IS 1760-2 (1991): Methods of chemical analysis of limestone, dolomite and allied materials, Part 2: Determination of silica [MTD 13: Ores and Raw Materials]

BLANK PAGE


IS 1760 ( Part 2 ) : 1991
(Reaffirmed 2001)

भारतीय मानक

# चूना, पत्थर, डोलोमाइट एवं सम्बद्ध सामग्री का रासायनिक विश्लेषण

भाग 2 सिलिका ज्ञात करना

( पहला पुनरीक्षण )

*Indian Standard*

# CHEMICAL ANALYSIS OF LIMESTONE, DOLOMITE AND ALLIED MATERIALS

**PART 2 DETERMINATION OF SILICA**

*( First Revision )*

First Reprint SEPTEMBER 1996

UDC 553·551 : 543 [ 546·284 ]



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

*November* 1991

**Price Group 1**

Methods of Chemical Analysis of Ores, Minerals and Allied Materials for Metallurgical Industry Sectional Committee, MTD 29

## FOREWORD

This Indian Standard ( Part 2 ) ( First Revision ) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Chemical Analysis of Ores, Minerals and Allied Materials for Metallurgical Industry Sectional Committee had been approved by the Metallurgical Engineering Division Council.

IS 1760 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. The committee decided to revise this standard into different parts covering determination of each element in a separate part, which on publication would supersede the determination of that element given in IS 1760 : 1962. 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

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values ( *revised* )'.

*Indian Standard*

# CHEMICAL ANALYSIS OF LIMESTONE, DOLOMITE AND ALLIED MATERIALS

## PART 2 DETERMINATION OF SILICA

*( First 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 are necessary adjunct to this standard:

| *IS No.* | *Title* |
|---|---|
| 1070 : 1977 | Water for general laboratory use ( *second revision* ) |
| 2109 : 1982 | Methods of sampling of dolomite, limestone and other allied materials |

### 3 SAMPLING

3.1 The sample shall be drawn and prepared in accordance with IS 2109 : 1982.

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 : 1977 ) 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 hydrofluorization.

#### 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 filter 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 hydrochoric 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 hydrofluorization;

$B$ = mass in g, of platinum crucible with residue obtained after repeated hydrofluorization;

$C$ = 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. Collectt he 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.

**Bureau of Indian Standards**

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 1986 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

**Copyright**



**Review of Indian Standards**

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards Monthly Additions'.

This Indian Standard has been developed from Doc: No. MTD 29 ( 3801 )

**Amendments Issued Since Publication**

| Amend No. | Date of Issue | Text Affected |
|---|---|---|
| | | |
| | | |
| | | |

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 323 01 31, 323 83 75, 323 94 02

Telegrams: Manaksanstha
(Common to all offices)

| Regional Offices: | | Telephone |
|---|---|---|
| Central | : Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110002 | 323 76 17, 323 38 41 |
| Eastern | : 1/14 C.I.T. Scheme VII M, V.I.P. Road, Maniktola CALCUTTA 700054 | 337 84 99, 337 85 61<br>337 86 26, 337 91 20 |
| Northern | : SCO 335-336, Sector 34-A, CHANDIGARH 160022 | 60 38 43<br>60 20 25 |
| Southern | : C.I.T. Campus, IV Cross Road, MADRAS 600113 | 235 02 16, 235 04 42<br>235 15 19, 235 23 15 |
| Western | : Manakalaya, E9 MIDC, Marol, Andheri (East) MUMBAI 400093 | 832 92 95, 832 78 58<br>832 78 91, 832 78 92 |
| Branches | : AHMADABAD. BANGALORE. BHOPAL. BHUBANESHWAR. COIMBATORE. FARIDABAD. GHAZIABAD. GUWAHATI. HYDERABAD. JAIPUR. KANPUR. LUCKNOW. PATNA. THIRUVANANTHAPURAM. | |

Reprography Unit, BIS, New Delhi, India