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Salado Mass Concrete: Mixture Development and Preliminary Characterization

L. D. Wakeley, J. J. Ernzen, B. D. Neeley, F. D. Hansen

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ABSTRACT

A salt-saturated concrete proportioned with Class H oilwell cement, Class F fly ash, and a shrinkage compensating component was developed to meet performance requirements for mass placement as seal components at the Waste Isolation Pilot Plant. Target properties of the concrete included 8-in. slump 3 hr after mixing, no aggregate segregation, heat rise of <25°F 4 hr after mixing, compressive strength of 4,500 psi at 180 days, minimal volume change, and probable geochemical stability for repository conditions. Thermal and mechanical properties of promising candidate concrete mixtures were measured. Modulus of elasticity and creep behavior were similar to those of ordinary portland cement mass concretes. Thermal expansion for the salt-saturated concrete developed here was typical of ordinary concrete with similar silicate aggregates. Thermal conductivity, diffusivity, and specific heat approximated values measured for other mass concretes and were similar to values of the host salt rock.

MASTER

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1.0 BACKGROUND

The Waste Isolation Pilot Plant (WIPP) is a research and development facility of the US Department of Energy (DOE). Its purpose is to demonstrate the safe disposal of radioactive waste from US Defense activities. The WIPP repository facility is located in bedded evaporite rock (principally halite), approximately 2,150 ft (656 m) below the ground surface, near Carlsbad, New Mexico.

1.1 Previous Work

For nearly 20 years, the US Army Engineer Waterways Experiment Station (WES) in support of Sandia National Laboratories (SNL) has conducted research on cement-based grouts and concretes for use in the WIPP repository facility. This support for the WIPP Plugging and Sealing Program has focused on developing acceptable sealing technology for the WIPP in a technical program, including laboratory materials research and verification and field testing (Stormont, 1984).

A primary product of this research at the WES was an expansive salt concrete, developed and placed at the WIPP in 1985 and placed again in 1986, as reported previously (Wakeley and Walley, 1986; Wakeley, 1987a; 1987b; 1990; Gulick and Wakeley, 1989; Stormont, 1986; Stormont and Howard, 1986). This specialty concrete had been proportioned for small-volume placements and large and rapid volume increase (expansion), for tests of in situ permeability at early ages. The expansive concrete was not developed to be a long-term seal material, and its geochemical stability was not considered until after it had been placed. Expansive salt concrete met performance requirements of the Small-Scale Seal Performance Test (SSSPT) Series A and B (Peterson et al., 1987; Labreche and Van Sambeek, 1988). The concrete in several sizes of small plugs remained intact and well bonded to the host rock.

A major goal of the WES research on cement-based materials for the WIPP seals program during 1991 and 1992 was development of a concrete for mass placement underground in the bedded halite of the Salado Formation. The performance requirements for mass concrete differ markedly from those for small-scale seals. Mass concrete must function in placements large enough to require that measures be taken to cope with the heat generated during cement hydration and volume changes that accompany this heat generation.

According to Nowak et al. (1990), the concrete seal components will be load bearing and are intended to function for 100 years. The main function of these concrete components of the seal system is to confine and protect the reconsolidating crushed salt, which would form the primary long-term seals for periods beyond 100 years. Another function is to resist the flow of gases and to provide redundant barriers to the intrusion of brine from the Rustler Formation above the repository horizon.

During 1991, the WES conducted an extensive test program involving more than 10 different concrete mixtures. Three of these concrete formulations met the requirements of handling and placement and achieved the physical properties determined by the WES to be necessary for mass concrete for seals in the bedded salt. From these three, a single concrete henceforth called Salado Mass Concrete (SMC) was selected for extensive characterization of its thermal and mechanical properties.

1.2 Organization of This Report

This report covers two projects accomplished through laboratory research. The first was selecting materials for mass concrete for the Salado Formation, developing that concrete, and verifying its mixing and handling characteristics and early-age physical properties. Chapter 2 describes the first project. The second project, as described in Chapter 3, was to recommend the thermal and mechanical properties of mass concrete needed for modeling seal-system behavior and to measure these properties of the candidate concrete. Overall results are discussed in Chapter 4.

2.0 DEVELOPMENT OF SALADO MASS CONCRETE

2.1 Performance Requirements

The first step in the development of SMC was selecting properties for a mass concrete to be placed underground in bedded salt at the WIPP site. Because this work began before seal design or placement procedures were finalized, working constraints on performance were selected from WES experience and from previously published work (Wakeley and Walley, 1986; Deans and Andre, 1987; Gulick and Wakeley, 1989; Wakeley, 1990). Target properties during concrete development are listed in Table 2-1.

Table 2-1. Target Properties for Salado Mass Concrete

Property	Comment
4-hr working time	Indicated by 8-in. slump (ASTM C 143) after 3-hr intermittent mixing
Non-segregating	Aggregates do not readily separate from cement paste during handling
$\leq$25 °F heat rise prior to placement	Difference between initial condition and temperature after 4 hr
4,500 psi compressive strength (f_c')	At 180 days after casting (ASTM C 39)
Volume stability	Length change between +0.05 percent and -0.02 percent (ASTM C 490)
Probable geochemical stability during repository operations	Based on performance of similar concrete used in SSSPT Series A and B

The requirement for a 4-hr working time, during which the concrete is readily pumpable, self-leveling, non-segregating, and cohesive, was derived from experience during placement of the concrete in SSSPT Series A and B. That concrete was batched and mixed at the surface and placed underground in Rooms M, L1, and L2. Nearly four hours elapsed between the addition of water to the dry materials and casting of the last test specimen (Stormont, 1986). When the current study was undertaken, it was still assumed that concrete for seals would be batched and mixed at the surface and that 4-hr working time again would be required. It is clear, however, that large-volume emplacements required as seal components would be constructed differently from the SSSPT small volumes.

During development of SMC, a concrete slump (ASTM C 143) of 8 in. after 3 hr was assumed to indicate adequate workability. Having this slump also provides a concrete that is self-leveling and can be expected to fill forms even around embedded items such as gauges and cables without much in-place vibration. Requiring that the mixture be cohesive and non-segregating is good concrete practice, and ensures a more uniform product even after the extended time of setting that is common with salt-saturated concrete. The performance goal of a temperature increase of 25°F or less during handling was based on WES experience with mass concretes. If the concrete evolves heat so rapidly that its temperature increases more than 25°F, it is likely to experience rapid slump loss before hardening and excessive thermal stress after hardening. In this concrete work, these guidelines of slump, nonsegregation and flow, and temperature rise of less than 25°F were consistently obtained.

The target properties for hardened concrete are also listed in Table 2-1. The requirement for 4,500 psi at 180 days was based on a judgment of what strength is reasonably achievable by a salt-saturated concrete coupled with an estimate of strength that might be required for the panel seal to bear the load of the overlying rock. Creep closure by the rock and development of structural loads would require many years; therefore, early-age strength was not specified.

Volume stability or slight volume increase is recommended for all concrete seal components. If the concrete experiences measurable shrinkage at an early age, bonding to host rock is less likely. Although the concrete components of the seals were not recognized to be gas barriers when this concrete was developed, the WES judged that overall performance of the system would be enhanced with good bonding. Therefore, a tight tolerance on volume change was included in target properties, as indicated in Table 2-1.

Geochemical stability cannot be approximated by an ASTM test. Durability is not a measurable property of concrete. The term "durability" is used here to address the issue of degradation of the concrete in its particular service environment. For repository seals, the concrete is durable if it has adequate strength to resist design loads, maintains that strength throughout the operational life of the repository, and resists degradation that might increase permeability. A potential degradation mechanism is interaction with brines (Wakeley, 1987a). Brine is present in the repository and contact between concrete and brine is probable during the 100 years after placement. Concrete materials were selected with the demonstrated likelihood of withstanding deterioration in WIPP brines, within the constraints of the other properties needed.

2.2 Objectives of SMC Development

The starting point for selection of materials for the mass concrete was the experience gained during concrete placement in SSSPT Series A and B in 1985 and 1986. Materials selection for SMC began soon after recovery of core segments from plugs that had been placed vertically in the floor of the WIPP facility during SSSPT activities. This concrete was competent when recovered 6 years after placement. Since placement in 1985, this salt-saturated concrete had been exposed to an unknown quantity of brine. The new concrete was based on related materials to achieve the same stable phase assemblage.

The expansive concrete had been proportioned for a large volume increase (>0.1% at 14 days; Wakeley, 1987a) for early-age tests of in situ permeability in very small placements. It could not be used in large placements because it evolved too much heat during setting and its extreme expansion was difficult to achieve. But given its remarkably good condition in 1991, the expansive salt-concrete formulation was a natural starting point for selection of materials for SMC.

Desirable properties for SMC relative to previously developed salt-saturated concrete were lower heat generation, less expansion during and after setting, a simplified and reasonably reproducible system, and enhanced durability to magnesium-bearing brines. Properties to be maintained for SMC from previous concretes were an initially self-levelling quality with long working time, a low ratio of water to cementitious solids for a closed (as opposed to open) microstructure, and a salt-dominated phase assemblage for geochemical stability:

1. The first objective was generation of heat comparable to that of conventional mass concretes. Relative to the expansive concrete used in the small-scale seal tests, a concrete to be used in mass placements should generate less heat via cement hydration to decrease thermal stresses and related cracking. SMC was proportioned to be a mass concrete for large-volume placements.

2. Given that SMC will be mixed and placed in large volumes, field operations should be simplified if possible. SMC was formulated with fewer components than the previous concrete to simplify batching, mixing, and handling.

3. Demonstrating reproducibility was the third objective. It is important to show that consistent concrete properties can be produced over extended periods with similar

materials. It is probable that one or more of the materials used in SMC will become unavailable or available only in a different form during the operating life of the repository. Demonstrating reproducibility supports the reasonable expectation that adequate and defined properties can be achieved in the future from commercially available materials of the recommended chemical composition. It is essential to understand the contribution made by each concrete component to overall material performance so that modifications to the concrete can be made if concrete seal requirements change. The contributions to concrete performance of the shrinkage-compensating component and chemical admixtures will be discussed in subsequent reports.

4. The fourth objective of the experimental program was to enhance the likelihood of geochemical stability of the concrete. Components of concrete other than the cement, specifically the pozzolans and aggregates, influence the overall resistance of the concrete to the degrading effects of magnesium compounds or other brine components (Wakeley et al., 1994; Wakeley et al., 1992). The materials selected for use in mass concrete in the Salado Formation at the WIPP were chosen to be the best balance between materials for resistance to chemical attack and materials to achieve adequate workability, volume stability, and strength.

2.3 Materials to Meet the Performance Requirements

This section introduces all of the materials used in the experimental matrix for development of SMC. A complete list of materials considered and their sources is included in Appendix A. The formulation for SMC first recommended by the WES in September 1991 and currently being refined **does not include all of these materials**. Mixtures proportioned with some of these materials failed to meet performance requirements, after which that material was eliminated from further laboratory studies. The reason each material was considered is given here. After each material is introduced, Section 2.5 on proportioning explains how the materials and amounts thereof were adjusted to meet performance requirements and research goals.

2.3.1 Selection of Cement

Of the performance requirements listed in Table 2-1, those most affected by choice of cement are heat generation and resistance to chemical attack. Cement hydration—the chemical

reactions between cement and water that result in strength-giving phases—are highly exothermic for the first three or four days after placement and continue to generate heat at a diminishing rate thereafter. Large-volume concrete placements must minimize heat generation to avoid cracking due to contraction on cooling that follows early thermal expansion.

To help alleviate thermal effects during the first critical days after concrete placement, an API Class H moderate sulfate-resisting cement was selected for SMC. Class H cement is more coarsely ground (has a lower surface-to-volume ratio) than ordinary portland cements and therefore hydrates more slowly and generates heat more slowly than ordinary portland cement, resulting in less heat-related early expansion. Concrete is better able to withstand stresses later, after it has had time to gain more strength. Avoiding excess thermal tensile stresses early in its history, when its strength is still low, is the best way to avoid cracking young concrete.

