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

व्यापक परिचालन मसौदा

हमारा संदर्भ: सीईडी 04/टी-46 **29 नवंबर 2024**

तकनीकी समिति: इमारती चूना और जिप्सम उत्पाद विषय समिति, सीईडी 04

प्राप्तकर्ता:

क) सिविल इंजीनियरी विभाग परिषद्, सीईडीसी के सभी सदस्य
ख) सीईडी 04 के सभी सदस्य
ग) रूचि रखने वाले अन्य निकाय.

प्रिय महोदय/महोदया,

निम्नलिखित भारतीय मानक का मसौदा संलग्न हैं:

प्रलेख संख्या	शीर्षक
सीईडी 04 (27003) WC	**बिल्डिंग लाइम्स के लिए परीक्षण के तरीके** **भाग 1 तनु अम्ल और क्षार में अघुलनशील अवशेषों का निर्धारण, ज्वलन पर हानि, हाइड्रोक्लोरिक एसिड में अघुलनशील अवशेष, सिलिकॉन डाइऑक्साइड, फेरिक और एल्यूमीनियम ऑक्साइड, कैल्शियम ऑक्साइड और मैग्नीशियम ऑक्साइड** **का भारतीय मानक मसौदा** [IS 6932 (**भाग** 1) **का** ***पहला पुनरीक्षण***] ICS 91.100.10

कृपया इस मानक के मसौदे का अवलोकन करें और अपनी सम्मतियॉ यह बताते हुए भेजे कि यदि यह मानक के रूप में प्रकाशित हो तो इस पर अमल करने में आपके व्यवसाय अथवा कारोबार में क्या कठिनाइयाँ आ सकती हैं ।

सम्मतियाँ भेजने की अंतिम तिथि : **30 दिसंबर 2024**

सम्मति यदि कोई हो तो कृपया अधोहस्ताक्षरी को उपरिलिखित पते पर संलग्न फोर्मेट में भेजें या manoj@bis.gov.in पर ईमेल कर दें ।

यदि कोई सम्मति प्राप्त नही होती है अथवा सम्मति में केवल भाषा सम्बन्धी त्रुटि हुई तो उपरोक्त प्रलेख को यथावत अंतिम रूप दिया जाएगा। यदि सम्मित तकनीकी प्रकृति की हुई विषय समिति के अध्यक्ष के परामर्श से अथवा उनकी इच्छा पर आगे की कार्यवाही के लिए विषय समिति को भेजे जाने के बाद प्रलेख को अंतिम रूप दे दिया जाएगा ।

यह प्रलेख भारतीय मानक ब्यूरो की वैबसाइट www.bis.gov.in पर भी उपलब्ध हैं।

धन्यवाद ।

भवदीय,

(द्वैपायन भद्र)
प्रमुख (सिविल इंजीनियरी)

संलग्नक : उपरिलिखित

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

DRAFT IN WIDE CIRCULATION

Our Ref: CED 04/T-46 **29 November 2024**

TECHNICAL COMMITTEE: Building Limes and Gypsum Products Sectional Committee, CED 04

ADDRESSED TO:

a) All Members of Civil Engineering Division Council, CEDC
b) All Members of CED 04
c) All others interests.

Dear Sir/Madam,

Please find enclosed the following document:

Doc No.	Title
CED 04 (27003)WC	**Draft Indian Standard** **Methods of Tests for Building Limes** **Part 1 Determination of Insoluble Residue in Dilute Acid and Alkali, Loss on Ignition, Insoluble Residue in Hydrochloric Acid, Silicon Dioxide, Ferric and Aluminium Oxide, Calcium Oxide and Magnesium Oxide** [*First Revision* of IS 6932 (Part 1)] ICS 91.100.10

Kindly examine the draft standard and forward your views stating any difficulties which you are likely to experience in your business or profession if this is finally adopted as National Standard.

Last Date for Comments: **30 December 2024**

Comments if any, may please be made in the attached format and mailed to the undersigned at the above address or preferably through e-mail to manoj@bis.gov.in.

