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APPENDIX-A

CHEMICAL ANALYSIS OF BUILDING LIMES

A-1 INTRODUCTION

Lime has been used in India as a material of construction from very ancient days. The manner in which lime structures of about 2000 years old have withstood the ravages of time bear irrefutable evidence to the durability of lime mortars. The vastness of this country and its varied geological character has made available several types of lime stones suitable for burning to obtain building limes. Due to the variability of lime stone from place to place the resulting lime may be expected to vary in quality. While it is not possible to make strict comparison of characteristics of many types of limes obtained, an attempt has been made here to compare the chemical properties of limes from three different sources which are used in the experiments. The method of manufacturing building limes and the manner in which they are used in construction work differ from one part of the country to another. Besides, defects caused by adulteration with undesirable admixtures, differences in the method of burning, slaking, storing and using will appreciably affect the quality of lime. Hence suitable standards are necessary
to check the quality of lime so that it can be confidently used in the building construction. At a time when necessity has been felt to make full use of our construction material resources, the formulation of a specification for building limes and its adoption are expected to bring about a more judicious use of limes for construction purposes.

A.2 Chemical Requirements

Chemical requirements of building limes are given in Table A-1. (IS: 712, 1973).

A.3 Notation

The following notation is used throughout the chemical analysis.

<table>
<thead>
<tr>
<th>S.No.</th>
<th>Source of lime and type</th>
<th>Notation</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Cuddapah-unsalted</td>
<td>C-A</td>
</tr>
<tr>
<td>2.</td>
<td>Cuddapah - slaked</td>
<td>C-B</td>
</tr>
<tr>
<td>3.</td>
<td>Tirupati - unsalted</td>
<td>T-A</td>
</tr>
<tr>
<td>4.</td>
<td>Tirupati - slaked</td>
<td>T-B</td>
</tr>
<tr>
<td>5.</td>
<td>Pakala - unsalted</td>
<td>P-A</td>
</tr>
<tr>
<td>6.</td>
<td>Pakala - slaked</td>
<td>P-B</td>
</tr>
</tbody>
</table>
### TABLE A1 CHEMICAL REQUIREMENTS

*Class 4.1*

<table>
<thead>
<tr>
<th>Sr. No</th>
<th>Type of Test</th>
<th>Requirements for Building Limes</th>
<th>Methods of Tests (Ref to IS:6932, 1973)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Class A Hydrated</td>
<td>Class B</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Quick</td>
<td>Hydrated</td>
</tr>
<tr>
<td>(1)</td>
<td>(2)</td>
<td>(3)</td>
<td>(4)</td>
</tr>
<tr>
<td>i)</td>
<td>Calcium and magnesium oxides, percent, Min</td>
<td>60</td>
<td>70</td>
</tr>
<tr>
<td>ii)</td>
<td>Magnesium oxide, Max percent</td>
<td>5</td>
<td>5</td>
</tr>
<tr>
<td>iii)</td>
<td>Silica, alumina and ferric oxide, percent, Min</td>
<td>25</td>
<td>15</td>
</tr>
<tr>
<td>iv)</td>
<td>Unhydrated oxides, percent, Max</td>
<td></td>
<td></td>
</tr>
<tr>
<td>v)</td>
<td>Insoluble residue in hydrochloric acid less than silica, percent, Max</td>
<td>2</td>
<td>5</td>
</tr>
<tr>
<td>vi)</td>
<td>Insoluble matter in sodium carbonate solution, percent, Max</td>
<td>5</td>
<td>5</td>
</tr>
<tr>
<td>vii)</td>
<td>Loss on ignition, percent, Max</td>
<td></td>
<td></td>
</tr>
<tr>
<td>viii)</td>
<td>Carbon dioxide, percent, Max</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Notes:**
- Percentages shall be expressed on ignited basis in respect of items (i) to (vi).
- The sign (—) means no requirements for the test.

**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.
- Part II Determination of carbon dioxide content.
- Part V Determination of unhydrated oxide of quicklime.

