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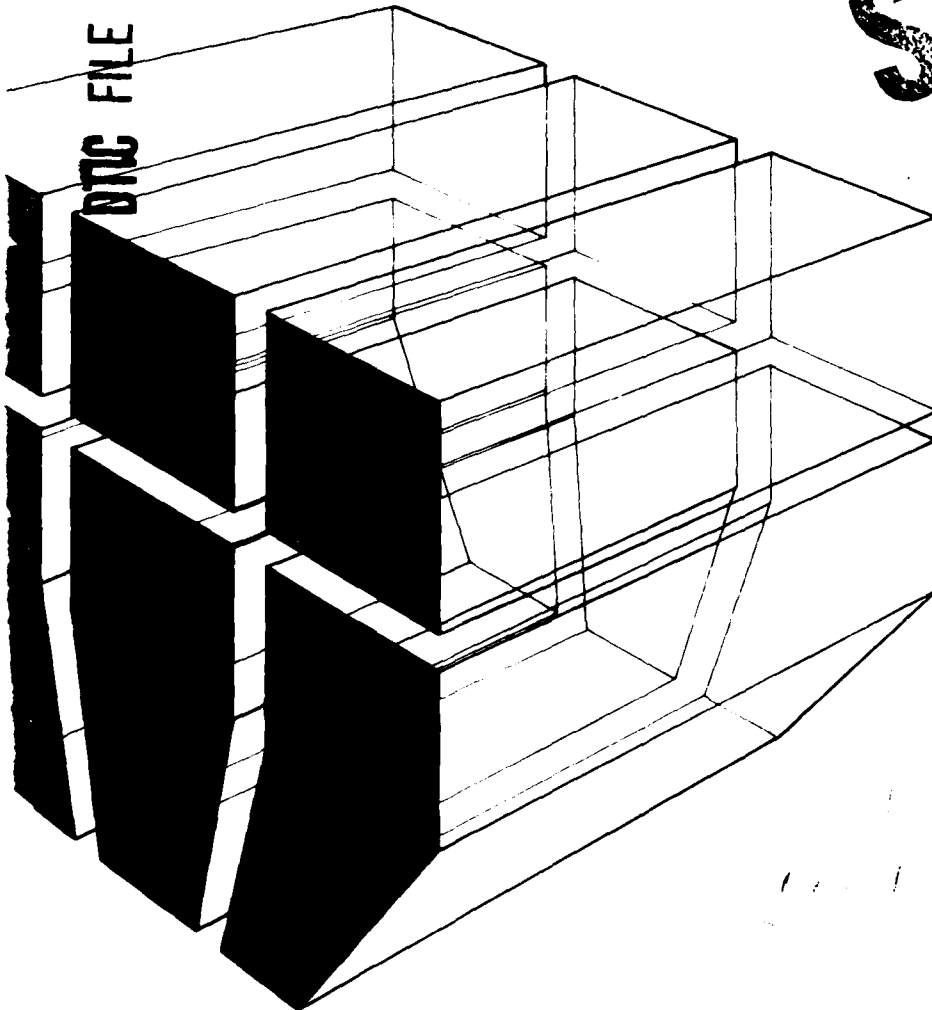
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CORPS OF ENGINEERS CONCRETE QUALITY MONITOR:
OPERATIONS GUIDE

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by
P. A. Howdyshell



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test. The results of validation tests indicate the CQM system is as accurate as the previously developed Kelly-Vail (KV) and CERL/KV methods; and that CQM water/cement ratios can be used to estimate both compressive and flexural strengths.¹

¹R. T. Kelly and J. W. Vail, "Rapid Analysis of Fresh Concrete," *Concrete* (April 1968), pp 140-145 and (May 1968), pp 206-210; and P. A. Howdysshell, *Operations Guide -- Water and Cement Content of Fresh Concrete*, Technical Report (TR) M-177/ADA022697 (U. S. Army Construction Engineering Research Laboratory [CERL], September 1975).

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FOREWORD

This work was performed for the Directorate of Military Programs, Office of the Chief of Engineers (OCE), under Project 4A762731AT41, "Military Facilities Engineering Technology"; Task A, "Military Construction"; Work Unit 039, "Rapid Testing/Plastic PCC." The OCE Technical Monitor was Mr. E. Hunt, DAEN-MPC-E.

The research was performed by the Engineering and Materials Division (EM), U. S. Army Construction Engineering Research Laboratory (CERL). Dr. R. Quattrone is Chief of CERL-EM.

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CORPS OF ENGINEERS CONCRETE QUALITY MONITOR: OPERATIONS GUIDE

1 INTRODUCTION

Background

This report describes how to set up and use the Corps of Engineers Concrete Quality Monitor (CQM), a system for determining the water and cement contents of fresh concrete. The CQM system makes it possible to judge the quality of concrete as it is being placed, thereby helping avoid the high cost of replacing defective concrete.

The CQM water test consists of mixing a known weight of concrete with a known volume and strength of a salt solution. The strength of the intermixed salt solution is then determined and directly related to the water in the concrete sample. The CQM cement test consists of separating the aggregate from the cement, uniformly suspending the cement in a fixed volume of water, dissolving a fixed volume sample of the suspended cement in dilute nitric acid, and determining the calcium strength of the dissolved solution. The calcium content of the dissolved solution is proportional to the cement content of the concrete. Both tests have proven to be rapid (the water test takes 3 to 4 minutes and the cement test takes 6 to 7 minutes), simple, field worthy, and reliable.

The CQM is the third generation of a method originally proposed by Dr. R. T. Kelly and Mr. J. W. Vail of the Greater London Council.² All three generations use the water and cement tests described above, but vary the equipment and analytical technique. The original Kelly-Vail (KV) method (Generation 1) relied on volumetric chloride ion titration to determine water content, and flame photometry (calcium signature) to determine cement content.³ The CERL/KV method (Generation 2) performed the calcium analysis (cement test) by titrating with an ethylene diaminetetra-acetate (EDTA) solution in the presence of a buffer and an eriochrome black-T indi-

cator.⁴ The CQM system (Generation 3) uses slightly different equipment to separate the aggregate and cement to obtain a representative sample of the cement suspension. It also relies on a commercially available calcium analyzer and chloride meter to determine calcium and chloride solution strengths,* respectively.

Extensive tests proved the Generation 1 and 2 KV methods to be rapid (7 to 8 minutes for each water and cement test), field worthy, and sufficiently accurate to estimate the strength potential of fresh concrete.⁵ However, these same tests indicated several deficiencies in the methods, the major one being the amount of central laboratory support required to operate in the field. Other deficiencies related to the use of fragile glassware, reagent cost, reagent stability and degradation, calibration, clean-up requirements between tests, and ease of transportation.

The CQM system has significantly improved on the Generation 1 and 2 KV methods. With the exception of a 5 percent nitric acid solution and a 0.5 normal (N) salt solution, all reagents can be purchased in small prepackaged vessels from the chloride meter and calcium analyzer manufacturer. And because only a small quantity of prepackaged reagents is required, the reagent cost per test is insignificant. Reagent degradation, calibration, clean-up, and transportation are also easier with the CQM than with the Generation 1 or 2 KV methods.

Purpose

The purpose of this report is to describe (1) the equipment, reagents, and procedures for using the CQM system, (2) the system's capabilities and limitations, and (3) the results of laboratory and field validation tests which compared the CQM system to the Generation 2 KV method.

Mode of Technology Transfer

The information in this report is applicable to the Corps of Engineers *Handbook for Concrete and Cement* (U. S. Army Waterways Experiment Station, 1949), and has potential application as an American Society for Testing and Materials (ASTM) standard test method.

²P. A. Howdyshell, *Revised Operations Guide for a Chemical Technique to Determine Water and Cement Content of Fresh Concrete*, TR M-212/ADA039120 (CERL, April 1977).

*Corning Distributors, Model 940 calcium analyzer and Model 920M chloride meter.

⁵P. A. Howdyshell, "Concrete Quality Control - 28 Days - 24 Hours - 15 Minutes," ACI Special Publication (SP-56), *Accelerated Strength Testing* (American Concrete Institute, 1978).

²R. T. Kelly and J. W. Vail, "Rapid Analysis of Fresh Concrete," *Concrete* (April 1968), pp 140-145 and (May 1968), pp 206-210.

³P. A. Howdyshell, *Operations Guide - Water and Cement Content of Fresh Concrete*, TR M-177/ADA022697 (CERL, September, 1975).

2 CQM SYSTEM — EQUIPMENT AND OPERATIONS

Equipment

Tables 1 and 2 list the kinds of equipment (and their cost) needed for the CQM water and cement content tests (also see Figures 1 and 2). In general, these apparatus are the recommended minimum needed for the CQM analysis. Several items can be replaced by other pieces of equipment that perform the same function. For example, the cement suspension tank (Item 5, Table 2) can be replaced by the commercially produced washing machine (Figure 3) that was specified for the original KV and CERL/KV methods. A cement suspension tank was chosen for the Generation 3 system because it is smaller and more portable than the washing machine. The triple-beam scale (Item 1, Tables 1 and 2) can be replaced by a more versatile, rugged (and expensive) digital scale. Also, if the CQM is to be used extensively at the same site, it probably would be cost effective to obtain or make some type of end-over-end mixer similar to those specified for the original KV and CERL/KV methods to mechanically mix the water test sample in the wide-mouth jar.

