IS 1514 (1990): Methods of sampling and test for quick lime and hydrated lime [CHD 1: Inorganic Chemicals]

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IS 1514 : 1990
(Reaffirmed 2010)
Edition 2.1
(1998-01)

भारतीय मानक

अनबुझे और बुझे चूने के नमूने लेने और परीक्षण पद्धतियाँ

(पहला पुनरीक्षण)

Indian Standard

METHODS OF SAMPLING AND TEST FOR QUICKLIME AND HYDRATED LIME

(First Revision)

(Incorporating Amendment No. 1)

UDC 666.924.1/.3.620.113

BUREAU OF INDIAN STANDARDS
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FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards on 25 May 1990, after the draft finalized by the Acids, Alkalis and Halides Sectional Committee had been approved by the Chemical Division Council.

Quicklime and hydrated lime are among the most widely used raw materials in the chemical industry. Owing to the diversity of sources and variations in the practice of manufacture and storage, there exists a considerable variation in the quality of lime.

Lime in large quantities is used for softening of water. Besides, lime is used in calcium carbide, pulp and paper, tanneries, glass and sugar industries and in the manufacture of bleaching powder and greases. Since the consumption of lime has increased enormously, the industries require a standardized raw material to produce quality products.

This standard was first published in 1959. In this revision, methods for the determination of free calcium oxide and moisture have been incorporated.

This edition 2.1 incorporates Amendment No. 1 (January 1998). Side bar indicates modification of the text as the result of incorporation of the amendment.

In reporting the result 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 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS OF SAMPLING AND TEST FOR QUICKLIME AND HYDRATED LIME

(First Revision)

1 SCOPE

1.1 This standard prescribes the methods of sampling and test for quicklime and hydrated lime used for softening water and in various chemical industries for the manufacture of calcium carbide, paper, glass, sugar, greases, bleaching powder and in the tanning of leather. It covers the methods of determining carbonate, volatile matter at 105°C, loss on ignition, available lime, acid insoluble matter, (as SiO_2) iron oxide, alumina, total calcium oxide, magnesium oxide, sulphates, dead burnt lime, moisture and free calcium oxide. Screen analysis of the material is also prescribed.

1.2 This standard does not deal with materials but specifies the methods for determining whether they comply with the requirements of individual standards.

1.3 Should any inconsistency exist between the requirements of this standard and those of the standard for an individual material, the latter shall prevail.

2 REFERENCES

2.1 The following Indian standards are necessary adjuncts to this standard:

IS No.	*Title*
265 : 1987	Specification for hydrochloric acid (*third revision*)
296 : 1986	Specification for sodium carbonate, anhydrous (*third revision*)
323 : 1959	Specification for rectified spirit (*revised*)
1070 : 1977	Specification for water for general laboratory use (*second revision*)

3 TERMINOLOGY

3.0 For the purpose of this standard, the following definitions shall apply.

3.1 Available Lime

Denotes such of those constituents of quicklime and hydrated lime as enter into reaction under the conditions of the method prescribed under **9.**

3.2 Total Calcium Oxide

Denotes such of those constituents of quicklime and hydrated lime as enter into reaction under the conditions of the method prescribed under **12.**

3.3 Dead Burnt Lime

Denotes such of those constituents of quicklime and hydrated lime as are calculated by the method prescribed under **15.**

3.4 Free Calcium Oxide

Denotes such constituents of hydrated lime which are capable of being hydrated by steam at atmospheric pressure.

4 SAMPLING

4.1 General Precautions

4.1.1 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

4.1.2 Samples shall not be taken at a place exposed to weather or from damaged or broken packages

4.1.3 The sampling instrument shall be clean and dry when used.

4.1.4 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

4.1.5 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

4.1.6 The samples shall be placed in clean, dry and air-tight glass or other suitable containers on which the material has no action.

4.1.7 The sample containers shall be of such a size that they are almost completely filled by the sample.

4.1.8 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling,

month of manufacture, and other important particulars of the consignment.

4.2 Sampling from Bulk in Heaps or Wagons

4.2.1 Samples shall be taken from each heap or wagon.

4.2.2 *Procedure*

Unless otherwise agreed to between the purchaser and the vendor, draw several samples from each heap or wagon by means of a scoop of about 0.5 kg capacity from different parts, namely, the front, middle and back and at different depths so that the sample taken is fully representative of the bulk. Place the separate samples thus collected together in a covered cask to obtain an average sample. Empty out the contents of the cask on a level, clean and hard surface, spread them out flat and scoop the mass together into a cone. Flatter the cone and divide the mass obtained into four equal parts. Remove the two diagonally opposite parts. Mix the two remaining parts together and form a cone out of it again. Repeat the operation of coning and quartering until finally about one kilogram of the average sample representative of the heap or wagon is left.

4.3 Sampling from Packages

4.3.1 *Scale of Sampling*

4.3.1.1 *Lot*

All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute the lot. If a consignment is declared or known to consist of different batches of manufacture, the batches shall be marked separately and the containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot. In the case of a consignment drawn from a continuous process, 1 000 containers (or 100 metric tonnes) of the material shall constitute the lot.

NOTE — Samples shall be tested from each lot for ascertaining the conformity of the material to the requirements of the specification.

