1. **SCOPE**

   1.1. **Description of Test**

       This method covers the determination of percentage of cement in freshly mixed cement treated base. Determinations are based on chemical titration which relates cement concentration to a known laboratory prepared mix of cement and aggregate which is being used on the job. Sufficient acid is added during a specified time period to just neutralize the OH ion which is liberated during the hydration of the cement.

2. **APPARATUS AND MATERIALS**

   2.1. **Equipment**

       22.7 L plastic carboy.

       250 ml and 650 ml graduated cylinders.

       600 ml beakers.

       Stop watches.

       100 ml titrating burette and stand.

       2 L plastic containers.

       Stainless steel stirring rods.

       Balance - sensitive to 0.1 g.

       Eye dropping bottles.

       Graph paper - 10 x 10.
2.2. **Materials**

2.7 kg bottles HCL.

Hydrochloric acid.

Phenolphthalein indicator.

3. **PROCEDURE**

3.1. **Reagent Preparation**

Add 13 L of the tap water into carboy. Carefully pour the contents of two full standard 2.7 kg bottles of concentrated hydrochlorine acid CP into the carboy. Add tap water slowly until 18 litres of solution is obtained. Note: the resulting reagent solution is in the strength of 4 parts water to 1 part acid.

3.2. **Determination of Standard Curve**

The standard curve establishes the relationship between the amount of hydrochloric acid used to neutralize the OH ion in the cement and the percent cement in the test specimen for the particular aggregates, cement and water used on the project.

Normally only one point is needed to establish the standard curve and this point is determined from duplicate tests on specimens containing 6% cement. However, if a small sample of untreated aggregate in water shows a red color on adding a few drops of phenolphthalein solution, indicating the presence of water soluable alkalis, then duplicate aggregate blank specimens should also be tested.

Make up calibration mix containing 6% cement and using aggregate from the job.

Determine moisture content of damp aggregate by using Method STP 206-3.

Obtain optimum moisture from the mix design used in the field site.

Use Table No. 1 to obtain the required quantities of aggregate, cement and water to produce a mix of 300 g.
Using the dry weight in the table calculate the wet weight of material required for 10% moisture. E.g. moisture in field sample is 5% therefore:

\[
\text{Wet Weight} \quad 257 \times 1.05 = 270 \text{ g} \\
\text{Weight of Water} \quad \frac{5}{100} \times 257 = 12.9
\]

If 10% is required 27 - 13 = 14 therefore another 14 ml of water is required to adjust the moisture to 10%.

**Table No. 1**

Quantities of Materials for Calibration Specimens containing 6% cement.

Always use 6% when establishing Standard Curve.

### 6% CEMENT

<table>
<thead>
<tr>
<th>% Moisture</th>
<th>Wt. of Dry Aggregate</th>
<th>Wt. of Cement</th>
<th>Wt. of Water</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>270</td>
<td>16.2</td>
<td>14</td>
</tr>
<tr>
<td>6</td>
<td>267</td>
<td>16.0</td>
<td>17</td>
</tr>
<tr>
<td>7</td>
<td>264</td>
<td>15.9</td>
<td>20</td>
</tr>
<tr>
<td>8</td>
<td>262</td>
<td>15.7</td>
<td>22</td>
</tr>
<tr>
<td>9</td>
<td>260</td>
<td>15.6</td>
<td>25</td>
</tr>
<tr>
<td>10</td>
<td>257</td>
<td>15.4</td>
<td>27</td>
</tr>
<tr>
<td>11</td>
<td>255</td>
<td>15.3</td>
<td>30</td>
</tr>
<tr>
<td>12</td>
<td>253</td>
<td>15.2</td>
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<tr>
<td>13</td>
<td>250</td>
<td>15.0</td>
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<tr>
<td>14</td>
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<td>246</td>
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<tr>
<td>17</td>
<td>242</td>
<td>14.5</td>
<td>44</td>
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<td>18</td>
<td>240</td>
<td>14.4</td>
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<td>19</td>
<td>238</td>
<td>14.3</td>
<td>48</td>
</tr>
<tr>
<td>20</td>
<td>236</td>
<td>14.2</td>
<td>50</td>
</tr>
</tbody>
</table>

Note: % Moisture content calculation is based upon the weight of dry aggregate plus cement.

% Cement content calculation is based upon the weight of dry aggregate.