The approximate 1980 costs for the items listed in Tables 1 and 2 are \$2063 and \$4953, respectively. Excluding Items 1, 2, 3, and 9b in Table 2 (which are duplicates of items listed in Table 1), the total equipment cost for the CQM system is about \$6800.

Transportation and Field Operation Requirements

The CQM is easy to transport, simple to set up and take down, self-contained, and can be operated in a variety of environments. All its equipment can be carried in a car or pickup truck in a ready-to-use condition; this equipment does not have to be crated or packed with special shock isolation. For long-haul commercial transportation, all equipment can be crated in cardboard boxes small and light enough to ship as either excess baggage on most commercial airlines or by U. S. Parcel Post (Figure 4). (The crates and foam liners in which the calcium analyzer, chloride meter, and scales are packed when received from the manufacturer can be saved and used when commercially transporting the equipment.) The centrifuge and cement suspension tank are rugged mechanical items that do not require special packaging or shock isolation.

With the exception of the calcium meter and the cement suspension tank, all crated equipment only

needs to be uncrated when it arrives in the field and electrical lines connected and hooked to a 1.5 kW (or less) source of 115-volt AC electrical power. In addition, an EGTA reagent bottle must be installed in a side compartment of the calcium meter. After the reagent bottle's tubing is connected, a purge cycle must be run to remove air pockets in the tubing. It takes about 10 to 15 minutes to install and purge the EGTA system.

The cement suspension tank is normally shipped in two crates: the upper section is a 10-gal (37.6-L) polypropylene tank; the lower section is a base stand which has a water pump and stirrer motor (Figure 5). The tank is set up by (1) placing it on the base stand, (2) attaching the tubing connecting the water pump to the tank, and (3) connecting the Jiffy stirrer blade to the stirrer motor through the watertight bushing. It usually takes about 1 hour to uncrate (or crate) and set up (or take down) all the CQM equipment.

The CQM system, including preprepared reagents supplied by the equipment manufacturer, is completely self-contained, with the exception of 110 to 115 volts of AC current (less than 1.5 kW required), tap water, salt solution, and a 5 percent nitric acid solution. If not locally available, the current can be supplied by a small gasoline-driven alternator, and the tap water by an appropriately sized storage tank. The salt solution can be made from table salt. Thus, the only material or equipment that is not normally locally available (or shipped as part of the test system) is the nitric acid. Nitric acid is classified by the Department of Transportation as an oxidizer requiring an oxidizer-corrosive label and is subject to transportation restrictions. These restrictions include special packaging and forbid shipment on passenger-carrying aircraft and railcars.

All CQM equipment, including the calcium analyzer and chloride meter is rugged and reliable enough to operate in any interior or exterior environment in which concrete is normally placed.

Water Content Test

Reagents

The reagents needed to conduct the water content test are:

1. Sodium chloride (NaCl) solution (about 0.5 normal [N] in tap water)
2. Acid buffer solution.

The NaCl solution is made by dissolving 292 (± 3) g of dry NaCl in tap water and diluting to 10 L.* Each water test uses 250 ml of the 0.5 N NaCl solution; thus, 10 L is enough for 40 water tests.

The acid buffer solution is a preprepared reagent for the Corning 920M chloride meter. Replacements are available from Corning distributors. The reagent bottles, as shipped, contain 475 ml of solution. This is enough to load the meter's sample beaker 25 to 30 times. Each loading is good for 5 to 8 chloride readings. If each water content test requires two to three individual readings, the 475 ml should be enough for 60 to 100 water content tests.

Procedure

The CQM water content test consists of adding 250 ml of a 0.5 N NaCl solution to a 2-kg concrete sample, intermixing the two, and determining the chloride concentration of the intermixed supernatant salt solution using the Corning 920M chloride meter. If the concrete contains chlorides from other sources, both an actual and a blank sample (250 ml of distilled water added to a 2-kg concrete sample) must be used.

The steps for the CQM water content test are described below and outlined in Appendix A. (The outline in Appendix A should be posted near the equipment so operators can refer to it as needed):

Step 1. Obtain a 12- to 15-kg sample of fresh concrete, mix the sample to ensure homogeneity, and weigh out two subsamples of at least 2000 (± 200) g each.[†] Record the exact weight of each subsample to the nearest gram. Place one 2-kg subsample in a wide-mouth jar; then, using a volumetric flask, add 250 ml of distilled water. Secure the lid on the jar. This is the blank sample required for estimating chlorides in the concrete itself.

Step 2. Place the second 2-kg sample in another wide-mouth jar, add 250 ml of 0.5 N NaCl solution, and secure the lid.

*Dry NaCl crystals dissolve slowly, and mechanical agitation is recommended to ensure that the crystals completely dissolve.

[†]The reason for the wide range of sample weight is to prevent biasing the mortar/aggregate ratio of the sample by adjusting the sample size.

Step 3. Turn the two jars end-over-end, either by hand or in an end-over-end mixer. At least 75 complete revolutions are recommended if the jars are turned by hand; if turned by a 40 to 60 rpm mixer, at least 2 minutes are recommended.*

Step 4. After mixing, unfasten the lids and pour the water-cement slurry from the blank sample and the NaCl solution-cement slurry from the actual sample into the centrifuge tubes. Place the tubes in the centrifuge and run at 2000 to 3000 rpm for 3 to 4 minutes.

Step 5. Prepare the chloride meter for analysis by (a) placing the sample selector toggle switch on 100 μ l and switching the on/off switch to on, (b) placing 15 to 17 ml of acid buffer solution in the meter's 20 ml beaker, (c) placing the beaker on the stand, (d) lowering the silver electrodes, and (e) beginning the conditioning cycle by pressing the conditioning switch. (This step is required only at the start of each day or when the buffer solution sign indicates that it needs changing—about every 5 to 8 readings.)

Step 6. Determine the chloride strength of the blank sample by pipetting 100 μ l of the blank sample, using an Eppendorf, into the meter's 20 ml beaker. Press the titration switch. Record the result and repeat the test to ensure reproducibility. If the meter's blank light is on, no chlorides are present.

Step 7. Determine the chloride strength of the actual sample by pipetting 100 μ l of the actual sample, using an Eppendorf, into the meter's 20 ml beaker. Press the titration switch. Record the result and repeat the test to ensure reproducibility to ± 1 percent.

Step 8. Determine the chloride strength of the 0.5 N NaCl solution by pipetting 100 μ l of the .5 N NaCl solution, using an Eppendorf, into the 20 ml beaker. Press the titration switch. Record the results and repeat the test to ensure reproducibility to ± 1 percent. Water content is calculated as follows:

$$\text{Water Content (ml)} = 250 \left[\frac{\text{Std}}{\text{Sa} - \text{Bl} \left(\frac{\text{SaWt}}{\text{BlWt}} \right)} - 1 \right] \quad [\text{Eq 1}]$$

*Under no condition should the jars be turned so rapidly that the centrifugal force exceeds gravitational forces; this will prevent the salt solution and distilled water from completely mixing with the concrete samples.

Table 1
Water Tests

Item	Title	Quantity	Description	Source	Cost	
					Per Unit	Total
1	Scale	1	Triple-beam scale (2600 g capacity, 1 g sensitivity)	Laboratory equipment supplier	\$80	\$90
2	Hand scoop	1	Square-mouth scoop; bowl dimensions are 3 in. (76 mm) wide by 8 in. (203 mm); 1 mg; cast aluminum	Equipment supplies, concrete & soil testing	\$8	\$8
3	Sample tub	1	5 qt (4.7 L) polyethylene tub	Domestic food freezer goods supplier	\$1	\$1
4	Wide-mouth jar	2	½ gal (1.9 L) polyethylene wide-mouth jar with screw closure and lid	Laboratory equipment supplier	\$5	\$5
5	Centrifuge	1	Variable speed, 4-place centrifuge for 15 ml tubes	Same as Item #1	\$230	\$230
6	Centrifuge tube	2	Disposable, 15 ml, polystyrene centrifuge tubes (2 per test purchased in cases of 1000)	Same as Item #1	\$12/pair	\$60/1000
7	Eppendorf pipet (20µl)	1	Tip ejector fixed volume, pipet (20 µl capacity)	Same as Item #1	\$85	\$85
8	Disposable pipet	3	Disposable tips for 20 µl Eppendorf pipets; purchased in case lots of 1000	Same as Item #1	\$36/1000	\$36/1000
9	Chloride meter	1	Corning Model 920M chloride meter	Same as Item #1	\$1500	\$1500
10	Volumetric flask	2	Polypropylene, 250 ml cap	Same as Item #1	\$9	\$9
11	Carboy	1	Linear polyethylene, rectangular, with spigot, screw closure, 2 gal (7.6 L) capacity	Same as Item #1	\$30	\$30
12	Beakers	1	Polypropylene, Griffin low-form graduated, 250 ml capacity (sold in case lots of 6)	Same as Item #1	\$2	\$9
					TOTAL	\$2063

