

APPENDIX C

VTM-40, TITRATION & CEMENT PLANT CALIBRATION

Virginia Test Method - 40

Determining Cement Content of Freshly Mixed Cement-Aggregate Mixtures

November 1, 2000

1. Scope

- 1.1 This method of test is intended for determining the cement content of cement-aggregate mixtures sampled at the central mix aggregate plant.
- 1.2 This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Apparatus

- 2.1 Balance - A balance having a capacity of at least 1,000 grams with a sensitivity of at least 0.1 gram.
- 2.2 Timer - A timer with a capacity of 10 minutes or more and a sensitivity of at least 0.1 second.
- 2.3 Glassware - 25 ml graduated cylinder, 1,000 ml cylinder, 2000 ml volumetric flask, 50 ml burettes, 10 ml volumetric pipettes, 250 ml Erlenmeyer flasks, medicine droppers.
- 2.4 Plasticware - 2 qt. polyethylene containers with snap-on covers, 12 in. diameter plastic funnel, 5 gal. polyethylene bottles for ammonium chloride, 5 gal. polyethylene bottles for demineralized water.
- 2.5 Burette Stand for 50 ml burette.
- 2.6 Magnetic Stirrer and Stirring Bar.
- 2.7 Stirring Rods - Glass stirring rods approximately 12 in. long.
- 2.8 Indicator Paper - Supply of indicator paper, pH range from 10 to 14.
- 2.9 Pipette Filler.
- 2.10 Sample Splitter - Maximum size 1 1/2 in.

3. Reagents

- 3.1 Ammonium Chloride Solution (10%) - Transfer 1893 g of U. S. P. granular ammonium chloride (NH_4Cl) to a 5-gal. plastic bottle.
Make up to 5 gallons with distilled or demineralized water and mix well.
- 3.2 EDTA Solution (0.1 M) - Dissolve 74.5 g of reagent grade disodium (ethylenedinitrilo) tetraacetate dehydrate ($\text{Na}_2\text{C}_{10}\text{H}_{14}\text{N}_2\text{O}_8\cdot 2\text{H}_2\text{O}$) powder in about one litre of warm, distilled or demineralized water in a beaker. Cool to room temperature, transfer quantitatively to a 2-liter volumetric flask and make to the mark with distilled or demineralized water. Store in polyethylene bottle.
- 3.3 Hydroxy Naphthol Blue may be used as the indicator.
- 3.4 Sodium Hydroxide Solution (50%) - Add 500 g of reagent grade sodium hydroxide (NaOH) pellets in 600 ml of distilled or demineralized water and allow to cool to room temperature. Dilute to one litre with distilled or demineralized water. Store in plastic bottle. Dilute 1:1 with distilled or demineralized water for use. Caution: Solution shall be mixed in the order given to avoid spontaneous reaction.
- 3.5 Triethanolamine Solution (20%) - Dilute 100 ml of reagent grade triethanolamine (HOCH_2CH_2)₃ N to 500 ml with distilled or demineralized water.

4. Procedure for Preparing Calibration Curve

- 4.1 From the materials to be used for construction, prepare 3 sets of duplicate samples at the design moisture content and containing the following amounts of cement:
- Set 1. Two (2) samples at 75 percent of the design cement content.
- Set 2. Two (2) samples at 100 percent of the design cement content.
- Set 3. Two (2) samples at 125 percent of the design cement content.

Using a sample size of 600 grams for each sample, compute the quantities of aggregate, cement and water as follows:

$$W_a \text{ (total weight of aggregate, g)} = \frac{\text{Sample Size}}{(1 + M/100)(1 + C/100)}$$

$$W_r \text{ (weight of material retained on No. 4 sieve)} = \frac{R}{100} \times W_a$$

$$W_f \text{ (weight of material passing No. 4 sieve, g)} = W_a - W_r$$

$$W_c \text{ (weight of cement, g)} = \frac{C}{100} \times W_a$$

$$V_w \text{ (volume of water, ml)} = \frac{M}{100} (W_a + W_c)$$

Where:

M = design moisture content, percent by dry weight

C = cement content, percent by dry weight of aggregate, and

R = percent material retained on No. 4 sieve.

For each sample, mix the aggregate and cement thoroughly to a uniform color. Add the water and mix thoroughly.

Titrate each 600 g sample as described under Procedure for Titration. After titrating the 6 samples, construct a graph showing ml of EDTA solution vs. per cent cement by weight using average figures from Sets 1, 2, and 3.