4.3.1.2 The number n of containers to be chosen from lots of different sizes (N) shall be as indicated in Table 1.

Table 1 Number of Containers to be Selected for Sampling from Different Sizes of Lots

Lot Size (*N*)	No. of Containers to be Chosen (*n*)
(1)	(2)
2 to 15	2
16 to 40	3
41 to 65	4
66 to 110	7
111 and above	10

4.3.1.3 These containers shall be chosen at random from the lot and in order to ensure the randomness of selection, some random number table, as agreed to between the purchaser and the supplier, shall be used. In case such a table is not available, the procedure given in **4.3.1.4** shall be adopted.

4.3.1.4 Arrange all the containers in the lot in a systematic manner and starting from any container, count them as 1, 2 ..... etc, up to r, and so on. Every rth container thus counted shall be withdrawn from the lot to give a sample for test, where

$$r = \frac{N}{n}$$

NOTE — In case r comes out to be fractional number, its value shall be taken to be equal to the integral part of it.

4.4 Test Samples

4.4.1 *Test Samples from Heaps or Wagons*

A small but equal amount of material shall be collected from each average sample. The material so collected from every 10 average samples or parts thereof, shall be mixed together to form composite samples. Each composite sample shall weigh about 600 g and shall be divided into 3 equal parts, one for the purchaser, another for the supplier and the third for the referee. The remaining portions of each average sample form individual test samples and, like the composite sample, they too shall be divided into three equal parts.

4.4.2 *Test Samples from Packages*

To prepare a set of test samples, draw with an appropriate sampling instrument, small portions of the material from different parts of each container selected under 4.3.1.2 and freshly opened. The total quantity of the material drawn from each container shall be sufficient to conduct the tests for all the requirements of individual specification and shall not exceed one kilogram.

4.4.2.1 Thoroughly mix all portions of the material drawn from the same container. Out off these portions, a small but equal quantity shall be taken from each selected container and shall be well mixed so as to form a composite sample weighing not less than 600 g. This composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

4.4.2.2 The remaining portions of the material from each selected container (after a small quantity needed for formation of the composite sample has been taken out) shall be divided into three equal parts, each part weighing not less than 50 g.

These parts shall be immediately transferred in thoroughly dried bottles which shall then be sealed air-tight with stoppers and labelled with all the particulars of sampling given under **4.1.7.** The material in each such sealed bottle shall constitute a test sample. These individual samples shall be separated into three identical sets of test samples in such a way that each set has a test sample representing each container selected under **4.3.1.2**. One of these three sets shall be marked for the purchaser, another for the supplier and the third for the referee.

4.5 Referee Sample

Referee sample shall consist of the composite sample (*see* **4.4.1** and **4.4.2.1**) and a set of test samples (*see* **4.4.1** and **4.4.2.2**) marked for this purpose and shall bear the seals of the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of a dispute between the two.

4.6 Number of Tests

4.6.1 Tests for the determination of available lime and/or total calcium oxide content shall be conducted individually on each of the samples constituting a set of test samples (*see* **4.4.1** and **4.4.2.2**).

4.6.2 Tests for the determination of other characteristics shall be conducted on the composite sample prepared as given under **4.4.1** and **4.4.2.1.**

4.7 Criteria for Conformity

4.7.1 In the case of heaps or wagons, if the individual test samples (*see* **4.4.1**) satisfy the specification requirements for available lime and/or total calcium oxide content, the heap or wagon from which the test sample was made shall be deemed as conforming to the requirement(s) of the characteristic(s).

4.7.2 In the case of sampling from packages, for available lime and/or total calcium oxide content, mean ($\bar{X}$) and range (R) shall be computed. (Range is defined as the difference between the maximum and minimum of test results).

4.7.2.1 If the value of the expression ($\bar{X} - 0.6\,R$) is greater than or equal to the minimum value for the characteristic specified in the individual standard, then the lot shall be considered as conforming to the requirements of the particular characteristic.

4.7.3 As far as the remaining characteristic(s), is (are) concerned, the test result(s) on the composite sample (*see* **4.6.2**) shall meet the corresponding requirement(s) as specified in the individual standard.

4.7.3.1 If the requirements for any of the characteristics are not met, the lot/group of wagons or heaps shall be declared to have not satisfied the requirements of the specification.

5 QUALITY OF REAGENTS

5.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1977) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

6 PREPARED SAMPLE

6.1 Samples collected under **4.4.1** and **4.4.2** shall be individually ground well by a suitable mechanical device or by hand in an agate mortar and placed in an air-tight container for use for the chemical analysis.

7 DETERMINATION OF CARBONATE

7.1 Apparatus

The apparatus shall consist of the following components assembled as given in Fig. 1:

a) Funnel (*A*);
b) Two-way cock (*B*);
c) 50-ml flask (*C*);
d) Levelling bottle (*D*);
e) Condenser tube (*E*);
f) Water jacket *(F);*
g) Compensator tube (*G*);
h) Gas burette (*H*);
j) Manometer (*J*);
k) Sodium hydroxide bubbler (*K*);
m) Valve (*L*); and
n) Three-way cocks (*M, N, P, Q*).

7.2 The compensator tube is used to correct automatically for the changes in barometric pressure and temperature by balancing the pressure in the burette against that in the compensator tube. The manometer is used when balancing the pressure in the burette against that in the compensator tube.