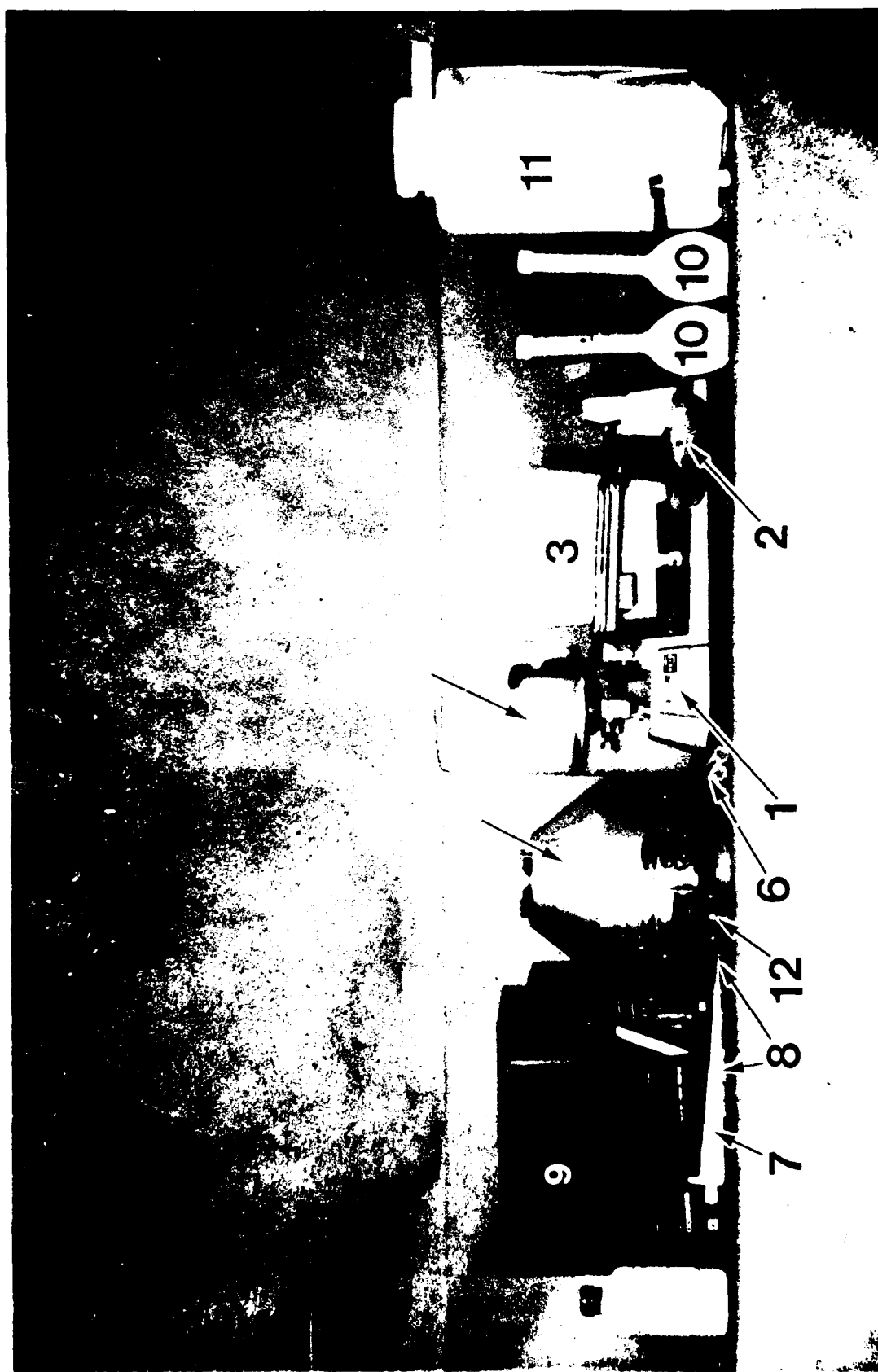


Figure 1. Water content test equipment.

Table 2
Cement Tests

Item	Title	Quantity	Description	Source	Cost	
					Per Unit	Total
1	Scale	1	Triple-beam scale (2600 g capacity, 1 g sensitivity)	Laboratory Equipment	\$90	\$90
2	Hand scoop	1	Square-mouth scoop bowl; 3 in. (76 mm) wide by 8 in. (203 mm) long; cast aluminum	Equipment supplier for concrete and soil testing	\$8	\$8
3	Sample tub	1	5 qt (4.7 L) polyethylene tub	Domestic food freezer goods supplier	\$1	\$1
4	Specimen tub	1	2 qt (1.9L) polyethylene tub	Same as Item #3	\$1	\$1
5	Cement suspension tank	1	Polypropylene, 10 gal (37 L) "Nalgene" tank including recirculating pump and hose, 1/20 hp DC motor with an AC/DC controller for use on 115/120-Volt AC lines, watertight bushing, and Jiffy mixing blade coupled through universal joint to 1/12 hp stirrer motor. Including cutout ring to hold 12 in. (304 mm) diameter sieves.	Tank, hoses, motors, etc. purchased from laboratory equipment supplier (locally fabricated)	\$900	\$900
6	Sieve nest		Standard stainless steel, 12 in. dia (304 mm)	Same as Item #2	\$85	\$85
	No. 4		4760 micron openings		\$80	\$80
	No. 50		297 micron openings		\$80	\$80
	No. 100		149 micron openings		\$80	\$80
7	Magnetic stirrer & stirring rod		Variable speed magnetic stirrer & non-stick coated stirring rod	Same as Item #1	\$80	\$80
8	Syringe type pipet	1	"Varipet," syringe-type variable volume transfer pipet, 30 ml capacity	Same as Item #1	\$60	\$60
9	Eppendorf pipet		Tip ejector, fixed volume, pipet	Same as Item #1	\$60	\$60
	20 µl capacity	1			\$85	\$85
	100 µl capacity	1			\$85	\$85
10	Disposable pipet tips	-	Disposable tips for 20 and 100 µl Eppendorf pipets, purchased in case lots of 1000	Same as Item #1	\$36/1000	\$36,1000
11	Flask			Same as Item #1		
	Erlenmeyer	1	Polycarbonate, 500 ml capacity		\$5	\$5
	volumetric	1	Polypropylene, 250 ml capacity		\$9	\$9
12	Calcium analyzer	1	Corning Model 940 calcium analyzer	Same as Item #1	\$3300	\$3300
13	Carboy	1	Linear polyethylene, rectangular, with spigot screw closure, 2 gal (7.6L) capacity	Same as Item #1	\$30	\$30
					TOTAL	\$4935

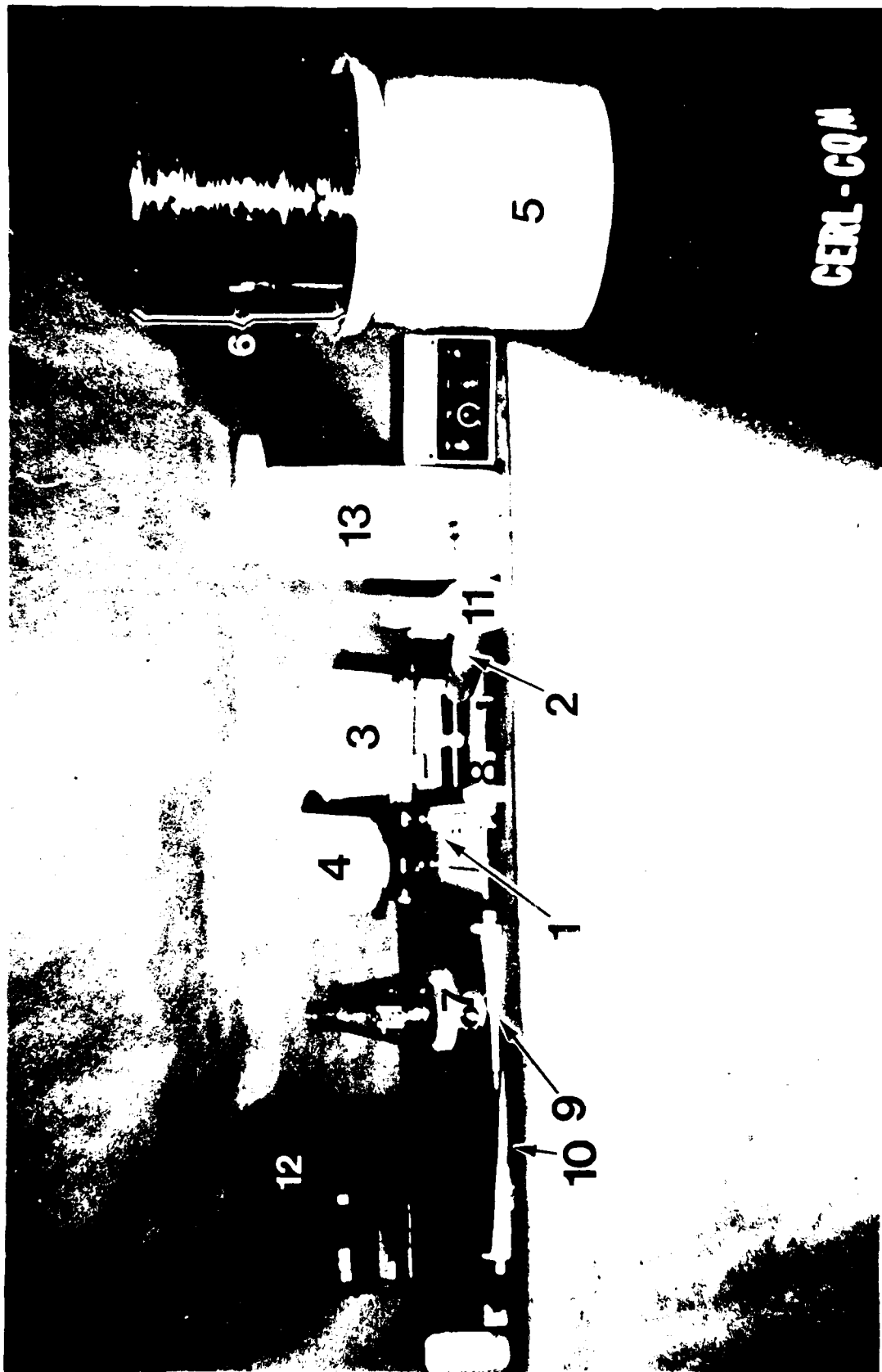


Figure 2. Cement content test equipment



Figure 3. Commercially produced washing machine and sieve nest used with the CERL/KV system.

where

Std = relative chloride strength of the 0.5 N NaCl solution
(Step 8) (milliequivalents per liter)

Sa = relative chloride strength of the sample solution
(Step 7) (milliequivalents per liter)

Bl = relative chloride strength of the blank solution
(Step 6) (milliequivalents per liter)

SaWt = weight of sample (nearest gram)

BlWt = weight of blank (nearest gram).

