THE UNIVERSITY OF CALGARY

USE OF CONTROL SPECIMENS DURING FREEZE-THAW DURABILITY TESTING

BY

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The undersigned certify that they have read, and recommend to the Faculty of Graduate Studies for acceptance, a thesis entitled, "Use of Control Specimens during Freeze-Thaw Durability Testing," submitted by John H. Rutherford in partial fulfillment of the requirements for the degree of Master of Science in Engineering

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Abstract

The principal objective of this investigation was to examine the use of *control specimens* to improve the interpretation of results from the ASTM C 666 — 84 (Procedure A) test. This test involves the resistance of concrete to rapid freezing and thawing in water.

The control specimens were tested over a range of durability, involving various curing histories and material ratios.

This investigation established that control specimens compensate for non-test related variations, including internal changes in the test specimens that other control methods cannot compensate for. These internal changes can create an uptake of water in the specimens causing an increase in weight and relative dynamic modulus readings which are not possible for a deteriorating concrete.

The control specimens provide a continuous reference for the test specimens giving a *before and after* picture for visual assessment.

The results indicate that use of control specimens during the ASTM C 666 — 84 (Procedure A) test improves the interpretation of the test data.

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Chapter 1

Introduction

1.1 Background

The ability to assess the potential freeze-thaw durability of concrete is particularly important in countries such as Canada where climatic conditions in the winter months are severe. Such assessments are important for long-term evaluation of new mix designs and of insitu performance of existing concrete.

The assessment should involve physical and visual testing. The physical testing can be divided into field and laboratory methods. The field testing is usually the most time consuming and costly but will give direct or absolute values for evaluation. Laboratory testing is usually more economical but produces only relative values for comparison. The visual assessment normally complements the physical testing (ie. observations of scaling, pitting or cracking).

The laboratory test which is the subject of this investigation is designed to emulate long-term field conditions in a rapid freeze-thaw simulation. It provides a relative assessment of the frost resistance of concrete which can be evaluated against the performance of control specimens or standards.

In 1928, Scholer [15] presented a paper describing "Some accelerated freezing and thawing tests on concrete". Since that presentation many papers have been written on the subject, with individual laboratories developing their own testing methods. Over the years efforts have been made to study factors that influence these tests and to standardize the method of testing.

In 1945, a newly developed apparatus for automatically performing rapid freeze-thaw tests in a laboratory setting was described in detail by Wuerpel and Cook [20]. The continual development of this apparatus and its associated specifications has lead to today's ASTM C 666 — 84 (C 666) [25] the standard test method for resistance of concrete to rapid freezing and thawing. During the life of the test, measurements can be taken on such concrete properties as expansion, weight loss, scaling and dynamic modulus of elasticity. It is the measurements of the dynamic modulus and its relative change during the period of the test that are the prime concerns in this investigation.

C 666 compares the relative change in the fundamental transverse frequency reading of a specimen during the test period to the specimen's initial frequency reading prior to the start of the test. The square of the specimen's frequency reading (n_1) at a given number of freeze-thaw cycles is divided by the square of the specimen's initial frequency (n) reading at 0 freeze-thaw cycles. This ratio is then multiplied by 100 to give a product called the *relative dynamic modulus of elasticity* (RDM) at the given number of freeze-thaw cycles. The equation is given below:

$$RDM = (n_1^2/n^2)\,(100)$$

This calculation of RDM is based on the assumption that the weight and dimensions of the specimen remain constant throughout the test.

The *durability factor* (DF) of the specimen can then be calculated as shown:

$$DF = RDM\,(N/M)$$

where:

- N is the lesser of the number of cycles at which RDM reaches the specified minimum value for discontinuing the test or the specified number of cycles at which exsposure is to be terminated and
- M is the specified number of cycles at which the exsposure is to be terminated.

The frequency is created by a driving circuit consisting of a variable frequency audio oscillator, an amplifier and a power source. The readings are taken transversely to the specimens length by the use of low frequency ultrasonic transducers (refer to ASTM C 215 — 85 and Appendix B for further descriptions).

The freeze-thaw test involves a cooling and heating cycle between 4.4 and $-17.8°C$ at a cycle rate of 2 to 5 hours. The specimen can be frozen in water (Procedure A) or in air (Procedure B). The thawing is in water for both procedures. This study deals only with Procedure A, acknowleged as the most aggressive and most commonly used of the two procedures. Since this procedure was developed from ASTM C 290 both standard reference numbers (C 666 and C 290) will be used when discussing a reference depending on which standard was used in the reference.

As previously mentioned the C 666 test is a relative test. This implies the test results require referencing to control results to reduce variations due to the operator, the equipment and the concrete itself.

Concrete is a time-dependent material, i.e. as it ages its properties change due to the influence of the immediate environment and its internal hydation process.

Since the C 666 procedure requires testing of the concrete to begin at a relatively young age (14 days) the potential for change is great. The standard test procedures do not involve any compensation for change in the specimen's frequency reading due to concrete changes such as weight variations through water uptake or fluctuations attributable to the dynamic testing apparatus.

1.2 Scope

This investigation examines the use of control specimens to improve the interpretation of the ASTM C 666 — 84 (Procedure A) test data. Specifically to reduce non-test related variations to the frequency readings caused by internal changes to the concrete specimens during the test.

Various air contents, compressive strengths and curing regimes were used to create a range of durabilities to study the effect of control specimens.

The following chapters of this thesis are briefly described below:

Chapter 2 is a literature review which covers the history of freeze-thaw durability testing of concrete and the development of the associated testing apparatus.

Chapter 3 describes the manufacturing and testing procedures used during this investigation. Specific characteristics of the materials and machines used are included.

Chapter 4 presents and discusses the results of the testing.

Chapter 5 summarizes the investigation by way of conclusions and recommendations for future study .

1.3 Applications

If control specimens are shown to be effective in reducing non-test related variations in frequency readings their inclusion in the ASTM testing procedure would compensate for differences in curing histories. This would reduce the present need for long soaking periods for dried specimens from field or steam cured concretes.

During testing, fluctuations of the instruments or a change in operators that would affect readings would be accommodated since the effect would be seen in the control specimen as well as in the test specimen.

The control specimen would give a continuous reference point for observation tests such as scaling. A *before and after* picture of the specimen condition would be readily available at all stages of testing.

Chapter 2

Literature Review

The effects of frost action on concrete have been studied since the early 1900's. Many of the early ideas about frost action were brought together by Powers in 1945 [13] when he developed "A Working Hypothesis for further Studies of Frost Resistance of Concrete". Working from the acquired knowledge of the freezing of water and the composition of concrete, a mechanism of frost action on concrete was developed. During his research Powers found that there were many variables that affect the resistance of concrete to frost action. The concrete variables he found most prominent were the degree of saturation, permeability, compressive strength and air content of the concrete as well as changes in these properties during the maturing process. These properties affected the hydraulic pressures generated during freezing which in turn affected the degree, distribution, and significance of cracking.

In 1955 Powers [14] reiterated the above and defined the mechanism of dilation, the first step in deterioration resulting from the hydraulic pressures produced by the growth of capillary ice and osmotic pressure within cement paste. After the initial dilation the disintegration process takes place and can be observed by the reduction in the concrete's weight, strength and resilience accompanied by surface deteriorations. Powers noted that this process in nature occurs under many conditions with no one condition being typical. The combination of concrete's various properties and external factors acting on the concrete in any given situation made absolute testing of frost resistance difficult. Powers therefore stressed the need for standard relative testing.

Since 1945, when Wuerpel and Cook [20] published their paper "Automatic Accelerated Freezing and Thawing Apparatus for Concrete", there have been numerous investigations into the control of machines to test the durability of concrete with great success. The control of the concrete being tested is another matter. The studies of concrete with regards to the testing and subsequent analysis have had mixed results. The apparatus used to test freeze-thaw durability of concrete has improved since 1945 but the variability of the test results and subsequent analysis is still a major concern. The standardization of the test procedure and control methods will reduce the variability of the test.

Cordon in 1966 [4] supported this, noting that C 290 was the most consistent of the testing methods used. The test was more severe than the natural environment since it could create many freeze-thaw cycles per day (the normal test involved 7 cycles per day) where nature may take one or more winters to create the same number of cycles. Cordon observed that this test could discriminate among concretes and the proposed materials for concrete work in a relatively short time. This made the test a very useful tool in the evaluation process of concrete.

One of the limits of the test was how it was affected by water-content change in the specimens. Both Powers and Cordon found the test results to vary due to the concrete's history, particularly the water content and its distribution. This variability was confirmed by in P. Kleiger's work in 1952 and 1956 [7,8] where he recorded relative dynamic elasticity modulus greater than 100% which is physically impossible for a deteriorating concrete subjected to such a severe test as C 290.

This brought about the need for an examination of the variability of the test procedure and apparatus. In 1954 the Corps of Engineers, U.S. Army, performed some "Cooperative Freezing and Thawing Tests of Concrete Specimens" [17] to identify the magnitude and causes of variation in results of tests of aggregate. They originally planned to develop data that would identify suitable revisions to the ASTM C 290 test method in order to reduce test variations within a single laboratory and between laboratories. Although they were able to identify the magnitude and some causes of the variations they found that reproduction of the data was inadequate to reduce the effect of the variables of the testing procedure.

They found the average durability factors for various laboratories involved in the study had a variance well beyond the standard deviation of the averages. The standard deviations of the average durability factors for the individual laboratories were also highly variable.

In 1959, the Highway Research Board [5] produced another cooperative study covering a range of concretes and laboratories. Their results also showed high variability with poor reproducibility. Large variations in durability factors were observed for the same concrete mixtures and for tests by the same method, both within and between laboratories. It was noted that these variations were probably due both to differences in the concretes as prepared in the labs and in treatment of the specimens by a given test method. The four test methods investigated were:

1. Resistance of concrete specimens to rapid freezing and thawing in water
2. Resistance of concrete specimens to rapid freezing in air and thawing in water
3. Resistance of concrete specimens to slow freezing and thawing in water or brine

4. Resistance of concrete specimens to slow freezing in air and thawing in water

The four test methods investigated varied in the way they differentiated among the concretes. Each of the tests was a different combination of test variables and these combinations affected the results differently. Examples are:

- The rapid tests generally created lower durability factors and less distinction between results than the slow tests for the same type of concrete.

- The freezing in air tests would allow drying of the specimens.

Due to the combinations of variables the individual user of each method could rationalize the data to favour his method.

This report established the rapid freezing and thawing in water test (ASTM C 290) to be the most severe. Extreme durability results, either poor or good had lower standard deviations which they surmised were probably due to the severity of the test. Once a concrete started to fail early in the test, it failed quickly. In the case of good freeze-thaw durability when all the required concrete properties (air content, degree of saturation, strength and permeability) were fulfilled the deterioration process created by the test had minimal effect on the concrete resulting in no durability factor loss.

The variability of results in the middle range of durability (durability factors of 40 to 80) was found to be the highest. This is caused by the heterogeneous nature of concrete and the many possible combinations of shortcomings of the concrete properties and their effect on the rate of deterioration. This is usually the range of most concern during testing since the effect of deterioration on the service life of concrete is not precise and the variability of the results is high.

The report's findings were that the C 290 test was a good indicator of relative concrete durability but that many factors including as air content and degree of saturation were negatively affecting the reproducibility of the results.

Tyne in 1966 [16] found that the opportunity for specimens to dry could drastically change the durability results. Test methods or curing histories that allowed the degree of saturation to vary for the same concrete would alter the final durability result.

In 1971, Blaine and Arni [1] extended Tyne's work by investigating a series of concrete mixes that were subjected to laboratory freeze-thaw tests. Their report was divided into three sections covering:

1. durability aspects,
2. water loss and absorption and
3. dynamic modulus of elasticity.

The freeze-thaw test method used was C 290. Their data indicated that the air content and degree of saturation of the concretes generally had the greatest effect on the durability results. Higher air content mixes gave lower initial dynamic modulus readings and allowed for greater change in the degree of saturation as moisture conditions changed. These changes were detailed in the second section. The amount of change (i.e. weight gain) was dependent on time and temperature with higher air contents having higher weight gains. The range of weight gain was between two and four percent. Associated with the weight gains was an increase in the dynamic modulus, (i.e. the greater the air content, the greater the opportunity for water gain, therefore the greater the dynamic modulus readings). Their results demonstrated the detrimental effect that increases in C_2S, K_2O, M_gO and the fineness of the cement can have on the dynamic modulus of concrete.

After freeze-thaw testing the deteriorated specimens were placed in a fog room where autogenous healing enabled the concretes to regain most or all of their original dynamic modulus.

The preceding work demonstrates that there are many factors that can affect the dynamic modulus reading and the subsequent calculation of durability factors. During the freeze-thaw test the degree of saturation and the opportunity for autogenous healing must be controlled or referenced.

The effects of the degree of saturation on freeze-thaw testing were reaffirmed in 1973 by Wong, Anderson and Hilsdorf [19]. They found that a short drying period prior to the commencement of the testing increased the durability factor of a concrete. This initial drying period affected the degree of hydration, freezable water content and permeability of the concrete. The main result was that the drying period reduced the water content well below saturation and created minute shrinkage cracks which incresed the air voids in the concrete allowing a greater reduction in the hydrostatic pressure during freezing with minimal or no effect on strength. This study established the optimal drying period for improved durability to be three days drying after an initial seven day wet curing. This indicated that concretes, due to curing history, can have improved freeze-thaw durability which suggests that the curing history must be considered in evaluation of test results.

2.1 Summary

The literature review has brought forward some of the factors that affect freeze-thaw durability results causing poor reproducibility. Many of the researchers referred to the degree of saturation or moisture content of specimens as one of the major factors. In accepting this the writer was of the opinion that control specimens could compensate for water uptake caused by variations in the degree of saturation of the specimens due to curing history or hydration during the test, since frequency readings and weight changes to the control specimens would be of the same type as those to the test specimens.

Chapter 3

Manufacturing and Testing

3.1 The Materials

3.1.1 Cement

Normal portland cement (*Type 10*) supplied by Canada Cement LaFarge Limited located at Exshaw, Alberta was used in all the mixes. The cement was received in bags and stored in overhead hoppers until required. The cement conformed to CSA 3-A23.1-M77 [21]. The chemical composition of the cement is given in Table 3.1.

Table 3.1: Chemical Composition of the Type 10 Cement, March 1986

Oxides			Compounds		
Notation		Name	Notation		Name
CaO	63.4%	lime	C_3S	55.7%	tricalcium silicate
SiO_2	21.3	silica	C_2S	18.9	dicalcium silicate
Al_2O_3	4.4	alumina	C_3A	7.7	tricalcium aluminate
Fe_2O_3	2.4	ferric oxide	C_4AF	7.1	tetracalcium aluminoferrite
M_gO	4.6	magnesia			
Na_2O	0.2	alkalis	LoI	1.7	loss on ignition
K_2O	0.9	alkalis			
SO_3	2.7	sulfur trioxide			

3.1.2 Water

Potable water from the University's main water supply was used for mixing. The water was allowed to stand for one hour to stabilize at room temperature prior to use. This ensured good control of the water temperature used in the mixes. The room temperature at mixing was $22 \pm 2°C$.

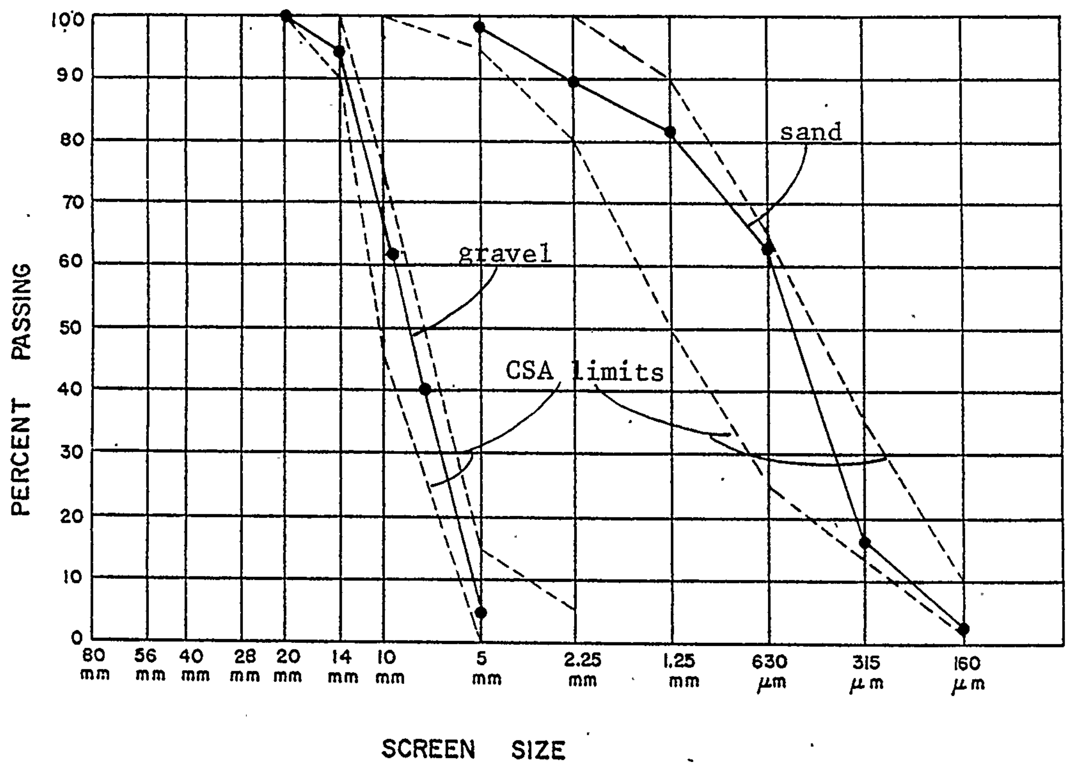
3.1.3 Admixture

The air entrainment admixture used during this investigation was a surface active chemical (called a surfactant). It was manufactured by Master Builders and is received as a clear dark brown liquid. Its designation is *M.B. A.E. 10* and it conformed to CSA 3-A266.1-M78 [23] and ASTM C 260-86 [25]. This admixture was blended with one litre of water before adding to the mix.

3.1.4 Fine Aggregate

The fine aggregate (sand) was mainly composed of quartzite and limestone. It was from a local alluvial deposit and was supplied by Consolidated Concrete Ltd. of Calgary. This aggregate was received dry in 1100 kg bags and stored in 45 gallon drums to minimize segregation and improve uniformity prior to use. The specific gravity and absorption of the sand were 2.64 and 1.4% respectively. The physical properties and gradation curve of the sand are given in Table A.1 of Appendix A and in Figure 3.1 respectively. All of the above properties were periodically checked and conformed to the requirements of Section 5 of CSA 3-A23.1-M77 [21].