7.3 Procedure

7.3.1 Weigh accurately about 3 to 4 g of finely powdered lime and place the sample in flask *C*. Also add a piece of iron wire about the size of a pin head and a drop of methyl orange indicator. Add water until the flask is about three-quarters full.

7.3.2 Open cock *M* for a moment so as to connect the compensator tube *G* to the atmosphere and turn it half anti-clockwise so as to connect the compensator tube with the manometer *J*. Open cocks *P* and *Q* to the atmosphere and level the mercury columns. Turn cocks *P* and *Q* anti-clockwise so as to connect them with the capillary tube leading to

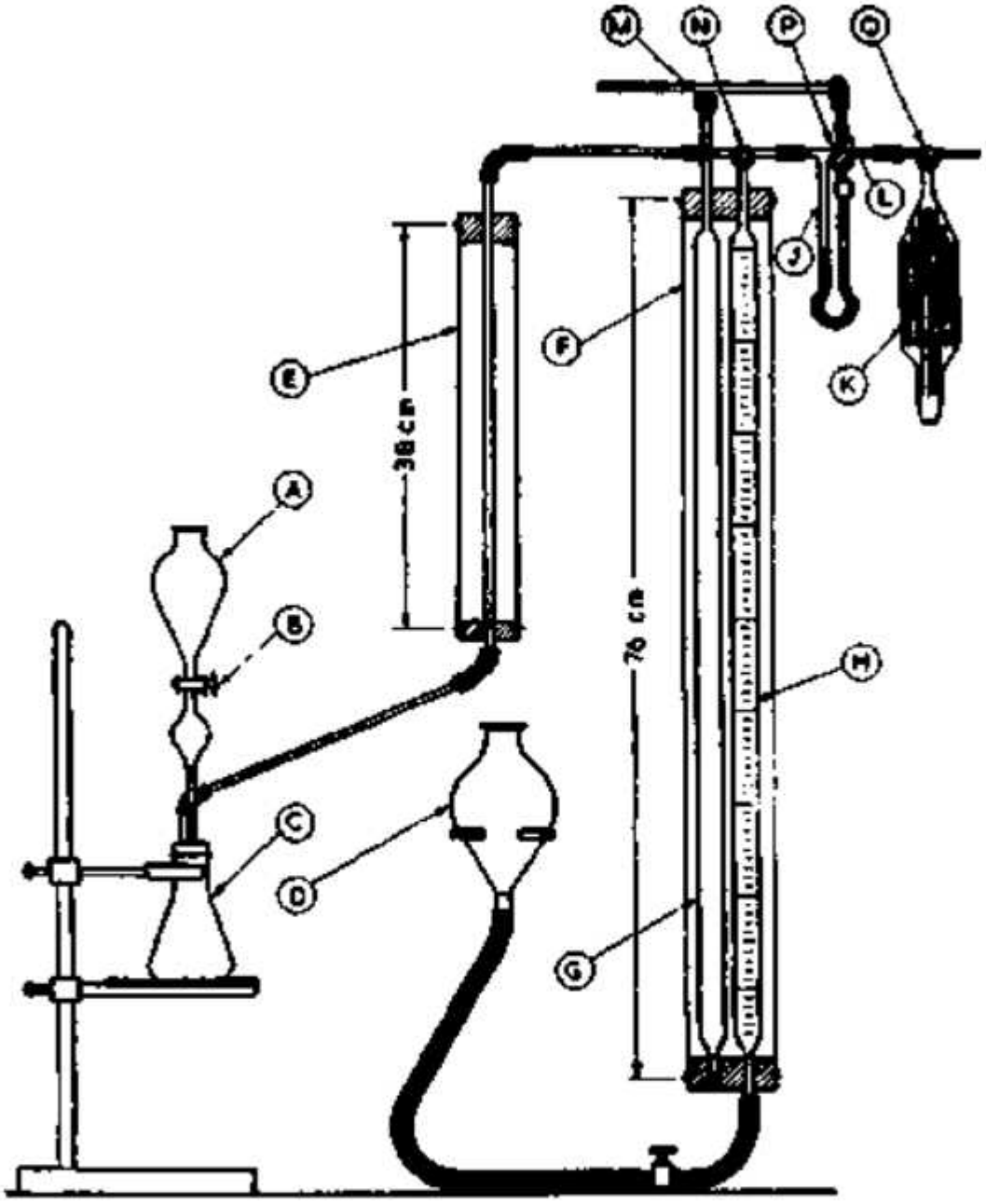

FIG. 1 ASSEMBLY OF APPARATUS FOR DETERMINATION OF CARBONATE

the flask *C*. Fill the burette *H* completely with acidulated water by connecting the cock *N* with the capillary tube leading to the flask and raising *D*. Connect *C* and close cock *B*. Pour excess of concentrated hydrochloric acid into *A*.

7.3.3 Now open cock *B* fully and lower *D* so as to let the acid drop slowly into C until very slightly in excess. Close *B* and fill *D* nearly full with water and lower *D* to the full extent. Heat the contents of *C* to boiling and continue boiling very gently for at least 3 minutes. Remove the burner and open *B* immediately until the water from *C* fills the capillary up to *N* by lowering *D* if necessary. Close *B* and connect *N* with manometer and adjust the mercury columns and read burette *H*.