If the concrete being tested does not contain chlorides (i.e., if the chloride strength of the blank sample equals zero), the blank sample tests can be discontinued.

Cement Content Test

Reagents

The reagents needed to conduct the cement content test are:

1. A 5 percent nitric acid solution made by adding 5 ml of nitric acid (specific gravity = 1.42) to 95 ml of tap water.
2. Tap water
3. EGTA solution

4. Calcein indicator solution

5. Calcium standard solution

6. Potassium hydroxide (1.0 N potassium hydroxide).

Reagents 3 through 6 are preprepared and are available from Corning (they are produced specifically for Corning's 940 calcium analyzer). Replacements are available from Corning distributors. Reagent 4, the calcein indicator, is shipped in a powder form; each prepackaged sample is dissolved in 10 ml of the calcium standard solution. The reconstituted calcein solution has a shelf life of 4 to 6 weeks, and should be replaced accordingly.

Calibration Requirements

Before conducting the cement content test, the procedure must be calibrated for the calcium in the tap water and the concrete. This is done by running the standard cement content test. To determine the tap water calibration constant, Step 2 of the cement test procedure is excluded, and the aggregate proportions present in a 2 kg concrete sample are obtained as the "2 kg sample". In Step 10, analyze a 100 μ l sample.

For the concrete calibration test, a 10-kg sample of concrete is hand mixed using the materials and mix proportions of the concrete to be tested. (The cement should be hand mixed until it is homogeneous.) A 2-kg sample of the concrete is taken and tested (Steps 1 through 10), and the results recorded.*

The cement calibration curve is a linear plot of cement content (g) vs the calcium analyzer reading (mg %), with zero cement being the water calibration result (mg %) divided by 5. The weight of the cement in the 2-kg concrete calibration sample and its calcium analyzer reading are the other set of coordinates. Figure 6 shows a typical calibration curve.

The concrete calibration test must be repeated each time the cement or aggregate source or type used to produce the concrete is changed (or on a weekly

*The sieve arrangement used in the calibration procedure should be consistent with those used in the test procedure. That is, if only the No. 4 and No. 50 sieves are used to calibrate the cement test, the same arrangement should be used during testing. If the No. 4, No. 50, and No. 100 sieves are used, they should be used for both calibrating and testing.

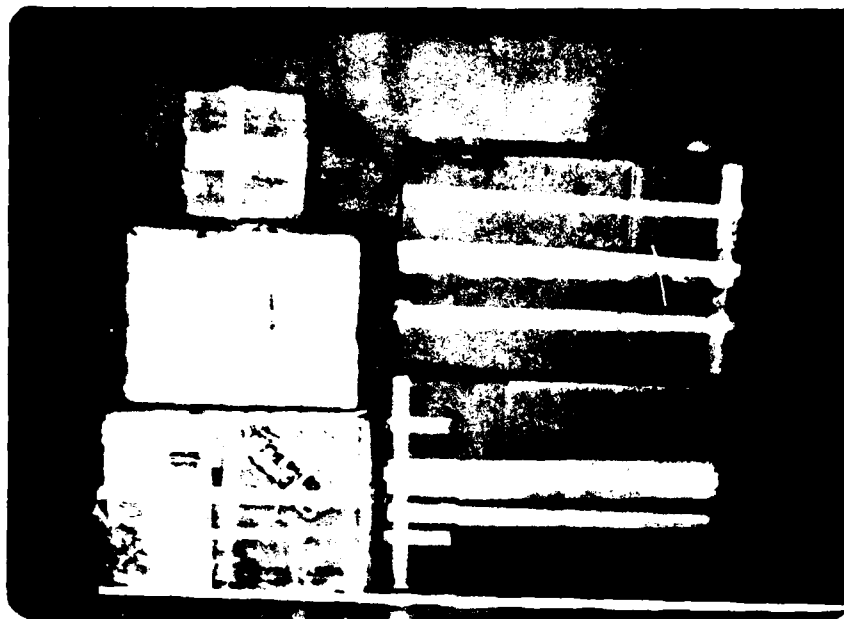


Figure 4. CQM equipment crated for commercial shipment.

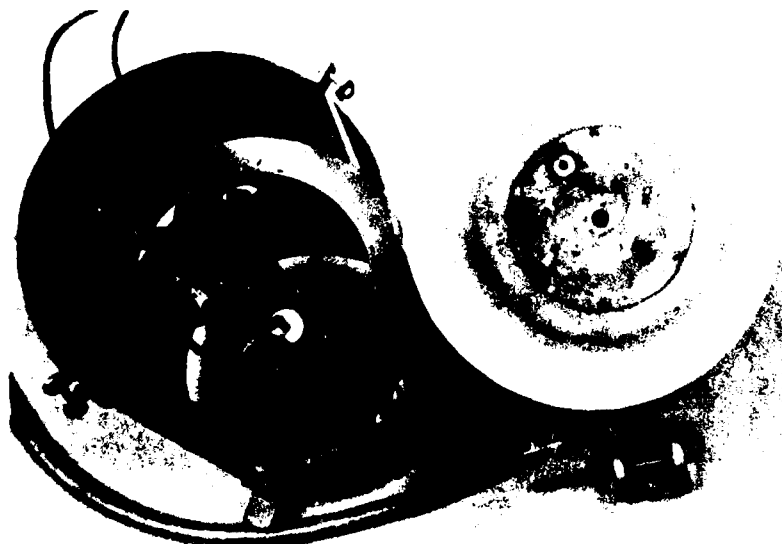


Figure 5. Cement suspension tank -- upper and lower sections separated.

basis if the aggregate and cement sources and type do not change). Both the concrete and tap water calibration tests must be repeated each time the water source changes.

Procedure

The CQM cement content test is based on the following assumptions:

1. Cement of a given type from a given source is uniform in calcium content; and the aggregates either do not contain calcium or are uniform in calcium content for that proportion of the aggregates that pass the finest sieve over the cement suspension tank.

2. When agitated, cement can be uniformly dispersed and suspended in water so that a representative sample can be obtained.

3. Stirring without external heat will produce a quantitative solution of cement in nitric acid.

4. The calcium content of the cement solution can be determined by titration with the Corning 940 calcium analyzer.

The steps for the cement content test are described below and outlined in Appendix B. (The outline in Appendix B should be posted near the test equipment so operators can refer to it as needed):

Step 1. Fill the cement suspension tank with tap water to the 10-gal (37.6-L) mark on the side of the tank. Place the nested sieves on the tank and turn on the tank's agitator.*

Step 2. Obtain the 12- to 15-kg concrete sample, mix the sample to ensure homogeneity (or remix, if used in conjunction with the water test), and weigh

*If calcareous fines are present, it is recommended that an additional No. 100 sieve be nested below the No. 50. The combination of sieves used for calibration and cement content testing must be consistent.

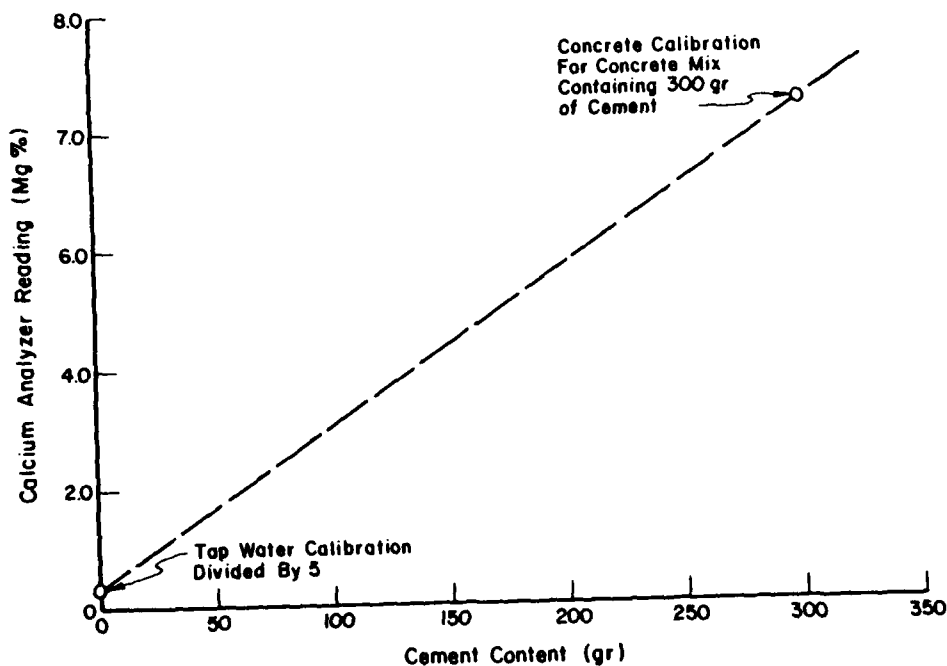


Figure 6. Cement content test -- typical calibration curve.

out 2000 ($\pm$ 200) g of fresh concrete. Record the weight to the nearest gram.