Figure 3.1: Gradation of Aggregate March 1986



3.1.5 Coarse Aggregate

The coarse aggregate (gravel) used in this project is referred to as 14 *mm* nominal size. This material is a natural river gravel containing approximately 75% quartz and 25% limestone or their associated minerals and is commonly used for ready-mix curb and gutter concrete in the Calgary area. It was supplied by Consolidated Concrete Ltd. of Calgary in a damp condition and stored in an overhead hopper. It had a specific gravity of 2.66 and an absorption of 1.0%. The average percent of crushed material was 5% and the received moisture content varied between 1 and 2 percent. The grading of the aggregate as received satisfied Section 5 of CSA 3-

A23.1-M77 [21]. The results of gradation analysis are given in Figure 3.1 and the physical properties are listed in Table A.2 of Appendix A.

3.2 The Mix Design

Two major objectives of the mix design were to:

1. have mixes that were comparable to local ready-mix curb and gutter designs which are commonly exposed to freeze-thaw conditions and to

2. create a range of concrete durabilities over which the use of control specimens could be evaluated.

To accomplish the *first* objective a group of trial mixes were run. These mixes were cast to assess the water requirements and the air entrainment dosage level of the concrete produced from the 0.05 M^3 batch size. There were three criteria governing the *water requirements* of the mixes:

1. The curb and gutter mix design of the local ready-mix producers.

2. The required workability for curb and gutter concrete and for laboratory compaction.

3. The compressive strength range used by the local producers.

The curb and gutter designs of the local ready-mix suppliers served as the starting point for the design of the test mixes. An average of the material ratios of cement to sand to gravel (**C-S-G**) from the local mixes were used in the test mixes. This would permit future comparison of the results by user groups with their own product.

The requirements of the workability were for proper compaction in the laboratory by the external vibratory table to ensure a uniform specimen density and to be close to the workability range of the local ready-mix producers. The workability for this investigation was defined by the slump test, CSA 3-A23.2-5C [22] and the slump range was established at 40 – 60 *mm.*

The third criteria of the water requirement was to create a compressive strength range that covered the strength ranges used by the local producers. Trial mixes were cast to ensure that the strength range normally used for local curb and gutter mixes was met. Although compressive strength can be secondary to air-content or permeability when dealing with freeze-thaw durability of concrete, it is still a requirement in most specifications at this time. In this study 7 and 28 day compressive strengths were recorded. A total of 5 trial mixes were cast.

The *air-content*, in conjunction with permeability, is the most important factor affecting the durability of concrete under freeze-thaw conditions [13] therefore the dosage control of air entrainment is critical. The trial mixes provided the opportunity to verify the dosages required to create concrete with two air-content ranges, a reasonable or *normal air* content series and a borderline or *low air* content series. This will be discussed at length in the following sections.

The *second* objective of the mix design, creating concrete mixes of various freeze-thaw durabilities, was accomplished by various combinations of *three* factors.

These factors were:

1. The use of two air-content ranges.

2. The use of three material ratio groups.

3. The use of various curing regimes.

The first factor, *air-content* was divided into two ranges, a normal total air-content (**NA**) 6 – 7% and a low total air content (**LA**) 3.5 – 4%. The normal range conformed to the locally used specifications of 5+% or 5 – 8% air-content in the concrete. Note that for severe exposure conditions such as during ASTM C 666 method A, ACI 201 [27] recommends a minimum of 6 – 7% air-content for this class of concrete. These air-contents should allow for more than the minimum 9% air-content in the mortar fraction of the concrete and should give high durability results. The low air-contents would result in lower than the 9% air content in the mortar thus creating lower durability results for comparison.

The second factor chosen to vary the durability of the concrete was the *compressive strength* of the mix. The compressive strengths were governed by the **C-S-G** ratios of the various mix designs used by the local ready-mix suppliers. The ratios used are shown in Table 3.2. In addition to the material ratios the compressive strength of the test mixes would vary due to the final water/cement ratio and air-content of the mix. The idea was to initially test the control specimens with durable mixes and then reduce the durability by lowering the air-content and compressive strength to observe how the they responded.

Table 3.2: Average Mix Designs used by Local Ready-Mix Suppliers

Material Ratio Cement-Sand-Gravel	Water/Cement Ratio	Strength*
1 - 1.4 - 2.5	0.40	35 MPa
1 - 2.3 - 4.0	0.48	27.5
1 - 2.3 - 3.3	0.50	25.0

The Air Content Averaged 6.5 %

The Slump Range was 20-40 mm

* The Strength is the Design Target 28 Day Compressive Strength used by the Suppliers.

The third factor, the *curing regimes*, is external to the mix design and is covered under Laboratory Procedures and Fresh Concrete Tests.

3.3 Mix Description

The laboratory work was divided into 2 series of mixes, the normal air-content (**NA**) and the low air-content (**LA**) series.

3.3.1 Normal Air-Content Series

The purpose of the five mixes of the **NA** series was to create a quality mix with respect to durability and to test control specimens of the mixes under various curing regimes. These mixes had a combination of two air-contents and two material ratios. The final mix designs are given in Table 3.3.

Table 3.3: Final Mix Designs — Normal Air-Content (NA)

Mix Material Ratio Cement-Sand-Gravel	Use in Mixes	Water/Cement Ratio	Air	Slump
1 - 1.4 - 2.4	NA 1	0.41	6.5 %	50 mm
	NA 2			
1 - 2.2 - 4.1	NA 3	0.50	6.5 %	50 mm
	NA 4			
	NA 5			

Note the only admixture used during this investigation was an air entraining agent whereas the ready-mix suppliers additionally use a water reducing agent. This required a slightly higher water/cement ratio by mass to attain the similar workabilities. This study examined the testing of the end product thus the water reducer was seen as an extra variable that was not required.

The proportioning of the mixes shown in Table 3.3 created a paste fraction which is slightly higher than the average curb and gutter mix. The supplier's paste range was 23 – 25% by volume while the investigation's paste range was 24 – 32% by volume. This was due to the choice of the 14 *mm* coarse aggregate rather than the 19 *mm* or 25 *mm* coarse aggregate used by some suppliers. The intent here was to reduce the influence of the aggregate during the testing and to create more paste which is the fraction of the concrete that is most affected by the freeze-thaw testing.

3.3.2 Low Air-Content Series

The LA series initially consisted of three strength groups cured in two regimes, a total of 7 mixes. These mixes are shown in Table 3.4. This series was designed to evaluate the control specimen method on low air-content mixes where the water uptake would differ due to the lower air-contents and lower durabilities.

Table 3.4: Final Mix Designs — Low Air-Content (LA)

Mix Material Ratio Cement-Sand-Gravel	Use in Mixes	Water/Cement Ratio	Air	Slump
1 - 1.4 - 2.4	NA 1	0.41	4.0 %	50 mm
	NA 2			
1 - 2.2 - 4.1	NA 3	0.50	4.0 %	50 mm
	NA 4			
1 - 2.6 - 3.8	NA 5	0.55	3.5 %	50 mm
	NA 6			
	NA 7			

3.4 Laboratory Procedures and Fresh Concrete Tests

3.4.1 Aggregate Preparation

The fine aggregate was air dried in the laboratory prior to batching to ensure a uniform moisture content at the time of use. Periodic sieve analyses ensured consistent grading.

The coarse aggregate was batched dry and pre-soaked for 24 hours. After the pre-soak period there was a one-hour drain period prior to mixing. At the start of mixing a sample was taken for the calculation of the extra (free) water on the coarse aggregate. This free water was included as part of the mix water. This procedure allowed for absorption by the coarse aggregate which in turn improved the repeatability of the mixes and reduced slump loss. The total 25 hour standing period of the aggregate in the laboratory also permitted temperature stability.

Both aggregates were weighed to an accuracy of $\pm 0.5\%$. All mixing water and admixtures were allowed to stand for one hour to stabilize at room temperature. The above was done in accordance with CSA 3-A23.2-2C-M77 [22].

3.4.2 The Mixing

A 0.085 M^3 horizontal rotary paddle mixer was buttered prior to each mixing to reduce the loss of mortar from the test mixes. The buttering consisted of coating the mixer with a small premix mortar which had the same proportions as the mortar fraction of the test mix. After the buttering the coarse aggregate, fine aggregate and cement (in that order) with approximately two thirds of the mixing water were then blended in the mixer for approximately 3 minutes to allow for initial absorption of water by the sand. The air entraining agent - water mixture and the remainder of the mixing water were then added. This took approximately one minute with the mixer operating. After all ingredients were added the mixing continued for three more minutes followed by a two minute soak period and a final two minute mixing period. The concrete was bottom dumped into a wheelbarrow for movement at the end of this final mixing period. The mixing procedure was in accordance with CSA 3-A23.2C-M77 [22].

3.4.3 Workability Test

The workability test used throughout this project was the slump test. The slump of each mix was measured to the nearest 5 *mm* just prior to casting. The slump test was performed twice as a check. After each test the sample was reblended into the batch. This is in compliance with CSA 3-A23.2-5C-M77 [22].

3.4.4 Density Test and Air Content

For each mix of concrete the plastic density was determined using the seven litre bowl of an air pressure meter in accordance with CSA 3-A23.2-6C-M77 [22]. Following the density test the air content of the concrete was determined by CSA 3-A23.2-4C-M77 [22]. As in the case with workability, these tests were repeated with two different samples to check the initial values. Compaction for both of these tests was achieved using a vibration table as per CSA 3-A23.2-3C-M77 [22]. The pressure-meter bowl was filled in two lifts in each case.

3.4.5 Casting and Moulds

During casting the moulds were held firmly on an Allen Standard vibrating table (refer to Appendix B), filled and vibrated in two approximately equal layers. Vibration was stopped when the surface became relatively level or the egress of entrapped air bubbles ceased.

The compressive strength specimens were formed in 150 mm diameter by 300 mm high cylindrical non-absorbent plastic moulds. These moulds were reusable, having a small hole in their base through which air pressure could be applied to release the specimen.

The freeze-thaw specimens were cast in 75 mm by 75 mm by 375 mm rectangular, steel moulds. A release agent was applied to the internal surfaces of all moulds prior to the casting of the specimens. The above procedures and moulds were in compliance with CSA 3-A23.2-3C-M77 [22].

3.4.6 Curing

At the end of casting the specimens were placed on a laboratory table and covered with a polyethylene sheet for 24 ± 2 hours, after which the compressive strength specimens were demoulded and placed in the fog room ($23 \pm 1.7°C$ and 90 – 100% relative humidity). The durability specimens were demoulded and transferred as a group to one of the following curing regimes.

1. 100% relative humidity and $23 \pm 1.7°C$
2. 50% relative humidity and $23 \pm 1.7°C$
3. 100% relative humidity and $5 \pm 1.7°C$

4. combination of 1 and 2.

Regime 1 was accomplished by immersion of specimens in water within the fog room. The specimens were placed in water-filled buckets and allowed to cure for 14 days.

Regime 2 was the result of specimen placement in an environment controlled room. This room had humidity and temperature controls which allowed the room to be set at $50 \pm 3\%$ relative humidity and $23 \pm 2°C$. Continuous monitoring of the relative humidity and temperature was by strip chart recorders.

Regime 3 was created by placing the specimens in a $5°C$ water bath. This water bath was a 0.6 M^3 stainless steel tank with a freon based refrigeration unit. A Camless temperature programer controlled the bath temperature and a thermocouple gave continuous monitoring of the bath temperature. This apparatus doubled as the conditioning bath. Additional information can be found in Appendix B.

Regime 4 was a combination of 7 days curing in the fog room and 7 days curing in the environment room. This regime was used to create a water uptake situation that was between Regime 1 and Regime 2.

The above conditions were selected to create a range of durability results when combined with the various mix strengths and air contents. The regimes also allowed a comparison between moist curing and air curing with respect to water uptake and variability in the readings during the testing. The length of curing was 14 days for all cast specimens.

3.5 Low Pressure Steam Cured Precast Mix

3.5.1 Background

In the precast concrete industry, steam curing allows increased production by reducing the turnover time of the moulds and formwork through shorter curing periods. The ACI Recommended Practice 517 [27] outlines low (atmospheric) pressure, steam curing of concrete.

The optimum temperature range is $65 - 80°C$, with the final temperature selection depending on the requirements of the product. These elevated temperatures during the curing process create a product that continually uptakes water during a freeze-thaw test which in turn affects the transverse fundamental frequency readings of the samples being tested. The inclusion of the low pressure steam cured precast mix in this investigation was to add a commonly used pretest curing history.

3.5.2 The Test Element

The procedure for the precast mix differed from the laboratory cast mixes. An element that will be subjected to freeze-thaw cycles during its use was received from a precast plant. The specimens were then cut from the element using a Highland Park Slāb (Model 24ss) Oil Saw with a 40 *cm* diamond blade. They were then cleaned of oil and presoaked for 48 hours in the fog room prior to the testing. Six specimens were cut, four for the freeze-thaw test and two as controls. A characteristic of this mix was the cutting of the specimens, creating a different boundary condition for water uptake and scaling during the testing. The mix particulars given in Table 3.5 were supplied by the precaster.

Table 3.5: Low Pressure Steam Cured Precast Mix Characteristics

Cement, TYPE 30	416 kg/m^3
Water	166
19 mm Aggregate	1140
Sand	645
Water Reducer	114 ml/cement weight
Air Entrainment	57 ml/cement weight
Slump	50 mm
Air Content	5.9 %
Unit Weight	2302 kg/m^3
Concrete Temp	22.2 °C
Curing Time	14.5 hrs.
Max Curing Temp	65.5 °C
Release Strength	32.6 MPa
28 Day Strength	54.0 MPa

3.6 Hardened Concrete Tests

3.6.1 Compressive Strength

Compressive strength testing was performed at 7 and 28 days. Two specimens were cast for the 7 day test and four specimens for the 28 day test. Cylinders for the strength testing were cast for each strength group. All cylinders were capped with a sulphur - flyash compound. This compound was 60% sulphur and 40% flyash by mass. It had a 2 hour strength of 39 MPa and a 24 hour strength of 52 MPa. The strength values were from a 50 mm cube compressive strength test as prescribed in ASTM C 109-86 and ASTM C 617-85 [25]. The concrete testing was carried out on a 2000 kN Amsler compression machine using the 1000 kN range. This testing was in accordance with CSA 3-A23.2-9C-M77 [22] and ASTM C 39-86 [25]. The compressive tests were used as a measure of quality control for the mixes.

3.6.2 Freeze-Thaw Durability

The reference specification for this test is ASTM C 666-84, Procedure **A** —(Resistance of Concrete to Rapid Freezing and Thawing in Water). There were five specimens cast from each batch of concrete for the durability testing. At the end of the 14 day curing period the weight was recorded for each specimen. All cast specimens were then transferred to a conditioning bath ($5.6 \pm 2.8°C$) for a period of 4 hours to allow for temperature stabilization with ambient conditions. The fundamental transverse frequency and weight (for a second time) were then recorded. After taking the readings, three specimens picked at random were placed in the freeze-thaw durability bath. The remaining two specimens were left in the conditioning bath as control specimens.

The precast specimens were transfered to the conditioning bath at the end of the 48 hour presoak where they were temperature stabilized for 4 hours. The weight and transverse fundamental frequency readings were then recorded and 4 specimens picked at random were placed in the durability bath with the two remaining specimens left in the conditioning bath as controls.

The durability bath continuously reproduced cycles within the temperature range of 4.4 to $-17.8°C$. One complete cycle took 3 hours. At intervals of 36 cycles or less, weight and frequency measurements were repeated for each specimen after a 2 hour conditioning period.

Visual observation of specimen scaling took place during each reading set. The condition of the surface was rated from 0 to 5, with 0 being no scaling and 5 being severe scaling. This rating is described in ASTM C 672-84 [25].

The equipment used for the transvers fundamental frequency readings consisted of a Hewlett Packard Spectrum Analyzer (Model 3580A) with James $24Khz$ ultrasonic transducers (Model C 4891), attached by coaxial cables. The freeze - thaw bath was manufactured by M & L Testing Equipment Company (Model 3185). Refer to Appendix B for detailed equipment descriptions.

Chapter 4

Results and Discussion

4.1 Introduction

The combination of air content, strength and curing conditions created a durability range to test the usefulness of *control specimens.* The results of the laboratory testing prior to the freeze-thaw test are tabulated in Table 4.1 and Table 4.2. Table 4.1 is the data on the individual mixes and Table 4.2 is the data on the mix groups.

Review of the tables indicates each of the mixes was consistent with design criteria and that the desired ranges of air content and strength were achieved.

The use of the two material ratios in the **NA** Series created two strength ranges with compressive strengths at 28 days of 34.7 MPa and 27.5 MPa. The series had a slump range of 50 – 60 mm and an air content of 6 to 7%.

Three material ratios were used in the **LA** Series to create three ranges of compressive strength, 37.5 MPa, 33.0 MPa, and 24.6 MPa at 28 days. This series had a slump range of 40 – 50 mm and an air content of 3.5 to 4.0%.

As a check on the consistency of the resulting mix characteristics some comparisons were made in terms of compressive strength vs water/cement ratio and density vs air content. As shown in Figures 4.1 on page 32 the characteristics were consistent between mixes and with published literature [11,12].