Class H cement has a long history of successful commercial use in the oil industry (Smith, 1990) and has been used in many grouts and concretes for the WIPP and other waste isolation projects (Gulick and Wakeley, 1989; Wakeley, 1990; Wakeley and Ernzen, 1992). The low alumina content of Class H cement also makes it more resistant to sulfate attack. This cement was used for all variants of SMC during concrete development. The chemical analysis and physical properties of the cement selected for SMC are shown in Table 2-2.

Table 2-2. Chemical and Physical Data for Class H Oilwell Cement

Chemical Analysis	(%)	Physical Properties	
SiO_2	22.4	Surface Area (Blaine)	235 m^2/kg
Al_2O_3	4.0		
Fe_2O_3	3.9	Autoclave	-0.03%
CaO	64.0	Expansion	
MgO	1.5		
SO_3	2.6		
Loss on Ignition	0.7	Initial Set (Gil)	155 min
Insoluble Residue	0.26	Final Set (Gil)	205 min
Na_2O	0.28		
K_2O	0.63	Air Content	9%
TiO_2	0.22		
P_2O_5	0.08	3-day Strength	1,490 psi*
		7-day Strength	2,420 psi

*Cement paste with water

2.3.2 Selection of Fly Ash

Both Class C and Class F fly ashes were tested during this program. Fly ash types are defined in ASTM C 618. Class C fly ash was considered because previous expansive salt-saturated concretes had been proportioned successfully with a large amount of this ash. It provided a source of reactive phases needed for the expansion that had been required in the small-scale seals. On the other hand, Class C fly ash usually contributes to early heat generation, and its hydration products are characteristically more susceptible to reactions with sulfates and other forms of chemical attack.

A large amount of Class F fly ash was used in proportioning most variants of SMC during mixture development. Class F fly ash is used commonly in Corps of Engineers mass concretes because it contributes heat more slowly than do most cements, but it ultimately contributes to strength gain and a closed microstructure. Its particle shape and slow chemical reaction also contribute to enhanced workability, ease of pumping, and longer working time. Ultimately, only concretes proportioned with the Class F ash met all the performance requirements and satisfied the project goals (see Sections 2.5 and 2.6). The chemical analysis and physical data for the Class F fly ash that was used in the concretes discussed in Section 3.0 are listed in Table 2-3. Comparative data for the Class C fly ash are included in Appendix A.

Table 2-3. Chemical and Physical Data for Class F Fly Ash

Chemical Analysis	(%)	Physical Properties	
SiO_2	46.0	Fineness (45 micron), % retained	23
Al_2O_3	22.9		
Fe_2O_3	5.0	Fineness variation,	3%
Total	73.9		
		Water Requirement,	94%
CaO	-		
R Factor	-	Density,	2.18 Mg/m^3
MgO	1.8	Density variation,	4%
SO_3	0.5		
		Autoclave Expansion,	-0.03%
Moisture Content	0.1		
Loss on Ignition	0.7	Pozzolanic Activity w/lime,	1,060 psi
Available Alkalies	1.14		
		Strength Activity Index w/cement, 7-day	81%

[1]Must be more than 70% (ASTM C618)

2.3.3 Other Concrete Components

Two additional cementitious materials used in the proportioning of SMC variants were Cal-Seal and Chem-Comp III. Both are proprietary materials designed to give early expansion to the fresh mixture for various reasons, including offsetting the effects of drying shrinkage. Both were included in the expansive concrete used in the small-scale tests. In the current study, only mixtures proportioned with Chem Comp III met performance requirements, as discussed in the next section. Table 2-4 gives data on the different lots of Chem Comp III used during 1991-1992.

Granular sodium chloride sufficient to saturate the mixing water was used in all variants of SMC. Field experience and laboratory studies conducted by the WES have shown that salt-free grouts and concretes, when placed in contact with the halite, form no bond with the host rock. The mixing water dissolves some of the host rock at the interface. By contrast, a salt-saturated cementitious material bonds well with the salt (Wakeley et al., 1993).

Sodium citrate functioned as a water-reducing admixture and set retarder for all variants of SMC tested in 1991-92. It had been used successfully in the small-scale tests, providing the extended working time needed for mixing at the surface and placing at the repository horizon. Previous work at the WES and elsewhere had shown that many commercial set-retarders and water-reducing admixtures contribute unacceptable properties to salt-saturated concretes. Many cause a very high air content, or foaming (Grutzeck, 1984), which could result in low strength and high permeability. Others can cause rapid slump loss or can retard setting for days or weeks (Wakeley and Walley, 1986). For additional assurance of strength and low permeability, a commercially available air de-training admixture (D-Air-1) was used to maintain the air content of SMC below 2%. This admixture also had been used successfully in the small-scale seals work (Wakeley and Walley, 1986). Appendix A includes a list of component materials and their sources.

2.3.4 Aggregates

The aggregates used in proportioning SMC were a siliceous river gravel, consisting principally of rounded chert particles, and a natural sand of similar composition. Both were obtained from commercial suppliers near the WES. A dolomitic aggregate available near Carlsbad, NM had been used in previous concrete shaft liners. But this type of aggregate had

Table 2-4. Chemical Data for Chem Comp III Used in Development of SMC

Chemical	Percentage			Range
SiO_2	7.1*	7.2**	5.0***	2.2
Al_2O_3	7.1	6.9	4.7	2.4
Fe_2O_3	0.9	0.9	0.6	0.3
CaO	41.7	41.1	38.0	3.7
MgO	0.8	0.8	0.6	0.2
SO_3	27.1	26.8	33.6	6.5
Loss on ignition	11.0	11.5	14.9	3.4
Insoluble residue	0.48	0.41	0.39	0.09
Na_2O	0.07	0.06	0.06	0.01
K_2O	0.48	0.41	0.32	0.16
Alkalies total as NaO_2	0.42	0.4	0.27	0.15

* Used in developmental mixtures with pea gravel.
** Used 040692 through 060592; see Section 3.2.
*** Used 081392 and 092192; see Section 3.2.

shown vulnerability to chemical alteration by reaction with brines in concrete of the WIPP waste-handling shaft liner (Wakeley et al., 1992). Siliceous aggregates have exhibited superior resistance to chemical attack in many environments and are under study at the WES for their durability in simulated WIPP brines.

2.4 Balancing Heat Rise, Workability, and Strength

The first concrete mixed in this study was essentially the expansive salt concrete recreated with 1991 materials. This demonstrated that the same concrete properties could be achieved using materials obtained from different sources and several years later, as long as the materials were well characterized and mixture proportions were adjusted carefully to accommodate changes in chemical composition or physical characteristics. This met some of the objectives of reproducibility. However, Class C fly ash contributes significantly to early heat rise in this concrete, exceeding the target temperature rise. The following sections explain the logic of trying to balance the requirements for low early heat, adequate workability, and adequate strength, as well as to achieve other target properties.

2.4.1 Decreasing Thermal Stresses

Class H cement from the same source as was used in the small-scale seals was selected for use in the SMC. As explained previously, it had a proven performance record and possessed properties necessary for the SMC, such as low surface area for low heat and extended workability, and proven resistance to sulfate and other chemical attack. To reduce the heat rise of the mixture, the proportions of cement and fly ash were adjusted to include more fly ash than had been used previously.

Class F fly ash hydrates at a slower rate than does portland cement. Therefore, less heat will be generated immediately after the concrete is placed if the concrete contains less cement and more Class F fly ash. This change in hydration will cause the concrete to have lower compressive strength at early ages (less than 28 days). However, high early strength is not required of a mass concrete for seals: the target value was 4,500 psi at 180 days (Table 2-1). This design consideration allows an increase in the ratio of fly ash to cement for SMC. Concretes containing Class F fly ash have a proven record of gaining strength later, so achieving the target compressive strength was a reasonable expectation even with a high percentage of fly ash.

Natural sand and 3/4-in. nominal maximum size natural chert coarse aggregates available near the WES were used in all trial batch mixtures. The mixtures were initially proportioned using 3/8-in. aggregates because this smaller aggregate size had been used in the small-scale seals concrete. Upon selection of the candidate SMC for testing of thermal and mechanical properties, concrete proportions were adjusted to accommodate 3/4-in. aggregate. Because larger aggregates have a smaller surface-to-volume ratio, a smaller volume of cement paste is required to coat all aggregates and form a continuous matrix of the mortar fraction of concrete. This allows reproportioning to reduce the total cement content of the concrete, and thus reduces the total heat output of the concrete for mass placements. Aggregate gradation was selected to meet American Concrete Institute requirements of ACI 304.R for proportioning pumpable concrete (American Concrete Institute, 1993).

Larger aggregates commonly are used in mass concretes to decrease early heat even more. Smaller aggregates were selected here in anticipation of difficult placement conditions and possibly long drop or pumping distances. The aggregate sizes chosen were a balance between achieving needed thermal properties and ensuring that the concrete would be placeable under a variety of conditions.

2.4.2 Simplifying the Mixture

The very high expansion required for small-scale tests was difficult to achieve. Both Chem Comp and Cal Seal had been needed, along with the Class C fly ash, to enhance expansive characteristics. These components, in turn, required a large dose of sodium citrate to maintain adequate workability for 3 to 4 hrs. In the present work as well as in previous experience, concretes with more components and with tight tolerances for admixture dosages are more difficult to control in field activities and more difficult to reproduce than simpler mixtures.

As described by Nowak et al. (1990), concrete used in seals does not need to function as a gas barrier at early ages. Although design guidance is still being developed, concrete panel and drift seals may not be required to be load bearing or gas-flow retarding until several months after placement. It is unlikely that seals will be exposed to measurable fluid pressures soon after placement. Volume stability or minimal expansion of the panel seal should be adequate to enhance initial bonding to the host rock. The creep closure of halite toward and into excavations (Van Sambeek, 1987) would then ensure a tight interface for longer term performance. In the current study, concretes were proportioned to achieve only slight positive expansion or volume stability. This was consistent with the selection of Class F fly ash for lower heat, but unlike

Class C ash, Class F ash has little potential to contribute to expansion. The experimental matrix still included two mixtures proportioned using a Class C fly ash for comparison.

2.4.3 Improving Resistance to Deterioration in Brine

The rights to manufacture a product under the patent for Chem Comp III have been held by several different companies, and the materials have undergone several manufacturing changes since Chem Comp III was used in concrete for the SSSPT in 1985. Because of the need for reproducibility, it was necessary to prove by testing that the new product would give essentially the same properties as had the previous formulation.

The need for improved resistance to possible chemical attack also suggested a reduction in the amount of Chem Comp III. It was determined that the target expansion for panel-seal concrete could be achieved with less Chem Comp III or Cal Seal in combination with a Class F fly ash, and it was not necessary to use both expansive components together. This reduction in Chem Comp III also served to reduce the concrete's vulnerability to chemical attack by reducing total reactive alumina. In addition, decreasing the amount of Chem Comp III reduced early heat generation to make longer working times easier to achieve and more consistent.

In addition to its ability to reduce temperature rise in concrete, Class F fly ash is widely known to contribute to resistance of concrete to chemical attack, when compared to concretes made with equal amounts of Class C fly ash (Dunstan, 1976; Helmuth, 1987; Day and Ward, 1988; Mehta, 1988). However, most of this previous work addressed resistance to sulfate attack and not the resistance of concrete to high concentrations of magnesium in groundwater. Recent and current work at the WES (Wakeley et al., 1992; Wakeley et al., 1994) indicates some improved resistance to magnesium-related deterioration for mortars made with Class F fly ash.

As listed previously, salt (NaCl), sodium citrate, and an air-detraining admixture also were components of all experimental SMC mixtures. Sources of these components are given in Appendix A.

2.5 Proportioning and Testing Trial Batches

Ten concrete mixtures and variants were proportioned in laboratory investigations to determine the combination of materials that would meet all requirements for the SMC.

Table 2-5 shows the cementitious components of the 10 mixtures with each component reported as a percentage of the total volume of cementitious solids. Table 2-6 reports the mass of each component required for a 1-yd^3 batch of concrete. For each mixture, the total volume of cementitious materials was held constant. The matrix was designed to explore which expansive admixtures to retain and what amounts were required to achieve target properties.