**The value is equal to**

\[
\frac{2.8P + 1.1Q + 0.7R}{1.0X + 1.4Y}
\]

**where**
- \(P\) = silica \((SiO_2)\) content, percent by weight;
- \(Q\) = aluminium oxide \((Al_2O_3)\), percent by weight;
- \(R\) = ferric oxide \((Fe_2O_3)\), percent by weight;
- \(S\) = calcium oxide \((CaO)\), percent by weight; and
- \(Y\) = magnesium oxide \((MgO)\), percent by weight.
A-4 EXPERIMENTATION

Lime has been obtained from three different sources (Art 2.2), sieved and prepared as explained earlier (Art 3.4). Experiments have been conducted for the determination of the following.

a) Insoluble residue  b) Loss on ignition  c) Insoluble matter  d) Silicon dioxide  e) Ferric and aluminium dioxide  f) Calcium Oxide and  g) Magnesium oxide.

A-5 DETERMINATION OF INSOLUBLE RESIDUE INCLUDING SILICON DIOXIDE IN HYDROCHLORIC ACID

A-5-1 REAGENTS: Dilute Hydrochloric acid is prepared by diluting Hydrochloric acid of Sp.Gr. 1.16 with an equal volume of distilled water (1:1) Sodium carbonate solution is prepared by dissolving 5 gm. of sodium carbonate in 100 ml of water.

A-5-2 PROCEDURE

In beaker of 250 ml capacity 1.0 gm of lime powder from one of the sources is weighed, to which 10 ml of distilled water is added and the contents are made into a slurry. Then 20 to 25 ml of dilute hydrochloric acid is added and the contents are digested for half an hour with occasional gentle agitation. The sides of the
beaker are rinsed with hot water thus diluting the solution to about 100 ml. The contents are redigested at a temperature just below the boiling point for about 15 minutes. The contents of the beaker are filtered through a Whatman filter paper. The residue and the filter paper are washed twice with dilute hydrochloric acid and then with hot water till they are free from chlorides.

The filter paper along with the residue are transferred to a porcelain dish and 30 to 40 ml of sodium carbonate solution is added to it. The dish is covered and its contents are digested for about half an hour at a temperature just below the boiling point. Hot water in small quantities are added during digestion to make up the loss of volume caused due to evaporation. The contents of the dish are filtered through a filter paper, the residue in the dish is washed a few times with hot water and filtered again. The residue with the filter paper is transferred into the dish, covered with sodium carbonate solution, and digested again for about 10 minutes. Then the contents are filtered through a filter paper, washed twice with hot sodium carbonate solution and then with hot water, till free from alkalies. Again the filter paper with the residue is washed twice with dilute hydrochloric acid and then with hot water.
till free from chlorides.

Finally the filter paper with the residue is placed in a crucible, dried, ignited and the contents are weighed.

A-5-3 PRESENTATION OF RESULTS

The insoluble residue in 5 per cent sodium carbonate solution is expressed as a percentage on the ignited mass. Table A-1 gives the results of insoluble residue test, conducted as above.

Table A-1 Results of insoluble residue test

<table>
<thead>
<tr>
<th>Source of lime</th>
<th>C-A</th>
<th>C-B</th>
<th>T-A</th>
<th>T-B</th>
<th>P-A</th>
<th>P-B</th>
</tr>
</thead>
<tbody>
<tr>
<td>Percentage</td>
<td>5.998</td>
<td>5.908</td>
<td>4.408</td>
<td>5.602</td>
<td>4.528</td>
<td>4.686</td>
</tr>
</tbody>
</table>

A-6 DETERMINATION OF LOSS ON IGNITION

A-6-1 PROCEDURE

One gram of lime powder from one of the sources is weighed and placed in a weighed silica crucible and covered with a lid. Then it is placed in a Muffle furnace at a temperature of about 600°C to constant mass. The crucible is cooled to room temperature and weighed with the contents, the difference between the original mass and the final mass of the sample represents the loss on ignition. This is expressed as a percentage of the mass
of the sample taken in the beginning.

A-6-2 PRESENTATION OF RESULTS

Table A-2 gives the results of loss on ignition.

