

IS : 6932 ( Part I ) - 1973

*Indian Standard*

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**

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**BUREAU OF INDIAN STANDARDS**  
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## *Indian Standard*

### 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

### 0. FOREWORD

**0.1** This Indian Standard ( Part I ) 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 chemical properties 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 I ) covers the methods of tests for determination of the following of building lime.

- a) Insoluble residue,
- b) Loss on ignition,
- c) Insoluble matter,

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

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- d) Silicon dioxide,
- e) Ferric and aluminium dioxide,
- f) Calcium oxide, and
- g) Magnesium oxide.

## 2. GENERAL

**2.1 Treatment of the Sample** — The sample, for carrying out various tests, shall be prepared in accordance with 7.2 of IS : 712-1973\*.

**2.2** The distilled water ( see 1070-1960† ) shall be used where use of water as a reagent is intended.

## 3. DETERMINATION OF INSOLUBLE RESIDUE INCLUDING SILICON DIOXIDE IN HYDROCHLORIC ACID

### 3.1 Reagents

**3.1.1 Dilute Hydrochloric Acid** — 1 : 1 ( v/v ). It shall be prepared by diluting hydrochloric acid sp gr 1.16 and conforming to IS : 265-1962‡ with an equal volume of distilled water.

**3.1.2 Sodium Carbonate Solution** — It shall be prepared by dissolving 5 g sodium carbonate ( conforming to IS : 296-1965§ ) in 100 ml of water.

### 3.2 Procedure

**3.2.1** Weigh 1 g of the sample obtained in accordance with 2.1 in a 250-ml beaker, add 10 ml of water to the lime in beaker and make the contents into a slurry. Then add 20 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 minutes. 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.

**3.2.2** Transfer the filter paper along with the residue to a porcelain dish and add 30 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

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

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

‡Specification for hydrochloric acid ( *revised* ).

§Specification for sodium carbonate, anhydrous ( *revised* ).

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 minutes. 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.

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

**3.3 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 4.

#### 4. DETERMINATION OF LOSS ON IGNITION

**4.1 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 be expressed as a percentage of the mass of the sample taken.

**4.2 Calculations** — 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.

#### 5. DETERMINATION OF INSOLUBLE MATTER INCLUDING SILICON DIOXIDE

##### 5.1 Reagents

**5.1.1 Concentrated Hydrochloric Acid** — sp gr 1.16 (conforming to IS : 265-1962\* ).

**5.1.2 Dilute Hydrochloric Acid** — 1 : 99 (v/v).

##### 5.2 Procedure

**5.2.1** Transfer the ignited sample from 4 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

\*Specification for hydrochloric acid (revised).

the solution to dryness on a water-bath. When the sample is nearly dry, place it in an oven for 1 hour. Maintain the temperature of the oven at 110 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 minutes. 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 to 120°C for 1 hour. Extract it with hydrochloric acid as before and filter through a second smaller filter paper. Reserve the filtrate for conducting further tests.

**5.2.2** 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.

**5.3 Report of Test Results** — The insoluble matter including silicon dioxide shall be expressed as a percentage by mass of the sample taken under 5.2.1.

## 6. DETERMINATION OF SILICON DIOXIDE

### 6.1 Reagents

**6.1.1 Hydrofluoric Acid** — approximately 40 percent (  $v/v$  ).

**6.1.2 Concentrated Sulphuric Acid** — sp gr 1.84 ( conforming to IS : 266-1966\* ).

**6.2 Procedure** — Treat the insoluble matter including silicon dioxide obtained under 5.2.1 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 to 3 minutes and weigh again. Repeat this procedure till the mass obtained is constant within  $\pm 0.1$  percent.

### 6.3 Report of Test Results

**6.3.1** The difference between the mass obtained under 6.2 and that obtained under 5.2.1 gives the mass of silicon dioxide.

