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Variability in Properties of Salado Mass Concrete

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Variability in Properties of Salado Mass Concrete

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ABSTRACT

Salado Mass Concrete (SMC) has been developed for use as a seal component in the Waste Isolation Pilot Plant. This concrete is intended to be mixed from pre-bagged materials, have an initial slump of 10 in., and remain pumpable and placeable for two hours after mixing. It is a mass concrete because it will be placed in monoliths large enough that the heat generated during cement hydration has the potential to cause thermal expansion and subsequent cracking, a phenomenon to avoid in the seal system. This report describes effects on concrete properties of changes in ratio of water to cement, batch size, and variations in characteristics of different lots of individual components of the concrete. The research demonstrates that the concrete can be prepared from laboratory-batched or pre-bagged dry materials in batches from 1.5 ft^3 to 5.0 yd^3, with no chemical admixtures other than the sodium chloride added to improve bonding with the host rock, at a water-to-cement ratio ranging from 0.36 to 0.42. All batches prepared according to established procedures had adequate workability for at least 1.5 hours, and achieved or exceeded the target compressive strength of 4500 psi at 180 days after casting. Portland cement and fly ash from different lots or sources did not have a measurable effect on concrete properties, but variations in a shrinkage-compensating cement used as a component of the concrete did appear to affect workability. A low initial temperature and the water-reducing and set-retarding functions of the salt are critical to meeting target properties.

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CONTENTS

ACRONYMS vi

1.0 INTRODUCTION 1
- 1.1 Previous WIPP Research at the WES 1
- 1.2 Objectives of this Study 2
- 1.3 Organization of the Report 3

2.0 PERFORMANCE REQUIREMENTS 5
- 2.1 Use of Salt-Saturated Concrete 5
- 2.2 Materials and Proportions of Salado Mass Concrete 6
- 2.3 Methods of Batching and Mixing 8

3.0 SMALL EXPERIMENTAL BATCHES 11
- 3.1 Performance Goals 11
- 3.2 Properties Measured During Mixing 12
- 3.3 Properties of Hardened Concrete 16
- 3.4 Thermal Screening 16
- 3.5 Conclusions from Experimental Batches 20

4.0 BATCHES MADE WITH PRE-BAGGED DRY MATERIALS 21
- 4.1 Materials Preparation and Batching 21
- 4.2 Large-Volume Batches 22
- 4.3 Properties Measured During and After Mixing 26
- 4.4 Properties of Hardened Concrete 26
  - 4.4.1 Individual Specimens Cast in the Laboratory 26
  - 4.4.2 Thermal Screening 30
  - 4.4.3 Casting and Monitoring Larger Monoliths 30
- 4.5 Conclusions from Large Batches of SMC-3 33

5.0 STUDIES OF EFFECTS OF SALT AND MIXTURE TEMPERATURE 35
- 5.1 Effect of Sodium Chloride on Slump and Time of Setting 35
- 5.2 Effect of Higher Initial Temperature on Workability 37

6.0 DISCUSSION AND CONCLUSIONS 39
- 6.1 Discussion 39
- 6.2 Conclusions 42

7.0 REFERENCES 45

APPENDIX A: SOURCES OF MATERIALS USED IN SMC BATCHES PREPARED AT THE WES A-1

APPENDIX B: MIXING SEQUENCE FOR LABORATORY BATCHES B-1

APPENDIX C: TEST METHODS AND STANDARDS APPLIED TO MATERIALS, BATCHING, MIXING, AND TESTING CONCRETE C-1

APPENDIX D: CHEMICAL ANALYSES OF CHEM COMP III D-1

Figures

3-1. Length change of SMC-3 and SMC-5, ASTM C 157 18
3-2. Calculated adiabatic temperature rise from Qdrum tests of batches 153SM3 and 153SM5 . . . 19
4-1. Pre-bagged materials in flexible intermediate bulk containers shown in laboratory storage . . . 23
4-2. Flexible intermediate bulk container positioned over mixer for discharge of materials 25
4-3. Discharge of concrete materials into mixer 25
4-4. Length change of prisms from batches 161SM3 and 231SM3, ASTM C157 29
4-5. Comparison of adiabatic temperature rise of SMC-3 batches and SMC-6R 31
6-1. Decrease in workability with time: SM3 series. 41
6-2. Decrease is workability with time: SM4 series. 41

Tables

1-1. Target Properties for Salado Mass Concrete 3
2-1. Proportions of Salado Mass Concrete 6
3-1. SMC-3 and SMC-5 Mixture Proportions 12
3-2. Trial Batches 13
3-3. Properties of SMC-5 Measured during Mixing 14
3-4. Properties of SMC-3 Measured during Mixing 15
3-5. Unconfined Compressive Strength, Laboratory Batches of SMC-5 and SMC-3 17
4-1. Batches from Pre-Bagged Materials 24
4-2. Properties Measured during Mixing of Batches Using Pre-Bagged Materials 27
4-3. Time of Setting of One Preliminary Batch and Two Pre-Bagged Batches of SMC-3 28
4-4. Unconfined Compressive Strength, Samples from Batches Using Pre-Bagged Materials . . . 28
4-5. Static Modulus of Elasticity, Samples from One Preliminary Batch and One Pre-Bagged Batch of SMC-3 28

4-6. Percentages of Coarse Aggregate and Air in Concrete Cores Taken from Laboratory-Cast Monoliths 32
5-1. Proportions of Batches to Demonstrate Effects of Sodium Chloride 35
5-2. Properties Measured during Mixing, with and without Sodium Chloride 36
5-3. SMC without Ice 38
6-1. Summary of 13 Batches of SMC-3 40

ACRONYMS

ACI	American Concrete Institute
ASTM	American Society for Testing and Materials
API	American Petroleum Institute
FIBC	flexible intermediate bulk container
DOE	Department of Energy (U.S.)
EPA	Environmental Protection Agency
ESC	expansive salt-saturated concrete
LSST	large-scale seal test
SMC	Salado Mass Concrete
SM3	SMC variety 3, from 1993
SM4	SMC variety 3, from 1994
SM5	SMC variety 5
SNL	Sandia National Laboratories
SSD	Saturated Surface Dry
SSSPT	small-scale seal performance test
TRU	transuranic
WES	Waterways Experiment Station
WIPP	Waste Isolation Pilot Plant
w/c	water to cement ratio

1.0 INTRODUCTION

This report describes the selection criteria for materials, target properties, and variations in the properties of concrete for use as a seal component at the Waste Isolation Pilot Plant (WIPP) and presents data verifying the laboratory performance of the seal-system concrete. The WIPP is a research and development facility operated by the U.S. Department of Energy (DOE) to demonstrate safe disposal of transuranic (TRU) wastes generated by defense-related activities. Compliance with relevant standards of the U.S. Environmental Protection Agency (EPA) must be demonstrated before the WIPP can become a disposal facility for TRU wastes. Performance of components of the WIPP seal system must be verified as part of the demonstration of compliance.

Sandia National Laboratories (SNL) supports the DOE by performing and directing scientific research necessary to understand the interaction of TRU waste with this geologic environment, to design and verify performance of the WIPP seal system, and to evaluate compliance with EPA standards for disposal of TRU wastes in a geologic repository. The Waterways Experiment Station (WES), the principal research laboratory of the U.S. Army Corps of Engineers, has supported SNL for nearly 20 years with research on repository-sealing materials. The current seal system specifies concrete components to provide structural support and to function as barriers to flow for 100 years or more after emplacement. A major element of WES support to SNL is development of concrete for these seal components and verification of properties and performance of this concrete.

1.1 Previous WIPP Research at the WES

The research reported here benefited from knowledge gained from ESC, an expansive salt-saturated concrete that was an earlier product of WES support to SNL. ESC had been developed and fielded in 1985 and 1986 for the Small-Scale Seal Performance Test (SSSPT) Series A and B (Stormont, 1986; Stormont and Howard, 1986; Wakeley and Walley, 1986; Stormont, 1987). Performance criteria for ESC had focused on a long working time and on high early-age expansion to create tight seals in boreholes for tests of in situ permeability beginning within a few days after concrete placement.

ESC met all of its performance criteria during and soon after placement. Although long-term geochemical stability of the concrete had not been a factor during ESC development, samples of ESC were recovered for analysis from a small portion of the SSSPT matrix in 1991 and were found to be in excellent condition. The concrete had maintained its physical and compositional integrity and had gained strength far in excess of its design value, in spite of exposure to brine (Wakeley et al., 1993).

Following this verification of the successful performance of ESC, program activity shifted to materials for mass placement in large-scale seal components and chemical durability of the large-scale seal concrete gained renewed emphasis. In 1991-92, the WES formulated a new series of candidate

concretes that differed fundamentally from ESC, using materials to increase chemical durability and developing the thermal and mechanical properties needed for a mass concrete. This series of concretes called Salado Mass Concrete (SMC) was refined through batching experiments and thermal and mechanical testing (Wakeley et al., 1994a). SMC component materials were also subjected to extensive geochemical tests (Poole et al., 1994; Wakeley et al., 1994b). The geochemical research showed that strength loss of cementitious solids in brines with strong concentrations of magnesium occurred by loss of calcium from cement hydration products, and not by direct replacement of calcium by magnesium, as has been reported elsewhere (Oberste-Padtberg, 1985; Bonen and Cohen, 1992).

1.2 Objectives of this Study

Two promising candidate SMC mixtures were tested during April through June 1993, as plans evolved for a large concrete placement demonstration. The materials and proportions used in the concrete called "SMC" have remained constant since late June 1993 and laboratory batching studies continued through February 1994. The properties of concrete prepared in WES laboratory studies between April 1993 and February 1994, the variability of those properties, and the factors that control them are the subjects of this report.

Refinement of the SMC formulation was focused on achieving the target properties listed in Table 1-1. Experience gained during preparation of volumes of concrete from laboratory mixers of 1.0 yd^3 or less indicated that placement characteristics and properties of SMC in an unhardened state, such as maintenance of slump with time, are sensitive to variables of materials and mixing. Batch size, mixing procedure, temperature, and compositional differences of the shrinkage-compensating cement (Chem Comp III) also were identified as factors influencing initial properties.

This report documents the investigation of mixing and handling characteristics and early-age properties of SMC produced in batches of up to 5 yd^3. Specific objectives were as follows:

- Identify batching and mixing procedures for large batches of SMC.
- Identify modifications to the SMC formulation needed to meet requirements of the batching, mixing, and placing procedures selected for underground construction of seal components.
- Evaluate probable effects on concrete handling and placing properties of small variations in water content, initial temperature, the composition of critical mixture components, or other variables revealed to influence properties of SMC in the unhardened state. Critical handling properties included changes in concrete slump and temperature with time after mixing, air content, and unit weight.
- Evaluate the effects of these changes on selected physical properties of hardened SMC.

Table 1-1. Target Properties for Salado Mass Concrete

Property	Comment
Initial slump 10 ± 1.0 in. Slump at 2 hr 8 ± 1.5 in.	ASTM C 143, high slump needed for pumping and placement
Initial temperature ≤ 60 °F Temperature @ 2 hr ≤ 68 °F	ASTM C 1064, using ice as part of mixing water
Air content ≤ 2.0%	ASTM C 231 (Type B meter), tight microstructure and higher strength
Self-levelling	Restrictions on underground placement may preclude vibration in forms
No separately batched admixtures	Simple and reproducible operations
Adiabatic temperature rise ≤ 60 °F at 28 days	CRD-C 38, to reduce thermally induced cracking
4500 psi compressive strength (f'_c)	ASTM C 39, at 180 days after placement
Volume stability	ASTM C 157, length change between + 0.05 and -0.02% through 180 days

1.3 Organization of the Report

This report contains seven Chapters and Appendices A through D. Chapter 2.0 is a discussion of the reasons that salt-saturated concrete, and particularly SMC, has been included in the WIPP seal designs. Chapter 2.0 supports the current recommended mixture proportions and performance requirements, outlines differences between the current SMC and its predecessors, and explains the choice of pre-bagging and underground mixing of SMC for field demonstrations. Chapter 3.0 describes small-scale batching and mixing operations from April 1993 through February 1994, and tabulates properties measured during mixing and handling, and physical properties of concrete specimens. Batch preparation up to 5 yd^3 from pre-bagged materials is described in Chapter 4.0. Chapter 5.0 covers special laboratory studies of the effects of salt and of elevated temperature on slump and workability. Data analyses are presented in Chapter 6.0, with discussions of the impact of important variables on concrete placing and performance. Chapter 7.0 is the list of references. Sources of materials, mixing procedures, standard test methods, and pertinent chemical analyses are given in the Appendices A through D.

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2.0 PERFORMANCE REQUIREMENTS

Because the WIPP is a unique facility, materials comprising components of the seal system require unprecedented documentation. In part, this report serves to document the attributes of a specially tailored concrete for seals application at the WIPP.

