

भारतीय मानक प्रारूप
चूना पत्थर, डोलोमाइट एवं सम्बद्ध सामग्री का
रासायनिक विश्लेषण

भाग 4 कार्बन डाइऑक्साइड ज्ञात करना

(दूसरा पुनरीक्षण)

Draft Indian Standard

CHEMICAL ANALYSIS OF LIMESTONE,
DOLOMITE AND ALLIED MATERIALS

PART 4 DETERMINATION OF CARBON DIOXIDE

(Second Revision)

ICS 73.080

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Methods of Chemical Analysis of Metals Sectional Committee, MTD 34

FOREWORD

This draft Indian Standard (Part 4) (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 and 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 different parts covering determination of each element 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 carbon dioxide and has been updated on the basis of experience gained during the past. The other parts in the series are as follows:

IS 1760 Chemical analysis of limestone, dolomite and allied materials:

Part 1 Loss on ignition

Part 2 Determination of silica

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

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 4 DETERMINATION OF CARBON DIOXIDE

*(Second Revision)***1 SCOPE**

1.1 This standard (Part 4) prescribes method for determination of carbon dioxide in the range from 42 to 52 percent in limestone, dolomite and allied materials.

2 REFERENCES

2.1 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.

<i>IS No.</i>	<i>Title</i>
266 : 1993	Sulphuric acid – Specification (<i>third revision</i>)
1070 : 1992	Reagent grade water – Specification (<i>third revision</i>)
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 sample, as given in 3.1 so that it passes through IS Sieve 15 (100 mesh). Dry to a constant mass at $105 \pm 2^\circ\text{C}$ and use it for the purpose of chemical analysis.

4 QUALITY OF REAGENTS

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

5 DETERMINATION OF CARBON DIOXIDE**5.1 Outline of the Method**

A known weight of the sample is made to react with an acid and the liberated carbon dioxide freed from impurities is absorbed in previously weighed soda asbestos bulbs and weighed. From the difference in weight, the percentage of carbon dioxide is calculated.

5.2 Apparatus

The assembly of apparatus is shown in Fig. 1. In order to obtain better pressure for drawing gases through the train, all joints should be mercury sealed.

5.3 Reagents

5.3.1 Dilute Hydrochloric Acid - 1:1 (v/v).

5.3.2 Concentrated Sulphuric Acid - rd = 1.84 (conforming to IS 266).

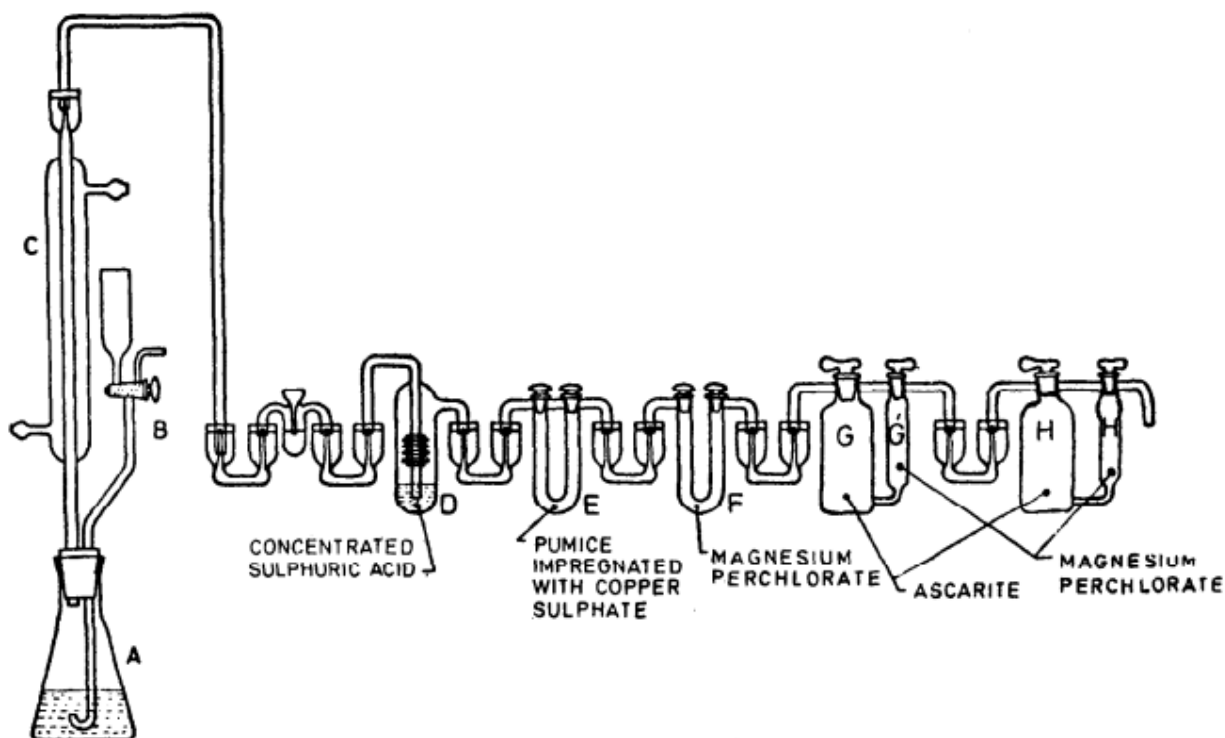
5.3.3 Ascarite or Soda Asbestos

5.3.4 Magnesium Perchlorate - Solid.

5.3.5 Pumice Impregnated with Copper Sulphate - Anhydrous. Crush pumice to approximately 5 mm size, sift free from dust, and transfer 60 g to a casserole. Cover with a concentrated solution of 30 to 35 g of copper sulphate. Evaporate to dryness while constantly stirring and then heat for 3 to 4 hours at 150 to 160°C in an air-bath. Cool in a dessicator and preserve in a glass-stoppered bottle.

5.4 Procedure

5.4.1 Transfer one gram of the accurately weighed sample to the flask *A* and cover it with water. Insert the stopper carrying the separatory funnel *B* and a condenser *C*. Connect the later with *D*, *E* and *F*. Pass air that is free from carbon dioxide through the system until it is judged that all carbon



NOTE - Tubes are compactly arranged along the edge of 150×230 mm board which is supported 130 mm from the table-top upon a tripod base.

FIG. 1 ABSORPTION TRAIN FOR CARBON DIOXIDE

dioxide is removed. Close the stopcock in the separatory funnel, and insert the weighed absorption bulbs *G* and *H* in the train; the latter acts as a guard tube. Half fill the separatory funnel with dilute hydrochloric acid, replace the stopper carrying the air, and see that there is free passage for gases through the train. Open the stopcock in the separatory funnel and run acid into the flask slowly if there is much carbon dioxide and rapidly if there is but little. When effervescence diminishes in the former case, at once in the latter, start a flow of water in the condenser, and heat the flask slowly so as to secure steady but quiet dissolution. When it is judged that carbon dioxide has been boiled out of the solution, remove the flame, increase the current of air and sweep out all carbon dioxide. Disconnect the weighed bulbs, close the inlet and outlet tubes, and place them in the balance case. When cool, open the stopper momentarily and weigh.

6 CALCULATION

$$\text{Carbon dioxide, percent by mass} = \frac{m_2 - m_1}{M} \times 100$$

where

m_1 = mass, in g, of the bulb before the test;

m_2 = mass, in g, of the bulb after the test; and

M = mass, in g, of sample taken.