Table 4.1: Individual Test Results prior to Freeze-Thaw Test

Group	Mix Number	Curing Regime	Slump mm	Density Kg/M3	Air %	W/C	Strength MPa
1.NA	NA1	fog	60	2282	6.0 %	0.41	35.4
	NA2	50 % RH	50	2266	6.5 %	0.40	34.0
2.NA	NA3	fog	60	2224	7.0 %	0.49	26.4
	NA4	50 % RH	55	2228	7.0 %	0.51	29.5
	NA5	5 °C	60	2251	6.5 %	0.49	26.9
1.LA	LA1	fog	50	2344	4.0 %	0.41	37.3
	LA2	50 % RH	45	2340	4.0 %	0.41	37.8
2.LA	LA3	fog	40	2366	4.0 %	0.50	33.4
	LA4	50 % RH	40	2358	4.0 %	0.52	32.6
3.LA	LA5	fog	45	2363	3.5 %	0.56	24.4
	LA6	50 % RH	45	2363	3.5 %	0.56	24.4
	LA7	fog&50 % RH	40	2370	3.5 %	0.55	25.0

Table 4.2: Group Test Results prior to Freeze-Thaw Test

Group	Slump mm	Density Kg/M3	Air %	W/C	Strength MPa
1.NA	50-60	2274	6.2 %	0.40	34.7
2.NA	50-60	2234	6.8 %	0.50	27.5
1.LA	50-60	2342	4.0 %	0.41	37.5
2.LA	40-50	2362	4.0 %	0.51	33.0
3.LA	40-50	2365	3.5 %	0.56	24.6

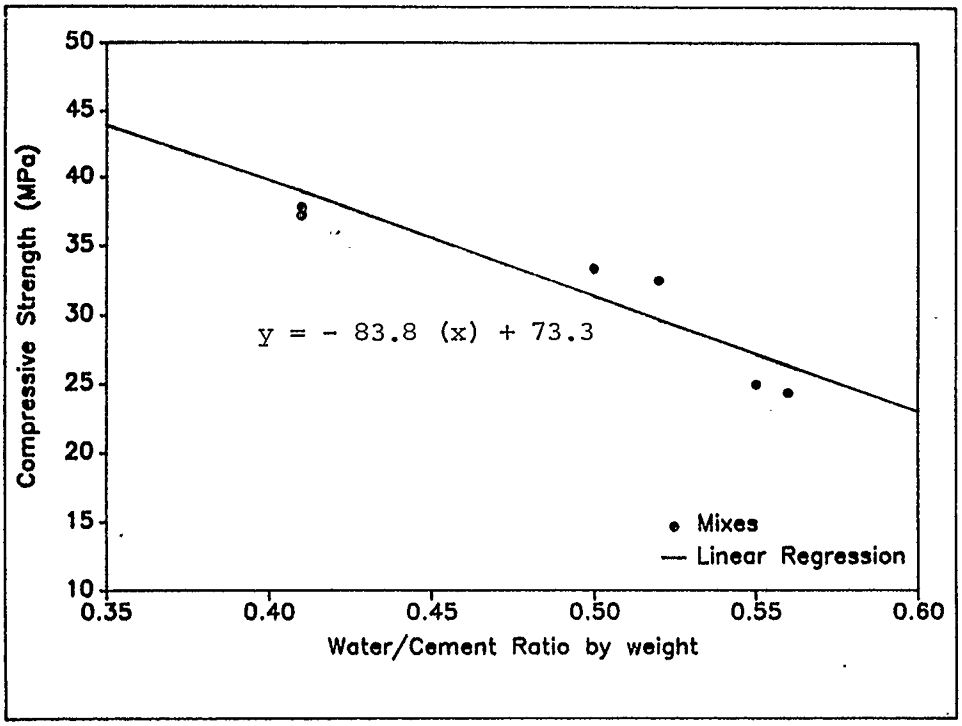


Figure 4.1: Strength vs Water/Content for LA Series

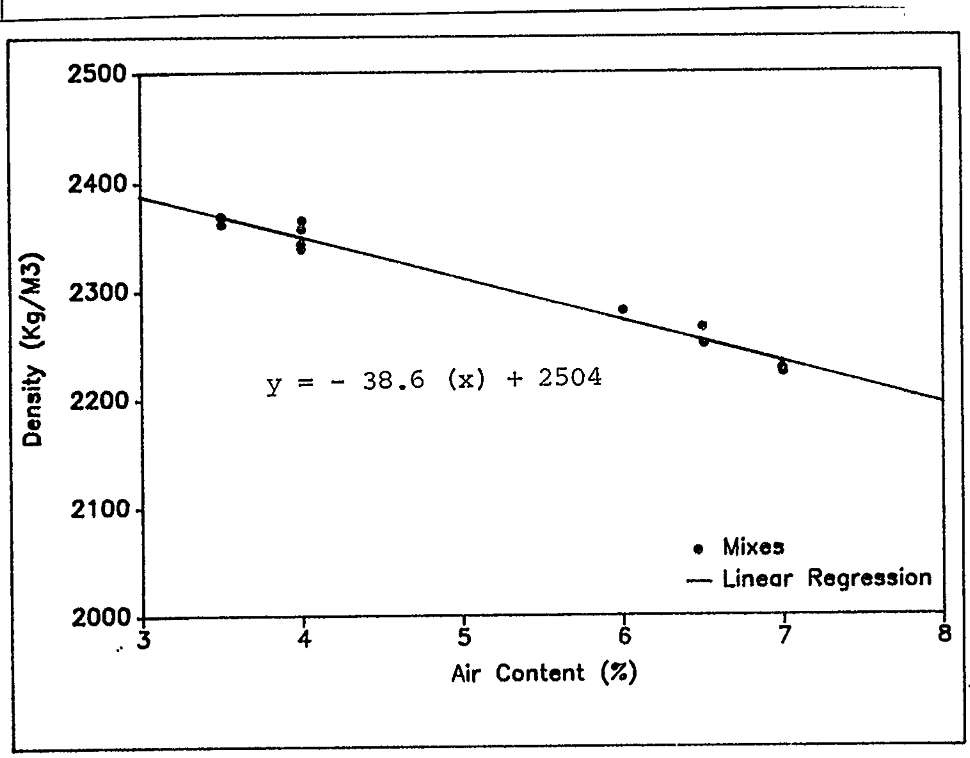


Figure 4.2: Density vs Air Content for All Mixes

4.2 The Freeze-Thaw Test Variations

There are many factors that cause variations in frequency readings during a freeze-thaw test that are independent of the deterioration effect on the readings. Some factors that the author observed were equipment stability, change in operator, curing regime effects and difficulty in obtaining a sound transducer coupling to the sample as the sample deteriorated during the test.

The *equipment* was calibrated initially ($\pm 3\%$) but as the test proceeded it was subject to electrical fluctuations resulting from noise in the connections and cables. To counter these variances, a reference was required. This reference would identify the variance of the readings that was due to the equipment.

During the study a second *experienced operator* was employed to take readings to study the effect of changing the operator. This change could occur in a single laboratory during follow-up testing or replacement of the first operator by a different operator. The variation in operator could be between laboratories as well as within a single laboratory. The various effects that operators can have on the test were described in the cooperative testing [17] discussed in the literature review.

The *specimen's internal structure* can create a number of variations. Since the concrete is immersed in water during the period of test, hydration will continue. This hydration or *maturity* is affected by both time and temperature, thus researchers have developed functions that express maturity of concrete in terms of time and temperature of curing. To apply the concept of maturity there must be a datum temperature below which there is *no* increase in strength with time. The value established by different researchers for this temperature has ranged from

$-10°C$ to $-20°C$ with the most common value being $-10°C$. These values are below the ASTM C 666 upper temperature of $5°C$ which suggests that some hydration is to be expected during the durability test which in turn would affect the fundamental frequency readings. [11]

Deterioration of the concrete surface as the test progresses can make transducer coupling to the specimen difficult. This can reduce the reception of the frequency signal and create more variation in the readings. Deterioration of the concrete as the test progresses can cause microcracking, creating voids which will affect the transverse fundamental frequency readings.

Water uptake due to unsaturated or short curing histories can increase weight and fundamental frequency readings with time, which can cause durability factors during the test to be greater than their initial value; this is not realistic. The frequency signal created by the equipment responds to the density of the specimen. If the specimen absorbs water during the test its density increases which in turn increases the signal. This signal increase usually takes the form of increased frequency readings until the water uptake is complete.

When time or cost restraints are involved it is not always feasible to soak the specimens for a sufficient length of time to ensure moisture equilibrium before testing. Field or precast steam-cured specimens are usually well below their saturated moisture content when cut for testing and would require long soaking times for the water uptake to stabilize. This not only delays the test results but changes the working condition of the specimens before the testing is started.

4.3 Reduction of Variation due to Control Use

4.3.1 The Control Methods Used

In this investigation three control methods were tested, the control specimen method, an aluminum block and an old concrete block of the same size as the test specimens. The aluminum block measured 100 mm by 100 mm by 510 mm and weighed 14.6 kg. The concrete specimen was 10 years old with a relatively consistent transverse fundamental-frequency reading. It measured 75 mm by 75 mm by 380 mm and had a weight of 5.0 kg. Both the aluminum block and old concrete have been used by other operators as control blocks.

4.3.1.1 The Control Blocks

Table 4.3 shows the statistical analysis of the frequency readings of the two control blocks. The statistical analysis involved a two part calculation,

- first, the application of standard statistical formulas to the average of the frequency readings taken on the blocks and
- second, the calculation of the relative dynamic modulus (RDM) of the blocks, averaging the RDM and applying the statistical formulas to the RDM average. The RDM calculation procedure was the same as in ASTM C 666.

The comparison of the frequency calculations indicates the average values for both operators on the aluminum block were similar and both were higher than the concrete block. In all cases the standard deviation over the life of the test was approxiately 1%.

Table 4.3: Statistical Analysis of Control Blocks

Transverse Fundamental Frequency Readings

Cycles	Operator 1 Aluminum Block	Operator 1 Concrete Block	Cycles	Operator 2 Aluminum Block
0	1755	1730	0	1783
2	1755	1700	44	1775
15	1755	1700	80	1754
36	1745	1700	115	1783
51	1750	1723	152	1769
66	1770	-	187	1776
95	1791	1700	223	1781
131	1792	1700	244	1758
167	1775	1685	277	1766
203	1760	1665	308	1736
238	1770	1665		
274	1760	1690		
295	1760	1682		
328	1750	1691		

Statistics in Terms of Frequency Readings

Average	1760 Hz	1696 Hz	1768 Hz
Stdev.	17	17	15
Stdev. in %	0.97	1.00	0.86
Variance	288	275	206

Statistics in Terms of Relative Dynamic Modulus

Average	101 %	93 %	98 %
Stdev.	2.08	1.59	1.68
Variance	3.78	3.35	2.53

The RDM calculations have a standard deviation between 1.5 and 2.1%. This slight increase is due to the power function involved in the calculation which would square any error in the readings. The 2.1% standard deviation applies to all the figures presented in the thesis which involve RDM.

The average RDM of the aluminum block varied between operators. This means that the control block varied differently for the two operators during the test. In this case the difference was 3% (101% – 98%). This variation would have to be accounted for if readings between operators are compared. The variability due to an operator change in this investigation would be a combination of the above.

4.3.1.2 The Control Specimen

As previously mentioned five durability specimens were cast from each mix. After the initial 4 hour conditioning of the specimens in the $5°C$ conditioning bath three specimens were placed in the freeze-thaw apparatus as *test specimens* and two specimens were left in the conditioning bath as *control specimens.* This was a random selection process. It was found during this investigation that the control specimens could account for the previously stated test variations. Since all five specimens have the same curing history until the start of the test, the control specimens act as a continually adjusting reference for the test specimens. This means that the control specimens are not only the initial state of the concrete, but represent the changes to the concrete that are independent of the deterioration of the concrete due to the imposed freeze-thaw conditions.

At present the control methods of Table 4.3 can evaluate an equipment variation or operator change but they do not reference the changes within the concrete during the testing ie. the frequency increase due to water uptake. Of the three methods only the control specimens could reference the variations to the readings due to changes in the concrete. One of the control blocks could be used if variances of the concrete and of the equipment were to be isolated but for normal use of the freeze-thaw test the resulting durability factor of the concrete is the main concern. With this in mind the control specimens separate the effect of variations on the test specimens from the actual deterioration of the test specimen during the freeze-thaw testing. This will be elaborated on in the following sections.

4.3.2 The Calculation Procedures

The calculations involved the comparison of the average of the test specimen weight and frequency readings to the average of the control specimen weight and frequency readings for a given mix and curing regime. These calculations were applied at selected cycles of freeze-thaw during the period of the test. The procedure and example calculations for the 105 cycle on Table 4.4 are shown below:

1. average the *test specimen* weight and frequency readings for a given cycle.
   - weight average $5279 + 5156 + 5273 = 15708/3 = 5236$
   - frequency average $1580 + 1560 + 1550 = 4689/3 = 1563$
2. average the *control specimen* weight and frequency readings for a given cycle.
   - weight average $5251 + 5225 = 10476/2 = 5238$
   - frequency average $1628 + 1627 = 3256/2 = 1628$

3. set the initial average of the test specimens equal to the initial average of the control specimens and adjust the following averages of the test specimens by the same amount. This is done for weight and frequency readings.

   - at 0 cylces the average weight of the test specimens is 5233 and the average weight of the control specimens is 5230. The difference, 3 is subtracted from the following averages of the test specimens. For the 105 cycle the *adjusted weight average* becomes $5236 - 3$ or 5233.
   - at 0 cycles the average of the test frequency is 1565 and the average of the control frequency is 1594. The difference, 29 is added to the following averages of the test specimens. For the 105 cycles the *adjusted frequency average* becomes $1563 + 29$ or 1592.

4. now the averages of the control specimens for each date are the initial readings (n) as described in ASTM C 666 and the adjusted averages of the test specimens are the fundamental tranverse frequency at various cycles of freezing and thawing (n_1).

   The formulas in ASTM C 666 are now applied using n and n_1. These formulas are:

$$RDMc = (n_1^2/n^2)\,(100)$$

   - RDMc = relative dynamic modulus of elasticity (RDM), after c cycles of freezing and thawing, in percent,
   - n = fundamental transverse frequency at 0 cycles of freezing and thawing, and
   - n_1 = fundamental transverse frequency after c cycles of freezing and thawing.

$$DF = RDMN\ (N/M)$$

- DF = durability factor of the test specimen average,
- RDMN = relative dynamic modulus of elasticity at N cycles, in percent,
- N = number of cycles at which RDM reaches the specified minimum value for discontinuing the test or the specified number of cycles at which the exposure is to be terminated, whichever is less, and
- M = specified number of cycles at which the exposure is to be terminated which is usually taken as 300 cycles.

5. compare the new procedure to the old procedure for RDM calculations.

6. convert the weight averages (adjusted test and control) into percentage and subtract the adjusted test weight from the control weight for the selected cycles to get the *net* weight change, (-ve is a loss and +ve is a gain on Table 4.4)

   - the adjusted average of the test specimens at 105 cycles is divided by the adjusted average of the test specimens at 0 cycles $(5233/5230 * 100) - 100 = 0.057\%$ shown as 0.06.
   - the subtraction is $0.153 - 0.057 = 0.096\%$ shown as -0.10.

The above procedure was found simpler and more efficient than other methods such as drying the specimen containers and weighing the material left in them.

The use of the averages rather than the individual readings in the calculations was to give a consistent or standard value for each of the mixes. The author believes that the mix rather than the individual specimen is the critical factor, therefore the values used should represent the mix.

Table 4.4: Example of Weight and RDM Calculations

NA1 35 MPa FOG ROOM

Cycles		0	36	68	105	142	175	216
1. Average of Test Specimens								
Weight	(gms)	5233	5237	5237	5236	5230	5229	5224
Frequency	(Hz)	1565	1549	1567	1563	1580	1567	1572
2. Average of Control Specimens								
Weight	(gms)	5230	5235	5238	5238	5240	5245	5244
Frequency	(Hz)	1594	1594	1628	1628	1626	1613	1615
3. Adjusted Test Averages								
Weight	(gms)	5230	5234	5234	5233	5227	5226	5221
Frequency	(Hz)	1594	1578	1596	1592	1609	1596	1601
4. RDM, New Procedure		100(%)	98	95	94	98	98	98
5. RDM, Old Procedure		100(%)	98	100	100	102	100	101
6. Weight Averages in Percent (%)								
test specimens		0.00	0.08	0.08	0.06	-0.06	-0.08	-0.17
control specimens		0.00	0.10	0.15	0.15	0.19	0.29	0.27
net change		0.00	-0.02	-0.08	-0.10	-0.25	-0.36	-0.44

During this investigation all specimens were tested to at least 300 cycles with no specimen dropping below the industry established termination relative dynamic modulus value of 60%. This means the relative dynamic modulus (RDM) and durability factor (DF) for a given average are the same. For clarity in this study the relative dynamic modulus will be used in the same context as the durability factor.

4.4 Application of Control Specimen Method

4.4.1 The Normal Air Content Series

As previously mentioned there were two strength ranges and three curing regimes used in this series totaling 5 mixes. Table 4.5 presents a summary of the results of the durability testing for the **NA** Series. Note that the results are presented at 300 cycles. This series exhibited high durability with the relative dynamic modulus ranging between 85% and 100%.

Table 4.5: NA Series Durability Test Results — New Procedure

Mix	RDM at 300 cycles (%)	Weight Loss (%)
NA1	98.5	0.8
NA2	102.0	0.4
NA3	88.0	1.0
NA4	94.0	2.3
NA5	95.0	4.1

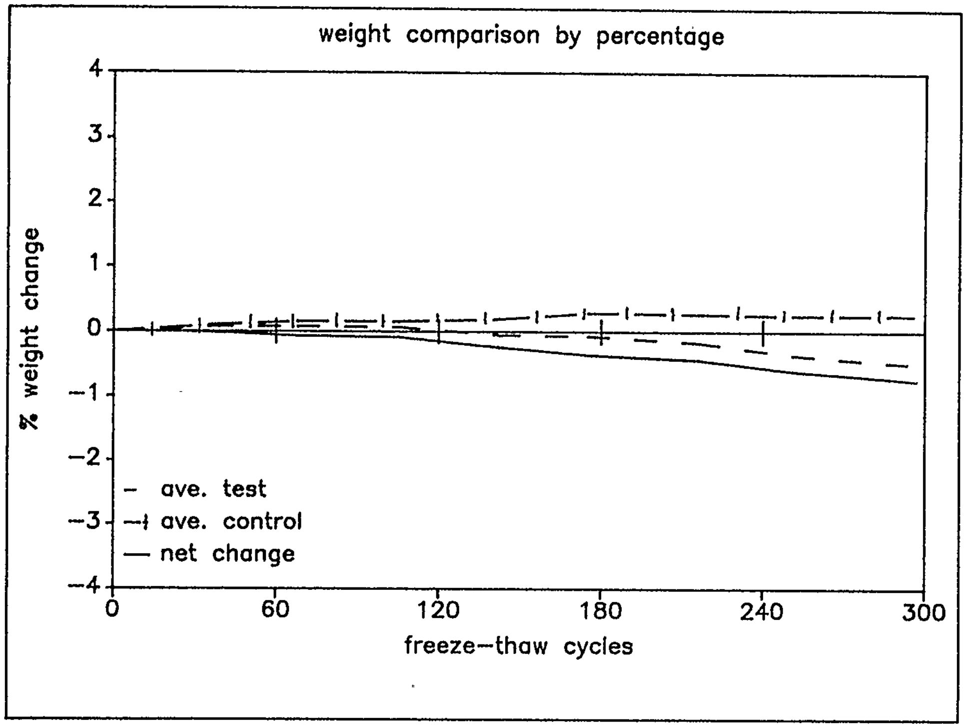
Weight Comparisons in Figures 4.3 to 4.7 indicate that control specimens of all the mixes had weight increases during the test period through water uptake. The enviroment-room mixes (Figure 4.4 and Figure 4.6), as expected, had the greatest uptake of water and associated weight increase. These mixes had a two part water uptake. The first part was a rapid rate of gain due to the concrete being well below saturation at the begining of the test. This rapid gain took place in the first 30 to 40 cycles and was in the order of 0.8% by weight. The first part of the water uptake was inversely proportional to the degree of saturation caused by the pretest curing regimes. The higher the degree of saturation, the lower the rate of water uptake. The second part of the water uptake occured over the duration of the test and was in the order of 0.2% by weight. The second part of the water uptake was principally due to the hydration requirements of the specimen, which is why the second part of the water uptake was essentially the same for all the mixes.