2.1 Use of Salt-Saturated Concrete

The need for salt-saturated concrete in the Salado Formation is dictated by the chemical composition of the host rock. Salt is used as a component of a cement-based material in halite rock sections to achieve bonding between the set product and the host rock salt (Smith, 1990). Freshwater concrete used in such a section would dissolve part of the host salt, prevent bonding, and enlarge the opening in the rock. This has been known since the 1940s, when salt was added as a component of cementitious grouts, used for completion of oilwells in salt domes along the Gulf Coast of the U.S. (Slagle and Smith, 1963). Salt-saturated grouts and concretes have been used for decades in bedded and domed salts, but post-placement properties for most projects are not as critical or scrutinized as extensively as they will be in a U.S. DOE facility in the 1990s. Thus, even though salt-saturated concretes are used widely in the U.S., experience with their use has not been documented well enough to provide objective evidence needed for the WIPP seal program.

When concrete is proportioned to be salt saturated in closely controlled operations, salt is added to the concrete as clean sodium chloride in an amount adequate to saturate the concrete mixing water. Mined rock salt such as that from the WIPP horizon cannot be used because it includes non-halite salts, which in small quantities can cause cement to hydrate quickly and thereby lose workability and slump (Kantro, 1975; Gartner and Gaidis, 1989). Other minerals in the host rock, such as clays and anhydrite, would decrease both workability and strength if added to the concrete.

The presence of sodium chloride in concrete improves its resistance to deterioration in high-magnesium brines, such as those known to be present at the WIPP repository horizon and higher in the geologic section. These brines represent a potential mechanism for concrete deterioration. Salt-saturated mixtures form a phase assemblage that is chemically more similar to the host environment than are conventional concretes. Improved durability has been demonstrated in laboratory studies, in which loss of calcium and strength were less severe for salt-saturated mixtures relative to their nonsalt counterparts (Wakeley et al., 1994b). The same effect has been shown in studies of the deterioration of concrete structures in the Arabian Gulf (Matta, 1993).

Salt, itself, imparts the benefits of set-retarding and water-reducing admixtures to a salt-saturated concrete. These properties were confirmed recently at the WES in comparison tests of initial slump and slump loss for two batches of concrete that were identical except that one was salt saturated. These tests are described in Chapter 5.0 of this report. Salt also acts as a dispersant (Smith, 1990), making salt-

saturated concretes more cohesive (sticky) during handling and placing and less prone to aggregate segregation.

Salt-saturated concrete can be proportioned to take full advantage of the benefits of salt to workability, cohesiveness, and water reduction. The concrete can be made to approach a self-levelling condition to simplify placement, with a comparatively low ratio of water to cement (w/c) for higher strength and low permeability, without the use of additional chemical admixtures.

2.2 Materials and Proportions of Salado Mass Concrete

The term "Salado Mass Concrete" was first applied to concretes developed at the WES during 1991 and 1992 (Wakeley et al., 1994a). "Salado" refers to the geologic unit enclosing the WIPP repository horizon, the Salado Formation. The American Concrete Institute (ACI) defines mass concrete as "any volume of concrete with dimensions large enough to require that measures be taken to cope with generation of heat from hydration of the cement and attendant volume change to minimize cracking" (ACI, 1990). Construction of concrete seals requires placements of 100 yd^3 or more and placement at the repository temperature, approximately 82 °F. This volume and placement temperature make heat a potential problem. Thus, SMC is proportioned to be compatible with salt and to minimize heat generation. Materials and proportions for the 1994 version of SMC are listed in Table 2-1. Selection of SMC component materials is described elsewhere (Wakeley et al., 1994a). Additional explanation of the current version of SMC is presented here.

Table 2-1. Proportions of Salado Mass Concrete

Material	lb/yd^3*
Portland cement, API Class H	278
Class F fly ash	207
Shrinkage-compensating cement	134
Fine aggregate**	1292
Coarse aggregate***	1579
Sodium chloride	88

* $kg/m^3 = (lb/yd^3) \times (0.59)$
** Assumes specific gravity =2.58, absorption = 0.63%
*** Assumes specific gravity = 2.55, absorption = 2.25%

Many common water-reducing admixtures are chemically incompatible with salt-saturated mixtures, causing either extraordinarily long setting times or high air contents (Wakeley and Walley, 1986). Air contents over 18% resulting from the chemical interaction of salt and some high-range water reducers have been reported (Grutzeck, 1984). Many dry powdered water-reducing admixtures that could be

added to pre-bagged dry concrete materials are not readily soluble in the presence of sodium chloride. Liquid admixtures would have to be measured separately into the concrete mixing water, which would add another step to the batching process and require more equipment. High-range water reducers are associated with rapid and unpredictable slump loss in many concretes, especially in combination with salt. Also, long-term properties of organic chemical admixtures are unknown, especially in a service environment in which they might be exposed to caustic brines. For these reasons and because the concrete achieved a 10-in initial slump without use of chemical admixtures other than salt, no water reducer was used in proportioning SMC for use at the WIPP.

A high air content reduces concrete strength making it more vulnerable to thermal stresses and cracking, can affect the time of setting, and may enhance diffusion and deterioration. Mixing a concrete with salt as a dry ingredient, rather than predissolving the salt and mixing the dry ingredients with brine, reduces the likelihood of foaming and a high air content (Slagle and Smith, 1963; Smith, 1990). This provides a more reproducible product, with low and constant air content, predictable cohesiveness and time of setting, and more constant strength.

The salt-saturated concrete (SMC) to be used as a seal component within the Salado Formation has been proportioned to make best use of the properties imparted by salt and to be a mass concrete. As currently designed, concrete seal components comprise a mass of concrete large enough to generate considerable heat from cement hydration and experience cracking from thermal stresses. Thus, it is a mass concrete and demands concrete practice to minimize heat rise. For this reason, SMC is prepared at an initial temperature below 60°F and a maximum placement temperature of 68°F. This mitigates potential thermal stresses by decreasing the maximum temperature the concrete experiences. A low initial temperature improves the potential of the concrete to be self-levelling and to flow for moderate distances during placement, by maintaining a higher slump for a longer time. Longer working time is a desirable property in any complex and closely scrutinized concrete placement (Wakeley, 1990). The effects of temperature on slump and flow are discussed in Chapter 5.0. Other benefits of a lower initial temperature for mass concrete include a more uniform solid product because of more uniform formation of hydration products and a higher ultimate strength (Mindess and Young, 1981; Troxell et al., 1968; Neville, 1981).

The Class F fly ash selected for SMC was chosen to evolve minimum heat at early ages in mass concrete. Class F fly ash hydrates more slowly than cement or some other pozzolans, contributing significantly to strength gain after 28 days. Use of Class F ash in SMC is consistent both with the demands of mass concrete and with the need for higher strength at later ages, when lithostatic loads on seal components will increase. The Class F fly ash also makes this concrete more resistant to chemical deterioration in high-magnesium brine (Wakeley et al., 1994b).

The SMC is proportioned to minimize shrinkage, promote tight sealing between concrete and host rock, and thus help avoid formation of a preferred pathway for fluid flow at the seal-rock interface. The

shrinkage of SMC is reduced by use of Chem Comp III, a shrinkage-compensating cement. Chem Comp III is more temperature sensitive than portland cement or fly ash and is much less effective as a shrinkage-compensating agent in warm and low-slump concretes. The low initial temperature and high slump of SMC favor the effectiveness of this component.

The component materials and initial concrete properties chosen for SMC create a concrete that is readily pumpable and placeable, is able to tolerate short-distance flow without segregation, and has a long working time to ease the demands of continuous placement. It has high slump with a low w/c for enhanced density and strength without using organic chemical admixtures. Fewer ingredients allow simpler and more reproducible batching. Use of salt batched as a dry ingredient and without a high-range water reducer gives the product a low and constant air content (2% or less) for consistent time of setting, strength, and density. With a low initial temperature, it achieves higher ultimate strength and reduces potential for thermal cracking in large placements. These properties were carefully balanced in proportioning and verifying the mixture to produce a highly placeable and reproducible salt-saturated mass concrete. Materials used in the current recommended version of SMC are listed in Appendix A.

2.3 Methods of Batching and Mixing

Placement of large volumes of SMC in the WIPP underground impacts selection of production methods and procedures. Initial experimental variables and methods of concrete preparation and curing in the laboratory were selected from WES experience and anticipated placement methods and conditions. Several approaches to batching and mixing concrete for large-volume placements were considered by the SNL Principal Investigator and personnel from Westinghouse (Managing Operating Contractor for the WIPP), Parsons Brinkerhoff, and the WES. Two options considered but rejected were 1) installing a batching and mixing facility underground, and 2) mixing at the surface and delivering concrete by pressurized pipe or drop-pipe to the repository level. The option selected requires pre-batching all dry ingredients off-site and mixing the pre-batched solids underground.

For pre-batching dry concrete materials, the aggregates are dried to meet requirements of ASTM C 387 and placed in flexible intermediate bulk containers (FIBCs). All other dry components then are either blended with the aggregates or placed separately into the FIBCs. Filled FIBCs are transported underground and stored at the nearly constant temperature and humidity of the repository horizon. The storage time underground depends on concrete production requirements and schedules. The minimum storage time is the few days required to equilibrate all materials to constant temperature, so that all batches of concrete can be produced using a constant procedure for cooling.

Many other advantages contributed to the selection of pre-batching at the surface and mixing underground. This process precludes the need for an underground batch plant, which is logistically difficult to operate and supply and can produce an unacceptable amount of dust. Underground operations

become a simple two-component system: 1) measure the water or ice and 2) mix it with the contents of FIBCs. Mixing close to the placement site without long-distance transport of fluid concrete produces a more uniform and reproducible product than a drop-pipe system does, is less complex than a long-distance pressurized pipe, and has less inherent risk.

Concrete production by surface batching and underground mixing alleviates the need for a four-hour working time. Delivery of fresh concrete to formwork is expected to take 30 minutes or less from the time of mixing. Allowing a generous contingency factor, the WES now specifies a 2-hour working time as a target property, defined as a slump of 8 $\pm$ 1.5 in. after two hours of intermittent mixing (Table 1-1). Previous specification (Wakeley et al., 1994b) called for 4-hour working time. Experiments conducted at the WES since May 1993 were designed assuming that the concrete would be dry-batched at the surface and mixed underground. This option should accommodate many possible choices of mixing and pumping equipment and many placement techniques.

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3.0 SMALL EXPERIMENTAL BATCHES

Fifteen batches of SMC were prepared in the laboratory between April 1993 and February 1994. Batch sizes ranged from 1.5 to 13.0 ft^3. This chapter gives the objectives of these batching experiments, and reports batching variables and properties of fresh and hardened concrete.

3.1 Performance Goals

A previous report (Wakeley et al., 1994a) discussed a variety of potential mixture proportions for SMC and focused on one mixture, designated SMC-6R, which was the variety that produced the least heat while achieving the required handling properties and compressive strength. The "R" at the end of the mixture designator indicated that it had been reproportioned with coarse aggregate more appropriate for mass concrete. All mixtures prepared since that work have been proportioned with the same size coarse aggregate, and "R" is no longer used in mixture designators.

The target compressive strength for this version of SMC was 4500 psi at 180 days after placement. Concrete specimens from four of the batches prepared during 1992 achieved 180-day compressive strengths of 3830 to the 4330 psi, lower than the 4500-psi target value. Calculations of lithostatic loads on panel seals indicated that concrete strength may need to be at least 4500 psi to resist cracking from stress concentrations at the ends of seals (Hansen et al., 1993). A decision was made to focus further concrete development on a higher strength version of SMC, recognizing that increased strength would be accompanied by increased heat evolution during the first few weeks after placement. From the previous work, the two best candidate mixtures for higher strength were SMC-3 and SMC-5, which in the present study, were proportioned with larger aggregate for mass concrete. Since June 1993, all laboratory studies have focused on SMC-3, which is considered the best candidate mixture because of its higher strength (over 6600 psi, Wakeley et al., 1994a). Materials and proportions for both SMC varieties prepared during this study are in Table 3-1.

Another target property of the previous work had been a four-hour working time to accommodate possible surface batching and mixing. A set-retarding admixture (sodium citrate) was used to extend the working time, but was not equally effective for all batches. Also, it appeared to cause foaming in some batches, a condition that was controlled by use of an air-detraining (anti-foaming) admixture. With the selection of underground mixing and an attendant shorter working-time requirement, experiments in the present study were designed to determine if a two-hour working time could be achieved and if the air content of the mixture could be kept low and constant without the use of either chemical admixture.

