NATIONAL COOPERATIVE
HIGHWAY RESEARCH PROGRAM REPORT **284**

EVALUATION OF PROCEDURES USED TO MEASURE CEMENT AND WATER CONTENT IN FRESH CONCRETE

TRANSPORTATION RESEARCH BOARD
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EVALUATION OF PROCEDURES USED TO MEASURE CEMENT AND WATER CONTENT IN FRESH CONCRETE

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AREAS OF INTEREST:

STRUCTURES DESIGN AND PERFORMANCE
CEMENT AND CONCRETE
CONSTRUCTION
GENERAL MATERIALS
(HIGHWAY TRANSPORTATION)
(PUBLIC TRANSIT)
(RAIL TRANSPORTATION)
(AIR TRANSPORTATION)

TRANSPORTATION RESEARCH BOARD
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NATIONAL COOPERATIVE HIGHWAY RESEARCH PROGRAM

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FOREWORD

By Staff
Transportation Research Board

This report documents the findings of side-by-side comparisons of several "rapid" methods for determining the cement and water content of fresh portland cement concrete. Comparisons were made under a variety of conditions. The report contains information that can be used to help make decisions on methods to be included in quality assurance programs. The report also contains useful data for further examination under other research projects. The report will be of particular interest to researchers, materials engineers, and construction managers.

Under NCHRP Project 10-25, "Measurement of Cement and Water Content of Fresh Concrete," the U.S. Army Engineer Waterways Experiment Station, Vicksburg, Mississippi, examined seven test procedures that determined either the cement or water content of fresh portland cement concrete. Although most of the test procedures are commonly referred to as being "rapid," none enable a decision to be made on the acceptance of the specifically sampled amount of fresh concrete prior to its placement in forms. However, these rapid methods can be used to audit the performance of concrete produced, and lead to quicker corrections of production problems.

Side-by-side comparisons of the techniques were conducted under several testing schemes. In addition to the cement and water content, variables such as siliceous and calcareous aggregates, fly ash, ground granulated iron blast furnace slag, high-range water-reducing admixtures, and calcium chloride as an accelerator were added to mixes of fresh concrete in varying amounts. Many of the tests performed well under specific conditions, but none performed satisfactorily under all schemes. Consequently, knowing the conditions in which the tests will be performed is essential to the selection of the proper testing technique in a quality assurance program. The tests are best suited for checking conformance with specified conditions, not for explaining the unexpected. The data and commentary in the report will help in making the proper selections.

The tests examined were: (1) U.S. Army Construction Engineering Research Laboratory Concrete Quality Monitor (cement and water), (2) Federal Highway Administration Nuclear Cement Gage (cement), (3) Rapid Analysis Machine (cement), (4) X-ray Emission Spectrometer (cement), (5) Hot Plate (water), (6) Microwave Oven (water), and (7) the modified U.S. Army Engineer Waterways Experiment Station Centrifuge (this was included in the comparisons for cement content, but the primary reason for its inclusion was to provide a possible technique for detecting the presence of slag or pozzolans). Many of the testing techniques will no doubt be studied further, individually and collectively. The data generated from this study should greatly assist such efforts.

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ACKNOWLEDGMENTS

The research reported here was performed under NCHRP Project 10-25 at the Concrete Technology Division, Structures Laboratory, U.S. Army Engineer Waterways Experiment Station. The work was accomplished under the general supervision of Messrs. Bryant Mather, Chief, Structures Laboratory; John M. Scanlon, Chief, Concrete Technology Division; and Richard L. Stowe, Chief, Materials and Concrete Analysis Group. Messrs. Stowe, Alan D. Buck, Tony B. Husbands, and Joe G. Tom were the co-principal investigators for the study. Mr. Tom prepared the report, except for the portions dealing with statistical analysis, which were written by Dr. A. Dale Magoun, Project Statistician.

The work was performed under the direct supervision of Mr. Tom with the assistance of Dr. Magoun. Assisting the co-principal investigators in carrying out the research were Kenneth L. Saucier, Billy D. Neeley, James L. Winters, Donald M. Smith, Alberto L. Laborda, G. Sam Wong, J. Pete Burkes, Joyce C. Ahlvin, Charles L. White, Toy S. Poole, Dennis L. Bean, Judy C. Madison, Shirley D. Griffin, Elizabeth E. O'Dell, Tommy G. Ray, Marvin O. Harrington, Michael K. Lloyd, Julies Mason, Tom W. Lee, Frank W. Dorsey, and Timothy D. Curtis. Phyllis Foster accumulated and processed the data.

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EVALUATION OF PROCEDURES USED TO MEASURE CEMENT AND WATER CONTENT IN FRESH CONCRETE

SUMMARY

This research program was carried out under NCHRP Project 10-25, "Measurement of Cement and Water Content of Fresh Concrete." Concrete producers and users generally agree on the need for tools and procedures to experimentally assess the quality of portland cement concrete before it is placed. They also generally agree that the water-cement ratio is the most important index of the quality of portland cement concrete. Several test methods and procedures have been developed to rapidly determine the water or cement content or both of freshly mixed concrete. The objective of this research was to establish the applicability, bias, and validity of these procedures.

The test procedures investigated were: (1) U.S. Army Construction Engineering Research Laboratory Concrete Quality Monitor; (2) Federal Highway Administration Nuclear Cement Content Gage; (3) Rapid Analysis Machine; (4) X-ray Emission Spectrometer; (5) modified U.S. Army Engineer Waterways Experiment Station centrifuge; (6) hot plate; and (7) microwave oven. Sixty-one different concrete mixtures containing different amounts of concrete materials such as portland cement, water, siliceous and calcareous aggregates, fly ash, ground granulated iron blast-furnace slag, high-range water-reducing admixtures, and accelerating admixture (calcium chloride) were used to evaluate the test procedures.

The major findings of this research were:

1. No one test procedure for determining water or cement content can rapidly and without bias determine the water or cement content of all the portland cement concrete mixtures studied.

2. The ingredients of portland cement concrete can significantly affect the ability of a test procedure to rapidly and accurately determine water or cement content of a freshly mixed concrete mixture.

3. If a procedure is to be used in a quality assurance program, the type or types of concretes proposed will determine which procedure or combination of procedures should be used.

4. None of the procedures evaluated has the ability to qualitatively detect the unexpected presence of ground granulated iron blast-furnace slag or fly ash.

CHAPTER ONE

INTRODUCTION AND RESEARCH APPROACH

BACKGROUND

Concrete is unusual among construction materials in that it is manufactured as used and cannot be tested for acceptance in advance. Because concrete gains strength over a long period of time and the design strength may vary from that attained for test periods ranging from 1 day to 3 months, acceptance is commonly based on strength tests at these advanced ages. There is, at any given time, a large amount of concrete in place, on most projects, the acceptability of which has not been determined. Clearly, a need exists to verify the quality of concrete much earlier, preferably before it is placed.

The water-cement ratio is universally agreed to be the most important parameter for determining the quality of concrete. If the quantities of cement and water can be determined in concrete before it hardens, or if the water-cement ratio can be determined directly by a reliable method, significant progress will have been made. A 1972 survey by the American Association of State Highway and Transportation Officials (AASHTO)-ARTBA-AGC Task Force on Rapid Testing cited the need for methods for measuring the cement content and water content of unhardened portland cement concrete (PCC). Two-thirds of the 43 state agency respondents affirmed the need for such methods.

Several methods have been developed or proposed. None of those developed to date are rapid enough to enable a decision on acceptance to be made before concrete is placed in the forms. Nevertheless, if these methods were reliable, they could be used to audit the performance of the concrete produced and could lead to a much quicker response to correcting production problems than is now possible. The Head and Phillippi report (*1*) is an excellent summary of the methods, their advantages and disadvantages, and various operating details.

A critical examination and evaluation of these methods is needed to assess their adequacy and to provide support data to allow the development of standardized procedures. Questions related to the reliability of the methods when used to test concretes with different aggregate types, cement factors, additives, and admixtures must be answered.

OBJECTIVES

NCHRP Project 10-25 was initiated in response to these needs. The primary objective of the project was to establish the applicability and accuracy, along with the limits of validity, of test methods for the determination of water-cement ratio or cement or water content of freshly mixed concrete. As a minimum, the test methods to be investigated were: (1) U.S. Army Construction Engineering Research Laboratory/Kelly-Vail (CERL K-V), Rapid Analysis Machine (RAM), Federal Highway Administration (FHWA) Nuclear Cement Content Gage (NCCG), and centrifuge test (such as the Willis-Hime method) for determination of cement content; and (2) CERL K-V and microwave oven for the determination of water content.

Attainment of the project objectives necessitated accomplishment of the following tasks:

Task 1—Assess existing field experience with the various methods and summarize available data.

Task 2—Perform statistically designed laboratory experiments in which each method is used to measure the cement and water content of the same concretes. At least the following variables shall be investigated: (a) portland cement concrete (PCC) containing siliceous and calcareous aggregates and mixtures of these; (b) portland cement contents, for a given aggregate type, throughout the range of 300 to 800 lb/cu yd; (c) the effect of additional calcareous fines produced by degradation during mixing; (d) the effect of fly ash and pulverized granulated slag used either as an admixture or as a component of a blended cement; (e) the effects of a low water-cement ratio made possible by the use of a high-range water-reducing admixture and the effects of prolonged mixing on water content; and (f) the addition of calcium chloride.

Task 3—Evaluate the methods to determine the qualitative ability of each method to detect the unexpected presence of slag or pozzolans.

Task 4—Make recommendations as to which procedures or combination of procedures are most suitable for use as a part of a quality assurance program, taking into account the size of project, ruggedness of equipment in the field environment, and required expertise of operators; and make recommendations on potential improvements to existing methods.

RESEARCH APPROACH

In accordance with the overall goal of the project, the research was divided into eight major tasks, which were an expansion of the four tasks specified in the Project Statement. Task 1 involved compiling the results of a comprehensive literature search relating all the available field experiences of seven test procedures and summarizing the available data (the portland cement content procedures under investigation were (1) CERL's Concrete Quality Monitor (CQM), (2) Rapid Analysis Machine (RAM), (3) FHWA's Nuclear Cement Content Gage (NCCG), (4) modified Willis-Hime/U.S. Army Engineer Waterways Experiment Station (WES) centrifuge test (CF), and (5) an X-ray Emission Spectrometer procedure (X-ray); for water content the procedures were (1) CQM, (2) Microwave-Oven method (MW), and (3) Hot Plate method (HP)). Tasks 2 through 6 involved a laboratory study of the seven test procedures in side-by-side comparisons. Task 7 involved qualitative detection of the unexpected presence of pulverized slag or fly ash. Task 8 involved the recommendations.

The experimental design of Task 2 was to evaluate the abilities of all seven test procedures to determine cement or water content of conventional PCC. The design required a series of tests and evaluations on known concrete mixtures containing various cement factors, aggregate types, and aggregate ratios. The experimental design of Task 3 was to evaluate only the ability of the five cement-content procedures to recover cement content from concrete mixtures that were excessively mixed to simulate the effect of prolonged mixing. This simulation was achieved by the addition of calcareous aggregate fines to the concrete mixtures.

Tasks 4 through 6 involved the use of admixtures in the concrete and the determination of the ability of the test procedures to determine cement or water content. The experimental design for Task 4 required the addition of two mineral admixtures to the basic concrete mixtures and determination of their effect on the ability of the test procedures to determine portland cement and other cementitious material contents. The experimental design for Task 5 involved adding two types of high-range water-reducing admixtures to the concrete mixtures to reduce the water-cement ratio to a minimum, and then determining whether the three water content procedures were affected by the minimal water content or by the chemical composition of the admixture. The experimental design for Task 6 required the addition of an accelerating admixture (calcium chloride) to the concrete mixture and evaluating the five cement-content procedures for a concrete mixture that is rapidly hardening.

Task 7 was an extension of Task 4 to determine the effects of mineral admixtures on cement-content determination. The task involved determining the ability of the test procedures to qualitatively detect the unexpected presence of pulverized, granulated iron blast-furnace slag or fly ash in a PCC mixture.

Task 8 involved evaluating the data, drawing conclusions, and making general recommendations as to which test procedure or combination of test procedures would be best suited for use as a part of a quality assurance (QA) program. The results will suggest recommendations for potential improvements and changes to existing procedures and provide input for development of new techniques for the rapid analysis of freshly mixed PCC.

TEST PROGRAM

General

To conduct a systematic and statistically valid testing program involving several different materials that are found in PCC, a system of evaluating each test procedure individually and versus each other was developed. The test program involved proportioning conventional PCC mixtures containing ingredients that may influence the cement or water content determination of the test procedures. The PCC mixtures resembled concrete conventionally used by state transportation departments and the U.S. Army Corps of Engineers and produced at ready-mixed concrete plants. The test program required a minimum of five statistical replications to detect a 10 percent difference among test procedures at the 90 percent confidence level as detailed in Appendix B. The experimental design considerations included evaluating known PCC mixtures in each test procedure from the same batch of PCC; performing five replications; comparing each of their cement or water content recoveries to each other, their mean, and to the known contents; and determining the effects of cement factor, aggregate factor, admixture factor, and prolonged mixing on the bias of the test procedure in determining cement or water content in freshly mixed PCC. In this project "bias" means getting an answer that is higher or lower than the correct value.

Materials

The concrete materials used for this project were: Type I portland cement, siliceous fine aggregate (American Society for Testing and Materials (ASTM) C 33 grading), calcareous fine aggregate (ASTM C 33 grading), 1-in. nominal maximum size siliceous coarse aggregate (ASTM C 33 No. 56 grading), 1-in. nominal maximum size calcareous coarse aggregate (ASTM C 33 No. 56 grading), Class F pozzolan (fly ash), ground granulated iron blast-furnace slag, a sulphonated naphthalene formaldehyde condensate high-range water-reducing admixture (HRWRA), a sulphonated melamine formaldehyde condensate HRWRA, the accelerating admixture calcium chloride, and water.

Sixty-one concrete mixtures were proportioned using these materials. The variables included three cement contents, three aggregate combinations, two fine-to-coarse aggregate ratios, three fly ash to total cementitious materials percentages, three slag to total cementitious materials percentages, four naphthalene-based HRWRA percentages, one melamine-based HRWRA percentage, two calcium chloride percentages, and two prolonged mixing simulations. Details of the concrete materials and the PCC mixtures are presented in Appendix C.

Test Procedures

The seven test procedures were evaluated for their ability to determine the cement or water content of PCC mixtures. These included the RAM (*2*), COM (*3*), NCCG (*4*), X-ray, modified Willis-Hime/WES CF (*5*), MW (*6*), and HP. Details of each test procedure, theoretical design, calibration, use, application, and discussions are presented in Appendix D.

Operators

Primary and alternate operators were chosen from the technical staff of the Concrete Technology Division, WES. One primary operator and two alternate operators were assigned to each test procedure or combination of test procedures. Appointment of operators was based on direct experience and background with a test procedure or similar technique. More detailed information about the operators is presented in Appendix E.

CHAPTER TWO

FINDINGS

FIELD ASSESSMENT

Information on the field experience for the seven test procedures was very limited. Most literature reviewed concerned performance of the equipment under strict laboratory controls to determine the accuracy and precision of the equipment and not the field worthiness of the equipment. In the reports pertaining directly to field use and field assessment, only four of the seven procedures had been evaluated under actual field conditions. These were the RAM, CQM, NCCG, and MW.

Most data on the RAM were accumulated from literature supplied from the Cement and Concrete Association (England). Other sources included Canada and the United States. North America had only four RAM's in use, while Europe had nearly 200 units in use on various field projects. When cement factors are specified as they are in Europe, there is a need to invent and devise rapid cement content analysis equipment. In the United States, specifications give requirements for the unconfined compressive strength of PCC at 28 days. The RAM originated in England and as shown by its strong market has been a useful test procedure. Apparently, the RAM is a field-worthy testing device on the European market. Reports indicated that RAM's have been located in a mobile van and transported about work sites and from site-to-site without adversely affecting the equipment.

In England, Dr. R. T. Kelly and Mr. J. W. Vail did research on a test procedure which became known as the Kelly-Vail. The K-V was introduced in the United States around the mid-1970's, when CERL obtained a unit and began extensive research to modify it for field applications. The use of metal and plastic instead of glass along with other minor changes produced the CERL K-V, the second generation K-V. More improvements followed, including electronic analyzers, that produced the CERL CQM, the third-generation K-V.

The CQM was built in 1981; there are a limited number of existing reports on the field assessment of the device. The reports contained data on conventional PCC and data for cement content of roller-compacted concrete (RCC) and soil-cement mixtures. The CQM is considered compact and is easily shipped by automobile, rail, and airplane. Assembly takes about 1 hour and about the same time to disassemble and repack. The CQM was used and evaluated on several Corps of Engineers projects; however, test results were not published. One advantage of the CQM is that both the cement content and the water content of PCC can be ascertained. Because of this advantage, the CQM was probably used more in testing programs than other apparatuses that have been used to determine cement and water content separately. The CQM unlike the RAM was developed to be operated with standard laboratory equipment. Most of the CQM component parts may be found, gathered, and assembled from within many large construction materials testing laboratories. The CQM reportedly has been used successfully on field projects. The CQM is primarily used in the United States; all the data reviewed were from projects in the United States.

The NCCG was developed in the early 1970's by the FHWA. Today's prototype was produced in 1975. The NCCG has been used on several highway construction projects under the supervision of FHWA personnel. One advantage of the NCCG is the complete portability of the equipment. No external supply of either electricity or water is required as with all of the other procedures. With its battery pack, the NCCG can be used at the concrete placement location. The radioactive source, americium 241, emits gamma rays that are absorbed by the PCC, and secondary X-rays are generated by each of the chemical elements present. The intensity of the secondary emission at the wavelength of an element characteristic of the element, such as calcium, is taken to be proportional to the cement content. The literature supports the field worthiness of the NCCG, and it can be transported from site-to-site in the back of a pickup truck without damage.

The MW procedure was not used frequently until the mid-1970's when these ovens were more common in laboratories. Up to that point, the hot plate was used extensively to remove moisture in hardened concrete, fresh PCC, soils, and aggregates; however, in the process of drying fresh PCC, cement hydration was believed to be hastened, making a portion of the water nonevaporable. The microwave has the advantage of heating from within each particle. The microwave procedure was used on several highway projects and the indications were that the microwave would withstand the hard usage in a field application. Preliminary laboratory testing, however, resolved the problem of greater weight loss during heating than that due to water in the concrete sample. The testing showed the cement particles themselves were melted by the intense microwaves after about 20 min at high power levels (*6*). This was apparently similar to what happens during a loss on ignition test which involves heating in a furnace at 950 ± 50°C. The problem was resolved by conducting the tests in a defrost mode or at a lower power level. No field experience could be found on the other test procedures—the CF, X-ray, and HP.

Table 1 summarizes the field assessment data.

EVALUATION OF TEST PROCEDURES

General

The seven test procedures were evaluated based on their ability to determine the cement or water content of a PCC mixture. The evaluation included determining which method or combination of methods would be best suited for use. Each test procedure was evaluated with samples obtained from the same batch of concrete. The five cement content procedures were tested and evaluated against each other as were the three water content

Table 1. Field assessment data.

	Percent Recovery Range	
	Cement Content, percent	Water Content, percent
CF	No Data	N/A[a]
CQM	91.7 to 106.8	98.8 to 129.5
HP	N/A	No Data
MW	N/A	94.6 to 99.9
NCCG	98.5 to 104.8	N/A
RAM	97.8 to 115.8	N/A
X-Ray	No Data	N/A

[a] Not applicable.

procedures. The CQM is capable of determining both the cement and water content. The CF procedure was evaluated fully in Task 2 and partially in Task 4. The CF procedure was not evaluated further when (1) the chemicals required for normal operation were determined to be too hazardous to the health of the operators, and (2) the procedure was determined to be too lengthy for the rapid analysis of fresh concrete.

Of the cement content procedures, none emerged as the single most unbiased procedure for all types and combinations of concrete mixtures. The same also holds true for the three water content procedures.

No one procedure proved to determine water content of PCC mixtures better than the others. Each test procedure had a distinctive character or quality with each different concrete mixture. All PCC mixtures had known quantities of concrete materials, particularly the portland cement and water, which were the basis of the evaluation. Known quantities of portland cement and water were added to each of the mixtures and were the base recovery factor of each mixture. All calculated amounts of cement and water recovered were in terms of percent recovery of the original cement or water content, respectively. Summary tables are included in Appendixes L, M, and N.

Task 2

In Task 2 the test procedures were evaluated with conventional concrete, varying only cement factors, water-cement ratios, aggregate types, and aggregate ratios. The test procedures were evaluated with respect to those mixtures containing calcareous aggregate, siliceous aggregate, or a blend of siliceous fine aggregate and calcareous coarse aggregates at cement factors of 350, 550, and 800 lb/cu yd. Because of the highly significant interaction among the variables, no single testing procedure proved to exhibit better measurement characteristics for determining percent cement recovery. For each statistical design variable, individual recommendations and considerations were made. For the calcareous aggregate mixtures at the 350-lb/cu yd level, the CF procedure was recognized as the only procedure to consider and recommend; at the 550-lb/cu yd level, the CQM and RAM emerged as not being significantly different from 100 percent recovery; and at the 800-lb/cu yd level, the CQM, NCCG, RAM, and X-ray were considered better because they were considered not significantly different from 100 percent recovery. For the siliceous aggregate mixtures at the 350-lb/cu yd level, the NCCG and X-ray emerged as the procedures to consider, but at the 550-lb/cu yd as well as at the 800-lb/cu yd levels the CQM, NCCG, RAM, and X-ray were considered not significantly different from 100 percent recovery. For the blended aggregate mixtures, the CF and RAM were considered better at the 350-lb/cu yd level, and the CQM, NCCG, RAM, and X-ray procedures were better at the 550-lb/cu yd and 800-lb/cu yd levels.

Also in Task 2, the procedures for determining water content were evaluated. The test procedures that were not considered significantly different from 100 percent recovery for the calcareous and blended aggregate mixtures were the HP and MW procedures; and for the siliceous aggregate mixtures only the MW procedure emerged as the test procedure to consider and recommend. Details are presented in Appendix F.