In case no comments are received or comment received are of editorial nature, you may kindly permit us to presume your approval for the above document as finalized. However, in case of comments of technical in nature are received then it may be finalized either in consultation with the Chairman, Sectional Committee or referred to the Sectional Committee for further necessary action if so desired by the Chairman, Sectional Committee.

The document is also hosted on BIS website www.bis.gov.in.

Thanking you,

Yours faithfully,

(**Dwaipayan Bhadra**)
Head (Civil Engineering)

Encl: As above

FORMAT FOR SENDING COMMENTS ON BIS DOCUMENTS

(Please use A-4 size sheet of paper only and type within fields indicated. Comments on each clause/sub-clause/table/fig etc. be started on a fresh box. Information in column 3 should include reasons for the comments and suggestions for modified working of the clauses when the existing text is found not acceptable. Adherence to this format facilitates Secretariat's work) (Please e-mail your comments to manoj@bis.gov.in)

Doc. No.: **CED 04 (27003)WC**

Title: **Draft Indian Standard Methods of Tests for Building Limes**
Part 1 Determination of Insoluble Residue in Dilute Acid and Alkali, Loss on Ignition, Insoluble Residue in Hydrochloric Acid, Silicon Dioxide, Ferric and Aluminium Oxide, Calcium Oxide and Magnesium Oxide
[*First Revision* of IS 6932 (Part 1)] ICS 91.100.10

LAST DATE OF COMMENT: **30/12/2024**

NAME OF THE COMMENTATOR/ORGANIZATION: ___________________________

Sl. No.	Clause/Para/Table/ Figure No. Commented	Comments/Modified Wordings	Justification of the Proposed Change

BUREAU OF INDIAN STANDARDS

DRAFT FOR COMMENTS ONLY
(*Not to be reproduced without the permission of BIS or used as an Indian Standard*)

Draft Indian Standard

METHODS OF TESTS FOR BUILDING LIMES
PART 1 DETERMINATION OF INSOLUBLE RESIDUE IN DILUTE ACID AND ALKALI, LOSS ON IGNITION, INSOLUBLE RESIDUE IN HYDROCHLORIC ACID, SILICON DIOXIDE, FERRIC AND ALUMINIUM OXIDE, CALCIUM OXIDE AND MAGNESIUM OXIDE

[*First Revision* of IS 6932 (Part 1)]
ICS 91.100.10

Building Lime and Gypsum Products Sectional Committee, CED 04	Last date of Comments: **30 December 2024**

FOREWORD

(*Formal clauses will be added later*)

The role of building limes in construction has been recognized and valued for centuries, from the ancient structures to modern structures. The use of lime as building materials is not only a testament to its versatility and durability but also to its sustainability and environmental benefits. As we continue to seek eco-friendly alternatives in construction, the relevance of lime-based products has become increasingly significant.

Building lime is used in construction for a variety of purposes such as lime washing, lime mortar, lime Plastering, lime Concrete, Rendering and Pointing, soil Stabilization, Restoration of Historic Buildings, Waterproofing and Decoration. Each of these forms of lime serves specific purposes in construction, from creating strong, durable mortar joints to providing breathable, flexible finishes that protect and preserve structures.

A number of Indian Standards on lime building materials covering specifications, code of practices, etc. have been prepared with a view to assisting the lime industry in its development. In line with that, methods of test for building lime, IS 6932 was prepared in eleven parts in the year 1973. In this revision it was decided to review and update the various existing test methods of building lime, taking into consideration the latest international practices and developments in this field and the current practices in the country. In this revision all the amendments are incorporated and reference of all Indian standards has been updated. Ambiguity in the procedure or reporting has been also removed.