Table 2-5. Cementitious Material Proportions

Mixture No.	% by Volume of Total Cementitious Materials				
	Cement	Class F Fly Ash	Cal Seal	Chem Comp III	Class C Fly Ash
1	38	27	9	26	0
2	38	36	0	26	0
3	38	43	0	19	0
4	38	48	0	14	0
5	31	50	0	19	0
6	27	54	0	19	0
7	38	42	9	0	11
8	38	26	9	0	27
9	38	39	0	23	0
10	38	48	14	0	0

2.5.1 Proportioning

Mixture 1 in Table 2-6 was a repeat of the expansive salt concrete but with Class F fly ash substituted for Class C fly ash. In mixtures 2, 3, and 4, Cal Seal was eliminated and the Chem Comp III was reduced in steps and replaced with equal volumes of fly ash, while the volume of cement remained constant at 38% of the cementitious materials. In mixtures 5 and 6, the proportion of Chem Comp III was held constant at a median value of 19% by volume, and the fly ash was increased while the volume of cement was reduced. The purpose of mixtures 1 through 4 was to determine if mixtures containing Class F fly ash could be made non-shrinking by use of Chem Comp III, and to identify the minimum acceptable level of Chem Comp III. Mixtures 5 and 6 were proportioned to minimize heat.

Table 2-6. Trial Mixture Proportions for Salado Mass Concrete

Mixture No.	Batch Weights, lb/yd³*										
	Cement	Class F Fly Ash	Cal Seal	Chem Comp III	Class C Fly Ash	Fine Aggregate**	Coarse Aggregate**	Water	Salt	Sodium Citrate	Air-Detraining Admixture
1	317	156	64	184	0	1,433	1,283	292	100	5.9	7.2
2	317	208	0	184	0	1,433	1,283	292	100	5.2	7.1
3	317	248	0	134	0	1,433	1,283	292	100	2.1	7.0
4	317	273	0	98	0	1,444	1,293	288	99	1.6	6.8
5	252	281	0	131	0	1,456	1,303	284	97	2.1	6.6
6	216	299	0	129	0	1,467	1,313	280	96	3.2	6.4
7	304	233	62	0	76	1,467	1,313	280	96	0.7	6.8
8	304	144	62	0	185	1,467	1,313	280	96	1.2	7.0
9	317	225	0	163	0	1,433	1,283	292	100	3.6	7.1
10	304	266	95	0	0	1,467	1,313	280	96	1.3	6.7

* To obtain Kg/m³ divide lb/yd³ by 1.6875.

** Saturated surface-dry condition.

In mixtures 7 and 8, the Chem Comp was eliminated and the original 9% by volume of Cal Seal was restored. The volume of cement was restored to 38% as in mixtures 1-4, and the remaining cementitious materials were split between Class F and Class C fly ash. This was done to determine if using Cal Seal alone could provide adequate expansion, which would be advantageous because Chem Comp reacts more quickly and reduces workability sooner. Mixture 9 was a step between mixtures 2 and 3. Mixture 10 used a larger amount of Cal Seal (14 percent by volume) and eliminated both Chem Comp and Class C fly ash.

The ratio of water to cementitious solids was held constant at 0.35 as calculated by the ACI 211.1 absolute volume equivalency method for all mixtures. This resulted in a ratio of water to cementitious materials of 0.41 to 0.43 by mass. Chilled water was used to lower the initial concrete temperature to approximately 55°F, and thus increase working time. The water content was lowered as the volume percentage of fly ash was increased (it has a lower mass per unit volume than cement). Sodium citrate was varied to provide sufficient workability. In general, the amount of sodium citrate was proportional to the amount of Chem Comp, that is, larger amounts of sodium citrate were required to achieve adequate working time in mixtures having larger amounts of Chem Comp. The other cementitious materials did affect the sodium citrate dosage, but percentage of Chem Comp was the controlling variable. The disadvantage to using large amounts of sodium citrate was that the setting time was also delayed as the dosage was increased and early age properties became less predictable.

The concrete mixtures were batched and mixed by procedures given in ASTM C 192. After the prescribed mixing time, the concrete was tested for slump (ASTM C 143), unit weight (ASTM C 138), air content (ASTM C 231), and temperature (ASTM C 1064). The concrete was then mixed continuously until the slump decreased to less than 8 in. The slump was measured at 1 hr, 2 hr, 2-1/2 hr, and 3 hr. If the concrete did not have at least an 8-in. slump at 3 hr, the mixture was discarded. If the concrete maintained at least an 8-in. slump for 3 hr, the unit weight, air content, and temperature were again measured and specimens were cast.

Twelve cylindrical specimens (4 in. diameter by 8 in.) were cast (ASTM C 192) from each mixture for tests of unconfined compressive strength (ASTM C 39) at 7, 28, 90, and 180 days. Three unrestrained prisms (3 in. cross section by 10 in. long) were cast for determining length change as an indicator of expansion (ASTM C 157). Additional cylindrical specimens were cast for aging in simulated brines.

2.5.2 Properties of Unhardened Concretes

The required workability was easily attained in all mixtures for up to 2 hrs and in some mixtures for up to 2-1/2 hrs. The 2-to-2-1/2 hr time frame is a critical period. Within this period, the slump usually started to decrease rapidly for some mixtures. For example, the slump of mixture 1 decreased from 8.5 in. at 2-1/2 hr to just over 2 in. at 3-1/2 hr. For most mixtures, it was difficult to maintain an 8-in. slump beyond 3 hours, especially in the mixtures containing Chem Comp.

The difficulty in maintaining acceptable workability increased as the amount of Chem Comp increased. Only mixture 10, which did not contain Chem Comp, maintained an 8-in. slump for the required 4 hours, and it required more than 2 days to attain final set. It is probable that the slump requirement could have been met if larger doses of sodium citrate had been used, but severe retardation of setting would have resulted. Concrete slump to 3-1/2 hr, air content, unit weight, time of setting, and other properties of the unhardened concretes are given in Table 2-7.

Initial and final setting times were determined as described in ASTM C 403 with one change. Due to the extended setting times of these concretes, a penetration resistance value of 2,000 psi was used to define the condition of "final set," rather than the 4,000 psi as required in ASTM C 403. Rationale for this change was that the 4,000 psi requirement was not appropriate for salt-saturated concretes for which the time of setting is retarded significantly and the rate of strength gain is slow after set has been achieved. Some of the concretes might not have reached the ASTM C 403 value for 2 to 3 days.

2.5.3 Properties of Hardened Concretes

Due to the characteristics of the cementitious materials used in the SMC, early-age compressive strengths were low, generally near 1,000 psi at 7 days. However, six of the mixtures were near the target compressive strength by 90 days. Only mixtures 7 and 10 failed to reach 4,500 psi by 180 days. All other mixtures met or exceeded this value. Results of tests of compressive strength are given in Table 2-8.

Volume stability of the candidate concretes was monitored for 8 months. Concrete prisms were stored in a chamber for which temperature was controlled at 80 $\pm$ 2°F, to approximate conditions at the repository horizon. Relative humidity could be maintained only

Table 2-7. Unhardened Concrete Properties

Mixture No.	Slump, in.						Unit Weight, lb/ft^3		Air, %		Mixture Temperature, °F		Time of Setting, hr	
	Initial	1 hr	2 hr	2-1/2 hr	3 hr	3-1/2 hr	Initial	Final	Initial	Final	Initial	Final	Initial	Final
1	10-1/4	9-3/4	9-1/4	8-1/2	8	2-1/4	139.2	143.6	3.6	2.6	69	78	7:40	9:45
2	11	10-1/2	10	9-1/4	9-1/4	4-3/4	140.8	143.2	3.6	2.6	63	80	8:30	11:50
3	11	10-1/2	10	9-3/4	9	7	141.6	142.4	1.5	1.6	67	82	12:55	22:55
4	10-3/4	10-1/4	10	9-1/2	9	7	140.4	143.2	2.1	1.9	60	81	17:05	26:30
5	10-1/2	10-1/2	10	10	9	7-1/2	140.8	142.8	2.0	1.9	62	80	14:10	28:55
6	10-3/4	10-1/2	10	8-3/4	8	*	140.0	142.0	2.9	2.6	55	81	19:30	44:05
7	10-3/4	10-1/4	9-1/4	9	8	*	137.6	143.0	4.9	2.4	64	82	22:35	26:30
8	10-3/4	10-1/2	9-3/4	9-1/4	9	7-1/4	138.0	143.2	5.2	2.9	50	82	40:35	47:30
9	10-1/2	10-1/2	10	9-3/4	8-1/4	*	142.0	142.8	1.8	1.8	57	80	11:55	32:35
10	10-3/4	10-1/2	10-1/4	10-1/4	10	9-3/4	137.6	143.2	4.2	2.2	57	79	48:15	53:25

* Test not run; specimens cast at 3 hr age.

Table 2-8. Compressive Strength of 10 Original SMC Mixtures

Mixture No.	Compressive Strength psi*			
	7 Days	28 Days	90 Days	180 Days
1	820	2,400	-	4,870
2	1,090	2,550	3,660	4,950
3	1,540	3,160	4,450	6,650
4	1,320	2,900	4,550	5,690
5	1,070	2,630	4,570	5,400
6	770	2,450	4,640	5,650
7	1,320	2,250	3,310	4,450
8	1,800	3,790	5,940	6,830
9	1,230	2,710	4,930	6,410
10	870	1,740	2,970	3,510

*To convert psi to MPa, multiply psi by 0.00689.

approximately in the chamber large enough to accommodate all specimens and ranged from 50 to 75%. Three mixtures (1, 2, and 9) exhibited expansion greater than the target value (Table 2-1). Most mixtures attained volume stability within the first month after casting and remained constant thereafter. Figures 2-1 and 2-2 show these data.

2.6 Preliminary Thermal Screening

At this point in the investigation, four candidate concretes were selected for further investigation and initial thermal screening. Mixtures 5 and 6 were selected because they exhibited the best combination of good workability, low heat evolution during working time, adequate strength, volume stability, and components with enhanced probability of long-term stability. Mixture 3 was similar but had somewhat higher early strength, and more heat rise in thermal tests was expected. Mixture 8 had the highest strength, but its preliminary thermal screening was expected to reveal heat rise unacceptable for mass concretes. It was included in initial thermal screening as an outlier, to show quantitatively that further consideration of concretes with Class C fly ash was not warranted.

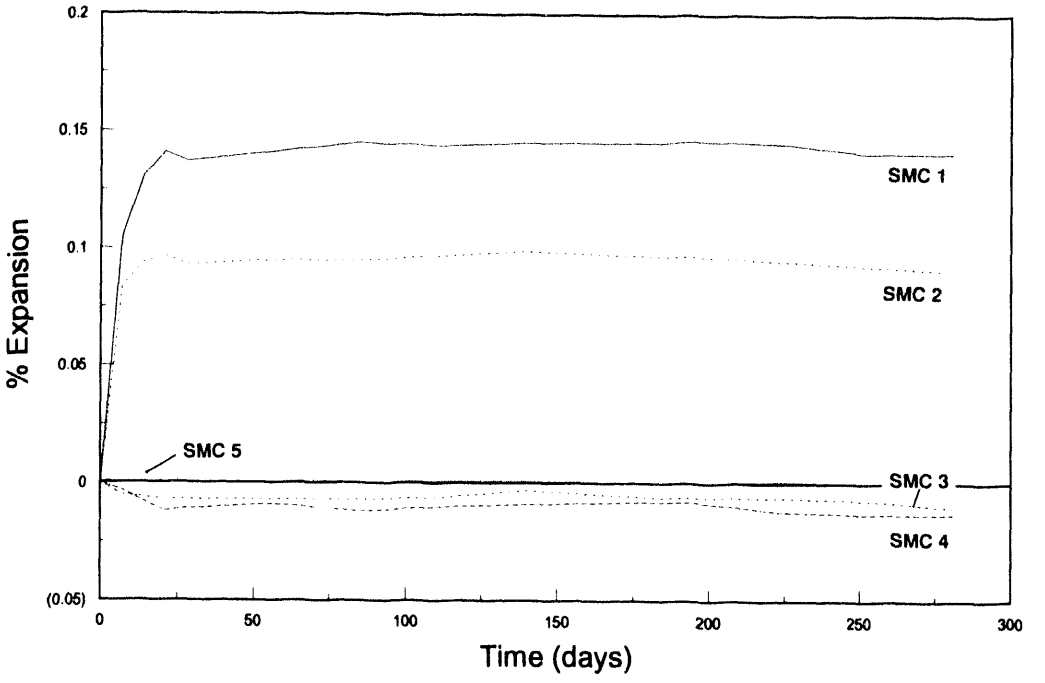

Figure 2-1. Length change of candidate mixtures 1 through 5.