Task 3

In Task 3, the test procedures were evaluated for their ability to determine cement content contaminated by calcareous aggregate fines generated by degradation of the coarse aggregate as a result of prolonged mixing. The test procedures were evaluated with respect to those mixtures containing calcareous aggregates at cement contents of 350 and 800 lb/cu yd by adding fines in amounts that could be developed by 15 and 60 min (7) of additional mixing. All procedures were conducted as they were in Task 2, using the basic calibration curves for their respective mixtures. The objective was to determine the effect of prolonged mixing and subsequent additional fines generated on the test procedures. PCC mixtures containing calcareous aggregate that were mixed longer exhibited significantly higher percent recovery values than those mixed for a shorter time period for the CQM, NCCG, RAM, and X-ray. This trend was not observed for the test procedures evaluated with the blended aggregate mixtures. The simulated mixing times did not affect the percent cement recovery for the blended aggregate mixtures. The test procedures displayed overall average percent recoveries significantly higher than 100 percent at both prolonged mixing times with the calcareous aggregate mixtures. Regardless of the nonsignificant effect of mixing time, the CQM and RAM had average percent recoveries that were not significantly different from 100 percent recovery; however, the NCCG did produce average percent recovery values that were significantly different from 100 percent recovery for the 60-min averages. Furthermore, the X-ray procedure was significantly different from 100 percent recovery with its average percent recovery values for both the 15- and 60-min averages. Nevertheless, for the calcareous aggregate mixtures, simulated prolonged mixing up to 60 min did tend to increase the percent recovery values. This increase in percent recovery, however, was only significantly different from 100 percent recovery for the average recovery values produced by the NCCG. In addition, if mixing times were increased past 60 min, the effect observed with the NCCG would most probably be demonstrated by each of the other test procedures. Details are presented in Appendix G.

Task 4

In Task 4, the test procedures were evaluated for their ability to determine cement content of a PCC mixture which contained fly ash or ground granulated iron blast-furnace slag as a cementitious material. The test procedures were evaluated with respect to those mixtures containing varying calcareous and siliceous aggregate types, initial cement contents of 550 and 800 lb/cu yd, and 15 and 40 percent of either fly ash or pulverized slag by volume of total cementitious material. Each test procedure was recalibrated by making up new calibration curves from mixtures containing the fly ash and slag replacement percentages. The fly ash mixtures tended to produce higher average percent cement content recovery values for both the calcareous and siliceous aggregate mixtures. The observed increase in percent recovery with the increase of fly ash from 15 to 40 percent was only significantly higher with the calcareous aggregate mixtures. At the 15 percent fly ash level, the CQM, NCCG, and X-ray had average percent recovery values that were not significantly different from 100 percent recovery. At the 40 percent fly ash level, all three test procedures tended to overestimate the cement content above the 100 percent recovery level. With the siliceous aggregate mixture, the nonsignificant increase in average percent recovery values did not affect the ability of the CQM or the RAM to estimate cement content. At both fly ash percentage levels, the CQM and RAM had average percent cement recovery values that were not significantly different from 100 percent recovery. The NCCG and X-ray, however, did produce recovery values that were considered significantly different and also lower than 100 percent recovery.

Ground granulated iron blast-furnace slag did not produce any significant effects with respect to either the calcareous or siliceous aggregate mixtures. The CQM had average percent recovery values that were not significantly different from 100 percent recovery at either the 15 or 40 percent slag levels. The NCCG had average percent recovery values that were not significantly different from 100 percent recovery only at the 40 percent slag level. The RAM, on the other hand, had average percent recovery values that were not significantly different from 100 percent recovery only at the 15 percent slag level. The X-ray procedure produced average percent recovery values that were significantly different from 100 percent recovery at both the 15 and 40 percent slag levels. Details are presented in Appendix H.

Task 5

In Task 5, the water content test procedures were evaluated for their ability to determine percent water recovery of PCC mixtures with very low water-cement ratios. The procedures were evaluated with respect to those mixtures containing a HRWRA, either naphthalene-based or melamine-based. Increasing the percentages of the naphthalene-based HRWRA from 0.5 to 5.0 percent exhibited mixed trends depending on the type of aggregate used. For calcareous-aggregate mixtures, the percent water recovery values increased for all three test procedures when the percent naphthalene-based HRWRA increased from 0.5 to 2.0 percent, but oscillated thereafter for the CQM and decreased for both the HP and MW procedures. However, only the CQM produced average percent water recovery values that were significantly different from and higher than 100 percent recovery. For the siliceous aggregate mixtures, the overall trend was an increase in percent water recovery as the percentage of naphthalene-based HRWRA increased. Only the percent water recovery values for the CQM and HP at 5.0 percent naphthalene-based HRWRA were significantly different from and higher than 100 percent recovery. For the melamine-based HRWRA comparison with the naphthalene-based HRWRA, neither admixture produced any significant effects; however, only the HP procedure produced average percent water recovery values that were not significantly different from 100 percent recovery. Details are presented in Appendix I.

Task 6

The test procedures, in Task 6, were evaluated for their ability to determine the cement content of a concrete mixture containing an accelerating admixture. The test procedures were evaluated with respect to those mixtures containing varying percentages of calcium chloride from 1.0 to 2.0 percent. The PCC mixtures used in this task were identical to those in Task 2 except for the use of an accelerator. This allowed the use of the basic calibration curves used in Task 2; test procedures were also conducted in the same manner as for Task 2. Only the calcareous aggregate mixtures with either percentage of calcium chloride exhibited a significant effect for all test procedures. The trend was a decrease in percent cement recovery with an increase in cement content and calcium chloride percentages. The CQM and NCCG produced average percent recovery values that were not different from 100 percent recovery at a cement content of 550 lb/cu yd; yet, only the X-ray procedure had average percent recovery values that were not different from 100 percent recovery at the 800-lb/cu yd cement content. For the siliceous aggregate mixtures, the percent recovery was not affected by the addition of calcium chloride. Also, the CQM and X-ray procedures were the only procedures that produced average recovery values that were not different from 100 percent recovery. Details are presented in Appendix J.

Task 7

Task 7, an evaluation and determination of the ability of the five test procedures to qualitatively detect the unexpected presence of fly ash or ground slag in a concrete mixture, was an extension of Task 4. None of the five cement-content test procedures under investigation had the qualitative ability to detect the presence of fly ash or ground granulated iron blast-furnace slag in a concrete mixture. The use of optical microscopy was available for detecting fly ash and slag in PCC by particle shape and crystallinity. Results of this task indicated that only the CQM, X-ray, and NCCG for fly ash mixtures can determine the actual cement content of PCC mixtures with a cement factor as low as zero. Details are presented in Appendix K.

Time Required

Time-required was the parameter next in importance to bias in the evaluation of methods for rapid analysis of freshly mixed PCC. Reported times (for comparison) are all from Head and Phillippi (*1*). The CQM, which was used to determine both water content and cement content, was reportedly performed within 15 min for both; but the average time from sample collection to results was approximately 30 min for this work. The NCCG reportedly required 10 to 15 min; the average time required here was 10 min. The RAM reportedly required 5 to

10 min, but the average time required here was 15 min. The X-ray procedure required 50 min. The CF procedure reportedly required 75 min, although the average time required was 110 min. The MW reportedly required 60 min in the defrost mode; the average time required here was 35 min. The HP procedure required 35 min.

CHAPTER THREE

INTERPRETATION, APPRAISAL, AND APPLICATION

PROBLEM SOLUTION

Lack of bias is the most important criterion involved in the rapid analysis of PCC. The seven test procedures evaluated can all be used accurately to determine the cement and water content of concrete under the conditions for which they were developed.

However, using Tukey's w-procedure and Dunnett's procedure (*8*), no one test procedure was capable of accurately determining the water or cement content for all the PCC mixtures studied. Tukey's w-procedure is a statistical method for making comparisons among a set of mean values and determining which ones can be grouped together as representing essentially the same result. Dunnett's procedure is a statistical method for comparing multiple sets of results to a control value in order to determine which ones differ significantly from the control. The detailed results of this study are outlined in Appendixes F through K.

The water-cement ratio is usually considered to be the single most important parameter for controlling PCC quality. Although no single device can directly determine the water-cement ratio, any combination of one of the five cement content procedures with one of the three water content procedures can produce numbers from which a water-cement ratio can be calculated. The CQM is actually two separate test procedures; one determines cement content and the other determines water content. The two procedures are completely independent of each other and can be used in conjunction with any of the other cement or water content determination procedures. However, the name CQM designates the two procedures as a single test method with separate procedures for cement and water content.

Time-required is probably the second most important criterion for the rapid analysis of PCC. Rapid analysis of freshly mixed concrete should ideally be performed before the concrete is placed in the forms. None of the test procedures that were evaluated in this project was capable of instantaneously determining the cement or water content of a PCC mixture. The NCCG proved to be the quickest in that a cement content can be obtained in 10 min from the time of sampling to testing to computing results. The only procedure that required more than an hour was the CF procedure, and it required 1 hr 45 min to as much as 2 hr depending on the concrete composition. This excessive time eventually led to the deletion of the CF procedure from further evaluations. The CQM proved to be the quickest for obtaining a water content result. It required only 10 min from sampling to testing to computing results. The CQM can be used to obtain a water-cement ratio in 30 min including sampling, conducting two tests, and computing results.

STANDARDS AND SPECIFICATIONS

A review of the results of bias for either cement or water content revealed that the seven test procedures cannot be used to accurately analyze all types of concrete. The development of a standard test method for each of the procedures is recommended through the ASTM, Corps of Engineers, or the AASHTO. However, the standard should be of a generalized nature to indicate that perhaps unacceptable results may occur when evaluating certain concrete mixtures. The findings gave credence to the fact that each test procedure does provide acceptable and significant test results of water or cement content of several PCC mixtures that are used in concrete construction. Project specifications are written to the construction of a structure. These specifications should be written with prior knowledge of concrete requirements and types of concrete to be produced in order to use the proper test procedures for quality control and quality assurance.

APPRAISAL AND APPLICATION

Concrete Quality Monitor

Although all seven test procedures were worthy and accurate for determining the parameter they were designed to determine, there were limitations associated with each procedure; in other words, they cannot be used to evaluate all types of concrete mixtures. The initial cost of the CQM was $7,000. The chemicals—sodium chloride, acid buffer, nitric acid, ethylenediaminetetraacetic acid (EDTA) solution, calcium indicator, calcium standard, and potassium hydroxide—were an additional cost. These items must be monitored closely in terms of lead time for reordering and shelf life; for example, the shelf life of calcium indicator was 4 to 6 weeks. During this project, the CQM required a chemist or an individual with a background in chemistry to operate the device with confidence. The CQM used two electronic analyzers for calcium and chloride. These were questionable both in being sufficiently rugged for field use and available for future users of this procedure. Other analyzers were available but unproven; however, the procedure may fall back on its predecessor which used the chemical titration sequence. In addition, the CQM cement test analyzed a small subsample, a 30-mL sample from a 37.6-L solution.

Nuclear Cement Content Gage

The NCCG had an undetermined initial cost associated with it because it was a prototype and not available to the general public. There were only two in existence; both were owned by the FHWA. Potential users of the equipment will have several problems and inherent limitations with which to contend when using the NCCG. It contained 0.014 curies (5.2E8 B_q) of americium 241, a radioactive material as its source of operation. Prior training in handling equipment containing radioactive material was required. Regardless, the borrowing agency must first obtain a license to possess and operate radioactive equipment from the U.S. Nuclear Regulatory Commission. The license can take up to 6 months to obtain. Very stringent requirements were imposed on the borrowers. The equipment or the radioactive source itself had to be kept secure at all times, during use or in storage. The operators wore radiation detection badges to monitor the amount of radiation exposure of the body, and the user must comply with special shipping regulations when moving from site-to-site or from FHWA to user and vice versa. The NCCG also had an extensive calibration requirement. The calibration was affected by small changes in fine and coarse aggregate ratios and mixture proportions. Three people were needed to perform each calibration, one operator and two laborers. The operator should have an adequate mathematics background to comprehend the least-squares fit equation that is needed for the prediction equation. When the NCCG was used outdoors, it was very sensitive to ambient temperature changes. However, the NCCG was the only test procedure evaluated that was completely and totally portable. The rechargeable batteries allowed the NCCG to be taken to remote sites without the need for a source of water or electricity.

Rapid Analysis Machine

The RAM had an initial cost of between $7,000 and $10,000 depending on the purchase location—in the United States or in England. Distributors in the United States have been reluctant to market the RAM, which has contributed to its limited use in the United States. The chemicals—aluminum potassium sulfate and Nalfloc N625, a proprietary chemical agent—were an additional cost. They must be monitored very closely in terms of an unknown shelf life, lead time when ordered from England, and reordering. They must be shaken to homogenize and disperse sediment; however, if unused chemicals are left in the RAM, there is no way to stir the chemicals sufficiently and the chemicals must be removed and fresh chemicals added. The RAM had an extensive and elaborate calibration procedure and is very sensitive to minor changes in the fine aggregate content and overall mixture proportion. The RAM uses less than 10 percent of the concrete sample, which may lead to possible errors. However, of the five cement content procedures studied, the RAM was, perhaps, the easiest and simplest to operate because it was almost totally automatic.

X-ray Emission Spectrometer

The X-ray Emission Spectrometer unit used during the conduct of this project was a laboratory unit. Portable X-ray units were available with sufficient capabilities and capacities to fulfill the requirements; however, the initial cost is unknown, but was quite likely $30,000 to $60,000. The operator must be trained and qualified to operate the X-ray unit. Radiation is generated internally; therefore, additional safety precautions must be taken. Equipment must be kept away from other personnel; the unit must be secure at all times, in use or in storage. Operators must wear radiation detection badges to monitor the amount of radiation the body is exposed to and must follow strict operational procedures to avoid exposure. The operator should have training in physical science in order to read, comprehend, and evaluate the X-ray patterns into elements present and their relative quantities.

Centrifuge

The CF procedure is a laboratory test procedure. Safety precautions for this procedure are very explicit. The chemicals—sodium dodecyl benzene sulfonate; SEPARAN NP10, a proprietary product; acetone; and tetrabromethane—must be handled carefully. The acetone and tetrabromethane are listed as highly flammable and highly toxic substances, respectively. Rubber gloves, rubber aprons, safety goggles, a 100-cu ft/min velocity fume exhaust hood, and medical surveillance must be maintained for the operator. Disposal of chemical waste must be through the U.S. Environmental Protection Agency (EPA)-approved disposal sites and under EPA guidelines. The CF procedure is also a lengthy test, requiring as much as 2 hr to complete. The operator should have the ability to accurately weigh and measure the chemicals and concrete sample. The CF procedure included a significant amount of operator judgment in the performance of the test.

Hot Plate

The HP procedure had a low cost associated with it and was an efficient test procedure. In determining water content for water-cement ratios, the absorption and mass of both fine and coarse aggregates must be known, as well as the amount of liquid admixture used. Care must be taken to avoid loss of material during the stirring and drying operation.

Microwave

The MW procedure had an initial cost associated with it of $200 to $500 for purchase of the oven. This procedure was used to determine the total water content and, therefore, requires that the aggregate absorption and mass be known as well as the liquid admixture volumes. The use of the defrost mode in the microwave oven rather than a higher power setting was recommended by earlier studies (*6*). The authors stated that the microwaves can actually decompose hydrated cement particles in the concrete sample, thus increasing the liquid content. The lower power setting or the intermittent turning on and off of high power settings to obtain a defrost mode apparently does not affect the cement particles.

CHAPTER FOUR

CONCLUSIONS, RECOMMENDATIONS, AND SUGGESTED RESEARCH

CONCLUSIONS

The following conclusions are believed warranted based on the research described in this report.

1. No single procedure evaluated proved to exhibit better measurement characteristics than any other procedure for determining the cement content of all the PCC mixtures studied.
2. No single procedure evaluated proved to exhibit better measurement characteristics than any other procedure for determining the water content of all the PCC mixtures studied.
3. PCC containing calcareous aggregates affected the bias of CQM, NCCG, and X-ray procedures more than the other procedures. The calcium content of the aggregate tended to affect those instruments that detected the element calcium through chemical reactions or radiation.
4. PCC containing siliceous aggregates affected the bias of the X-ray method, which detected the element silicon.
5. PCC containing a blend of siliceous fine aggregate and calcareous coarse aggregate tended not to affect the bias of test procedures as much as the straight aggregate mixtures.
6. More error was found using concrete of low cement contents than using concrete of high cement contents for all methods.
7. PCC subjected to simulated prolonged mixing affected the bias of the NCCG and X-ray methods more than it affected the CQM or RAM procedures.
8. PCC subjected to simulated prolonged mixing affected the bias of all test procedures as the mixing time increased.
9. PCC containing Class F pozzolan (fly ash) affected the bias of all the test procedures.
10. PCC containing fly ash tended to have more effect on the bias of all test procedures as the percentage of fly ash increased.
11. PCC containing ground granulated iron blast-furnace slag tended to affect the bias of the NCCG, RAM, and X-ray procedures more so than the bias of CQM procedure.
12. PCC with a low water-cement ratio achieved through use of a HRWRA tended to affect the bias of CQM water content determination more than it affected the bias of the hot plate and microwave procedures.
13. PCC containing different HRWRA did not tend to affect the bias of the test procedures.
14. PCC containing calcium chloride as an accelerator tended to affect the bias of the test procedures.
15. PCC containing calcium chloride as an accelerator affected the bias of all the test procedures more as cement factors increased.
16. None of the test procedures evaluated could qualitatively detect the unexpected presence of fly ash or ground granulated iron blast-furnace slag in a PCC. The use of optical microscopy appeared to be a viable method of detecting fly ash and ground granulated iron blast-furnace slag, and optical microscopy can be easily used in the field.
17. The centrifuge procedure should not be used as a field test procedure for the rapid determination of cement content in freshly mixed PCC, because it is too slow and requires the use of toxic chemicals.

RECOMMENDATIONS

The seven test procedures that were evaluated for their ability to rapidly and without bias determine the cement or water content of a freshly mixed PCC mixture are suitable for use as a part of a quality control and quality assurance program for a concrete construction project. The CF procedure because of its lengthy operation and strict safety requirements should be used only with extreme caution and guidance. However, any of the other six test procedures—CQM, NCCG, RAM, X-ray, HP, and MW—may be readily used in a QA program.

Any of the seven procedures used individually or in combination to determine cement or water content for the water–cement ratio determination may be used in conjunction with equipment and test methods currently implemented in a concrete QA program. However, many factors may affect the choice of procedure(s) which should be used for a particular project. Such factors may include ruggedness, required operator expertise, and overall size and construction time for the project.

The findings indicate that no single procedure, for cement or water content, can evaluate all types of PCC that may be used on a concrete construction project. Therefore, the requirements of each project should be considered in choosing a procedure or combination of procedures for use in a QA program. Work reported on in this report primarily determined statistically how well each test procedure could measure cement or water content or the percent recoverable by each procedure. There are no hard and fast statements recommending one test procedure over the other procedures for all types of concrete. A procedure may be recommended, however, for use on a particular project because the procedure works well or best for materials that will be used on the project.

Because of the use of laboratory rather than actual field conditions for evaluation, recommendations of equipment ruggedness were not applicable to this project except as a general comment and prediction of probable results. The CQM may have erratic readings from its calcium and chloride analyzers because they are susceptible to dust. The RAM must be level at all times to adequately follow its automatic sampling cycle. The constant handling of the cables for the NCCG may cause the cables to fray or pull loose, thus causing erratic readings. The ruggedness evaluation must be conducted under actual field conditions to fully determine overall field worthiness or ruggedness.

Ease of operation and required operator expertise should also be considered. Devices that are simple and straightforward usually have somewhat greater success in terms of acceptability by the users and their management. Procedures for operating the RAM, microwave, hot plate, and NCCG were easy to learn with minimal training. The CQM, CF, and X-ray methods were more complex and perhaps required the technical background level of an engineer or scientist or a senior technician. The skill and ability of field technicians may well be the most important parameters to consider.

The type of project is also important in deciding what instrument to use. For the large projects, where a well-equipped laboratory will be involved for several years, all of the procedures may be set up and used. Most samples will have to be brought to the test apparatus, but as the projects become smaller in concrete volumes and shorter in duration, the method of rapid testing becomes more restrictive. The smaller and shorter the project, the more portable the equipment for a procedure must be to adequately fulfill the requirements. In the case of central batch plant equipment the procedure could easily be set up in the laboratory where the quality control and quality assurance personnel perform their conventional physical tests. However, if the concrete is received from ready-mix concrete trucks at a remote site, the only procedure readily available as a portable unit is the NCCG because it requires no external power or water source to operate a series of tests. Many projects have minimal laboratory equipment to perform the essential tests and will have a portable laboratory that may be supplied by generators and water truck; these projects must be selective in their rapid test procedures. Here the RAM, CQM, NCCG, MW, and HP procedures could be used.

SUGGESTED RESEARCH

With twenty-one variables affecting the water content or cement content or both of any PCC mixture, the present program became too broad to control even a single variable with seven different test procedures. The five sample replications were a minimum to obtain statistical validity for this program only. It is possible the results would not be different if more samples were evaluated; however, those results that were significantly different from a 100 percent recovery may or may not remain the same. The following research is suggested:

1. Extend the studies to obtain greater depth in evaluating individual test procedures. Divide the broad program into smaller projects and examine the procedures in greater detail.
2. Look at each individual test procedure in order to recommend improvements rather than trying to improve the procedure as it is being evaluated.
3. Take the instruments to the field, determining their ruggedness by side-by-side comparison in actual field conditions where dust, vibration, and timing are critical. Expose the equipment to truck rides, weather changes, operator changes, water problems, electrical problems, and so on.
4. Determine some of the unknowns associated with each test procedure such as chemicals, safety hazards, and procedure sequences.
5. Develop or modify existing equipment to qualitatively detect the unexpected presence of foreign materials including fly ash, granulated iron blast-furnace slag, silica fume, chemical admixtures, as well as contaminants left in bulk tanks, aggregate trucks, railcars, etc.
6. Develop a more rapid or instantaneous method of determining water content or cement content or both or the water–cement ratio of freshly mixed PCC. An instantaneous method would involve direct results—no calibration curve, no mixing or weighing. A gage would simply be read in pounds per cubic yard or water–cement ratio.

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APPENDIX A

FIELD EXPERIENCE ASSESSMENT

The assessment was made of the available field data on the seven test procedures. The assessment was conducted through three major phases: (1) literature search of available reports and papers pertaining to the evaluation or direct use of any of the seven test procedures; (2) accumulation and review of the reports and papers that pertained to field use of any of the seven test procedures; and (3) summarizing of the data from pertinent reports and papers as shown in Table A-1.

