

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION R 311

METHODS OF CHEMICAL ANALYSIS OF MANGANESE ORES
DETERMINATION OF SILICON DIOXIDE

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BRIEF HISTORY

The ISO Recommendation R 311, *Methods of Chemical Analysis of Manganese Ores—Determination of Silicon Dioxide*, 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. 244) was circulated to all the ISO Member Bodies for enquiry. It was approved by the following Member Bodies:

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

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Two Member Bodies opposed the approval of the Draft:

France, India.

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.

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METHODS OF CHEMICAL ANALYSIS OF MANGANESE ORES

DETERMINATION OF SILICON DIOXIDE

(Atomic mass Si : 28.09, molecular mass SiO₂: 60.09)

This ISO Recommendation contains two parts:

- I. Introduction section 1,
 II. Gravimetric hydrochloric acid method of determination of silicon dioxide . . sections 2 to 5,

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I. INTRODUCTION

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- 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 silicon dioxide, take three more test samples for the determination of hygroscopic moisture.

Calculate the content of silicon dioxide in ore which is absolutely dry by multiplying the numerical results of the determination of silicon dioxide 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 silicon dioxide 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 silicon dioxide 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 silicon dioxide 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 silicon dioxide content should not differ from the result shown in the certificate by more than the \pm value of the permissible tolerance (for the corresponding interval of silicon dioxide 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 samples and the silica 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.

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

- 1.7 The following symbols and abbreviations are used:

<i>d</i>	relative density
<i>g</i>	gramme
<i>g/l</i>	grammes per litre
<i>ml</i>	millilitre
<i>mm</i>	millimetre
PFA	pure for analysis

II. GRAVIMETRIC HYDROCHLORIC ACID METHOD

2. PRINCIPLE OF METHOD

The sample of ore is dissolved in hydrochloric acid; the insoluble residue is fused with sodium carbonate and added to the main solution. From this, silicic acid is then precipitated by means of dehydration effected by evaporating twice to dryness the initial hydrochloric solution and the filtrate.

3. REAGENTS REQUIRED

3.1 *Ammonium thiocyanate*, PFA, solution (10 g/l).

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

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

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

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

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

3.7 *Sodium carbonate* (anhydrous), PFA.

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4. PROCEDURE

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4.1 Weigh a sample of 0.5 to 2 g of manganese ore, depending on silicon dioxide content, into a 100 ml beaker, add 15 to 30 ml of hydrochloric acid (*d* 1.19) and heat until the attack is complete. Evaporate the solution until dry and until the smell of hydrochloric acid has been driven off, digest the dry residue in 10 to 15 ml of hydrochloric acid (*d* 1.19), dilute with hot water to 20 to 30 ml, heat to boiling and filter off the insoluble residue.

4.2 First wash the insoluble residue on the filter 3 or 4 times with hydrochloric acid, diluted 1 : 50, and then 6 to 8 times with hot water. Place the filter with the residue in a platinum crucible and ignite. Fuse the residue in the crucible with 2 g of sodium carbonate at a temperature of 900 to 1000 °C. Extract the fusion in 50 ml of hot hydrochloric acid, diluted 1 : 4, thoroughly wash the crucible and add the solution to the original filtrate. Evaporate the combined solutions until dry and heat until the smell of the hydrochloric acid has been driven off. Moisten the dry residue with 5 ml of hydrochloric acid (*d* 1.19) and again evaporate until dry by keeping the beaker containing the residue at 120 to 130 °C for 40 to 60 min.

Add 10 to 15 ml of hydrochloric acid (*d* 1.19) to the dried residue thus obtained, and place on a hot plate for 3 to 5 min. Add 40 ml of hot water, heat without allowing the mixture to boil, and filter off the silicic acid.

4.3 First wash the filter with the residue with hydrochloric acid, diluted 1 : 50, until removal of the iron (test with ammonium thiocyanate), and then 2 or 3 times with hot water.

Retain the filter with the residue. Evaporate the filtrate to dryness, and repeat the evaporation with 5 ml of hydrochloric acid (*d* 1.19), as before.

- 4.4 Take up the dried residue in 10 ml of hydrochloric acid (*d* 1.19) and 40 ml of water; heat the solution to boiling, filter and wash the residue of silicic acid thus obtained, as stated above.

Place the filters and the residues together in a platinum crucible, and ignite at a temperature of 1000 to 1050 °C to constant weight.

- 4.5 Moisten the dry residue in the crucible with some drops of water, add 2 drops of sulphuric acid (*d* 1.84) and 5 to 7 ml of hydrofluoric acid, and evaporate until vapour of sulphuric acid has been driven off. Then place the crucible in a muffle and ignite it at a temperature of 1000 to 1050 °C to constant weight. The loss in weight corresponds to the weight of silicon dioxide.

Note. After the removal of silicic acid, the filtrate may be used for the determination of phosphorus, aluminium, nickel, cobalt and other constituents.

5. EXPRESSION OF RESULTS

5.1 Method of calculation

The percentage content of silicon dioxide is calculated from the following formula:

$$\text{SiO}_2 = \frac{(A - B) \times 100}{G} \text{ per cent}$$

where

A = mass of crucible with residue of silicic acid before treatment with hydrofluoric and sulphuric acids, in grammes;

B = mass of crucible after treatment with hydrofluoric and sulphuric acids, in grammes;

G = mass of sample of ore, in grammes.

5.2 Accuracy of method

The permissible tolerances, per cent (absolute value), are given in the table below:

Silicon dioxide content		Permissible tolerance (in absolute value)
from (over)	to	
	2.00 per cent	± 0.04 per cent
2.00 per cent	5.00 per cent	± 0.08 per cent
5.00 per cent	10.00 per cent	± 0.12 per cent
10.00 per cent	15.00 per cent	± 0.15 per cent