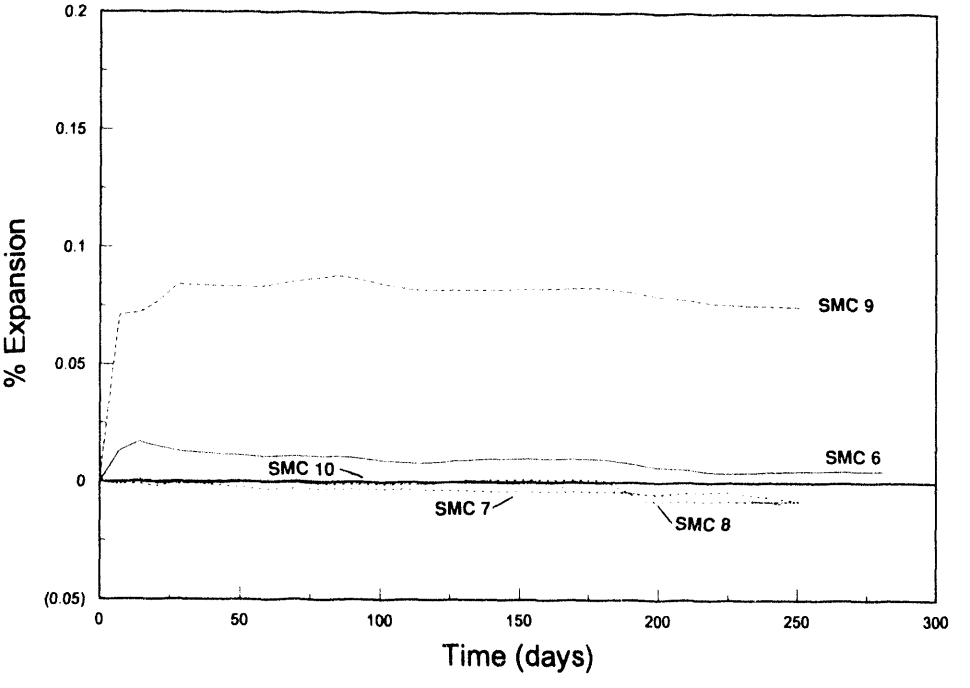

Figure 2-2. Length change of candidate mixtures 6 through 10.

Initial thermal screening was performed using a patented commercial device called 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 Haybox[1] 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. This test was used to screen the four candidate mixtures to select two that would be reproportioned using 3/4-in. coarse aggregate and undergo additional testing. The target value for maximum mixture temperature was 40°F above the initial mixture temperature of approximately 55°F. Figure 2-3 shows the adiabatic temperature rise calculated by the Haybox system for these four mixtures. As anticipated, the temperature rise for mixture 3 was marginal, at 45°F above the starting temperature, and the temperature rise for mixture 8 was deemed unacceptably high at 50°F above the starting temperature.

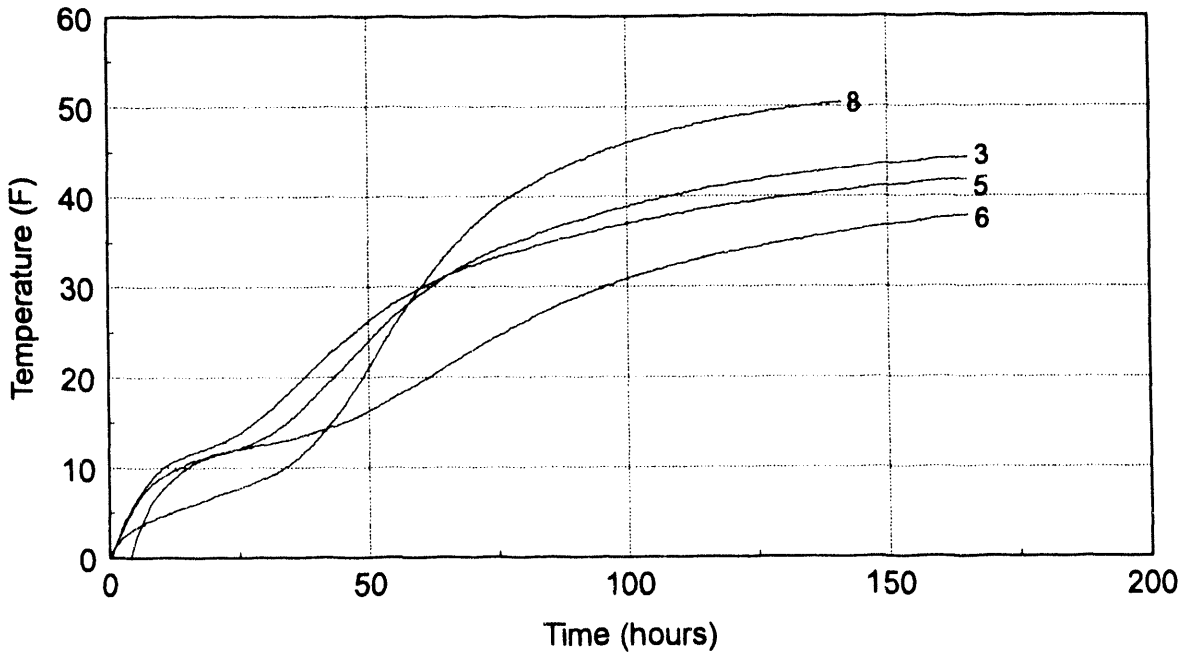

Figure 2-3. Calculated adiabatic temperature rise for SMC mixtures 3, 5, 6, and 8.

1 "Computer Interactive Maturity System (CIMS)" by Digital Site Systems, Inc.

2.7 Selection of Concrete for Mechanical and Thermal Tests

At this time in concrete development, the target value for strength was still the 4,500 psi defined initially. A 4-hr working time and a temperature rise of 40°F during the first 3 to 4 days was considered the deciding factor in selecting a mass concrete. From the results of the preliminary thermal tests, mixtures 5 and 6 were selected for reproportioning and further testing.

Mixtures 5 and 6 were reproportioned using 3/4-in. coarse aggregate, which is considered more appropriate for mass concrete. Proportions are given in Table 2-9 as 5R and 6R. These two mixtures were batched, mixed, and tested again to verify that target properties were achieved with the new proportions. Both mixtures met project requirements for fresh concrete properties and volume stability to 60 days. Figure 2-4 shows their adiabatic thermal signatures calculated from Haybox tests. Calculated temperature increases for the reproportioned mixtures 5R and 6R are appreciably less than those of their predecessors.

Table 2-9. Mixture Proportions for SMC 5R and SMC 6R

Mixture Compound	SMC 5R lb/yd^3*	SMC 6R lb/yd^3
Class H cement	221	190
Class F fly ash	247	264
Fine aggregate	1,283	1,294
Coarse aggregates (3/4" max)	1,645	1,658
Salt	86	84
Chem Comp III	112	110
Na citrate	4.7	3.5
Water	250	246
D-Air	5.6	5.6

*To obtain kg/m^3 divide lb/yd^3 by 1.69.

Based on test results available at 28 days, mixture 6R was selected for full-scale testing of thermal and mechanical properties. Compressive strength of cylinders cast from trial batches

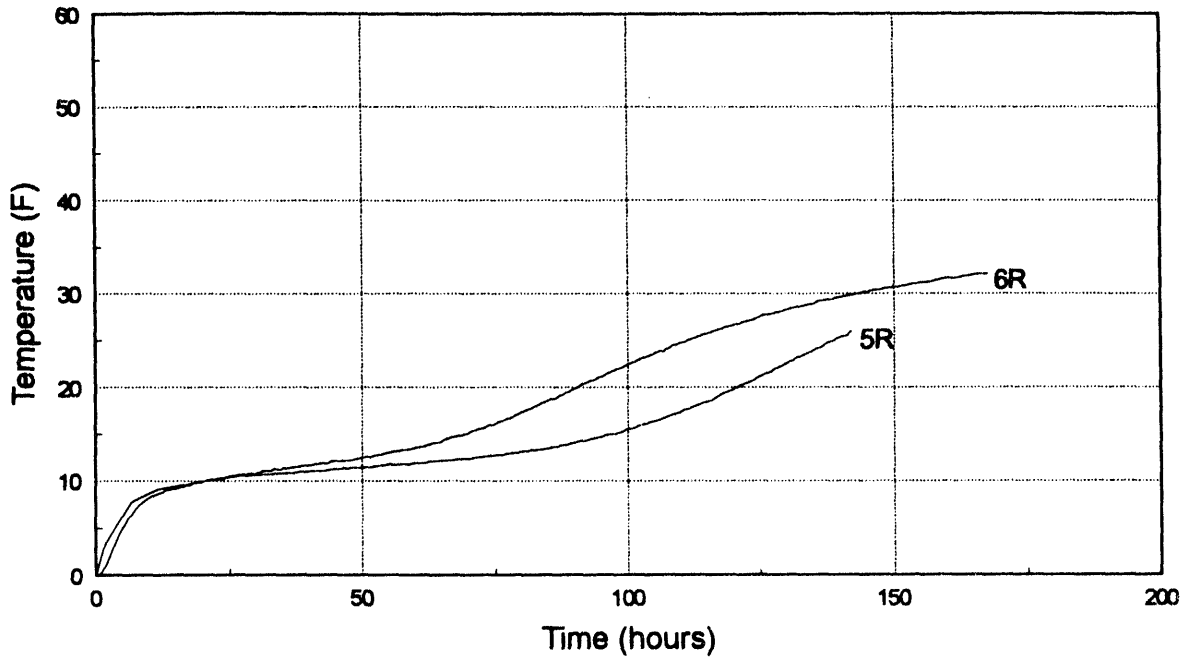

Figure 2-4. Calculated adiabatic temperature rise data for mixtures 5R and 6R.

of mixture 6R was about 1,800 psi at 28 days and 3,700 psi at 90 days. The rate of strength gain approximated from these data indicated that strength would achieve the target value of 4,500 psi by 180 days, as had mixture 6 before reproportioning.

Shrinkage of mixture 6R also was acceptable at 28 days for trial batches. Assumed design requirements at that time were that the low-heat requirement should be emphasized over higher strength in mixture proportioning. Based on preliminary values for heat, strength, and shrinkage, mixture 6R was selected for extensive testing of mechanical and thermal properties.

Tests at later ages and on other batches showed that mixture 6R experienced additional shrinkage after 28 days and exceeded the target limit of 0.02% shrinkage by 90 days. Some subsequent batches also failed to reach the target compressive strength. The variability introduced by differences in chemical composition of Chem Comp III and the possibility of the strength gain being excessively retarded by citrate were not understood at that time. The effects of these and other variables on the properties of SMC-related concretes will be explored in future work.

3.0 MECHANICAL AND THERMAL PROPERTIES

3.1 Considerations of Mass Concrete

The use of concrete as part of a multiple barrier system requires that the concrete monoliths remain as free of cracks as is reasonably achievable. The proposed size of panel seals, approximately 120 yd^3, necessitates careful consideration of thermal stresses. Exothermic reactions of the hydrating cement generate heat and cause thermal expansion of concrete soon after placement. Following this early exothermic period, the concrete cools and contracts while the matrix still has fairly low strength. In a large monolith, this volume change can exceed the strain capacity of the concrete and cause cracking. Concretes that generate less heat are at less risk of cracking.