Table A-2 Results of loss on ignition

<table>
<thead>
<tr>
<th>Source of Lime</th>
<th>C-A</th>
<th>C-B</th>
<th>T-A</th>
<th>T-B</th>
<th>P-A</th>
<th>P-B</th>
</tr>
</thead>
<tbody>
<tr>
<td>Percentage</td>
<td>1.30</td>
<td>1.08</td>
<td>1.32</td>
<td>1.17</td>
<td>2.57</td>
<td>1.79</td>
</tr>
</tbody>
</table>

A-7 DETERMINATION OF FERRIC AND ALUMINIUM OXIDES

A-7-1 REAGENTS

Dilute Hydrochloric acid - 1:99 (v/v); 1:1 and 1:3 (v/v) Methyl Red Indicator - 0.1 per cent (m/v). It is prepared by dissolving 0.1 gm of the sodium salt of the methyl red in 100 ml of water.

Ammonium chloride solution: 2 per cent (m/v).

A-7-2 PROCEDURE

The ignited sample from Art A-6 is transferred into an evaporating dish and made into a slurry with distilled water. Then 10 ml of concentrated hydrochloric acid is added to the slurry and the contents are digested by heating and agitating simultaneously until the contents are dissolved. Then the solution is evaporated to dryness
on a water-bath. When the sample is nearly dry it is placed in an oven for one hour, while the temperature of the oven is maintained at 110° to 120°C. Then the sample is taken out, cooled to room temperature and 10 ml of concentrated hydrochloric acid is added to the same. After allowing it to stand for a few minutes, an equal volume of water is added, covered with a dish and heated on the water-bath for 10 minutes. It is filtered, the residue is washed with hot dilute hydrochloric acid and finally twice with hot water. The filtrate is evaporated to dryness and then baked at 110° to 120°C for one hour. It is extracted with hydrochloric acid as before and filtered through a second filter paper. The filtrate is used for conducting further tests.

A-7-3 To the filtrate reserved in A-7-2 a few drops of concentrated nitric acid is added and the solution is boiled until all traces of chlorine are gone. About 10 to 15 ml of concentrated hydrochloric acid is added and the solution is diluted to 200 ml. Then a few drops of methyl red solution is added, the solution is heated to boiling and neutralized with ammonium hydroxide solution until the colour of the liquid changes to a distinct yellow. The solution is then boiled for one to two minutes, allowed to settle and filtered through a filter paper.
The precipitate is washed immediately 2 or 3 times with
hot ammonium chloride solution and dried by suction.
The filtrate is used for conducting further tests.

The precipitate on the filter paper is dissolved
in hot dilute hydrochloric acid (1:3) and the solution
is collected in the beaker in which the precipitation
was made. The filter paper is thoroughly washed with hot
water into the same beaker. The solution is boiled to
expel any traces of chlorine and is treated with ammonium
hydroxide solution for precipitation. Then it is fil-
tered and washed with hot ammonium chloride solution.
This filtrate is combined with the previous filtrate and
used for the determination of calcium oxide.

/.7-4 PRESENTATION OF RESULTS

Table 7-3 gives the results of the tests conducted
for ferric and aluminium oxides.

Table 7-3 Results of tests for Ferric and Aluminium
Oxides.

<table>
<thead>
<tr>
<th>Source of lime</th>
<th>C-A</th>
<th>C-B</th>
<th>T-A</th>
<th>T-B</th>
<th>P-A</th>
<th>P-B</th>
</tr>
</thead>
<tbody>
<tr>
<td>Percentage</td>
<td>8.66</td>
<td>8.55</td>
<td>6.28</td>
<td>6.02</td>
<td>9.164</td>
<td>9.18</td>
</tr>
</tbody>
</table>
Determination of Calcium Oxide Content

A-8-1 REAGENTS

Ammonium oxalate solution: 0.1 per cent (m/v) and saturated. Dilute sulphuric acid: 1:10 (v/v).

Standard potassium permanganate solution: It is prepared by dissolving 0.563 gm of potassium permanganate in 100 ml of distilled water. It is allowed to stand for a week, filtered and standardized against sodium oxalate solution, prepared by dissolving 0.75 gm of sodium oxalate in 250 ml of water.