**6.3.2** The silicon dioxide content shall be expressed as a percentage of the mass of the sample taken under 5.2.1.

## 7. DETERMINATION OF FERRIC AND ALUMINIUM OXIDES

### 7.1 Reagents

**7.1.1 Dilute Hydrochloric Acid** — 1 : 1 and 1 : 3 (  $v/v$  ).

\*Specification for sulphuric acid ( revised ).

**7.1.2 Concentrate Nitric Acid** — sp gr 1.42 (conforming to IS: 264-1968\* ).

**7.1.3 Concentrated Hydrochloric Acid** — see 5.1.1.

**7.1.4 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.

**7.1.5 Ammonium Hydroxide Solution** — sp gr 0.90 (conforming to IS: 799-1955† ).

**7.1.6 Ammonium Chloride Solution** — 2 percent (*m/v*).

## 2 Procedure

**7.2.1** To the filtrate reserved in 5.2.1 add a few drops of concentrated nitric acid and boil the solution until all traces of chlorine are gone. If necessary, add 10 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 to 2 minutes, allow to settle, filter. Wash the precipitate immediately 2 or 3 times with hot ammonium chloride solution and dry by suction. Reserve the filtrate.

**7.2.2** 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 7.2.1 for the determination of calcium oxide.

**7.2.3** Ignite the moist filter paper containing the precipitate in a weighed platinum crucible to constant weight at 1100°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 ( $Mn_2O_4$ ) and titanium dioxide ( $TiO_2$ ).

**7.3 Report of Test Results** — The ferric and aluminium oxide content shall be reported as a percentage of the mass of the sample taken in 5.2.1.

## 7.4 Determination of Ferric Oxide

**7.4.1 Apparatus** — Bunsen valve or Jone's reductor.

\*Specification for nitric acid (*first revision*).

†Specification for ammonia, liquor, technical.

### **7.4.2 Reagents**

**7.4.2.1 Sodium pyrosulphate** ( $\text{Na}_2\text{S}_2\text{O}_7$ ) — solid. Alternatively potassium pyrosulphate ( $\text{K}_2\text{S}_2\text{O}_7$ ) may also be used.

**7.4.2.2 Dilute sulphuric acid** — approximately 5 N.

**7.4.2.3 Standard potassium permanganate solution** ( $\text{KMnO}_4$ ) — 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 0.7 500 g of sodium oxalate in 250 ml of water.

**7.4.2.4 Hydrofluoric acid**

### **7.4.3 Procedure**

**7.4.3.1** Fuse the combined ferric and aluminium oxides obtained in 7.2.3 in a platinum crucible at a very low temperature with 3 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 redissolving 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.

**7.4.3.2** 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.

**7.4.3.3 Evaluation** — One millilitre of the 0.05 N potassium permanganate solution is equal to 0.003 992 g of ferric oxide ( $\text{Fe}_2\text{O}_3$ ). Accordingly the ferric oxide content shall be calculated, which shall be expressed as a percentage of the mass of the sample taken in 5.2.1.

**7.5 Determination of Aluminium Oxide** — The mass of ferric oxide shall be deducted from the total mass of ferric and aluminium oxides obtained under 7.3.

## **8. DETERMINATION OF CALCIUM OXIDE CONTENT**

### **8.1 Gravimetric Method**

#### **8.1.1 Reagents**

**8.1.1.1 Dilute hydrochloric acid** — *see* 3.1.1.

**8.1.1.2 Ammonium hydroxide solution** — sp gr 0.90 approximately.

**8.1.1.3 Ammonium oxalate solution** — 0.1 percent (*m/v*) and saturated.

**8.1.1.4 Ammonium chloride solution** — see 7.1.6.

### **8.1.2 Procedure**

**8.1.2.1** The combined filtrate reserved under 7.2.2 shall be used. The filtrate shall be diluted with distilled water so as to obtain a volume of 500 ml. Use 25 ml of this filtrate in the analysis given under 8.2.

**8.1.2.2** 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 minutes 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.