Placement of concrete underground and almost completely enclosed by rock from early age will mitigate differential thermal effects. If these were free-standing monoliths with large surface areas exposed to different thermal conditions, they would be more likely to experience serious thermal stresses. However, all portions of a panel seal or other large placement in the Salado Formation will be at a constant service temperature and eventually will experience circumferential compressive forces due to creep of the enclosing salt rock (Van Sambeek, 1987).

The WES and other offices of the Corps of Engineers have had extensive experience proportioning and characterizing mass concrete for placement in large structures. Previous research and field experience with mass concretes have indicated that the critical material properties affecting cracking in mass concrete are strength, elastic modulus, creep, shrinkage, heat production, and thermal properties (US Army Corps of Engineers, 1989; Hammons et al., 1989; Garner et al., 1991; Smith, 1991; Bombich et al., 1991). Some of these material properties may be used for structural calculations of the seal system.

Although the environment of placement and service for panel seals is markedly different from that of conventional mass concrete structures, the same material properties of concrete will be important. Little of the collective experience of measuring thermal and mechanical properties or performing thermal-stress analysis has involved salt-saturated mass concrete. It was necessary to determine whether the SMC material itself behaved similarly to other mass concretes so that its behavior could be predicted reliably when it was placed. To this end, the WES characterized mixture 6R for its thermal and mechanical properties over a wide but common range of test ages from 7 to 180 days after casting.

3.2 Mechanical Properties

3.2.1 Multiple Batches of Mixture 6R

The data reported here include measurements of compressive strength for 7 different batches of mixture 6R cast on 5 dates between April and September 1992. Multiple batches were prepared to explore effects on workability and physical properties of variables such as batch size, continuous vs. intermittent mixing, and variations in component materials. For this discussion, mixing dates are identified by 6 digits representing month, day, and year. All materials were batched within 24 hr before mixing. Table 3-10 lists the mixing dates and differences among batches, which are discussed in the following paragraphs.

Table 3-10. Differences Among Batches of SMC 6R

Mixing Date	Variables or Unique Conditions
040692	First large batch of 6R, 9 cu ft mixed continuously. Unacceptable slump loss at 2 hr, followed by rapid strength gain.
043092	Two batches of 1.7 and 9 cu ft, continuous mixing. Same Chem Comp as 040692, but improved slump with smaller batches.
060592	Two batches of 1.7 and 9 cu ft, intermittent mixing, increased dose of citrate. Less early heat, less slump loss, slower strength gain than previous batches.
081392	New batch of Chem Comp, same citrate dose as 060592. Setting was unacceptably retarded. Very slow strength gain.
092192	Same Chem Comp as 081392, reduced citrate dose. Strength low at 7 days but increased acceptably.

The 040692 and 081392 batches represent the extremes of workability and rate of strength gain. The 040692 batch gave the shortest working time and most rapid strength gain. The 081392 batch had an extended working time, but gained strength so slowly that samples could not be demolded until 5 days after casting. Differences between these batches resulted principally from use of Chem Comp III from two different shipments. The change from continuous to intermittent mixing also contributed to differences in time of setting and strength gain.

The amount of sodium citrate needed to achieve workability for more than 3 hr had been determined for materials on hand before the 040692 batching. With continuous mixing and a large batch size, the citrate dose proved to be inadequate to extend the working time beyond 2 hr with the Chem Comp used. This dose was adequate with intermittent mixing, which imparts less shear to the mixture and causes less increase in mixture temperature. Intermittent mixing was used for all batches after 043092.

Beginning with the activities of 081392, concrete was batched using a new shipment of Chem Comp. The material in this shipment was atypical. It contained less of the expansive calcium sulfoaluminate phase and more gypsum than did five other lots of Chem Comp that have been used in concretes by the WES. Using the amount of sodium citrate determined to be adequate for other lots of Chem Comp when the concrete was mixed intermittently caused excessive retardation of setting and a decrease in strength for the 081392 concrete. Concrete was prepared on 092192 using less citrate. With this set-retarding component decreased from 3.5 to 2.6 lb/yd^3, time of setting and strength gain returned to the more typical range (Figure 3-5).

3.2.2 Compressive Strength and Modulus of Elasticity

Tables 3-11 and 3-12 list the individual and average values measured for unconfined compressive strength of this series of concretes at various ages. Figure 3-5 shows strength for concretes prepared on these five dates as a function of time.

Two batching dates, 043092 and 060592, represent two batches each of 1.7 and 9 cu ft. Strength values from the larger batch were slightly lower in both cases, but these differences were not significant for such small populations of data. Values for compressive strength from the two batches on each of these dates are averaged together in Table 3-12. The strength value for 092192 concrete at 90 days was not measured. It was estimated to conform to the group of strength-gain curves shown in Figure 3-5 as a reasonable intermediate value between the results of tests performed at 28 and 205 days after samples were cast.

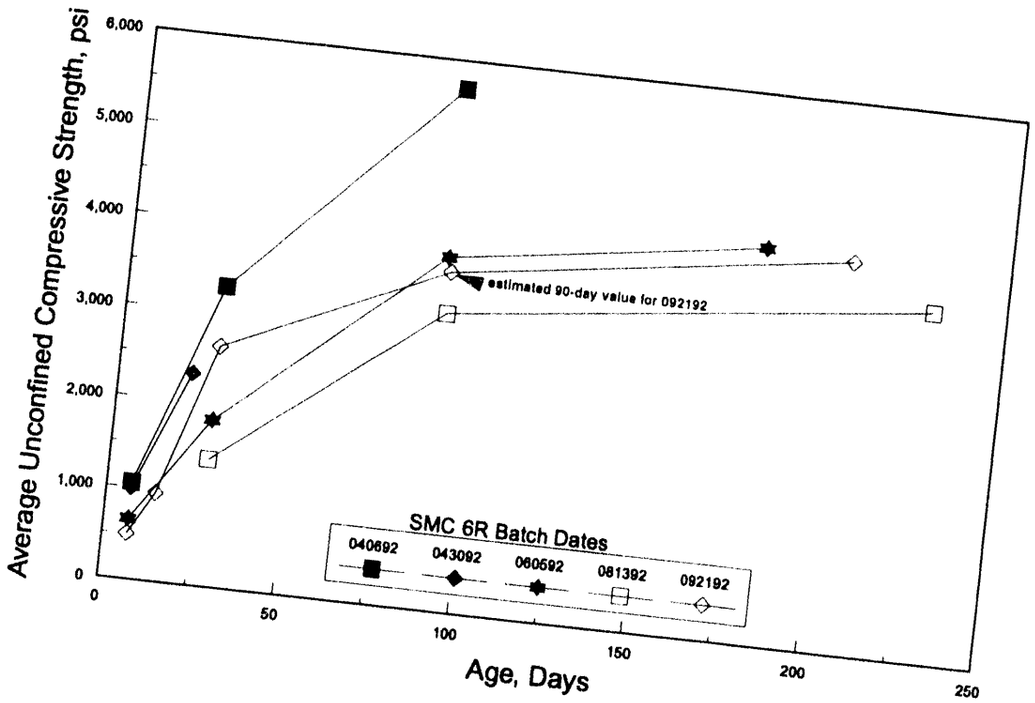

Figure 3-5. Unconfined compressive strength versus age for 5 batches of SMC 6R.

Table 3-11. Unconfined Compressive Strength and Modulus of Elasticity for Mixture 6R Mixed 040692 and 081392.

	040692			
	Unconfined Compressive Strength, psi[1]		Modulus of Elasticity, psi x 10^6	
Age, days	Individual	Average	Individual	Average
7	1063	1060	2.3	2.6
	1050			
28	3260	3280	4.2	4.2
	3300		4.2	
90	5810	5690	6.0	5.6
	5570		5.2	

	081392			
	Unconfined Compressive Strength, psi		Modulus of Elasticity, psi x 10^6	
Age, days	Individual	Average	Individual	Average
	1350		2.2	
28	1430	1390	2.3	2.4
	1380		2.8	
	3220		3.9	
90	3260	3250	3.9	3.9
	3260		4.0	
	3820		4.7	
230	3680	3830	3.9	4.3
	3980		4.4	

[1] To convert compressive strength values to MPa, multiply psi by 0.00689.

Table 3-12. Unconfined Comprehensive Strength of Other Batches of Mixture 6R

	Batched 043092[1]		Batched 060592[1]			Batched 092192	
	Unconfined Compressive Strength, psi[2]					Unconfined Compressive Strength, psi[2]	
Age, days	Individual	Average	Individual	Average	Age, days	Individual	Average
7	1060 1000	1030	670 680 660 640	660	7	510 490	500
21	2530 2430 2150 2120	2310			14	980 950	970
28			1930 1830 1720 1790	1820	28	2640 2610	2630
90			3790 3920	3860	90		
180			4480 4810 4120 3920	4330	205	4340 4240 4270	4280

[1] Two batches of 1.5 and 9.0 cu ft prepared simultaneously. Individual values from both batches are reported and averaged.
[2] To convert compressive strength values to MPa, multiply psi by 0.00689.

Values for modulus of elasticity were calculated for samples from the 040692 and 081392 batches, representing the high and low values for strength for this series of concretes (Table 3-11). From these batches, the static modulus of elasticity was calculated for each specimen tested in compression, in accordance with ASTM C 469. Strain gauges were applied to the cylinders, and the longitudinal strain was monitored by a computer-driven data acquisition system until the specimen failed. Strain corresponding to 40% of the ultimate stress and the stress corresponding to a strain of 50 millionths were determined from the data. A modulus was then calculated from the data according to the following equation:

$$E = (S_2 - S_1)/(\epsilon_2 - 0.000050) \tag{1}$$

where:

E = modulus of elasticity, psi
S_1 = stress corresponding to a longitudinal strain of 50 millionths, psi
S_2 = stress corresponding to 40% of ultimate load, and
ϵ_2 = longitudinal strain produced by stress S_2.

Individual stress vs. strain curves (2 or 3 per age) are shown in Figure 3-6 for the highest strength batch. Figure 3-7 illustrates the stress vs. strain curves for the lowest strength batch. There is some noise in the data for two of the stress-strain curves in Figure 3-7. Overall, the trend of the moduli of the concrete is to increase with age. The modulus of the stronger mix increased from 2.6 (10^6) psi at 7 days to 5.6 (10^6) psi at 90 days. The modulus of the weakest mix ranged from 2.4 to 4.3 (10^6) psi over equal time periods. Increase in secant modulus with age and strength is typical of all concrete.

