

DETERMINATION OF PORTLAND CEMENT CONTENT IN CEMENT TREATED BASE MATERIAL

(An Arizona Method)

SCOPE

1. (a) This test procedure covers a method for the determination of the percentage of portland cement in cement treated base material.

(b) This test method may involve hazardous material, operations, or equipment. This test method does not purport to address all of the safety concerns associated with its use. It is the responsibility of the user to consult and establish appropriate safety and health practices and determine the applicability of any regulatory limitations prior to use.

(c) See Appendix A1 of the Materials Testing Manual for information regarding the procedure to be used for rounding numbers to the required degree of accuracy.

(d) Metric (SI) units and values are shown in this test method with English units and values following in parentheses. Values given for metric and English units may be numerically equivalent (soft converted) for the associated units, or they may be given as rounded or rationalized values (hard converted). Either the metric or English units along with their corresponding values shall be used in accordance with applicable specifications. See Appendix A2 of the Materials Testing Manual for additional information on the metric system.

APPARATUS

2 Requirements for the frequency of equipment calibration and verification are found in Appendix A3 of the Materials Testing Manual. Apparatus for this test procedure shall consist of the following:

- (a) Burette - 50 mL capacity with teflon stopcock and burette cover.
- (b) Burette clamps.

- (c) Ring stand.
- (d) Pipette - 10 mL capacity, accurate to 0.05 mL.
- (e) Plastic dropper dispenser - 100 mL.
- (f) Plastic wash bottles - 100 mL capacity, 500 mL capacity, 1000 mL capacity.
- (g) Graduated cylinders - (transparent plastic), 100 mL capacity, 500 mL capacity, 1000 mL capacity.
- (h) A balance or scale capable of measuring the maximum weight to be determined and conforming to the requirements of AASHTO M 231, except the readability and sensitivity of any balance or scale utilized shall be at least 0.1 gram.
- (i) An analytical balance capable of measuring the maximum weight to be determined and conforming to the requirements of AASHTO M 231, except the readability and sensitivity of any balance utilized shall be at least 0.0001 gram.
- (j) Beakers - (plastic), 300 mL capacity, 2000 mL capacity, 4000 mL capacity.
- (k) Stirring rods - stainless steel, 305 mm (12") long; glass, not over 150 mm (6 inches) long.
- (l) Erlenmeyer flasks - (plastic), 500 mL capacity with stoppers.
- (m) Variable control hot plate.
- (n) Spatulas, cleaning brushes, watch glasses.
- (o) Stop watch or equally efficient timing device.
- (p) Plastic bottles - 3.8 liter (1 gal.), 1 liter (1 qt.).
- (q) 19.0 liter (5 gallon) polyethylene jug.

REAGENTS

3. Reagents shall be prepared as described below. Distilled or de-ionized water shall be used in their preparation.

(a) 2-Propanol (Analyzed Reagent)

(b) Tartaric Acid 72% Solution (TAS).

1) For a 19.0-liter (5 gallon) batch, weigh 13680 grams of reagent grade D-Tartaric Acid into a 19.0 liter (5 gallon) polyethylene jug, or other suitable non-reactive container, precalibrated to indicate a 19.0 liter (5 gallon) volume.

2) Add boiling water to fill the jug to the 19.0 liter (5 gallon) mark. Mix thoroughly and let set overnight.

3) Filter into another 19.0 liter (5 gallon) jug and dilute to the 19.0 liter (5 gallon) mark.

(c) Potassium Hydroxide 1-Normal Titrating Solution (TS).

1) Weigh 56.1 grams reagent grade Potassium Hydroxide into a one liter polyethylene jug, or other suitable non-reactive container, precalibrated to indicate a one liter volume. The Potassium Hydroxide shall not be placed in a glass container.

2) Add sufficient water to dissolve the Potassium Hydroxide. Mix thoroughly and allow to come to room temperature. Dilute to the one liter mark. This solution shall not be stored in a glass container.

(e) 1% Methyl Red Indicator.

1) For a 50 mL batch, weigh 0.5 gram methyl red dye into a 100 mL beaker.

