CONCRETE CORES CHEMICAL LAB. PROCEDURE ; M/T C-12.5
REFERENCE: ASTM C-1084-92

*** *** *This procedure does not purport to address all the safety concerns associated with its use. It's the responsibility of the user of this procedure to establish appropriate health and safety practices and dispose of hazardous materials in an approved manner.*

Test: Portland Cement Content of Hardened Hydraulic-cement Concrete

Equipment: Standard Lab and Safety Equipment, Substantial Degree of chemical skill using relatively elaborate instrumentation.

Preliminary: See **Calculations.**

Sample Preparation;
The concrete core is cut into slices 0.5"-0.75" using a masonry chop saw(water-cooled). The slices of core are submitted to the chemical Lab. If powdered samples are submitted go to step no. 6.

1. Dry the sliced sample with compressed air and place it in a large, clean mortar and pestle. Place a piece of brown paper under the pestle to catch spills.

2. Crush the sample carefully until it passes a 4.75mm(No.4) sieve and mix thoroughly. Thoroughly mix by coning from one brown paper to another at least 10 times. This will assure there is very little segregation of the different sizes of aggregate. Other techniques will work also. The objective is to get a thoroughly mixed cone of sample to split in the next step.

3. Using a four way splitter, split the sample until 10-20 grams is leftover. One may choose to save the waste generated for additional testing. No more than 20 grams may be used in the next step.

4. Place the 10-20 gram sample into a Spex wrist-action ball mill container(Tungsten Carbide type) . Add some Genetron 113 reagent or other grinding aid. Add the balls, gasket, close and grind for 15 minutes. Remove container from mill. This step will reduce the sample size to less than 200 mesh.

5. Using a spatula spread the sample to air dry on a 'manila' folder.

6. Any submitted powdered sample should be sieved through a 300-um(No.50) mesh screen. Ball mill ground samples don't need to be sieved through the 300-um(No.50) mesh screen.

Chemical Procedure:
1. Place in a metal container and dry in an oven at 220 to 240°F (105 to 115°C) for 1 h.

2.. Desiccate the sample for 30 min. and weigh out 2.0000 grams into a 400 ml beaker in duplicate.

3. Add 50 ml of 1:1 Hydrochloric Acid using a graduated cylinder. Observe the reaction which occurs. Considerable effervescence indicate a substantial amount of calcite or carbonated paste. Delayed effervescence suggests a dolomitic aggregate.
Place on a hot plate for 15 minutes.

4. Prepare funnels with #41 Whatman inside a #40 Whatman into a 400 ml beaker.

5. Decant gently the hot liquid into the filter paper. Rinse with hot distilled water several times and rapidly police and rinse the beaker. Discard the filter paper. Reserve the filtrate.

6. The following steps are similar or the same as the regular portland cement analysis procedure as found in the cement lab. Add 2-3 drops of Methyl Red indicator to the filtrate and bring to a boil.

7. Remove from boiling and place in a fume hood. Add 1:1 Ammonium Hydroxide with a dropping bottle until the solution is basic (yellow). This reduces the Fe^{3+} to Fe^{2+}
Cool 5-7 minutes and filter through a #40 Whatman paper into 600 ml beakers to remove the Fe^{2+} and Al^{3+} compounds.

8. Police rapidly and rinse the precipitate four to six times by directing the stream of reagent carefully into the apex of the paper starting from the top edge of the paper with a circular motion. Use 20 gram/L Ammonium Nitrate(hot).

9. Acidify the filtrate with 5 mL of concentrated HCl and bring to near boiling. Add 40 mL of hot 5% Ammonium Oxalate, stir, and make solution basic with 1:1 Ammonium Hydroxide. Add 10 drops in excess. Allow to digest for 60 min ± 5 min.

10. Filter through #40 Whatman filter paper into waste beakers or a clean 600 ml beaker if magnesium oxide analysis is desired. Rinse precipitate with about 100 ml total of hot distilled water. Add filter pulp if creeping of precipitate occurs. Police well.

11. Add 200 mL distilled water and 10 ml Sulfuric Acid (conc) to mother beakers and bring to near boiling. Place the filter paper into the acidic solution and carefully macerate with a stirring rod.

12. With gentle stirring titrate to a pink endpoint which persists for at least 30 seconds. Use standardized potassium permanganate.

Calculations:

1. Calculate the percentages of cement, sand, flyash and stone in the cores using weights provided by the concrete lab mix design. If a constituent does not have a value for calcium oxide in the chemical lab records then a calcium oxide determination must be done.

2. (mL of $KMnO_4$ x Factor) / 4 = % CaO (divided by 4 for a 2.0 gram sample)

Then….. %CaO- %CaO(aggregate) - %CaO(sand) - %CaO(flyash) - %CaO(other) = %CaO corrected

gives….. (%CaO corr) / (%CaO in cement) = Actual cement in percent in core.

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