3.2.3 Creep Tests in Compression

Creep is time-dependent deformation caused by sustained load. Creep in concrete is differentiated from shrinkage strains and elastic strains. Upon initial application of load, the material response is primarily elastic but may include a nonelastic component. The nominal elastic strain is governed by the elastic modulus at the time of loading. Shrinkage of the creep specimen is measured by monitoring the deformation of identically prepared unloaded specimens. Thus, the creep strains are calculated from the total measured strains as follows:

$$\epsilon_{creep} = \epsilon_{total} - \epsilon_{elastic} - \epsilon_{shrinkage}. \tag{2}$$

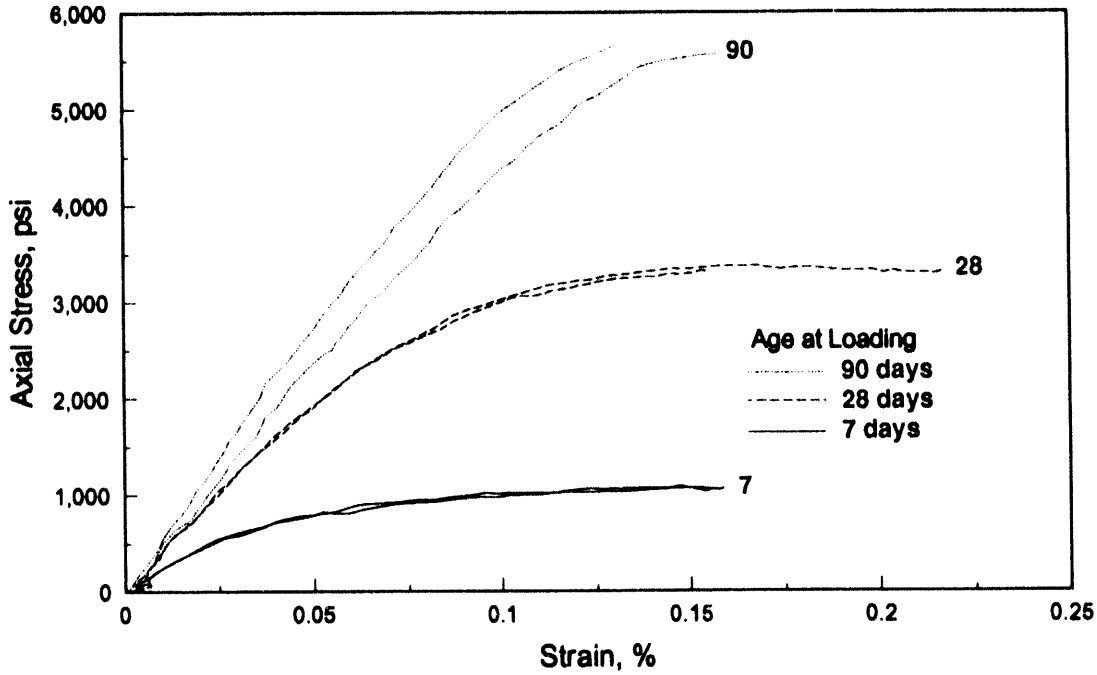

Figure 3-6. Stress-strain response of mixture 6R batched 040692.

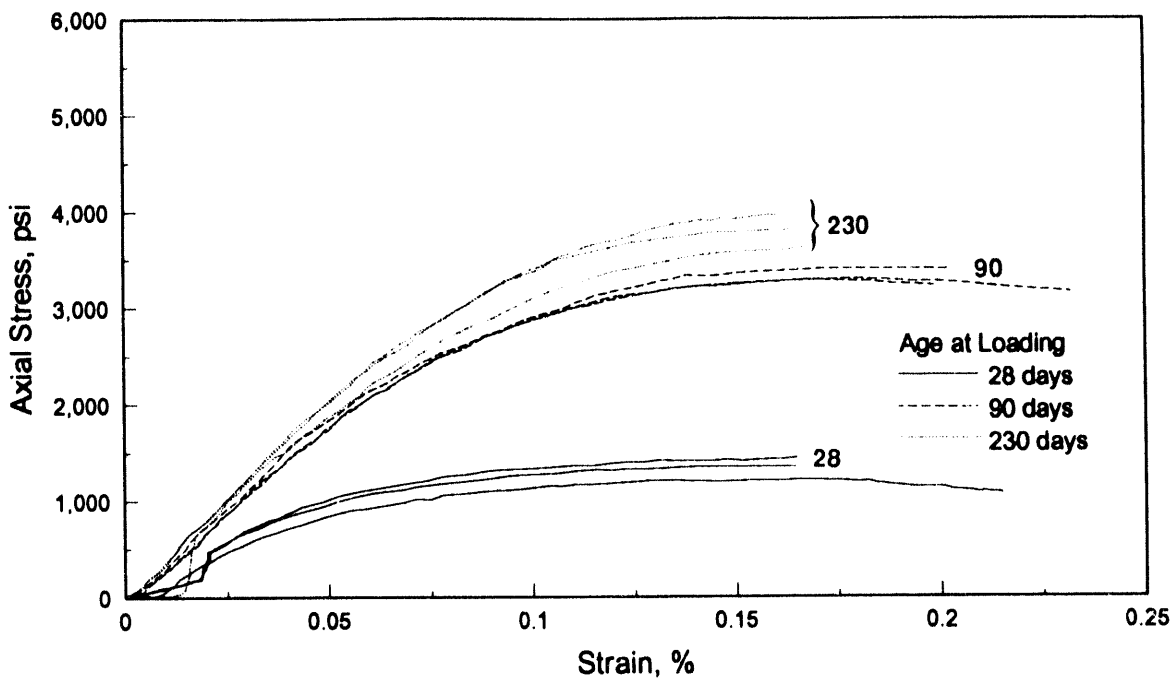

Figure 3-7. Stress-strain response of mixture 6R batched 081392.

Using these concepts, creep tests were conducted at various ages according to ASTM C 512 with all measurements recorded by continuous computer-controlled data acquisition. The specimens tested were 6 by 16 in. The creep specimens were cast in steel forms with the longitudinal axis horizontal. These forms accommodated Carlson strain gauges embedded in the center of the specimens oriented along the longitudinal axis of the cylinder. Steel bearing plates were attached to the ends of each specimen by embedded mechanical anchors. These plates provided a smooth plane surface for applying the compressive force. A bituminous moisture barrier was applied to the surface of the creep specimen immediately after the forms were removed to prevent moisture from entering or leaving the specimen.

The apparatus used to perform the creep tests was a hydraulic loading frame designed to maintain a constant stress by means of a gas pressure regulator in series with a gas/oil accumulator and hydraulic ram. The desired applied stress was set by the gas pressure regulator. The test device accommodated two specimens loaded in series held at constant temperature. Two control cylinders were also monitored to determine the strains not associated with the applied loads. The creep specimens were loaded to 40% of the unconfined compressive strength at the age of loading as determined from unconfined compressive tests on companion cylinders. The following measurements were recorded using a digital data acquisition unit:

- Applied stress, by a pressure transducers located in the gas pressure regulator output line.
- Strain and temperature in the loaded specimen, by Carlson strain gauges embedded in the center of the specimen.
- Strain and temperature in the control specimen, by Carlson strain gauges embedded in the center of the specimen.
- Time, by an internal clock in the computer data acquisition unit.

The strains recorded by the shrinkage-compensation cylinders were subtracted from the measured strains. The elastic strains at time of loading also were subtracted to obtain creep strains. These corrected strains were then divided by the applied stress to obtain specific creep strain. Data for concrete from 040692 loaded at 28 and 90 days of age are shown in Figure 3-8. Figure 3-9 shows creep strain for 081392 concrete loaded at 28, 90, and 230 days of age. Values for specific creep often are used in time-dependent material models. Specific creep is calculated by dividing creep strain by total stress. Stresses for each creep strain curve also are shown in Figures 3-8 and 3-9. Creep values for mixture 6R are similar to published values for concrete loaded at later ages (Hammons et al., 1991; Smith, 1991).

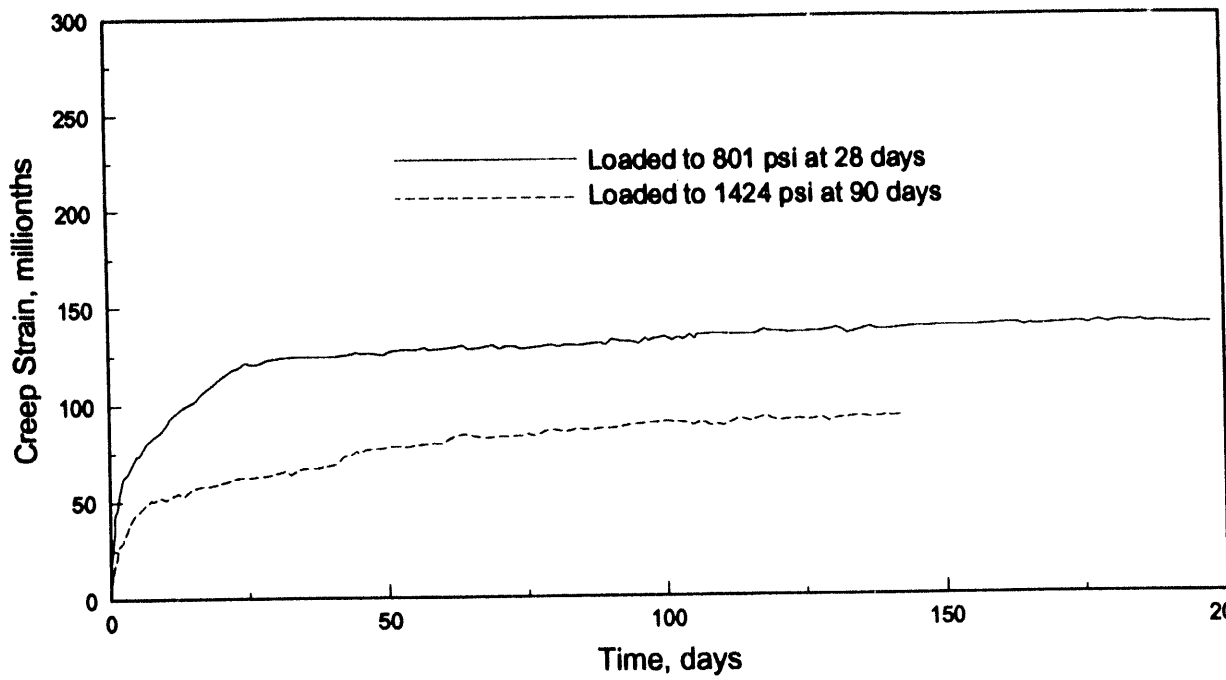

Figure 3-8. Creep strain data for the highest strength SMC 6R loaded at 28 and 90 days after casting.

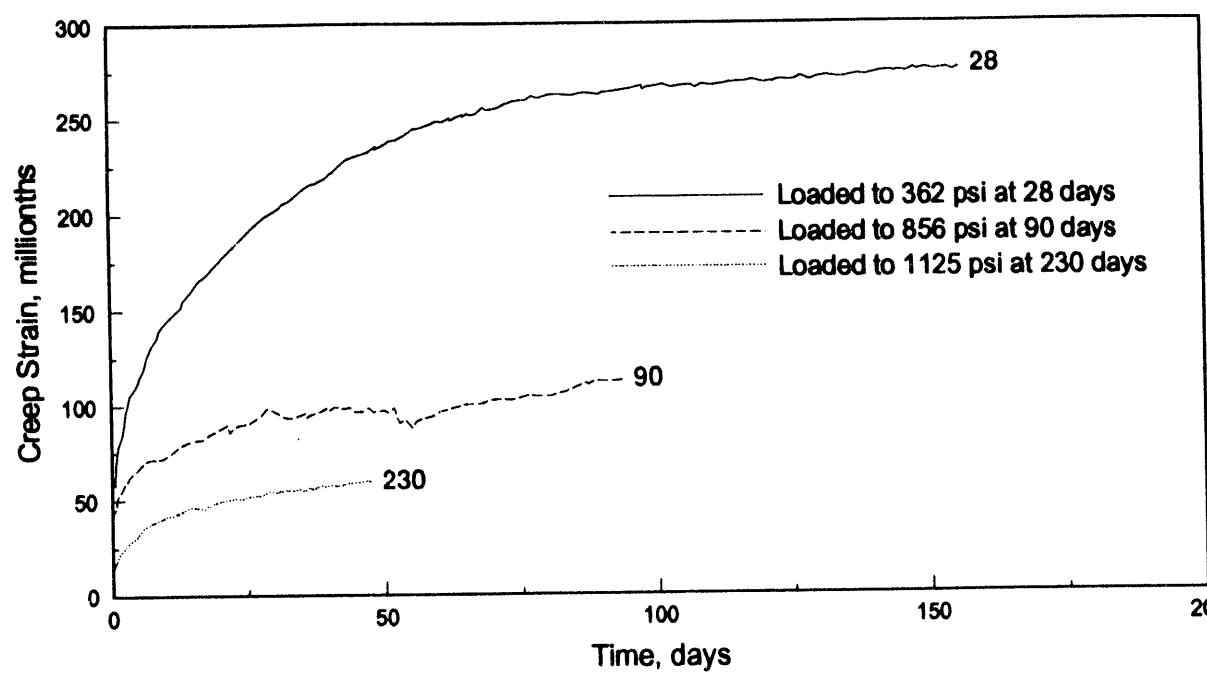

Figure 3-9. Creep strain data for the lowest strength SMC 6R loaded at 28, 90, and 230 days after casting.