Step 3. Transfer the 2000-g sample to the sieves over the tank. Turn on the tank's recirculating pump and wash the residue from the 2000-g sample container into the tank using the water jet from the recirculating pump hose.

Step 4. Wash the plus No. 4 aggregate carefully, using the water jet from the recirculating pump hose. After all the cement has been washed from the aggregate retained on the No. 4 sieve (this takes about 1 to 1.5 minutes), remove the No. 4 sieve.

Step 5. Wash the aggregate retained on the No. 50 sieve until all cement has been washed from the aggregate (this takes about 1 to 1.5 minutes). Remove the No. 50 sieve.*

Step 6. Obtain a representative sample of the cement suspension in the tank using the 30 ml syringe pipet. Place the suspended material in a 500-ml Erlenmeyer flask. Refill the syringe pipet with 5 percent nitric acid, and add the acid solution to the content of the Erlenmeyer flask. While discharging the acid solution from the syringe pipet, shake it occasionally to ensure that all cement that settled out when the cement samples were taken has dissolved and is flushed out with the acid solution. Use a volumetric flask to add 250 ml of tap water to the Erlenmeyer flask.

Step 7. Put a magnetic stirring bar in the Erlenmeyer flask and place it on a magnetic stirrer. Turn on the stirring motor and check to see that stirring has begun.

Step 8. Prepare the calcium analyzer by switching the power on, and filling the cuvette to mark with 1.0 N potassium hydroxide and 100 μ l (Eppendorf) of reconstituted calcein reagent. Put the cuvette in the analyzer, add 100 μ l (Eppendorf) of calcium standard solution, and push the titration button to condition the cuvette for analysis. (This step is required only after the cuvette is filled with new potassium hydroxide solution. A single cuvette filling is sufficient for 15 to 20 readings.)

*When both the No. 50 and No. 100 sieves are used, the aggregate retained on the No. 100 sieve should be washed an additional 1 to 1.5 minutes after the No. 50 sieve has been removed. Remove the No. 100 sieve when complete.

Step 9. Begin the analysis by placing the *mEq/mg*[†] toggle switch on *mg*[†] and adding 100 μ l (Eppendorf) of the calcium standard to the cuvette. Press the titration button. Record the result and repeat the test by adding another 100 μ l sample. Continue repeating until consecutive results are less than 1.5 percent apart. Push the calibration button and run an additional 100 μ l sample of the calcium standard to ensure that the calcium standard readout value is 10 ($\pm$ 0.1) mg percent.

Step 10. Determine the strength of the cement solution in the Erlenmeyer flask by analyzing a 20 μ l sample in the calcium analyzer. Repeat this test until all values are less than 1.5 percent apart. Determine the cement content by referring to the calibration graph.

3 VALIDATION TESTS

Validation tests were conducted under both laboratory and field conditions to determine how simple, fast, robust, and accurate the CQM system was relative to the Generation 2 CERL/KV method. The comparison of the two systems included accuracies for determining water and cement content and estimates of strength potential. The strength potential estimates included, for the first time, both compressive and flexural strengths.

Laboratory Test

During the laboratory validation series, CQM and CERL/KV tests were run on air- and nonair-entrained concrete samples. The concrete was Type 1 cement, siliceous river sand, and a 1 in. (25.4 mm) maximum calcareous coarse aggregate. On the air-entrained mixes, vinsol resin was used to entrap air. Five different concrete mixes were batched with water/cement ratios varying from 0.4 to 0.8; cement content ranged from 21.5 to 10.9 percent for the nonair-entrained concrete and 19.6 to 10.0 percent for the air-entrained concrete. Two-cubic-foot (0.05 m³) mixes were batched and mixed in a rotary drum with a capacity of 3-1/2 cu ft (1 m³). Besides samples for CQM and CERL/KV testing, two 6 x 12 in. (152 x 204 mm) cylinders and two 6 x 6 x 21 in. (152 x 152 x 533 mm) beams were cast from each concrete batch. Slump tests were also taken on each batch, and the air contents determined for the air-entrained batches. The cylinders and beams were moist-cured for 28 days, then broken in compres-

sion and flexure, respectively. The slump, air content, and beam and cylinder tests were all conducted according to ASTM standards.

Four CQM and CERL/KV tests were conducted on each concrete batch. The test procedure consisted of weighing out four 2-kg samples for water content cement content analysis. Two hundred fifty milliliters of 0.5 N NaCl solution were added to each of the four water content samples, and the concrete and solution were intermixed in an end-over-end mixer. After settling, the chloride strength of the resulting supernatant of each sample was determined by the CQM and CERL/KV methods, respectively, and related to the water content of the mix. Similarly, each of the four cement content test samples were washed over the No. 4 and No. 50 sieves with the recirculating water from the cement suspension tank. After the aggregate and cement separated, a representative CQM sample was obtained with a 30 ml syringe pipet; a representative CERL/KV sample was taken with the 125 ml linked pipet. The CQM and CERL/KV tests were then completed.

Table 3 summarizes the results of the CQM and CERL/KV water and cement content estimates. The table is based on percent recovery (CQM or CERL/KV value divided by the actual mix proportions times 100.)* The overall mean and standard deviation values

indicate that the accuracy of the CQM system is equal to or slightly better than the CERL/KV for both water and cement content. For all tests (air- and nonair-entrained), the mean and standard deviations for the CQM tests are 97.8 and 5.2 percent and 106.1 and 8.5 percent, respectively, for cement and water content recovery. Similarly, the CERL/KV results were 100.3 and 5.7 percent and 101.4 and 8.6 percent, respectively, for cement and water content recovery. The water recovery values for the air-entrained concrete mixes were the only results that differed significantly from similar results on previously reported tests.⁶ The mean and standard deviation for these CQM tests were 112.9 and 7.56 percent; the CERL/KV results were 100.65 and 10.9 percent. It is not known precisely what caused the high recovery values for the CQM water test, but they probably related to the presence of suspended solids and/or entrapped air not completely settled out or removed from the 20 μ l sample used to analyze the chloride strength of the water sample. Suspended solids and/or entrapped air in the 20 μ l sample would result in lower chloride and higher water content results. Because most air-entraining agents, besides entrapping air, are also dispersives, the suspension of solids was significantly greater in the air-entrained tests than in the nonair-entrained tests. It is assumed that this problem would not be as significant if the supernatant of the water test sample was centrifuged. The laboratory tests were not centrifuged

*Free water (total water minus the absorption capacity of the aggregates) was used as the actual water mix proportion in computing recovery for the CQM and CERL/KV water content tests.

⁶P. A. Howdyshell, *Revised Operations Guide for a Chemical Technique to Determine Water and Cement Content of Fresh Concrete*, TR M-212/ADA039120 (CFRL, April 1977).

Table 3
Laboratory Validation Series (Recovery)

	CQM		CERL/KV	
	Cement	Water	Cement	Water
Nonair-Entrained				
$\bar{X}$	97.3	101.9	100.7	101.8
S_x	5.4	5.9	5.7	6.8
n	39	39	39	38
Air-Entrained				
$\bar{X}$	98.5	112.9	99.7	100.6
S_x	4.8	7.6	5.8	10.9
n	24	24	24	24
All Tests				
$\bar{X}$	97.8	106.1	100.3	101.4
S_x	5.2	8.5	5.7	8.6
n	63	63	63	62

because both the CQM and CERL/KV tests were conducted on the same sample; thus, it was not possible to obtain a supernatant sample large enough to centrifuge.

Figures 7, 8, and 9 depict the relationships among flexural strength and the CQM, CERL/KV, and actual water/cement ratios for the nonair-entrained mixes. Similarly, Figures 10, 11, and 12 depict the relationships for the air-entrained data. The linear regression analysis conducted on each data set indicates excellent correlation. Correlation coefficients were greater than 0.9 and 0.8 for the nonair-entrained and air-entrained mixes, respectively.

The standard error for flexural strength prediction was computed by:

$$\sigma^2 = \frac{\sum \Delta^2}{(n - 3)} \quad [\text{Eq 2}]$$

where

σ = standard error

Δ = the difference between the actual and estimated flexural strength in the "i" case

n = sample number.