After it was determined that the required properties could be achieved from SMC-3 proportions and without the use of chemical admixtures, additional experiments were designed to increase the strength by decreasing the water content, to prepare batches using different lots of Chem Comp III, and to gain

Table 3-1. SMC-3 and SMC-5 Mixture Proportions

Material	SSD Batch Quantities, lb/yd³*	
	SMC-3	SMC-5
Cement, API Class H	278	221
Class F Fly Ash	207	247
Chem Comp III	134	112
Fine Aggregate	1255 to 1292**	1283
Coarse Aggregate	1579 to 1615**	1645
Salt	88	86
Water	216 to 260 ***	226 to 295***

* $kg/m^3 = (lb/yd^3) \times (0.59)$
** Quantities may change with aggregate density or grading; see ACI, 1991.
*** Changes with w/c

more experience with larger batch sizes. Table 3-2 lists all batches by batch designator, date of mixing, w/c, and batch volume, where "cement" includes [portland cement + fly ash + Chem Comp III]. The first three digits of the batch designator give the Julian date, followed by SM5 for SMC-5 proportions or SM3 for SMC-3 proportions. SM4 identifies batches with SMC-3 proportions prepared in 1994. Batches containing chemical admixtures also are indicated in Table 3-2.

3.2 Properties Measured During Mixing

The charging sequence of materials into the mixer is summarized in Appendix B. Ice was substituted for half of the mixing water, by mass, so that initial concrete temperatures after mixing were below 60°F. Each batch was mixed intermittently for two hours. Concrete slump and temperature were measured within a few minutes after mixing was started (see Tables 3-3 and 3-4) and at 1, 1½, and 2 hours after mixing began. Air content and unit weight were recorded initially and after two hours of intermittent mixing. Table 3-3 lists these properties for five batches of SMC-5 and Table 3-4 lists properties for 11 SMC-3 batches prepared during this segment of the study. Appendix C lists the standard test methods followed in making these measurements.

For batches 113SM5, 133SM5, and 153SM5, total slump loss was approximately 2½ in. during the two-hour mixing period. Batch 174SM3 maintained its initial slump for 1½ hours after mixing was initiated, illustrating the delay in slump loss attributable to the sodium citrate added to that batch. Slump loss of batches 113SM3, 133SM3, and 153SM3 was approximately 2 in. during the mixing period. For batches with w/c of 0.42 or higher, the final slump met or exceeded the target values of 8 in.

Table 3-2. Trial Batches

Batch Designator	Mixing Date	w/c*	Batch Volume, ft^3****
097SM5**	04/07/93	0.51	2.0
106SM5**	04/16/93	0.39	1.5
113SM5	04/23/93	0.43	2.0
133SM5	05/13/93	0.43	2.0
153SM5	06/02/93	0.43	2.5
106SM3**	04/16/93	0.39	1.5
113SM3	04/23/93	0.42	2.0
133SM3	05/13/93	0.42	2.0
153SM3	06/02/93	0.42	2.5
174SM3***	06/23/93	0.42	1.5
190SM3	07/09/93	0.42	1.5
024SM4	01/24/94	0.35	1.5
025SM4	01/25/94	0.36	14.0
032SM4 M*	02/01/94	0.36	13.0
032SM4 A**	02/01/94	0.36	9.0
040SM4	02/09/94	0.36	12.5

* C = all cementitious materials, Cement + Fly Ash + Chem Comp III
** Air-detraining admixture added at 1.0% by mass of cementitious material
*** Sodium citrate added at 2.1 lb/yd^3 of concrete
**** $m^3 = (ft^3) \times (0.028)$

Table 3-3. Properties of SMC-5 Measured during Mixing

Batch Designator	Slump, in.				Unit Weight, lb/ft³*		Air, %		Mixture Temperature, °F	
	Initial	1 hr	1½ hr	2 hr	Initial	Final	Initial	Final	Initial	Final
097SM5	10	10	9½	8½	141.6	142.6	3.3	2.7	56	63
106SM5	9	8	7	5½	144.0	144.0	2.4	2.4	57	68
113SM5	11	10	9½	8½	146.4	146.4	2.0	1.3	52	66
133SM5	10	10	9½	8	145.6	146.0	1.2	1.6	57	68
153SM5	11	10	9½	8½	144.0	146.0	2.2	1.5	56	70

097SM5 - with D-Air, w/c=0.51
106SM5 - with D-Air, w/c=0.39
113SM5, 133SM5, 153SM5 - w/c=0.43

* $kg/m^3 = (lb/ft^3) \times (16.02)$

Table 3-4. Properties of SMC-3 Measured during Mixing

Batch Designator**	Slump, in.				Unit Weight, lb/ft³*		Air, %		Mixture Temperature, °F	
	Initial	1 hr	1½ hr	2 hr	Initial	Final	Initial	Final	Initial	Final
106SM3	9½	8	6½	5	145.6	144.0	2.4	1.9	57	70
113SM3	10	9½	9	8½	143.6	144.0	1.9	1.9	52	65
133SM3	10	10	9	8	144.8	145.0	1.7	1.9	56	68
153SM3	10	9	9	8	142.4	146.0	3.4	2.0	55	70
174SM3	10½	10½	10½	8½	145.6	148.0	1.3	1.1	60	73
190SM3	11	10½	9½	8½	Not tested				55	72
024SM4	10	8½	6½	5	144.4	146.0	2.0	2.0	54.3	68.0
025SM4	10	8½	5½	5½	144.0	145.2	2.2	2.1	51.4	61.8
032SM4 M	10	9½	8	7½	144.4	144.8	1.7	1.8	46.9	57.6
032SM4 A	9½	8½	7	6	144.8	144.8	1.9	1.8	48.2	58.3
040SM4	9½	7	5½	4	144.8	145.2	2.0	2.3	51.6	61.8

106SM3 - with D-Air, w/c = 0.39
113SM3, 133SM3, 153SM3, 190SM3 - w/c = 0.42
174SM3 - with citrate, w/c = 0.42

032SM4 M = morning
A = afternoon

* $kg/m^3 = (lb/ft^3) \times (16.02)$
** All SM4 batches w/c = 0.36

Differences in slump between SM3 and SM4 batches indicate predictable changes in slump with changing amounts of mixing water. Lower w/c gave lower initial slump and more rapid slump loss. The lower w/c used for SM4 batches was based on the w/c of 0.36 used successfully with 5-yd^3 batches (see Chapter 4.0), although in these small batches at this w/c workability was sufficient for only one hour after initial mixing.

All batches maintained a narrow range of temperature throughout the mixing period. The concrete temperature at the start of mixing for all trial batches was 60°F or less. Final temperature was 70°F or less except where citrate was used (174SM3, Table 3-4).

3.3 Properties of Hardened Concrete

Specimens were cast from most trial batches for measurements of unconfined compressive strength and linear expansion. SM5 and SM3 specimens were cured and stored in simulated repository conditions (80°F and 50% relative humidity). SM4 specimens were cured at laboratory ambient conditions. Table 3-5 reports data from compressive strength tests conducted at four ages from seven through 180 days. The static modulus of elasticity determined for samples cast from 153SM3 ranged from 2.2 (10^6) psi at seven days to 5.7 (10^6) psi at 180 days. Figure 3-1 shows the length change of prisms cast from batches 106SM3, 106SM5, 113SM3, and 113SM5. These tests indicate the tendency of a concrete to expand or shrink and are used for comparisons among mixtures, although the test does not indicate expected field performance of concrete in a large placement. All test prisms met the target value for volume stability through 180 days. Standard test methods used for properties of hardened concrete are listed in Appendix C.

3.4 Thermal Screening

Thermal screening was performed using a patented device called a "Qdrum" Calorimeter (previously known as a "Haybox" Calorimeter) (Hover, 1992). This device measures the heat signature from a 6-by-12-in. cylindrical concrete specimen in a calibrated calorimeter. The Qdrum couples this heat signature with heat-capacity information entered for the individual components of the concrete and calculates an adiabatic temperature rise for the mixture.

Using this device, the heat signature was measured and adiabatic temperature rise calculated for two of the preliminary small batches. Curves representing adiabatic temperature rise for the small batches are shown in Figure 3-2, and at seven days, for 153SM3 and 153SM5, are 35.3°F and 31.6°F, respectively.

Table 3-5. Unconfined Compressive Strength, Laboratory Batches of SMC-5 and SMC-3

Batch Number	Compressive Strength, psi*			
	7 Days	28 Days	90 Days	180 Days
097SM5	570	1510	2980	4090
106SM5	670	1890	3470	4880
113SM5	770	1990	4430	5140
133SM5	750	2050	4610	5220
153SM5	810	2240	4390	5480
106SM3	830	1930	3110	4540
113SM3	1040	2190	3770	4830
133SM3	960	2250	4580**	4510
153SM3	1060	2410	4350	5130
174SM3***	1170	2863	6050	7310
024SM4	1530	NT	4790	NT
025SM4	1220	3040	3720	NT
032SM4 (M)	910	2340	3970	4890
032SM4 (A)	1000	2230	3970	NT
040SM4	930	2430	3770	NT

* MPa = (psi) × (0.00689)
** At 133 days
*** Batch contained sodium citrate retarder
NT = not tested

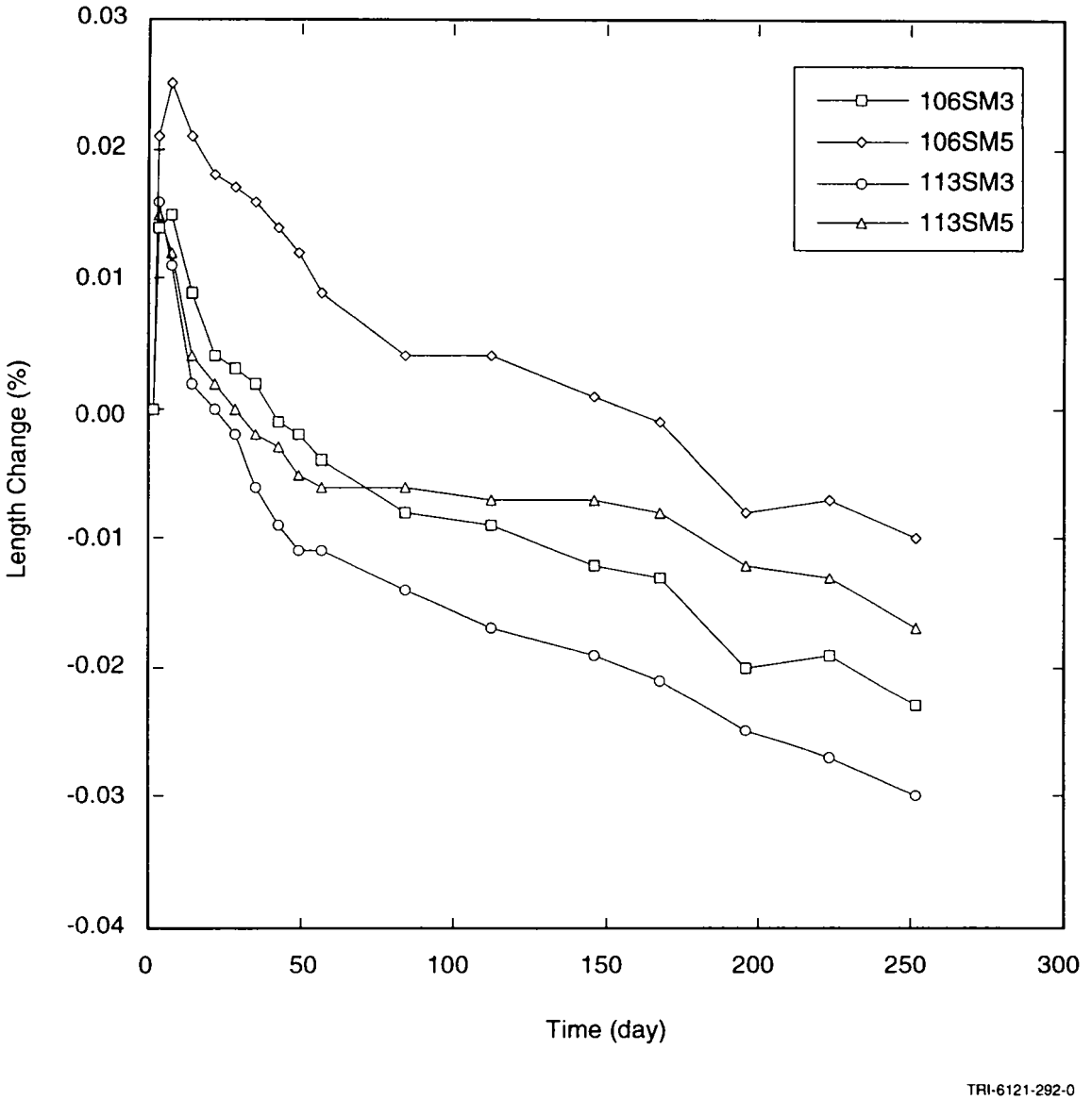

Figure 3-1. Length change of SMC-3 and SMC-5, ASTM C 157.