PHASE ONE - LITERATURE SEARCH

Part one of the literature search consisted of retrieving all existing reports retrievable through the use of the following information retrieval services:

1. Highway Research Information Service (HRIS)
2. DIALOG Information Service
3. Engineering Index
4. National Technical Information Service (NTIS)
5. Defense Technical Information Service (DTIC)
6. Ohio College Library Consortium (OCLC)
7. Concrete Technology Information Analysis Center (CTIAC)

The Technical Information Center at the WES used these seven information retrieval services. A series of keywords including the names of the seven procedures and words such as "cement content," "water content," "fresh concrete," "rapid analysis," "concrete," and "testing" were keyed into the information services and all available reports were sought with these words in the title or in the keyword section of the report. Several thousand reports were located with these keywords; however, only a limited number pertained to the seven test procedures under investigation.

Many of the abstracts that were reviewed appeared to be reprints or duplications of items previously found. Most of the abstracts indicated the instruments were prototypes and early models before modifications were made. For three of the procedures--X-ray, CF, and HP--no experience data were found. The abstracts indicated the instruments were evaluated but under laboratory conditions rather than field conditions as required by the terms of this project. Prototypes and special purpose instruments are almost always tested and evaluated in controlled laboratory conditions to determine requirements for revisions. This was apparently the case for many of the procedures for determining cement and/or water content of freshly mixed PCC.

Part two of the literature search consisted of obtaining additional reports that were not available through the information retrieval services. For many construction projects instruments and equipment are used, but reports of their use are not given as for a research project. Therefore, the manufacturers or current owners of the test procedures were contacted for additional information on recent and current projects that have used and are using any of the test procedures. These include the CERL, FHWA, and Wexham Development, Ltd., London, England. Several representatives of these organizations provided additional information by means of letter reports, unpublished papers, raw data, and names of other organizations that had used the instruments or procedures, and also who presently has possession of any of the test procedures or instruments.

PHASE TWO - FIELD EXPERIENCE

The published reports, letter reports, unpublished papers, and raw data accumulated from the literature search were reviewed to delineate those items that did not pertain to or that were not specifically related to any field exercise or project. Only the reports that contained data from the field where a procedure or instrument was actually on site and was used as part of a quality control or quality assurance program, or was used as an experimental device in a research project, were evaluated. The field assessment, therefore, contained only those reports that were field related. All other reports that featured any of the test procedures or instruments but which were exercises in controlled laboratory situations were not included in the field assessment.

Additional field data were obtained from several construction projects of state transportation departments, Corps of Engineers Districts and Divisions, and through the Cement and Concrete Association in England.

PHASE THREE - DATA ANALYSIS

Rapid Analysis Machine

Field data for the RAM were very limited. There were only three RAM's in the United States and one in Canada. Most data were obtained from England, where some 160 units are in use throughout Great Britain and Europe.

The RAM reportedly has field mean recoveries ranging from 97.8 to 104.8 percent of cement content from data obtained from London to a Canadian mean recovery ranging from 100.0 to 102.8 percent to United States field mean recoveries of 115.8 percent of cement content as shown in Table A-1. The data indicate the RAM has been used widely and quite successfully in Great Britain and Europe.

The British have developed a unique concept with the RAM. The "RAM in a Van" concept permits the unit to travel from site to site testing

and evaluating PCC. This concept actually begins to determine the ruggedness and reliability of the instrument in actual field conditions. No longer a laboratory-based instrument, the RAM may become a more usable testing apparatus in the United States.

The RAM in a Van concept was primarily used to obtain the data and results from projects in England. The manufacturers have been going from project to project analyzing the PCC. All contracts specify cement contents rather than compressive strengths as the controlling factors in concrete acceptance. This type of specification lends more to the actual rapid analysis of PCC for cement content or even water content. The original Kelly-Vail was also invented in the United Kingdom.

Additional procedures recommended for the RAM included adding additional wash water to the preweighed sample of PCC to aid in removing cement particles from the coarse aggregate; adding a defoaming agent for air-entrained PCC (2); and redetermining correction factors and equations when any changes of aggregate or quantity of aggregate were made or suspected in the PCC mixture (2).

Difficulties associated with the RAM have been electrical shorts, faulty toggle switches, sticky solenoids, leaky water valves, shortages of chemicals, and limited repair knowledge in the United States (9).

Concrete Quality Monitor

Data for the CQM were limited. The CQM, the third generation of the Kelly-Vail procedure, has been available only since 1980 (3). Most of the data were obtained through unpublished reports, test reports, and unprocessed raw data from current field projects. Field mean recovery of the CQM ranged from 91.7 percent to 106.8 percent recovery of cement content in PCC to 100.0 percent recovery at a soil-cement project, as shown in Table A-1. Field mean recovery of the CQM on water content ranged from 98.8 percent recovery to 129.5 percent recovery, as shown in Table A-2.

Field experience has shown the actual test performance took 20 min, exclusive of sampling and cleanup time. Major difficulties encountered in the field included faulty test procedure (10), undetected equipment malfunctions (10), spurious readings (10), faulty suspension tanks, stirring motors, and equipment calibration, primarily the calcium analyzer. Special procedures needed for the CQM included forced water pressure to aid in washing, refilling, and cleaning (10), and additional centrifuging steps to compensate for air-entrained PCC (2).

Nuclear Cement Content Gage

The NCCG is the property of the FHWA (4). The FHWA controls the use of the two prototype NCCG's with respect to who uses the NCCG, where it will be used, and when it can be used. Data on the field use of the NCCG were limited to field data obtained from state transportation departments in Georgia, North Dakota, Wyoming, Pennsylvania, West Virginia, and Virginia. Field mean recoveries of the NCCG ranged from 98.5 percent to 104.8 percent of cement content in PCC mixtures, as shown in Table A-1.

The NCCG requires nuclear materials licensing for the 14 millicuries of americium 241; therefore the operator must be a qualified technician, scientist, or engineer with a radiation license. The requirements are strict and limit the numbers of operators available to use the NCCG. Most data examined were performed under the general guidance and direction of the FHWA. The projects included general highway construction, bridge construction, and research. Usually

two individuals were required to perform the calibration and two to batch the concrete and lift the concrete-filled container.

Field experiences have shown that the NCCG must be recalibrated for even slight changes in the ratio of fine to coarse aggregates, and separate calibration curves and equations are required for each change in mixture proportion, aggregate types, admixtures, concrete density, and changes in ambient temperature (4).

Difficulties encountered with the prototype NCCG included major shifts in nuclear-gage readings, cables becoming loose causing erratic readings, faulty equipment, frequent recalibration due to field adjustments of the PCC mixture, inadequate significant digits in the counter, low illumination of readings from the LED digital display, and accidental changes of the preset dials and counters located on the working surface of the counter (11).

Centrifuge

The Willis-Hime centrifuge procedure was introduced by R. A. Willis and W. G. Hime in the early 1950's to determine cement content of PCC using the principle of heavy medium separation. The cement is separated from the remaining concrete materials by means of a heavy liquid whose density is between that of cement at 3.15 Mg/m^3 and that of the aggregates which typically have a maximum density of 2.84 Mg/m^3. Although introduced as a possible field test procedure, the centrifuge procedure has been used primarily as a laboratory procedure. The procedure has a reported recovery bias on tests performed in the laboratory of ±1/4 bag of cement (1 bag equals 94 lb) using the mass method and ±1/2 bag of cement using the volumetric method.

The US Army Corps of Engineers has had since 1959 a test procedure based on the Willis-Hime centrifuge procedure, CRD-C 72-59 (12). There is also in the Corps Handbook for Concrete and Cement a test method involving the Willis-Hime centrifuge procedure, CRD-C 55-83 (13). The CRD-C 55-83 procedure is a modified form of the Willis-Hime centrifuge procedure which determines the relative percentage of cement in cement mortar for the evaluation of concrete mixer performance.

X-ray Emission Spectrometer

The X-ray Emission Spectrometer procedure has been used as a laboratory technique for determining the bulk elemental analysis of an unknown material or substance. The bulky size of the X-ray unit has limited its use to the laboratory. Smaller portable X-ray units have become available that could be used in the field. No evaluation data were found for the X-ray.

Microwave Oven

The MW procedure of drying samples in determining the water content of a PCC mixture has been used extensively in the field as well as in the laboratory. Field mean recovery of the microwave procedure ranged from 94.6 percent to 99.9 percent of water content in PCC, as shown in Table A-2.

Major difficulties associated with the microwave oven included obtaining a large representative sample because of the nominal maximum size of the coarse aggregate; additional calculations due to absorbed water in the aggregate; additional calculations for the evaporable liquid in liquid admixtures; apparent decomposition of the hydrated cement particles during the test (6); and popping of aggregate particles during the heating of the sample, resulting in lost sample material.

Hot Plate

The HP procedure of determining the water content of a sample has been one of the standard test methods used on almost all field projects where moisture contents of concrete, soils, rock, and other materials were required. The hot plate is a standard piece of field laboratory equipment. No evaluation data were found.

The major difficulty reported to have been encountered in using the hot plate as a procedure in determining the water content of a freshly mixed PCC sample has been the acceleration of the hydration of the cement particles, making a portion of the water nonevaporable.

Table A-1

Detailed Field Assessment Summary for Cement Content

Test Procedure	Reference	Mean Cement Recovery, %	Standard Dev., %	No. of Samples	Recovery Range, %
CQM	(14) Beecroft & Dominick, 1982	102.7	21.5	19	84.0-129.0
	(15) Lawrence, 1983a	97.4[a]	23.9	37	
		98.7[a]	19.6	45	
		101.9[a]	18.5	48	
		94.0[a]	13.7	40	
		91.7[a]	9.3	12	
		100.0[b]	9.4	70	
	(10) Head & Phillippi, 1982	98.9	5.3	4	
		103.1	6.3	4	
	(16) Lawrence, 1983b	106.8	8.8	10	
NCCG	(11) Gulden, 1975	104.8	2.4	39	98.6-109.8
		99.2	3.7	38	91.1-108.2
		98.5	4.1	46	91.8-107.4
	(17) Howdyshell, 1976	101.9	6.4	65	87.4-117.2
RAM	(18) Wexham, 1984	99.8	2.1	3	97.8-102.0
	(19) Wexham, 1981a	97.8	1.7	2	96.7- 99.0
	(20) Costain, 1981	104.8	5.0	10	95.3-112.7
	(21) Wexham, 1981b	98.9	5.2	10	88.8-108.2
	(22) Southern, Water Authority 1981	98.9	3.8	13	89.2-105.7
	(23) Wexham, 1983	100.0	2.4	4	97.6-102.0
	(24) Roumillac, Hicks, & Mahoney, 1982	115.8	14.2	12	92.6-136.1
	(25) Bickley & Mukherjee, 1979	100.0	6.7	119	
		102.1	5.3	134	
		102.8	4.9	172	

[a] Roller-compacted concrete.
[b] Soil-cement concrete.

Table A-2

Detailed Field Assessment Summary for Water Content

Test Procedure	Reference	Mean Cement Recovery, %	Standard Dev., %	No. of Samples	Recovery Range, %
CQM	(17) Howdyshell, 1976	115.7	8.3	126	
	(10) Head & Phillippi, 1982	112.5	8.0	11	
		98.8	8.2	7	
		120.6	7.3	8	
	(15) Lawrence, 1983a	106.8	8.8	10	
	(14) Beecroft & Dominick, 1982	129.5	12.3	19	101-147
MW	(14) Beecroft & Dominick, 1982	94.6	13.8	48	65-124
	(6) Peterson & Leftwich, 1978	99.9	0.6	35	

APPENDIX B

EXPERIMENTAL DESIGN CONSIDERATIONS

SAMPLING DESIGN

During the initial phase of the NCHRP project, an experience assessment was conducted in which data pertaining to the performance of the test procedures were reviewed and tabulated. From this information, estimates of average cement content recovery and their associated variances were tabulated and used in the determination of the required sample size for each of the experiments in this project. The requirement for sample size was established as the minimum number of observations which would be needed to detect a 10 percent difference among treatment means with a confidence of 90 percent. From this investigation, it was determined that a sample size of five replicate measures would suffice.

STATISTICAL MODELS

Each of the experiments, which are defined in the Test Program as Tasks 2 through 5, is summarized in Table B-1. To evaluate the goals of each task, experiments were designed so that the hypotheses of interest could be tested using analysis of variance (ANOVA) techniques. The hypotheses of interest for each task are summarized in Table B-2. Since each task involved the use of several controlled factors which were used in the preparation of the experimental units (concrete mixtures) and testing procedures which were used in the determination of cement content or water content or both, the underlying statistical model inherent in each of the research experiments was a factorial model with five replicate measures per treatment. Table B-3 displays the relevant model for each task.

ANALYSIS OF VARIANCE

In order to determine the significance of the hypotheses of interest an ANOVA, that is, a statistical procedure which partitions total variance into known sources of variation, was computed for each task. Each of the effects was tested using Snedecor's F test, that is, the ratio of variance estimate of the factor to the experimental error variance. Significance of these effects was measured by computing the probability of obtaining a larger F-ratio. Any value of this probability, p, was judged to be significant if the value of p was smaller than 0.05.

POSTANALYSIS OF VARIANCE TECHNIQUES

The ANOVA procedure indicated that when a concrete mixture factor was found to be significant, then Tukey's w-procedure should be used to delineate the significant differences. If the significant effect was testing procedure, then Dunnett's method was used to compare all treatment averages to the absolute control of 100 percent recovery.

This, the second phase, involved the experimental design of the PCC test mixtures to be used in the evaluation of the seven test procedures. Known quantities of portland cement and water in each mixture provided the basis of each single evaluation. This testing phase provided the input data to statistically evaluate the freedom from bias of each procedure. The operators provided their input to the reliability and operator expertise needed for each procedure.

The second phase of the overall project plan was divided into five experimental design tasks as shown in Figures B-1 through B-5. Each design task involved a specific purpose in the overall evaluation of the test procedures. Specific test mixtures were proportioned for each task. Task 2 involved 18 basic PCC mixtures containing conventional concrete materials and proportioned similarly to those mixtures used in the construction industry. Task 3 evaluated the test procedures for their ability to determine cement content of a PCC mixture that was mixed an additional 15 and then 60 min in which fines generated from the calcareous aggregate and the prolonged mixing affected the estimate of cement content. Task 3 involved the eight concrete mixtures from Task 2 that were proportioned using the calcareous coarse aggregate. Task 4 involved 18 mixtures containing pulverized granulated iron blast-furnace slag or fly ash. Task 4 also involved two mixtures containing no portland cement and only fly ash or slag to determine what the effects were on the cement content determinations of the five test procedures. Task 5 involved concrete mixtures with very low water-cement ratios with the use of high-range water-reducing admixtures to determine the ability of the three test procedures for water content determination. Also in Task 5 was a mixture containing a melamine-based admixture to evaluate chloride-sensitive test procedures. Task 6 involved the use of calcium chloride as an accelerating admixture in the concrete mixtures to determine what effects it had on the five cement content determination procedures.

Statistical determinations dictated five replicate samples be tested to meet the 90 percent confidence level needed to critically evaluate the seven test procedures. The five replications involved 61 unique concrete mixtures totaling 305 individual tests.

Several factors dictated the number of replications to be performed per day. Among the factors were mixing time, cleanup time, personnel schedules, and mixer availability, and most important was the testing time which, for the centrifuge procedure, was as much as 2 hr.

Allotments were provided for each factor, which eventually limited the number of tests per normal workday to four.

Mixing time included weighing the materials, batching the materials, mixing the concrete, performing physical properties tests on each batch of concrete, and cleaning up the mixer between batches. Materials were usually weighed during the preceding procedure evaluation except for the first batch of the day. The first batch of the day involved blending the mass of fine aggregate and mass of coarse aggregate to be used during the course of the day; this provided for a more constant moisture control of the aggregate. The first batch also involved a quick determination of aggregate moisture contents for batch weight adjustments.

Batching the materials involved the standard mixing sequence and timing for the materials as they were introduced into the mixer. ASTM Test Method C 192 (26) was followed as guidance for mixing and batching the PCC. The ASTM mixing procedure was followed to ensure that the batch-to-batch variation was minimized.

The concrete was discharged from the mixer into a sample pan and was remixed by use of hand tools to correct segregation of the mixture caused by discharge. Tests including slump (27), air content (28), and unit weight (29) were performed on each batch prior to releasing the batch to the operators. These tests provided assurance that the batches of concrete were proper replicates of the initial batch and representative of the intended concrete mixture. Following the release of the batch to the operators, the mixer was cleaned if necessary and buttered with a small volume of concrete similar to the next batch.

Cleanup time included the proper thorough cleaning of all instruments to prevent possible malfunctions and hardening of concrete on the instruments. The proper disposition of waste materials accumulated during the day was important with the RAM, CQM, X-ray, MW, HP, and especially the CF procedure. Materials such as aggregates, hardened concrete, wash water, the sample of concrete, and in some cases chemicals were disposed of in an approved manner.

Personnel schedules took into account the time allocated for lunch and also for the times alternate operators needed to familiarize themselves with the procedure before starting. Alternate operators were used sparingly. Primary operators were used as much as possible; however, when a primary operator was absent (for whatever reason) an alternate was notified immediately and the alternate performed the evaluation until the primary operator returned. Continuity and smoothness in the evaluation of the procedures were regarded as important. Alternate operators were as efficient and confident in their testing as were the primary operators.

Mixer availability involved alternately scheduling the mixing equipment with the other research projects requiring concrete. More than 20 project engineers and project managers were scheduling work either directly or indirectly associated with proportioning and making PCC.

Testing time was the time needed to perform one series of evaluations. Although actual testing varied with each procedure, test time was controlled by the procedure requiring the most time to sample, test, clean up, and prepare for the next sample, which for this project was the CF procedure. The CF procedure required a drying time of almost 1 hr and required 1 hr 40 min to as much as 2 hr to complete one cycle; that is, time from receiving one sample until the next sample was ready.

Testing time was actually the governing factor on how many series of evaluations could be performed in a normal workday. The result was four series of evaluations per day.

All tests and evaluations were performed according to the manufacturer's specifications and requirements set for the seven procedures. Each operator obtained a representative sample of each batch of concrete in accordance with ASTM Test Method C 172 (30). They performed each procedure accordingly and accumulated data on a daily basis. The data calculations were checked and entered into computer storage. The accumulation of data was held in the computer until the completion of each task at which time the data were sent to the project statistician for analysis.

Table B-1

NCHRP Research Tasks

Task	Description
2	Basic Concrete Mixture Evaluation
3	The Effects of Mixing
4	The Effects of Fly Ash and Pulverized Iron Blast-Furnace Slag
5	The Effects of High-Range Water-Reducing Admixture
6	The Effects of An Accelerator

Table B-2

NCHRP Statistical Hypotheses

Task	Hypotheses
2	a. Percent recovery of cement content is independent of the actual cement content used in mixture preparation.
	b. Percent recovery of cement content is independent of the aggregate ratio used in mixture preparation.
	c. Percent recovery of cement content is independent of the method used to determine cement content.
	d. Percent water recovery is independent of testing method.
3	a. Simulated prolonged mixing has no effect on percent recovery of cement content.
	b. Percent recovery of cement content subjected to prolonged mixing has no effect on a testing method's ability to determine percent recovery.
4	a. The use of fly ash has no effect on percent recovery of cement content.
	b. The use of pulverized iron blast-furnace slag has no effect on percent recovery of cement content.
	c. Percent recovery of cement content is independent of the method used to determine cement content and the amount of cementitious material.
5	a. The use of a high-range water-reducing admixture has no effect on percent water recovery.
	b. Percent water recovery is independent of the method used to determine percent water recovery and admixture.
6	a. The use of calcium chloride has no effect on percent recovery of cement content.
	b. Percent recovery of cement content is independent of the method used to determine cement content and the amount of calcium chloride admixture.

Table B-3

NCHRP Statistical Models

Task	Mathematical Model
2	Percent recovered = K + R + C + RC + M + MR + MC + MRC + e
3	Percent recovered = K + C + T + CT + M + MC + MT + MCT + e
4	Percent recovered = K + C + A + CA + M + MC + MA + MCA + e
5	Percent recovered = K + A + M + MA + e
6	Percent recovered = K + C + A + CA + M + MC + MA + MCA + e

Where:

K = Population mean of percent recovery
R = Aggregate ratio
C = Cement content
M = Test procedure
T = Simulated mixing time
A = Admixture
  Task 4 = Fly Ash or Pulverized Slag
  Task 5 = Naphthalene or Melamine
  Task 6 = Calcium Chloride
e = Experimental Error

Combination of letters: Interaction terms among factor combinations

Instrument/ Procedure	CQM	RAM	NCCG	CF	X-ray	MW	HP
Aggregate Type	Calcareous	Siliceous	Blended				
Aggregate Proportion, fine/coarse	I 35/65	II 50/50					
Cement Content, lb/cu yd	350	550	800				
Batches	A	B	C	D	E		
Replicate	1						

Figure B-1. Experimental design of Task 2, 18 mixtures, 90 batches of concrete

Instrument/ Procedure	CQM	RAM	NCCG	CF	X-ray
Aggregate Type	Calcareous	Blended			
Aggregate Proportion	I				
Simulated Degradation Fines/time	15 min	60 min			
Cement Content, lb/cu yd	350	800			
Batches	A	B	C	D	E
Replicate	1				

Figure B-2. Experimental design of Task 3, 8 mixtures, 40 batches of concrete

Instrument/ Procedure	CQM, RAM, NCCG, CF, X-RAY
Aggregate Type	Calcareous, Siliceous
Aggregate Proportion	II
Percentage	15, 40, 100	15, 40, 100
Cementitious Material	Pulverized Slag, Fly Ash
Cementitious Content, lb/cu yd	550, 800
Batches	A, B, C, D, E
Replicate	1

Figure B-3. Experimental design of Task 4, 18 mixtures, 90 batches of concrete (includes only 10 batches with 100 percent replacement)

Instrument/ Procedure	CQM, MW, HP
Aggregate Type	Calcareous, Siliceous
Aggregate Proportion	II
Dosage of HRWRA	Low/ 0.5%, Medium/ 2%, High/ 3%, Double Dose/5%
Type of HRWRA	Naphthalene, Melamine*
Cement Content, lb/cu yd	800
Batches	A, B, C, D, E
Replicate	1

Figure B-4. Experimental Design, Task 5, 9 mixtures, 45 batches of concrete

* Includes only five additional batches.