A separate calibration curve shall be prepared for each brand, type and source of cement. When Type I-P is used, a separate calibration curve shall be prepared for each shipment in which the percent of fly ash varies by more than ± 3.0 per cent from the quantity for which a curve has been established.

5. Procedure for Test Samples

- 5.1 At the central mix aggregate plant, samples of the cement-aggregate mixture shall be taken at the completion of mixing. The samples are to be tested immediately or placed in covered plastic containers and tested within one hour of the completion of mixing.

For testing, weigh a 600 g portion and titrate as described under Procedure for Titration.

Note 1 - If a correction is to be made for variations in moisture content, determine the moisture content (M') of a separate portion of the material passing a No. 4 sieve. Computation for the correction are given under Calculations, Note 4.

6. Procedure for Titration

- 6.1 Place each 600 g sample in a 2-qt. polyethylene container and add 1,200 ml ammonium chloride solution. Place cover on the container and shake the mixture for 2 minutes (± 2 seconds). Allow the mixture to settle for 4 minutes (± 2 seconds). Pipette a 10 ml aliquot of the supernatant solution into a 250 ml Erlenmeyer flask and add 100 ml of distilled or demineralized water. While thoroughly mixing on a magnetic stirrer, add drops of sodium hydroxide solution until a pH between 13.0 to 13.5 is obtained as measured by the indicator paper. Use stirring rod to transfer drops of solution to indicator paper, add 4 drops of triethanolamine solution and then add about 0.2 g of the indicator powder. While the solution is being stirred on the magnetic stirrer, titrate with EDTA and record the quantity in ml to a pure blue endpoint.

Note 2 - A sharper endpoint may sometimes be obtained by adding approximately half of the anticipated quantity of EDTA solution before the addition of sodium hydroxide.

Note 3 - All equipment must be kept scrupulously clean by thorough rinsing with distilled or demineralized water. All reagents must be stored in polyethylene containers.

7. Calculations

Read the cement content by dry weight directly from the calibration curve corresponding to the titration results in ml of EDTA for the test sample.

Note 4 - Variations of moisture content (above 2%) will have a slight effect on the accuracy of test. Correction for moisture variation may be computed as follows:

$$C' = \frac{1 + M'/100}{1 + M/100} C$$

Where: C' = percent cement corrected for moisture variation,

C = percent cement determined from test sample,

M' = percent moisture of test sample as determined in Paragraph 5, Note 1, and

M = design moisture content.

GENERAL INFORMATION

8. Miscellaneous

8.1 Size of sample - Obtain a 10 lb. sample. Split this sample over a splitter until about a 600 g sample is obtained. Weigh exactly 600 g as sample size for testing.

8.2 Sampling

8.2.1 In all cases, samples shall be taken in a stratified random manner.

8.2.2 When sampling from a truck, the truck should be divided into quadrants and the 4 samples shall be taken from randomized quadrants.

VTM - 40

VIRGINIA TEST METHOD FOR
DETERMINING CEMENT CONTENT
OF FRESHLY MIXED
CEMENT-AGGREGATE MIXTURES

SCOPE

- 1.1 This method of test is intended for determining the cement content of cement-aggregate mixtures sampled at the central mix aggregate plant

SCOPE

- 1.2 This standard may involve hazardous materials , operations and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

APPARATUS

- 2.1 Balance - minimum capacity 1000 grams
sensitivity 0.1 gram
- 2.2 Timer - minimum capacity 10 min. sensitivity
0.1 second
- 2.3 Glassware - 25 ml graduated cylinder, 1000 ml
cylinder, 2000 ml volumetric flask, 50 ml
burettes, 10 ml volumetric pipettes, 250 ml
Erlenmeyer flask, medicine droppers

APPARATUS

- 2.4 Plasticware - 1.9 l polyethylene containers with
snap on covers, 305 mm diameter plastic funnel,
1.9 l bottles for ammonium chloride, 1.9 l bottles
for demineralized water
- 2.5 Burette Stand for 50 ml burette
- 2.6 Magnetic Stirrer and Stirring Bar
- 2.7 Stirring Rods - glass stirrings rods approximately
305 mm long

APPARATUS

- 2.8 Indicator Paper - Supply of indicator paper,
pH range from 10 - 14
- 2.9 Pipette Filler
- 2.10 Sampler Splitter - Maximum size 37.5 mm (1½")