This standard (Part 1) covers, the methods of tests for building limes for determination of Insoluble residue in dilute acid and alkali, loss on ignition, Insoluble residue in hydrochloric acid, silicon dioxide, ferric and aluminium oxide, calcium oxide and magnesium oxide. The others standards in the series are:

Part 2	Determination of carbon dioxide content

Part 3	Determination of residue on slaking of quicklime
Part 4	Determination of fineness of hydrated lime
Part 5	Determination of unhydrated oxide
Part 6	Determination of volume yield of quicklime
Part 7	Determination of compressive and transverse strengths
Part 8	Determination of workability
Part 9	Determination of soundness
Part 10	Determination of popping and pitting of hydrated lime
Part 11	Determination of setting time of hydrated lime

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 the same as that of the specified value in this standard.

Draft Indian Standard

METHODS OF TESTS FOR BUILDING LIMES
PART 1 DETERMINATION OF INSOLUBLE RESIDUE, LOSS ON IGNITION, INSOLUBLE MATTER, SILICON DIOXIDE, FERRIC AND ALUMINIUM OXIDE, CALCIUM OXIDE AND MAGNESIUM OXIDE

(*First Revision*)

1 SCOPE

This standard (Part 1) covers the methods of tests for determination of the following requirements of building lime:

a) Insoluble residue in dilute acid and alkali,
b) Loss on ignition,
c) Insoluble residue in hydrochloric acid,
d) Silicon dioxide,
e) Ferric and aluminium oxide,
f) Calcium oxide, and
g) Magnesium oxide.

2 REFERENCES

The standards listed in Annex A contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated in Annex A.

3 SAMPLE PREPARATION

3.1 Sampling shall be carried out as quickly as possible so that the material does not deteriorate. The total time occupied in mixing and preparation of the sample for the test should not exceed two hours. The samples shall be placed immediately in clean, dry, airtight containers. When testing is not to be carried out at once, the samples shall be kept in the airtight containers. Tools such as Shovel, auger, metal or plastic containers shall be of material free from rust and shall be alkali resistant.

3.2 If the sample contains lumps, crush the lime using a mortar and pestle or mechanical grinder to achieve a fine powder. Sieving is used to achieve a uniform particle size for accurate test results. Pass the dried and pulverized lime through a 2.36 mm sieve [*see* IS 460 (Part 1)]. This is a standard procedure for many tests, although specific tests may require different sieve sizes (for example, 300-micron sieve). Use a precision balance to weigh the required quantity of lime for each test. The amount of lime needed will vary depending on the test being conducted. Typically, chemical tests may require 0.5 g to 5.0 g of sieved lime. Use distilled (*see* IS 1070) or deionized water to avoid contamination. Follow the specific water-to-lime ratio as required by the test method. The mixing can be done manually using a spatula or mechanically using a mixer. Ensure that the mixture is homogeneous and free of lumps. For some tests, a paste like consistency may be required.

4 DETERMINATION OF INSOLUBLE RESIDUE IN DILUTE ACID AND ALKALI

4.1 Objective

This test method is intended to determine the portion of lime that does not dissolve in acid and is typically composed of siliceous and other impurities. The determination of insoluble residue is important because a high level of insoluble materials can indicate poor quality lime, which may affect its binding properties, workability, and overall performance in construction.

4.2 Reagents

4.2.1 *Dilute Hydrochloric Acid* (1:1, *v Iv*) – It shall be prepared by diluting hydrochloric acid sp gr 1.16 and conforming to IS 265 with an equal volume of distilled water.

4.2.2 *Sodium Carbonate Solution*

It shall be prepared by dissolving 5 g sodium carbonate (conforming to IS 296) in 100 fill of water.

4.3 Procedure

4.3.1 Weigh 1 g of the sample in a 250 ml beaker, add 10 fill of water to the lime in beaker and make the contents into a slurry. Then add 20 ml to 25 ml of dilute hydrochloric acid and digest the contents for half an hour with occasional gentle agitation. Rinse the sides of the beaker with hot water thus diluting the solution to about 100 ml. Redigest the contents at a temperature just below the boiling point for about 15 min. Filter the contents of the beaker through a filter paper and wash the residue and filter paper twice with dilute hydrochloric acid and then with hot water till they are free from chlorides.