Instrument/ Procedure	CQM	RAM	NCCG	CF	X-ray
Aggregate Type	Calcareous	Siliceous			
Aggregate Proportion	II				
Calcium Chloride	1%	2%			
Cement Content, lb/cu yd	550	800			
Batches	A	B	C	D	E
Replicate					1

Figure B-5. Experimental design, Task 6, 8 mixtures, 40 batches of concrete

APPENDIX C

CONCRETE MATERIALS AND MIXTURES

GENERAL

The assessment of seven procedures required that a series of different PCC mixtures be used in the evaluations. The test procedures had to be evaluated with mixtures containing a variety of materials available for use in PCC. The materials included portland cement, water, siliceous aggregate, calcareous aggregate, pozzolan, granulated iron blast-furnace slag, high-range water-reducing admixtures, and an accelerating admixture.

MATERIALS

The portland cement was Type I from the Dundee Cement Company in Clarksville, MO. The mixing water was tap water supplied by the City of Vicksburg Water Treatment Plant in Vicksburg, MS. The siliceous fine aggregate was concrete sand from the Monroe Sand and Gravel Company in Monroe, LA. The siliceous coarse aggregate was 1-in. nominal maximum size aggregate from the Lewis Miller Construction Company in Vicksburg, MS. Both the calcareous fine and coarse aggregates were crushed limestone from Vulcan Materials Incorporated in Calera, AL, but purchased through the Lewis Miller Construction Company in Vicksburg. The calcareous coarse aggregate was also 1-in. nominal maximum size aggregate. The pozzolan was a Class F fly ash from Trinity Materials Incorporated, Purvis, MS. The ground granulated iron blast-furnace slag was from the Atlantic Cement Company, Sparrows Point, MD. The first of two HRWRA's was WRDA-19, a sulphonated naphthalene formaldehyde condensate from W. R. Grace and Company, Cambridge, MA. The second HRWRA was Melment, a sulphonated melamine formaldehyde condensate from American Admixture and Chemical Company, Chicago, IL. The accelerating admixture was calcium chloride from Dow Chemical USA, Midland, MI.

MIXTURES

The PCC mixtures included varying amounts of portland cement, aggregates, fly ash, slag, HRWRA's, calcium chloride, and calcareous fines. The cementitious material contents were the volumetric equivalent of 350, 550, and 800 lb/cu yd of portland cement. The aggregate types included all siliceous, all calcareous, and a blend of siliceous fine aggregate and calcareous coarse aggregate. The ratios of fine to coarse aggregate were 35 to 65 percent and 50 to 50 percent. Both the fly ash and slag were used to make up 15 and 40 percent by volume of the cementitious material. Mixtures containing 100 percent fly ash or slag, no portland cement, were also used. The dosages of HRWRA were: low, 0.5 percent; medium, 2 percent; high, 3 percent; and a very high dose at 5 percent by mass of cementitious material. The amounts of calcium chloride were 1 and 2 percent by mass of cementitious material, calculated as anhydrous calcium chloride. Also included was a combination of mixtures for a simulation of prolonged mixing of a calcareous aggregate mixture for 15 and 60 min by adding crushed calcareous fines.

Sixty-one different PCC mixtures were proportioned and refined into conventional usable concrete mixtures as shown in Table C-1. As in all proportioning of concrete mixtures, some refinements are needed in the laboratory to locate and make minor adjustments to produce a workable and usable concrete mixture. Table C-2 represents the 61 mixtures used in the evaluations.

Table C-1

PCC Mixture Proportioning Summary

Mixture	Cement Content			Aggregate[a]			Ratio		Mixing Time min		percent: Slag			percent: Fly Ash			percent: Naphthalene				percent: Melamine	percent: Calcium chloride		Instrument	
	350	550	800	S	C	B	35/65	50/50	15	60	15	40	100	15	40	100	0.5	2	3	5	3	1	2	Cement	Water
2-1	X			X			X																	X	X
2-2	X			X				X																X	X
2-3		X		X			X																	X	X
2-4		X		X				X																X	X
2-5			X	X			X																	X	X
2-6			X	X				X																X	X
2-7	X					X	X																	X	X
2-8	X					X		X																X	X
2-9		X				X	X																	X	X
2-10		X				X		X																X	X
2-11			X			X	X																	X	X
2-12			X			X		X																X	X
2-13	X				X		X																	X	X
2-14	S				X			X																X	X
2-15		X			X		X																	X	X
2-16		X			X			X																X	X
2-17			X		X		X																	X	X
2-18			X		X			X																X	X
3-1	X					X	X		X															X	
3-2	X					X	X			X														X	
3-3			X			X	X		X															X	
3-4			X			X	X			X														X	
3-5	X				X		X		X															X	
3-6	X				X		X			X														X	
3-7			X		X		X		X															X	
3-8			X		X		X			X														X	
4-1		X		X				X			X													X	
4-2		X		X				X				X												X	
4-3		X		X				X					X											X	
4-4			X	X				X			X													X	
4-5			X	X				X				X												X	

[a] S - siliceous, C - calcareous, B - blend.

Table C-1 (Concluded)

	Cement Content			Aggregate			Ratio		Mixing Time min		percent: Slag			Fly Ash			Naphthalene				Melamine	Calcium chloride		Instrument	
Mixture	350	550	800	S	C	B	35/65	50/50	15	60	15	40	100	15	40	100	0.5	2	3	5	3	1	2	Cement	Water
4-6		X			X			X			X													X	
4-7		X			X			X				X												X	
4-8			X		X			X			X													X	
4-9			X		X			X				X												X	
4-10		X		X				X						X										X	
4-11		X		X				X							X									X	
4-12		X		X				X								X								X	
4-13			X	X				X						X										X	
4-14			X	X				X							X									X	
4-15		X			X			X						X										X	
4-16		X			X			X							X									X	
4-17			X		X			X						X										X	
4-18			X		X			X							X									X	
5-1			X	X				X									X								X
5-2			X	X				X										X							X
5-3			X	X				X											X						X
5-4			X	X				X												X					X
5-5			X		X			X									X								X
5-6			X		X			X										X							X
5-7			X		X			X											X						X
5-8			X		X			X												X					X
5-9			X	X				X													X				X
6-1		X		X				X														X		X	
6-2		X		X				X															X		
6-3			X	X				X														X		X	
6-4			X	X				X															X	X	
6-5		X			X			X														X		X	
6-6		X			X			X															X	X	
6-7			X		X			X														X		X	
6-8			X		X			X															X	X	

Table C-2

NCHRP Mixture Proportions

Mixture No.	Cement Content lb	Water Content lb	Fine Aggregate lb	Coarse Aggregate lb	
2-1	350	280	1,171	2,116	
2-2	350	322	1,618	1,575	
2-3	550	286	1,107	2,002	
2-4	550	319	1,539	1,498	
2-5	800	320	1,003	1,813	
2-6	800	320	1,434	1,396	
2-7	350	280	1,172	2,317	
2-8	350	322	1,619	1,724	
2-9	550	286	1,108	2,191	
2-10	550	303	1,561	1,662	
2-11	800	320	1,004	1,985	
2-12	800	304	1,455	1,549	
2-13	350	280	1,239	2,317	
2-14	350	280	1,769	1,782	
2-15	550	286	1,171	2,191	
2-16	550	286	1,673	1,685	
2-17	800	270	1,110	2,076	
2-18	800	320	1,516	1,527	
					Fines Added, lb
3-1	350	280	1,172	2,308	9.3
3-2	350	280	1,172	2,285	32.4
3-3	800	320	1,004	1,977	7.9
3-4	800	320	1,004	1,958	27.8
3-5	350	280	1,219	2,306	27.9
3-6	350	280	1,161	2,283	109.2
3-7	800	320	1,045	1,976	23.8
3-8	800	320	995	1,956	93.6
					Slag Added, lb
4-1	467.4	319	1,539	1,498	74.4
4-2	329.9	319	1,539	1,498	198.3
4-3	-0-	319	1,539	1,498	495.7
4-4	680.0	320	1,433	1,395	108.1
4-5	480.0	320	1,433	1,395	288.3
4-6	467.4	282	1,678	1,691	74.3
4-7	329.9	282	1,678	1,691	198.3
4-8	680.0	315	1,522	1,533	108.1
4-9	480.0	315	1,522	1,533	288.3

(Continued)

Table C-2 (Concluded)

Mixture No.	Cement Content lb	Water Content lb	Fine Aggregate lb	Coarse Aggregate lb	
					Fly Ash Added, lb
4-10	467.4	319	1,540	1,498	59.0
4-11	330.0	319	1,540	1,498	157.2
4-12	-0-	319	1,540	1,498	392.9
4-13	680.0	320	1,434	1,396	85.7
4-14	480.0	320	1,434	1,396	228.5
4-15	467.4	286	1,673	1,685	59.0
4-16	330.0	286	1,673	1,685	157.2
4-17	680.0	320	1,515	1,526	85.7
4-18	480.0	320	1,515	1,526	228.5
					HRWRA Added, lb
5-1	800	240	1,539	1,498	4.0
5-2	800	232	1,549	1,508	16.0
5-3	800	215	1,572	1,530	24.0
5-4	800	200	1,591	1,549	40.0
5-5	800	248	1,616	1,627	4.0
5-6	800	240	1,627	1,638	16.0
5-7	800	216	1,660	1,672	24.0
5-8	800	200	1,682	1,694	40.0
5-9	800	215	1,572	1,530	24.0
					Calcium Chloride Added, lb
6-1	550	319	1,539	1,498	5.5
6-2	550	319	1,539	1,498	11.0
6-3	800	320	1,434	1,396	8.0
6-4	800	320	1,434	1,396	16.0
6-5	550	286	1,673	1,685	5.5
6-6	550	286	1,673	1,685	11.0
6-7	800	320	1,516	1,527	8.0
6-8	800	320	1,516	1,527	16.0

APPENDIX D

EQUIPMENT AND TEST PROCEDURES

The WES had equipment to perform five of the seven test procedures. These included the RAM, X-ray, CF, MW, and the HP. The CQM was borrowed from the CERL in Champaign, IL. The NCCG was on loan from the Office of Research, FHWA in McLean, VA.

The WES has used the five test procedures on numerous research projects dealing with the analysis of fresh concrete, hardened concrete, and concrete materials. The WES has also been involved in research projects and programs pertaining to the use of the CQM and the NCCG. The CQM along with the RAM has been under evaluation by the US Army Corps of Engineers for several years dating back to the mid 1970's when the original Kelly-Vail and RAM were introduced in the United States. The Corps of Engineers, primarily at CERL and WES, have sought to standardize these systems into normal everyday testing and evaluation tools. A segment of WES's Concrete Technology Training Course introduces the CQM and the RAM to concrete technologists, engineers, scientists, laboratory supervisors, and senior technicians throughout the Corps of Engineers. Demonstrations of both procedures have been given during these courses by CERL and WES personnel.

The NCCG was evaluated by the Louisiana Department of Transporation (31) with cold temperature test specimens evaluated at the WES. Test specimens were from PCC that was evaluated with the NCCG in the cold temperature facilities at WES. All equipment and materials were placed in the cold room and batched out of a 1.5-cu-ft mixer and then evaluated by the NCCG while still at 40° F.

Therefore, it can be said that the WES has experience with all of the procedures involved in this evaluation.

RAPID ANALYSIS MACHINE

The RAM was a fully self-contained testing apparatus, requiring only a source of electricity and water to perform a single test. The theory behind the RAM was that water would be displaced with cement to determine cement content. The procedure involved obtaining an 8-kg sample of concrete, washing the cement from the aggregate through Stokes' law of settling, flocculating the cement particles, and weighing a fixed volume of cement and water. The RAM used calibration graphs or equations or both as do all the other test procedures.

The RAM uses a fully automatic system between introducing the sample into the RAM and weighing the finished product. A 6-min timer is used to direct the sequence of events taking place during the test. After the 8-kg sample of PCC is introduced into the elutriation column, a single button is pushed to activate the timer. The sample is washed by a high volume and high velocity of water, separating the cement from the aggregate. The velocity is such that Stokes' law of particle settling causes the very small cement and cement size particles to be carried upward away from the larger aggregate particles that remain stationary or sink. The cement is carried to a sampler which directs a 10 percent sample to a vibrating 150-μm sieve through to a conditioning vessel where two flocculating agents are added, a stirrer agitates the solution, and a siphon removes the excess water to a standardized volume. There are several stagnant periods throughout that enhance chemical reaction as well as physical reaction of the solution. The final waiting period allows all the heavy cement particles to fall through the solution into a constant-volume vessel (CVV) for weighing. Two predetermined calibration curves are used to determine the total amount of solids in the CVV and then the amount of silt or noncement materials trapped with the solids. The curves are a CVV calibration curve and a silt correction curve. The calibration curve is developed from a series of PCC mixtures identical to the PCC mixtures to be evaluated at the project site, except (1) the cement content is varied from 0 through 750 to 1500 g for an 8-kg mixture, and (2) the aggregate, both fine and coarse, is washed free of any and all silt particles prior to mixing and testing. The calibration curve is drawn as a straight line with the weight of the CVV as the ordinate and cement content as the abscissa. A statistical least-squares linear regression curve can also be used to determine the cement content. The calibration line does not change from mixture to mixture; however, the silt correction changes as aggregate types and aggregate ratios are changed. The silt correction is developed from mixtures containing no cement; only the aggregates for each PCC mixture in exact ratios are evaluated for their silt content. Pozzolan and other cementitious materials are usually considered as silt. Each unique PCC mixture must have a separate silt correction curve if and only if the aggregate is adjusted or changed.

The total cement content per unit volume of PCC is calculated from the calibration curve as cement content minus the silt, divided by the initial weight of the PCC sample, times the plastic PCC density.

X-RAY EMISSION SPECTROMETER

The X-ray used was a General Electric/Diano/Bausch and Lomb XRD-6 unit upgraded to use a XRD-700 detector and generator. The X-ray procedure has been used to determine the bulk elemental analysis of unknown

materials. The X-ray has been used in the research and testing of cements, aggregates, soils, concretes, and other known and unknown materials. This unit was a laboratory device because of its size and capabilities. Smaller portable X-ray units have become available with sufficient capacity and capabilities to perform the analysis of PCC samples in the field.

The X-ray procedure involved irradiating a sample of PCC with gamma rays causing it to fluoresce, generating secondary X-ray characteristics of the chemical elements present. Radiation produced by one or another of three elements common to portland-cement--silicon, calcium, and sulfur--was used for the analysis. The X-ray unit measured the amount of secondary X-ray emission by the individual element thus indicating the relative amount of that element present in the sample tested. The element or elements selected for each analysis were based on the elemental composition of the aggregate used in the concrete mixture. For instance, if a siliceous aggregate was used in the PCC, then the element to detect was calcium because the silicon in the siliceous aggregate was detected as well as the silicon in the cement. When the calcareous aggregate was used, silicon was detected because of the calcium in the aggregate. However, with the blend of siliceous fine aggregate and calcareous coarse aggregate in the PCC, the emission by sulfur was detected and the amount determined. Sulfur was not used throughout because of its limited amount in portland cement. The higher the percentage of an element in the cement, the more reliable and free of bias the results will be.

Calibration involved making a set of standard curves from known cement content mixtures. This step included making synthetic mixtures with known amounts of cement, fly ash, or granulated blast-furnace slag. Three synthetic mixtures were used for each material. One mixture was a pure sample of the material and two with different amounts, which generated a series of linear curves. The three points were established by emitting Kα (39) lines into each of the mixtures and determining the amounts of silicon, calcium, and sulfur for each mixture. These standard curves thus become useful for all types of PCC.

The X-ray procedure now becomes useful in the determination of cement content in both known and unknown concretes as long as the X-ray pulse count is recorded and used to determine what percent of that material was present in the sample. The procedure involved taking a 300- to 350-mL sample and drying to a constant weight. The dried concrete and sieve is then broken up over No. 4, 50, and 100 sieves to remove the large aggregate. The sample containing the mortar was ground with 1.5 g taken to make a test pellet for the X-ray emission spectrometer. The X-ray peaks for silicon, sulfur, and calcium at 30 sec are recorded and plotted on the calibration curves to determine cement content.

CENTRIFUGE

The CF used was a Sargent International Centrifuge, Size 1, Model CM, a standard piece of laboratory equipment. Its primary use has been the determination of relative cement contents in PCC. In the evaluation of mixer efficiency for determining the adequacy of mixing of a batch of concrete, the relative cement content was determined as a percentage of the mortar in the concrete rather than a true cement content of concrete. However, with the incorporation of the Willis-Hime centrifuge procedure for freshly mixed concrete, the centrifuge was used in a modified combined procedure.

The WES CF procedure is described in CRD-C 55-83 (13). The Willis-Hime CF procedure is the basis of CRD-C 72-59 (12). Most of the chemicals used in CRD-C 72-59 have been banned by the EPA and the Occupational Safety and Health Administration (OSHA), and some of the other chemicals were no longer available from manufacturers. Therefore, for NCHRP Project 10-25, the co-principal investigators decided to incorporate the viable phases of both CRD-C 55-83 and CRD-C 72-59 into a modified CF procedure.

The theory behind the CF procedure was separation by density of the materials in the PCC. The procedure involved removing the coarser aggregates, flocculating the cement and very fine aggregate, drying the sample, separating the cement whose density was 3.15 Mg/m^3 from the remaining materials whose maximum density was 2.84 Mg/m^3 by a heavy medium whose density was 2.95 Mg/m^3, and determining the amount of cement in the sample.

The CF calibration charts were established with small batches of PCC encompassing mixture with cement factors ranging from 350 to 800 lb/cu yd. Each individual mixture was batched three times to establish an average point for cement recovered. The graph has ordinates and abscissas of pounds per cubic yard of cement and grams of cement recovered, respectively. The calibration curve becomes a linear line which relates the cement factor to the amount of cement recovered.

The procedure involves taking a 2-kg concrete sample and introducing it to a 2,500-mL solution of 1 g Separan NP10, 2 mL Calsoft L-40, and water. The sample was washed in the solution using a No. 16 sieve basket to remove large aggregate. The liquid was removed by decantation and heating to a constant weight. A 20-g sample was taken and poured into a centrifuge tube, followed by tetrabromoethane up to a 40-mL volume. The tube was spun for 3-, 4-, and 4-min intervals at relative centrifugal forces (RCF) of 190, 525, and 525 with tubes rotated 180 deg between intervals. After settling for 10 min, the material that remained floating was added to 75 cc of acetone, stirred, and settled for 40 sec. The rinse was repeated twice more with only 50 cc of acetone and 20 sec settling time. Following a drying period to constant weight, the sample weight is plotted on the calibration curve of calculated cement content against actual cement content.

The CF procedure in its present form has two major disadvantages: (1) toxic chemicals are required for testing, and (2) the testing time does not meet the needs of a rapid test procedure. The chemical tetrabromoethane, the separating medium, and the solvent acetone require special handling and attention such as high volume exhaust hoods, rubber gloves, rubber aprons, face and skin protection, as well as additional surveillance of the operator by other laboratory personnel. Testing time generally was between 1-1/2 hr to as much as 2 hr for each individual test.

MICROWAVE

The MW procedure used a standard 625-w domestic microwave oven, the Sears Kenmore Model 566. Several types of microwave ovens have been previously used at WES for this testing. Microwave ovens were first used at the WES in the determination of water content of freshly mixed concrete in 1972 (32). Microwave testing has been used for moisture content of soils and aggregates as well as other materials containing appreciable amounts of liquids and water.

The theory of the microwave oven was production of high-frequency microwaves and the absorption of the microwaves by water molecules. As the microwaves were generated, they were transmitted to the sample containing the water molecules. The water molecules absorb the microwaves, thus exciting themselves into generating heat from their movement in the excited state. Therefore, as more heat was generated, more water molecules were being removed until the sample became dry. The heat from the excited water molecules was internally generated heat which did not affect the hydration process of the cement particle as much as externally generated heat would have. The microwave was supplied with scales, sample dishes, and stirrers for the operators. The area was adjacent to the hot plate test area. Testing procedure included obtaining 500-g PCC samples weighed initially and dried to a constant weight. Calculations included initial weight minus dry weight, division by the dry weight, and multipliction by 100; however, aggregate moisture corrections were also applied to obtain total mixing water.

The literature recommended operation of the microwave oven in the defrost mode to avoid an apparent melting of the cement particles which would increase the evaporated water loss greater than the known weight of the water in the sample. The defrost mode in the microwave oven was 30 percent of full power; however, the MW procedure was performed at 50 percent of full power for this project.

HOT PLATE

Hot plates have been standard laboratory and field equipment for many years. Hot plates have been used for determining surface moisture of fine and coarse aggregates for correcting the concrete material batch weights at batch plants as specified in ASTM C 566-78 (33). They have been used as standard laboratory equipment in PCC, soils, and asphaltic concrete testing laboratories.

The hot plates used on the NCHRP Project 10-25 research were two standard 1,100-w solid coil element hot plates. The hot plate procedure required an initial sample weight, drying of the PCC sample on the hot plate, determination of the final constant weight, correction for aggregate absorption, and calculation of the water content. Loss of moisture was through evaporation.

CONCRETE QUALITY MONITOR

The CQM has been assembled and demonstrated at the WES in conjunction with the US Army Corps of Engineers Training Session on Concrete Technology, in which procedures for the rapid analysis of concrete have been presented. CERL has been the most recent innovator of the CQM. CERL engineers initiated research on a new test procedure developed by R. T. Kelly and J. W. Vail (34,35). CERL made several modifications in 1977 to upgrade, improve, and increase the ruggedness of the Kelly-Vail method, which was renamed the CERL/Kelly-Vail method. The most significant change that occurred was the replacement of a flame photometer with a titration procedure using an EDTA solution. These improvements were used until 1981 when CERL again sought to change its version of the Kelly-Vail Method into a simpler, quicker, and more field worthy unit by introducing electronic analyzers into the system. This third-generation system is called the Concrete Quality Monitor (CQM).

The CQM procedure was actually two test methods. One method determined cement content and the other determined water content. The cement content method involved calibration of the equipment for the calcium content of the job cement and mixing water. The calibration curve is a linear curve plotted with cement content versus calcium analyzer reading. Following calibration, which was repeated weekly or as cement, aggregate, or water changed whichever occurred first, the procedure involved a 2-kg concrete sample placed into a 37.6-L volume of water through a nest of No. 4, 50, and 100 sieves. Following a 3- to 4-min washing from a recirculating water pump, a 30-mL sample was taken and mixed with 30-ml of 5 percent nitric acid; then 250 mL of water was added and stirred. A 20-μL sample was taken and analyzed in the electronic calcium analyzer with the reading plotted on the calibration curve to determine cement content of the PCC mixture.