7.3.4 Next turn cock *P* clockwise so as to connect with sodium hydroxide bubbler *K* and raise and lower *D* for three to four times repeatedly until all carbon dioxide is absorbed. Close *Q* when sodium hydroxide solution reaches the original mark. Connect *P* with *H* to adjust mercury levels and take the burette reading in the same manner as before. Note the temperature of water in the water jacket *F* and the atmospheric pressure.

7.4 Determination of Factor(S)

The factor may be determined theoretically, but more conveniently by a series of actual tests on a sample of known carbon dioxide content. It is recommended that the analytical reagent sodium carbonate (*see* IS 296 : 1986) be used.

7.4.1 *Procedure*

Weigh 2.00 g of the sodium carbonate, dissolve it in 25 ml of carbon dioxide free water, make up to 100 ml in a volumetric flask. Use 10 ml aliquot portions of this, measured by means of a standard pipette, for determining the amount of carbon dioxide it contains by the method given in **7.3**. Calculate the factor as follows:

$$S = \frac{\text{Percentage purity of sodium carbonate} \times M}{\text{ml of carbon dioxide} \times 240.86}$$

where

M = mass of sodium carbonate present in the aliquot of sample taken for Factor Test.

7.5 Calculation

7.5.1 For large number of determinations to be carried out over a short period, calculate as follows:

a) Carbon dioxide, percent by mass $= \frac{100\ VS}{M}$

where

V = observed volume in ml of carbon dioxide;

S = factor as determined in **7.4**; and

M = mass in g of the material taken for the test (*see* **7.3.1**).

b) Calcium carbonate, percent by mass = percentage of carbon dioxide × 2.274 3

7.5.2 For small number of determinations to be carried out over a long period, the separate calculation of factor (*S*) may not be necessary and for such determinations, calculate the percentage of carbon dioxide as follows:

Carbon dioxide, percent by mass $= \frac{0.000\ 705\ 8\ VP}{(273 + t) \times M}$

where

V = observed volume, in ml, of carbon dioxide;

P = atmospheric pressure in mm;

t = temperature, in degree Centrigrade, of water in the jacket *F;* and

M = mass, in g, of the material taken for the test (*see* **7.3.1**).

8 DETERMINATION OF VOLATILE MATTER AT 105°C, AND LOSS ON IGNITION

8.1 Procedure for Determination of Volatile Matter at 105°C

Take one gram (*see* Note) of the powdered lime, accurately weighed, in a platinum crucible. Place it in an oven maintained at 105 ± 2°C until, on cooling and weighing, constant mass is obtained. Preserve the dried material for test under **8.2.**

NOTE — In the case of low magnesium limes, starting with one gram of the material may not give an accurately weighable amount of magnesium pyrophosphate (*see* **13.1.2**). In such a case proportionately larger mass of the material may be taken.

8.2 Procedure for Determination of Loss on Ignition

Heat the crucible containing the dried material (*see* **8.1**), first gently and then ignite in a muffle furnace at 1 000°C. Cool the crucible in a desiccator and weigh to the constant mass. Preserve the ignited material for subsequent determinations.

8.3 Calculation

a) Volatile matter at 105°C, percent by mass $= \frac{100\ (M_1 - M_2)}{M_1}$

where

M_1 = mass, in g, of the material taken for the test (*see* **8.1**); and

M_2 = mass, in g, of the material after drying at 105°C (*see* **8.1**).

b) Loss on ignition, percent by mass = $\frac{100\,(M_1 - M_3)}{M_1}$

where

M_1 = mass, in g, of the material taken for the test (*see* **8.1**); and

M_3 = mass, in g, of the material after ignition (*see* **8.2**).

9 DETERMINATION OF AVAILABLE LIME (as CaO)

9.0 Two methods have been prescribed for the determination of available lime. For routine analysis, the iodine method prescribed in **9.1** may be used but in case of dispute, the sugar method prescribed in **9.2** shall be used.

9.1 Iodine Method

9.1.1 *Reagents*

9.1.1.1 *Standard sodium thiosulphate solution,* 0.1 N, recently standardized.

9.1.1.2 *Standard iodine solution*

0.1 N, recently standardized against standard sodium thiosulphate solution.

9.1.1.3 *Starch indicator solution*

Triturate 5 g of starch and 0.01 g of mercuric iodide with 30 ml of cold water and slowly pour it, with stirring, into 1 litre of boiling water. Boil for three minutes. Allow the solution to cool and decant off the supernatant clear liquid.

9.1.2 *Procedure*

Take 1 g of the material, accurately weighed, in a glass-stoppered conical flask and add about 30 ml of boiling water. Shake for 5 to 10 minutes. Cool the solution and add an amount of iodine solution sufficient to provide an excess of about 5 ml and stir occasionally until the lime has gone in solution. Any insoluble silica present is easily distinguished from the milky-appearing lime. When the solution of lime is complete (about 10 minutes are required), dilute the solution in a volumetric flask to 500 ml. Pipette out 25 ml of the solution and titrate the excess iodine with standard sodium thiosulphate solution, adding starch indicator towards the end of the titration. Carry out a blank determination using the same amount of iodine solution as with the sample.