2) Add 25 mL 2-propanol and dissolve.

3) Fill to 50 mL mark with distilled water.

4) Transfer to the dropper dispenser.

PREPARATION OF STANDARD SAMPLES

4. (a) Standard samples shall be prepared using the same materials and mix design formula as the job from which field samples will be taken. A representative minimum 3000 grams of soil/aggregate material with no added cement shall be used to prepare the standard samples. From this, three representative portions to be used for preparing standard samples shall be obtained using a standard reduction method such as given in AASHTO T 248. The standard samples shall be prepared with differing cement contents bracketing the mix design cement content as follows:

- (1) 0.0% cement content.
- (2) The mix design cement content, expressed to the nearest 0.1%.
- (3) 5.0% more cement than the design content.

(b) The three components of the standard samples are cement, soil/aggregate material, and water. The total sample weight shall be 300 grams. The weight of water from the job is 30 grams. The dry components of the standard sample are cement and soil/aggregate material with a combined weight of 270 grams. The state of dryness of the soil/aggregate material used in preparing standard samples may be either saturated-surface-dry, air-dry, or oven-dry. It is important that the same state of dryness be obtained for all standard samples and for field test samples, [See paragraph 8(a)]. Use the following formulas to determine the dry components of each standard sample:

$$W_C = 300 \times \frac{C}{100}$$

$$W_{S/A} = 270 - W_C$$

Where: W_C = Weight of cement, grams
 $W_{S/A}$ = Weight of soil/aggregate, grams
 C = Cement content, % of total sample

NOTE: In case a large quantity of large aggregate is present in a cement treated base job, a minimum of 15,000 grams of soil/aggregate material should be obtained for preparation of standard samples, and the following procedure followed:

- (1) For each standard sample, 5000 grams of soil/aggregate material is obtained using the standard reduction method and separated into coarse and fine fractions.
- (2) The coarse fraction is washed with distilled water and its adhering fines are added to the fine fraction.
- (3) From the resulting combined fine fraction, a representative portion is obtained using the standard reduction method.

TEST PROCEDURE FOR STANDARD SAMPLES

5. (a) Transfer the dry components to the 2000 mL plastic beaker.

NOTE: It is preferable to weigh the cement as accurately as possible. Use an analytical balance to weigh the desired amount of cement. Then transfer the cement to the aggregate already in the plastic beaker.

- (b) Mix thoroughly. AVOID PRODUCING DUST.
- (c) Add 30 grams of water that is used on the job for mixing the batch.

NOTE: At this point it may be desirable to thoroughly mix the standard sample and transfer it to a suitable container and allow it to cure for a sufficient time to approximate the expected condition of the field sample at the time it is tested, after which the standard sample must be broken down as necessary to facilitate mixing and intimate contact with the reagent (TAS). An apparatus such as the one described in AASHTO T 87 may be utilized for this purpose. Care should be taken to avoid reducing the size of individual natural particles. Quantitatively transfer the standard sample to the 2000 mL beaker.

- (d) Add 400 ± 0.1 mL of distilled water to the beaker.
- (e) Stir thoroughly and vigorously for 3 ± 1 minutes.

(f) Cover the 2000 mL beaker with a watch glass. Keep the beaker covered with a watch glass at all times, except when actually in use.

(g) Let stand undisturbed for 40 ± 5 minutes.

(h) Stir thoroughly and vigorously for 2 minutes. Let stand undisturbed for 1 to 2 minutes.

(i) Accurately measure 300 mL Tartaric Acid 72% Solution (TAS) into a 500 mL graduated cylinder.

NOTE: Keep all containers of all reagents covered when not in use. Keep all stock containers sealed.

(j) Add TAS slowly and with continued stirring to the beaker.

NOTE: If effervescence occurs, stop addition of TAS and stir vigorously until bubbling ceases, complete the addition of TAS, then stir at least 2 minutes continuously.

(k) Let the sample stand undisturbed for 10 minutes.

(l) Accurately measure 710 mL of 2-propanol into a 1000 mL graduated cylinder.