The difference between the average weight readings of the control specimens and the average weight readings of the test specimens during the durability test was dependent on the degree of saturation of the specimens at the begining of the test. This in turn was dependent on the curing regime, with the enviroment room cured mixes experiencing the greater difference.

This weight gain of the control specimens, as shown in Figure 4.6, continued after the test specimens started losing weight through deterioration due to the effect of the test. This means that after adjusting for the water requirements of hydration the actual material loss from the specimens would be the difference between the average weight readings of the control and test specimens at a given cycle count. This difference was greater than the material loss recorded by the weight readings of the test specimens. This was found to occur with mixes cured in the fog room as well as the mixes cured in the enviroment room.

The amount of material lost from a specimen depended on:

1. The severity of the pretest curing regime of the specimen. The greater the severity, the greater the material loss for a given strength group. In this study the the $5°C$ bath was the most severe curing regime with the fog room



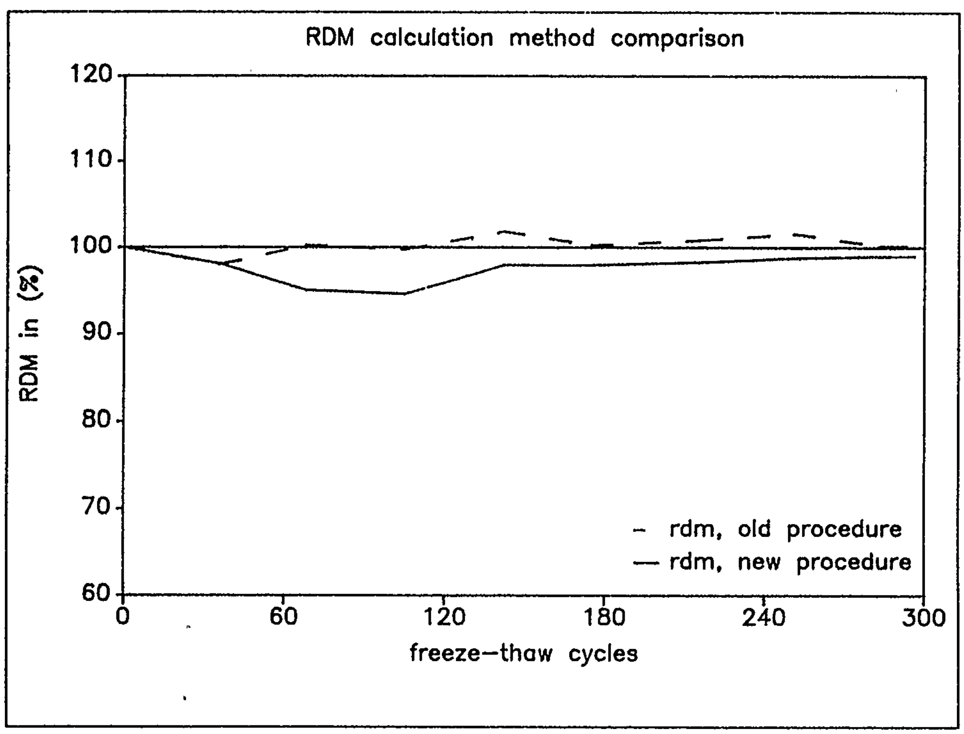


Figure 4.3: Weight and RDM Calculation Method Comparisons of NA 1

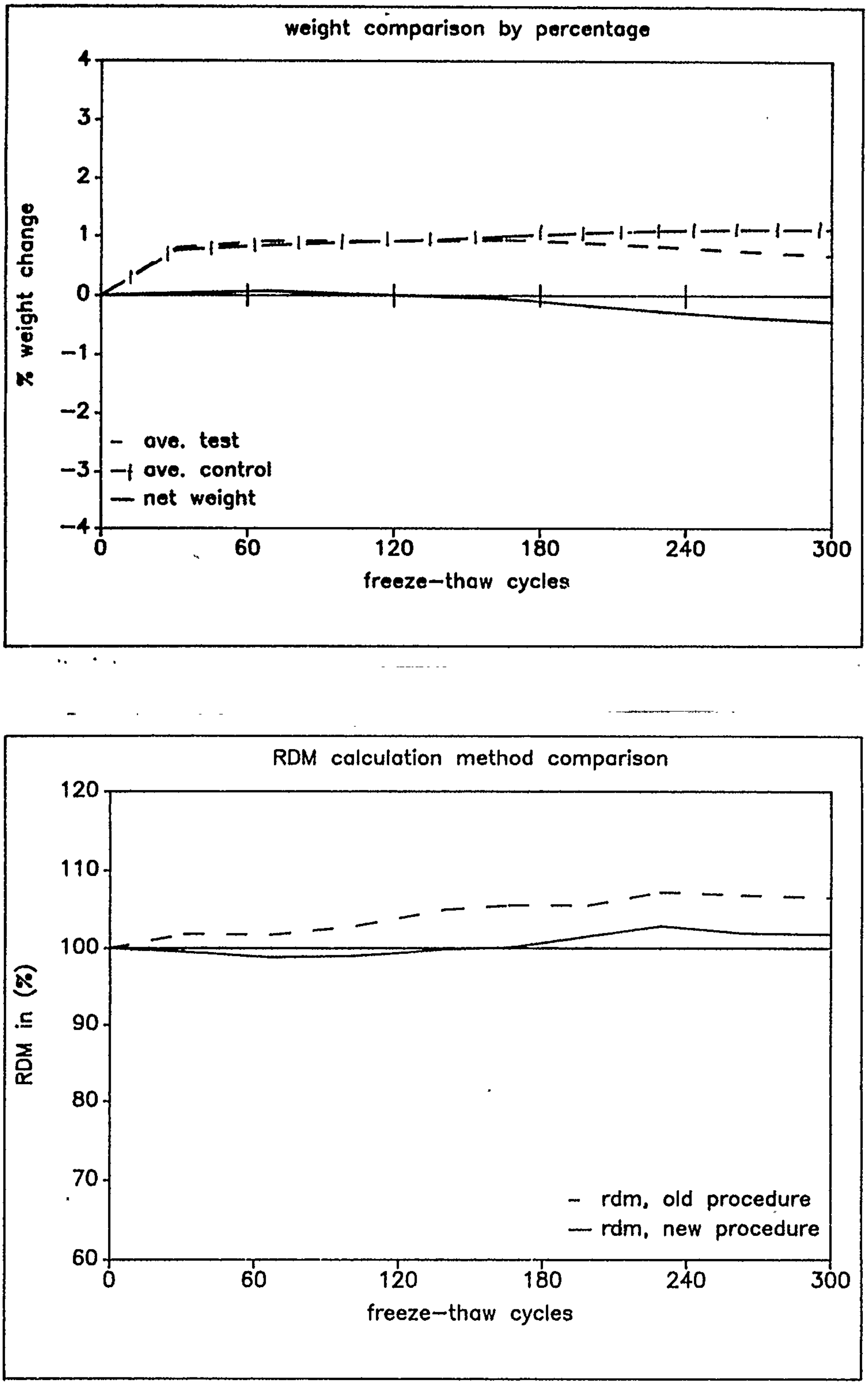


Figure 4.4: Weight and RDM Calculation Method Comparisons of NA 2

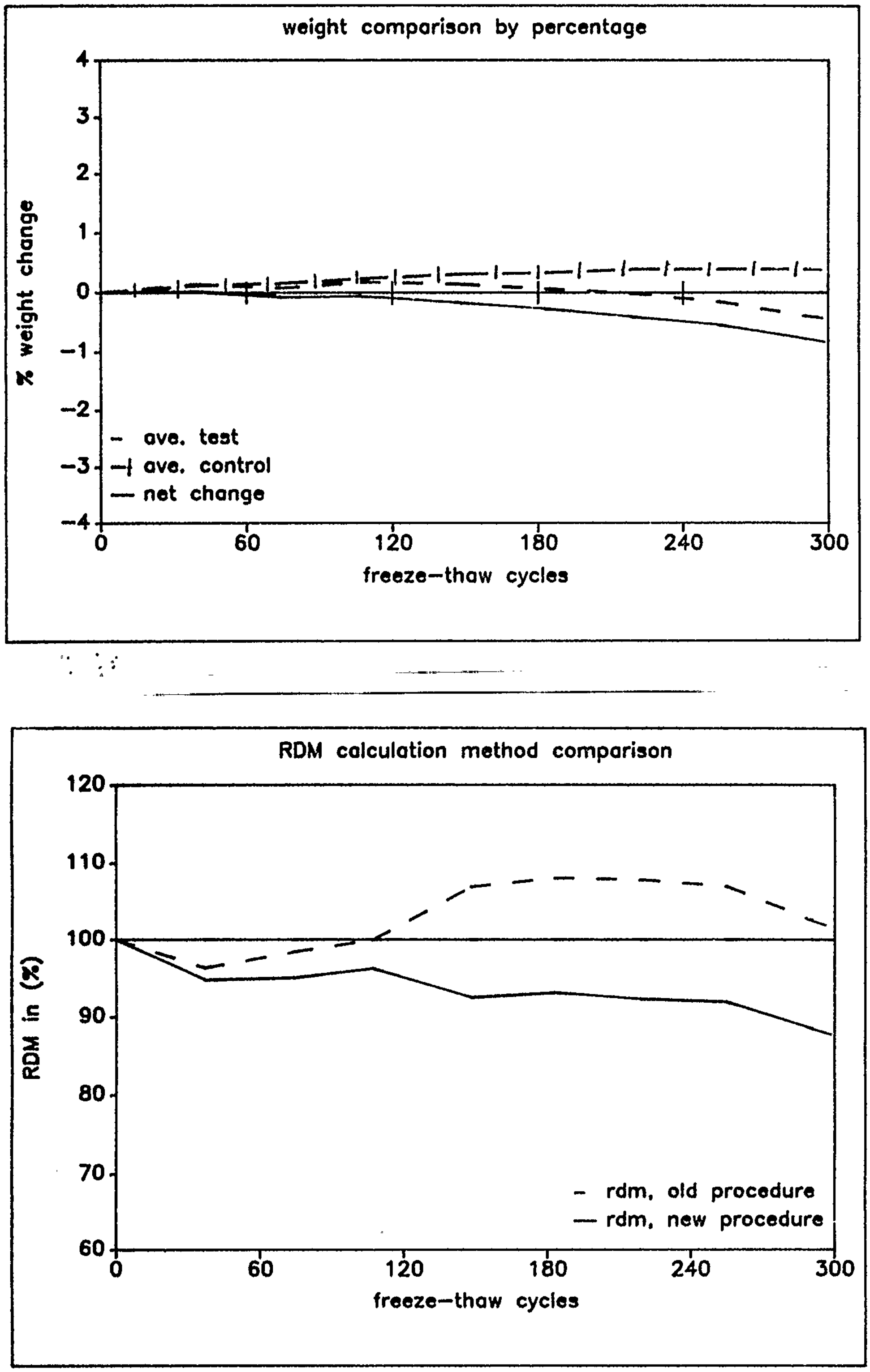
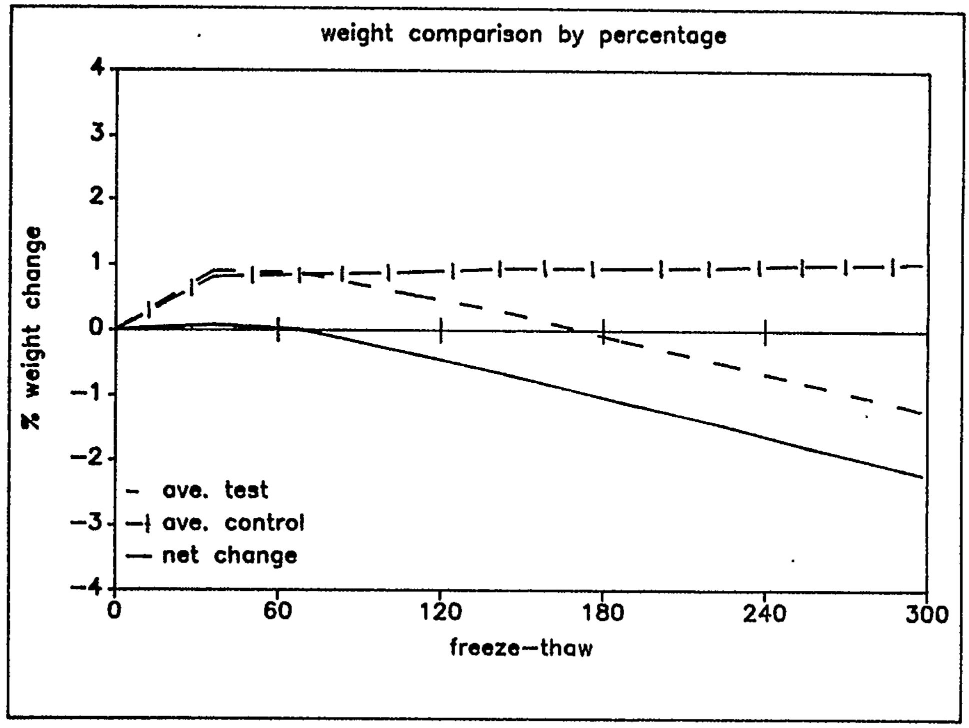


Figure 4.5: Weight and RDM Calculation Method Comparisons of NA 3



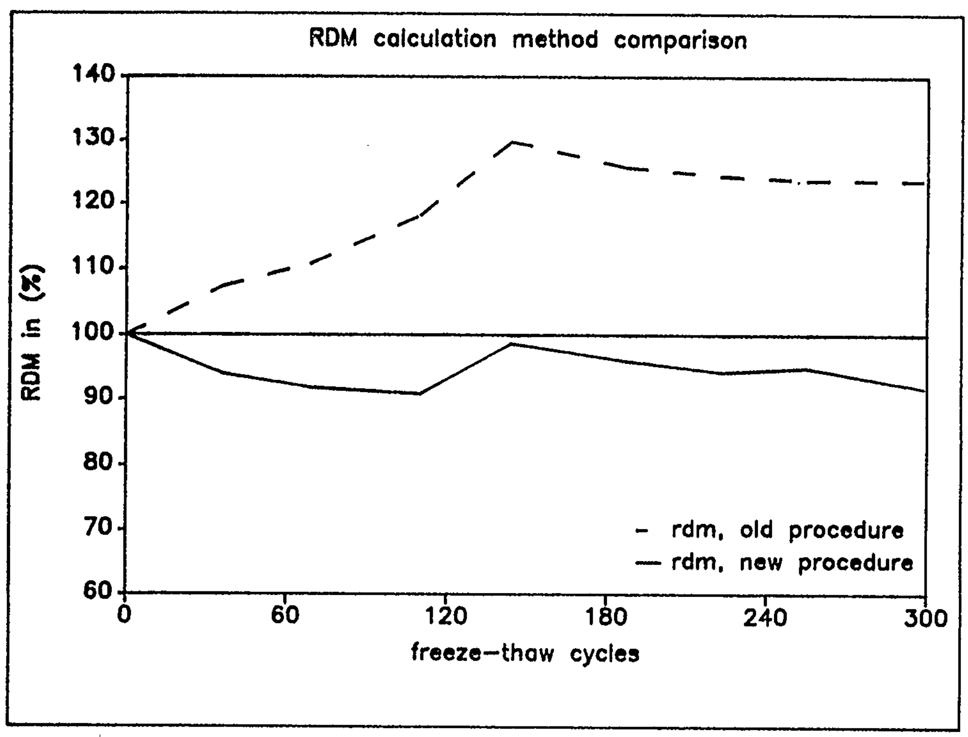
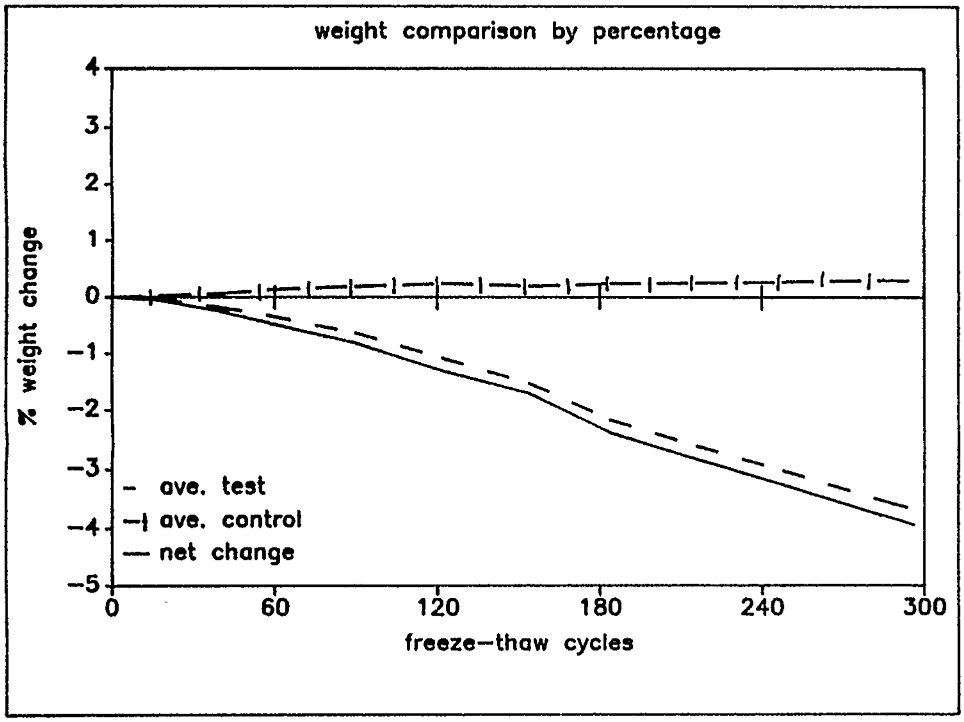


Figure 4.6: Weight and RDM Calculation Method Comparisons of NA 4



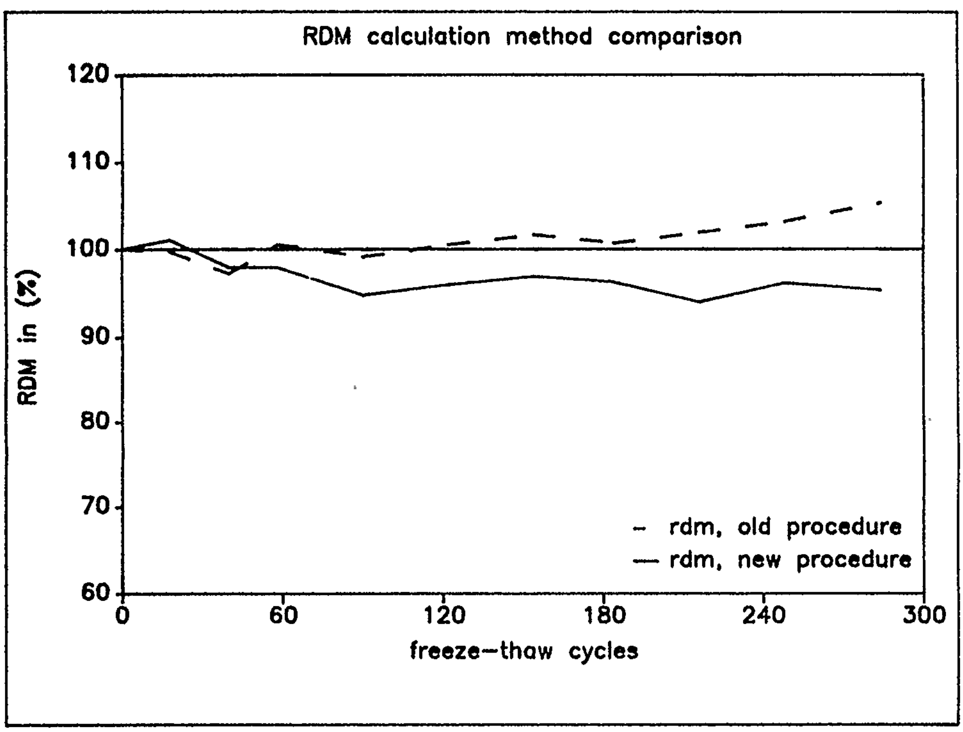


Figure 4.7: Weight and RDM Calculation Method Comparisons of NA 5

being the least severe. Refer to Figures 4.5 and 4.7 for example comparisons.