The standard errors for the flexural strength predictions of the nonair-entrained mixes for the CQM water/cement ratios were 31.1 and 36.7 psi (214.6 and 253.2 kPa), respectively, for the regression curve developed from the CQM and actual water/cement ratios. The standard errors for the flexural strength predictions of the nonair-entrained mixes for the CERL/KV water/cement ratios were 34.3 and 33.7 psi (236.7 and 232.5 kPa), respectively, for the CERL/KV and actual water/cement ratio regression curves. Similarly, the standard errors for the air-entrained mixes based on CQM water/cement ratios were 71.0 and 123.6; for the CERL/KV water/cement ratios, they were 65.7 and 69.1. These results indicate that both the CQM and CERL/KV systems can estimate flexural strength potential if the actual water/cement ratio strength relationships are known for the material constituents being used. The relatively large error for the CQM air-entrained mixes relates directly to the previously described high water test recovery for the CQM air-entrained mixes. Additionally, the accuracies of these strength predictions were somewhat improved

when predictions were based on regression analyses based on CQM and CERL/KV water/cement ratios.

Compressive strengths were predicted from the CQM and CERL/KV water/cement ratios and the air content data. These predictions were compared to the actual compressive strengths and standard errors computed for both the CQM and CERL/KV predictions. The compressive strength estimates were based on a regression equation developed previously in this research.

$$f_c = Af_{ca} \quad [\text{Eq 3}]$$

where

A = the mix factor constant (constant for each set of materials)

f_c = the estimated strength

$$f_{ca} = 9551 - 7847(W/C) - 733.7(a) + 760.1(a)(W/C)$$

W/C = CERL/KV or CQM water cement ratio

a = air content (percent).

The standard error for the strength prediction was computed by Eq 2. The standard errors for the compressive strength predictions for the CQM water/cement ratios were 674 and 728 psi (4650.6 and 5023.2 kPa), respectively, for the mix factor constants developed from the CQM and actual water/cement ratios. The standard errors for the CERL/KV compressive strength predictions were 648 and 648 psi (4471.2 kPa), respectively, for the mix factor constants developed from the CERL/KV and actual water/cement ratios.

An overall evaluation of the laboratory validation series indicates that, with the exception of the high CQM water contents for the air-entrained mixes, the accuracies of the CQM and CERL/KV methods are about equal for determining water and cement content and for estimating flexural and compressive strength. The accuracy of the CQM and CERL/KV flexural and compressive strength predictions (based on actual water/cement ratio to strength relationships) makes it possible to estimate strength from CQM water/cement ratios without a CQM water/cement ratio to strength relationship. Additional laboratory tests have indicated that if the CQM water tests are centrifuged they concur much more closely with actual water content for air-entrained mixes.

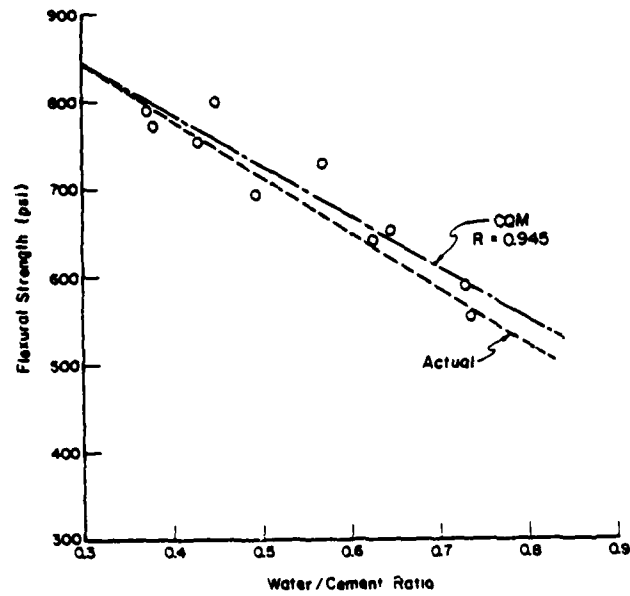


Figure 7. Flexural strength vs CQM water/cement ratios (nonair-entrained). Metric conversion: 1 psi = 6.895 kPa.

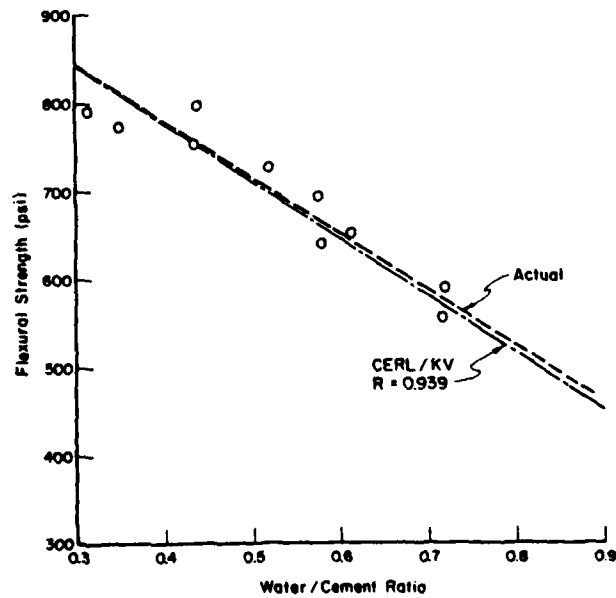


Figure 8. Flexural strength vs CERL/KV water/cement ratios (nonair-entrained). Metric conversion: 1 psi = 6.895 kPa.

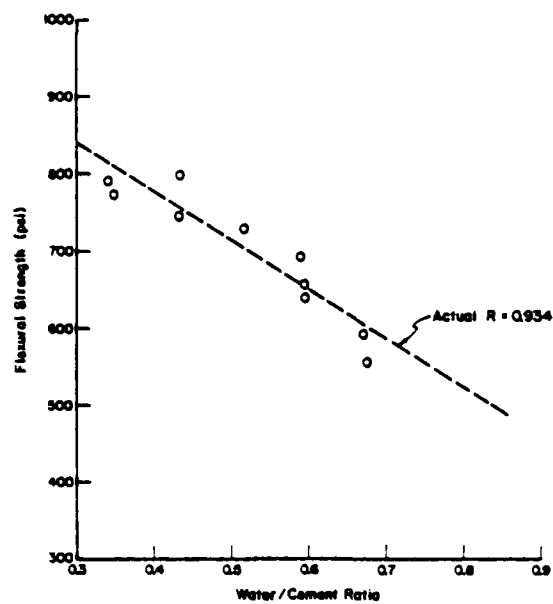


Figure 9. Flexural strength vs actual water/cement ratios (nonair-entrained). Metric conversion: 1 psi = 6.895 kPa.

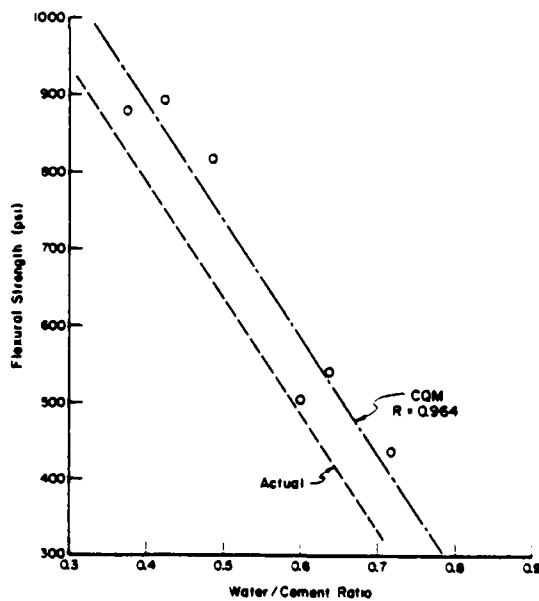


Figure 10. Flexural strength vs CQM water/cement ratios (air-entrained). Metric conversion: 1 psi = 6.895 kPa.

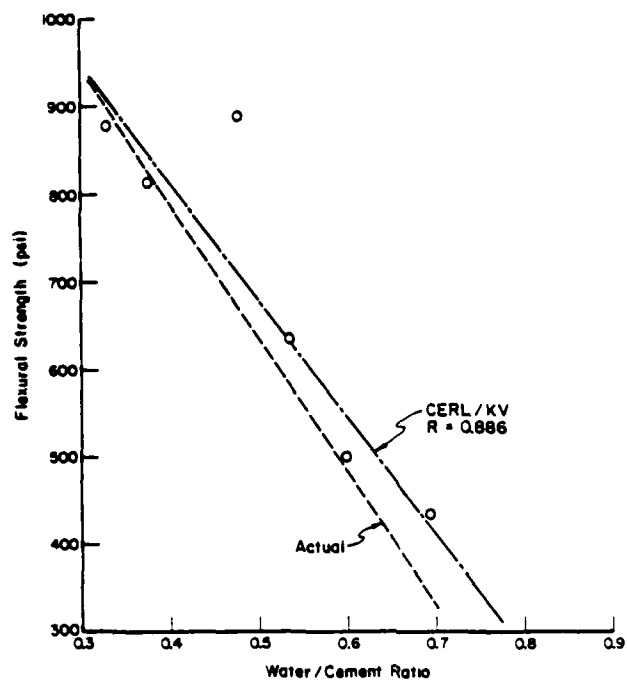


Figure 11. Flexural strength vs CERL/KV water/cement ratios (air-entrained). Metric conversion: 1 psi = 6.895 kPa.