The water content method included obtaining two 2-kg samples of concrete and adding 250 mL of 0.5 normal sodium chloride solution to one sample and 250 mL of distilled water to the other sample. Following 2 min of mixing, slurries from the two samples were removed and placed into centrifuge tubes and spun for 3 to 4 min at 2,000 to 3,000 rpm. Chloride strengths of both slurry samples were determined from 100 μL of sample. Water content is calculated from the formula:

$$\text{Water Content (mL)} = 250 \frac{\text{std}}{S - B \frac{S\ wt}{B\ wt}} - 1$$

where

std = chloride strength of sodium chloride solution, (0.5N)

S = chloride strength of sample, milliequivalents/L

B = chloride strength of blank, milliequivalents/L

S wt = sample weight, g

B wt = blank weight, g

The CQM was loaned by CERL for the duration of the NCHRP Project 10-25. There were several CQM's in use in the United States, yet the CQM has not been packaged, marketed, or sold in units as a testing device; rather, CERL has introduced the CQM as a test procedure with commercially available equipment recommended, most of which were standard laboratory equipment but not field laboratory equipment. ASTM Committee C 9 on Concrete and Concrete Aggregate is considering standardizing the CQM. Problems associated with the setup and operation of the CQM were a leaky washing container, faulty stirring motor, shorted calcium analyzer, shortage of chemicals, and very short shelf life of the indicator chemical.

NUCLEAR CEMENT CONTENT GAGE

The NCCG was one of two prototype gages introduced by the Department of Transportation, FHWA, Office of Research and Development, in 1975 for the purpose of rapidly, simply, and accurately measuring the cement content of PCC. T. M. Mitchell (36,37,38) has been the leading collaborator in the design of nuclear gages for cement content determinations dating back to 1973. The FHWA and the Atomic Energy

Commission initiated basic research and a prototype in 1968 that eventually led to the NCCG.

The theory behind the NCCG was a backscatter and absorption of low-energy gamma rays. The gamma rays were transmitted from a 14-millicurie (5.2E8 B_c) americium 241 source into a sample of PCC that attenuated the gamma rays through the photoelectric absorption process. The calcium in the portland cement absorbed much of the gamma rays as they passed through the sample. Those gamma rays that were not absorbed by the calcium were scattered back toward the center of the sample and counted by a sodium-iodide scintillation crystal detector. The amount of radiation backscattered, detected, and counted was inversely proportional to the cement content in the freshly mixed PCC. Calcium in the aggregates and other constituents was compensated for through the use of calibration graphs and equations.

The calibration involved an extensive process of developing and mixing three PCC mixture that bracket each individual target cement factor. For example, if the target was 500 lb/cu yd, then the three calibration cement factors were 500 ± 94 lb/cu yd or 406, 500, and 594 lb/cu yd. For each calibration mixture laboratory control of batching was essential. Each mixture was proportioned to 2.5-cu-ft batches and evaluated by taking six samples from each and determining the 18 data points and the least-squares fit curve or equation.

The actual procedure involved establishing a standard count on a standard polymer-impregnated concrete specimen. The concrete was placed into the sample bucket following the standard procedure for filling a unit weight bucket. The probe containing the radiation sources, a crystal detector, and a photomultiplier are inserted into a plastic spacer preplaced in the concrete. The counter is started and for 20 sec at each of three depths the backscatter records the amount of radiation detected, which is inversely proportional to the cement content as recorded on the calibration curve.

The NCCG had a special requirement unlike the other procedures in that due to the americium 241 source, a license from the Nuclear Regulatory Commission had to be obtained prior to the gage being shipped to the WES. Operators wore badge indicators that recorded the amount of radiation the body was subjected to from the source.

APPENDIX E

OPERATORS

One primary operator was appointed to each test procedure. The operators were responsible for performing and evaluating their respective test procedure. An operator was assigned to a procedure based upon knowledge of the equipment and procedure, knowledge of similar equipment and procedures, experience, and educational background. The basic idea was to have someone operating the instrument capable of realizing and recommending sound engineering judgment on procedural deficiencies and discrepancies, possible problems, mechanical deficiencies and discrepancies, improvements, changes, and repairs when needed.

Because of several external constraints, procedures were combined and performed by single primary operators. The CQM was designated to be two individual test procedures, a cement test and a water test, which warranted two primary operators; yet reports indicated the CQM cement test and the CQM water test have always been performed by a single operator both in the field and in the laboratory. Two operators were used to inprove performance, however. The microwave and hot plate procedures were so similar to each other as well as being simple to perform that the two procedures were performed by a single primary operator.

The length of the actual testing and evaluation was scheduled to be more than 6 months of routine testing. In addition to the routine testing, the equipment had to be set up and calibrated by the operators, which required time and effort of five primary operators continuously for more than 7 months. To alleviate problems of scheduling with numerous other major projects, a minimum of two alternate operators were assigned to each primary operator. The alternate operators were in some cases more experienced with the instrument and procedures than were the primary operators but having prior commitments were listed as alternates. However, the primary operators proved to be an asset in quickly learning and performing the procedures with great efficiency and providing valuable input to the project. The number of primary operators was increased, greatly benefiting the project.

APPENDIX F

TASK 2: BASIC CONCRETE MIXTURE EVALUATION

INTRODUCTION

In order to determine how well the CF, CQM, NCCG, RAM, and X-ray test procedures estimated the cement content, concrete mixtures were produced using three aggregate types (calcareous, siliceous, and blended) with two fine-to-coarse aggregate ratios (35/65 and 50/50) and three cement factors (350, 550, and 800 lb/cu yd). Each mixture was replicated five times, and cement content expressed as percent recovery was determined by each of the five test procedures. A three-factor factorial ANOVA was used to judge significant differences among the factors for mixtures using each of the types of aggregate. In conjunction with this experiment, percent water recovery for each of the replicated mixtures was determined by the HP, MW, and CQM test procedure.

CALCAREOUS AGGREGATE MIXTURES: MAIN EFFECTS

The hypotheses listed in Table B-2 proved to be significant with regard to percent recovery of cement for each main effect. The significant cement content effect ($F = 26.92$, $p = 0.0001$) exhibited average recoveries of 111.59, 104.41, and 98.49 percent for the 350-, 550-, and 800-lb/cu yd mixtures, respectively. Tukey's w-procedure (8) indicated that the 350-lb/cu yd mixtures exhibited a significantly higher average percent recovery than the 550- and 800-lb/cu yd mixtures which were also significantly different. The significant aggregate ratio effect ($F = 12.00$, $p = 0.0007$) exhibited average recoveries of 107.36 and 102.30 percent for the 35/65 and 50/50 aggregate ratio mixtures, respectively. In contrasting these effects, the 35/65 aggregate ratio mixtures exhibited a significantly higher average percent recovery than the 50/50 aggregate ratio mixtures. The significant test procedure effect

(F = 17.84, p = 0.0001) exhibited average percent recoveries of 112.45 for X-ray, 107.77 for CQM, 106.71 for NCCG, 103.23 for RAM, and 94.00 for CF. Dunnett's procedure (8) indicated that RAM was the only test procedure which was not significantly different from the control of 100 percent recovery. However, since the interaction of cement content by test procedure is also significant, further investigation is warranted.

CALCAREOUS AGGREGATE MIXTURES:
INTERACTION EFFECTS

The only significant interaction effect was cement content by test procedures (F = 2.12, p = 0.0390). The average percent recovery of cement for this significant effect is displayed in Table F-1. As can be observed from this table, the test procedures which were not significantly different from 100 percent recovery were CF for the 350-lb/cu yd mixtures; CF, CQM, and RAM for the 550-lb/cu yd mixtures; and CQM, NCCG, RAM, and X-ray for the 800-lb/cu yd mixtures. The nonsignificant interaction effects of aggregate ratio by test procedures (F = 1.79, p = 0.1346) and cement content by aggregate ratio by test procedures (F = 1.75, p = 0.0946) average percent recoveries are summarized in Tables F-2 and F-3, respectively. Dunnett's comparison procedure is not appropriate for these effects since they are nonsignificant; hence, conclusions drawn from Table F-1 are valid across other factor combinations.

Tables F-2 and F-3 summarize the average percent recovery values of the two nonsignificant interaction terms of aggregate ratio by test procedure and cement content by aggregate ratio by test procedure.

Table F-4 summarizes Dunnett's procedure for the calcareous aggregate mixtures by cement content. An "ns" response in the body of the

table indicates that the test procedure was not significantly different from 100 percent recovery.

SILICEOUS AGGREGATE MIXTURES:
MAIN EFFECTS

The hypotheses listed in Table B-2 proved to be significant with regard to percent recovery of cement content for each main effect. The significant cement content effect (F = 21.40, p = 0.0001) exhibited average percent recoveries of 104.38, 98.79, and 96.00 for the 350-, 550-, and 800 lb/cu yd mixtures, respectively. Tukey's w-procedure indicated that the 350-lb/cu yd mixtures exhibited a significantly higher average percent recovery from the 550- and 800-lb/cu yd mixtures which were not significantly different. The significant aggregate ratio effect (F = 15.93, p = 0.0001) exhibited average percent recovery values of 101.69 and 97.77 for the 35/65 and 50/50 fine-to-coarse aggregate ratio mixtures, respectively. In contrasting these effects, the 35/65 fine-to-coarse aggregate ratio mixtures exhibited a significantly higher average percent recovery than the 50/50 aggregate ratio mixtures. The significant test procedure effect (F = 8.27, p = 0.0001) exhibited average percent recoveries of 102.15 for RAM, 101.55 for CQM, 100.79 for NCCG, 99.65 for X-ray, and 94.26 for CF. Dunnett's procedure indicated that CF was the only test procedure which was significantly different from the control of 100 percent recovery. However, since both first-order interaction terms are significant, further investigation is warranted.

SILICEOUS AGGREGATE MIXTURES:
INTERACTION EFFECTS

The significant interaction effects were cement content by test procedures (F = 8.13, p = 0.0001) and aggregate ratio by test procedure

(F = 20.49, p = 0.0001). The average percent recoveries of cement for the significant effects are displayed in Tables F-5 and F-6. As can be observed from these tables, the test procedures which were not significantly different from 100 percent recovery are CF, NCCG, and X-ray for the 350-lb/cu yd mixtures; CQM, NCCG, RAM, and X-ray for 550 and 800-lb/cu yd mixtures. Furthermore, CF was the only test procedure which was consistently different from the 100-percent recovery for both the 35/65 and 50/50 fine-to-coarse aggregate ratio mixtures. Table F-7 summarizes the average percent recoveries for the second-order interaction effect which was nonsignificant. Table F-8 summarizes Dunnett's comparison procedure for the siliceous mixtures.

BLENDED AGGREGATE MIXTURES:
MAIN EFFECTS

The hypotheses listed in Table B-2 proved to be significant with regard to percent recovery of cement for each main effect. The significant cement content effect (F = 3.52, p = 0.0328) exhibited average percent recoveries of 103.14, 105.14 and 101.19 for the 350-, 550-, and 800-lb/cu yd mixtures, respectively. Tukey's w-procedure indicated that the 550-lb/cu yd mixtures exhibited a significantly higher average percent recovery from the 800-lb/cu yd mixtures. The 350-lb/cu yd mixture was not significantly different from either the 550- or 800-lb/cu yd mixtures. The significant aggregate ratio effect (F = 13.43, p = 0.0004) exhibited average percent recoveries of 100.93 and 105.38 for the 35/65 and 50/50 fine-to-coarse aggregate ratio mixtures, respectively. In contrasting these effects the 35/65 aggregate ratio mixtures exhibited a significantly smaller average percent recovery than the 50/50 aggregate ratio mixtures. The significant test procedure effect (F = 9.02,

p = 0.0001) exhibited average percent recoveries of 107.38 for CQM, 106.23 for CF, 104.11 for X-ray, 100.51 for RAM, and 97.55 for NCCG. Dunnett's procedure indicated that RAM and NCCG were the only two test procedures which were not significantly different from the control of 100 percent recovery. However, since both the first-order and the second-order interaction effects were significant, further investigation is warranted.

BLENDED AGGREGATE MIXTURES:
INTERACTION EFFECTS

The significant first-order interaction effects were cement content by test procedure (F = 6.71, p = 0.0001) and aggregate ratio by test procedure (F = 11.04, p = 0.0001). The average percent recoveries of cement for the significant effects are displayed in Tables F-9 and F-10. Dunnett's procedure was not performed on these averages, due to the significant second-order interaction effect which is summarized in Table F-11. The test procedures which were not significantly different from 100 percent recovery are summarized in Table F-12. For the 35/65 fine-to-coarse aggregate ratio mixtures CF, NCCG, RAM, and X-ray were not significantly different from 100 percent recovery for the 350-lb/cu yd mixtures and all test procedures for the 550- and 800-lb/cu yd mixtures. For the 50/50 aggregate ratio mixtures CF, CQM, and RAM for the 350-lb/cu yd mixtures and all methods except for CF for the 550- and 800-lb/cu yd mixtures were not significantly different from 100 percent recovery.

PERCENT WATER RECOVERY

In conjunction with the extensive investigation into percent recovery of cement content, the water content for each of 18 design mixtures was estimated using the HP, CQM, and MW test procedures. As with the percent cement content recovery experiments, each mixture was evaluated for percent water recovery, and delineation of any differences among design factors and test procedures was performed by means of a factorial ANOVA using cement content, aggregate ratio, and test procedures as the controlled factors. For the mixtures in which aggregate type was available, significant differences were observed with test procedures. Table F-13 summarizes the average percent water recoveries by aggregate type. As can be seen for the blended and calcareous mixtures, the HP and MW percent water recoveries were not significantly different from 100 percent recovery; and for the siliceous mixtures, the MW was the only test procedure to exhibit an average percent water recovery which was not significantly different from 100 percent recovery.

SUMMARY

Because of the highly significant interaction terms, no one test procedure proved to exhibit better measurement characteristics for estimating content. With each of the mixture factors, individual recommendations would have to be made. For the calcareous mixtures, the test procedures of CF for the 350-lb/cu yd mixtures, CQM and RAM for 550-lb/cu yd mixtures, and CQM, NCCG, RAM, and X-ray for 800-lb/cu yd mixtures are considered better because each of the average percent cement content recoveries were not considered significantly different from the 100 percent recovery value. For the siliceous mixtures, the test procedures NCCG and X-ray for 350-lb/cu yd mixtures and CQM, NCCG, RAM, and X-ray for the 550- and 800-lb/cu yd mixtures are considered better. For the blended mixtures, CF and RAM for the 350-lb/cu yd mixtures and CQM,

NCCG, RAM, and X-ray test procedures for the 550- and 800-lb/cu yd mixtures are considered better.

Table F-1

Average Percent Recovery, Calcareous Aggregate Mixtures, Cement Content by Test Procedure ($F = 2.12$, $p = 0.0390$), $N = 10$, LL = 93.1[a], UL = 106.9[a]

Cement Content lb/cu yd	Test Procedure CF	CQM	NCCG	RAM	X-Ray
350	97.6	114.5[b]	109.0[b]	113.7[b]	123.2[b]
550	93.9	106.3	110.0[b]	98.5	113.5[b]
800	90.5[b]	102.6	101.2	97.6	100.7

Note: Dunnett's interval is a 95 percent confidence interval which is used to compare all averages to the control of 100 percent recovery.

[a] LL: Lower limit on Dunnett's confidence interval for 100 percent recovery
UL: Upper limit on Dunnett's confidence interval for 100 percent recovery

[b] Significant difference from 100 percent recovery.

Table F-2

Average Percent Recovery, Calcareous Aggregate Mixtures, Aggregate Ratio by Test Procedure ($F = 1.79$, $p = 0.1346$), $N = 15$

Fine-to-Coarse Aggregate Ratio	Test Procedure CF	CQM	NCCG	RAM	X-Ray
35/65	96.0	109.4	111.5	107.9	112.1
50/50	92.0	106.2	101.9	98.6	112.8

Table F-3

Average Percent Recovery, Calcareous Aggregate Mixtures, Cement Content by Aggregate Ratio by Test Procedure ($F = 1.75$, $p = 0.0946$), $N = 5$

Fine-to-Coarse Aggregate Ratio	Cement Content lb/cu yd	Test Procedure CF	CQM	NCCG	RAM	X-Ray
35/65	350	105.0	120.1	109.8	122.7	124.3
	550	95.4	104.7	115.8	103.2	114.1
	800	87.6	103.2	109.0	97.7	97.9
50/50	350	90.2	108.9	108.2	104.6	122.1
	550	92.5	107.8	104.2	93.7	112.8
	800	93.4	101.9	93.3	97.5	103.4

Table F-4

Calcareous Aggregate Mixtures, Cement Content by Test Procedure, Summary of Results of Dunnett's Procedure

Cement Content lb/cu yd	Test Procedure CF	CQM	NCCG	RAM	X-Ray
350	ns	sig	sig	sig	sig
550	sig	ns	sig	ns	sig
800	sig	ns	ns	ns	ns

Note: An "ns" response indicates no significant difference from 100 percent cement recovery. A "sig" indicates a significant difference from 100 percent cement recovery.

Table F-5

Average Percent Recovery, Siliceous Aggregate Mixtures, Cement Content by Test Procedure ($F = 8.13$, $p = 0.0001$), N = 10[a], LL = 93.4, UL = 106.6

Cement Content lb/cu yd	Test Procedure CF	CQM	NCCG	RAM	X-Ray
350	102.7	109.3[b]	93.7	112.2[b]	104.1
550	92.3[b]	98.0	105.6	99.1	98.3
800	86.9[b]	97.3	103.1	95.2	96.6

[a] The averages within the 550- and 800-lb/cu yd mixtures for the test procedure of CF had nine observations each.

[b] Significant difference from 100 percent recovery.

Table F-6

Average Percent Recovery, Siliceous Aggregate Mixtures, Aggregate Ratio by Test Procedure ($F = 20.49$, $p = 0.0001$), N = 15[a], LL = 94.9, UL = 105.1

Fine-to-Coarse Aggregate Ratio	Test Procedure CF	CQM	NCCG	RAM	X-Ray
35/65	105.4[b]	100.6	99.7	104.2	98.5
50/50	81.4[b]	102.6	101.9	100.1	100.8

Note: Dunnett's interval is a 95 percent confidence interval which is used to compare all averages to the control of 100 percent recovery.

[a] The averages within the 35/65 and 50/50 mixtures for the test procedure CF had 14 observations each.

[b] Significant difference from 100 percent recovery.

Table F-7

Average Percent Recovery, Siliceous Aggregate Mixtures, Cement Content by Aggregate Ratio by Test Procedure ($F = 0.88$, $p = 0.5329$), N = 5[a]

Fine-to-Coarse Aggregate Ratio	Cement Content lb/cu yd	Test Procedure CF	CQM	NCCG	RAM	X-Ray
35/65	350	113.6	110.6	92.0	115.6	102.5
	550	103.7	96.2	106.1	104.1	97.8
	800	98.9	94.8	101.1	92.9	95.3
50/50	350	91.7	108.0	95.4	108.8	105.6
	550	78.0	99.9	105.2	94.0	98.7
	800	71.9	99.7	105.0	97.5	98.0

[a] The averages within the 50/50 aggregate ratio mixtures for the test procedure of CF had only four observations each for the 550- and 800-lb/cu yd content levels.

Table F-8

Siliceous Aggregate Mixtures, Cement Content by Test Procedure, Summary of Results of Dunnett's Procedure

Factor	Test Procedure CF	CQM	NCCG	RAM	X-Ray
Cement Content lb/cu yd					
350	ns	sig	ns	sig	ns
550	ns	ns	ns	ns	ns
800	sig	ns	ns	ns	ns
Fine-to-Coarse Aggregate Ratio					
35/65	sig	ns	ns	ns	ns
50/50	sig	ns	ns	ns	ns

Note: An "ns" response indicates no significant difference from 100 percent cement recovery. A "sig" indicates a significant difference from 100 percent cement recovery.

Table F-9

Average Percent Recovery, Blended Aggregate Mixtures, Cement Content by Test Procedure ($F = 6.71$, $p = 0.0001$), N = 10

Cement Content lb/cu yd	Test Procedure: CF	CQM	NCCG	RAM	X-Ray
350	103.7	112.1	86.7	104.1	109.1
550	111.8	107.1	103.2	99.4	104.1
800	103.1	103.0	102.8	98.0	99.1

Table F-10

Average Percent Recovery, Blended Aggregate Mixtures, Aggregate Ratio by Test Procedure ($F = 11.04$, $p = 0.0001$), N = 15

Fine-to-Coarse Aggregate Ratio	Test Procedure: CF	CQM	NCCG	RAM	X-Ray
35/65	96.8	106.1	99.7	101.1	101.0
50/50	115.7	108.6	95.4	99.9	107.3

Table F-11

Average Percent Recovery, Blended Aggregate Mixtures, Cement Content by Aggregate Ratio by Test Procedure ($F = 3.40$, $p = 0.0015$), N = 5, LL = 87.9, UL = 112.11

Fine-to-Coarse Aggregate Ratio	Cement Content lb/cu yd	Test Procedure: CF	CQM	NCCG	RAM	X-Ray
35/65	350	102.3	113.1[a]	98.5	107.2	104.8
	550	95.5	106.4	102.8	99.2	102.4
	800	92.5	98.9	97.8	96.9	95.7
50/50	350	105.2	111.1	74.9[a]	100.9	113.4[a]
	550	128.2[a]	107.7	103.6	99.7	105.9
	800	113.7	107.0	107.7	99.1	102.5

Note: Dunnett's interval is a 95 percent confidence interval which is used to compare all averages to the control of 100 percent recovery.

[a] Significant difference from 100 percent recovery.

Table F-12

Blended Aggregate Mixtures, Cement Content by Test Procedure, Summary of Results of Dunnett's Procedure

Fine-to-Coarse Aggregate Ratio	Cement Content lb/cu yd	Test Procedure: CF	CQM	NCCG	RAM	X-Ray
35/65	350	ns	sig	ns	ns	ns
	550	ns	ns	ns	ns	ns
	800	ns	ns	ns	ns	ns
50/50	350	ns	ns	sig	ns	sig
	550	sig	ns	ns	ns	ns
	800	sig	ns	ns	ns	ns

Note: An "ns" response indicates no significant difference from 100 percent cement recovery. A "sig" indicates a significant difference from 100 percent cement recovery.

Table F-13

Percent Water Recovery

Aggregate Type	F	p	Test Procedure CQM	Hot Plate	Microwave
Blended	4.72	0.0116	92.8[a]	103.0	101.1
Calcareous	4.60	0.0130	96.0[a]	102.7	99.4
Siliceous	13.32	0.0001	95.7[a]	105.1[a]	101.2

[a] Indicates that average percent water recovery was significantly different from 100 percent recovery.