A-8-2 PROCEDURE

The combined filtrate reserved under Art A-7-3. is used in the determination of Calcium oxide. The filtrate is diluted with distilled water so as to obtain a volume of 200 ml. Out of this 25 ml of this filtrate is used in the analysis. This filtrate is made alkaline with hydroxide solution. This solution is boiled to which 35 ml of boiling saturated solution of ammonium oxalate is added, stirred vigorously and allowed to stand until the precipitate is settled. It is filtered through a filter paper and the precipitate washed 10 times with hot water. Care is taken to see that the total quantity of water used for this wash does not exceed 125 ml. The filter paper along with the precipitate is transferred
to the beaker in which the precipitation was done, the paper being spread out against the upper portion of the beaker. The precipitate on the paper is washed with a jet of hot water, the paper is folded and left adhering to the upper portion of the beaker. To this beaker is added 50 ml of dilute sulphuric acid and the solution is diluted to a volume of 250 ml with hot water. The beaker is heated to 90° to 90°C. It is titrated with the standard potassium permanganate solution until the pink end point is obtained. Then the folded filter paper when dropped into the liquid will discharge the pink colour. The titration is again continued by adding potassium permanganate solution drop by drop until the pink end point is again obtained.

### 7-0-3 PRESENTATION OF RESULTS

One millilitre of standard potassium permanganate solution is approximately equivalent to 0.005 gm of Calcium oxide. Table A-4 gives the results of the tests conducted for Calcium oxide.

<table>
<thead>
<tr>
<th>Source of lime</th>
<th>C-A</th>
<th>C-B</th>
<th>T-A</th>
<th>T-B</th>
<th>P-A</th>
<th>P-B</th>
</tr>
</thead>
<tbody>
<tr>
<td>Percentage</td>
<td>36</td>
<td>56</td>
<td>48</td>
<td>48</td>
<td>44</td>
<td>44</td>
</tr>
</tbody>
</table>
DETERMINATION OF MANGANESE OXIDE

7.3-1 Reagents

Dilute Hydrochloric acid: 1:1 and 1:4 (v/v). Diammonium hydrogen phosphate solution: 25 per cent (m/v). Ammonium Nitrate wash solution: It is prepared by diluting ammonium hydroxide with distilled water until the solution contains 2.5 per cent of ammonia by mass, to this is added 3 or 4 drops of concentrated Nitric acid.

7.3-2 PROCEDURE

The combined filtrate obtained in Art 7.3-2 is acidified with hydrochloric acid. Then 10 ml of diammonium hydrogen phosphate solution is added to it and the solution is cooled to room temperature. To this solution, ammonium hydroxide solution is added drop by drop, the contents stirred constantly until crystalline precipitate of magnesium ammonium orthophosphate begins to form. Excess of ammonium hydroxide solution is added to the extent of 5 to 10 per cent of the volume of the solution. It is stirred continuously for several minutes and the liquid is allowed to stand in a cool atmosphere for 12 to 48 hours and then filtered through a filter paper.

The precipitate is dissolved in hot dilute hydrochloric acid (1:4) and the solution is diluted to about
100 ml. Then one ml of diammonium hydrogen phosphate solution is added and then ammonium hydroxide solution drop by drop with constant stirring until the precipitate again begins to form as described earlier. Ammonium hydroxide is added moderately in excess quantity. It is allowed to stand in a cold atmosphere for 12 to 48 hours, then filtered and washed with ammonium nitrate wash solution. The filter paper is burnt at a low temperature and finally the residue is ignited to constant mass.

I.-9-3 PRESENTATION OF RESULTS

The magnesium oxide content is calculated as follows:

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

$A =$ mass of magnesium pyrophosphate

$B =$ mass of the ignited sample represented by the aliquot taken for the estimation of Calcium oxide under I.-8-2.

I.-9-4 PRESENTATION OF RESULTS

Table I.-5 gives the results of the tests conducted for magnesium oxide.