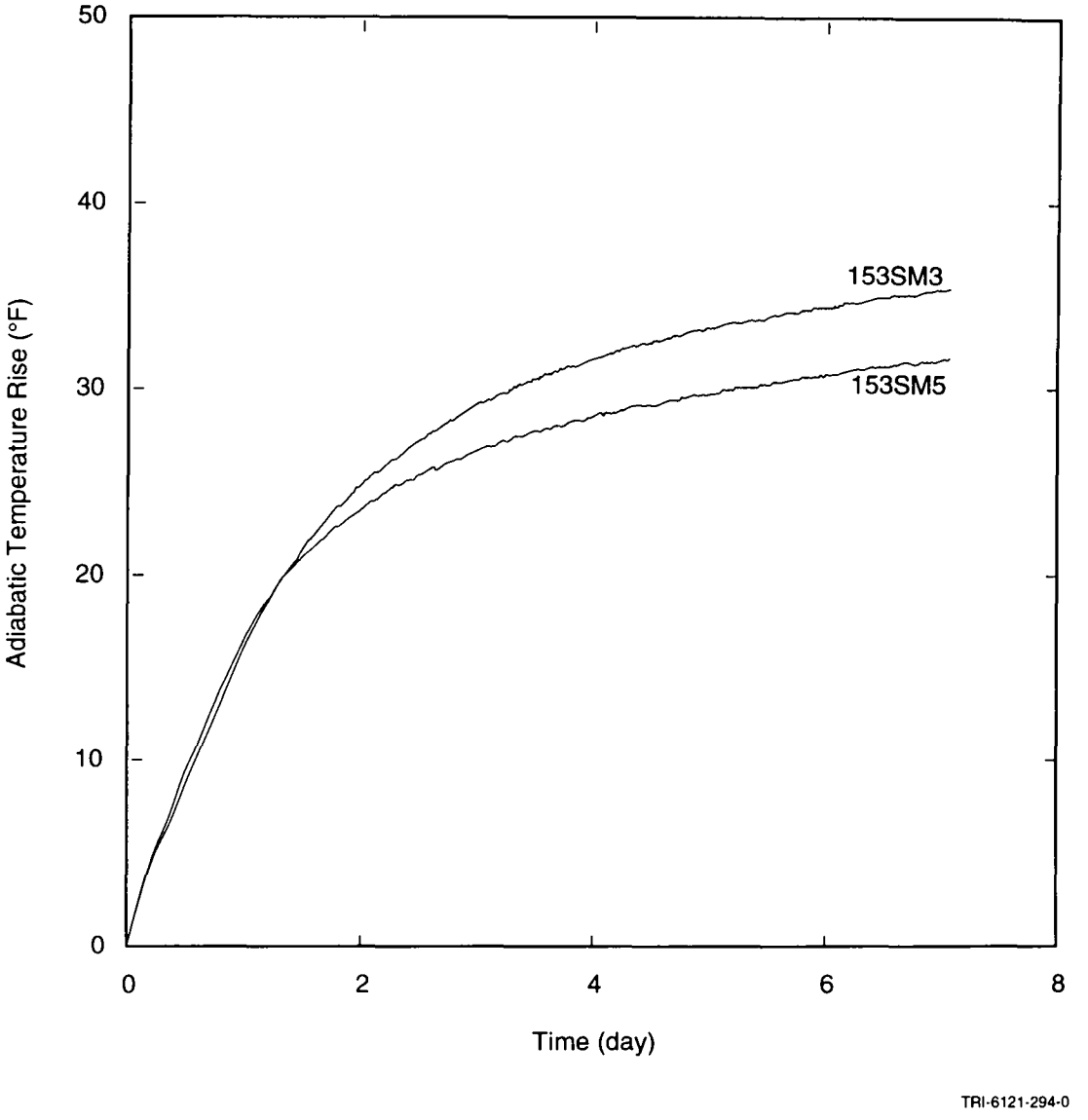

Figure 3-2. Calculated adiabatic temperature rise from Qdrum tests of batches 153SM3 and 153SM5.

3.5 Conclusions from Experimental Batches

Both SMC-3 and SMC-5 met requirements for workability and compressive strength. Elimination of sodium citrate and air-detraining admixture did not adversely affect unhardened properties. Data from volume stability measurements depict similar expansion characteristics through 30 days age, with less shrinkage by SMC-5 thereafter. Maximum shrinkage was about of 0.02% through 180 days age, which is considered volumetrically stable. A large mass of concrete protected from low humidity by being enclosed on all sides, as will be the case with a concrete seal, is likely to experience even less shrinkage.

The compressive strength measured for specimens cast from batch 174SM3 indicates that use of the set retarder, sodium citrate, may increase the compressive strength. Strength of concrete from all batches except 097SM5, which had the highest w/c, exceeded the target value of 4500 psi at 180 days. Once the option of using pre-bagged dry solids was selected, use of citrate was seen as adding unnecessary complication to underground operations. It would have to be weighed for each batch and added to the mixing water rather than to the pre-bagged material, because its solubility is decreased in the presence of salt and it is not an effective set retarder unless it is predissolved. The option of using sodium citrate could be considered again if higher slump for a longer time is determined to be essential during placement or if higher strength is required.

To determine whether or not differences in strengths of these two concretes were indicated statistically, 180-day strength values were compared for the SM5 and SM3 batches. Strength values for 174SM3 were omitted from the data set because that was the only batch containing citrate, and 097SM5 values were omitted because of its high water content. Statistically there is no difference between the ultimate strengths of SMC-3 and SMC-5. Average strength of the SM3 batches at 180 days is 4960 $\pm$ 530 psi and for the SM5 series is 4750 $\pm$ 290 psi, both above the target value of 4500 psi. The seven-day strengths of SMC-3 are significantly higher, which may be advantageous during construction. Heat signatures for these two mixtures are similar as seen in Figure 3-2 and in previous work (Wakeley et al., 1994a). Because SMC-3 had slightly higher early strength with virtually the same heat generation, larger volume batches were prepared with the SMC-3 formulation, as discussed in Chapter 4.0.

4.0 BATCHES MADE WITH PRE-BAGGED DRY MATERIALS

The objective of this part of the investigation was to demonstrate the feasibility of producing large batches of concrete from dry-batched and pre-bagged solids, while still meeting all performance requirements. This work simulated the concrete batching and mixing procedures planned for panel-seal construction at the WIPP site. Aggregates were dried and bagged by a commercial producer of pre-bagged products. The cementitious materials and salt were added at the WES and mixed in the laboratory with commercial equipment.

4.1 Materials Preparation and Batching

Cementitious materials and salt used for pre-bagging were from the sources listed in Appendix A. Aggregates were from a source convenient to the contractor who performed drying and bagging operations. Mineralogic composition and aggregate gradations met specifications and overall, the aggregates were similar to those used previously. Standard methods used for aggregate characterization are listed in Appendix C.

After being dried to meet the requirements of ASTM C 387, aggregates were blended into 15-ft^3 FIBCs. An FIBC is a large sack made of a woven fabric and used commonly for shipping large and pre-measured quantities of cements, aggregates, mineral admixtures, and pre-blended dry concrete materials. They are available in a variety of shapes and sizes, with many different options for lifting and discharging. FIBCs provide a convenient way to handle pre-batched dry solids for storage and mixing underground at the WIPP. The sacks have a duffel top, four corner lifting straps, and an integral polyethylene liner. The underside of each sack has a discharge chute. The target weight for each sack was 646 lb of fine aggregate and 790 lb of coarse aggregate. Actual batch quantities of aggregates as recorded at the commercial pre-bagging facility were within $\pm$ 1.0% by mass of design batch values. The sacks of aggregates were delivered to the laboratory shrink-wrapped on wooden pallets. The storage temperature inside the laboratory was approximately 75°F.

Dry cementitious materials and salt were added to each sack in batching operations at the WES, in amounts required for 0.5 yd^3 of fresh concrete. Cement, fly ash, and salt were the same for all sacks. However, previous studies (Wakeley et al., 1994a) of SMC-6R indicated that differences in the chemical composition of different shipments of Chem Comp III caused notable changes in concrete properties. For this reason, the sacks to be used in a preliminary batch and the first large batch were charged with Chem Comp III from one shipment and a second shipment of Chem Comp III was used in the sacks for the second large batch. This provided the opportunity to further assess the differences in characteristics of the concrete attributable to variability of this component.

Dry materials were not preblended. After all components had been added, each sack was closed and shrink wrapped for storage until mixing. Figure 4-1 shows the shrink-wrapped sacks in laboratory storage.

4.2 Large-Volume Batches

Three batches of concrete were mixed using pre-bagged materials. Actual batch weights for each component of this batch are given in Table 4-1. To establish mixing and handling procedures for the pre-bagged materials, a trial batch (159SM3) was prepared in a stationary revolving drum mixer using the contents of one FIBC. The initial slump of this 0.5-yd^3 batch was 11 in., higher than the required slump and the highest slump recorded for any batch of SMC-3. This indicated that the ratio of water to cementitious materials could be lower for large batches and still give the required slump. It is fairly common for a larger batch to require less water to achieve the same slump as a smaller batch of the same mixture. Considering the high slump of this batch, it was decided to try a lower w/c with subsequent larger batches.

Following the 0.5-yd^3 trial batch, two batches each of 5.0 yd^3 were prepared. Pre-bagged material had been stored in FIBCs for seven days in laboratory conditions prior to preparation of the first large batch (161SM3). The FIBCs remained in storage for 44 days in the same conditions before the second large batch was mixed (231SM3). Each batch was prepared in a truck-mounted concrete mixer with a capacity of 10.0 yd^3.

Because of the high initial slump of the trial batch, 10% by weight of the mixing water was withheld from batch 161SM3, reducing the w/c from 0.42 to 0.36. With this ratio, the concrete had an initial slump of 10 in. and appeared homogeneous and non-segregating. Therefore, a w/c of 0.36 was maintained for the second large batch.

Procedures followed during concrete production were the same for both 5.0-yd^3 batches. The truck-mounted mixer was inspected and positioned outdoors as shown in Figures 4-2 and 4-3. Ice (to replace 65% by mass of the mixing water) and a metered portion of the water were introduced into the mixer. Then each sack was lifted by its corner straps by a fork lift and positioned with the discharge chute over the mixer opening, as shown in Figure 4-2.

Dry materials were released from each sack by opening the discharge chute (Figure 4-3). The process of emptying 10 sacks needed for a 5.0-yd^3 batch required approximately 20 minutes. Then, additional water was metered into the mixer to bring the total water content to the required amount. The truck was moved into the laboratory at a controlled temperature and the batch was mixed intermittently for two hours at 78°F, approximately the same as the repository ambient temperature.

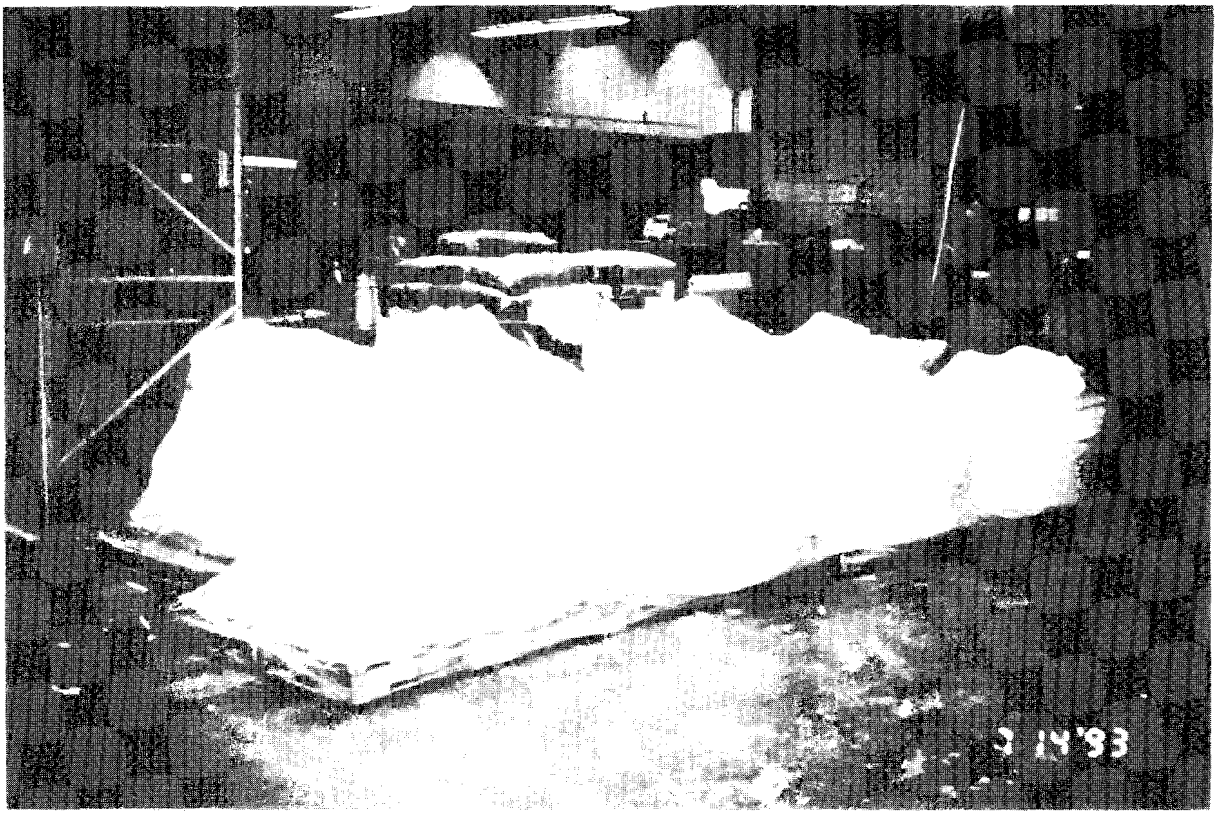

Figure 4-1. Pre-bagged materials in flexible intermediate bulk containers shown in laboratory storage.

