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Draft Indian Standard

CHEMICAL ANALYSIS OF LIMESTONE, DOLOMITE AND ALLIED MATERIALS

PART 2 DETERMINATION OF SILICA

(Second Revision)

ICS 73.080

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

XXXX **2022** **Price Group**

Methods of Chemical Analysis of Metals Sectional Committee, MTD 34

FOREWORD

This draft Indian Standard (Part 2) (Second Revision) subject to its finalization, is to be adopted by the Bureau of Indian Standards on recommendation of the Methods of Chemical Analysis of Metals Sectional Committee and approval of the Metallurgical Engineering Division Council.

This standard was first published in 1962. It covers the determination of different elements in various grades of minerals like limestone, dolomite, calcite and magnesite. It also covers the methods for magnesite refractories. Subsequently, the first revision was issued to bifurcate this standard into five different parts covering determination of various elements in separate parts. This revision has been brought out to bring the standard in the latest style and format of the Indian Standards. This part covers determination of silica by gravimetric method. The other parts in the series are as follows:

Part 1 Loss on ignition

Part 3 Determination of iron oxide, alumina, calcium oxide and magnesia

Part 4 Determination of carbon dioxide

Part 5 Determination of chlorides

Part 6 Determination of free silica

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’. The number of significant places retained in the rounded off value should be same as that of the specified value in this standard.

Draft Indian Standard

CHEMICAL ANALYSIS OF LIMESTONE, DOLOMITE AND ALLIED MATERIALS

PART 2 DETERMINATION OF SILICA

(Second Revision)

1 SCOPE

This standard (Part 2) describes the method for determination of silica in limestone, dolomite and allied materials.

2 REFERENCES

The following Indian Standards contain provisions, which through reference in this text, constitute provision of this standard. At the time of the publication, the editions indicated below were valid. All the standards are subject to revision, and parties to agreement based on this standard are encouraged to investigate the possibility of applying the most recent editions of these standards indicated below.

IS No.	*Title*
1070 : 1992	Reagent grade water – Specification (*third revision*)
2109 : 1982	Methods of sampling dolomite, limestone and other allied materials

3 SAMPLING

3.1 The sample shall be drawn and prepared in accordance with IS 2109.

3.2 Grind 5 to 10 g of the prepared sample drawn under **3.1**, so that it passes through IS sieve 15 (100 mesh). Dry to constant mass at 105 ± 2°C and use it for the purpose of chemical analysis.

4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and distilled water (*see* IS 1070) shall be employed for the test.

5 DETERMINATION OF SILICA BY GRAVIMETRIC METHOD

5.1 Outline of the Method

Sample is dissolved in dilute hydrochloric acid and baked. The baked mass is extracted with dilute hydrochloric acid. The insoluble residue is then fused with anhydrous sodium carbonate and the

melt is extracted with dilute hydrochloric acid. The silica is dehydrated and determined by hydro-fluorization.

5.2 Reagents

5.2.1 *Dilute Hydrochloric Acid*, 40 percent (*v/v*).

5.2.2 *Fusion Mixture*

Mix carbonates of sodium and potassium in equal proportion.

5.2.3 *Dilute Sulphuric Acid*, 1:4 and 1:1 (*v/v*).

5.2.4 *Hydrofluoric Acid,* 40 percent (*v/v*).

5.3 Procedure

5.3.1 *For other than Magnesite Refractory Materials*

5.3.1.1 Weigh accurately 1.0 g of the test sample into a beaker or a porcelain dish and add to it 40 to 50 ml of dilute hydrochloric acid. Cover the beaker by means of a suitable cover glass immediately after the addition of the acid. As soon as effervescence stops, wash the lower surface of the cover glass into the beaker and set contents for drying and baking at 110° to 115°C. After baking for about 20 to 25 minutes, cool to room temperature, add 25 to 30 ml dilute hydrochloric acid, boil and filter. Thoroughly transfer all the residue as well as that adhering to the sides of the beaker, to the filter by hot water. Wash the residue free from chlorides by means of hot water. Collect the filtrate and washings in the same beaker and preserve it.

5.3.1.2 Transfer the filtrate with its residue into a previously heated platinum crucible and char at low temperature. Finally ignite at 900° to 950°C and cool.

5.3.1.3 Fuse the residue in the platinum crucible with about 3 g of fusion mixture. Cool and extract the melt in about 50 ml of dilute hydrochloric acid. Mix with the filtrate preserved under **5.3.1.1** and repeat the process of drying and baking. Extract the baked mass with about 30 to 40 ml of dilute hydrochloric acid and filter. Transfer all the silica in the beaker to the filter thoroughly by means of hot water. Wash silica on the filter free from chloride by means of hot water. Collect the filtrate and washings in the same beaker and preserve the filtrate.

5.3.1.4 Transfer the filter with its residue into a platinum crucible and smoke off the filter paper at a low heat without burning the paper. Finally ignite at 900° to 950°C to a constant mass. Moisten the residue with few millilitres of dilute sulphuric acid (1:1) and add to it about 10 ml of hydrofluoric acid. Evaporate to dryness, ignite, cool and weigh.

5.3.1.5 Carry out a blank determination following the procedure specified in **5.3.1.1** to **5.3.1.4** using the same amount of reagents but without the sample.

5.3.1.6 *Calculation*

$$\text{Silica, percent by mass} = \frac{[(A - B) - C]}{D} \times 100$$

where

A = mass in g, of platinum crucible with residue of silica before hydro-fluorization;

B = mass in g, of platinum crucible with residue obtained after repeated hydro-fluorization;

C = mass in g, of silica obtained in blank determination, and

D = mass in g, of the sample taken.

5.3.2 *For Magnesite Refractory Materials*.

5.3.2.1 Weigh accurately one gram of the sample in a platinum crucible and fuse it with 6 to 8 g of pure anhydrous sodium carbonate. Extract the melt carefully with 0 to 50 ml of dilute hydrochloric acid in a 500-ml beaker and when dissolution is complete, wash the crucible thoroughly with hot water. Evaporate the solution to dryness on hot-plate and bake for about 20 minutes.

5.3.2.2 Cool the beaker, add 25 to 30 ml of dilute hydrochloric acid, boil and filter. Transfer thoroughly all the residue in the beaker to the filter paper by a jet of hot water and wash it free from acid by means of hot water. Collect the filtrate and washings in the same beaker and preserve it. Further, complete the estimation as described under **5.3.1.2** to **5.3.1.6**.