2. The strength of the specimen at the start of the durability test. The lower the compressive strength of the specimen the greater the material loss. Examples of this are Figures 4.4 and 4.6.

The Relative Dynamic Modulus of a specimen was affected by the water uptake of the specimen. In some cases this increase was enough to raise RDM to greater than the initial 100%, which should not happen in this type of test. Review of Figures 4.3 to 4.7 shows the RDM calculated by the old procedure are at or above the 100% value while the RDM calculated by the new procedure plots below the 100% value due to the correction made by using the adjusted test and control averages in the calculation procedure. In cases where water uptake was a major variable the difference in the fundamental frequency readings and resulting RDM is significant. Figure 4.6 is one such example where calculation by the old procedure gives a RDM of 123% and calculation by the new procedure gives a RDM of 93% at 300 cycles — a difference of 30%. Differences up to 10% were noted with the mixes cured in the fog room. This indicates that even under saturated conditions the RDM can be affected by water uptake.

4.4.2 The Low Air Content Series

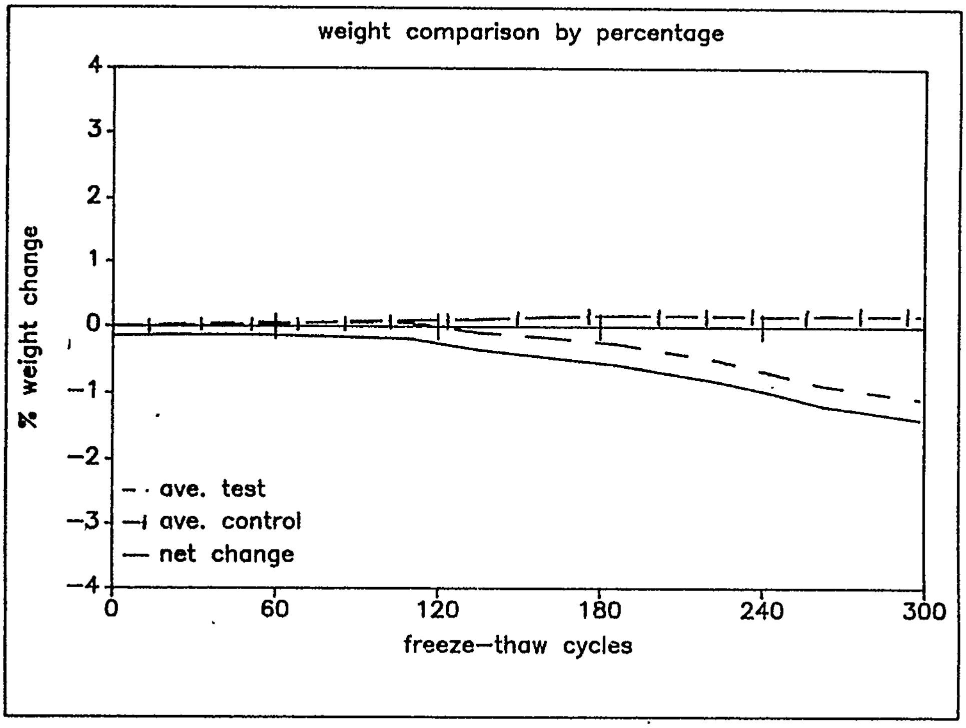
This **LA** Series initially consisted of three strength groups and three curing regimes for a total of seven mixes. As previously stated this series was to create a group of low durability mixes with various water gaining capacities to test the control specimen method. The reduction of the transverse fundamental frequency and weight data followed the same procedure as described in the previous section. A summary of the results of the durability testing at 300 cycles is shown in Table 4.6.

Table 4.6: LA Series Durability Test Results — New Procedure

Mix	RDM at 300 cycles (%)	Weight Loss (%)
LA1	90.0	1.4
LA2	86.0	1.2
LA3	88.0	2.1
LA4	68.0	9.8
LA5	87.0	2.8
LA6	86.0	4.5
LA7	70.0	2.9

Weight Comparisons between the test and control specimens are presented in Figures 4.8 to 4.14. All mixes in the **LA** Series showed small water gains (up to 1%) compared to the control specimens. The rate and amount of the water uptake was affected by the concretes strength and curing regime.

- For a given curing regime the lower the strength of a mix the faster the rate of the first part of the water uptake (0 to 40 cycles). This is due to the higher permeability of the lower strength mixes.
- For a given curing regime the higher the strength of a mix the greater the total water uptake. The higher strength mixes (more cement) are usually less permeable than lower strength mixes but require more water to complete the hydration process.



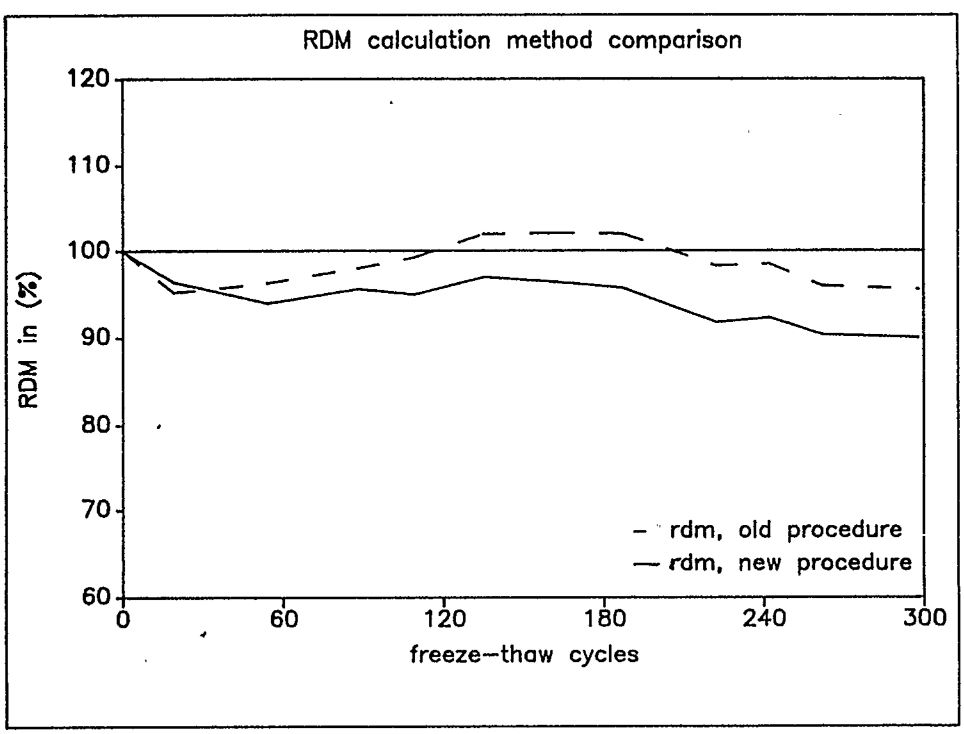
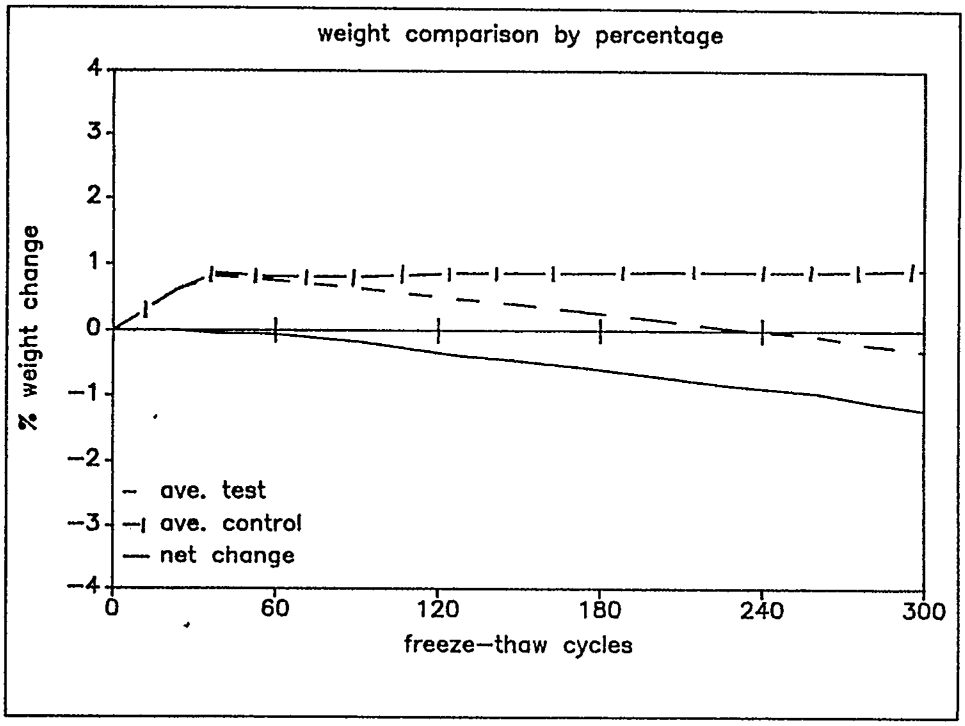


Figure 4.8: Weight and RDM Calculation Method Comparisons of LA 1



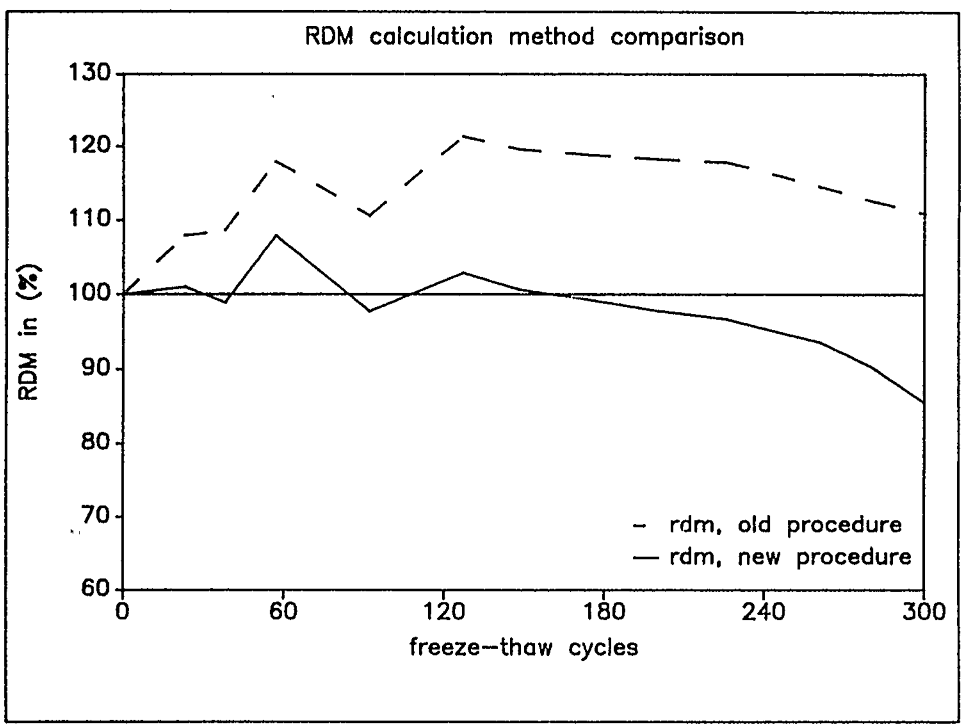
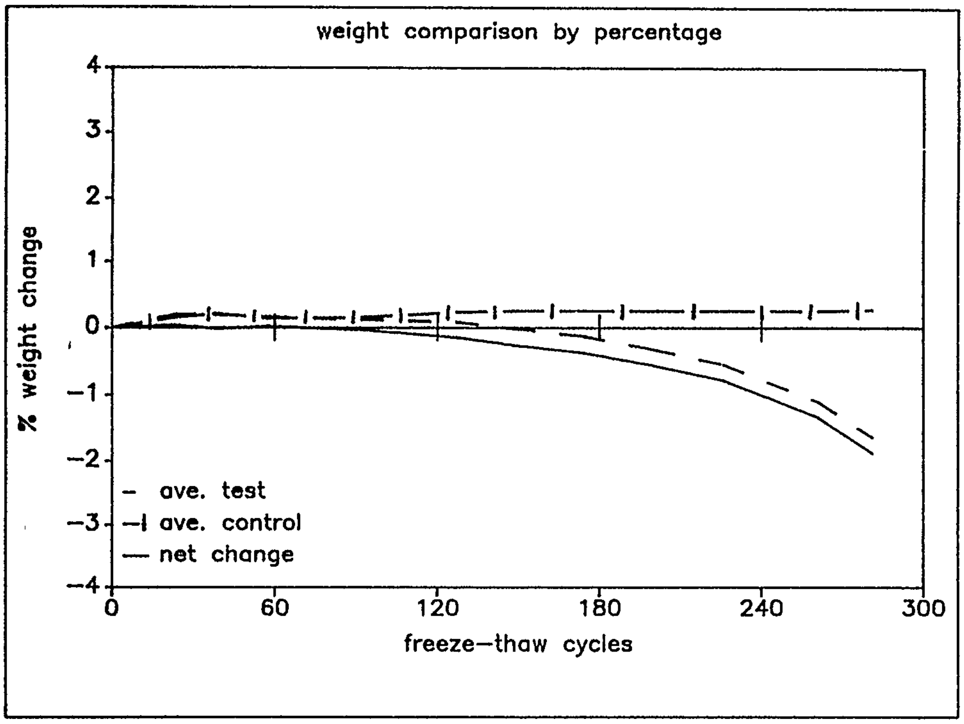


Figure 4.9: Weight and RDM Calculation Method Comparisons of LA 2



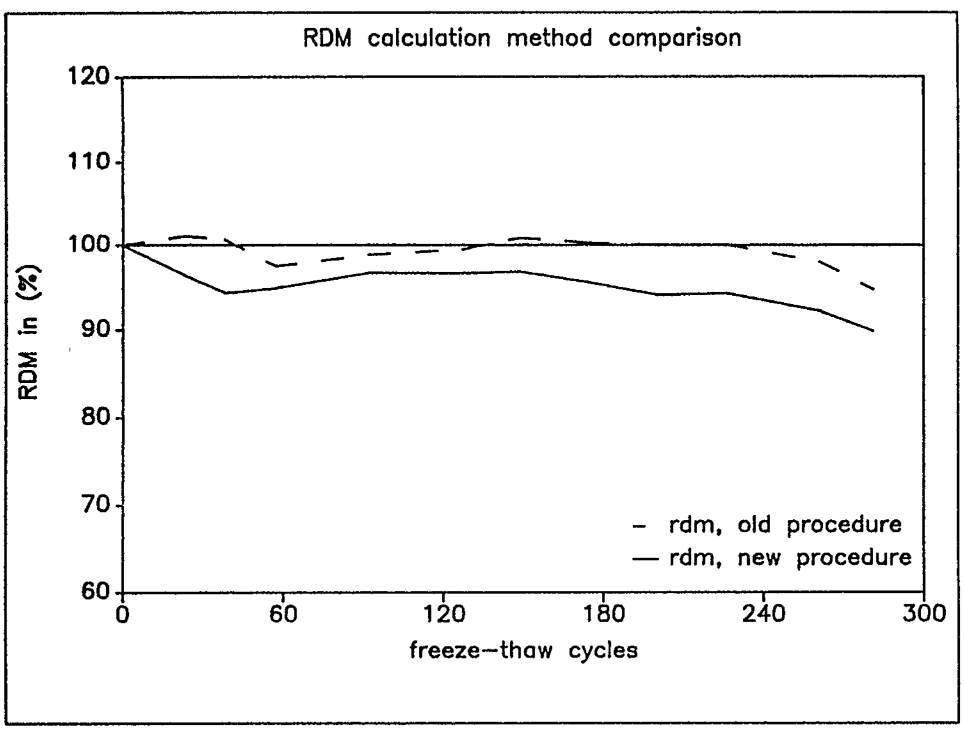
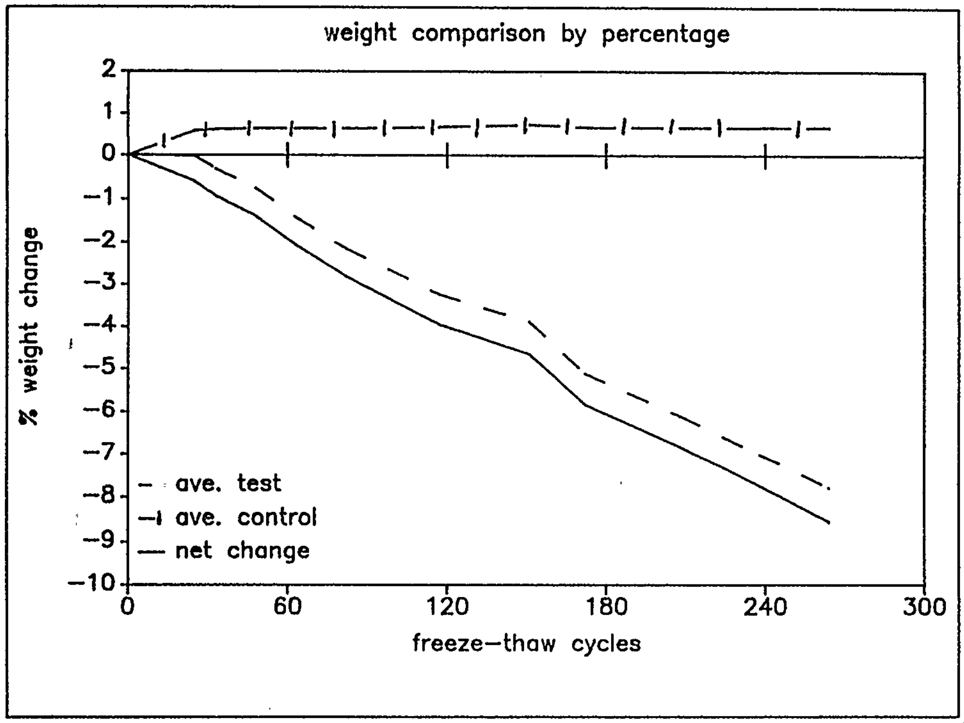