4.3.2 Transfer the filter paper along with the residue to a porcelain dish and add 30 ml to 40 ml of sodium carbonate solution. Cover the dish and digest its contents for about half an hour at a temperature just below the boiling point. Add hot water in small quantities during digestion to make up the loss of volume caused by evaporation. Decant off the supernatant liquid through a filter-paper and wash the residue in the dish a few times with hot water decanting off the liquid into the filter paper. Cover the residue in the dish with sodium carbonate solution and digest again for about 10 min. Filter the contents and wash the residue on the filter paper twice with hot sodium carbonate solution and then with hot water till free from alkali. Again wash the filter paper and the residue twice with dilute hydrochloric acid and then with hot water till free from chlorides.

4.3.3 Transfer the filter paper and the residue to a crucible, dry, ignite and weigh the contents.

4.4 Report of Test Results

The insoluble residue in 5 percent sodium carbonate solution shall be expressed as a percentage on the ignited mass basis. For doing this the total loss on ignition shall be obtained as determined under **5**.

5 DETERMINATION OF LOSS ON IGNITION (LOI)

5.1 Objective

This test method is intended to determine the percentage of weight loss of lime when it is heated to a high temperature, typically due to the decomposition of carbonates (like calcium carbonate) into oxides and the release of gases such as carbon dioxide. Loss on Ignition (LOI) is significant because it provides insight into the purity of lime, the presence of carbonates, and the amount of volatile substances. High LOI values may indicate the presence of unreacted carbonates or organic materials, which can influence the lime's reactivity and strength.

5.2 Equipment

5.2.1 *Muffle Furnace*

The muffle furnace shall capable to provide a high temperature, typically between 900 °C to 1 200 °C, for a specified period of time.

5.2.3 *Analytical Balance*

The analytical balance shall have the range of 200 g with a least count of 0.001 g.

5.2.2 Silica Crucibles and Desiccator

5.3 Procedure

Place 1 g of the prepared sample in a weighed platinum crucible and cover with a lid. Then ignite it at a temperature not less than 1 000 °C to constant mass. The difference between the original mass and the final mass of the sample represents the loss on ignition. This shall he expressed as a percentage of the mass of the sample taken.

5.4 Report of Test Results

$$\text{Loss on ignition, percent by mass} = \frac{100\ (M_1 - M_2)}{M_1}$$

where

M_1 = mass of the original sample, and
M_2 = mass of the sample after ignition.

6 DETERMINATION OF INSOLUBLE RESIDUE IN HYDROCHLORIC ACID

6.1 Objective

This test method is intended to determine the portion of lime that does not dissolve in acid and is typically composed of siliceous and other impurities. The determination of insoluble residue is important because a high level of insoluble materials can indicate poor quality lime, which may affect its binding properties, workability, and overall performance in construction.

6.2 Reagents

6.2.1 *Concentrated Hydrochloric Acid* – sp gr 1.16 (conforming to IS 265).

6.2.2 *Dilute Hydrochloric Acid* – 1:99 (vlv).

6.3 Procedure

6.3.1 Transfer the ignited sample from **5** into an evaporating dish and mix with water into a thin slurry. Add about 10 ml of concentrated hydrochloric acid into the dish and digest the contents with the acid by heating and agitating simultaneously until dissolved. Then evaporate the solution to dryness on a water bath. When the sample is nearly dry, place it in an oven for 1 h. Maintain the temperature of the oven at 110 °C to 120 °C. Take out the sample, cool and add about 10 ml of concentrated hydrochloric acid. Allow it to stand for a few minutes. Add an equal volume of water, cover the dish and place on the water-bath for 10 min. Filter and wash the residue with hot dilute hydrochloric acid and finally twice with hot water. Evaporate the filtrate to dryness and then bake at 110 °C to 120 °C for 1 h. Extract it with hydrochloric acid as before and filter through a second smaller filter paper. Reserve the filtrate for conducting further tests.