APPENDIX G

EFFECTS OF SIMULATED PROLONGED MIXING

INTRODUCTION

In order to determine what effects simulated prolonged mixing had on the ability of estimating cement content, concrete mixtures with cement contents of 350- and 800-lb/cu yd were subjected to simulated mixing times of 15 and 60 min by adding an appropriate amount of fines to the samples. After the mixtures were produced, samples were collected and the cement content was estimated using the four test procedures of CQM, NCCG, RAM, and X-ray. The concrete mixtures used in this experiment consisted of two of those in which aggregate type was a variable (calcareous and blended). The underlying experimental design was a three-factor factorial for each aggregate type: cement content, simulated mixing times, and test procedures.

CALCAREOUS AGGREGATE MIXTURES: MAIN EFFECTS

With the calcareous aggregate mixtures, the three main effects of cement content, simulated mixing time, and test procedures were significant. Cement content ($F = 11.61$, $p = 0.0011$) indicated that the 350-lb/cu yd mixtures exhibited a significantly higher percent recovery than the 800-lb/cu yd mixtures. The average percent recoveries for each of these factors were 116.29 and 105.56 for the 350- and 800-lb/cu yd mixtures, respectively. The significant simulated mixing time factor ($F = 6.30$, $p = 0.0146$) indicated that both simulated mixing times exhibited average percent recovery values which were significantly higher than 100 percent recovery. The observed averages were 106.97 and 114.88 for the 15- and 60-min simulated mixing times, respectively. The significant test procedure effect ($F = 8.01$, $p = 0.0002$) indicated that CQM and RAM exhibited average percent recoveres which were not significantly different from 100 percent recovery; whereas, X-ray and NCCG were significantly higher. The average values were 123.40 for X-ray, 111.12 for NCCG, 105.79 for CQM, and 103.40 for RAM. However, since the first-order interaction effect of cement content by test procedures was significant, further investigation is warranted.

CALCAREOUS AGGREGATE MIXTURES:
INTERACTION EFFECTS

The first-order interaction effect of cement content by test procedure was the only significant interaction effect observed. Table G-1 displays the average percent recovery values for this effect; whereas, Tables G-2 and G-3 display the average percent recovery values for the nonsignificant interaction effects of simulated mixing time by test procedure and cement content by simulated mixing time by test procedure. As can be seen from Table G-1, CQM, NCCG, and RAM exhibited averages which were not significantly different from 100 percent recovery for the 350-lb/cu yd mixtures; whereas, CQM, RAM, and X-ray exhibited averages which were not significantly different from 100 percent recovery for the 800-lb/cu yd mixtures.

BLENDED AGGREGATE MIXTURES:
MAIN EFFECTS

With the blended aggregate mixtures, the only significant effects were cement content and test procedures. Simulated mixing times did not affect the percent recovery. The significant cement content effect ($F = 33.79$, $p = 0.0001$) exhibited average percent recoveries of 106.23 for the 350-lb/cu yd mixtures and 93.40 for the 800-lb/cu yd mixtures. Tukey's w-procedure indicated that the average percent recovery for the 350-lb/cu yd mixtures was significantly higher than for the 800-lb/cu yd

mixtures. The nonsignificant simulated mixing time effect ($F = 0.39$, $p = 0.5370$) exhibited average percent recoveries of 100.50 and 99.13 for the 15- and 60-min simulated mixing times, respectively. The significant test procedure effect ($F = 7.85$, $p = 0.0002$) exhibited average percent recovery values of 108.85 for NCCG, 98.75 for CQM, 96.25 for RAM, and 95.43 for X-ray. Of the four test procedures, only NCCG exhibited an average percent recovery value which was significantly different from 100 percent recovery.

BLENDED AGGREGATE MIXTURES:
INTERACTION EFFECTS

In contrast to the calcareous aggregate mixtures, none of the interaction effects for the siliceous aggregate mixtures was significant. Hence, the interpretations and findings of the main effects are consistent over the various levels of the factor combinations. Tables G-4 through G-6 summarize the average percent recovery for each of the interaction effects.

SUMMARY

Simulated prolonged mixing of 60 min tended to produce samples which displayed significantly higher percent recoveries than samples which were subjected to a simulated mixing time of 15 min for the calcareous aggregate mixture samples. It was also noticed that, within the calcareous aggregate mixtures, the overall average percent recovery for both the 15-min and 60-min simulated mixing times was significantly higher than 100 percent recovery. However, when considering the averages displayed in Table G-2, the X-ray test procedure appears to be the primary reason for this behavior. Furthermore, when considering the effects of simulated prolonged mixing, it appears that, for the calcareous aggregate mixtures, the numerical trend is an increase in percent recovery with increased mixing. However, in testing the non-significant mixing time by test procedure effect using Dunnett's procedure to determine whether the prolonged mixing averages were different from 100 percent, the following conclusions were made:

1. For the CQM and RAM methods neither the 15-min nor the 60-min averages were significantly different from 100 percent recovery.

2. For the X-ray method, both the 15-min and 60-min averages were significantly different from 100 percent recovery.

3. For the NCCG method, the 15-min average was not significantly different from 100 percent recovery, but the 60-min average was significantly different from 100 percent recovery.

It must be reemphasized that for the blended aggregate mixtures, the above conclusions were not substantiated. In summary, for the calcareous aggregate mixtures, simulated prolonged mixing of up to 60 min did tend to increase percent recovery; however, this increase in percent recovery only produced averages which were significantly different from 100 percent recovery for the NCCG method. If mixing time were increased past 60 min, the effect which was observed for NCCG would most probably be demonstrated by each of the other test procedures.

Table G-1

Average Percent Recovery, Calcareous Aggregate Mixtures, Cement Content by Test Procedure (F = 6.38, p = 0.0008), N = 10, LL = 87.5, UL = 112.5

Cement Content lb/cu yd	Test Procedure CQM	NCCG	RAM	X-Ray
350	106.6	108.6	111.2	138.8[a]
800	105.0	113.7[a]	95.6	108.0

Note: Dunnett's interval is a 95 percent confidence interval which is used to compare all averages to the control of 100 percent recovery.

[a] Significant difference from 100 percent recovery.

Table G-2

Average Percent Recovery, Calcareous Aggregate Mixtures, Simulated Mixing Time by Test Procedure (F = 0.46, p = 0.7146), N = 10

Simulated Mixing Time, min	Test Procedure CQM	NCCG	RAM	X-Ray
15	99.3	109.3	98.5	120.8
60	112.3	113.0	108.3	126.0

Table G-3

Average Percent Recovery, Calcareous Aggregate Mixtures, Cement Content by Simulated Mixing Time by Test Procedure (F = 0.02, p = 0.9918), N = 5

Cement Content lb/cu yd	Simulated Mixing Time min	Test Procedure			
		CQM	NCCG	RAM	X-Ray
350	15	101.2	108.9	107.8	137.9
	60	112.0	108.3	114.6	139.7
800	15	97.4	109.7	89.2	103.7
	60	112.5	117.6	102.0	112.3

Table G-4

Average Percent Recovery, Blended Mixtures, Cement Content by Testing Methods (F = 0.46, p = 0.7180), N = 10

Cement Content	Testing Method			
	CQM	NCCG	RAM	X-Ray
350	105.6	116.2	103.5	99.6
800	91.9	101.5	89.0	91.2

Table G-5

Average Percent Recovery, Blended Mixtures, Simulated Mixing Time by Testing Methods (F = 0.39, p = 0.7624), N = 10

Simulated Mixing Time, min	Testing Method			
	CQM	NCCG	RAM	X-Ray
15	98.9	111.6	96.0	95.6
60	98.7	106.1	96.5	95.3

Table G-6

Average Percent Recovery, Blended Mixtures, Cement Content Simulated Mixing Time by Testing Methods (F = 1.24, p = 0.3041), N = 5

Cement Content lb/cu yd	Simulated Mixing Time min	Testing Method			
		CQM	NCCG	RAM	X-Ray
350	15	105.6	124.0	103.2	101.6
	60	105.6	108.3	103.8	97.7
800	15	92.1	99.1	88.8	89.6
	60	91.7	103.9	89.2	92.9

APPENDIX H

EFFECTS OF FLY ASH AND GROUND GRANULATED

IRON BLAST-FURNACE SLAG

INTRODUCTION

In order to determine what effects the fly ash and ground granulated iron blast-furnace slag had on the estimation of percent cement recovery, concrete mixtures were produced using calcareous aggregate and siliceous aggregate with either fly ash or ground slag at cementitious material contents of 550- and 800-lb/cu yd. The test procedures CQM, NCCG, RAM, and X-ray were then evaluated to determine the cement content of the concrete samples. A three-factor factorial ANOVA was used to ascertain significant differences among the factors of this experiment.

CALCAREOUS AGGREGATE AND FLY ASH MIXTURES: MAIN EFFECTS

The ANOVA indicated that the two main effects of cement content and percent fly ash were significant, and that the main effect of test procedure was nonsignificant. The significant cement content effect ($F = 15.63$, $p = 0.0002$) exhibited average percent recovery values of 122.32 and 109.59 for the 550- and 800-lb/cu yd mixtures, respectively. Tukey's w-procedure indicated that the 550-lb/cu yd mixtures exhibited a significantly larger average percent recovery than the 800-lb/cu yd mixtures. The significant percent fly ash effect ($F = 27.54$, $p = 0.0001$) exhibited average percent recoveries of 124.66 for the 40-percent fly ash mixtures and 106.85 for the 15-percent fly ash mixtures. Furthermore, not only did the increase of from 15 to 40 percent fly ash produce a significant effect, but also both average percent cement content recoveries were significantly different from 100 percent recovery. Also, since the percent fly ash by test procedure interaction effect was significant, a more detailed investigation is warranted. The nonsignificant test procedure effect ($F = 0.33$, $p = 0.8029$) exhibited average

percent cement recoveries of 118.44 for X-ray, 116.66 for CQM, 114.90 for RAM, and 114.14 for NCCG. In comparing each of these averages to 100 percent recovery, it was observed that each test procedure produced averages which were significantly higher than 100 percent recovery.

CALCAREOUS AGGREGATE AND FLY ASH MIXTURES: INTERACTION EFFECTS

The only interaction effect which was significant was the first-order interaction effect of percent fly ash by test procedures. The average percent cement recoveries for this significant effect are summarized in Table H-1. As can be observed from this table, CQM, NCCG, and X-ray estimated percent cement recoveries which were not significantly different from 100 percent recovery within the 15 percent fly ash replacement level. All test procedures were significantly larger than 100 percent recovery within the 40 percent fly ash replacement levels. Tables H-2 and H-3 summarize the average percent recoveries for the nonsignificant first-order interaction effect of cement content by test procedure and cement content by percent fly ash by test procedure, respectively.

CALCAREOUS AGGREGATE AND GROUND SLAG MIXTURES: MAIN EFFECTS

The ANOVA indicated that the two main effects of cement content and percent slag were not significant, and the main effect of test procedure was significant. The nonsignificant cement content effect ($F = 0.28$, $p = 0.5985$) exhibited average percent recoveries of 93.97 and 93.32 for the 550- and 800-lb/cu yd mixtures. The nonsignificant percent slag effect ($F = 1.56$, $p = 0.2168$) exhibited average percent recoveries of 94.6 for the 40-percent slag mixtures and 92.67 for the 15-percent mixtures. Furthermore, the average percent cement content recoveries were significantly different from 100 percent recovery. Also, since the percent slag by test procedure interaction effect was significant, a more detailed investigation is warranted. The significant test procedure effect (F = 19.17, p = 0.0001) exhibited average percent cement recoveries of 103.71 for CQM, 93.44 for NCCG, 92.00 for RAM, and 85.01 for X-ray. In comparing each of these averages to 100 percent recovery, it was observed that the CQM test procedure was the only method not significantly different from 100 percent recovery and that the other four methods were significantly different and lower than 100 percent recovery. As with the percent slag effect, a closer evaluation of this significance relation is warranted.

CALCAREOUS AGGREGATE AND GROUND SLAG MIXTURES: INTERACTION EFFECTS

The only interaction effect which was significant was the first-order interaction effect of percent slag by test procedure. The average percent cement content recoveries for this significant effect are summarized in Table H-4. As can be observed from this table, only CQM and RAM estimated percent cement content recovery values which were not significantly different from 100 percent recovery within the 15-percent slag level; and within the 40-percent slag CQM and NCCG were not significantly different from 100 percent recovery. Tables H-5 and H-6 summarize the average percent recoveries for the nonsignificant first-order interaction effect of cement content by test procedure and cement content by percent slag by test procedure, respectively.

SILICEOUS AGGREGATE AND FLY ASH MIXTURES: MAIN EFFECTS

The ANOVA indicated that the two main effects of cement content and percent fly ash were not significant, and that the main effect of test procedures was significant. The nonsignificant cement content effect (F = 0.19, p = 0.6622) exhibited average percent recoveries of 97.45 and 96.31 for the 550- and 800-lb/cu yd mixtures, respectively. The nonsignificant percent fly ash effect (F = 2.33, p = 0.1324) exhibited average percent recoveries of 98.02 for the 40 percent fly ash mixtures and 95.69 for the 15 percent fly ash levels. Furthermore, according to Dunnett's procedure, the 40 percent fly ash replacement level was not significantly different from 100-percent recovery, but the 15 percent level of fly ash was significantly lower. The significant test procedure effect (F = 10.45, p = 0.0001) exhibited average percent cement recoveries of 101.78 for CQM, 100.45 for RAM, 92.84 for NCCG, and 91.64 for X-ray. In comparing each of these averages to 100 percent recovery, it was observed that CQM and RAM produced averages which were not significantly different from 100 percent recovery, and that NCCG and X-ray produced averages which were significantly smaller than 100 percent recovery.

SILICEOUS AGGREGATE AND FLY ASH MIXTURES: INTERACTION EFFECTS

All of the interaction effects for this experiment were judged to be nonsignificant by the ANOVA. Tables H-7 through H-9 summarize the average percent cement content recoveries for each of these effects.

SILICEOUS AGGREGATE AND GROUND SLAG MIXTURES: MAIN EFFECTS

The ANOVA indicated that the two main effects of cement content and test procedures were significant, and that the main effect of percent slag was not significant. The significant cement content effect (F = 11.37, p = 0.0013) exhibited average percent recoveries of 96.00 and 89.96 for the 550- and 800-lb/cu yd mixtures. Tukey's w-procedure indicated that the 500-lb/cu yd mixtures exhibited a significantly higher average percent cement content recovery than the 800-lb/cu yd mixtures. The nonsignificant percent slag effect (F = 0.11, p = 0.7449) exhibited average percent recoveries of 93.31 for the 40 percent slag mixtures and 92.63 for the 15 percent slag mixtures. Furthermore, the average percent cement content recoveries were significantly smaller than 100 percent recovery. Also, since the percent slag by test procedure interaction effect was significant, a more detailed investigation is warranted. The significant test procedure effect (F = 40.37, p = 0.0001) exhibited average percent cement recoveries of 104.36 for CQM, 98.55 for RAM, 93.85 for NCCG, and 74.78 for X-ray. In comparing each of these averages to 100 percent recovery, it was observed that the CQM and RAM were the only methods not significantly different from 100 percent recovery and that the other two methods were significantly different and lower than 100 percent recovery, but as with the percent slag effect, a closer evaluation of this significance relation is warranted.

SILICEOUS AGGREGATE AND GROUND SLAG MIXTURES: INTERACTION EFFECTS

The two first-order interaction effects were significant. Tables H-10 and H-11 summarize the average percent cement content recovery for each of these effects. From these tables it can be observed that within the 800-lb/cu yd mixtures and 15 percent slag CQM and RAM, within the 550-lb/cu yd mixtures NCCG and RAM, and for the 40 percent slag CQM and NCCG exhibited average percent recoveries which were not significantly different from 100 percent recovery. Table H-12 summarizes the average percent recoveries for the nonsignificant second-order interaction effect of cement content by percent slag by test procedure.

SUMMARY

Fly ash tended to produce increased average percent cement content recoveries for both the calcareous and siliceous aggregate mixtures; however, the observed increase in percent recovery with increased percent fly ash was only significant with the calcareous mixtures. At the 15 percent fly ash levels, the test procedures CQM, NCCG, and X-ray produced average percent recoveries which were not significantly different from 100 percent for the calcareous aggregate mixtures, but with the 40 percent fly ash levels, each of the above-mentioned test procedures tended to overestimate 100 percent recovery. With the siliceous mixtures, the nonsignificant increase in average percent recovery did not affect the estimability of the CQM and RAM. At both levels of fly ash replacement, CQM and RAM produced averages which were not significantly different from 100 percent recovery. The test procedures of NCCG and X-ray produced estimates which were considered to be significantly different and lower than 100 percent recovery. Ground granulated iron blast-furnace slag did not produce any significant effects with respect to calcareous and siliceous aggregate types. For the calcareous-aggregate mixtures, CQM produced average percent recoveries which were not significantly different from 100 percent recovery at both the 15 and 40 percent slag levels. NCCG and RAM produced average percent recovery values which were not significantly different from 100 percent recovery at the 40 and 15 percent slag levels, respectively. With regard to the siliceous aggregate mixtures, the same test procedure conclusions which the calcareous mixtures produced were valid for the siliceous mixtures.

Table H-1

Average Percent Recovery, Calcareous Aggregates and Fly Ash Mixtures, Percent Cementitious Material by Test Procedure

($F = 5.96$, $p = 0.0014$), N = 10[a]

Percent Cementitious Material	Test Procedure			
	CQM	NCCG	RAM	X-Ray
15	110.4	94.8	114.1[b]	108.6
40	122.9[b]	133.4[b]	115.7[b]	127.1[b]

Note: The confidence intervals vary according to sample size:
if N = 10, then LL = 87.4, UL = 112.6;
if N = 8, then LL = 85.9, UL = 114.1; and
if N = 7, then LL = 84.9, UL = 115.1.

Dunnett's interval is a 95 percent confidence interval which is used to compare all averages to the control of 100 percent recovery.

[a] Averages for X-ray consisted of seven and eight observations for 15 and 45 percent cementitious material, respectively.

[b] Significant difference from 100 percent recovery.

Table H-2

Average Percent Recovery, Calcareous Aggregates and Fly Ash Mixtures, Cement Content by Test Procedure ($F = 2.46$, $p = 0.0704$), N = 10[a]

Cement Content lb/cu yd	Test Procedure			
	CQM	NCCG	RAM	X-Ray
550	125.3	122.4	113.9	130.1
800	108.1	105.9	115.9	108.3

[a] Averages for X-ray contained seven and eight observations for the cement content levels of 550- and 800-lb/cu yd, respectively.

Table H-3

Average Percent Recovery, Calcareous Aggregate and Fly Ash Mixture, Cement Content by Percent Cementitious Material by Test Procedure

(F = 0.59. p = 0.6282), N = 5[a]

Cement Content lb/cu yd	Percent Cementitious Material	Test Procedure			
		CQM	NCCG	RAM	X-Ray
550	15	117.5	99.1	114.6	121.3
	40	133.0	145.7	113.2	136.6
800	15	103.3	90.6	113.6	99.0
	40	112.8	121.2	118.2	117.6

[a] Averages for X-ray consisted of three and four observations for 550 lb/cu yd, respectively and four observations for 800 lb/cu yd, cement content.

Table H-4

Average Percent Recovery, Calcareous Aggregate and Pulverized Slag Mixture, Percent Cementitious Material by Test Procedures

(F = 5.15, p = 0.0031) N = 10[a]

Percent Cementitious Material	Test Procedure			
	CQM	NCCG	RAM	X-Ray
15	103.6	87.7[b]	95.8	82.6[b]
40	103.9	99.1	88.2[b]	87.2[b]

Note: The confidence intervals vary according to sample size:
if N = 10, then LL = 93.0, UL - 107.0; and
if N = 8, then LL = 92.6, UL = 107.4.

Dunnett's interval is a 95 percent confidence interval which is used to compare all averages to the control of 100 percent recovery.

[a] Averages for X-ray consisted of seven and eight observations for 15 and 40 percent cementitious material, respectively.

[b] Significant difference from 100 percent recovery.

Table H-5

Average Percent Recovery, Calcareous Aggregate and Pulverized Slag Mixtures, Cement Content by Test Procedure (F = 1.32, p = 0.2746), N = 10[a]

Cement Content lb/cu yd	Test Procedure			
	CQM	NCCG	RAM	X-Ray
550	105.2	91.3	94.5	84.9
800	102.2	95.6	89.5	85.1

[a] The average from X-ray contained nine observations within the 800-lb/cu yd level.

Table H-6

Average Percent Recovery, Calcareous Aggregate and Ground Slag Mixture, Cement Content by Percent Cementitious Material by Test Procedure

(F = 1.78; p = 0.1592), N = 5[a]

Cement Content lb/cu yd	Percent Cementitious Material	Test Procedure			
		CQM	NCCG	RAM	X-Ray
550	15	103.9	81.3	99.0	83.2
	40	106.4	101.3	90.0	86.6
800	15	103.2	94.2	92.6	81.8
	40	101.3	97.0	86.4	87.4

[a] The average from X-ray contained four observations for 15 percent cementitious material within the 800 lb/cu yd level.

Table H-7

Average Percent Recovery, Siliceous Aggregate and Fly Ash Mixture, Cement Content by Test Procedure (F = 0.10, p = 0.9568), N = 10[a]

Cement Content lb/cu yd	Test Procedure			
	CQM	NCCG	RAM	X-Ray
550	101.7	93.0	101.5	92.1
800	101.9	92.7	99.4	91.3

[a] The average for X-Ray contained seven observations within the 550-lb/cu yd level of cement content.

Table H-8

Average Percent Recovery, Siliceous Aggregate and Fly Ash Mixture, Percent Cementitious Material by Test Procedure (F = 0.39, p = 0.7631), N = 10[a]

Percent Cementitious Material	Test Procedure			
	CQM	NCCG	RAM	X-RAY
15	99.2	92.5	99.7	90.3
40	104.4	93.2	101.2	92.8

[a] Averages for X-ray consisted of eight and seven observations for 15 and 40 percent cementitious material, respectively.

Table H-9

Average Percent Recovery, Siliceous Aggregate and Fly Ash Mixture, Cement Content by Percent Cementitious Material by Test Procedure (F - 0.04, p = 0.9826), N = 5[a]

Cement Content lb/cu yd	Percent Cementitious Material	Test Procedure			
		CQM	NCCG	RAM	X-Ray
550	15	99.4	92.4	100.4	90.3
	40	103.9	93.6	102.6	93.5
800	15	98.9	92.6	99.0	90.4
	40	104.8	92.7	99.8	92.2

[a] The averages for X-ray within the 550-lb/cu yd cement content level contained three and four observations, respectively, for the 15 and 40 percent cementitious material levels.

