भारतीय मानक प्रारूप

चूना पत्थर, डोलोमाइट एवं सम्बद्ध सामग्री का रासायनिक विश्लेषण

भाग 5 क्लोराइड ज्ञात करना

(दूसरा पुनरीक्षण)

Draft Indian Standard

CHEMICAL ANALYSIS OF LIMESTONE, DOLOMITE AND ALLIED MATERIALS

PART 5 DETERMINATION OF CHLORIDES

(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 5) (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 five 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 chlorides by volumetric and turbidimetric methods. The turbidimetric method has been specified when the chloride content is present in ppm level. The other parts in the series are as follows:

Part 1 Loss on ignition

Part 2 Determination of silica

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

Part 4 Determination of carbon dioxide

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 5 DETERMINATION OF CHLORIDES

(Second Revision)

1 SCOPE

This standard (Part 5) prescribes methods for determination of chloride content, up to 0.1 percent by volumetric method and in ppm level by turbidimetric method, 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*
266 : 1993	Sulphuric acid – Specification (*third revision*)
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 sample (**3.1**) so that it passes through IS Sieve 15 (100 mesh). Dry to a 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 CHLORIDE BY VOLUMETRIC METHOD

5.1 Outline of the Method

Chloride is precipitated with silver nitrate solution. Excess of silver nitrate is titrated with ammonium thiocyanate solution.

5.2 Reagents

5.2.1 *Dilute Nitric Acid*, 1:2.5 (*v/v*) and 6N.

5.2.2 *Silver Nitrate Solution*, 0.025 N.

Weigh about 2.15 g silver nitrate, dissolve in water and make up to 500 ml in a volumetric flask. Standardize the solution against 0.025 N sodium or potassium chloride solution using potassium chromate solution as indicator. Adjust the normality exactly to 0.025 N.

5.2.3 *Nitrobenzene*

5.2.4 *Ferric Alum Indicator Solution*

Cold saturated solution containing few drops of dilute nitric acid (6 N).

5.2.5 *Ammonium Thiocyanate Solution* (0.025 N)

Weigh about 2.0 g ammonium thiocyanate and dissolve in 1 litre of water in a volumetric flask. Shake well and standardise by titrating against silver nitrate solution 0.025 N using ferric alum indicator. Adjust the normality to exactly 0.025 N.

5.3 Procedure

Weigh accurately about 3.0 g of sample in a 250 ml beaker, add 25 ml nitric acid (6 N) and enough hot water so as to make the volume to about 150 ml. Stir until dissolution is complete. Heat the solution on a hot plate for 2-3 minutes and filter through medium textured filter paper into a 500 ml conical flask. Wash the residue 3 to 4 times with hot water. Add 10 ml of silver nitrate solution (0.025 N) from the burette. Add 2-3 ml of nitrobenzene and 1 ml ferric alum indicator solution and shake vigorously to coagulate the precipitate. Titrate the excess silver nitrate with ammonium thiocyanate solution (0.025 N) until a permanent faint reddish brown colouration appears. From the volume of silver nitrate solution added, subtract the volume of thiocyanate solution required. Take the average of two determinations.

5.4 Calculation

$$\text{Chloride, percent by mass} = \frac{0.0008865 \times V}{M} \times 100$$

where

V = volume, in ml, of silver nitrate used; and

M = mass, in g, of sample taken.

6 DETERMJNATION OF CHLORIDE BY TURBIDIMETRIC METHOD

6.1 Outline of the Method

Silver nitrate is added to the solution and turbidity produced by silver chloride is measured. (This method is applicable only when chloride content is in ppm level).

6.2 Apparatus, Turbidimeter

6.3 Reagents

6.3.1 *Dilute Nitric Acid*, 1:3 (*v/v*).

6.3.2 *Silver Nitrate* 0.025 N.

6.3.3 *Standard Sodium Chloride Solution*, (100 ppm)

Weigh accurately 0.1649 g of sodium chloride previously dried at 105 to 110°C for 2 hours. Dissolve in a small quantity of distilled water and transfer to 1 litre volumetric flask. Make up the volume to the mark.

6.4 Procedure

6.4.1 *Calibration of the Turbidimeter*

Take 5 ml of dilute nitric acid (1:3) in a 100 ml volumetric flask add 5 ml of silver nitrate solution and make up the volume with distilled water. Shake well and use the solution as blank for adjusting the zero of the galvanometer. Take 20 ml of the standard sodium chloride solution in a 100 ml volumetric flask, add 5 ml of dilute nitric acid (1:3) and 50 to 60 ml distilled water. Shake well and add 5 ml silver nitrate solution and make up the volume with distilled water. Shake well and use this turbid solution to adjust the galvanometer deflection to full scale. Run 1.0 ml, 2.5 ml, 5.0 ml, 7.5 ml, 10.0 ml, 15.0 ml, 17.0 ml and 20.0 ml standard chloride solution from a burette into separate 100 ml volumetric flasks. Take the first flask, add 5 ml of dilute nitric acid and 50 to 60ml distilled water. Shake well, add 5 ml of silver nitrate solution and make up the volume with distilled water. Shake well and measure the turbidity after checking the galvanometer 'zero' again. Repeat the above procedure with the remaining solutions. Plot the galvanometer readings against chloride concentration in ppm.

6.4.2 Determination of Chloride Content in Test Sample Weigh accurately 2.0 g of sample and boil with 100 to 150 ml distilled water. Filter and wash with hot distilled water. Collect the filtrate and washings into a 500 ml volumetric flask and make up the volume. Take 50 ml of this solution into a 100 ml volumetric flask, add 5 ml dilute nitric acid and 5 ml silver nitrate solution and make up the volume with distilled water. Shake well and measure the turbidity after checking the galvanometer 'zero'. Read the chloride ion concentration in ppm from the calibration graph prepared earlier and then calculate the chloride content in the sample.

NOTE - Suitable dilutions may have to be carried out such that the galvanometer reading falls within the range 2 to 15 ppm chloride concentration, whenever it is found necessary.

6.5 Calculations

$$\text{Chloride content, percent by mass} = \frac{m}{M} \times 100$$

where

m = mass, in g, of chloride present, and

M = mass, in g, of sample take