*, PFA (*d* 0.91).

3.2 *Ammonium nitrate*, PFA, solution (20 g/l). Dissolve 20 g of ammonium nitrate in 1 litre of hot water. To the solution, add ammonia solution in the presence of methyl red, until the colour turns yellow.

3.3 *Ammonium acetate*, PFA, solution (200 g/l).

3.4 *Ammonium phosphate di-hydrogen* ($(NH_4)_2HPO_4$), PFA, solution (100 g/l).

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

3.6 *Hydrochloric acid*, PFA (*d* 1.19).

3.7 *Hydrochloric acid*, PFA, diluted 1 : 4.

3.8 *Hydrochloric acid*, PFA, diluted 1 : 50.

3.9 *Hydrofluoric acid*, PFA (40 per cent).

3.10 *Acetic acid* (glacial), PFA, diluted 1 : 1.

3.11 *Oxalic acid*, PFA.

3.12 *Methyl red indicator*, alcoholic solution (1 g/l).

3.13 *Sodium thiosulphate* ($Na_2S_2O_3 \cdot 5H_2O$), PFA, solution (200 g/l).

3.14 *Sodium carbonate*, PFA, anhydrous.

3.15 *Sodium carbonate*, PFA, solution (10 g/l).

3.16 *Oxyquinoline*, PFA, solution (50 g/l). Dissolve 50 g of oxyquinoline in 1 litre acetic acid 2 N.

3.17 *Ethyl alcohol*, PFA.

4. PROCEDURE

4.1 Weigh 0.5 to 1 g of manganese ore into a 100 ml beaker, and dissolve while heating in 10 to 15 ml of hydrochloric acid (*d* 1.19). Evaporate the solution until dry, moisten the residue with 5 ml of hydrochloric acid (*d* 1.19), and again evaporate until dry; keep the dry residue at 120 to 130 °C for 40 to 60 min. Add to the dry residue 10 to 15 ml of hydrochloric acid (*d* 1.19), and heat the solution for 3 to 5 min. Then add 30 to 40 ml of hot water, heat to boiling, and filter off the residue of silicic acid.

4.2 Wash the residue on the filter 3 or 4 times with hot hydrochloric acid, diluted 1 : 50, and then 6 to 8 times with hot water. Place the filter with the residue in a platinum crucible, dry, and ignite at 500 to 600 °C.

4.3 Cool the residue, moisten it with 2 to 3 drops of water, add 2 to 3 drops of sulphuric acid (*d* 1.84) and 5 to 6 ml of hydrofluoric acid, and evaporate to dryness. Heat the residue at 450 to 500 °C and cool. Mix with 4 g of anhydrous sodium carbonate, and fuse the mixture at 900 to 1000 °C for 20 to 25 min. Place the crucible with the cold melt in a 150 ml beaker, add 50 to 60 ml of hydrochloric acid, diluted 1 : 4, and heat until the melt has completely dissolved. Wash the crucible with water, remove it from the beaker, and add the solution obtained from the fusion to the original filtrate.

4.4 Add to the combined filtrate 20 ml of ammonium phosphate di-hydrogen solution (100 g/l) and 4 or 5 drops of methyl red solution (1 g/l), and add ammonium hydroxide until the colour of the indicator changes; at the same time, the liquid begins to yield a precipitate and becomes turbid.

4.5 Add 4 ml of hydrochloric acid (*d* 1.19) to the solution, and stir until the precipitate dissolves. Dilute with warm water to 300 to 400 ml. If the solution is still turbid, add, drop by drop, hydrochloric acid (*d* 1.19) until the precipitate dissolves completely; stir the solution carefully after each drop. Then add, in consecutive order, 60 ml of sodium thiosulphate solution (200 g/l), 25 ml of acetic acid, diluted 1 : 1, and 15 ml of ammonium acetate solution (200 g/l); heat the solution and keep it boiling vigorously for 10 to 15 min until the sulphur coagulates and the solution becomes clear.

4.6 Filter off the residue on a fast filter containing a small quantity of paper pulp.

Wash the sides of the beaker, and then the filter and residue 6 to 8 times with a hot neutral solution of ammonium nitrate (20 g/l).

Place the filter and the residue in a platinum crucible, dry, and ignite at 500 to 600 °C.

Cool the crucible, add 3 to 4 g of sodium carbonate, and fuse the mixture at 950 to 1000 °C.