9.1.3 *Calculation*

Available lime (as CaO), percent by mass = $\frac{56.08\,(V_1 - V_2)\,N}{M}$

where

V_1 = volume, in ml, of standard sodium thiosulphate solution used in the blank;

V_2 = volume, in ml, of standard sodium thiosulphate solution used with the material;

N = normality of standard sodium thiosulphate solution; and

M = mass, in g, of the material taken for the test.

9.2 Sugar Method

9.2.1 *Reagents*

9.2.1.1 *Rectified spirit, see* IS 323 : 1959.

9.2.1.2 *Standard hydrochloric acid,* 0.1 N.

9.2.1.3 *Standard sodium hydroxide solution,* 0.1 N.

9.2.1.4 *Sugar solution,* approximately 15 percent, in carbon dioxide free water.

9.2.1.5 *Phenolphthalein indicator solution*

Dissolve 0.5 g of phenolphthalein in 100 ml of rectified spirit.

9.2.2 *Procedure*

Weigh accurately about 1 g of the finely powdered material into a 500-ml volumetric flask and wet it with the minimum amount of rectified spirit. Shake with about 400 ml of sugar solution for three hours and dilute to the mark. Filter through a coarse dry filter paper (Whatman No. 40 or its equivalent) into a dry flask. Discard the first 25 ml of the filtrate. Pipette out 50 ml from the filtrate into another flask, add 50 ml of standard hydrochloric acid and titrate with standard sodium hydroxide solution using phenolphthalein as indicator.

9.2.2.1 Run a blank using 50 ml of sugar solution and 50 ml of standard hydrochloric acid.

9.2.3 *Calculation*

Available lime (as CaO), percent by mass = $\frac{28.04\,(A - B)\,N}{M}$

where

A = volume, in ml, of standard sodium hydroxide solution used in blank;

B = volume, in ml, of standard sodium hydroxide solution used with the material;

N = normality of standard sodium hydroxide solution; and

M = mass, in g, of the material taken for the test.

10 DETERMINATION OF ACID INSOLUBLE MATTER

10.1 Reagent

10.1.1 *Concentrated Hydrochloric Acid* (*see* IS 265 : 1987).

10.2 Procedure

Transfer the ignited lime, preserved under **8.2** to a beaker, add 50 ml of water and mix well, add 10 to 15 ml of concentrated hydrochloric acid, and boil for 15 minutes. Filter, wash with hot water, ignite and weigh to constant mass.

NOTE — The result thus obtained contains most of the silica and any acid insoluble silicates. It is the figure usually taken in routine work. If an actual determination of the silica is desired evaporate the solution to dryness in a suitable dish, as for as possible on the steam bath, then cover and transfer to a hot high plate or oven and heat for one hour, at 200°C in case of a high-calcium lime, or at 120°C for high magnesium lime. Cool, moisten thoroughly with concentrated hydrochloric acid, let stand for few minutes, add an equal volume of water, cover the dish and heat on the steam bath for 10 to 15 minutes. Filter out the silica, wash well with dilute hydrochloric acid and then twice with hot water. Evaporate the filtrate to dryness, take up with hydrochloric acid as before and filter through a small paper. Transfer both filters to a weighed platinum crucible, smoke off the paper without flaming and ignite the silica over a blast to constant mass.

10.2.1 Preserve the filtrate for test prescribed under **11.**

10.3 Calculation

Express the mass of silica, obtained as described in **10.2** or in the Note under it, as percentage of the mass of the material taken for test in **8.1.**

11 DETERMINATION OF IRON OXIDE AND ALUMINA

11.1 Reagents

11.1.1 *Concentrated Hydrochloric Acid (see* IS 265 : 1987).

11.1.2 *Ammonium Hydroxide,* approximately 4N.

11.1.3 *Standard Potassium Permanganate Solution,* 0.1 N.

11.2 Procedure

To the filtrate, preserved under **10.2.1,** add concentrated hydrochloric acid, if necessary, in sufficient amount to make the total amount equivalent to 10 to 15 ml of concentrated hydrochloric acid. Add few drops of nitric acid and boil. Dilute to 200-250 ml. Add slight excess of ammonium hydroxide and keep just below the boiling point until the odour of ammonia is barely perceptible. Filter off the iron and aluminium hydroxide while hot, collecting the filterate in a 250-ml volumetric flask. Wash with hot water. Ignite the residue in a platinum crucible, blast, cool in a desiccator and weigh as aluminium oxide and ferric oxide. Make up the filtrate to 250 ml and preserve the filtrate for test under **12.**

11.2.1 *Iron Oxide*

Fuse the combined ferric and aluminium oxides in a platinum crucible with 3 to 4 g of potassium bisulphate at a low temperature. Dissolve in water, add sufficient sulphuric acid to make a 5 percent solution and pass through the Jones reductor. Cool and titrate with standard potassium permanganate solution.

11.2.1.1 *Calculation*

$$\text{Iron oxide (as } Fe_2O_3\text{), percent by mass} = \frac{7.984\ V\ N}{M}$$

where

V = volume, in ml, of standard potassium permanganate solution used;

N = normality of standard potassium permanganate solution; and

M = mass, in g, of the material taken for the test (*see* **8.1**).