6.3.2 Transfer the wet filter papers containing the residue to a weighed platinum crucible, char without allowing the paper to catch fire and finally ignite to constant mass at 1 100 °C. This gives the mass of insoluble matter including silicon dioxide in the sample taken.

6.4 Report of Test Results

The Insoluble residue in hydrochloric acid, including silicon dioxide shall be expressed as a percentage by mass of the sample taken under **6.3.1**.

7 DETERMINATION OF SILICON DIOXIDE

7.1 Objective

This test method is intended to determine Silicon dioxide (SiO_2), which is a major component of sand and other siliceous materials. In lime, the content of SiO_2 is significant because it influences the hydraulic properties of the lime. In hydraulic lime, a certain amount of SiO_2 is necessary for the formation of calcium silicates, which contribute to the setting and hardening properties of the lime. However, too much SiO_2 can make the lime too hydraulic or decrease its workability.

7.2 Reagents

7.2.1 *Hydrofluoric Acid* – approximately 40 percent (v/v).

7.2.2 *Concentrated Sulphuric Acid* – sp gr 1.84 (conforming to IS 266).

7.3 Procedure

7.3.1 Treat the insoluble matter including silicon dioxide obtained under **6.3.1** in the crucible with 5 ml each of water and hydrofluoric acid and one or two drops of concentrated sulphuric acid and then evaporate to dryness. Ignite the residue for 2 min to 3 min and weigh again. Repeat this procedure till the mass obtained is constant within ± 0.1 percent.

7.4 Report of Test Results

7.4.1 The difference between the mass obtained under **7.3** and that obtained under **6.3.1** gives the mass of silicon dioxide.

7.4.2 The silicon dioxide content shall be expressed as a percentage of the mass of the sample taken under **6.3.1**.

8 DETERMINATION OF FERRIC AND ALUMINIUM OXIDES

8.1 Ferric and Aluminium Oxides

8.1.1 *Objective*

This test method is intended to determine the presence of ferric oxide (Fe_2O_3) and aluminum oxide (Al_2O_3) in lime, which is important for understanding its hydraulicity. These oxides contribute to the formation of calcium aluminates and calcium ferrites, which help in the setting process of hydraulic limes. Their content must be carefully controlled, as excessive amounts can affect the color, strength, and setting time of the lime.

8.1.2 *Reagents*

8.1.2.1 *Dilute hydrochloric acid* – 1:1 and 1:3 ($v\ Iv$).

8.1.2.2 *Concentrate nitric acid* – sp gr 1.42 (conforming to IS 264).

8.1.2.3 *Concentrated hydrochloric acid* – sp gr 1.16 (conforming to IS 265).

8.1.2.4 *Methyl red indicator* – 0.1 percent (m/v). It shall be prepared by dissolving 0.1 g of the sodium salt of the methyl red in 100 ml of water. In case the acid is available, 0.1 g is dissolved in 60 ml alcohol and made up to 100 ml with water.

8.1.2.5 *Ammonium hydroxide solution* – sp gr 0.90 (conforming to IS 799).

8.1.2.6 *Ammonium chloride solution* – 2 percent (mlv).

8.1.3 *Procedure*

8.1.3.1 To the filtrate reserved in **6.3.1** add a few drops of concentrated nitric acid and boil the solution until all traces of chlorine' arc gone. If necessary, add 10 ml to 15 ml of concentrated hydrochloric acid and dilute to 200 ml. Then add a few drops of methyl red solution, heat the solution to boiling and neutralize with ammonium hydroxide (diluted towards the end) until the colour of the liquid changes to a distinct yellow. Boil the solution for 1 min to 2 min, allow to settle, filter. Wash the precipitate immediately 2 or 3 times with hot ammonium chloride solution and dry by suction. Reserve the filtrate.

8.1.3.2 Dissolve the precipitate on the filter paper in hot dilute hydrochloric acid (1:3) and collect the solution in the beaker in which the precipitation was made. 'Thoroughly wash the filter paper with hot water into the same beaker. Boil the solution to expel any trace of chlorine and treat the solution with ammonium hydroxide solution for precipitation. Then filter and wash with hot ammonium chloride solution. Combine this filtrate and the one reserved in **8.3.1.1** for the determination of calcium oxide.