**8.1.2.3** 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 7.5.

**8.1.2.4** 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 8.1.2.2 and reserve for conducting further tests.

**8.1.3 Report of Test Results** — The calcium oxide content shall be reported as a percentage of mass of the sample under 5.2.1.

## **8.2 Volumetric Method**

### **8.2.1 Reagents**

**8.2.1.1 Ammonium hydroxide solution** — see 8.1.1.2.

**8.2.1.2 Ammonium oxalate solution** — saturated.

**8.2.1.3 Dilute sulphuric acid** — 1 : 10 (*v/v*).

**8.2.1.4 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.



**8.2.2 Procedure** — Make second half of the filtrate ( see 8.1.2.1 ) alkaline with ammonium hydroxide solution. Boil and add 35 ml of boiling saturated solution of ammonium oxalate, stir vigorously and allow to stand until the precipitate 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 and 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 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.

**8.2.2.1 Evaluation** — One millilitre of the standard potassium permanganate solution would be approximately equivalent to 0.005 g of calcium oxide.

**8.3 EDTA Method** — For non-hydraulic type of lime, EDTA method as mentioned in IS : 5949-1970\* may also be used.

## 9. DETERMINATION OF MAGNESIUM OXIDE

### 9.1 Reagents

**9.1.1 Dilute Hydrochloric Acid** — 1 : 1 and 1 : 4 ( v/v ) ( see 3.1.1 ).

**9.1.2 Diammonium Hydrogen Phosphate Solution** — 25 percent ( m/v ).

**9.1.3 Ammonium Hydroxide** — see 8.1.1.2.

**9.1.4 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 nitric acid ( sp gr 1.42 ).

### 9.2 Procedure

**9.2.1** Acidify the combined filtrate obtained in 8.1.2.4 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

\*Method for volumetric determination of calcium and magnesium using EDTA.

solution to the extent of 5 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 to 48 hours and then filter.

**9.2.2** 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 **9.2.1**. Add ammonium hydroxide in moderate excess. Allow it to stand in a cold atmosphere for 12 to 48 hours, 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 1100°C ( A ).

**9.3 Report of Test Results** — The magnesium oxide content shall be calculated from the following formula:

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

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 **8.1.2.1**.

**9.3.1** The magnesium oxide content shall be expressed as a percentage of mass of the sample taken under **5.2.1**.

**9.4 EDTA Method** — For non-hydraulic type of lime, EDTA method as mentioned in IS : 5949-1970\* may also be used.

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\*Method for volumetric determination of calcium and magnesium using EDTA.

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**AMENDMENT NO. 2      MARCH 1984**

**TO**

**IS : 6932 ( Part I ) - 1973    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**

**Alterations**

( Page 1, clause 1.1 ) — Substitute the following for the existing clause:

**‘1.1** This standard ( Part I ) 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.’

( Page 2, clause 3, heading ) — Substitute the following for the existing heading:

**‘DETERMINATION OF INSOLUBLE RESIDUE IN DILUTE ACID  
AND ALKALI’**

( Page 3, clause 5, heading ) — Substitute the following for the existing heading:

**‘DETERMINATION OF INSOLUBLE RESIDUE IN HYDROCHLO-  
RIC ACID’**

( Page 4, clause 5.3, line 1 ) — Substitute ‘residue’ for ‘matter’.

( Page 7, clause 8.1.2.1, line 3 ) — Substitute ‘250 ml’ for ‘25 ml’.

( Page 8, clause 8.2.2, first and second sentences ) — Substitute the following for the existing sentences:

**‘8.2.2 Procedure** — Take an aliquot of 100 ml from the second half of the filtrate (8.1.2.1), 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.'

( Page 8, clause 8.2.2.1 ) — Substitute the following for the existing clause:

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

$$E = \frac{0.31385}{V}$$

where

$E$  = Calcium oxide equivalent of potassium permanganate solution in g/ml, and

$V$  = Millilitres of potassium permanganate solution required for titration of 0.7560 g of sodium oxalate.

( BDC 4 )