11.2.2 *Aluminium Oxide*

Subtract the ferric oxide value from the combined oxides value (*see* **11.2**).

12 DETERMINATION OF TOTAL CALCIUM OXIDE

12.0 General

Two methods have been prescribed, namely, Method A and Method B. Method A is the classical method and Method B is the complexometric method. In case of dispute, Method A shall be followed.

12.1 Method A

12.1.1 *Reagents*

a) *Ammonium oxalate solution,* saturated.

b) *Dilute ammonium hydroxide solution,* approximately 5 N and 1 N.

c) *Dilute hydrochloric acid,* approximately 5 N.

d) *Dilute sulphuric acid,* approximately 5 N.

e) *Dilute potassium permanganate solution,* approximately 0.01 N.

f) *Standard potassium permanganate solution,* 0.1 N.

g) *Diammonium hydrogen phosphate solution,* approximately 25 percent (*m/v*).

h) *Ammonium hydroxide,* relative density 0.90.

12.1.2 *Procedure*

Pipette out 50 ml of the filtrate preserved under **11.2** in a beaker and dilute to 100 ml. Heat to boiling and add slowly about 35 ml of boiling ammonium oxalate solution. Continue boiling for 2 or 3 minutes and allow the precipitated calcium oxalate to settle for half an hour. Filter the precipitated calcium oxalate through filter paper. Wash thoroughly with small portions of dilute ammonium hydroxide (1 N) and then with hot water until the washings do not decolourize a hot dilute potassium permanganate solution in the presence of dilute sulphuric acid. Preserve the filtrate and washings for test in **13.1**. Puncture the filter paper and transfer the precipitate to the beaker already used for precipitation with a fine jet of hot water. Add about 50 ml of dilute sulphuric acid, heat to 60°C and titrate with standard potassium permanganate solution with constant stirring. Towards the end of the titration, introduce the filter paper which was used for filtration into the titration vessel and carry out the titration till the end point is reached.

Carry out a blank with all reagents following the procedure as outlined for this method.

12.1.2.1 In the case of magnesium limes, decant through a filter paper, redissolve the calcium oxalate in the beaker and in the filter paper with dilute hydrochloric acid and wash the filter paper four times with hot water and finally with dilute ammonium hydroxide (5 N) in a slight excess and proceed as in **12.1.2**. For the second filtration, use the same filter paper as was used before.

12.1.2.2 If it is desired to complete the analysis in as short a time as possible, a portion of 50 ml of the filtrate from the ferric and aluminium oxide determination should be precipitated in the usual way with excess of ammonium oxalate. Boil for about 5 minutes and let the calcium oxalate settle clear. Decant through a qualitative filter paper and cool the filtrate (with ice water if possible). Add diammonium hydrogen phosphate solution in large excess and 5 to 10 ml of ammonium hydroxide solution. Stir rapidly with rubber 'policeman'. From the amount of precipitate thus formed one can judge whether the lime contains sufficient magnesium oxide to require a double precipitation or not. For accurate work, if there is more than a slight amount of magnesium oxide, a double precipitation should be carried out, using a fresh 50 ml aliquot.

12.1.3 *Calculation*

$$\text{Total calcium oxide (as CaO), percent by mass} = \frac{14.02\,(V_1 - V_2)\,N}{M}$$

where

V_1 = volume, in ml, of standard potassium permanganate solution used for the test;

V_2 = volume, in ml, of standard potassium permanganate solution used for the blank;

N = normality of standard potassium permanganate solution; and

M = mass, in g, of the material taken for the test (see **8.1**).

12.2 Method B

12.2.1 *Reagent*

a) *Concentrated hydrochloric acid (see* IS 265 : 1987).

b) *Standard disodium ethylene diamine tetra-acetate dihydrate (EDTA) solution*

Weigh 7.44 g of disodium ethylene diamine tetra-acetate dihydrate and dissolve in water. Make up the volume to two litres. Standardize the solution against standard calcium chloride solution according to the procedure given in **12.2.2.**

c) *Standard calcium chloride solution*

Dry about 2 g of calcium carbonate at 120° C to constant mass. Dissolve 1.00 g of the dried calcium carbonate in minimum quantity of concentrated hydrochloric acid and then make up the volume to one litre. One millilitre of this solution is equivalent to 0.000 56 g of calcium oxide.

d) *Eriochrome black 'T' indicator (for calcium and magnesium)*

Dissolve 0.1 g of the dye in 20 ml of methanol. This solution shall be prepared freshly every week.

e) *Murexide indicator (for calcium alone)*

Grind 0.2 g of murexide with 10 g of sodium chloride until the mixture is homogeneous.

or

Calcein indicator (for calcium alone)

Mix 0.01 g of calcein thoroughly with one gram of potassium chloride.

f) *Buffer solution*

Dissolve 67.5 g of ammonium chloride in a mixture of 520 ml of ammonium hydroxide (relative density 0.90) and 250 ml of water. Dissolve a mixture of 0.931 g of disodium ethylene diamine tetra-acetate dihydrate and 0.616 g of magnesium sulphate heptahydrate ($MgSO_4$. $7H_2O$) in 50 ml of water, combine both the solutions and make up to one litre. Five millilitres of buffer solution added to 50 ml of water should not consume more than one drop of EDTA solution to change to distinct blue with eriochrome black 'T' indicator.

g) *Standard sodium hydroxide solution,* 5 N.