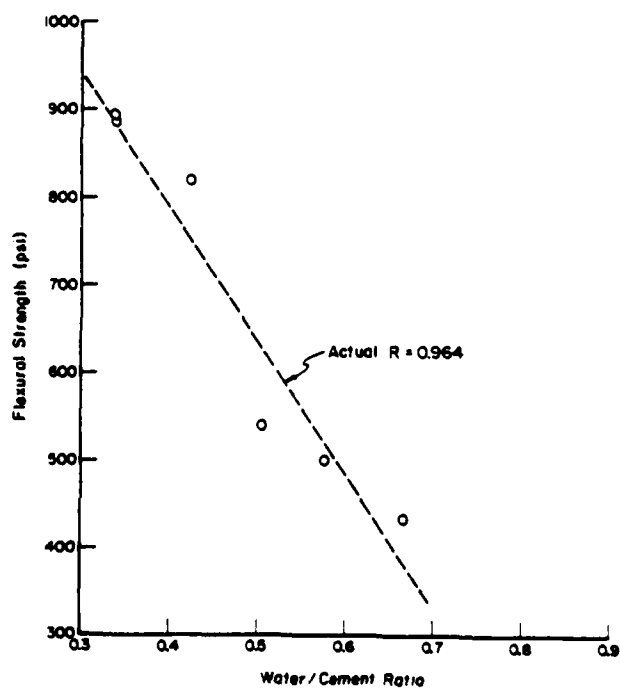


Figure 12. Flexural strength vs actual water/cement ratios (air-entrained). Metric conversion: 1 psi = 6.895 kPa.

Field Tests

Three different field tests have been conducted on the CQM system. The first two field tests evaluated the robustness and transportability of the system. One test proved that it was possible to use a car or truck to carry all equipment in a ready-to-use configuration. For this test, CQM equipment was transported in a sedan from Champaign to Springfield, IL (85 miles [136 km]) and demonstrated to the Materials Laboratory staff at the Illinois Department of Transportation. It took less than ½ hour to set up the demonstration; all equipment worked reliably at the demonstration site.

The second field test consisted of crating the equipment in cardboard boxes and shipping it as excess baggage on a commercial airline from Champaign, IL to Portland, OR. The equipment was then used for a field test and demonstration for the Federal Highway Administration Region 10 staff at Vancouver, WA. It took about 1 hour to uncrate and set up the equipment. Four concrete batches were tested using both the CQM and CERL/KV methods. Correlations between the CQM and CERL/KV test results were excellent. Also, the CQM water test was significantly more rapid than the CERL/KV water test, because of the centrifuge separation of the cement-salt solution slurry.

The third field test was conducted with the help of the Oregon Department of Transportation (ODOT). The objective of this test was to use non-CERL personnel to determine (1) the robustness, (2) required operator skills, and (3) the accuracy of the CQM system.

In the ODOT test, two undergraduate engineering students were hired to obtain concrete samples from ODOT construction sites throughout the greater Portland area. They were asked to cast cylinders and conduct the CQM and CERL/KV tests on the samples at a central field laboratory (a small trailer).

CERL delivered the CQM equipment and spent 3 days training the two students and other personnel from ODOT. Of the 3 days scheduled for training, less than ½ day was really available; the rest of the time was spent investigating an EDTA titration problem with the CERL/KV system that had never occurred before. The restricted training time did not permit any assessment of operator skills or equipment performance.

Mean and standard deviation values for CQM water and cement content recovery were 121.4 and 24.5, and 97.4 and 20.0 percent, respectively. These standard deviations are considerably greater than those obtained in the laboratory validation series. (Some increase in error would be expected when laboratory tests are compared to field data, because actual field mix proportions are only *assumed* to be the mix design proportions.) The degree of error in these tests was significantly greater than that normally associated with field tests.⁷ Thus, the cause of the error must have been in the technique or the equipment. The two student operators said they found it difficult to calibrate the calcium meter for the cement test (Step 9, cement content test procedure). They had the same trouble during the entire test series; near the end of the series, the calcium meter failed completely. After the calcium meter was returned to CERL for repair, it was discovered that the drive shaft that operates a syringe-type plunger in the analyzer had a loose coupling. This allowed the shaft to turn without moving the plunger. After the set screws on the coupling were tightened, the problem was corrected. A check of the calibration procedure (Step 9, cement content test procedure) indicated that the meter was operating satisfactorily. Thus, it seems that a large proportion of the cement content errors were related to field calibration problems probably caused by the slipping shaft and plunger in the calcium analyzer.

The water test results also had significantly greater errors than the laboratory results. There was also a high mean recovery value (121.4 percent). Before returning the chloride meter to CERL, the field operators performed a water analysis check on the chloride meter. This check consisted of running a standard CQM water content test on a sample containing only a known amount of water and comparing the results with the actual amount of water in the sample. Their analysis check provided a recovery value similar to that obtained from their concrete tests. But an identical test done at CERL by CERL personnel indicated near perfect agreement between the actual and computed water content. This indicates some problem or inconsistency in the field operators' procedure, but the specific nature of this procedural problem was not determined.

⁷P. A. Howdyshell, *Revised Operations Guide for a Chemical Technique to Determine Water and Cement Content of Fresh Concrete*, TR M-212/ADA039120 (CERL, April 1977).

With the exception of the loose coupling problem on the calcium analyzer, the overall field test results indicate that the CQM system can be transported reliably. The inconsistencies in the ODOT test indicate that more field tests are needed. The training approach should also be reviewed and refined, as necessary. In general, it is recommended that field evaluations should continue, but with close initial supervision and support from CERL to develop an effective training approach and to assess the causes of potential problems.

4 RECOMMENDED PROCEDURES FOR ANALYSIS OF CQM DATA

Comparison With Mix Design Values

The water and cement contents determined by the CQM method should be compared with the batch proportion values. If the CQM and batch proportion values vary by less than 10 percent, it is assumed that the CQM system is operating properly, that the concrete batch is homogeneous, and that the batched proportion values are correct. If the results vary by more than 10 percent, a second complete CQM test should be run. The 2-kg test samples for the rerun should be taken from the original 12 to 15 kg sample collected for the initial runs. Extreme care should be exercised on the reruns to ensure no procedural errors are made. If the second test agrees closely with the batch proportion values, it should be assumed that it is correct and that the initial test was in error. If the second test is significantly different from both the batch proportion and first test results, or if the second test agrees closely with the first, one of three things has occurred:

1. The concrete sample is not representative of the bulk (indicating poor mixer efficiency and nonhomogeneity).
2. The batch is not the same as indicated by the batch proportions.
3. The CQM system is incorrectly calibrated.

Figure 13 shows a series of analytical steps for determining which of these three problems has occurred. It is recommended that an inquiry be made

as to possible changes or problems that may have occurred at the batch plant at the same time as the test outlined in Figure 13 is run.

Determining Concrete Strength

Figures 7 through 12 and the results obtained from Eq 3 indicate the validity of the relationships between CQM water/cement ratios, air content, and 28-day flexural and compressive strengths. But since several other factors can contribute to a greater or lesser extent to concrete strength and strength-gain characteristics, it is recommended that the water/cement ratio to strength relationships be developed for each concrete material system used. (Although, as indicated by the laboratory results discussed in Chapter 3, the actual water/cement ratio to strength relationships can be used to accurately predict strength from CQM water/cement ratios.) With the exception of minor concrete, all Corps of Engineers specifications (both Civil Works and Military Construction) require either Government- or contractor-developed water/cement ratio to strength relationships for concrete placed on Corps of Engineers' projects. Thus nearly all concrete placed on Corps of Engineers' projects has water/cement ratio to strength data that can be used directly with the CQM water/cement ratios to estimate strength potential. If such information does not exist, Eq 3 can be used to estimate compressive strength by assuming the mix factor constant A is equal to 1 (A equal to 1 normally produces a conservative strength estimate.) Since flexural strength is more sensitive to parameters other than the water/cement ratio influencing strength potential, it is recommended that flexural strength estimates not be made without a specific water/cement ratio to strength relationship for the materials being used.

5 CONCLUSIONS

This report describes the equipment and reagents needed for the CQM system and outlines the system's test procedures (Chapter 2).