Table 4-1. Batches from Pre-Bagged Materials

Batch Number	Batch Volume, yd^3	w/c**	SSD Batch Amounts, lb/yd^3*						
			Cement	Fly Ash	Chem Comp III	Fine*** Aggr.	Coarse*** Aggr.	Salt	Water
159SM3	0.5	0.42	278	207	134	1306	1592	88	260
161SM3	5.0	0.36	278	207	134	1301	1601	88	225
231SM3	5.0	0.36	278	207	134	1303	1605	88	225

* $kg/m^3 = (lb/yd^3) \times (0.59)$

** Water Mass, lb ÷ (Mass of Cement + Fly Ash + Chem Comp III, lb)

*** Batch Design Mass for Fine Aggregate is 1292 lb, for Coarse Aggregate is 1579 lb

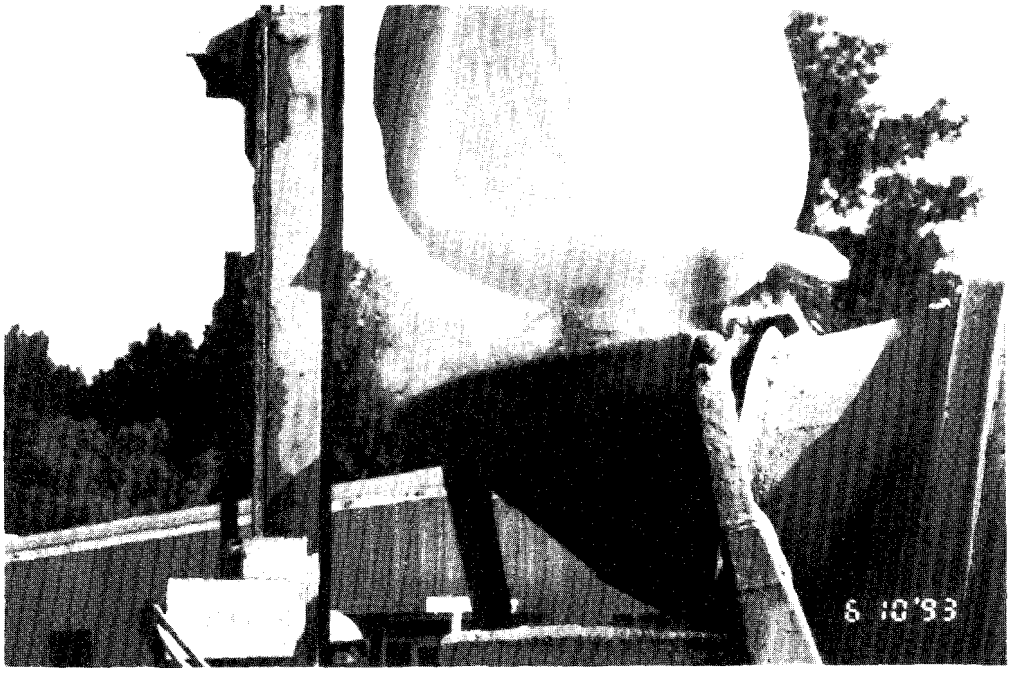

Figure 4-2. Flexible intermediate bulk container positioned over mixer for discharge of materials.

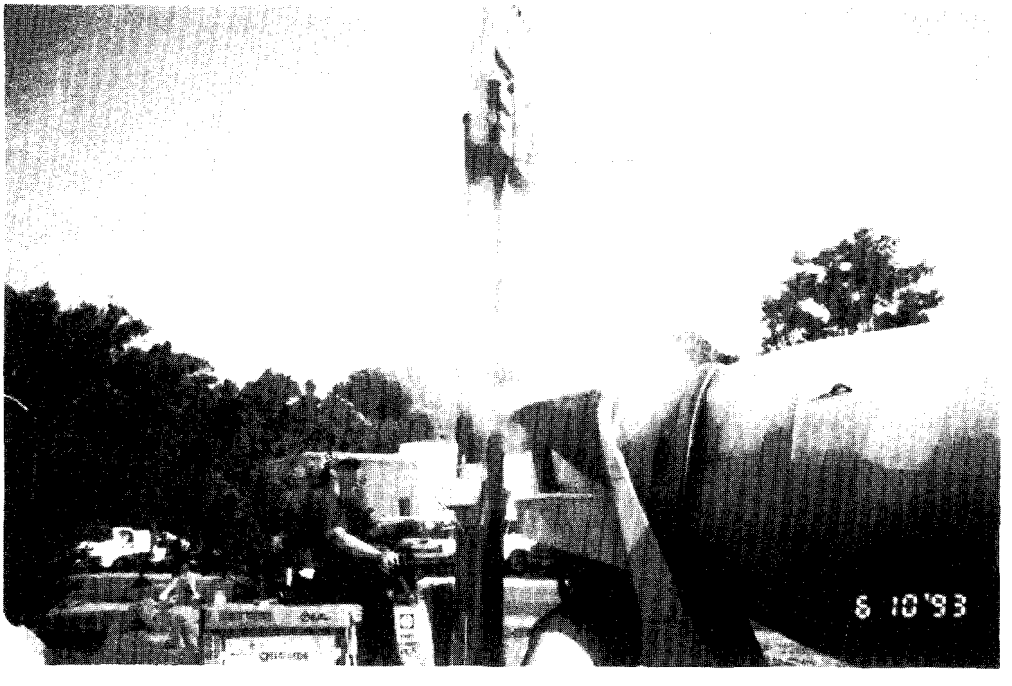

Figure 4-3. Discharge of concrete materials into mixer.

4.3 Properties Measured During and After Mixing

Initial measurements of concrete slump, unit weight, air content, and mixture temperature were made 30 minutes after the first introduction of pre-bagged solids into the mixer because of the time required to discharge the sacks. Table 4-2 reports concrete slump, monitored at half-hour intervals, and initial and final measurements for the other properties. Standard methods used for these tests are listed in Appendix C.

With a w/c of 0.36, batch 161SM3 had an initial slump of 9½ in. and an eight-in. slump after two hours of intermittent mixing (Table 4-2). No major difference in the rate of slump loss was anticipated for batch 231SM3, even though it included Chem Comp III from a different lot. Small batch 190SM3 (Table 3-4) was prepared with the same Chem Comp III used in 231SM3 and the former had maintained the required slump. In fact, batch 231SM3 had a slightly higher initial slump than 161SM3, had the same eight-in. slump at 1-1/2 hours, and then lost slump rapidly to five in. at two hours (Table 4-2).

The initial and final temperatures of batches 159SM3, 161SM3, and 231SM3 also are shown in Table 4-2. All three batches maintained a narrow range of temperature throughout the mixing period and the temperature of each was 68°F after two hours of intermittent mixing in a 78°F laboratory. The smaller batch (159SM3) had the lowest initial temperature and gained heat the fastest (17°F in two hours).

The time of setting was measured for both large batches prepared from pre-bagged materials and is listed in Table 4-3. The time of setting is also listed for preliminary batch 153SM3 for comparison of properties of batches of different sizes.

4.4 Properties of Hardened Concrete

4.4.1. Individual Specimens Cast in the Laboratory

Properties measured for samples cast from these pre-bagged batches included unconfined compressive strength and length change as an indicator of volume stability. Table 4-4 lists data from compressive-strength tests. Figure 4-4 shows length change as a percentage of initial length for prisms cast from batches 161SM3 and 231SM3, with the latter showing better shrinkage compensation. Along with measurements of unconfined compressive strength, the static modulus of elasticity was determined for batch 161SM3. Table 4-5 lists values for modulus of elasticity of 161SM3 and compares them to values from preliminary batch 153SM3, discussed previously (Section 3.3). Standard methods for measuring properties of hardened concrete are listed in Appendix C.

Table 4-2. Properties Measured during Mixing of Batches Using Pre-Bagged Materials

Mixture	Slump, in.				Unit Weight, lb/ft³*		Air, %		Mixture Temperature, °F	
	Initial	1 hr	1½ hr	2 hr	Initial	Final	Initial	Final	Initial	Final
159SM3	11	11	10	8½	145.2	145.2	1.6	1.6	51	68
161SM3	9½	8½	8	8	145.2	145.2	1.7	2.0	60	68
231SM3	10	8	8	5	150.4	147.2	2.0	2.4	56	68

* $kg/m^3 = (lb/ft^3) \times (16.02)$

Table 4-3. Time of Setting of One Preliminary Batch and Two Pre-Bagged Batches of SMC-3

Mixture	Initial	Final
153SM3	8 hr 50 min	14 hr 35 min
161SM3	8 hr 49 min	16 hr 15 min
231SM3	4 hr 13 min	8 hr 49 min

Table 4-4. Unconfined Compressive Strength, Samples from Batches Using Pre-Bagged Materials

	Compressive Strength, psi*			
Mixture	7 Days	28 Days	90 Days	180 Days
159SM3	1000	2240	3930	5050
161SM3	1380	3030	4730	5430
231SM3	1890	4560	6270	6280

* MPa = (psi) × (0.00689)

Table 4-5. Static Modulus of Elasticity, Samples from One Preliminary Batch and One Pre-Bagged Batch of SMC-3

Age (days)	Batch 153SM3*	Batch 161SM3**
	E, psi	E, psi
7	2.2×10^6	2.6×10^6
14	4.2×10^6	3.6×10^6
28	-	4.3×10^6
90	5.7×10^6	4.9×10^6
180	5.7×10^6	5.3×10^6