SMC salt-saturated concrete becomes much more creep resistant with age. In the tests plotted in Figures 3.8 and 3.9, the specific creep decreased by a factor of 10 between 28 and 230 days. Despite being subjected to greater loads, the older samples deformed less than the fresher samples. When structural calculations are made to support the seal system design, creep parameters will be included for the salt-saturated concrete. Based on initial results shown here, salt-saturated concrete is expected to creep much less than the surrounding salt; however, a small amount of concrete creep deformation will greatly influence the magnitude of stress and the stress history. A more complete constitutive relationship for salt-saturated concrete is desirable and will be pursued in future work.

3.3 Thermal Properties

3.3.1 Adiabatic Temperature Rise

The temperature rise of concrete in an adiabatic condition is primarily due to hydration or pozzolanic reaction of the cementitious materials. The magnitude and shape of the curve generated by plotting adiabatic temperature rise versus time is an important measure of the heat-generating potential of a concrete mixture. In addition to the adiabatic temperature rise values calculated from Haybox data (Figure 2-4), one test of true adiabatic temperature rise of Mixture 6R was conducted as described in CRD-C 38 (US Army Corps of Engineers, 1949). In this test, temperature rise is measured for a specimen of concrete for which no loss of heat to the surroundings is permitted. The specimen was a cylinder of 30-in. diameter by 29-in. height cast from a single batch of concrete (092192). The placement temperature for the concrete at the start of the test was 55°F and the test was conducted for 28 days.

The adiabatic temperature rise of mixture 6R is shown in Figure 3-10. Its heat generation as a function of time (rate of heat generation) is plotted in Figure 3-11. Figure 3-10 also compares data from mixture 6R to data from Figure 2-3 and to temperature rise of two conventional mass concretes tested by Hammons et al. (1991). The total temperature rise of this mixture 6R was approximately 10°F higher than that of the comparison mass concretes during the first month after casting. The higher total heat rise of this mixture can be explained by at least two factors. The nominal maximum size of coarse aggregate used in mixture 6R is smaller than is common in mass concretes used in construction of waterways structures, such as those described by Hammons et al. (1991), in which 3-in. aggregates were used. The smaller aggregate size necessarily increased the content of cementitious materials (cement + fly ash +

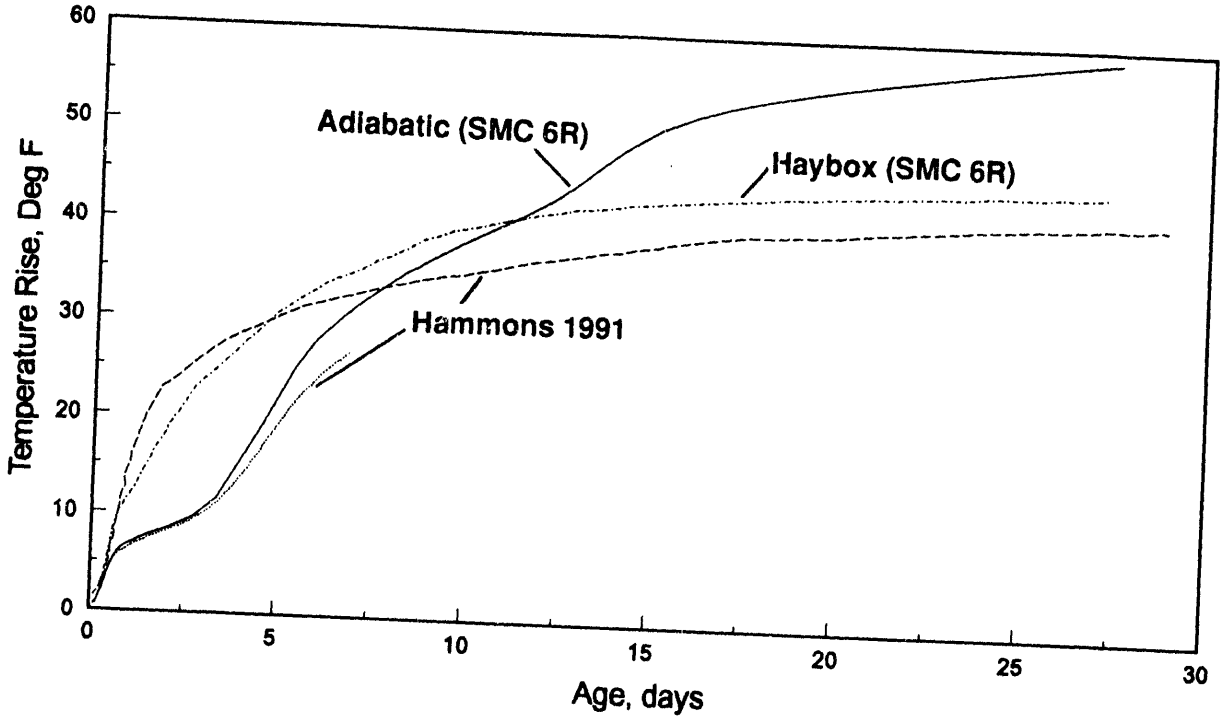

Figure 3-10. Adiabatic temperature rise of SMC 6R (092192) compared to values for other mass concretes.

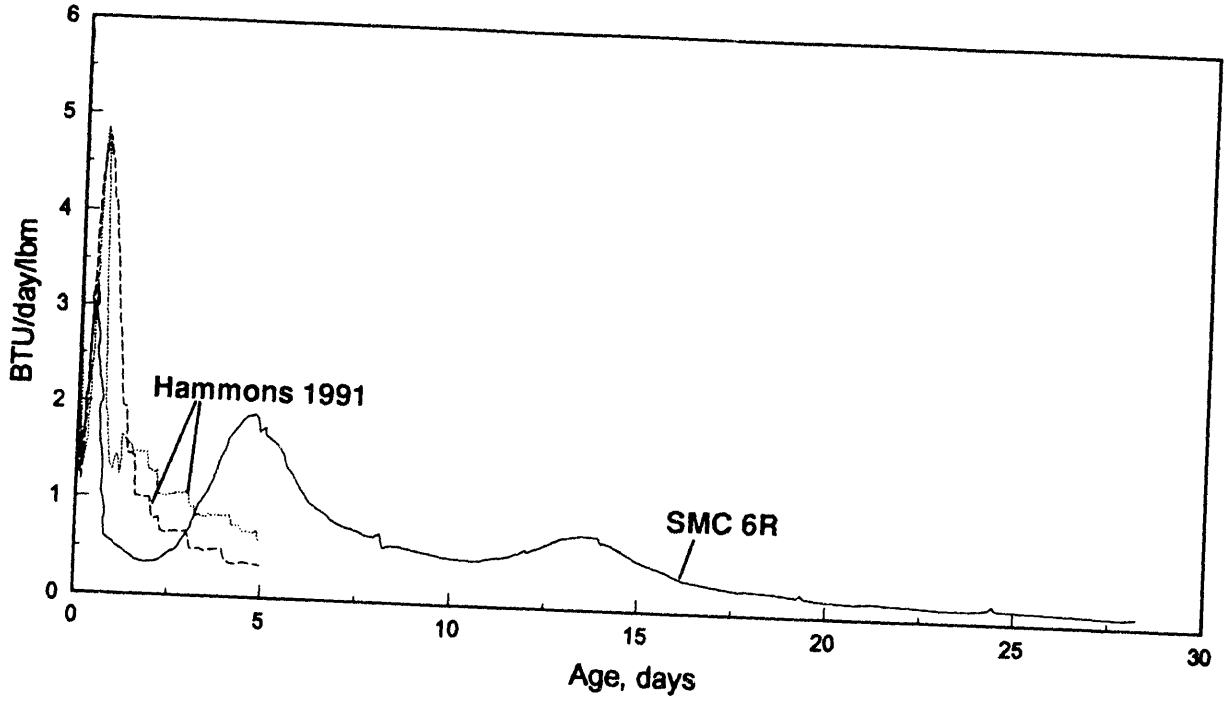

Figure 3-11. Heat generation plot for mixture 6R (092192).

Chem Comp III) in mixture 6R relative to the comparison concretes. For the two comparison mass concretes indicated in Figure 3-10, the total amount of cementitious material is about 300 lb/yd^3, or about half the content of cementitious materials in mixture 6R (see Table 2-6).

The second factor is the presence of Chem Comp III. This component evolves more heat per unit volume than do the cements and fly ashes that are used commonly in mass concretes. No references were found describing use of Chem Comp III in mass concrete, so there were no data for comparison of the heat generation of mixture 6R to data from other mass concretes with this component.

Figure 3-11 shows the rate of heat generation for the comparison mass concretes (Hammons et al., 1991). The actual rate of heat generation in BTU/day per lb-mass of cementitious materials was slower for mixture 6R. This slower rate of heat evolution is likely to create smaller thermal stress gradients, despite the higher total heat output. As seen in Figure 3-11, the comparison concretes evolved most of their heat during the first day after casting. The SMC reached a second heat-output peak 5 days after casting and a third peak at 14 days. This suggests delayed reaction of fly ash or the expansive component, attributable to the presence of salt and sodium citrate.

3.3.2 Other Thermal Properties

The linear coefficient of thermal expansion, thermal diffusivity, specific heat, and thermal conductivity were determined for laboratory-cast specimens of mixture 6R (092192) at ages near 20, 28, and 90 days. The time required for these tests plus the need for testing replicate samples resulted in test ages spanning several days and usually bracketing the scheduled test date. The results of these tests are reported and discussed below. To avoid leaching salt from the concrete samples, we substituted NaCl-saturated brine for water in the test baths.

3.3.3 Thermal Expansion

The coefficient of linear thermal expansion was determined in accordance with CRD-C 39 (US Army Corps of Engineers, 1949). Two 6-by-16-in. cylindrical specimens were cycled between water baths maintained at 40 and 100°F. The strains and temperatures in the specimens were measured with embedded Carlson strain meters. These meters were located at the centroid of the specimen and oriented along the longitudinal axis of the cylinders. The temperatures of

the water baths were measured with thermocouples placed in the brine. All data were monitored and recorded with a digital data-acquisition system and microcomputer. Results of tests at each age are given in Table 3-13. Values for thermal expansion, approximately 6.5 (10^{-6}/°F), reported for the SMC 6R mixture are higher than values for conventional mass concretes reported by Hammons et al. (1991), which averaged 3.85 (10^{-6}/°F). Much of this difference can be attributed to the difference in coefficient of thermal expansion of the aggregates used. The aggregate in mixture 6R was chert, with a coefficient of thermal expansion between 6.0 and 7.0 (10^{-6}/°F). The comparison concretes had limestone aggregates, with a comparable coefficient of 3.3 (10^{-6}/°F). This result indicates that aggregate type exerts a strong influence on the expansion coefficient of the composite material. It should be noted that the values of thermal expansion for chert-aggregate salt-saturated concrete are identical to those reported for ordinary concrete of similar aggregates (Neville, 1963).

3.3.4 Thermal Diffusivity

The thermal diffusivity is equal to the thermal conductivity divided by the product of heat capacity per unit volume and density. It may be used as an index of the ease with which the concrete will undergo temperature change. It is obtained by submerging a specimen in a water bath at a known temperature and measuring the time it takes for the specimen temperature to equilibrate to the temperature of the bath and alternating between hot and cold water baths.

Table 3-13. Summary of Thermal Properties of Mixture 6R

Property[1]	Nominal Age of Concrete		
	20 Days	28 Days	90 Days
Thermal Diffusivity ft^2/h (10^{-6} m^2/s)	0.0379 (0.978)	0.0390 (101)	0.0360 (0.923)
Specific heat BTU/lb-°F (J/kg-K)	0.237 (992)	0.232 (971)	0.209 (874)
Thermal Conductivity[2] BTU/(ft-h-°F) (W/m-K)	1.268 (2.195)	1.240 (2.145)	1.045 (1.809)
Coefficient of thermal expansion 10^{-6}/°F(10^{-6}/°C)	6.50 (11.7)	6.59 (11.9)	6.59 (11.9)

[1] Each value represents an average of 2 or 3 tests per age.
[2] Derived based on measured thermal diffusivity and heat capacity for an average unit weight of 139 lb/ft^3.