1. Laboratory and field tests showed that the CQM system is significantly easier to operate in the field than the Generation 2 KV system (Chapter 3) because the CQM uses less glassware and fewer reagents. With the exception of a nitric acid and salt solution, all reagents can be purchased prepackaged from the

PROCEDURE FOR CHECKING CAUSE OF ERROR IN RESULTS

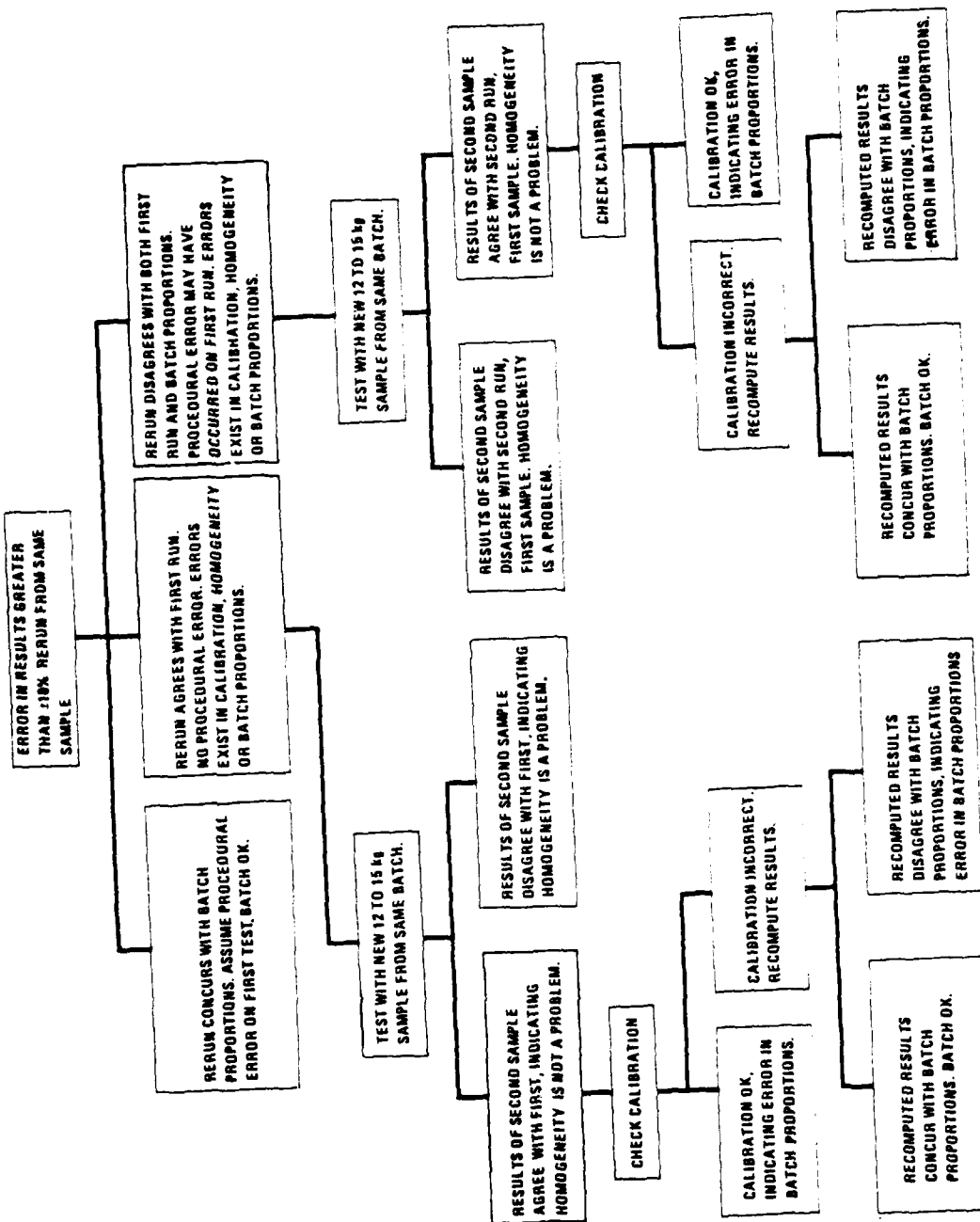


Figure 13. Procedure for checking cause of error in results.

equipment manufacturer. The CQM is also easier to transport than the Generation 2 KV system (Chapter 3).

2. The only significant limitation to the CQM system relates to the calcium signature of the cement content that passes the No. 50 and No. 100 sieves. Thus, nonuniformity of calcium concentrations in cement, aggregate, or water passing the No. 50 or No. 100 sieve can cause significant error (Chapter 4).

3. Validation test results indicate that the CQM system is as accurate as the Generation 2 KV system (Chapters 3 and 4).

4. The CQM system can be used to estimate both compressive and flexural strengths (Chapters 3 and 4).

Since this report may not answer all the questions of potential users, CERL will provide technical assistance to any Corps of Engineers facility interested in setting up and using the CQM system. This assistance includes field demonstrations and help in procuring equipment, training operators, analyzing results, and troubleshooting. For more information, contact:

Department of the Army
Construction Engineering Research Laboratory
ATTN: Debbie Lawrence or P. A. Howdyshell
P. O. Box 4005
Champaign, IL 61820
Telephone: 958-7224 (FTS) or 217-352-6511,
Extension 224 (Commercial).

APPENDIX A: COM WATER CONTENT TEST (BLANK SAMPLE)

1. Place a 2000g (± 200 g) concrete sample in wide-mouth jar. Record the weight of the sample to the nearest gram.

2. Use a volumetric flask; add 250 ml of distilled water to the concrete sample in the jar. Seal the lid.

3. Agitate the concrete and distilled water solution mixture by hand or in an end-over-end mixer. Hand mix for at least 75 revolutions; mix for 2 minutes if using an end-over-end mixer.

4. After agitation, unfasten the jar lid and pour the water-cement slurry from the blank sample into the centrifuge tubes (two tubes). Place the tubes in the centrifuge and centrifuge at 2000 to 3000 rpm for 3 to 4 minutes.

5. Prepare the chloride meter for analysis (this step is required only when buffer solution is changed).

- Turn the power switch on and selector switch to 100 μ l sample size.
- Add 15 to 17 ml of acid buffer to the meter's beaker.
- Begin the conditioning cycle by pressing the conditioning switch.

6. Determine the chloride strength of blank sample by pipetting 100 μ l sample from the centrifuge tubes into meter's beaker. Press the titration switch. Record the result and repeat the test to ensure reproducibility. If the blank sample light is on, no chlorides are present and the sample water content equation is correct. If chlorides are present, the equation is modified as follows:

[Eq A1]

$$\text{water content (ml)} = 250 \left[\frac{\text{Std}}{\text{Sa} - \text{Bl} \left(\frac{\text{SaWt}}{\text{BlWt}} \right)} - 1 \right]$$

where

Bl = chloride strength of blank sample solution obtained in Step 6 (milliequivalents per liter)

SaWt = weight of sample

BlWt = weight of blank.

COM WATER CONTENT TEST (ACTUAL SAMPLE)

1. Place a 2000 g (± 200 g) concrete sample in the wide mouth jar. Record the weight of the sample to the nearest gram.

2. Use a volumetric flask; add 250 ml of 0.5 N NaCl solution to the concrete sample in the jar. Seal the lid.

3. Agitate the concrete and the NaCl solution mixture by hand or in an end-over-end mixer. Hand mix for at least 75 revolutions; mix for 2 minutes if using an end-over-end mixer.

4. After agitation, unfasten the lid and pour the NaCl solution-cement slurry from the sample into the centrifuge tubes (two tubes). Place the tubes in the centrifuge and centrifuge at 2000 to 3000 rpm for 3 to 4 minutes.

5. Prepare the chloride meter for analysis:

- Turn the power switch on and selector switch to 100 μ l sample size.
- Add 15 to 17 ml of acid buffer to the meter's beaker.
- Begin the conditioning cycle by pressing the conditioning switch. (This step is required only when the buffer solution is changed.)

6. Determine the chloride strength of the sample by pipetting a 100 μ l sample from the centrifuge tubes into the meter's beaker. Press the titration switch. Record the result and repeat the test to ensure reproducibility.

7. Determine the chloride strength of the 0.5 N NaCl solution as described in Step 6.

$$8. \text{Water content (ml)} = 250 ([\text{Std}/\text{Sa}] - 1) [\text{Eq A2}]$$

Std = chloride strength of the 0.5 N NaCl solution obtained in Step 7 (milliequivalents per liter)

Sa = chloride strength of intermixed sample solution obtained in Step 6 (milliequivalents per liter).

APPENDIX B: COM CEMENT TEST

1. Fill the cement suspension tank to the 10 gal (37.6 L) mark. Place the nested sieves on the tank and turn on the tank's agitator.

2. Place a 2000 g (± 200 g) sample on the sieves over the tank. Record the sample weight to the nearest gram. Turn on the recirculating hose and wash the plus No. 4 aggregate carefully using the water from the recirculating hose. After all the cement has been washed from the aggregate retained on the No. 4 sieve, remove the sieve. Repeat the washing and sieve removal process for the No. 50 and No. 100 sieves.

3. Take a representative 30 ml sample of cement suspension in the tank by using 30 ml syringe pipet. Place the 30 ml sample in the Erlenmeyer flask. Refill the syringe pipet with 5.0 percent nitric acid, and add the acid solution to the flask. While discharging the acid solution from the syringe, shake it occasionally to ensure that all the cement residue in the syringe has been dissolved and flushed out of the syringe. Use a volumetric flask to add 250 ml of tap water to the flask.

4. Mix the contents of the flask by magnetically stirring.

5. Prepare the calcium analyzer (this step is required only when the cuvette solution is changed):

a. Fill the cuvette to the mark with 1.0 N potassium hydroxide.

b. Add 100 μ l of reconstituted calcein solution to the cuvette.

c. Place the cuvette into the analyzer and add 100 μ l of calcium standard. Close the cuvette door and push the titration button.

d. At the end of Step 5c, a ready light will come on indicating that the instrument is ready for analysis.

6. Repetitively analyze the 100 μ l samples of the calcium standard solution until consecutive readings are less than 1.5 percent apart. Push the calibration button and run an additional 100 μ l sample of the calcium standard to ensure that the calcium standard readout is 10 ± 1 (mg %). If it is not, repeat the calibration cycle.

7. Determine the relative calcium strength of the unknown cement solution in the Erlenmeyer flask by analyzing a 20- μ l sample in the calcium analyzer. Repeat this test to ensure reproducibility. The readings should be less than 1.5 percent apart. Determine the cement content by referring to the calibration graph.

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