8.1.3.3 Ignite the moist filter paper containing the precipitate in a weighed platinum crucible to constant weight at 1 100 °C. This gives the mass of ferric and aluminium oxide (*see* Note) in the sample taken.

NOTE – The precipitate may contain in case of some limes small quantities of phosphorus pentoxide (P_2O_5), manganese oxide (Mn_3O_4) and titanium dioxide (T_iO_2).

8.1.4 *Report of Test Results*

The ferric and aluminium oxide content shall be reported as a percentage of the mass of the sample taken in **6.3.1**.

8.2 Determination of Ferric Oxide

8.2.1 *Apparatus* – Bunsen valve or Jone's reductor.

8.2.2 *Reagents*

8.2.2.1 *Sodium pyrosulphate* ($Na_2S_2O_7$) – solid. Alternatively potassium pyrosulphate ($K_2S_2O_7$) may also be used.

8.2.2.2 *Dilute sulphuric acid* – approximately 5 N.

8.2.2.3 *Standard potassium permanganate solution* ($KMnO_4$) – approximately 0.05 N. It shall be prepared by dissolving 1.6 g of potassium permanganate in 1 000 ml of water. It shall be allowed to stand for a week or more and then filtered through purified asbestos and standardized against standard sodium oxalate solution, prepared by dissolving exactly 0.750 0 g of sodium oxalate in 250 ml of water.

8.2.2.4 *Hydrofluoric acid*

8.2.3 *Procedure*

8.2.3.1 Fuse the combined ferric and aluminium oxides obtained in **8.1.3.3** in a platinum crucible at a very low temperature with 3 g to 4 g of sodium pyrosulphate. Treat the melt with sufficient dilute sulphuric acid to ensure the presence of not less than 5 g of absolute acid and enough water to effect the solution on heating. Evaporate the solution and heat until it fumes copiously. After cooling and dissolving in water, filter, wash and ignite the small amount of silicon dioxide appearing as precipitate in the solution. Then weigh and correct (*see* Note) by volatilizing silicon dioxide by treating with hydrofluoric and sulphuric acid. Add the mass so corrected to the mass of silicon dioxide previously found and deduct from the gross mass of the ferric and aluminium oxides.

8.2.3.2 Reduce the filtrate after removal of silicon dioxide precipitate by zinc using Bunsen valve or Jone's reductor. Filter it through Whatman No. 41 filter paper, wash with hot water and titrate with 0.05 N potassium permanganate solution.

> NOTE – This correction for impurities shall not be made when the hydrofluoric acid correction of the silicon dioxide determination (*see* **6**) has been omitted.

8.2.3.3 *Evaluation*

One millilitre of the 0.05 N potassium permanganate solution is equal to 0.003 992 g of ferric oxide (Fe_2O_3). Accordingly the ferric oxide content shall be calculated, which shall be expressed as a percentage of the mass of the sample taken in **6.3.1**.

8.3 Determination of Aluminium Oxide

The mass of ferric oxide shall be deducted from the total mass of ferric and aluminium oxides obtained under **8.1.4**.

9 DETERMINATIONOF CALCIUM OXIDE CONTENT

9.1 Objective

This test method is intended to determine Calcium oxide (CaO), also known as quicklime, which is the primary active ingredient of lime. Calcium oxide is a crucial parameter because it directly affects the reactivity, setting time, and strength of lime based materials. High levels of CaO are desirable for good quality lime, as they ensure effective carbonation and strength development in lime. Insufficient CaO can lead to poor performance and durability.

9.2 Gravimetric Method

9.2.1 *Reagents*

9.2.1.1 *Dilute hydrochloric acid* – 1:1 ($v\ Iv$). It shall be prepared by diluting hydrochloric acid sp gr 1.16 and conforming to IS 265 with an equal volume of distilled water.

9.2.1.2 *Ammonium hydroxide solution* – sp gr 0.90 approximately.

