IS 6932-5 (1973): Method of test for building limes Determination of unhydrated oxide, Part v: Determination of unhydrated oxide [CED 4: Building Limes and Gypsum Products]

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IS : 6932 (Part V) - 1973
(Reaffirmed 2000)

Indian Standard

METHODS OF TESTS FOR BUILDING LIMES

PART V DETERMINATION OF UNHYDRATED OXIDE

(Second Reprint APRIL 1990)

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Gr 1 *February* 1974

AMENDMENT NO. 1 MAY 2010
TO
IS 6932 (PART 5) : 1973 METHODS OF TESTS FOR BUILDING LIMES

PART 5 DETERMINATION OF UNHYDRATED OXIDE

(*Page* 1, *clause* **2.1**, *line* 2) — Substitute 'IS 712 : 1984†' *for* 'IS : 712-1973†'.

(*Page* 1, *clause* **2.2**, *line* 1) — Substitute 'IS 1070 : 1992‡' *for* 'IS : 1077-1960‡'.

(*Page* 1, *footnote marked* †) — Substitute the following for the existing:

'†Specification for building limes (*third revision*).'

(*Page* 1, *footnote marked* ‡) — Substitute the following for the existing:

'‡Specification for reagent grade water (*third revision*).'

(*Page* 3, *clause* **3.6.2.1**, *line* 2) — Substitute 'IS 712 : 1984‡' *for* 'IS : 712-1973‡'.

(*Page* 3, *footnote marked* ‡) — Substitute the following for the existing:

'‡Specification for building limes (*third revision*).'

(CED 4)

Reprography Unit, BIS, New Delhi, India

Indian Standard

METHODS OF TESTS FOR BUILDING LIMES

PART V DETERMINATION OF UNHYDRATED OXIDE

0. FOREWORD

0.3 This Indian Standard (Part V) was adopted by the Indian Standards Institution on 22 March 1973, after the draft finalized by the Building Limes Sectional Committee had been approved by the Civil Engineering Division Council.

0.2 Hitherto, methods of tests for assessing qualitative requirements of building limes were included in IS : 712-1964. For facilitating the use of these tests it has been decided to print these tests as different parts of a separate Indian Standard. This part covers determination of unhydrated oxide of building limes.

0.3 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*.

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1. SCOPE

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

2. GENERAL

2.1 Preparation of the Sample — The sample shall be prepared in accordance with **7.2** of IS : 712-1973†.

2.2 The distilled water (*see* IS : 1077-1960‡) shall be used where use of water as a reagent is intended.

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*Rules for rounding off numerical values (*revised*).

†Specification for building limes (*second revision*).

‡Specification for water, distilled quality (*revised*). (Since revised).

3. DETERMINATION OF UNHYDRATED OXIDE

3.1 The sample shall be slaked at a specified temperature of 25±2°C [*see* **2.3** of IS : 6932 (Part III)-1973*], 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.

3.2 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.

3.3 Apparatus

3.3.1 *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.

3.3.2 The arrangement for stopping the temperature rise at any point shall be provided within this set-up.

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

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

3.3.5 *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.

3.3.6 *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/cm² with scale divisions of not more than 0·5 kg/cm². The heating may be controlled such that pressure of 21 kg/cm² can be obtained in 45 to 75 minutes. The autoclave shall be designed to permit the gauge pressure to drop from 21 kg/cm² to less than 1 kg/cm² in 1½ hours after the supply has been cut off.

3.4 Procedure

3.4.1 Dry the hydrated magnesiun lime powder in a vacuum desiccactor for 1 hour at a pressure of 10 mmHg (0·0136 kg/cm²). Accurately weigh

*Method of tests for building limes: Part III Determination of residue on slaking of quicklime.

1·000 g of the sample in a crucible and analyse 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 minutes and continue the programme until the mass finally becomes constant.

3.4.2 Accurately weigh 1·000 g portion from the dried hydrated magnesium lime sample (*see* **3.2**) 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/cm² in about 3 hours 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 analyse thermogravimetrically as before.

3.5 Calculation

i) $[\text{ Loss from (280 to 380) }] \frac{111}{100} \times 3{\cdot}238 = \text{mass of } Mg(OH)_2 \text{ in the hydrate} = A$

ii) mass of $Mg(OH)_2$ in the autoclaved hydrate $= B$

iii) increase in mass of $Mg(OH)_2 = (B - A)$

iv) Unhydrated $MgO = (B - A) \times 0{\cdot}692\,0$

3.6 Alternative Method Based on Chemical Analysis

3.6.0 The amount of unhydrated magnesium oxide in the sample may also be determined by the following alternative procedure.

NOTE — This method is being included in the standard primarily because it is felt that the instruments required for the earlier test method may not be immediately available in the testing laboratories.

3.6.1 *Principle of Method* — From the percentage of the loss on ignition, calcium oxide, magnesium oxide [*see* IS : 6932 (Part I) - 1973]*, carbon dioxide [*see* IS : 6932 (Part II) - 1973]† and sulphur trioxide, (*see* **3.6.2**) the value of unhydrated magnesium oxide may be calculated as given in **3.6.2.5**.

3.6.2 *Determination of Sulphur Trioxide Content*

3.6.2.1 *Sample for analysis* — The sample selected in accordance with **7.3** of IS : 712-1973‡ shall be used for analysis.

*Methods of tests for building limes: Part I Determination of insoluble residue, loss on ignition, insoluble matter, silicon dioxide, ferric and aluminium oxide, calcium oxide and magnesium oxide.

†Methods of tests for building limes: Part II Determination of carbon dioxide content.

‡Specification for building limes (*second revision*).

3.6.2.2 *Reagents*

a) *Dilute hydrochloric acid* — 1 : 1 (*v/v*).

b) *Barium chloride solution* — 10 percent (*m/v*).

3.6.2.3 *Procedure* — 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 diosolution is complete. Filter through a small filter paper and wash the residue thoroughly with hot water.

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 minutes. 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_3.

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

3.6.2.5 *Calculations*

a) Subtract carbon dioxide from the loss on ignition. This gives the 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 (Y).

c) Subtract Y from X to obtain the remaining combined water (Z), and calculate the magnesium oxide equivalent to it by multiplying with 2·238.

d) Subtract Z from the total magnesium oxide obtained by estimation. This gives the percentage of unhydrated magnesium oxide.

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