* Trial batch w/c = 0.42 (see Section 3.4)
** Large volume batch w/c = 0.36

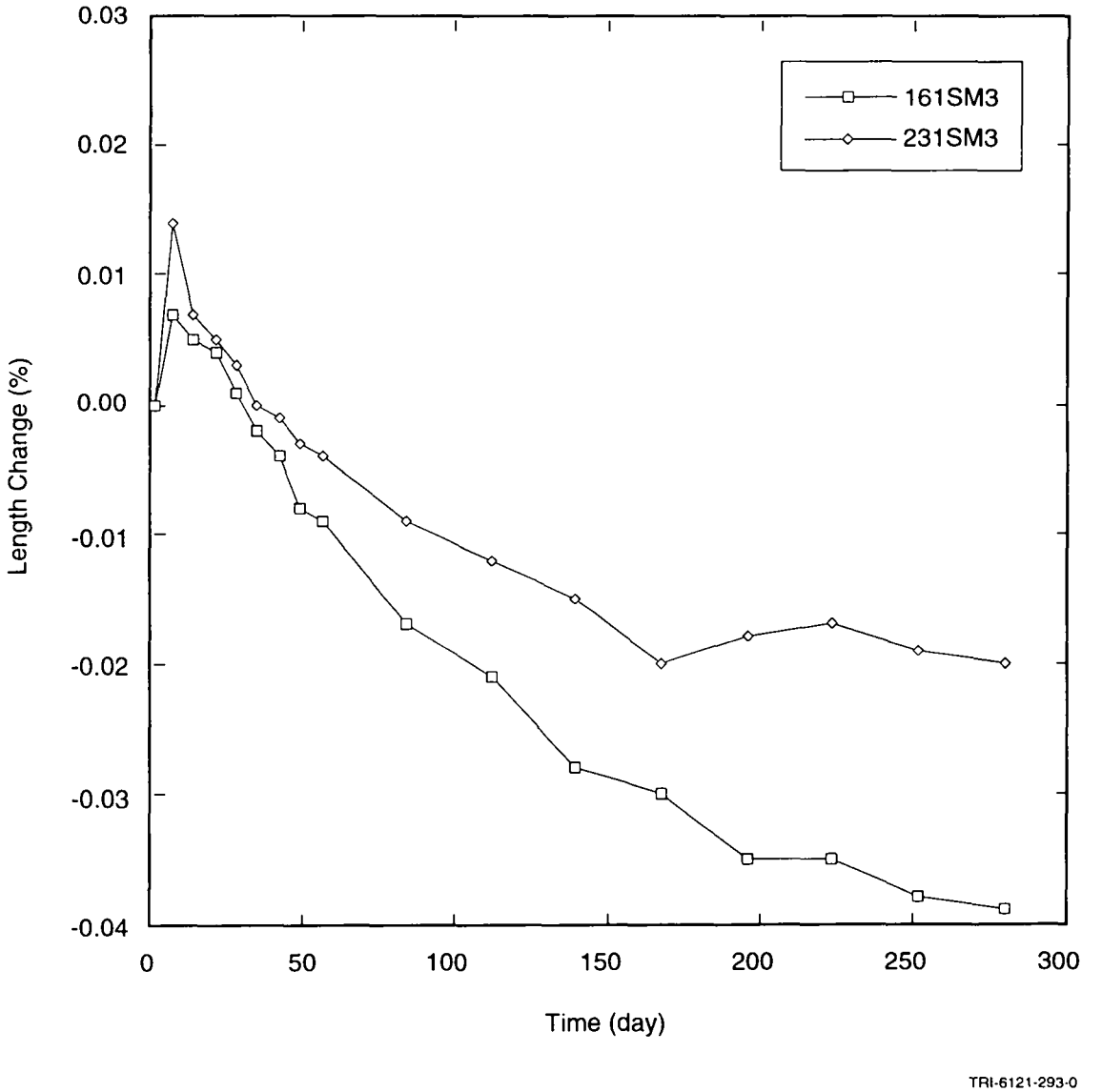

Figure 4-4. Length change of prisms from batches 161SM3 and 231SM3, ASTM C 157.

4.4.2 Thermal Screening

Heat generation of both large batches from pre-bagged materials was measured using the Qdrum device as described in Section 3.4 and approximate values for adiabatic temperature rise were calculated from these measurements. Figure 4-5 shows calculated adiabatic temperature rise of 161SM3 and 231SM3. The figure also shows comparable data for preliminary batch 153SM3 and for SMC-6R as reported previously (Wakeley et al., 1994a). At seven days, the calculated adiabatic temperature rise is 38.2°F and 45.0°F for 161SM3 and 231SM3, respectively. The difference noted in Section 3.1 can be seen here: a design choice to increase compressive strength includes increased temperature rise due to hydration of cementitious constituents.

4.4.3 Casting and Monitoring Larger Monoliths

A portion of each batch, 161SM3 and 231SM3, was discharged from the chute of the truck-mounted mixer directly into oval galvanized metal tanks with approximate dimensions four ft by eight ft by three ft. The concrete was allowed to flow freely to the opposite end of the tank. There were differences in the placing and consolidating procedures on the two occasions, as follows:

a) For 161SM3, the concrete was placed in one continuous lift, three ft deep and received minimal vibration. An internal vibrator having a three-in.-diameter head was inserted four times after the tank was filled, and each insertion lasted approximately three seconds.

b) For 231SM3, concrete was placed in two lifts each 1.5 ft deep and vibrated according to common practice. The same vibrator was inserted into both lifts 12 times and the duration of each insertion was six to eight seconds.

These two placement tests showed that properly prepared SMC is a flowable self-levelling mixture, capable of flowing at least seven ft in laboratory conditions. The energy contributed by flow down the inclined chute of the truck mixer may have contributed to the lateral flow of concrete in the tanks. Pumping tests were not conducted during these investigations.

The upper surface of each tank monolith was hand trowelled. The monoliths were covered with plastic and maintained in laboratory ambient conditions (approximately 78°F) for six months. During this time, they were observed periodically for evidence of cracking, and no visible cracks were recorded.

During planning meetings for the Large-Scale Seal Test (LSST), questions arose about effects on the concrete of using placement techniques that might allow or require SMC to flow in the forms for distances of five ft or more, and whether or not the concrete would be consolidated adequately if it could not be vibrated in the forms. The two tanks of cast concrete were the only examples of SMC that had been allowed to flow. Petrographic studies were undertaken on cores from these monoliths, to determine

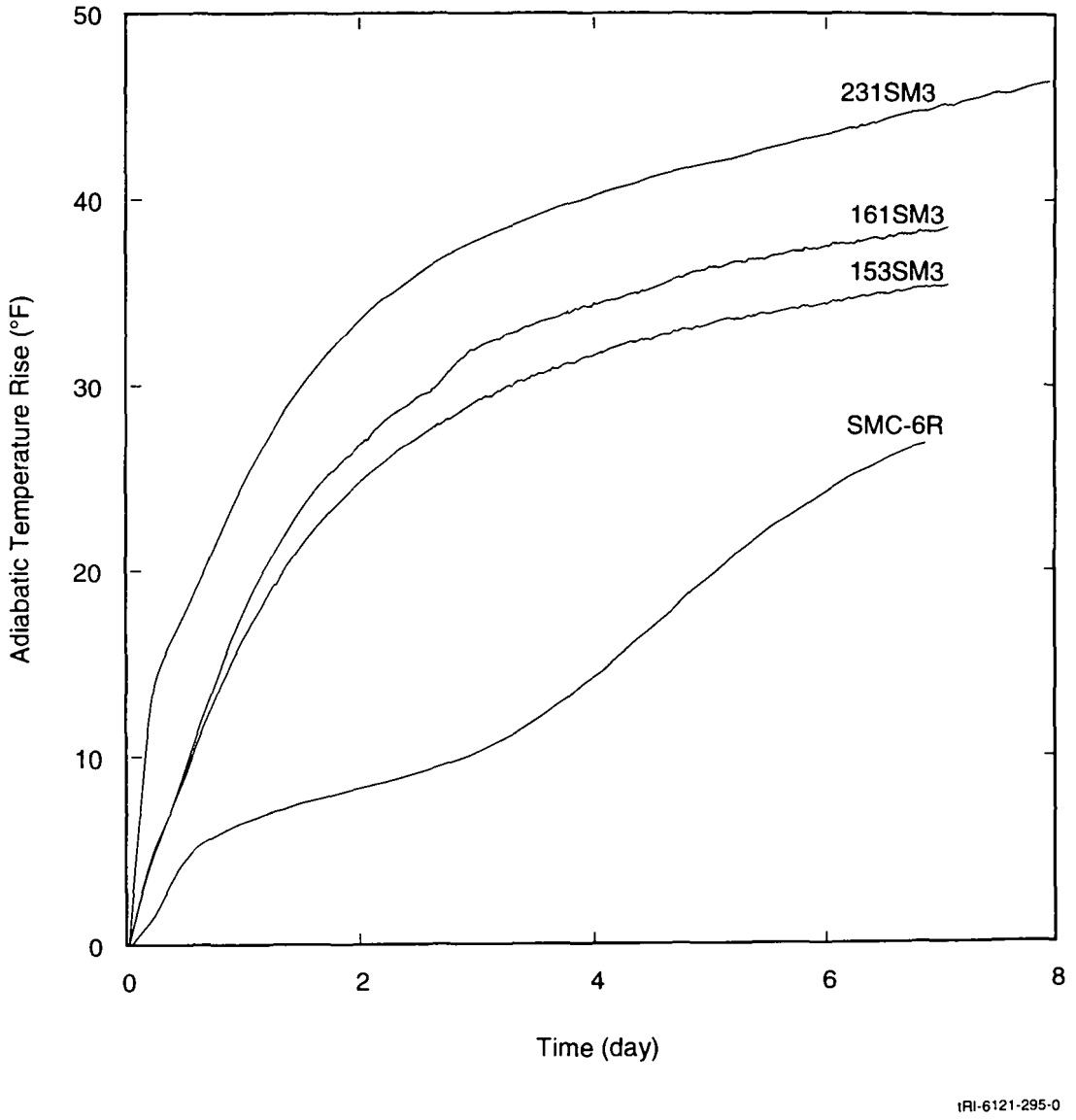

Figure 4-5. Comparison of adiabatic temperature rise of SMC-3 batches and SMC-6R (from Wakeley et al., 1994a).

if the aggregates became segregated from the cement paste when the concrete was allowed to flow, or if it had entrapped an unacceptable amount of air when it was placed with minimal vibration. Cores were recovered from four locations in each of the two tank monoliths, representing the discharge zone, the center of the tank, and two locations at the far end to which concrete had been allowed to flow. These six-in.-diameter cores were further subdivided into top and bottom portions for petrographic studies of total air content and distribution of aggregates.

Comparing the percentages of coarse aggregates in cores from the discharge area and from the far end of each tank would indicate whether or not aggregates had settled out of the paste as the concrete flowed. Values for total air content of hardened concrete from each tank were compared to air content measured for the unhardened concrete by the ASTM C 231 method (Table 4-2). If total air content counted on slabs cut from cores was far higher than the values given in Table 4-2, it is likely that air was entrapped during discharge and flow. This would indicate the need for vibration in the forms when the concrete is placed underground.

The petrographic method described in ASTM C 457 was used to determine percentages of coarse and fine aggregates, cement paste, and air voids for these 16 slabs of SMC (2 tanks X 4 core locations X 2 sections each, top and bottom). Results from these counts are in Table 4-6. They indicate a higher total air content in the concrete that was only minimally vibrated; although the air content is still acceptably low. Data from the far end of the tank for 231SM3 suggest that there may have been some aggregate segregation with this combination of free-flow and vibration. However, there is no strong evidence for aggregate segregation with flow toward the far end or from top to bottom of the monoliths.

Table 4-6. Percentages of Coarse Aggregate and Air in Concrete Cores Taken from Laboratory-Cast Monoliths

		At Discharge		Middle		Far End*	
		Coarse Aggregate	Air	Coarse Aggregate	Air	Coarse Aggregate	Air
1615M3	Top	40.3	3.7	28.2	3.3	34.5	4.4
	Bottom	47.2	4.2	27.5	4.4	35.8	4.5
2315M3	Top	27.8	2.4	34.7	1.5	29.5	2.1
	Bottom	30.5	3.2	32.0	2.5	32.7	1.7

* Values from far end are averages from two cores

4.5 Conclusions from Large Batches of SMC-3

Concrete materials were successfully pre-bagged and stored in the laboratory. The large batch size permitted a reduction in w/c to 0.36, while maintaining a high initial slump. Uniform and reproducible concrete resulted from mixing in a commercial mixer, even without preblending the dry materials before bagging. Although slump loss and time of setting varied between large batches, all batches attained the desired placement properties and exceeded the target strength at 180 days. Monoliths of 5 yd^3 apparently were crack-free during six months of laboratory storage. Concrete that was allowed to flow more than five ft during placement and was only minimally vibrated had a higher air content than adequately-vibrated concrete or concrete that did not flow. Increased vibration during placement decreased entrapped air. Aggregate segregation appeared to be minimal for this flow distance. Although the large batches involved in this prototype work are only a fraction of the volume of a seal component, all relevant mass concrete properties are favorable.

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5.0 STUDIES OF EFFECTS OF SALT AND MIXTURE TEMPERATURE

Several questions arose during planning for the LSSTs about the function of concrete components and recommended procedures. Two important issues were the effects on placing properties of use of sodium chloride in the mixture and the effects on workability of initial concrete temperature. These issues warranted special investigations within the overall framework of this study and this chapter describes experiments to answer questions about them.

5.1 Effect of Sodium Chloride on Slump and Time of Setting

It is common in concrete technology to use a high-range water-reducing admixture (ASTM C 1017) in concretes to be pumped, especially if they are expected to be self-levelling when placed. The materials selected for SMC-3 do not include an organic water-reducing admixture of a type common in concrete construction practice. Sodium chloride functions as a water-reducing admixture. A mixture that has a 10-in. initial slump at a w/c of 0.36 needs no additional water reducer.

To demonstrate the effectiveness of sodium chloride as a water reducer in SMC-3, two batches were prepared as listed in Table 5-1. The two batches have identical components, proportions, and initial material temperatures. The only difference is that the sodium chloride (salt) was omitted from one batch. Both were batched at the same time in the same conditions in 1.5 ft^3 volume. Both were intermittently mixed for two hours or until the slump was less than four in.