Table H-10

Average Percent Recovery, Siliceous Aggregate and Ground Slag Mixtures, Cement Content by Test Procedure (F = 5.65, p = 0.0018), N = 10[a]

Cement Content lb/cu yd	Test Procedure			
	CQM	NCCG	RAM	X-Ray
550	108.5[b]	102.9	101.7	72.2
800	100.7	84.9[b]	95.4	77.6[b]

Note: The confidence intervals vary according to sample size:
if N = 10, then LL = 92.2, UL - 107.8; and
if N = 9, then LL - 91.7, UL = 108.3.

Dunnett's interval is a 95 percent confidence interval for 100 percent recovery.

[a] The average from X-ray contained nine observations for within the 800-lb/cu yd level.

[b] Significant difference from 100 percent recovery.

Table H-11

Average Percent Recovery, Calcareous Aggregate and Ground Slag Mixture, Percent Cementitious Material by Test Procedure

(F = 16.75, p = 0.0001) N = 10[a]

Percent Cementitious Material	Test Procedure			
	CQM	NCCG	RAM	X-Ray
15	107.2	83.8[b]	107.4	71.5[b]
40	101.8	104.0	89.7[b]	77.7[b]

Note: The confidence intervals vary according to sample size:
if N = 10, then LL = 92.2, UL = 107.8; and
if N = 8, then LL = 91.7, UL = 108.3.

Dunnett's interval is a 95 percent confidence interval which is used to compare all averages to the control of 100 percent recovery.

[a] Averages for CQM and X-ray consisted of nine observations within the 15-percent cementitious material level.

[b] Significant difference from 100 percent recovery.

Table H-12

Average Percent Recovery, Siliceous Aggregate and Ground Slag Mixture, Cement Content by Percent Cementitious Material by Test Procedure

(F = 0.78, p = 0.5115), N = 5[a]

Cement Content lb/cu yd	Percent Cementitious Material	Test Procedure			
		CQM	NCCG	RAM	X-Ray
550	15	110.4	93.8	112.8	67.9
	40	107.0	111.9	90.6	76.5
800	15	104.6	73.8	102.0	76.0
	40	96.7	96.1	88.8	79.0

[a] Averages for CQM within the 550-lb/cu yd mixtures at 15 percent slag and for X-ray within the 800-lb/cu yd mixtures at 15 percent slag consisted of four observations.

APPENDIX I

EFFECTS OF HIGH-RANGE WATER-REDUCING ADMIXTURES ON PERCENT WATER RECOVERY

INTRODUCTION

In order to assess what effects the HRWRA, naphthalene base and melamine base, had on the ability to determine percent water recovery by testing procedure CQM, HP, and MW, concrete mixtures were proportioned consisting of either calcareous aggregate or siliceous aggregate with four levels of naphthalene-based HRWRA (0.5, 2.0, 3.0 and 5.0 percent by mass of the total cementitious material) and one mixture using a melamine-based HRWRA at 3.0 percent. For each of the mixtures, percent water recovery was estimated using the above-mentioned test procedures.

CALCAREOUS AGGREGATE MIXTURES: MAIN EFFECTS

The ANOVA procedure indicated that both main effects of percent admixture and test procedure were significant. The significant test procedure effect (F = 7.87, p = 0.0011) exhibited percent water recovery values of 116.1 for the CQM, 103.5 for HP, and 103.0 for MW procedures. Dunnett's comparison procedure indicated that CQM produced an average percent water recovery which was significantly larger than 100 percent recovery. The significant percent admixture effect (F = 4.16, p = 0.0107) exhibited average percent recovery values of 115.5 for the 2.0 percent level, 106.00 for the 3.0 percent level, 108.1 for the 5.0 percent level, and 100.5 for the 0.5 percent level. Of these four percent admixture levels, only the 2.0 percent level produced an average percent water recovery which was significantly larger than 100 percent recovery.

CALCAREOUS AGGREGATE MIXTURES:
INTERACTION EFFECTS

The interaction effect of percent admixture by test procedure was not significant. Table I-1 summarizes the average percent water recovery values for this effect.

SILICEOUS AGGREGATE MIXTURES:
MAIN EFFECTS

The ANOVA procedure indicated that both the main effects of test procedure and percent admixture were significant. The significant test procedure effect ($F = 6.06$, $p = 0.0045$) exhibited percent water recovery values of 110.3 for HP, 105.6 for CQM, and 101.3 for MW. Of these three procedures, only the MW procedure produced an average percent water recovery value which was not significantly different from 100 percent recovery. The significant percent admixture effect exhibited average percent water recoveries of 113.4 for the 5.0 percent level, 109.3 for the 3.0 percent level, 100.7 for the 2.0 percent level, and 99.4 for the 0.5 percent level. The 0.5 and 2.0 percent level average values were not different from 100 percent; whereas, the 3.0 and 5.0 percent levels were significantly higher than 100 percent recovery. Since the interaction effect of percent admixture by test procedure is significant, further investigation into the effects is warranted.

SILICEOUS AGGREGATE MIXTURES:
INTERACTION EFFECTS

The interaction effect of percent admixture by test procedure was judged to be significant by the ANOVA procedure. Table I-2 summarizes the average percent water recovery for this effect. From this table it is readily observed that the only average percent water recovery values which were significantly larger than 100 percent recovery occurred

within the 5.0 percent admixture replacement levels for the CQM and HP procedures.

NAPHTHALENE VERSUS MELAMINE

The ANOVA procedure indicated that the main effect of test procedure was the only effect which was significant. The nonsignificant admixture effect exhibited average percent water recoveries of 97.1 for the naphthalene-based HRWRA and 96.2 for the melamine-based HRWRA. Both of these averages were significantly different from 100 percent recovery. The significant test procedure effect exhibited average percent recoveries of 101.9 for HP, 96.0 for MW, and 92.0 for CQM. Dunnett's procedure indicated that HP produced an average percent water recovery which was not significantly different from 100 percent recovery; whereas, MW and CQM produced averages which were smaller than 100 percent recovery.

SUMMARY

Increasing the percent naphthalene-based HRWRA from 0.5 to 5.0 percent exhibited mixed trends depending on the type of aggregate mixture. For the calcareous aggregate mixtures, percent water recovery increased when the percent admixture increased from 0.5 to 2.0 percent, but oscillated thereafter. This trend existed regardless of the test procedure. Furthermore, only the CQM proved to produce average percent water recoveries which were significantly higher than 100 percent. For the siliceous aggregate mixtures, the overall trend was increased percent water recovery as percent admixture increased. All average values except for the 5.0 percent admixture with CQM and HP were not significantly different from 100 percent recovery. As far as melamine-based versus naphthalene-based, the two HRWRA's did not prove to produce any adverse effects; however, with these admixtures only the HP produced average percent water recovery values which were not significantly different from 100 percent recovery.

Table I-1

Percent Water Recovery, Calcareous Aggregate Mixture, Naphthalene-based HRWRA, Percent Admixture by Test Procedure ($F = 1.02$, $p = 0.4228$), $N = 5$

Percent Admixture	Test Procedures		
	CQM	HP	MW
0.5	102.4	100.0	99.1
2.0	124.1	108.4	114.0
3.0	114.1	104.4	99.5
5.0	123.7	101.1	99.4

Table I-2

Percent Water Recovery, Siliceous Aggregate Mixture, Naphthalene-based HRWRA, Percent Admixture by Test Procedure ($F = 2.36$, $p = 0.0444$), $N = 5$, LL = 89.3, UL = 110.7

Percent Admixture	Test Procedures		
	CQM	HP	MW
0.5	91.8	109.9	96.5
2.0	107.6	102.8	91.8
3.0	110.4	110.5	106.9
5.0	112.4[a]	117.8[a]	109.8

[a] Significantly different from 100 percent recovery.

APPENDIX J

EFFECTS OF AN ACCELERATOR

INTRODUCTION

In order to determine what effects the accelerator, calcium chloride, had on the ability to estimate percent cement recovery by test procedures CQM, NCCG, RAM, and X-ray, concrete mixtures were produced consisting of either calcareous aggregate or siliceous aggregate with two levels of admixture percentages (1.0 and 2.0 percent). Samples were obtained and percent recovery of cement was determined using each of the above test procedures. This experiment involved three factors for mixtures using each type of aggregate: cement content, percent admixture, and test procedures.

CALCAREOUS AGGREGATE MIXTURES

The ANOVA procedure indicated that all the main effects were significant. The significant cement content effect (F = 36.33, p = 0.0001) exhibited average percent cement content recoveries of 101.63 for the 550-lb/cu yd mixtures and 94.09 for the 800-lb/cu yd mixtures. Tukey's w-procedure indicated that the 550-lb/cu yd mixtures produced average values which were significantly larger than the 800-lb/cu yd mixtures. The significant percent admixture effect (F = 9.80, p = 0.0026) exhibited average percent cement content recoveries of 99.82 for the 1 percent calcium chloride admixture level and 95.91 for the 2 percent level. In contrasting these to 100 percent recovery, only the 1 percent level is not significantly different from 100 percent. The significant test procedure effect (F = 12.88, p = 0.0001) exhibited average percent recoveries of 103.62 for X-ray, 98.72 for CQM, 96.05 for NCCG, and 93.05 for RAM. Of these four instruments, only CQM produced an average which was not significantly different from 100 percent recovery. However,

since the interaction of cement content by test procedure effect is also significant, further investigation is warranted. Tables J-1, J-2, and J-3 summarize the average percent cement recoveries for each of the interaction effects of cement content by test procedure, percent admixture by test procedure, and cement content by percent admixture by test procedure, respectively. As can be seen from Table J-1, CQM and NCCG produced average percent recoveries which were not significantly different from 100 percent for the 550-lb/cu yd mixtures and only X-ray produced an average percent recovery which was not significantly different from 100 percent recovery for the 800-lb/cu yd mixtures.

SILICEOUS AGGREGATE MIXTURES

The ANOVA procedure indicated that the two main effects of cement content and test procedure were significant and that the effect of percent admixture was not significant. The significant cement content effect (F = 33.29, p = 0.0001) exhibited average percent cement recoveries of 104.8 for the 550-lb/cu yd mixtures and 97.88 for the 800-lb/cu yd mixtures. Tukey's w-procedure indicated that the 550-lb/cu yd mixtures had a significantly larger average percent recovery than the 800-lb/cu yd mixtures. The significant test procedure effect (F = 46.91, p = 0.0001) exhibited average percent recoveries of 111.88 for NCCG, 103.35 for CQM, 97.14 for X-ray, and 93.00 for RAM. Dunnett's procedure indicated that only CQM and X-ray produced averages which were not different from 100 percent recovery. NCCG produced an average recovery value which was significantly larger than 100 percent and RAM produced an average which was significantly smaller than 100 percent. The non-significant percent admixture effect (F = 0.19, p = 0.6661) exhibited average recoveries of 101.60 for the 1 percent level and 101.08 for the 2 percent level. Each of these average values was not significantly different from 100 percent recovery. Since the interaction effects were not significant, the conclusions pertaining to the main effects are valid across all combinations of the factors. Tables J-4, J-5, and J-6 summarize the average percent recoveries for each of these interaction effects.

SUMMARY

The two levels of calcium chloride exhibited a significant effect only when used in the calcareous aggregate mixtures. The trend was a decrease in percent cement recovery with an increase in percentage of calcium chloride. Furthermore, with the calcareous aggregate mixtures, CQM and NCCG produced average percent recoveries which were not different from 100 percent recovery for the 550-lb/cu yd mixtures, and only X-ray produced average percent recoveries which were not different from 100 percent recovery for the 800-lb/cu yd mixtures. For the siliceous aggregate mixtures, the percent admixture did not affect percent recovery. Also, CQM and X-ray were the only procedures which produced average percent recoveries which were not different from 100 percent recovery.

Table J-1

Average Percent Recovery, Calcareous Aggregate Mixture, Calcium Chloride, Cement Content By Test Procedure ($F = 4.24$, $p = 0.0086$), $N = 10$, LL = 95.0, UL = 105.0

Cement Content lb/cu yd	Test Procedure			
	CQM	NCCG	RAM	X-Ray
550	102.9	103.0	93.8[a]	106.8[a]
800	94.6[a]	89.1[a]	92.3[a]	100.4

[a] Significant difference from 100 percent recovery.

Table J-2

Average Percent Recovery, Calcareous Aggregate Mixture, Calcium Chloride, Percent Admixture by Test Procedure ($F = 2.17$, $p = 0.0990$), $N = 10$

Percent Admixture	Test Procedure			
	CQM	NCCG	RAM	X-Ray
1	101.4	95.9	94.2	107.7
2	96.0	96.2	91.9	99.5

Table J-3

Average Percent Recovery, Calcareous Aggregate Mixture, Calcium Chloride, Cement Content by Percent Admixture by Test Procedure (F = 0.10, p = 0.9562)

Cement Content lb/cu yd	Percent Admixture	Test Procedure CQM	NCCG	RAM	X-Ray
550	1	106.5	103.7	96.6	112.2
	2	99.3	102.4	91.0	101.4
800	1	96.3	88.2	91.8	103.3
	2	92.8	90.0	92.8	97.6

Table J-4

Average Percent Recovery, Siliceous Aggregate Mixture, Calcium Chloride, Cement Content by Test Procedure (F = 0.33, p = 0.8089), N = 10

Cement Content lb/cu yd	Test Procedure CQM	NCCG	RAM	X-Ray
550	106.6	115.7	95.6	101.3
800	100.1	108.1	90.4	93.0

Table J-5

Average Percent Recovery, Siliceous Aggregate Mixture, Calcium Chloride, Percent Admixture by Test Procedure (F = 0.12, p = 0.9414), N = 10

Percent Admixture	Test Procedure CQM	NCCG	RAM	X-Ray
1	103.8	112.6	92.7	97.3
2	102.9	111.2	93.3	96.9

Table J-6

Average Percent Recovery, Siliceous Aggregate Mixture, Calcium Chloride, Cement Content by Percent Admixture by Test Procedure (F = 1.06, p = 0.3712)

Cement Content lb/cu yd	Percent Admixture	Test Procedure CQM	NCCG	RAM	X-Ray
550	1	107.5	117.8	94.2	100.4
	2	105.7	113.6	97.0	102.2
800	1	100.1	107.3	91.2	94.3
	2	100.1	108.8	89.6	91.6

APPENDIX K

QUALITATIVE DETECTION

The seventh major task involved an evaluation of the test procedures to determine the qualitative ability of the procedures to detect the unexpected presence of a ground granulated iron blast-furnace slag or fly ash pozzolan in a PCC mixture. Both fly ash and ground slag have become conventional concrete-making materials. Fly ash possesses little or no cementing or cementitious value when used alone in concrete; ground granulated iron blast-furnace slag is, however, a hydraulic cement.

The qualitative detection of fly ash and ground slag in PCC involved examining each test procedure and determining its ability to detect what cementitious materials other than portland cement were present in a concrete mixture. Two series of concrete mixtures, one containing 100 percent fly ash and no portland cement and the other mixture containing 100 percent ground slag and no portland cement, were proportioned and batched to simulate the possible error of batching concrete with no portland cement, but rather with all fly ash or ground slag. This activity demonstrated the most extreme error in batching PCC.

The 100 percent fly ash and ground slag evaluations revealed several problems with the test procedures. None of the five cement content determination procedures could qualitatively detect the unexpected presence of fly ash or ground slag in a concrete mixture, as shown in Table K-1. Two procedures, the CQM and X-ray, revealed little or no portland cement; however, they could not determine what material other than the portland cement was in the concrete. These two procedures in one sense had 100 percent freedom of bias in determining the actual cement content, 0 lb/cu yd. The procedures did exactly what they were

supposed to do--determine the cement content. One procedure, the NCCG, found little or no portland cement in only the mixtures containing fly ash. The RAM detected very high percentages of a material it saw as portland cement in both the fly ash and ground slag mixtures, indicating its inability to detect a cementitious material other than portland cement in a concrete mixture as different from portland cement. The NCCG also detected high percentages of what it saw as portland cement, but only in the 100 percent ground slag mixtures. This is not surprising since ground slag is a hydraulic cement consisting largely of calcium silicates. The CF procedure detected high percentages of portland cement in the 100 percent fly ash mixtures. The CF procedure was deleted from further testing in lieu of determining other means of qualitatively detecting the unexpected presence of fly ash and ground slag in a concrete mixture. None of the cement content determination test procedures had the ability to qualitatively detect the unexpected presence of fly ash or ground slag in any concrete mixture. The centrifuge procedure was very erratic in the separation process when the slag or fly ash was present. No clear separation could be made. Several avenues were sought to create or locate a basic technique to quickly but qualitatively detect the presence of ground slag or fly ash; however, no such technique was found during this project. Laboratory methods which can qualitatively detect ground slag or fly ash in concrete mixture are optical microscopy using the petrographic microscope or the scanning electron microscope, and phase identification using an X-ray diffractometer. Chemically, no quick method was found due to the similarities in the composition of the ground slag, fly ash, and portland cement.

Basically the best method was perhaps a modification of the CF procedure. The densities of the portland cement, ground granulated slag, and fly ash were different enough at 3.11, 2.84, and 2.25 Mg/m^3, respectively, so that separation by heavy liquid media was the most likely prospect. However, color charts, particle shape references, and particle size references would still be needed to actually distinguish the ground slag or fly ash from all the other materials in the concrete. Although the prospect of a modified CF procedure without the initial drying phase to reduce the time is more likely, hazardous chemicals must be used to perform the evaluation.

This task provided additional important information concerning the ability of the procedures to determine the cement content of a PCC mixture no matter what the cement content was. There was zero cement factor in both the fly ash and ground slag mixtures; however, only the CQM and X-ray could determine that data for both mixtures and the NCCG for the fly ash mixtures. All other procedures were erratic in determining the actual cement content. Therefore the CQM, X-ray, and NCCG are better for PCC projects using fly ash or ground slag as a replacement material.

Table K-1
100 Percent Replacement Results, Percent Recovery

Material	RAM	CQM	NCCG	X-ray	CF	Calibration Curve Used
Fly Ash	54.4		8.0			0 percent
	57.5		-8.6			15 percent
	59.5		-3.0			40 percent
		0.3		0.1	44.2	Fly Ash
Ground Slag	89.9		66.9			0 percent
	102.2		85.5			15 percent
	84.4		172.4			40 percent
		0.1		6.1		Slag

APPENDIX L
INDIVIDUAL PERCENT RECOVERY—MIXTURE

Table L-1

Individual Bias per Test Procedure per Mixture

	Cement Content					Water Content		
PCC Mixture No.	Concrete Quality Monitor	Nuclear Cement Content Gage	Rapid Analysis Machine	WES Centrifuge Procedure	X-ray Emission Spectrometer	Concrete Quality Monitor	Hot Plate Procedure	Microwave Oven Procedure
2-1	110.6	92.0	115.6	113.6	102.5	95.6	97.7	96.7
2-2	108.0	95.4	108.8	91.7	105.6	96.0	102.0	103.8
2-3	96.2	106.1	104.1	103.7	97.8	91.0	103.8	96.5
2-4	99.9	105.2	94.0	78.0	98.7	98.5	109.8	101.7
2-5	94.8	101.1	92.9	98.9	95.3	89.4	95.2	90.4
2-6	99.7	105.0	97.5	71.9	98.0	99.3	99.9	102.6
2-7	113.1	98.5	107.2	102.3	104.8	96.4	99.0	96.1
2-8	111.1	74.9	100.9	105.2	113.4	92.1	101.8	101.6
2-9	106.4	102.8	99.2	95.5	102.4	93.0	100.0	95.5
2-10	107.7	103.6	99.7	128.2	105.9	95.9	111.1	98.3
2-11	98.9	97.8	96.9	92.5	96.1	85.0	97.9	92.1
2-12	107.0	107.7	99.1	113.7	102.5	93.0	106.1	100.7
2-13	120.1	109.8	122.7	105.0	124.3	94.9	104.7	97.5
2-14	108.9	108.2	104.6	90.2	122.1	99.0	111.3	102.1
2-15	104.7	115.8	103.2	95.4	114.1	93.7	110.0	109.3
2-16	107.8	104.2	93.7	92.5	112.8	99.5	107.3	103.3
2-17	103.2	109.0	97.7	87.6	97.9	94.3	100.3	98.0
2-18	101.9	93.3	97.5	93.4	103.4	92.6	103.0	97.7
3-1	105.6	124.0	103.2		101.6			
3-2	105.6	108.3	103.8		97.7			
3-3	92.1	99.1	88.8		89.6			
3-4	91.7	103.9	89.2		92.9			
3-5	101.2	108.9	107.8		137.9			
3-6	112.0	108.3	114.6		139.7			
3-7	97.4	109.7	89.2		103.7			
3-8	112.5	117.6	102.0		112.3			
4-1	110.4	93.8	112.8		67.9			
4-2	107	111.9	90.6		76.5			
4-3	-	-	-	100% Slag mixture	-			
4-4	104.6	73.8	102.0		76.0			
4-5	96.7	96.1	88.8		79.0			
4-6	103.9	81.3	99.0		83.2			
4-7	106.4	101.3	90.0		86.6			
4-8	103.2	94.2	92.6		81.8			
4-9	101.3	97.0	86.4		87.8			
4-10	99.4	92.4	100.4		90.3			
4-11	103.9	93.6	102.6		93.5			

(Continued)

Table L-1 (Concluded)

PCC Mixture No.	Cement Content: Concrete Quality Monitor	Nuclear Cement Content Gage	Rapid Analysis Machine	WES Centrifuge Procedure	X-ray Emission Spectrometer	Water Content: Concrete Quality Monitor	Hot Plate Procedure	Micro-wave Oven Pro-cedure
4-12	-	-	-	100% Fly ash mix-ture	-			
4-13	98.9	92.6	99.0		90.4			
4-14	104.8	92.7	99.8		92.2			
4-15	117.5	99.1	114.6		121.3			
4-16	133.0	145.7	113.2		136.6			
4-17	103.3	90.6	113.6		99.0			
4-18	112.8	121.2	118.2		117.6			
5-1						91.8	87.8	96.6
5-2						107.6	102.8	91.8
5-3						110.4	110.5	106.9
5-4						112.4	117.8	109.8
5-5						97.7	109.4	106.1
5-6						124.1	108.4	114.0
5-7						114.1	104.4	99.5
5-8						123.7	101.1	99.4
5-9						108.6	122.3	111.8
6-1	107.5	117.8	94.2		100.4			
6-2	105.7	113.6	97.0		102.2			
6-3	100.1	107.3	91.2		94.3			
6-4	100.1	108.8	89.6		91.7			
6-5	106.5	103.7	96.6		112.2			
6-6	99.3	102.4	91.0		101.4			
6-7	96.3	88.2	91.8		103.3			
6-8	92.1	90.0	92.8		97.6			