12.2.2 *Standardization of Disodium Ethylene Diamine Tetra-acetate Dihydrate (EDTA) Solution*

Standardize as given below:

a) *With murexide or calcein indicator*

Pipette out 50 ml of standard calcium chloride solution (*see* Note 1) in a 250-ml conical flask and add to it one millilitre of standard sodium hydroxide solution to adjust the *p*H of the solution to about 12. Add 0.1 to 0.2 g of murexide indicator (*see* Note 2) and titrate the solution against standard EDTA solution until the colour changes from pink to purple.

b) *With eriochrome black 'T' indicator*

Pipette out 50 ml of standard calcium chloride solution (*see* Note 1) in a 250-ml conical flask and add 5 ml of buffer solution to adjust the *p*H of the solution to approximately 10. Add 5 drops of eriochrome black 'T' indicator and titrate against standard EDTA solution until the wine red colour changes to pure blue. (The volume of the solution to be titrated against standard EDTA solution should be approximately 50 ml and the strength of the standard EDTA solution should be adjusted in such a way that almost 50 ml of it is required for the titration.)

NOTES

1 The aliquot for titration shall be neutral so that by adding standard sodium hydroxide solution or buffer solution the desired *p*H could be adjusted in the solution.

2 If calcein indicator is to be used in place of murexide indicator, add 0.07 g of calcein. The colour change in this case will be from greenish yellow fluorescence to brown.

12.2.3 *Prepared Sample Solution*

Dissolve about 2.5 g of the material, accurately weighed, in a limited quantity of concentrated hydrochloric acid (*see* IS 265 : 1987) and make up the volume to 1 000 ml. Reserve the *prepared sample solution* for the determination of magnesium oxide in **13.2.**

12.2.4 *Procedure*

Pipette out 10 ml of the *prepared sample solution* (*see* **12.2.3**) in a 250-ml conical flask and dilute with 40 ml of water. Add 1.5 ml of standard sodium hydroxide solution followed by a small quantity (0.1 to 0.2 g) of murexide indicator (or 0.07 g of calcein indicator). Titrate against standard EDTA solution till the colour changes from wine red to purple. (In the case of calcein indicator the colour change will be from greenish yellow fluorescence to brown.) Note the burette reading.

12.2.5 *Calculation*

$$\text{Total calcium oxide (as CaO), percent by mass} = \frac{10\,000\,V_1\,A_1}{M}$$

where

V_1 = volume, in ml, of standard EDTA solution used;

A_1 = mass, in g, of calcium oxide equivalent to one millilitre of standard EDTA solution as obtained in **12.2.2** (a); and

M = mass, in g, of the material taken as in **12.2.3.**

13 DETERMINATION OF MAGNESIUM OXIDE (as MgO)

13.0 General

Two methods have been prescribed, namely Method A and Method B. Method A is the classical method and Method B is the complexometric method. In case of dispute, Method A shall be followed.

13.1 Method A

13.1.1 *Reagents*

a) *Dilute hydrochloric acid,* approximately 4 N.

b) *Dilute ammonium hydroxide,* approximately 1:2.

c) *Diammonium hydrogen phosphate solution, see* **12.1.1** (g).

d) *Rectified spirit, see* 323 : 1969.

13.1.2 *Procedure*

Acidify the filtrate set apart under **12.1.2** (or the combined filtrate, in case of a magnesium lime) with dilute hydrochloric acid and evaporate until the salts begin to crystallize. Dilute until

the salts are again in solution. Add a volume of dilute ammonium hydroxide equal to a third of the volume of the solution. Chill the solution and add slowly with constant stirring 10 ml of diammonium hydrogen phosphate solution. Let stand until precipitation is complete. Four hours are usually sufficient, but if possible, it is better to let the solution stand overnight. If the analysis is urgent, stir for half an hour and the precipitation will be complete. Filter through a weighed Gooch crucible (previously weighed) and wash with a mixture of one part dilute ammonium hydroxide, one part rectified spirit and three parts water. Dry at 105°C in an air-oven, char the filter paper by gently heating over a low flame and then slowly increase the flame and finish by heating at 900 to 950°C till the precipitate is almost white, indicating that the salt has been converted to magnesium pyrophosphate. Cool in a desiccator and weigh.

13.1.3 *Calculation*

$$\text{Magnesium oxide (as MgO), percent by mass} = \frac{181.2\,m}{M}$$

where

m = mass, in g, of magnesium pyrophosphate; and

M = mass, in g, of the material taken for the test in **8.1.**

13.2 Method B

13.2.1 *Reagents*

a) *Buffer solution, see* **12.2.1** (f),

b) *Eriochrome black 'T' indicator, see* **12.2.1**(d), and

c) *Standard disodium ethylene diamine tetra-acetate dihydrate (EDTA) solution — see* **12.2.1** (b).

13.2.2 *Procedure*

Pipette out 10 ml of the *prepared sample solution* (*see* **12.2.3**) in a 250-ml conical flask and dilute with 35 ml water. Add 5 ml of buffer solution and 5 drops of eriochrome black 'T' indicator. Titrate against standard EDTA solution till the wine red colour of the solution changes to blue.