Table 5-1. Proportions of Batches to Demonstrate Effects of Sodium Chloride

Material	No-Salt Batch, lb*	Salt Batch, lb*
Class H Cement	10.3	10.3
Class F Fly Ash	7.7	7.7
Chem Comp III	5.0	5.0
Fine Aggregate	46.5	46.5
Coarse Aggregate	59.8	59.8
Salt	3.3	0.0
Water	9.6	9.6

* SSD batch quantities per 1.0 ft^3 of fresh concrete

The Chem Comp III was from the lot used in batches with SM4 designators and was chemically similar to the Chem Comp III used in 161SM3. The chert aggregates used for this demonstration were from a source near Vicksburg, MS and were proportioned to match the original mixture proportions as reported in Table 3-1 for SMC-3. For the batch containing salt, salt was added as a dry material with the other dry components following the procedure used for all SMC prepared during these investigations.

Table 5-2 reports the properties measured during the mixing of these two batches. Initial temperature and air content for both were similar. Initial unit weights differed slightly, as would be expected from eliminating the salt component in one. Slump values differed markedly. The salt-saturated version had an initial slump of 10 in., consistent with previously tested batches of SMC-3. It maintained a slump of more than eight in. for two hours of intermittent mixing. The salt-free version had an initial slump of only six in. This difference in initial slump indicates that the salt acts as an effective water-reducing admixture; that is, it increases the slump at a constant w/c.

Table 5-2. Properties Measured during Mixing, with and without Sodium Chloride

Property	No-Salt Batch		Salt Batch			
	Initial	30 min.	Initial	60 min.	90 min.	120 min.
Mixture Temperature, °F	51.4	58.0	51.3	56.8	61.0	62.8
Slump, in.	6	2½	10	8½	8½	8
Unit Weight, lb/ft^3	144.0	---	145.2	---	---	---
Air Content, %	1.1	---	1.4	---	---	---

--- Not Tested

The salt-free mixture lost slump rapidly, its slump decreasing to 2½ in. within 30 minutes after mixing began. Rapid slump loss is common in concretes with a large content of cementitious materials (in this case, 619 lb/yd^3) and with no set-retarding admixture to delay the early effects of cement hydration and extend workability. Sodium chloride in an amount to saturate the mixing water serves this function, as demonstrated by the eight-in slump of the salt-saturated concrete after two hours (Table 5-2). Thus, the sodium chloride functions as both a water reducer, giving higher initial slump, and a set retarder to extend the working time of the concrete.

Rapid slump loss might be considered beneficial if it caused the concrete to set faster so forms could be removed sooner. However, rapid slump loss is not always associated with more rapid setting (see Section 5.2) and is more likely to cause a situation in which concrete suddenly loses slump and becomes unplaceable during pumping operations. The two main reasons for using a water-reducing admixture are to achieve higher slump or a lower w/c. However, the initial slump is 10 in. without the additional admixture and a slump higher than 10 in. is likely to lead to segregation of aggregates. Any lower w/c would have a negative impact on the shrinkage-compensating component, which requires more water per unit volume to hydrate than portland cement. Without enough water, it would provide little if any shrinkage compensation. Thus, the use of salt and the w/c of this mixture both contribute positively to the properties of the concrete.

5.2 Effect of Higher Initial Temperature on Workability

In addition to demonstrating the effectiveness of salt as an admixture to improve and extend workability, the effect of mixture temperature on workability was also demonstrated in the laboratory. All mixing of SMC at the WES has involved cooling the mixture with ice as a replacement for a portion of the mixing water, usually at 50% by mass of water. This results in initial mixture temperatures between 52°F and 60°F and final temperatures (after two hours) between 63°F and 70°F. Use of ice to achieve required workability is a common field practice, especially where placement temperature would be about 82°F. In mass concrete placements, cooling the concrete is common to decrease the difference between maximum concrete temperature and ambient temperature, thus mitigating thermal stress and cracking potential.

To demonstrate the effect on placement properties of not cooling the mixture initially, a small batch (2.0 ft^3 volume) of SMC was prepared in a temperature-controlled facility to simulate conditions at the WIPP repository horizon. No effort was made to cool the mixture initially other than using chilled mixing water. Mixture proportions were based on those in Table 4-1, with a w/c of 0.36. All dry materials were allowed to reach 85°F prior to batching. The batch was intermittently mixed until slump loss indicated lack of reasonable workability.

Room temperature at which the batching occurred fluctuated between 82°F and 88°F during the demonstration. The batch was intermittently mixed for 1½ hours. The mixer and all tools and scales were placed in the temperature-controlled facility during the day prior to batching, also to simulate repository conditions.

The initial temperature of this batch was over 75°F. Table 5-3 reports the slump and mixture temperature at 30-minute intervals during intermittent mixing. The mixture temperatures were notably higher than the temperature of previous cooled batches. Initially, there was no difference in slump between this warm batch and cooled batches documented previously. However, significant and

Table 5-3. SMC without Ice

Time Duration, min.	Mixture Temperature °F	Slump, in.
Initial	75.1	10
30	77.3	8½
60	80.4	5½
90	82.5	< ½

Initial and final unit weight 145.2 lb/ft^3
Initial and final air content 1.8%

progressive loss of slump occurred until virtually no slump remained less than 90 minutes after mixing was initiated. Specimens were cast at that time and with difficulty, intended for measurements of compressive strength. These specimens were not vibrated because plans for underground testing at the time this study was conducted called for concrete placement without vibration. Specimens were poorly consolidated and subsequent tests of compressive strength were considered meaningless.

The time of the initial setting was approximately seven hours and the final setting was 12 hours. The initial setting of batches 153SM3 and 161SM3 required between eight and nine hours and the final setting between 14 and 16 hours. Thus, the rapid slump loss with a higher temperature did not correspond to a similar decrease in the time required for the concrete to set.

This study demonstrated that mixture temperature has a significant effect on workability and placement properties of the fresh concrete. Maintaining a cooled mixture is critical to guarantee extended and predictable placeability of this material in the WIPP repository.

6.0 DISCUSSION AND CONCLUSIONS

6.1 Discussion

Following the decision to focus on SMC-3, 13 batches of this concrete were prepared using standardized procedures for batching, mixing, and temperature control. Variables were batch size, water content, and changes in component materials. Table 6-1 summarizes batch size, w/c, slump, and temperature at intervals after initial mixing for the 13 batches. The initial slump values (t=0) of all batches were remarkably constant and within one in. of the target value of 10 in., regardless of the temperatures and water contents. However, the decrease in slump was not constant during the two hours after mixing, during which the slump was monitored.

Data in Table 6-1 suggest that both w/c and temperature affect slump loss. Statistical analyses of the data indicate that slump loss correlated with initial temperature for batches with a low w/c (0.36), but not correlated with initial temperature when the w/c was higher (0.42). However, slump loss strongly correlates with final temperature at both w/c ratios. If materials and mixture proportions are constant, it is expected that the initial temperature and the final temperature will indicate the same phenomena and show similar correlations with slump loss. Because they did not show the same correlation with slump loss, some other factor, such as variability of materials, may be controlling slump loss.

The method described in ASTM C 359 (listed in Appendix C) was used to investigate the variation in workability caused by different lots of Chem Comp III. This test method is designed to detect false setting in hydraulic cements, but is also commonly used by concrete laboratories to investigate many types of workability problems because it correlates well with behavior of concrete. The method calls for measuring the penetration of a 10-mm Vicat needle into a mortar at prescribed intervals up to 11 minutes. The mortar is not mixed or agitated during the test unless the penetration falls to a standard low level, at which point the mortar is remixed and additional penetration readings are taken to determine whether the loss of workability is recoverable by mechanical mixing action. The effect of mechanical action on recovery of workability gives information on the mechanism by which workability is lost. For this application, penetration readings were continued past the 11-minute interval to examine the effects on workability at longer times. Proportions of cement, fly ash, and Chem Comp III are the same as in SMC. Water-cement ratio (0.50), laboratory sand (50% standard sand, 50% 20-30 sand, as per ASTM C 778), and cement-sand ratio (1.00) are specified by the method.

Figures 6-1 and 6-2 illustrate performance of two lots of Class H oilwell cement and two lots of Chem Comp III used in many of the 13 batches of SMC-3, as measured by the ASTM C 359 method. Both cements showed a tendency toward early stiffening in the first few minutes of mixing, which was removed by further mixing, after which workability slowly declined as hydration proceeded. False setting is caused by the presence of calcium sulfate hemihydrate (plaster of paris) in the cement and its effects

Table 6-1. Summary of 13 Batches of SMC-3

Mixture	Batch Size	w/c	Slump, t=0	Slump, t=1 hr	Slump t=2 hr	Temp t=0	Temp t=2 hr	Chem Comp III	Comments
106SM3	1.5 ft^3	0.39	9.5	8	5	57	70	930192	deair
113SM3	2.0 ft^3	0.42	10	9.5	8.5	52	65	"	
133SM3	2.0 ft^3	0.42	10	10	8	56	68	"	
153SM3	2.5 ft^3	0.42	10	9	8	55	70	"	
159SM3	0.5 yd^3	0.42	11	11	8.5	51	68	"	One FIBC
161SM3	5 yd^3	0.36	9.5	8.5	8	60	68	"	truck
174SM3	1.5 ft^3	0.42	10.5	10.5	8.5	60	73	"	citrate
190SM3	1.5 ft^3	0.42	11		8.5	55	72	930283	Chem Comp III trial
231SM3	5 yd^3	0.36	10	8	5	56	68	"	truck
025SM4	14 ft^3	0.36	10	8.5	5.5	51	62	940023	
032SM4	13 ft^3	0.36	10	9.5	7.5	47	58	"	
032SM4	9 ft^3	0.36	9.5	8.5	6.0	48	58	"	
040SM4	2.0 ft^3	0.36	9.5	6.75	4.0	52	62	"	

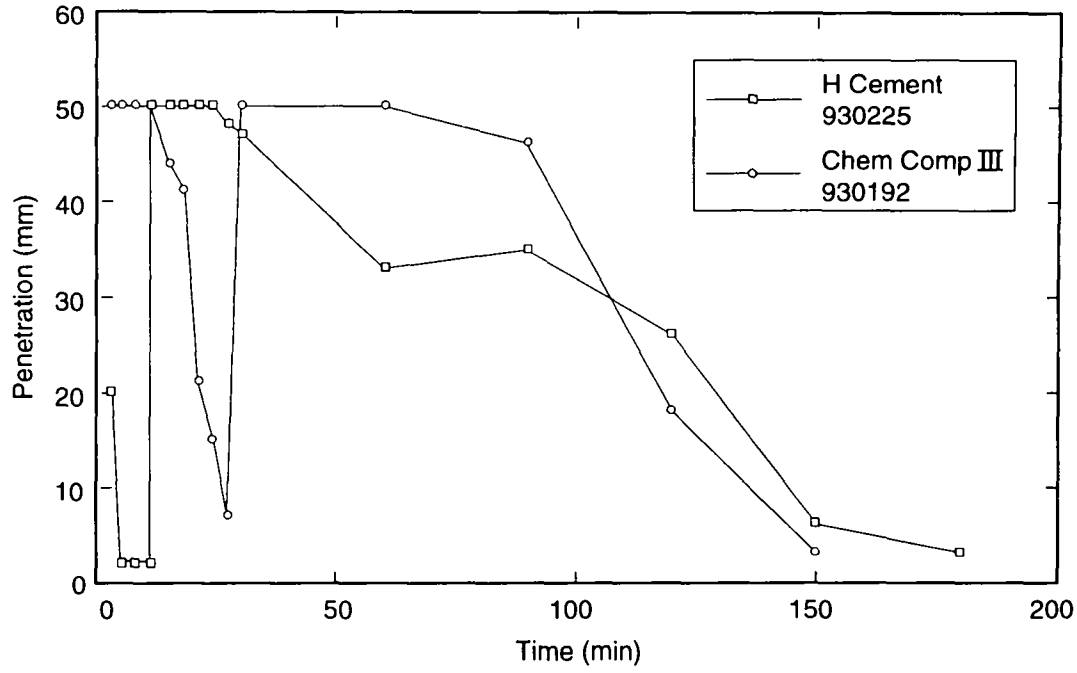

Figure 6-1. Decrease in workability with time: SM3 series.

