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

व्यापक परिचालन मसौदा

हमारा संदर्भः सीईडी 04/टी-50 **04 दिसंबर 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 (27011)WC	**बिल्डिंग लाइम्स के लिए परीक्षण के तरीके भाग 5 निर्जलित ऑक्साइड का निर्धारण का भारतीय मानक मसौदा** [IS 6932 (भाग 5) 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 : **04 जनवरी 2025**

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-50 **04 December 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 (27011)WC	**Draft Indian Standard** **Methods of Tests for Building Limes** **Part 5 Determination of Unhydrated Oxide** [*First Revision* of IS 6932 (Part 5)] 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: **04 January 2025**

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 (27011)WC**

Title: **Draft Indian Standard Methods of Tests for Building Limes**
Part 5 Determination of Unhydrated Oxide
[*First Revision* of IS 6932 (Part 5)] ICS 91.100.10

LAST DATE OF COMMENT: **04/01/2025**

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
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Draft Indian Standard

METHODS OF TESTS FOR BUILDING LIMES PART 5 DETERMINATION OF UNHYDRATED OXIDE

[*First Revision* of IS 6932 (Part 5)]
ICS 91.100.10

Building Lime and Gypsum Products Sectional Committee, CED 04	Last date of Comments: **04 January 2025**

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 5) covers the methods of tests for building limes for determination of unhydrated oxide. The others standards in the series are:

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
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 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 5 DETERMINATION OF UNHYDRATED OXIDE

(First Revision)

1 SCOPE

This standard (Part 5) covers the method of test for determination of unhydrated oxide contents of building lime.

NOTE –In case of dispute the result obtained from physical method is the deciding.

2 REFERENCES

The standards given below 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 agreement based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standard indicated below:

IS No.	*Title*
IS 460 (Part 1) : 2020	Test sieves — Specification: Part 1 Wire cloth test sieves (*fourth revision*)
IS 712 : 1984	Specification for building limes (*third revision*)
IS 1070 : 2023	Reagent grade water – Specification (*fourth revision*)
IS 6932 (Part 1) : 1973 (Part 2) : 1973 (Part 3) : 1973	Method of test for building limes: Determination of insoluble residue, loss on ignition, insoluble matter, silicone dioxide, ferric and aluminium oxide, calcium oxide and magnesium oxide Determination of carbon dioxide content Determination of residue on slaking of quicklime

3 OBJECTIVE

The objective of determining the unhydrated oxide content in hydrated lime is to assess the completeness and effectiveness of the hydration process by quantifying the amount of unreacted calcium oxide (CaO) or magnesium oxide (MgO) remaining in the final product. High levels of unhydrated oxides may indicate inadequate hydration and leading to reduced reactivity.

4 PRINCIPLE

4.1 For Physical Method

The amount of unhydrated oxide in hydrated lime is determined by reacting a known mass of the sample with a specific reagent that selectively reacts with unhydrated oxides. The resulting products are then quantified, typically through titration or gravimetric analysis, to determine the content of unhydrated oxide.

4.2 For Chemical Method

From the percentage of the loss on ignition, calcium oxide, magnesium oxide [*see* IS 6932 (Part 1)], carbon dioxide [*see* IS 6932 (Part 2)] and sulphur trioxide, the value of unhydrated magnesium oxide may be calculated.

5 APPARATUS AND REAGENTS

5.1 Thermobalance – The thermobalance shall consist of an arrangement wherein the sample can be heated, automatically weighed at regular intervals and the temperature of heating and the mass of the sample can be automatically recorded. The arrangement for stopping the temperature rise at any point shall be provided within this set-up.

5.2 Crucible – The crucible shall be made of a material which does not in any way react with lime up to 1 400 °C.

5.3 Support – The support over which the crucible is mounted before sliding over into the furnace shall be made of sintered or fused alumina.

5.4 Furnace – The furnace should be such that its temperature can be raised at a sufficient controllable rate to 1 100 °C, preferably 1 400 °C. This furnace may be raised and lowered as required with the help of a chain arrangement.

5.5 Autoclave – The autoclave shall be made of a high pressure steam vessel provided with a thermometer well. The autoclave cover lid shall be equipped with an automatic pressure control and a safety valve device. A vent valve shall be provided on the lid of the autoclave so as to allow the steam to escape out whenever required. The pressure gauge shall have a dial with 114 mm diameter and shall be graduated from 0 to 40 kg/cm^2 with scale divisions of not more than 0.5 kg/cm^2. The heating may be controlled such that pressure of 21 kg/cm^2 can be obtained in 45 min to 75 min. The autoclave shall be designed to permit the gauge pressure to drop from 21 kg/cm^2 to less than 1 kg/cm^2 in $1\frac{1}{2}$ h after the supply has been cut off.