Figure 4.10: Weight and RDM Calculation Method Comparisons of LA 3



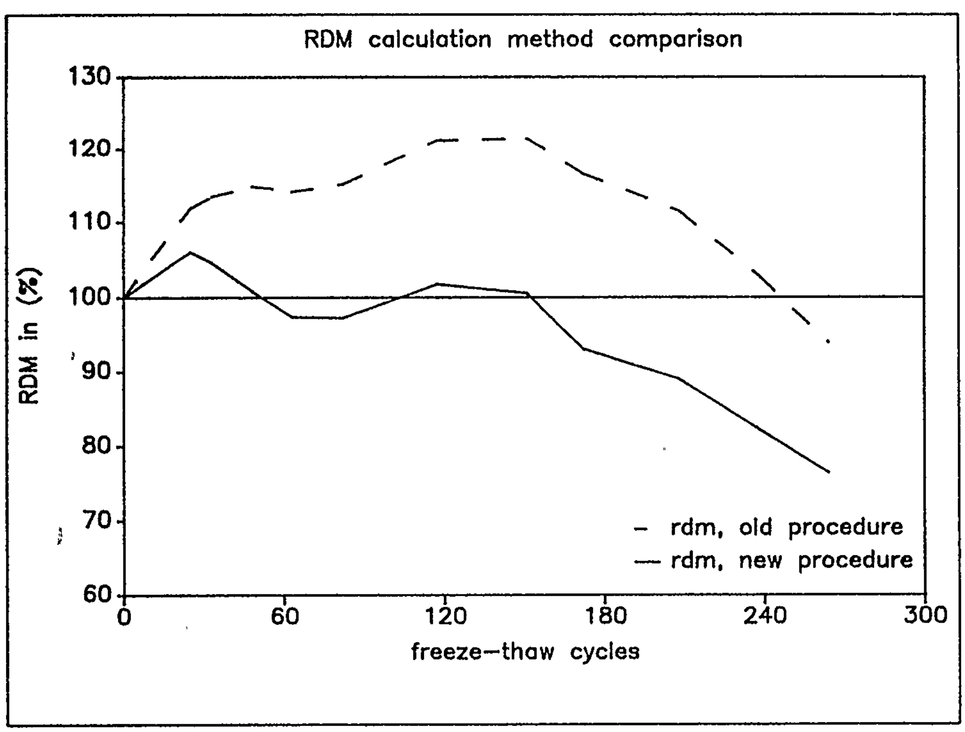
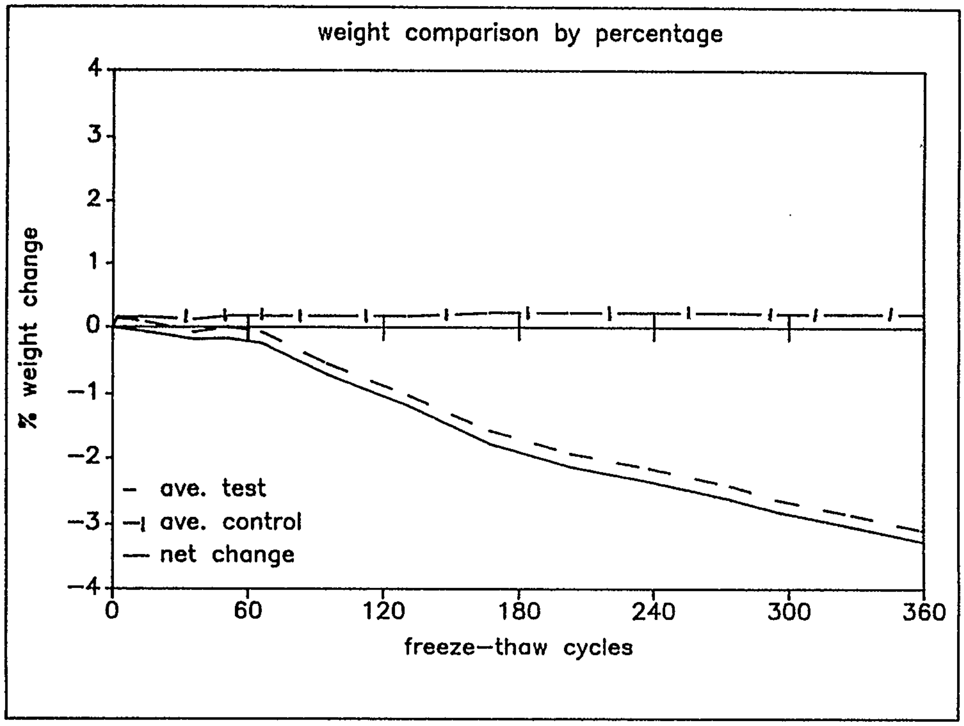


Figure 4.11: Weight and RDM Calculation Method Comparisons of LA 4



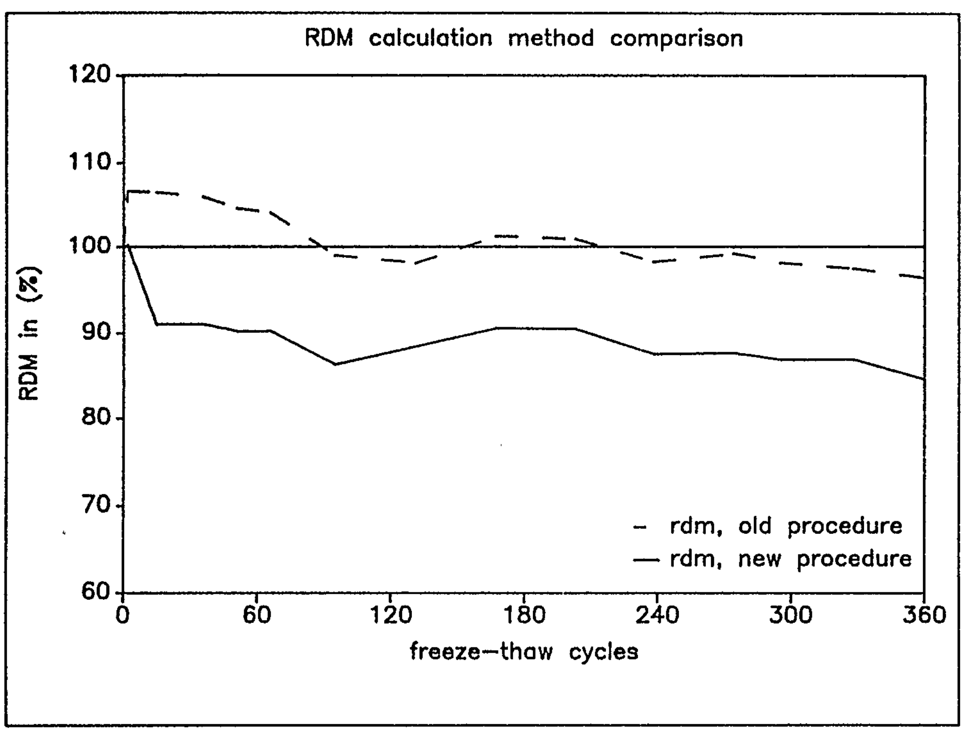
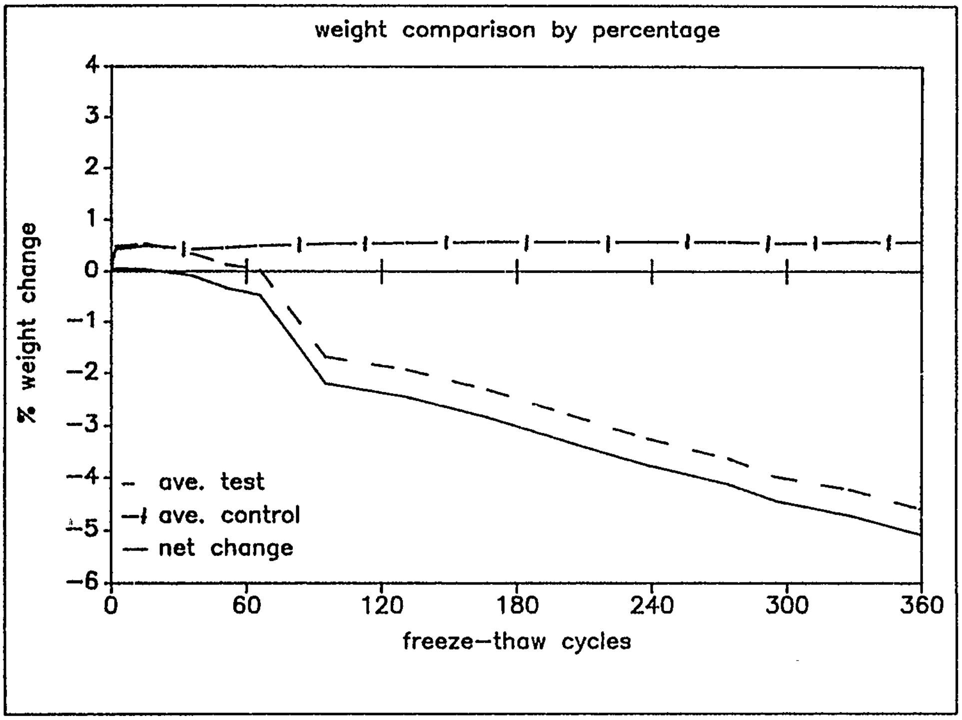


Figure 4.12: Weight and RDM Calculation Method Comparisons of LA 5



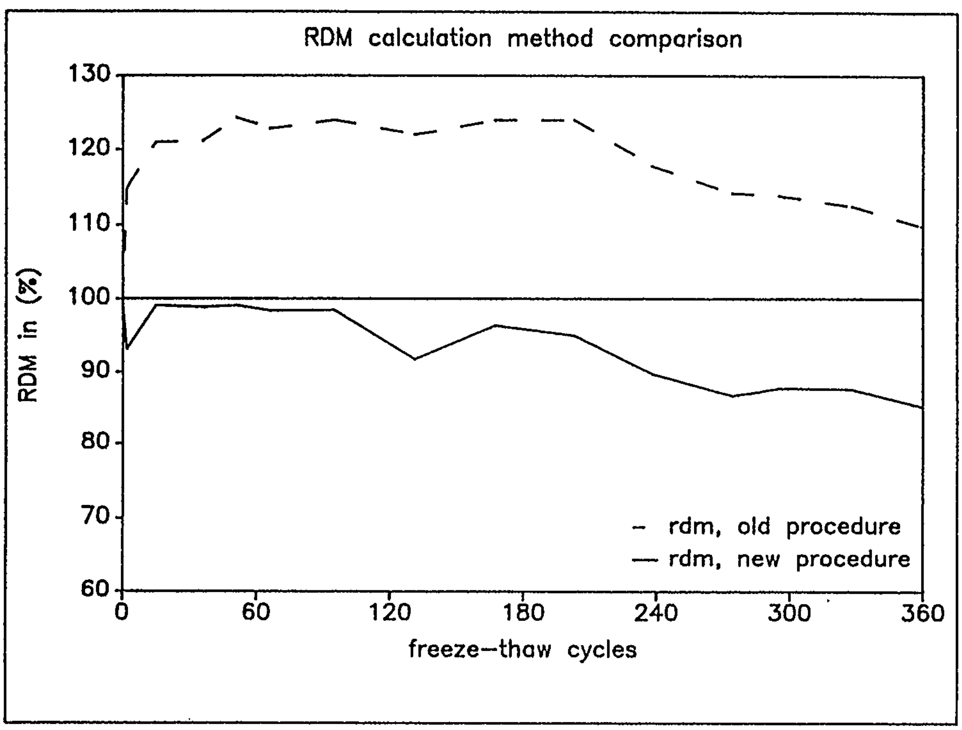


Figure 4.13: Weight and RDM Calculation Method Comparisons of LA 6

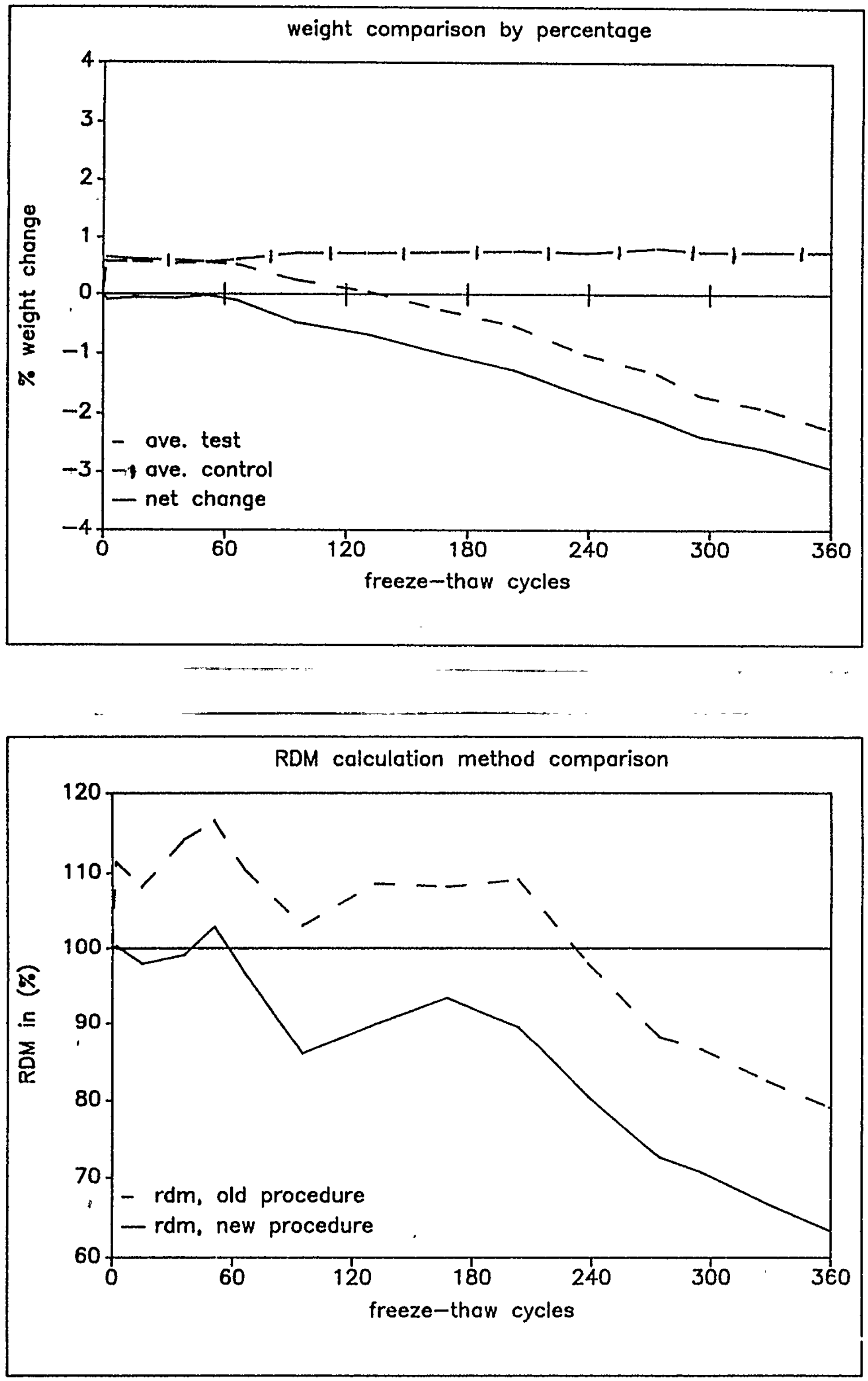


Figure 4.14: Weight and RDM Calculation Method Comparisons of LA 7

- The mixes cured in the environment room gain more water than the fog room cured mixes during the durability test since they have a lower water content that the fog room cured mixes at the begining of the durability test.

These water uptakes were again of two parts which were related to the curing regimes. The first part of the water uptake was a quick, short-term gain of $\frac{1}{2}$ – 1% by weight over less than 40 cycles of freeze-thaw and was observed in specimens from the enviroment room curing regime. The second part of the water uptake was a slow, long-term gain of less than $\frac{1}{2}$% by weight which continued for the duration of the test (300 cycles). As with the **NA** Series mixes the first part of the water uptake was inversely related to the degree of saturation and the second part was principally attributable to the hydration process.

As before the amount of weight loss was dependent on the curing regimes and the mix strength. The mixes cured in the enviroment room had a greater material loss than the mixes cured in the fog room.

Mix LA7, Figure 4.14, which had a combination of 7 days fog room and 7 days enviroment room curing had a greater water uptake than Mix LA5, the fog room cured mix, but lost the same small amount of material during the test. This indicates that even with 7 days fog room curing material loss due to scaling can be reduced, another indication of the effect of curing history on the test data. This result was found by others [16].

Relative Dynamic Modulus comparisons are presented in Figures 4.8 to 4.14. The RDM calculation method comparison showed the same type of result for the **LA** Series as was noted in the **NA** Series. The values calculated by the old procedure were at or higher than 100% due to water uptake and the use of the control specimens adjusted for the water uptake lowering the RDM to 100% or less.

This series did not produce the desired lower durability concrete (near or lower than 60% RDM at 300 cycles) therefore the test was continued to 360 cycles on LA5, LA6 and LA7 mixes to create some lower values as the deterioration process continued. The LA7 mix Figure 4.14 did approach 60% RDM at 360 cycles. At these lower values the new procedure was consistent with the previous testing.

4.4.3 Surface Scaling

Scaling of concrete surfaces as described in ASTM C 672-84 [25] can be a major concern when freeze-thaw cycles such as those produced by ASTM C 666 cause deterioration of a concrete. The use of control specimens during the ASTM C 666 test allowed a continuous scaling reference for the test specimens. This referencing can be used for scaling caused by mechanical (ASTM C 666) or chemical (deicing salts) mechanisms. The rating of the scaling used in this study is listed in Table 4.7.