9.2.1.3 *Ammonium oxalate solution* – 0.1 percent (m/v) and saturated.

9.2.1.4 *Ammonium chloride solution* – 2 percent (mlv).

9.2.2 *Procedure*

9.2.2.1 The combined filtrate reserved under **8.1.3.2** shall be used. The filtrate shall be diluted with distilled water so as to obtain a volume of 500 ml. Use 250 ml of this filtrate in the analysis given under **9.2**.

9.2.2.2 Add a few drops of ammonium hydroxide solution to the aliquot taken and allow the solution to boil. Add 35 ml of a saturated solution of ammonium oxalate to the liquid and continue boiling until the precipitated calcium oxalate assumes a granular form. Then allow to stand for 20 min or until the precipitate has settled and the supernatant liquid is clear. Filter and wash it moderately with ammonium oxalate solution (0.1 percent). Reserve the filtrate.

9.2.2.3 Transfer the wet filter paper and precipitate to a weighed platinum crucible. Burn the filter paper gently over a small flame and later ignite at 1 100 °C until calcium oxalate is converted into calcium oxide. Dissolve the contents, in 10 ml of hot dilute hydrochloric acid and make up the volume to 250 ml. Add ammonium hydroxide to the solution in slight excess which is indicated by persistent smell of ammonia and boil the liquid. If a small amount of aluminium hydroxide separates out, filter it, wash with ammonium chloride; ignite and weigh. Add the weight of aluminium oxide so determined to that found under **8.3**.

9.2.2.4 After the precipitate settles, filter the solution, wash the residue with ammonium oxalate solution (0.1 percent) and ignite in a weighed covered platinum crucible to constant mass. The difference shall give the mass of calcium oxide content in the sample. Combine the filtrate with that reserved in **9.2.2.2** and reserve for conducting further tests.

9.2.3 *Report of Test Results*

The calcium oxide content shall be reported as a percentage of mass of the sample under **6.3.1**.

9.3 Volumetric Method

9.3.1 *Reagents*

9.3.1.1 *Ammonium hydroxide solution* – sp gr 0.90 approximately.

9.3.1.2 *Ammonium oxalate solution* – saturated.

9.3.1.3 *Dilute sulphuric acid* – 1:10 (vlv).

9.3.1.4 *Standard potassium permanganate solution* – It shall be prepared by dissolving 5.634 g of potassium permanganate in 1 000 ml of water. It shall be allowed to stand for a week or more before use, filtered through purified asbestos and then standardized against sodium oxalate solution, prepared by dissolving 0.750 0 g of sodium oxalate in 250 ml of water.

9.3.2 *Procedure*

Take an aliquot of 100 ml from the second half of the filtrate (**9.2.2.1**), make it alkaline with ammonium hydroxide solution, boil vigorously and add 35 ml of boiling saturated solution of ammonium oxalate. Stir vigorously and allow to stand until the precipitation has settled. Filter through a filter paper of 11 cm diameter and wash the precipitate 10 times with hot water. Alternatively, use a Gooch or Sintered glass crucible instead of filter paper. The total quantity of water used for this wash shall not exceed 125 ml. Transfer the filter paper along with the precipitate to the beaker in which the precipitation was done, spreading the paper out against the upper portion of the beaker. Wash the precipitate from the paper with a jet of hot water, fold the paper arid leave it adhering to the upper portion of the beaker. Add 50 ml of dilute sulphuric acid to the beaker, dilute the solution to a volume of 250 ml with hot water, and then heat to 80 °C to 90 °C. Titrate it with the standard potassium permanganate solution until the pink end point is obtained. Drop the folded filter paper (which has been adhering to the side of the beaker) into the liquid, the pink colour of the latter will be discharged. Complete the titration by adding potassium permanganate solution drop by drop until the pink end point is again obtained.