Reagents

- 3.1 Ammonium Chloride Solution (10%)
- 3.2 EDTA Solution (0.1 M)
- 3.3 Hydroxy Naphthol Blue may be used as indicator
- 3.4 Sodium Hydroxide Solution (50%)
- 3.5 Triethanolamine Solution (20%)

Preparing a Calibration Curve

- Set 1. Two samples at 75% of the design cement content
- Set 2. Two samples at 100% of the design cement content
- Set 3. Two samples at 125% of the design cement content

Sample size 600 grams

Calculate amount of water
 $600 / (1 + \%M / 100) = \text{aggr. \& cement}$
 $600 / (1 + 5.7 \% / 100) = \text{aggr. \& cement}$
 $600 / (1 + .057) = \text{aggr. \& cement}$
 $600 / 1.057 = \text{aggr. \& cement}$
 $567.64 = \text{aggr. \& cement}$
 $600 - 567.64 = 32.36 \text{ grams of water}$

Sample size 600 grams

Calculate amount of cement
 $567.64 / (1 + \% C / 100) = \text{aggregate}$
 $567.64 / (1 + 3.0 \% / 100) = \text{aggregate}$
 $567.64 / (1 + .030) = \text{aggregate}$
 $567.64 / (1.030) = \text{aggregate}$
 $551.11 = \text{aggregate}$
 $567.64 - 551.11 = 16.53 \text{ grams of cement}$

Sample size 600 grams

Calculate amount of + and - 4
 $551.11 * (.4\% / 100) = - 4 \text{ material}$
 $551.11 * (.48\% / 100) = - 4 \text{ material}$
 $551.11 * .48 = - 4 \text{ material}$
 $264.53 = - 4 \text{ material}$
 $551.11 - 264.53 = 286.58 = + 4 \text{ material}$

Sample size 600 grams

Totals for 3%
- 4 material = 264.53
+ 4 material = 286.58
Cement = 16.53
Water = 32.36
Total = 600.00

Sample size 600 grams

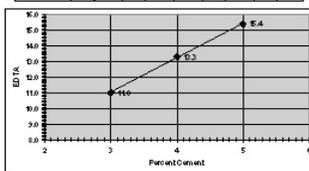
Totals for 4%
 - 4 material = 261.99
 + 4 material = 283.82
 Cement = 21.83
 Water = 32.36
 Total = 600.00

Sample size 600 grams

Totals for 5%
 - 4 material = 259.49
 + 4 material = 281.12
 Cement = 27.03
 Water = 32.36
 Total = 600.00

Titration Calibration Curve

TIME	2 Sp. C = 3%	100% C = 4%	2 Sp. C = 5%
	AXIS 1	AXIS 1	AXIS 2
FINISH	103 22.0	107 26.8	115.0 30.3
START	00 103	0.0 107	0.0 115.0
DIFFERENCE	103 112	107 12.8	115.0 15.4