5.6 Balance – Analytical balance with a least count of 0.001 g.

5.7 Oven – Capable of maintaining a temperature of 105 °C ± 5 °C.

5.8 Vacuum Desiccactor – Capable of maintaining a pressure of 10 mm Hg (0.0136 kg/cm^2)

5.9 Hydrochloric Acid (HCl) – Concentrated, reagent-grade, for reacting with unhydrated oxides 1:1 (*v/v*).

5.10 Barium Chloride Solution – 10 percent (*m/v*).

5.11 Distilled Water – For diluting reagents and cleaning apparatus.

6 SAMPLE PREPARATION

6.1 General

6.1.1 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.

6.1.2 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 (e.g., 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 and physical test require 100 g to 5 000 g. Use distilled (*see* IS 1070) or deionized water to avoid contaminaton. 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.

6.2 The sample shall be slaked at a temperature of 25 ± 2°C, the quantity of water will be sufficient to make a stiff putty. The sample shall then be dried in vacuum till its mass becomes constant.

6.3 Before carrying out the analysis, the hydrated lime sample shall be dried. For this purpose about 25 g of the sample shall be required to be kept in vacuum until its mass becomes constant.

7 PROCEDURE BASED ON PER PHYSICAL METHOD

7.1 Dry the hydrated magnesiun lime powder in a vacuum desiccactor for 1 h at a pressure of 10 mm Hg (0.136 kg/cm^2). Accurately weigh 1.000 g of sample in a crucible and analyze thermogravimetrically at any heating rate less than 200 °C/h. When the temperature reaches 380 °C, discontinue the heating programme and keep the temperature constant until no further loss in mass takes place. Restart heating after 10 min and continue the programme until the mass finally becomes constant.

7.2 Accurately weigh 1.000 g portion from the dried hydrated magnesium lime sample (*see* **6.4**) in a platinum crucible. Cover this with another loosely fitting crucible. Place inside the autoclave. Heat the autoclave carefully to raise the pressure to 20.746 kg/cm^2 in about 3 h and keep it constant for another hour. Release the pressure gradually. Dry the crucible in vacuum, as previously, until the mass becomes constant. Transfer the contents of the platinum crucible into the small crucible quantitatively and analyze thermogravimetrically as before.

7.3 Report of Test Results

a) $[\text{Loss from (280 to 380)}]\frac{111}{100} \times 3.238 = \text{mass of } \text{Mg(OH)}_2 \text{ in the hydrate } = A$;
b) mass of Mg(OH)_2 in the autoclaved hydrate $= B$;
c) increase in mass of $\text{Mg(OH)}_2 = (B - A)$; and
d) Unhydrated $\text{MgO} = (\text{B} - \text{A}) \times 0.692\,0$.

8 PROCEDURE BASED ON CHEMICAL METHOD

8.1 Determination of Sulphur Trioxide Content

Accurately weigh about 2.5 g of the sample, transfer it into a beaker and add 10 ml of cold water. Stir with a glass rod to ensure that all lumps are broken. Add 20 ml dilute hydrochloric acid and heat carefully until the dissolution is complete. Filter through a small filter paper and wash the residue thoroughly with hot water.

8.2 Dilute the filtrate to about 250 ml. Heat this solution carefully and bring it to boiling. Add 10 ml of hot barium chloride solution drop by drop with constant agitation. Boil for further 10 min. Stir well and allow to stand for overnight. Filter through filter paper No. 42 Whatman or its equivalent filter paper and wash with boiling water. Place the filter paper along with its contents in a weighed platinum crucible. Slowly incinerate the paper without inflaming. Ignite to constant mass and weigh as barium sulphate ($BaSO_4$). Multiply by 0.343 to get SO_2.

8.3 The sulphur trioxide content shall be reported as a percentage of mass of the sample taken.

8.4 Calculations

a) Subtract carbon dioxide from the loss on ignition amount of chemically combined water (X);
b) Calculate the calcium oxide equivalents of carbon dioxide and sulphur trioxide by multiplying their determined values by 1.275 and 0.700 respectively, and subtract the resultants from the total calcium oxide obtained by estimation. Calculate the water equivalent of the remaining calcium oxide by multiplying it with 0.321 3 (γ);
c) Subtract γ from X to obtain the remaining combined water (Z), and calculate the magnesium oxide equivalent to it by multiplying with 2.238; and
d) Subtract Z from the total magnesium oxide obtained by estimation. This gives the percentage of unhydrated magnesium oxide.