Table I.-5 Results of tests for Magnesium oxide

<table>
<thead>
<tr>
<th>Source of lime</th>
<th>C-A</th>
<th>C-B</th>
<th>T-A</th>
<th>T-B</th>
<th>P-A</th>
<th>P-B</th>
</tr>
</thead>
<tbody>
<tr>
<td>Percentage</td>
<td>7.22</td>
<td>6.84</td>
<td>4.74</td>
<td>4.90</td>
<td>4.64</td>
<td>435</td>
</tr>
</tbody>
</table>
The cementation value is calculated as follows.

\[
\text{Cementation value} = \frac{2.8 P + 1.1 Q + 0.7 R}{1.0 X + 1.4 T}
\]

where

- \( P \) = Silica (\( \text{SiO}_2 \)) content, per cent by weight,
- \( Q \) = Aluminium oxide (\( \text{Al}_2\text{O}_3 \)), per cent by weight,
- \( R \) = Ferric oxide (\( \text{Fe}_2\text{O}_3 \)), per cent by weight,
- \( S \) = Calcium oxide (\( \text{CaO} \)), per cent by weight,
- \( T \) = Magnesium oxide (\( \text{MgO} \)), per cent by weight.

In Table 8-6 are shown the cementation values for each source of lime.

<table>
<thead>
<tr>
<th>Source of lime</th>
<th>C-A</th>
<th>C-B</th>
<th>T-A</th>
<th>T-B</th>
<th>P-A</th>
<th>P-B</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cementation value</td>
<td>0.372</td>
<td>0.371</td>
<td>0.329</td>
<td>0.385</td>
<td>0.414</td>
<td>0.427</td>
</tr>
</tbody>
</table>

SUMMARY OF THE RESULTS OF CHEMICAL ANALYSIS

Table 8-7 summarises the results of chemical analysis.

Table 8-7 Test results of chemical analysis

<table>
<thead>
<tr>
<th>Source of lime</th>
<th>C-A</th>
<th>C-B</th>
<th>T-A</th>
<th>T-B</th>
<th>P-A</th>
<th>P-B</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Insoluble residue including silicon dioxide in hydrochloric acid; percentage:</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>5.998</td>
<td>5.908</td>
<td>4.408</td>
<td>5.602</td>
<td>4.528</td>
<td>4.686</td>
<td></td>
</tr>
<tr>
<td>2. Percentage of loss on ignition:</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1.300</td>
<td>1.000</td>
<td>1.320</td>
<td>1.170</td>
<td>2.570</td>
<td>1.790</td>
<td></td>
</tr>
</tbody>
</table>

contd.
3. Percentage of Ferric and Aluminium oxide:
4. Percentage of Calcium oxide:
   56.00 56.00 48.00 48.00 44.00 44.00
5. Percentage of Magnesium oxide:
   7.22 5.840 4.74 4.90 4.64 4.35
6. Cementation values:
   0.372 0.371 0.329 0.395 0.414 0.427

1. CONCLUSIONS

1. From the test results in Table A-7 it can be observed that the percentage of chemical compounds determined are comparable to the chemical requirements shown in Art 3-2 as per IS standards.

2. The percentage of Calcium oxide is the highest for Cuddapah lime, (C-A and C-B), some what less for Tirupati lime (T-A and T-B), and least for Pakala lime (P-A and P-B). From the test results of Chapter-II, it can be concluded that as the percentage of Calcium oxide increases the strength of the mortar decreases.

3. From Table A-6 it can be observed that the cementation values of Cuddapah lime are the least, some what higher for Tirupati lime and highest for Pakala lime. Hence, it can be concluded that the strength of lime mortar
increases along with the increase in cementation value.

4. The chemical analysis helps to classify the lime from classes A to E, and suggests the purpose for which it can be utilized as explained in Art 2.1.1.(2).
APPENDIX – B

List of Codes published by ISI on Lime, Lime mortar, Lime concrete, Concrete masonry units and related topics.

<table>
<thead>
<tr>
<th>S.No.</th>
<th>Code Number</th>
<th>Year of publication</th>
<th>Title of the code</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>IS-712</td>
<td>1973</td>
<td>Specifications for Building limes</td>
</tr>
<tr>
<td>3.</td>
<td>IS-1624</td>
<td>1960</td>
<td>Method of field testing of building lime.</td>
</tr>
<tr>
<td>8.</td>
<td>IS-2185</td>
<td>1979</td>
<td>Specifications for concrete masonry units, Hollow and solid concrete blocks (Second revision).</td>
</tr>
<tr>
<td></td>
<td>Part I</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

contd...


15. IS-3495 1976 Methods of tests for burnt clay building bricks, Part I to IV.


17. IS-4139 1967 Sand-lime bricks.


20. IS-6932 1973 Method of tests for building limes, parts I to XI.