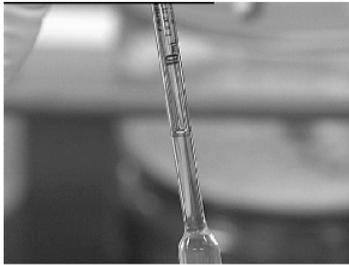




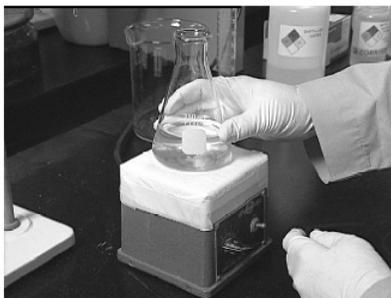


















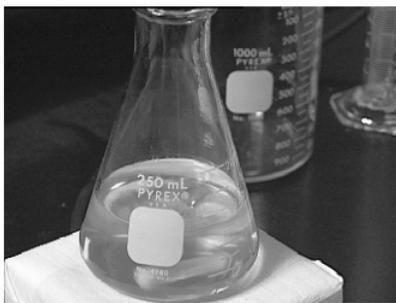


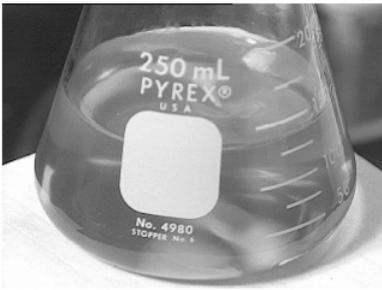




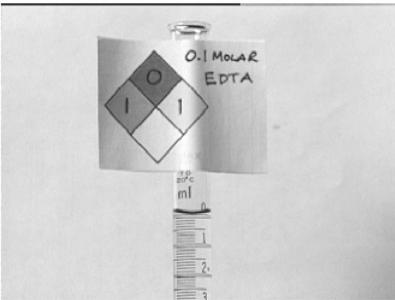


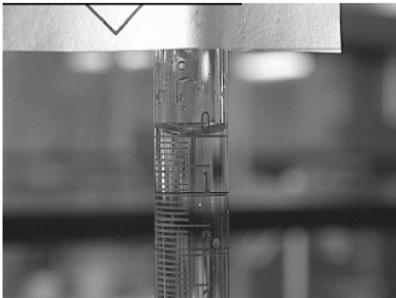








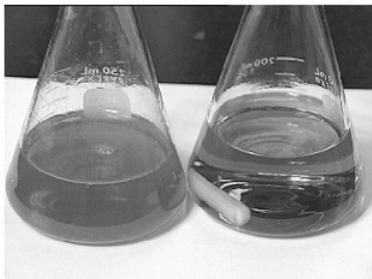


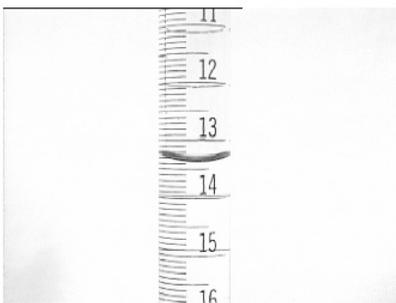


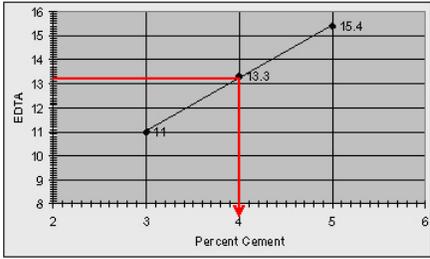


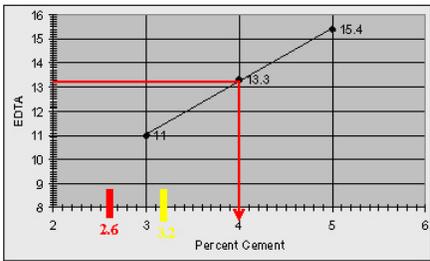












Cement Content Test Reagents and Steps

Step	Reagents	Reaction	Danger	Sensitivity
1	600 g Aggregate			0.1 g
2	1200 ml Ammonium	Extracts Calcium (Ca)	No	No
3	100 ml Distilled - Demineralized Water	Dilutes	No	No
4	Sodium Hydroxide	To bring pH to 13-13.5 because change in color will only take place at this pH.	Yes	No
5	4 drops Triethanolamine	Takes care of possible interference by (Fe) and (Mg) by forming a complex compound.	No	No
6	0.2 g Cal Red or Hydroxynaphthol Blue	An indicator absorbs all but pink color; therefore, looks pink to begin with.	No	No
7	EDTA	Reacts with Calcium (Ca) then the first excess drop will react with indicator to change its absorption of light. Now it will absorb all but blue; therefore it looks blue.	No	No

A central-mix aggregate plant is a relatively simple operation. Here two or more aggregates, having different gradations, are blended together to produce an aggregate mixture that meets Gradation and Atterberg Limits specifications for a particular application or size.

Aggregate is removed from storage, or stockpiles, proportioned and thoroughly mixed. This aggregate mixture is then stockpiled for future use or hauled to the job site.