The final ratings of the mixes are given in Table 4.8. The amount of scaling that occured to a particular mix is determined by the combined effect of many factors. During this investigation the major factors were air content, curing conditions and compressive strength of the mix. As shown by Table 4.8, the air content was the most important factor, which coincides with the majority of the research on this subject.

Table 4.7: Visual Rating of Surface Scaling

Rating	Condition of Surface
0	no scaling
1	slight scaling
2	slight to moderate scaling
3	moderate scaling
4	moderate to severe scaling
5	severe scaling

Table 4.8: Final Ratings of Surface Scaling for Mixes

THE NA SERIES

Mix	Rating
NA1	0
NA2	2
NA3	0
NA4	2
NA5	3

THE LA SERIES

Mix	Rating
LA1	3
LA2	4
LA3	4
LA4	5
LA5	5 *
LA6	5 *
LA7	5 *

The ratings were at 300 cycles except for the (*) mixes. The testing on these mixes continued to 360 cycles but the scaling was severe at 300 cycles. The freeze-thaw testing of LA3 and LA4 were terminated at 281 cycles and 264 cycles respectively. This was due to equipment problems.

4.4.4 Low-Pressure-Steam-Cured Precast Mix

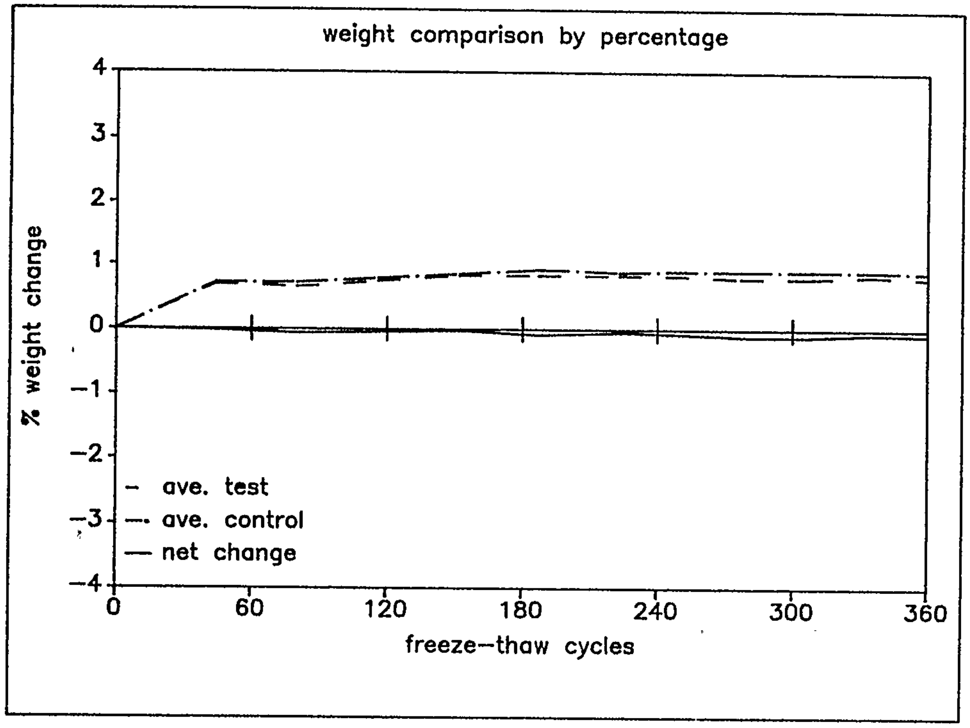
4.4.4.1 Present Testing

This mix was included in this study because the cut-steam-cured precast specimens differred from the cast specimens in their curing and boundary conditions. They were also cast and cured in the precast plant.

The resulting observations created by the freeze-thaw testing of this mix were much the same as the previous **NA** Series mixes cured in the enviroment room (unsaturated condition prior to start of testing) during this investigation.

Figure 4.15 shows the weight comparisons of the control and test specimens. This figure is much the same as Figure 4.4 where the weight gain due to water uptake was in two parts, an initial high rate gain over the first 40 cycles of the test followed by a minimal gain over the remaining test duration. The initial gain was 0.8%. After the adjustments had been applied to the weight gain data by use of the control specimen method the result was no *net* weight change of the specimens over 300 cycles. There was no material accummulation observed in the specimen containers during the test, which served as a check on the weight calculation results.

The water uptake caused an associated gain in RDM increasing the values to greater than 100% when calculated by the old procedure see Figure 4.15. As with the previous calculations the application of the new procedure resulted in a reduction of the water uptake effect on density with the result that the RDM dropped from 105% to near 90% at 300 cycles.



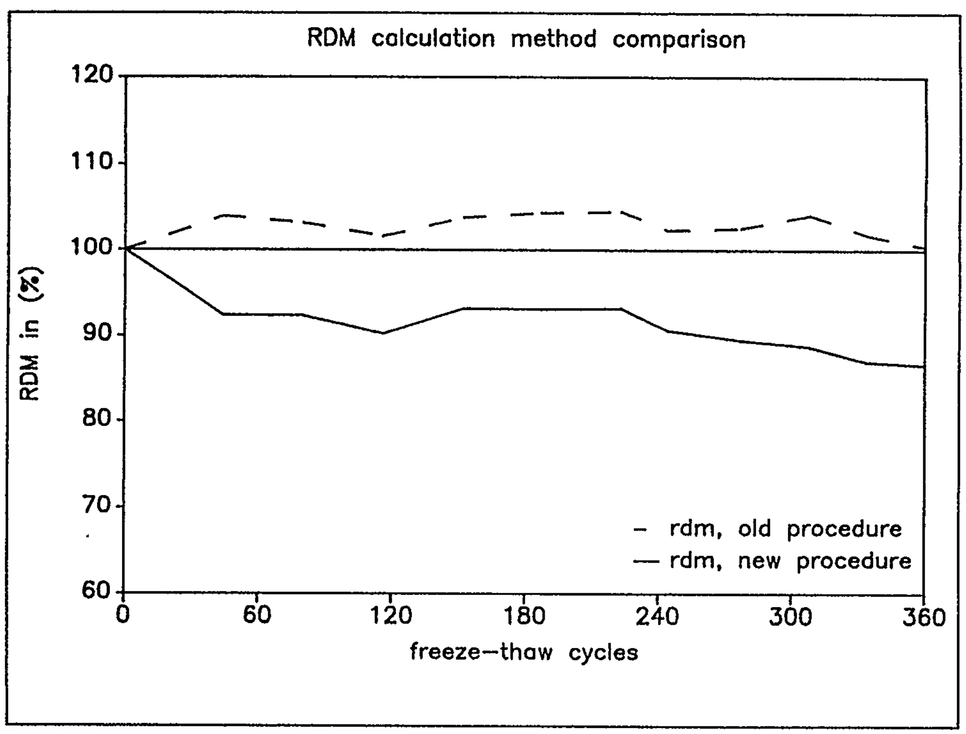
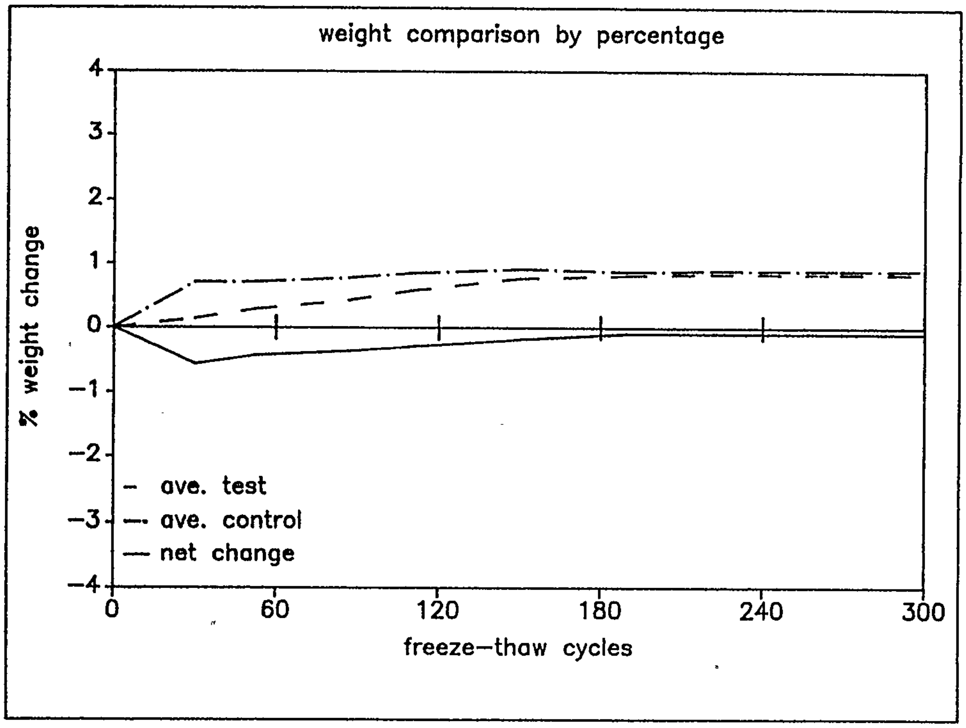


Figure 4.15: Weight and RDM comparisons of the Precast concrete

4.4.4.2 Application to Previous Testing

When applying the new procedure to previous relative dynamic modulus results from Precast Steam Cured concrete the effect of the water uptake and associated gain in the RDM was reduced. Figure 4.16 shows the weight change and RDM comparisons. Using the old procedure a 0.8% weight gain was measured together with a RDM of 108% at 300 cycles. The new procedure acknowledges that there is no *net* water uptake during the test which gives a RDM of 98% at 300 cycles. The new procedure provides a more realistic result for the durability test. When applying the new procedure to the previous work the precast control specimens from the current investigation were used. This caused some unreferenced variation in the results but since the mix design and curing history of the two sets of specimens were the same these variations were minimal once the test was past the first part of the water uptake.



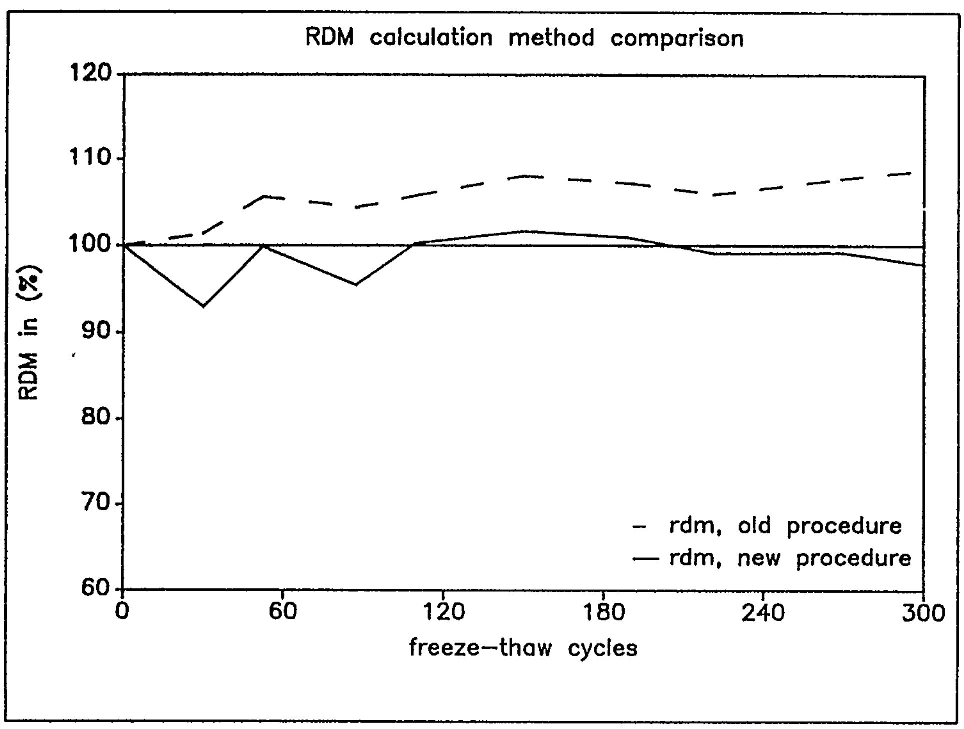
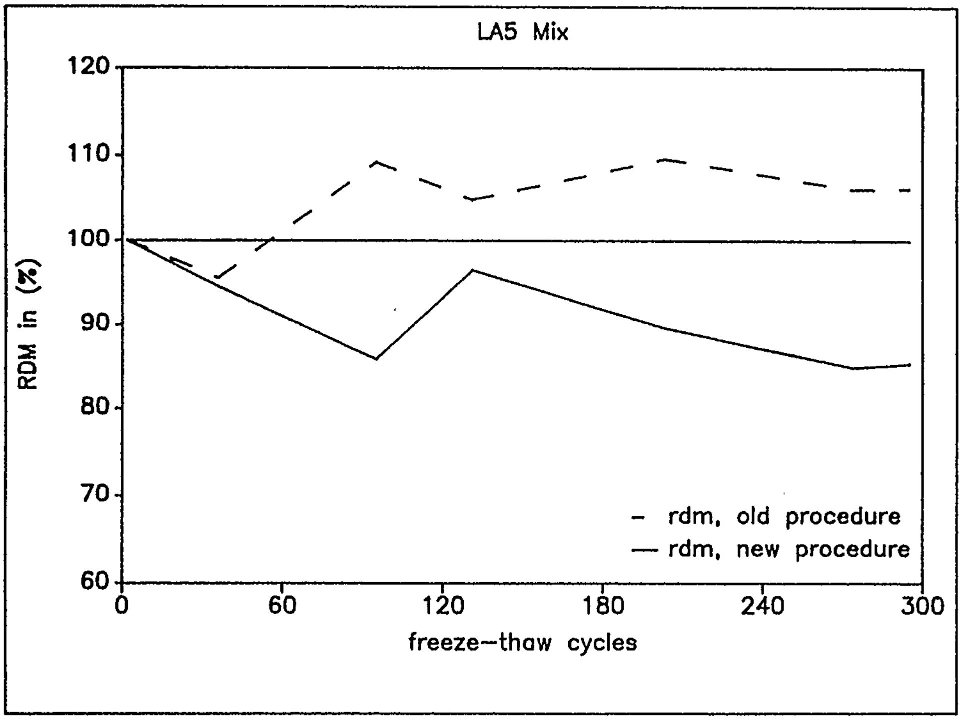


Figure 4.16: Weight and RDM of previous testing on Precast Concrete

4.5 Second Operator Comparison

This section deals with durability data collected by a second operator on the LA1 and Precast Steam Cured mixes. This data was collected to compare the effect of the control specimen method on the introduction of a second operator as a check on the results obtained by the calculation of the first operator's data. Figure 4.17 presents the results of the application of the control specimen method. As with the first operator, the new procedure reduces the effect of the water uptake lowering the plot of the RDM.

Operator comparison of the RDM calculated by the new procedure for the LA5 and Precast Mixes is given in Figure 4.18 The difference between the two operator plots for both figures is approximately 5% which is the calculated error due to operator and equipment in this experiment. This is covered under the section on reduction of variation due to control use.



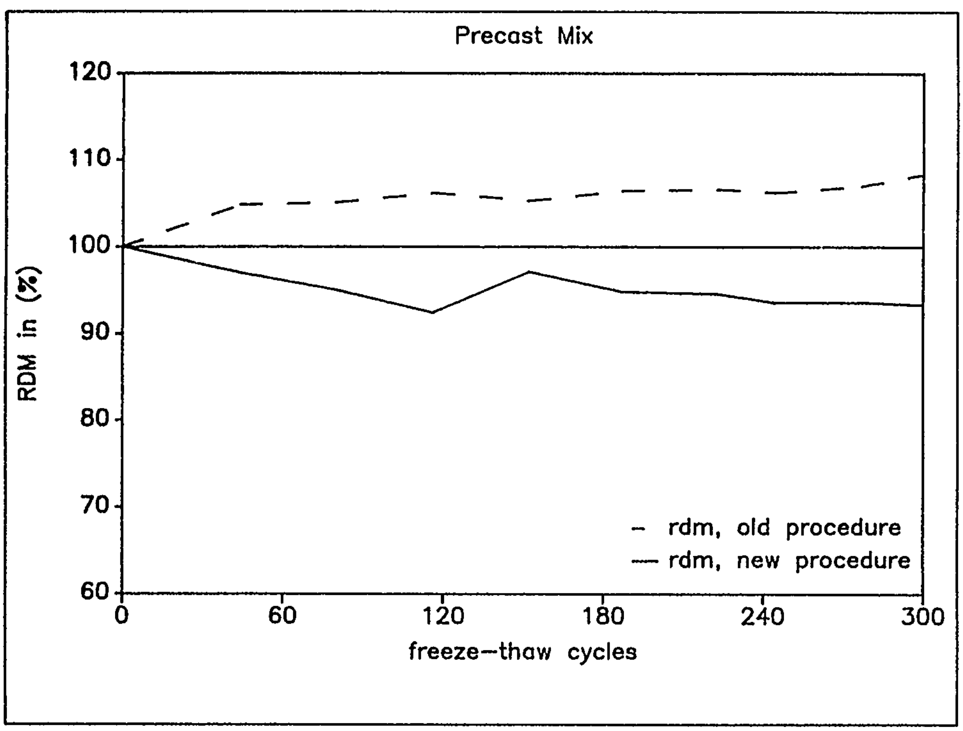
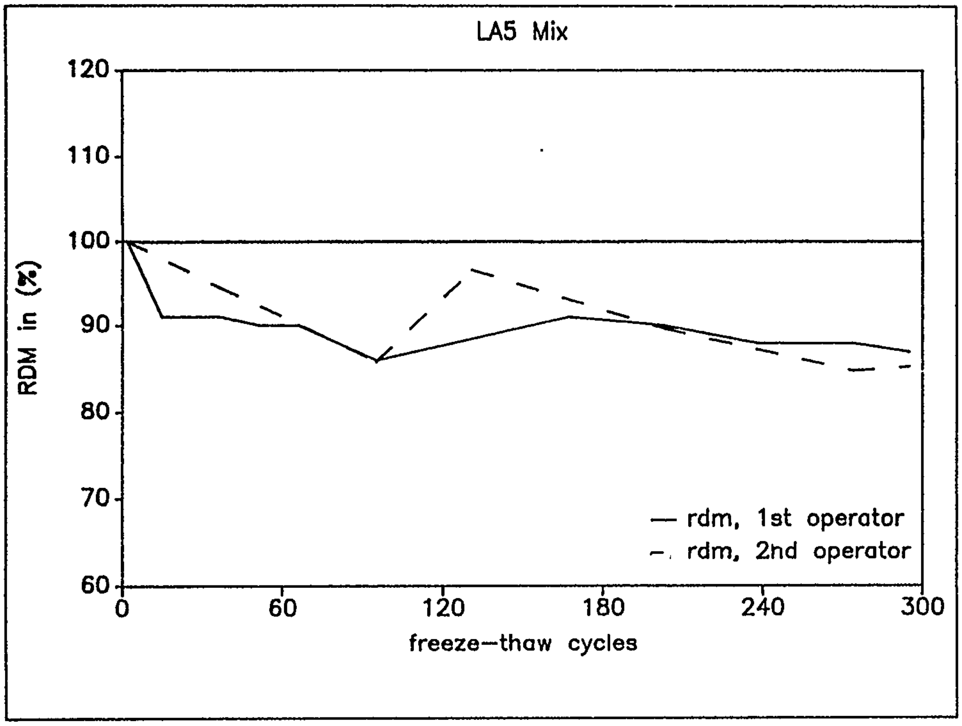