(m) Add the 2-propanol, all at once, to the beaker, completely draining the cylinder. Immediately stir, rapidly and thoroughly, for exactly 2 minutes, so that all the contents of the beaker are washed with 2-propanol. Cover with a watch glass, and let the beaker stand undisturbed until a clear liquid layer forms.

(n) Carefully pour only the clear liquid into a 500 mL plastic Erlenmeyer flask. Tightly stopper and seal the flask.

TITRATION PROCEDURE

6. (a) Fill the 50 mL burette completely with Potassium Hydroxide 1-Normal Titrating Solution (TS). Open the stopcock and let the burette drain completely.

(b) Refill the burette with TS, and adjust the bottom of the meniscus to read 0.0 mL.

NOTE: Make sure that there are no air bubbles anywhere in the system. Always check just below the stopcock for air bubbles when the burette is draining. Keep the top of the burette covered.

(c) Transfer an aliquot of 10.00 ± 0.05 mL of the clear liquid from the flask to a 300 mL plastic beaker using a 10 mL pipette.

(d) Add 100 mL distilled water to the beaker, using a 100 mL graduated cylinder.

(e) Stir the contents of the beaker with a glass stirring rod. Do not remove the stirring rod until the titration is completed.

(f) Add 2 drops of 1% Methyl Red Indicator and stir the solution. The test solution should now be a clear, dark-red color. The color change is as follows:

Start: Clear, dark-red solution.

To: Clear, pure orange solution.

End: Clear, pure lemon-yellow solution.

(g) Rapidly, with stirring, add TS until the solution turns to an orange color.

(h) Stir the solution.

(i) Set the burette stopcock to deliver the TS one drop at a time, with stirring between each addition. As the end point is approached, the solution should turn to a clear red-orange or clear yellow-orange color.

(j) Decrease the rate of addition of TS, and stop addition when one drop produces a pure lemon-yellow clear solution. Allow the solution to stand undisturbed for one minute to check for color fading.

(k) If the color fades, add one more drop and again allow to stand undisturbed for one minute. Repeat until the color does not fade.

(l) Read the volume of TS used to the nearest 0.1 mL at the bottom of the meniscus.

(m) If time is available, run at least two titrations for each sample and obtain the average volume of TS used.

PREPARATION OF THE STANDARD CURVE

7. (a) The standard curve is prepared using linear graph paper.
- (b) Plot the number of mL of TS, along the X-axis.
- (c) Plot the percentages of portland cement, along the Y-axis.
- (d) Draw the straight line of best fit for the three points.

TEST PROCEDURE FOR FIELD SAMPLE

8. (a) From a representative minimum 1000 gram field sample of the cement treated base material to be tested, at the same state of dryness as the standard samples, obtain a 270 ± 0.1 gram test sample using the standard reduction method used with the standard samples and transfer it to a 2000 mL plastic beaker.

NOTE: As necessary, the field sample must be broken down to facilitate weighing, mixing, and intimate contact with the reagent (TAS). An apparatus such as the one described in AASHTO T 87 may be utilized for this purpose. Care should be taken to avoid reducing the size of individual natural particles.

NOTE: If much large aggregate is present, a representative minimum of 5000 gram field sample should be used. It should be separated into coarse and fine fractions and the coarse fraction washed with distilled water to obtain a combined fine fraction from which the test sample is prepared as above. [See note following paragraph 4(b).]

- (b) Add 400 ± 0.1 mL of distilled water to the beaker.
- (c) Stir thoroughly and vigorously for 3 ± 1 minutes.
- (d) Cover the beaker with a watch glass. Keep the beaker covered with a watch glass at all times, except when actually in use.

- (e) Let the beaker stand undisturbed for 40 ± 5 minutes.
- (f) Follow the procedure as described in paragraphs 5(h) through 5(n).

TITRATION PROCEDURE

- 9. Titrate in accordance with Section 6.

REPORT

- 10. Read results directly from the Standard Curve as percent portland cement in the field sample. Report the result to the nearest 0.1%.