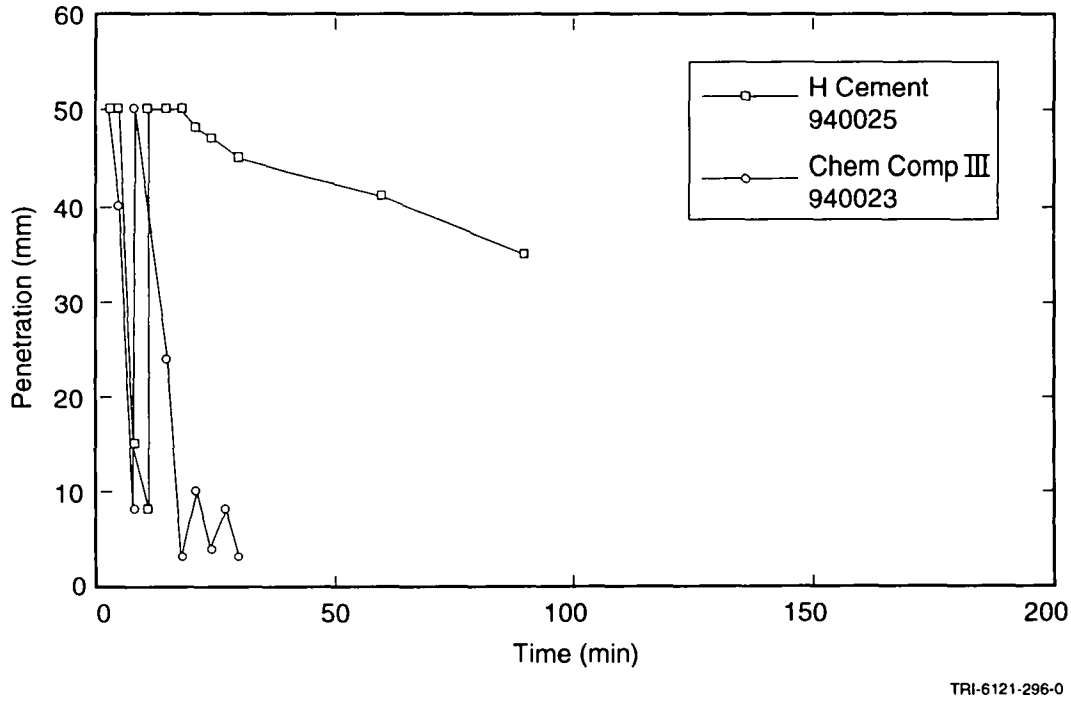

Figure 6-2. Decrease is workability with time: SM4 series.

usually can be corrected by making small changes in mixing protocol. The lot of Chem Comp III (930192) used in SM3 series batches when tested by this method showed a tendency to false set, but at a somewhat longer time (about 20 minutes). This was recoverable by remixing and normal workability was maintained for about two hours. The other Chem Comp III (940023), used in the SM4 series, caused early loss of workability that could not be counteracted by remixing. This early stiffening phenomenon is caused by accelerated hydration of cementitious components of the cement or Chem Comp III or both, and is likely to decrease working time and cause problems with concrete placement. This difference in the stiffening behavior of Chem Comp III could explain the more rapid slump loss of SMC-3 batches in the SM4 series, relative to the SM3 series.

Four different lots of Chem Comp III were tested by ASTM C 359. The two described in the preceding paragraph represent the extremes in the effect on workability. When workability properties and chemical compositions among the four were compared, no single factor of the chemical compositions of Chem Comp III emerged as a direct indicator of expected workability. This test method is probably the most practical way to detect workability problems in cementitious components and is known as a reliable indicator of field performance.

The specifications for SMC, completed in April 1994, allow w/c to vary from 0.35 to 0.42. The repository placement temperature of 82°F is expected to cause concrete mixture temperature to increase more rapidly than it did in the laboratory. Premature slump loss can be a problem during placement, particularly because the concrete will be placed by pumping and must be flowable and self-levelling for placement without vibration to be successful.

Use of sodium citrate as a water reducer for SMC was shown to increase ultimate strength. However, it also causes a condition that can be called delayed accelerated hydration. That is, mixtures proportioned with citrate maintain a high and constant slump for a longer time relative to concrete without citrate, after which slump loss is rapid as hydration accelerates. This condition was experienced during underground activities of the SSSPT Series A. The concrete, which contained sodium citrate, lost slump suddenly, during casting of individual test specimens (Wakeley and Walley, 1986). Rapid slump loss could be a useful property if it could be controlled such that the time of its occurrence was always predictable. However, it is not easily controlled and, thus, not practical in the field. Also, citrate would have to be measured separately and added separately to the mixing water, introducing another complicating factor to underground operations. Thus, in spite of the higher ultimate strength, its use is not recommended.

6.2 Conclusions

A version of SMC was developed that met the requirements for workability and placement and exceeded the strength requirement of 4500 psi at 180 days. This version, SMC-3, was proportioned with

sodium chloride as the only chemical admixture. No commercial organic chemical set retarder, water reducer, or air-detraining agent was required to achieve a uniform, non-segregating, flowable concrete with an initial slump of 10 in. and a low and constant air content. Sodium chloride was shown to function as both a set retarder and a water reducer. No air-detraining agent was needed as long as the salt was batched as a dry ingredient and was not predissolved in brine. At lower w/c, salt content was greater than the amount required to saturate the mixing water with no apparent adverse effects.

One significant concern about the SMC-3 described here is that it evolves more total heat than its predecessor SMC-6 and its heat-generation rate is faster in the first few days after placement. These thermal characteristics increase the risk of cracking from thermally induced stresses. Thermally induced cracking is not considered likely because large concrete monoliths have been constructed in salt without cracking.

Batches as large as 5 yd^3 were produced successfully from dried aggregates and other materials pre-bagged in FIBCs, and mixed in commercial ready-mixed concrete equipment. Uniform batches were achieved without pre-blending of the dry materials, although the minimum mixing time in this study was five minutes. Larger batch sizes permitted a reduction in water content, such that the concrete achieved the necessary workability at a w/c of 0.36. This w/c defined the low end of the allowable range of water-to-cement-ratios for field operations with SMC-3. Rapid slump loss was expected with lower w/c. The FIBCs were judged to be reasonably easy to fill at a commercial facility and to empty into a 10-yd^3 mixer.

Initial mixture temperature is another key to extended workability of SMC-3. All batches were cooled with ice as part of the mixing water, so that the temperature at the time of placement was 68°F or lower. Concrete with an initial temperature of 75°F that was mixed with chilled water, but with all other components at repository temperature and with no ice, lost slump rapidly and could not be cast without internal vibration. The time of setting did not decrease commensurate with loss of slump, however.

Concrete from 5-yd^3 batches was able to flow seven ft from the concrete discharge chute without measurable segregation of aggregates. Air content remained low even with minimal vibration. This suggests that placement without vibration may result in an acceptable product. Aggregate grading and particle shape can have a strong effect on concrete flow properties. Flow distances and the tendency for aggregate segregation should be tested further using the aggregate proposed for use at the WIPP and in pumping tests.

SMC-3 achieved the required properties at w/c's ranging from 0.36 to 0.42. Loss of slump is more pronounced at a lower w/c, whereas concrete at a higher w/c might not attain the required strength. The amount of water used will also affect the mixing time required, a variable that will have to be established

through mixer-uniformity tests with the materials, proportions, and mixing equipment selected specifically for field operations at the WIPP.

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APPENDIX A: SOURCES OF MATERIALS USED IN SMC BATCHES PREPARED AT THE WES

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Table A-1. Materials Used in SMC-3 and SMC-5 Laboratory Batches < 0.5 yd^3

Material	CTD Number	Description	Source
Portland cement	930225 (1993) 940025 (1994)	API Class H oilwell cement	Lone Star Industries, Maryneal, TX
Expansive cement	930192 (1993)[1] 940023 (1994)	Chem Comp III proprietary cement	CTS Cements, Los Angeles, CA
Fly ash	930226 (1993)	ASTM C 618 Class F fly ash	Pozzolanic International, Centralia, WA
	940011 (1994)		Western Ash, St. John's, AZ
Salt	laboratory stock (1993) 940043 (1994)	Food grade sodium chloride	Merchant's Co., Jackson, MS
Coarse aggregate	laboratory stock (1993)	Natural chert gravel	Mississippi Materials, Vicksburg, MS
	940040 (1994)		Green Brothers, Crystal Springs, MS
Fine aggregate	laboratory stock (1993)	Natural chert sand	Mississippi Materials, Vicksburg, MS
	940041 (1994)		Green Brothers, Crystal Springs, MS

[1] Chem Comp III numbered 930283, manufactured in a different lot, was used for batch 190SM3

Table A-2. Materials Used in Batches Made from Pre-Bagged Materials, 1993

Material	CTD Number	Description	Source
Portland cement	930225	API Class H oilwell cement	Lone Star Industries, Maryneal, TX
Expansive cement	930192 (161SM3) 930283 (231SM3)	Chem Comp III proprietary cement	CTS Cements, Los Angeles, CA
Fly ash	930226	ASTM C 618 Class F fly ash	Pozzolanic International, Centralia, WA
Salt	930276	food grade sodium chloride	Merchant's Co., Jackson, MS
Aggregates	930277	Coarse and fine aggregates bagged commercially	Quickcrete, Jackson, MS

APPENDIX B: MIXING SEQUENCE FOR LABORATORY BATCHES

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Intermittent Mixing
Laboratory Batches of 0.5 yd^3 or Smaller

Concrete batches were mixed intermittently for two hours, and properties of the fresh concrete were monitored at fixed intervals during this period. The mixing and testing sequence consisted of the following steps:

A. Coarse aggregate, half of the fine aggregate, ice (50% by mass of total mixing water), and about 20% of the remaining water are placed in the mixer and mixed for one minute. If sodium citrate is used, it is added to these materials prior to start of mixing.

B. Portland cement, fly ash, Chem Comp III, and the remaining sand are added to the mixer. With the mixer drum rotating, the remaining water and salt are added to the mixer.

C. The mixer drum is rotated for three minutes, rested for two minutes, rotated for two minutes, then initial properties are measured.

D. Following measurement of properties, uncontaminated concrete from slump test is returned to the mixer drum, and the drum is rotated for one minute.

E. The mixer drum remains stationary for approximately 25 minutes until the next series of property measurements.

F. At one hour after initiation of mixing and at 30-minute intervals thereafter, the mixer drum is rotated for three minutes prior to property measurements. Following property measurements, concrete from slump tests is returned to the drum and the drum is rotated for approximately one minute before the next period of rest.

All small batches (except those prepared for tests of effects of elevated temperature) were prepared in an air-conditioned laboratory with air temperature and materials at approximately 74°F.

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APPENDIX C: TEST METHODS AND STANDARDS APPLIED TO MATERIALS, BATCHING, MIXING, AND TESTING CONCRETE

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Table C-1. Standard Specifications for Concrete Materials

Material	Applicable Standard Tests and Specifications	Comments
Class H oilwell cement	American Petroleum Institute Spec. 10	Chemical composition determined according to ASTM C 114
Class F fly ash	ASTM C 618 , Standard Specification for Fly Ash	Composition and properties determined according to ASTM C 311
Chem Comp III	Similar to ASTM C 845; parts of standard not applicable	Composition determined according to ASTM C 114
Salt	ASTM E 534, Chemical Analysis of Sodium Chloride	Batched as dry ingredient, not as admixture
Coarse and fine aggregates	ASTM C 33, Standard Specification for Concrete Aggregates; ASTM C 294 and C 295 also applied	Moisture content determined by ASTM C 566; aggregates for pre-bagged materials meet ASTM C 387 specification

Table C-2. Test Methods Used for Measuring Concrete Properties during and after Mixing

Property	Test Method	Title
Slump	ASTM C 143	Slump of Portland Cement Concrete
Unit weight	ASTM C 138	Unit Weight, Yield, and Air Content (Gravimetric) of Concrete
Air content	ASTM C 231	Air Content of Freshly Mixed Concrete by the Pressure Method
Mixture temperature	ASTM C 1064	Temperature of Freshly Mixed Concrete

Table C-3. Test Methods Used for Measuring Properties of Hardened Concrete from Experimental Batches

Property	Test Method	Title
Compressive strength	ASTM C 39	Compressive Strength of Cylindrical Concrete Specimens
Modulus of elasticity	ASTM C 469	Static Modulus of Elasticity and Poisson's Ratio of Concrete in Compression
Volume stability	ASTM C 157	Length Change of Hardened Cement Mortar and Concrete

Table C-4. Additional Methods Used to Analyze Hardened Concrete from Large Monoliths of Concrete from Pre-Bagged Materials and to Characterize Chem Comp III

Property	Standard Method	Title
Aggregate distribution	ASTM C 856	Standard Practice for Petrographic Examination of Hardened Concrete
Total air content	ASTM C 457 (also used to quantify aggregate distribution)	Standard Test Method for Microscopical Determination of Parameters of the Air-Void System in Hardened Concrete
Loss of workability attributable to Chem Comp III	ASTM C 359	Standard Test Method for Early Stiffening of Portland Cement

APPENDIX D: CHEMICAL ANALYSES OF CHEM COMP III

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Table D-1. Chemical Analyses of CCIII Samples

Sample	Dates Used	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	LOI	Na_2O	K_2O
930192	4-6/93	8.8	9.4	0.9	40.0	0.6	25.8	11.3	0.09	0.50
930283	7-8/93	7.2	10.1	1.0	40.4	0.5	25.2	7.7	0.08	0.58
940023	1-3/94	13.2	7.8	0.9	40.5	0.9	27.4	11.1	0.06	0.50

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