Figure 4.17: RDM calculation method comparison on Second Operator Data



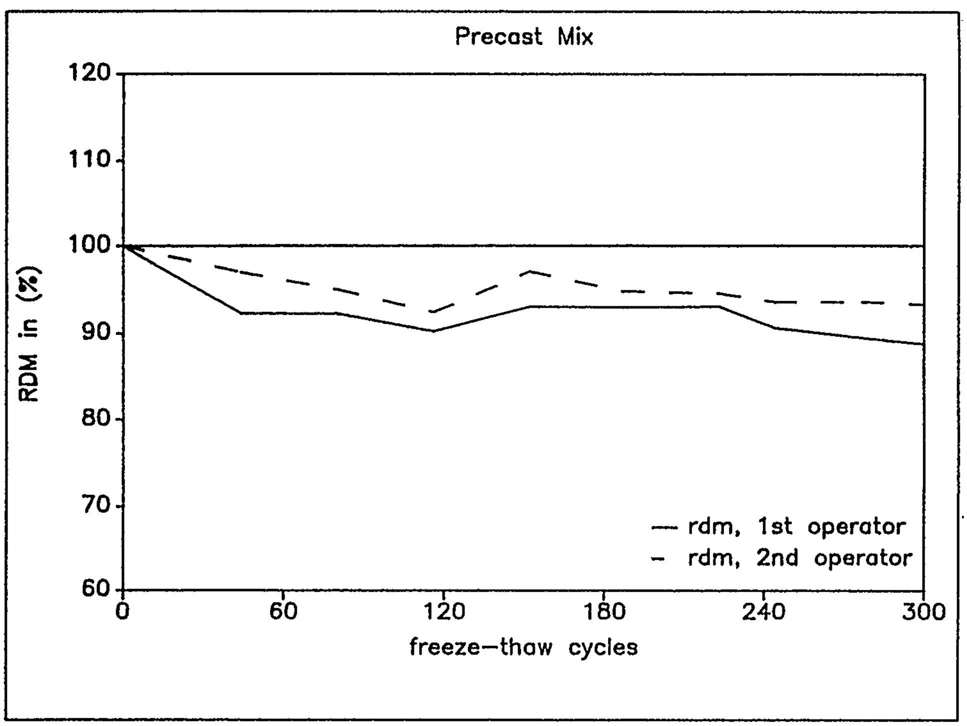


Figure 4.18: Operator Comparison of RDM calculated by the new procedure

4.6 Limitations

Although the use of control specimens distinguishes between test and non-test variations, it does not distinguish between the individual non-test variables. This grouping is usually of minor concern since it is the effect of the test on the specimens that is important and the ability to separate these effects from the non-test variables.

The procedure does not adjust for a change in operators but can reference the operators in the same way as the aluminum block. This allows the RDM readings of the operators to be compared. During this study the RDM readings on the aluminum block produced a 3% difference between the operators.

When applying the maturity concept the requirements and ability to hydrate will differ slightly between the control specimens and the test specimens due to the change of temperature of the test specimens during the freeze-thaw cycle. This difference would affect the water uptake of the specimen which in turn would affect the transverse fundamental frequency readings. This should be accounted for when analyzing durability results. The difficulty in calculating this difference is the determination of the temperature limits for hydration and the measurement of these limits during the test.

Use of the control specimen method does reduce the judgement required by the analyzer of the test data. It is suggested as a useful tool to improve the analyzer's knowledge of the concrete's history and the effects that the ASTM C 666 test has on the concrete.

Chapter 5

Conclusions and Recommendations

5.1 Conclusions

5.1.1 Comparison of Control Methods

Standard or control blocks such as the aluminum block used in this investigation cannot reference the variations due to the concrete. They can only reference the operator and the equipment. The control specimens reference the total variation which includes the concrete variations as well as the above. Since the net change in the RDM due to the test is of major concern the referencing of the total variation rather than the individual variations is adequate. If the analyzer requires the individual variations then a control block can be used in conjunction with the control specimen method.

In this investigation the single operator and equipment variation was shown to be small (2% standard deviation) when compared to the total variation (up to 30%) between the old and new procedures. With a saturated curing regime such as the fog room there was up to 10% variation between the procedures. This means that the variability of the concrete is greater than the variability caused by the operator and equipment and therefore should be referenced.

5.1.2 The Control Specimen Method

The objective of this investigation was to examine the use of control specimens to improve the interpretation of the ASTM C 666 test data. The results indicate that the use of the control specimen method does improve the referencing of the test specimens, helping to compensate for the non-test related variations in the transverse fundamental frequency readings for the duration of the test. The conclusions are:

1. The control specimens help to distinguish the weight change due to curing conditions of the concrete from the weight change due to the deterioration process caused by the freeze-thaw test. The net weight change of the test specimens can be monitored by the control specimens which are cast from the same mix and kept in a conditioning bath at $5°C$.

2. Use of the control specimens revealed a two stage water gain process. The first stage was due to the type of curing regime. This rate of gain was inversely proportional to the degree of saturation of the specimen at the beginning of the freeze-thaw test. The second stage of the water gain was principally caused by the continuing hydration process of the concrete.

3. When the control specimens are used during an ASTM C 666 test the effect of internal changes of the concrete on the transverse fundamental frequency readings of the test specimens can be distinguished from the effect of the deterioration process in the test on those readings.

The use of air content, curing regime and material ratios facilitated the investigation of the control specimen method for a variety of concrete durabilities and curing histories. Some of the conclusions are:

1. The procedure works for high and low of air contents.
2. The procedure works for a range of durabilities.
3. The effects of various curing histories are referenced by the control specimens.
4. The procedure can be applied to precast work and used to compare previous work to present work.
5. The use of control specimens allows a continuous reference for visual observation of the deterioration process such as scaling ratings.

5.2 Recommendations

Continued research in the standardization and analysis of the ASTM C 666 Procedure A Test could include:

1. Further investigation of the water uptake and the two stage effect could help to distinguish between the water requirements of the hydration of the concrete and the water uptake caused by the curing history.
2. Hydration and maturity differences between the control and test specimens due to the temperature change and deterioration process within the test specimens should be investigated to better understand the effects of the test on the test specimens.

3. Research of a more complete referencing of operator change could improve the comparison of results between groups of tests.

4. Study of the analytical procedures of using the control specimen method could improve on the knowledge of the effect of the deterioration process, when it starts to take place and how it is related to the water gain and subsequent material loss.

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Appendix A

Additional Information on the Materials

Table A.1: **Analysis of Fine Aggregate**

Sieve Analysis

Sieve Size	Weight Retained	Percent Retained	Percent Passing	CSA Standard	FM Calcs
5000 μm	11.6 gr	1.7%	98.3%	95-100%	1.7
2500	59.8	8.6	89.7	80-100	10.3
1250	56.1	8.1	81.6	50- 90	18.4
630	130.4	18.8	62.8	25- 65	37.2
315	317.5	45.8	17.0	10- 35	83
160	96.2	13.9	3.1	2- 10	96.9
Pan	21.9	3.1			

FINENESS MODULUS $\frac{2475}{100} = 2.475$ FM = 2.48

Petrographic Analysis

Quartz Quartzite	66.6 %
Feldspar	9.7
Chert	2.5
Limestones	3.5
Sandstones	6.1
Siltstone	2.7
Granite	2.9
Other	6.0
	100%

Table A.2: Analysis of Coarse Aggregate

Sieve Analysis

Sieve Size	Weight Retained	Weight Passed	Percent Passing	CSA Standard
20 μm	0 gm	1620.7 gm	100 %	100 %
14	78.7	1542.0	95.1	90-100
10	487	996.9	61.5	45- 75
8	284.2	712.7	44.0	0- 15
5	636.6	76.1	4.7	0- 5
Pan	76.1			

Petrographic Analysis

Quartz Quartzite	75.7 %
Feldspar Chert	1.3
Limstones	20.4
Sandstone	1.6
Siltstone	1.0
	100 %

Table A.3: **Detailed Analysis of Mixes**

	NA Series		LA Series		
	NA1	NA2	LA1	LA2	LA3
Materials (kg/m^3)					
cement	430	290	441	297	295
water	176	145	180	149	162
sand	590	660	605	676	760
gravel	1070	1200	1097	1230	1120
w/c	0.41	0.50	0.41	0.50	0.55
Air Content (%)					
total	6.5	6.5	4.0	4.0	3.5
mortar	12.1	13.3	7.3	8.0	6.4
paste	20.8	27.4	12.5	16.5	13.7
Paste Content (%)					
	31.3	23.7	32.0	24.3	25.6
Density (kg/m^3)					
	2266	2295	2323	2352	2337

Appendix B

Additional Information on the Equipment

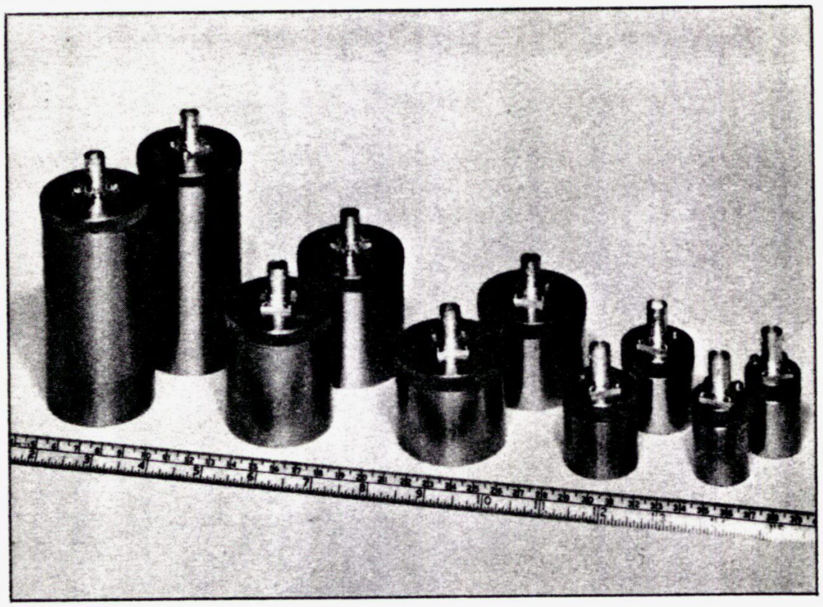
Table B.1: **The Vibration Table**

The vibration table was made by Equipment Consultants and Sales of Mississauga, Ontario. It measured 2 meters by 0.8 meters with a capacity of 454 kg. The table used two Vibco US 900 vibrators with the following specifications:

Model	US 900	Max Speed	10000 vpm
Force	900 lbs	Weight	18 lbs
Amps	3.5	Enclosed Construction	
Volts	115 ac/dc		

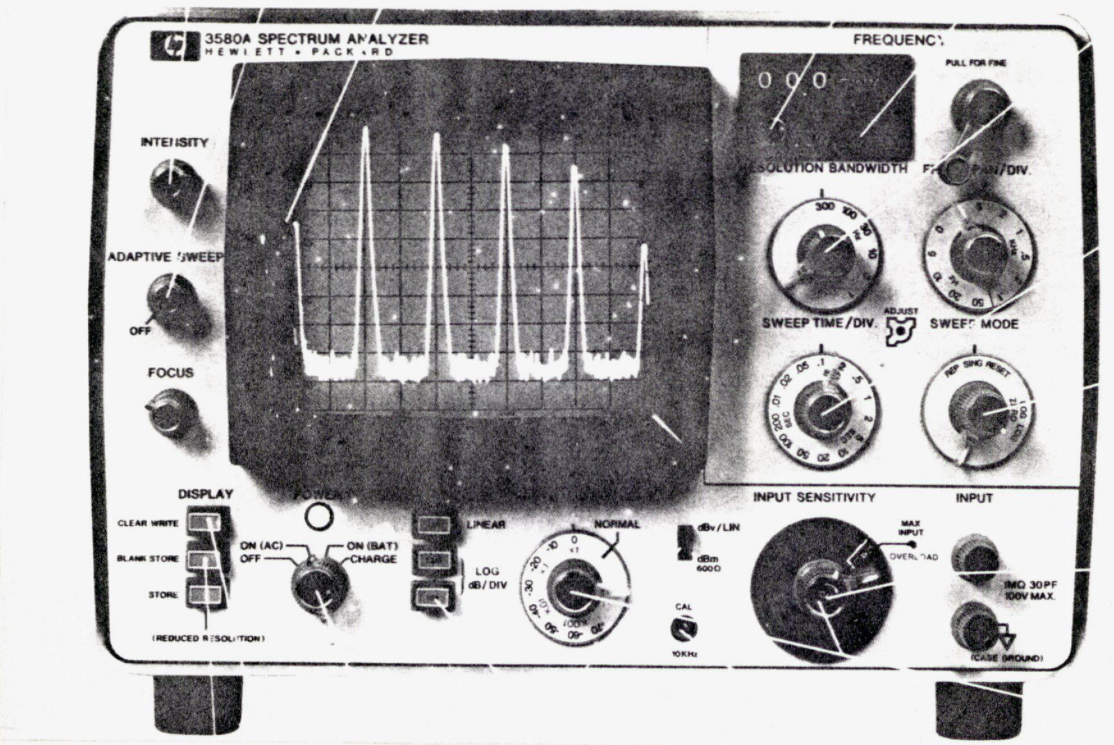
Table B.2: **The Slab Saw**

Table B.3: **The Transducers**



Model No.	Resonant Frequency	Diameter in.	Diameter mm.	Length in.	Length mm.
C-4891	24 kHz	1.97	50	3.90	99
C-4892	37 kHz	1.97	50	2.17	55
C-4898	54 kHz	1.97	50	1.65	42

Table B.4: **The Spectrum Analyzer**



Frequency Characteristics

- Range: 5 Hz to 50 kHz
- Frequency dial accuracy: 20°C to 30°C $\pm$ 100 Hz
- Display accuracy: $\pm 2\%$ of indicated point separation
- Typical stability: after 1 hour $\pm 10Hz/hour$, $\pm 5\ Hz/°C$
- Frequency dial resolution: 20 Hz on dial

Sweep Characteristics

- Scan width: 50 Hz to 50 kHz
- Log sweep: 20 Hz to 43 kHz ±20%
- Sweep times: .1 sec to 2000 sec ±5%
- Sweep error light indicates a sweep that is too fast to capture the full response. When the light is on the response will be greater than 5% lower than it should.

Input Characteristics

- Impedence: 1 $M\Omega$
- Maximum input: 100 V rms $\pm 100 V dc$

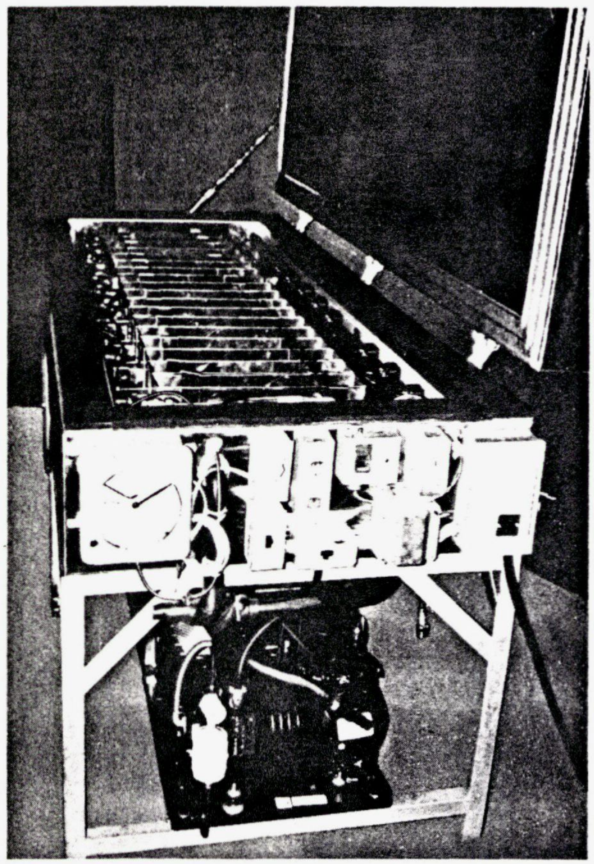
Output Characteristics

- Range: 0 to 2 V rms
- Frequency response: 5 Hz to 50 kHz ±3%
- Frequency accuracy: relative to center of filter $\pm 2.5 Hz$
- Impedance: 600 ohms

Amplitude Characteristics

- Overall range: 20 V to 100 nV full scale
- Accuracy : frequency 5 Hz to 50 kHz ±5%
- Switching : ($25°C$) 3 Hz to 300 Hz ±5%
- Dynamic range: 80 dB

Table B.5: **The Freeze-Thaw Bath**



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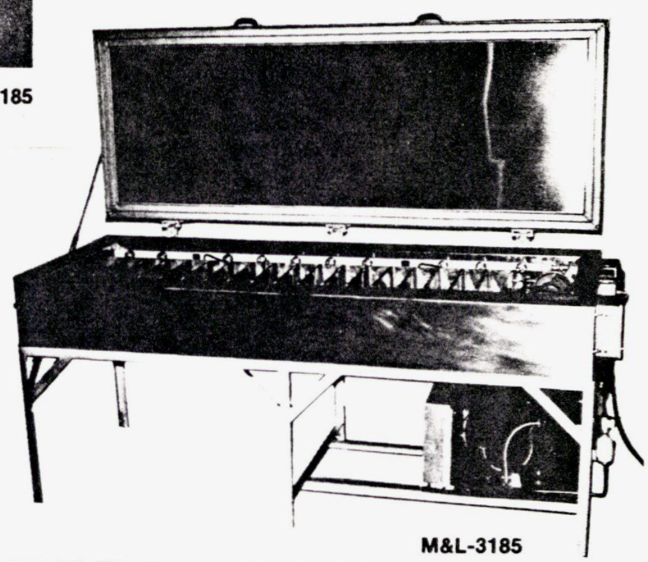

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