13.2.3 *Calculation*

$$\text{Magnesium oxide (as MgO), percent by mass} = \frac{7\,190\,(V_2 A_2 - V_1 A_1)}{M}$$

where

V_2 = volume, in ml, of standard EDTA solution used in **13.2.2;**

A_2 = mass, in g, of calcium oxide equivalent to one millilitre of standard EDTA solution as obtained in **12.2.2** (b);

V_1 = volume, in ml, of standard EDTA solution used in **12.2.4;**

A_1 = mass, in g, of calcium oxide equivalent to one milliiitre of standard EDTA solution as obtained in **12.2.2** (a); and

M = mass, in g, of the material in **12.2.3.**

14 DETERMINATION OF SULPHATES

14.1 Reagents

14.1.1 *Dilute Hydrocloric Acid,* approximately 1 : 2 by volume.

14.1.2 *Barium Chloride Solution,* approximately 10 percent (*m/v*).

14.2 Procedure

Weigh accurately about 2 g of the material and transfer it to a small beaker. Stir with 10 ml of cold water until all lumps are broken up and the lighter particles are in suspension. Add 15 ml of dilute hydrochloric acid and heat until reaction ceases. Filter and wash thoroughly with hot water. Dilute the filtrate to 250 ml, heat to boiling and add 10 ml of boiling barium chloride solution drop by drop with constant stirring. Boil for half an hour and allow to stand for four hours. Filter through a tared Gooch or tared sintered glass crucible (G. No. 4). Wash the precipitate thoroughly with hot water till free from chlorides and dry to constant mass at 105 to 110°C.

14.3 Calculation

$$\text{Sulphates (as } So_4\text{), percent by mass} = \frac{41.15\,A}{M}$$

where

A = mass, in g, of the barium sulphate precipitate; and

M = mass, in g, of the material taken for the test.

15 DETERMINATION OF DEAD BURNT LIME

15.1 Procedure

Determine the carbonates, available lime, total calcium oxide and sulphates as prescribed in **7, 9, 12** and **14** respectively.

15.2 Calculation of Result

15.2.1 Convert the percentage of carbonates present to its equivalent percentage of CaO by multiplying with the factor 0.560 3.

15.2.2 Convert the percentage of sulphates present to its equivalent percentage of CaO by multiplying with the factor 0.583 7.

15.2.3 To obtain dead burnt lime (as CaO) subtract the sum total of available lime (as CaO) and the values obtained in **15.2.1** and **15.2.2** from the percentage of total calcium oxide (as CaO) obtained in **12.1.3** or **12.2.5.**

16 DETERMINATION OF FINENESS

16.1 Procedure

Place 50 g of the sample on the appropriate sieve and wash by means of a stream of water from faucet until the water coming through the sieve is clear. Dry the residue to constant mass in an atmosphere free from carbon dioxide in a drying oven at 100 to 120°C. Calculate the percentage of sample passing through the sieve from the difference obtained by subtracting the mass of the residue from the original mass of the material.

17 DETERMINATION OF FREE CALCIUM OXIDE (as CaO)

17.0 Principle

Free calcium oxide is determined by subtracting calcium oxide present as calcium hydroxide from the total available calcium oxide.

17.1 Apparatus

17.1.1 *Platinum Crucible*

17.1.2 *Muffle Furnace*

17.2 Procedure

Weigh about 1 g of the sample in a platinum crucible. Place the crucible in a muffle furnace. Ignite the sample in the muffle furnace at 1 000°C for one hour. Cool the crucible in a desiccator and weigh. Repeat the process of heating, cooling, and weighing till constant mass is obtained.

17.3 Calculation

Water of hydration,
percent by mass = $L - (A + B)$

Calcium oxide present as calcium hydroxide (C), percent by mass, = $[L - (A + B)] \frac{56}{18}$

Free Calcium oxide (as CaO), percent by mass = $D-C$

where

A = carbon dioxide (percent by mass) equivalent of calcium carbonate percent in the sample (*see* **7.5.1** or **7.5.2**);

B = moisture content (percent by mass) of the sample (*see* **18.2**);

C = calcium oxide (percent by mass) present as calcium hydroxide;

D = Available calcium oxide in the sample (see **9.1.3** or **9.2.3**); and

L = Loss on ignition [*see* **8.3** (c)].

18 DETERMINATION OF MOISTURE

18.1 Procedure

Weigh about 5 g of the material in a weighing dish with lid (squat form) and place it in a vacuum desiccator with the lid open. Alongwith the sample, place another dish containing about 10 g of phosphorus pentoxide as a dehydrating agent. Connect the vacuum pump and evacuate to a pressure of 760 mm of Hg. Then disconnect the vacuum pump and allow the sample to remain in the desiccator for four hours. Then release the vacuum, take out the dish and weigh accurately.

18.2 Calculation

Moisture, percent by mass = $\frac{M_1 - M_2}{M_1} \times 100$

where

M_1 = mass, in g, of the material before drying; and

M_2 = mass, in g, of the material after drying.

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This Indian Standard has been developed from Doc : No. CHD 02 (9238)

Amendments Issued Since Publication

Amend No.	Date of Issue
Amd. No. 1	January 1998

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