Aggregate Feed Supply

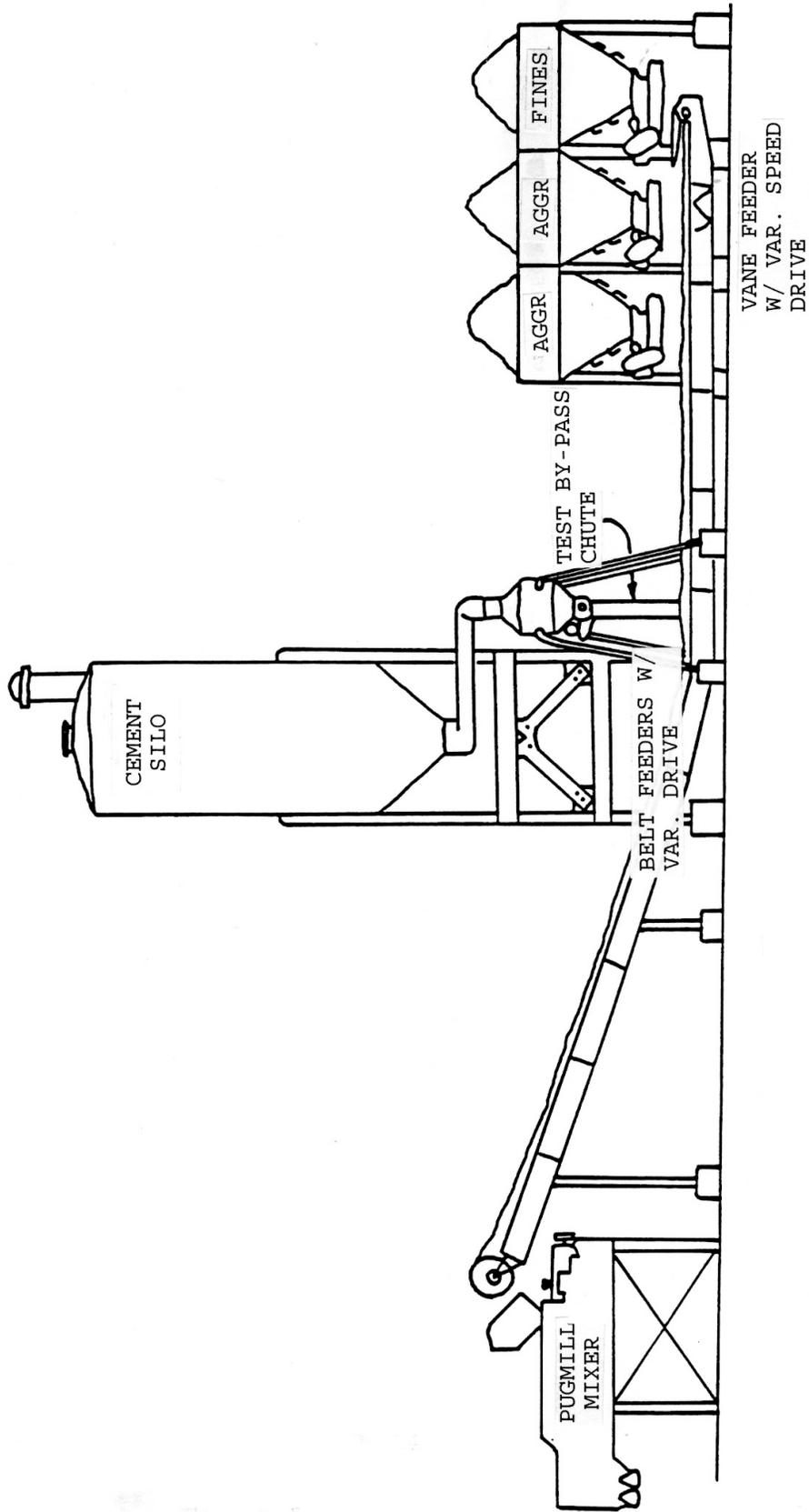
Aggregate feed is the first major component of the central-mix aggregate plant. The aggregate feeder may be charged by one or a combination of the following methods:

1. Open top bins with two, three, or four compartments, usually fed by a crane with a clamshell bucket or a front-end loader.
2. Tunnel under stockpiles separated by bulkheads. Materials are stockpiled over the tunnel by belt conveyor, truck, crane, or front-end loader.
3. Bunkers or large bins. These are usually fed by trucks, car unloaders, or bottom-dump freight car emptying directly into the bunkers.

The aggregate feed is one of the critical control points in the production flow-line. It is significant that, while most of the problems in central-mix aggregate plant production occur in the mixer or pugmill, the causes can usually be traced back to the aggregate feed.

When charging the aggregate bins, care should be exercised to minimize segregation and degradation of the aggregate. These can be prevented by taking the same precautions as for proper stockpiling. There should be sufficient material in all bins to provide a positive and uniform flow. Except for very large bins, no single bin should be run less than half-full, nor should any bin be loaded to overflow.