Table L-2

Task 2 Individual Mean Recoveries

PROCEDURE	MEAN RECOVERY	N	STANDARD DEVIATION
MIX 1			
CQM	110.6	5	5.6
NCCG	92.0	5	3.8
CF	113.7	5	5.3
X-RAY	102.5	5	4.0
RAM	115.6	5	4.6
MIX 2			
CQM	108.0	5	12.5
NCCG	95.4	5	22.9
CF	91.7	5	15.3
X-RAY	105.6	5	7.0
RAM	108.8	5	6.4
MIX 3			
CQM	96.2	5	4.8
NCCG	106.1	5	1.3
CF	103.7	5	4.0
X-RAY	97.8	5	3.7
RAM	104.1	5	3.0
MIX 4			
CQM	99.2	5	2.6
NCCG	105.2	5	3.6
CF	78.0	4	2.0
X-RAY	98.7	5	4.9
RAM	94.0	5	3.4
MIX 5			
CQM	94.8	5	1.9
NCCG	101.1	5	2.7
CF	98.9	5	7.2
X-RAY	95.3	5	3.4
RAM	92.9	5	3.7
MIX 6			
CQM	99.8	5	5.7
NCCG	105.0	5	2.9
CF	71.9	4	1.3
X-RAY	98.0	5	3.3
RAM	97.5	5	4.2

Table L-2 (Continued)

PROCEDURE	MEAN	N	STANDARD DEVIATION
MIX 7			
CQM	113.1	5	8.9
NCCG	98.4	5	9.0
CF	102.2	5	3.7
X-RAY	104.8	5	5.8
RAM	107.3	5	5.2
MIX 8			
CQM	109.7	5	4.5
NCCG	74.9	5	5.9
CF	105.2	5	10.5
X-RAY	113.4	5	13.8
RAM	100.9	5	3.2
MIX 9			
CQM	106.4	5	2.2
NCCG	102.8	5	5.3
CF	95.5	5	10.0
X-RAY	102.4	5	6.8
RAM	99.3	5	1.0
MIX 10			
CQM	107.7	5	4.4
NCCG	103.6	5	1.8
CF	128.1	5	7.5
X-RAY	105.9	5	9.7
RAM	99.7	5	2.9
MIX 11			
CQM	98.9	5	3.4
NCCG	97.8	5	3.0
CF	92.5	5	4.0
X-RAY	95.7	5	8.5
RAM	96.9	5	1.6
MIX 12			
CQM	107.0	5	3.1
NCCG	107.7	5	5.2
CF	113.7	4	22.6
X-RAY	102.5	5	1.7
RAM	99.1	5	4.7

Table L-2 (Continued)

PROCEDURE	MEAN	N	STANDARD DEVIATION
MIX 13			
CQM	120.1	5	4.3
NCCG	109.8	5	13.7
CF	105.0	5	11.1
X-RAY	124.3	5	6.0
RAM	122.7	5	5.3
MIX 14			
CQM	108.9	5	9.3
NCCG	108.2	5	7.5
CF	90.2	5	11.9
X-RAY	122.1	5	7.5
RAM	104.6	5	6.2
MIX 15			
CQM	104.7	5	6.3
NCCG	115.8	5	13.1
CF	95.4	5	8.3
X-RAY	114.1	5	6.3
RAM	103.2	5	8.9
MIX 16			
CQM	107.8	5	4.2
NCCG	104.2	5	2.6
CF	92.5	5	9.2
X-RAY	112.8	5	5.3
RAM	93.7	5	21.9
MIX 17			
CQM	103.2	5	1.2
NCCG	109.0	5	14.0
CF	87.6	5	3.8
X-RAY	97.9	5	4.3
RAM	97.7	5	1.6
MIX 18			
CQM	101.9	5	3.8
NCCG	93.3	5	3.3
CF	93.4	4	7.3
X-RAY	103.4	5	16.3
RAM	97.5	5	5.4

Table L-2 (Continued)

PROCEDURE	MEAN	N	STANDARD DEVIATION
MIX 1			
HOT-PL	97.7	5	15.0
MICRO	96.7	5	12.4
CQM	95.6	5	3.4
MIX 2			
HOT-PL	106.0	5	1.7
MICRO	103.8	5	4.8
CQM	96.0	5	1.4
MIX 3			
HOT-PL	103.8	5	1.8
MICRO	96.5	5	5.2
CQM	91.0	5	6.8
MIX 4			
HOT-PL	109.8	5	21.7
MICRO	101.7	4	4.6
CQM	98.5	5	4.3
MIX 5			
HOT-PL	95.2	5	2.4
MICRO	90.4	5	8.7
CQM	89.4	5	2.1
MIX 6			
HOT-PL	99.9	5	4.0
MICRO	102.6	5	5.9
CQM	99.3	5	3.3
MIX 7			
HOT-PL	99.0	5	7.1
MICRO	96.1	5	7.8
CQM	96.4	5	3.3
MIX 8			
HOT-PL	101.8	5	4.8
MICRO	101.6	5	7.1
CQM	92.1	5	3.6

Table L-2 (Concluded)

PROCEDURE	MEAN	N	STANDARD DEVIATION
MIX 9			
HOT-PL	100.1	5	5.3
MICRO	95.5	5	3.2
CQM	93.0	5	2.5
MIX 10			
HOT-PL	111.1	5	22.5
MICRO	98.3	5	11.6
CQM	95.9	5	5.2
MIX 11			
HOT-PL	97.9	5	4.8
MICRO	92.1	5	6.8
CQM	85.0	5	2.9
MIX 12			
HOT-PL	106.1	5	7.6
MICRO	100.7	5	5.4
CQM	93.0	5	3.7
MIX 13			
HOT-PL	104.7	5	14.6
MICRO	97.5	5	8.0
CQM	94.9	5	6.0
MIX 14			
HOT-PL	111.3	5	9.1
MICRO	102.1	5	11.6
CQM	99.0	5	11.8
MIX 15			
HOT-PL	110.0	5	6.4
MICRO	109.3	5	9.2
CQM	93.7	5	5.1
MIX 16			
HOT-PL	107.3	4	4.5
MICRO	103.3	4	6.6
CQM	99.5	5	6.9
MIX 17			
HOT-PL	100.3	5	7.6
MICRO	98.0	5	6.8
CQM	94.3	5	3.4
MIX 18			
HOT-PL	103.0	5	3.6
MICRO	97.7	5	1.1
CQM	92.6	5	2.1

Table L-3

Task 3 Individual Mean Recoveries

PROCEDURE	MEAN	N	STANDARD DEVIATION	C.V.
MIX 1				
NCCG	124.0	5	6.2	5.0
X-RAY	101.6	5	13.0	12.8
CQM	105.6	5	11.4	10.8
RAM	103.2	5	16.8	16.3
MIX 2				
NCCG	108.3	5	10.5	9.7
X-RAY	97.7	5	11.2	11.4
CQM	105.6	5	9.4	8.9
RAM	103.8	5	19.4	18.7
MIX 3				
NCCG	99.1	5	6.7	6.8
X-RAY	89.6	5	3.2	3.6
CQM	92.1	5	8.1	8.8
RAM	88.8	5	4.2	4.7
MIX 4				
NCCG	103.9	5	7.5	7.2
X-RAY	92.9	5	4.5	4.8
CQM	91.7	5	2.1	2.3
RAM	89.2	5	4.7	5.2
MIX 5				
NCCG	108.9	5	23.4	21.5
X-RAY	137.9	5	10.3	7.5
CQM	101.2	5	22.7	22.4
RAM	107.8	5	11.1	10.3
MIX 6				
NCCG	108.3	5	27.5	25.4
X-RAY	136.3	5	24.3	19.4
CQM	112.0	5	13.7	12.2
RAM	114.6	5	11.3	9.8
MIX 7				
NCCG	109.7	5	6.0	5.4
X-RAY	103.7	5	5.8	5.6
CQM	97.4	5	7.4	7.6
RAM	89.2	5	7.5	8.4
MIX 8				
NCCG	117.6	5	5.0	4.3
X-RAY	112.3	5	6.3	5.6
CQM	112.5	5	4.8	4.3
RAM	102.0	5	4.4	4.3

Table L-4

Task 4 Individual Mean Recoveries

PROCEDURE	MEAN	N	STANDARD DEVIATION	C.V.
MIX 1				
NCCG	93.8	5	0.3	0.3
X-RAY	72.3	5	3.0	12.4
COM	110.3	4	3.1	2.8
RAM	112.8	5	5.3	4.7
MIX 2				
NCCG	111.9	5	6.0	5.4
X-RAY	76.5	5	8.6	11.3
CQM	107.0	5	9.3	8.7
RAM	90.6	5	3.5	3.9
MIX 4				
NCCG	73.8	5	1.3	1.8
X-RAY	73.7	5	5.6	7.6
CQM	104.6	5	10.1	9.6
RAM	102.0	5	8.2	8.0
MIX 5				
NCCG	96.1	5	9.6	10.0
X-RAY	79.0	5	6.7	8.5
CQM	96.7	5	18.3	21.5
RAM	88.8	5	15.5	17.5
MIX 6				
NCCG	81.3	5	9.6	11.8
X-RAY	83.2	5	10.2	12.3
CQM	103.9	5	9.3	9.0
RAM	99.0	5	10.2	10.4
MIX 7				
NCCG	101.3	5	6.1	6.0
X-RAY	86.6	5	10.5	12.1
CQM	106.4	5	9.5	8.9
RAM	90.0	5	4.6	5.2

Table L-4 (Continued)

PROCEDURE	MEAN	N	STANDARD DEVIATION	C.V.
MIX 8				
NCCG	94.2	5	8.2	8.7
X-RAY	81.8	5	6.2	25.4
CQM	103.2	5	3.1	3.0
RAM	92.6	5	3.4	3.6
MIX 9				
NCCG	97.0	5	5.5	5.7
X-RAY	87.8	5	5.4	6.1
CQM	101.3	5	7.9	7.8
RAM	86.4	5	9.2	10.6
MIX 10				
NCCG	92.4	5	3.0	3.3
X-RAY	90.3	3	15.4	58.2
CQM	99.4	5	5.9	5.9
RAM	100.2	5	7.0	7.0
MIX 11				
NCCG	93.6	5	2.9	3.0
X-RAY	93.5	4	9.0	9.7
CQM	103.9	5	8.3	8.0
RAM	102.6	5	10.9	10.7
MIX 13				
NCCG	92.6	5	2.2	2.4
X-RAY	90.4	5	2.2	5.4
CQM	98.9	5	9.1	9.2
RAM	99.0	5	7.8	7.9
MIX 14				
NCCG	92.7	5	2.2	2.3
X-RAY	92.2	5	7.2	15.9
CQM	104.8	5	8.6	8.2
RAM	99.8	5	2.9	3.0
MIX 15				
NCCG	99.1	5	7.1	7.2
X-RAY	121.3	3	9.1	11.0
CQM	117.5	5	4.2	3.6
RAM	114.6	5	14.2	12.4

Table L-4 (Concluded)

PROCEDURE	MEAN	N	STANDARD DEVIATION	C.V.
MIX 16				
NCCG	145.7	5	34.8	23.9
X-RAY	136.6	4	26.8	17.5
CQM	133.0	5	24.0	18.1
RAM	113.2	5	7.6	6.7
MIX 17				
NCCG	90.6	5	4.5	5.0
X-RAY	99.0	4	11.6	
CQM	103.0	5	3.5	3.4
RAM	113.6	5	4.2	3.7
MIX 18				
NCCG	121.2	5	6.1	5.0
X-RAY	117.6	4	3.7	
CQM	112.8	5	8.4	7.4
RAM	118.2	5	4.4	3.8

Table L-5

Task 5 Individual Mean Discoveries

PROCEDURE	MEAN	N	STANDARD DEVIATION
TASK 5			
MIX 1			
HOT-PL	109.9	5	2.7
MICRO	96.6	5	7.0
CQM	91.8	5	6.0
MIX 2			
HOT-PL	102.8	5	9.2
MICRO	91.8	5	8.6
COM	107.6	5	2.6
MIX 3			
HOT-PL	110.5	5	5.6
MICRO	106.9	5	6.6
CQM	110.4	5	4.6
MIX 4			
HOT-PL	117.8	5	16.4
MICRO	109.8	5	8.4
CQM	112.4	5	10.4
MIX 5			
HOT-PL	100.0	5	5.9
MICRO	99.1	5	8.7
CQM	102.4	5	5.3
MIX 6			
HOT-PL	108.4	5	7.9
MICRO	114.0	5	22.3
CQM	124.1	6	7.9
MIX 7			
HOT-PL	104.4	5	10.0
MICRO	99.5	5	6.9
CQM	114.1	6	3.5
MIX 8			
HOT-PL	101.1	5	9.8
MICRO	98.9	5	5.0
CQM	126.3	6	23.6

Table L-5 (Continued)

PROCEDURE	MEAN	N	STANDARD DEVIATION
TASK 5			
MIX 9			
HOT-PL	121.0	5	13.8
MICRO	111.6	5	8.0
CQM	106.7	5	24.3
MIX 10			
HOT-PL	109.1	5	2.7
MICRO	106.1	5	7.0
CQM	97.7	6	6.1

Table L-6
Task 6 Individual Mean Discoveries

PROCEDURE	MEAN	N	STANDARD DEVIATION	C.V.
MIX 1				
NCCG	117.8	5	8.9	
X-RAY	100.4	5	8.4	
CQM	107.5	5	5.1	
RAM	94.2	5	4.1	
MIX 2				
NCCG	113.6	5	4.9	
X-RAY	102.2	5	8.8	
CQM	105.7	5	5.6	
RAM	97.0	5	2.8	
MIX 3				
NCCG	107.3	5	1.6	
X-RAY	94.3	5	1.0	
CQM	100.1	5	3.9	
RAM	91.2	5	1.9	
MIX 4				
NCCG	108.8	5	4.1	
X-RAY	91.7	5	6.3	
CQM	100.1	5	5.7	
RAM	89.6	5	3.8	
MIX 5				
NCCG	103.7	5	3.2	
X-RAY	112.2	5	8.6	
CQM	106.5	5	9.3	
RAM	96.6	5	5.2	
MIX 6				
NCCG	102.4	5	7.1	
X-RAY	101.4	5	5.7	
CQM	99.3	5	4.5	
RAM	91.0	5	5.6	
MIX 7				
NCCG	88.2	5	4.8	
X-RAY	103.3	5	5.6	
CQM	96.3	5	4.9	
RAM	91.8	5	2.0	
MIX 8				
NCCG	90.0	5	4.4	
X-RAY	97.6	5	5.4	
CQM	92.8	5	4.7	
RAM	92.8	5	3.1	

APPENDIX M

INDIVIDUAL PERCENT RECOVERY - TASK

Table M-1

Individual Bias per Test Procedure per Task

	CQM/C[b]	NCCG	RAM	CF	X-ray	CQM/W[b]	HP	MW
Task 2 Mean, %	105.6	101.7	102.0	97.7	105.4			
Standard Deviation, %	6.4	9.1	7.8	13.1	8.6			
N[a]	18	18	18	18	18			
Task 3 Mean, %	102.3	110.0	99.8		109.4			
Standard Deviation, %	8.1	7.7	9.7		19.4			
N[a]	8	8	8		8			
Task 4 Mean, %	106.7	98.6	101.5		92.5			
Standard Deviation, %	8.8	16.5	10.3		18.3			
N[a]	16	16	16		16			
Task 5 Mean, %						110.0	107.2	104.0
Standard Deviation, %						10.6	10.0	7.5
N[a]						9	9	9
Task 6 Mean, %	101.0	104.0	93.0		100.4			
Standard Deviation, %	5.4	10.5	2.7		6.2			
N[a]	8	8	8		8			

[a] Sample size, N, represents the total number of mixtures used in their respective tasks.

[b] CQM/C designates the cement portion of the test procedure and CQM/W designates the water portion of the test procedure.

APPENDIX N

INDIVIDUAL PERCENT RECOVERY - PROCEDURE

Table N-1

Individual Bias per Test Procedure

	Mean Recovery, %	Standard Deviation %	Sample Size, N[a]
CQM/C	104.7	7.5	50
NCCG	102.4	12.3	50
RAM	100.0	8.8	50
CF	97.7	13.1	18
X-ray	101.1	15.2	50
CQM/W	99.6	10.0	27
HP	104.6	7.1	27
MW	100.7	6.0	27

[a] Sample size, N, represents the total number of mixtures in which the procedure was evaluated.

Table N-2

Individual Bias per Individual Factor for COM - Cement Method

	Mean Recovery, %	Standard Deviation, %	Sample Size, N[a]
Low cement factor	110.5	8.0	14
Moderate cement factor	99.4	23.5	18
High cement factor	99.9	5.4	18
Calcareous aggregate	106.6	9.0	22
Siliceous aggregate	102.7	4.9	18
Blended aggregate	103.9	7.3	10
Fly ash	109.2	11.5	8
Ground slag	104.2	4.1	8
Naphthalene HRWRA	N/A		
Melamine HRWRA	N/A		
Calcium chloride	101.0	5.4	8
Prolonged mixing	102.3	8.1	8

[a] Sample size, N, represents the total of the individual mixtures where each factor was included.

Table N-3

Individual Bias per Individual Factor for NCCG

	Mean Recovery, %	Standard Deviation, %	Sample Size, N[a]
Low cement factor	105.8	16.4	14
Moderate cement factor	102.7	10.0	18
High cement factor	99.4	10.5	18
Calcareous aggregate	104.5	13.7	22
Siliceous aggregate	100.0	10.6	18
Blended aggregate	102.1	12.2	10
Fly ash	103.5	19.8	8
Ground slag	93.7	11.7	8
Naphthalene HRWRA	N/A	-	-
Melamine HRWRA	N/A	-	-
Calcium chloride	104.0	10.5	8
Prolonged mixing	110.0	7.7	8

[a] Sample size, N, represents the total of the individual mixtures where each factor was included.

Table N-4

Individual Bias per Individual Factor for CF

	Mean Recovery, %	Standard Deviation, %	Sample Size, N[a]
Low cement factor	101.3	8.9	6
Moderate cement factor	98.9	16.6	6
High cement factor	93.0	13.7	6
Calcareous aggregate	94.0	6.0	6
Siliceous aggregate	93.0	15.8	6
Blended aggregate	106.2	13.1	6
Fly ash	N/A		
Ground slag	N/A		
Naphthalene HRWRA	N/A		
Melamine HRWRA	N/A		
Calcium chloride	N/A		
Prolonged mixing	N/A		

[a] Sample size, N, represents the total of the individual mixtures where each factor was included.

Table N-5

Individual Bias per Individual Factor for RAM

	Mean Recovery, %	Standard Deviation, %	Sample Size, N[a]
Low cement factor	106.1	9.0	14
Moderate cement factor	99.6	8.6	18
High cement factor	95.7	6.3	18
Calcareous aggregate	101.5	10.7	22
Siliceous aggregate	98.9	7.8	18
Blended aggregate	98.8	5.9	10
Fly ash	107.7	7.9	8
Ground slag	95.3	8.8	8
Naphthalene HRWRA	N/A		
Melamine HRWRA	N/A		
Calcium chloride	93.0	2.7	8
Prolonged mixing	99.8	9.7	8

[a] Sample size, N, represents the total of the individual mixtures where each factor was included.

Table N-6

Individual Bias per Individual Factor for X-ray

	Mean Recovery, %	Standard Deviation, %	Sample Size, N[a]
Low cement factor	110.2	19.6	14
Moderate cement factor	99.3	14.1	18
High cement factor	95.9	8.4	18
Calcareous aggregate	108.9	17.0	22
Siliceous aggregate	91.8	10.4	18
Blended aggregate	100.7	6.9	10
Fly ash	105.1	17.7	8
Ground slag	79.9	6.5	8
Naphthalene HRWRA	N/A		
Melamine HRWRA	N/A		
Calcium chloride	100.4	6.2	8
Prolonged mixing	109.4	19.4	8

[a] Sample size, N, represents the total of the individual mixtures where each factor was included.

Table N-7

Individual Bias per Individual Factor for CQM - Water Method

	Mean Recovery, %	Standard Deviation, %	Sample Size, N[a]
Low cement factor	95.7	2.2	6
Moderate cement factor	95.3	3.3	6
High cement factor	102.9	12.4	15
Calcareous aggregate	93.4	26.3	10
Siliceous aggregate	100.1	8.3	11
Blended aggregate	92.6	4.1	6
Fly ash	N/A		
Ground slag	N/A		
Naphthalene HRWRA	110.2	11.3	8
Melamine HRWRA	108.6	-	1
Calcium chloride	N/A		
Prolonged mixing	N/A		

[a] Sample size, N, represents the total of the individual mixtures where each factor was included.

Table N-8

Individual Bias per Individual Factor for HP

	Mean Recovery, %	Standard Deviation, %	Sample Size, N[a]
Low cement factor	102.8	4.9	6
Moderate cement factor	107	4.3	6
High cement factor	104.5	8.6	15
Calcareous aggregate	106.0	3.8	10
Siliceous aggregate	104.5	10.0	11
Blended aggregate	102.7	5.0	6
Fly ash	N/A		
Ground slag	N/A		
Naphthalene HRWRA	105.3	8.8	8
Melamine HRWRA	122.3	-	1
Calcium chloride	N/A		
Prolonged mixing	N/A		

[a] Sample size, N, represents the total of the individual mixtures where each factor was included.

Table N-9

Individual Bias per Individual Factor for MW

	Mean Recovery, %	Standard Deviation, %	Sample Size, N[a]
Low cement factor	99.6	3.3	6
Moderate cement factor	100.8	5.1	6
High cement factor	101.2	7.3	15
Calcareous aggregate	102.7	5.6	10
Siliceous aggregate	100.8	7.0	11
Blended aggregate	97.4	3.5	6
Fly ash	N/A		
Ground slag	N/A		
Naphthalene HRWRA	103.0	7.4	8
Melamine HRWRA	111.8	-	1
Calcium chloride	N/A		
Prolonged mixing	N/A		

[a] Sample size, N, represents the total of the individual mixtures where each factor was included.

APPENDIX O

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