9.3.2.1 *Evaluation*

The calcium oxide equivalent of one millilitre of the standard potassium permanganate solution in g/ml shall be calculated as follows:

$$E = \frac{0.315\ 85}{V}$$

where

E = Calcium oxide equivalent of potassium permanganate solution in g/ml, and
V = Millilitres of potassium permanganate solution required for titration of 0.756 0 g of sodium oxalate.

9.4 EDTA Method

For non-hydraulic type of lime, EDTA method as mentioned in IS 5949 may also be used.

10 DETERMINATION OF MAGNESIUM OXIDE

10.1 Objective

This test method is intended to determine Magnesium oxide (MgO), which is often present in lime as a minor component. The content of MgO is significant because it influences the setting time and durability of lime. While some MgO can improve the flexibility and workability of lime, excessive MgO can lead to expansion and cracking in lime-based materials.

10.2 Reagents

10.2.1 *Dilute Hydrochloric Acid* – 1:1 ($v\ Iv$) – It shall be prepared by diluting hydrochloric acid sp gr 1.16 and conforming to IS 265 with an equal volume of distilled water.

10.2.2 *Diammonium Hydrogen Phosphate Solution* – 25 percent (m/v).

10.2.3 *Ammonium Hydroxide Solution* – sp gr 0.90 approximately.

10.2.4 *Ammonium Nitrate Wash Solution*

It shall be prepared by diluting ammonium hydroxide with distilled water until the solution contains 2.5 percent of ammonia by mass, then adding 3 or 4 drops of concentrated acid (sp gr 1.42).

10.3 Procedure

10.3.1 Acidify the combined filtrate obtained in **9.2.2.4** with hydrochloric acid and concentrate to about 150 ml. Add 10 ml of diammonium hydrogen phosphate solution and cool it by placing in a beaker of ice water. After the solution cools down add ammonium hydroxide solution drop by drop, stir the contents constantly until crystalline precipitate of magnesium ammonium orthophosphate begins to form. Add excess of ammonium hydroxide solution to the extent of 5 percent to 10 percent of the volume of the solution. Continue stirring for several minutes. Allow the liquid to stand in a cold atmosphere for 12 h to 48 h and then filter.

10.3.2 Dissolve the precipitate in hot dilute hydrochloric acid (1:4) and dilute the solution to about 100 ml. Add 1 ml of diammonium hydrogen phosphate solution and then ammonium hydroxide solution drop by drop with constant stirring until the precipitate again begins to form as described in **10.3.1**. Add ammonium hydroxide in moderate excess. Allow it to stand in a cold atmosphere for 12 h to 48 h, filter and wash with ammonium nitrate wash solution. Burn the filter paper off at a low temperature and finally ignite the residue to constant mass at 1 100 °C (A).

10.4 Report or Test Results

The magnesium oxide content shall be calculated from the following formula:

$$\text{Magnesium oxide, percent by max} = \frac{A}{B} \times 36.2$$

where

A = mass of magnesium pyrophosphate, and
B = mass of the ignited sample represented by the aliquot taken for the estimation of calcium oxide under **9.2.2.1.**

10.4.1 The magnesium oxide content shall be expressed as a percentage of mass of the sample taken' under **6.3.1**.

10.5 EDTA Method

For non-hydraulic type of lime, EDTA method as mentioned in IS 5949 may also be used.

ANNEX A

(*Clause* 2)

LIST OF REFERRED INDIAN STANDARDS

IS No.	*Title*
IS 264 : 2005	Nitric acid – Specification (*third revision*)
IS 265 : 2021	Hydrochloric acid – Specification (*fifth revision*)
IS 266 : 1993	Sulphuric acid – Specification (*third revision*)
IS 296 : 2023	Specification for sodium carbonate, anhydrous (*fourth revision*)
IS 460 (Part 1) : 2020	Test sieves — Specification: Part 1 Wire cloth test sieves (*fourth revision*)
IS 799 : 2023	Specification for ammonia, liquor (*third revision*)
IS 1070 : 2023	Reagent grade water – Specification (*fourth revision*)
IS 5949 : 1990	Methods for volumetric determination of calcium and magnesium using EDTA (*second revision*)