Pour the weighed aggregates and cement into the 2 L plastic containers and dry mix thoroughly with a stainless steel stirring rod.
Add the quantity of water given in Table No. 1 and again mix thoroughly.

Allow the mixture to stand in the covered 2 L plastic container for a period of time which corresponds to the time required to obtain a field treated sample, transport it to the testing location and prepare it for test. Normally the standing period should not be less than 30 minutes nor more than 90 minutes.

At the end of the standing period add 1000 ml of tap water to each test sample and start timer.

Use the constant neutralization test procedure in accordance with the procedure given in testing of field samples.

Plot the grams of acid used against percent cement for duplicate 6% cement specimens.

Draw a straight line from the origin of the chart to the average of the tests on the two 6% specimens. This is the standard curve for determining the cement content of the field mixed cement treated specimens.

3.3. Sample Preparation

Secure one 3 kilogram sample of the cement treated aggregate. When central plant mix methods are used the samples should be taken directly from the output conveyor belt or alternately from various locations in a loaded truck.

3.4. Test Procedures

Prepare two 300 gram test specimens.

Place each specimen in a plastic container and allow to stand in the covered containers to complete the curing time established as a standard for the project.

At the end of the standing period add 1000 ml of tap water to each test sample and start timer.

Stir the sample thoroughly.

Add two full droppers (approximately 40 drops) of phenolthalein solution to each sample. The water will turn red due to the presence of cement.

At ten minutes after the addition of water, start adding the 3N hydrochloric acid with a titrating burette while stirring continuously. Add the acid carefully and slowly. The
objective of adding the acid is to neutralize the OH ions as they are released into solution. When all of the free OH ions in the solution have been neutralized, the red color will disappear.

During the first minute of stirring and adding acid, stir the test sample continuously and add acid only as necessary to cause the color to disappear.

Make subsequent additions of acid at random as the red color reappears using only the minimum amounts necessary to cause the color to just disappear and maintain a neutral solution.

Besides the stirring accomplished during the initial introduction of the acid, additional light stirring (3 seconds) should be performed every minute with every fifth minute being devoted to deep stirring (6 seconds).

Continue this procedure of adding acid and stirring until one hour after the initial addition of water to the specimens.

Read and record to the nearest 0.5 ml the amount of acid added to each test sample.

Locate the amount of acid added to the test sample on the ordinate of the plotted calibration curve. Extend a horizontal line from this point to its intersection with the calibration curve. From this point extend a vertical line until it intersects the abscissa of the curve plotted for calibration samples. This intersection indicates the amount of cement in the sample.

4. ADDITIONAL INFORMATION

4.1. Precautions

This test should not be performed by persons subject to color blindness.

It is very important that the operator vigilantly maintain a neutral solution by repeatedly adding acid as soon as the pink color reappears. Neglect of this item will cause inaccuracies in the cement determinations and uncomparable test results.

When adding acid, use only the amount necessary to just eliminate the red color. Excess acid may attack the aggregates, particularly in the later phases, when the amount of hydration products from the cement remaining in the mixture may be low, resulting in erroneous cement determinations. It is also possible that the use of excessive acid in the final stages could cause the total quantity of acid to exceed that which would have normally been attained at the end of the one hour time period.
Try to maintain the same procedure for adding acid as variances in your titrating will tend to give erroneous results.

Keep all reagent bottles closed when not in use to prevent evaporation and the consequent change in acid concentration.

Rinse and drain all glassware and plasticware when the day’s work is done.

A standard curve should be determined at least once per day. In the event of any change in source of aggregate or cement.

When a new working solution of acid is introduced, a new standard curve should be determined.

5. **RESULTS**

5.1. **Reporting Results**

· Report % Cement on Form MR-84.
APPROVAL SHEET

New __ Revision X __ Date of Previous Document 89-04-26
Effective Date: __ - __

Description of Revision (Reason for Revision):
Format of test procedure updated.

Review/Implementation Process:
Reviewed by the Materials Section of the Technical Standards and Policies Branch.

Other Manuals/Policies Affected:
Nil

Follow Up/Training Required:
Nil

Comments/Concerns/Implications (Budget/Environment/Stakeholders):

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Approval Recommended by  A.R. Gerbrandt    __________
Dir., Technical Standards & Policies Br.   Date  92-07-23

Approved by  D.G. Metz    __________
Assistant Deputy Minister, Infrastructure   Date  92-07-25

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Update Mailed __ - __