When the aggregate feed is stockpiled over a tunnel and belt, care should be taken in handling material over the feeders. The use of bulldozers should be discouraged. The reason they are discouraged is that on stockpiles there is serious danger of segregation and degradation. Vibrations from the bulldozer can cause fine particles in the stockpile to filter down into a layer which will be pushed to the feeder. Also, continuous abrasive action by the aggregate particles being moved about by the bulldozer, and the grinding action of its tracks, can cause degradation in both the coarse and fine aggregate stockpiles.



If the stockpile level above the tunnel is maintained by a dragline or clamshell, the operator must be careful not to pick up material from the same position in the stockpile in successive withdrawals.

When a front-end loader is used, the operator should be cautioned not to pick up material from the storage stockpile at ground level.

When trucks are used to charge the bin, they should deposit their loads directly above the feeder.

When the stockpile is replenished by overhead belts, the flow of material should be controlled by baffles or perforated chimneys.

Aggregate Feeders and Controls

Aggregate feeder units are located beneath the storage bins or stockpiles or in positions that assure a uniform flow of aggregate. Feeder units have controls that can be set to produce a uniform flow to the aggregate elevator. Generally, belt and vibratory feeders are best for accurate metering of the fine aggregates. Coarse aggregates usually flow satisfactorily with any type of feeder.

For a uniform output from the central-mix aggregate plant, input must be accurately measured. The importance of feeding the exact amounts of each size aggregate into the pugmill at the correct rate of flow cannot be overemphasized.

Cement Silo and Proportioning Devices

On certain projects, the plans will specify that the aggregate to be used in the pavement structure is to be stabilized by a chemical agent, usually cement. In these cases, an additional component needs to be added to the central-mix aggregate plant. This possible component of a central-mix aggregate plant is relatively simple. It usually consists of a standard cement silo with some type of proportioning attachment. The proportioning attachments or devices are either a simple gate control on the bottom of the silo or a variable-speed screw feeder or vane feeder. These devices must be calibrated to proportion at the desired rate.

The cement silo and proportioning device are located so that the stabilizing material (cement) can be proportioned onto the aggregate elevator belt prior to the discharge of the material into the pugmill.



Central Mix Aggregate Pugmill with Cement Silo

Water Metering

All central-mix aggregates, prior to use in road construction, must have the proper moisture content (optimum moisture + 2% points) to aid in compaction of the material on the road. This moisture content also helps hydrate the cement in cement stabilized aggregates. To insure proper moisture, central-mix aggregate plants are equipped with some type of water proportioning system. Water proportioning can be achieved by either a sophisticated water metering device or a simple water control valve calibrated by trial and error.

Water is introduced to the aggregate mixture at the input end of the pugmill through a spray bar. This spray bar emits a fine spray of water for the full width of the pugmill.

Pugmill

A twin pugmill-type mixer is commonly used in all modern central-mix aggregate plants. This unit is mounted directly beneath the aggregate elevator, and high enough so that it may discharge the mixture into the truck or other hauling unit.

Mixing

When aggregates are discharged from the aggregate elevator and deposited in the pugmill, some mixing takes place. The mixing time begins when the flow of material from the aggregate elevator is deposited in the pugmill.

The mixing time should be as long as is necessary to get a uniform distribution of aggregate sizes. Speed of the mixer shafts and the arrangement and pitch of the paddles are factors governing mixing efficiency.

Upon completion of the mixing time, the bottom of the pugmill mixer opens up and discharges the contents into a truck or other hauling equipment.

Appendix C
Titration, VTM-40
Questions

1. The Contractor shall furnish a motorized screen shaker for:
 - A. Fine aggregate gradation analysis.
 - B. Coarse aggregate gradation analysis.
 - C. Coarse and fine aggregate gradation analysis.

2. To determine the cement content of cement aggregate mixtures by the Titration Method, samples shall be taken at the:
 - A. Beginning of mixing.
 - B. Completion of mixing.
 - C. Completion of compaction on the road.
 - D. Prior to mixing.

3. When dealing with sodium hydroxide solution, you should always pour the solution into distilled or demineralized water to prevent a spontaneous reaction.
 - A. True
 - B. False

4. The method used to determine the cement content of cement aggregate mixtures is:
 - A. Reflux Method.
 - B. Centrifuge Method.
 - C. Titration Method.
 - D. Marshall Method.

5. In determining the cement content by the Titration Method, the sample for testing should weigh 600 grams.
 - A. True
 - B. False