The thermal diffusivity of mixture 6R at 20, 28, and 90 days was determined in accordance with CRD-C 36 for cylindrical specimens 8 in. in diameter by 16 in. in length. The temperatures of the hot and cold water baths were maintained at 40°F and 140°F, respectively, rather than maintaining the hot bath at 212°F and the cold bath at essentially the temperature of the laboratory tap water, as called for in the standard method. This range of temperatures was chosen because it is the likely range of temperatures expected in the mass concrete. In this way, the test results for mixture 6R can be compared to data available for other mass concretes.

Temperatures of water baths and individual cylindrical specimens were monitored with thermocouples. All data were monitored and recorded using a digital data-acquisition system and microcomputer. Values for thermal diffusivity of mixture 6R were nearly identical to those reported for other mass concretes (Hammons et al., 1991), which ranged from 0.032 to 0.036 ft^2/h. The results of the tests are given in Table 3-13.

3.3.5 Specific Heat

The specific heat of mixture 6R was determined at ages of approximately 20, 28, and 90 days in accordance with CRD-C 124 (US Army Corps of Engineers, 1949). These tests were conducted on samples obtained from 4-by-8-in. cylinders. The temperatures of hot and cold brine baths were maintained at 100°F and 40°F, respectively, rather than at 125°F and 35°F as prescribed in CRD-C 124. All temperatures were measured with a precision thermistor thermometer. The average value of specific heat measured at each age is given in Table 3-13. Values are very close to those reported for other mass concretes, 0.22 BTU/lb-°F or 920 J/kg-K (Hammons et al., 1991). Values for mixture 6R ranged from 962 J/kg-K at 7 days to 874 J/kg-K at 90 days, somewhat higher than the 860 J/kg-K reported for WIPP rock salt by Kreig (1984).

3.3.6 Thermal Conductivity

The thermal conductivity of the concrete was calculated in accordance with CRD-C 44 (US Army Corps of Engineers, 1949). This method requires the results of the thermal diffusivity and specific heat tests as reported above. Also required is the actual unit weight of the concrete, calculated by comparing the mass of the concrete specimens in air and water. Values for thermal conductivity of mixture 6R at 20, 28, and 90 days range from 1.8 to 2.2 W/m-K, as shown in Table 3-13. This range of values is similar to those reported by

Hammons et al. (1991), approximately 1.9 W/m-K. It also is lower than the value used as input to a heat-transfer model by Garner et al. (1991), which was 3.98 W/m-K. The values for thermal conductivity of mixture 6R are within the range of values used by Van Sambeek (1987) to define boundary conditions for thermomechanical analyses of WIPP shaft seals.

4.0 DISCUSSION AND CONCLUSIONS

4.1 Mixture Proportioning, Batching, and Casting

Proportioning SMC was an exercise in balancing conflicting performance requirements. For mass concrete, low early heat and low total heat are desirable. Mixtures that evolve heat slowly also gain strength slowly. If 4,500 psi at 180 days is sufficient for design needs, reducing early strength and slowing heat-generated thermal stress does not adversely affect other properties. If higher strength or more rapid strength gain becomes essential as design requirements evolve, the balance between heat and strength should be reconsidered.

For chemical durability and good bonding to bedded salt, the concrete used in WIPP seals needs to be salt saturated. Many familiar chemical admixtures are chemically incompatible with salt-saturated concretes. This fact decreases the range of materials available to control such characteristics as long working time and low water content for a superior microstructure. Because low permeability is a desirable attribute for the concrete component seals, it may become necessary to decrease the water content of the concrete. The balance between salt-saturation, water content, and use of admixtures is part of ongoing research.

Values measured for strength or expansion in the laboratory do not indicate directly the field performance of concrete in a large placement. Specimens on which these properties are measured are small and have a high surface-to-volume ratio. When stored at simulated repository conditions, these small specimens are much more susceptible to moisture and heat loss from exposed surfaces than would be the bulk of the concrete in a seal. The relative humidity in the storage chamber used for these experiments varied from about 50 to 70% during the months of tests. Humidity below 80% is known to result in lower strength and expansion. In a mass placement such as a panel seal, the near end of the seal is virtually the only surface that will be exposed to low humidity and potential moisture loss, and the bulk of the concrete may achieve somewhat higher strength and expansion than were measured for laboratory specimens.

One very positive outcome of this study is that it revealed much about how each component of SMC affected its overall performance and suite of properties. This will simplify future efforts at reproportioning these materials to meet performance requirements.

The characteristics of the particular Chem Comp and the amount of sodium citrate used in the concrete are critical to the working time. The 040692 mixture experienced rapid slump

loss within 2 hr or less after mixing. Because the amount of citrate per cubic yard of concrete is very small (between 2 and 4 lb/yd^3 or 1.1 to 2.2 kg/m^3), it may be difficult to control the working time with citrate under field conditions. This study suggests that slump loss occurs almost instantly after a certain working time when citrate is used as a set retarder. An overdose of sodium citrate increased the working time, but for concrete prepared with one batch of Chem Comp III, this resulted in an extremely long setting time and decreased ultimate strength.

If placing procedures are established so that the required working time is reduced to 2 hr or less, it should be possible to attain adequate concrete workability without using citrate and improve the predictability of the mixture. Mixing underground using pre-batched dry materials is one way to shorten the time required for the concrete to remain workable. Omitting the citrate also would simplify the mixture, which is always desirable: having fewer components decreases the opportunity for batching errors.

Because all mixtures in this study were prepared at laboratory temperature, the effects of higher or lower temperatures on the working time are unknown. It is reasonable to assume that if the concrete is batched and mixed with the materials at elevated temperature, as would be the case if they were stored underground at the WIPP site (approximately 80°F), working time could be seriously decreased. Larger doses of citrate probably cannot overcome the effects of elevated temperature and may have other adverse affects. Storing materials at constant temperature is advisable. This would preclude the seasonal variations in concrete workability and other properties that would be encountered when concrete seal components are placed during different seasons.

The temperature of mixture 6R consistently rose 25°F during the 3-hr mixing time from an initial mixture temperature of approximately 55°F. Low initial temperatures typically extend the working time of concrete mixtures. When the SMC is placed in the field, the initial concrete temperatures should be less than 60°F to ensure that the same fresh concrete properties are attained. Depending on the temperature of the materials, this initial temperature could be achieved by using ice and chilled water.

4.2 Mechanical and Thermal Properties

Several important mechanical and thermal properties have been measured for mixture 6R of a family of mixtures collectively called Salado Mass Concrete. The concrete mixture for the actual in situ demonstrations and testing will be finalized during fiscal year 1994. It will

comprise the same major cementitious components as mixture 6R but in different proportions. Several batches of mixture 6R attained strengths lower than the target strength at 180 days, with a maximum temperature rise of 60°F over 25 days. Work undertaken in 1993 modified the concrete to attain higher strength at the cost of increased heat. This indicates a need for analyses to assess the influence of a larger thermal pulse on a typical seal arrangement. However, the values for thermal and mechanical properties tabulated here can be used in preliminary modeling and performance assessment.

The modulus measured for the highest-strength trial batch of mixture 6R increased from $2.6(10^6$ psi) to $5.6(10^6$ psi) between 7 and 90 days after casting, corresponding to a strength increase from approximately 1,000 to almost 5,700 psi. In contrast, for the lowest-strength trial batch, formulated with a less-reactive Chem Comp III and with its setting excessively retarded, the modulus increased from 2.4 to $4.3(10^6$ psi) between 28 and 230 days after casting. The compressive strength of this batch at 230 days was only 3,830 psi, below the target value of 4,500 psi. The value chosen for preliminary modeling should be somewhere between these extremes.

Cylinders of mixture 6R tested for creep exhibited very little deformation when loaded for 40 to 200 days at a constant stress equivalent to 40% of its instantaneous strength. The various test specimens deformed between 50 and 250 microstrains overall and became more creep resistant with age. It appears that creep rate of the concrete is much lower than the creep rate of the enclosing rock salt at these stress levels. However, it may be appropriate to run additional creep tests at higher loads, approximating design loads, rather than using the conventional 40% ratio of creep stress to unconfined compressive strength. Creep loads approaching lithostatic may be appropriate, under which concrete creep may be more significant especially at early ages.

The coefficient of thermal expansion measured for duplicate specimens of mixture 6R was constant at $6.5(10^{-6})/°F$ between 20 and 90 days after casting. This is higher than corresponding values measured for other non-salt mass concretes, but much of this difference is attributable to the difference in coefficients of thermal expansion for their respective aggregates. The coefficient of thermal expansion, along with heat evolution and strength, will be important factors in thermal-stress analyses because of the need to avoid thermally caused cracking of the concrete seals in this system. These properties and their influence on large-volume placements will be assessed during in situ demonstrations and subsequent testing.

The thermal properties of diffusivity, conductivity, and specific heat were found to undergo a reduction of 10 to 15% between 28 and 90 days.

4.3 Conclusions

Three of ten concretes proportioned with the materials selected in this study attained the target properties in preliminary screening. These were mixtures 3, 5, and 6. They cover a range of values for strength and early heat evolution. Even allowing for some changes in performance requirements as seal designs are finalized, a variant of one of these mixtures should serve as an acceptable concrete for the WIPP seals at the repository horizon.

Changes in properties among the concretes can be attributed to each material and the changes in its proportioning. Conflicting performance requirements must be balanced carefully when mixtures are modified. This study has identified some of the variables that affect concrete performance, an experience that should streamline all future concrete development. Factors such as batch size, mixer type, temperature and moisture condition of materials, and variations in the expansive admixture have been explored and their effects quantified during 1993; results of the subsequent studies will be documented in future reports.

The thermal and mechanical properties measured for mixture 6R indicate that it is potentially suitable for use as a mass concrete, as are candidate mixtures 3 and 5. Properties such as thermal diffusivity, specific heat, and thermal conductivity agree very well with other reported values for salt-free mass concretes, and should be useful in any effort to model the behavior of the seal system. They also compare favorably with properties reported for the halite host rock at the proposed repository horizon.

Variations among batches of mixture 6R are largely attributed to variations in the composition of and properties resulting from different batches of Chem Comp III, and to the interaction between Chem Comp III and sodium citrate. Subsequent research at the WES has shown that the chemical and physical properties of different batches of Chem Comp III can be related directly to the effect they have on heat evolution, setting time, expansion, and strength gain of the concretes made with them. Results from both projects will be used to establish proportioning criteria to compensate for variability in materials and to yield concrete properties that are reproducible within a narrower range.

This report does not specify the Salado Mass Concrete. Rather, it is a compendium of supporting test results that will eventually lead to a salt-saturated concrete specification for sealing applications. Factors influencing mechanical and physical properties have been tested in this study. Three candidate mixtures were tentatively identified for further development. In the course of the reported research, the formulation narrowed to one particular concrete mixture (SMC 6R), which was evaluated in detail.

As the various mixtures were examined in the evolution of the reported research, consideration was given to the seal applications. Trade-offs were considered here and will certainly be a part of the mass concrete placements underground. For example, if batching, mixing, and placing time could be reduced, the requirement for a 4-hr. working time could be relaxed. In addition, if the concrete cannot achieve strength requirements, more cementitious material may be required. The additional strength gain would have to be balanced against the propensity for thermally induced fracture. Lastly, the escalation in size from these research-sized batches to mass concrete emplacement will require further tailoring of the proposed mixture. The results covered in this report are encouraging. Based on these tests, we are on the track of a specialty concrete that is salt-saturated, has a low heat of hydration and has sufficient strength to survive in the WIPP seal environment.

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[1] Test methods have been updated each quarter to the present.

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APPENDIX A: SOURCES OF CONCRETE MATERIALS

Table A-1. Chemical and Physical Description of Class C Fly Ash

Chemical Analysis	(%)	Physical Properties	
SiO_2	28.7	Fineness (45 micron),	6
Al_2O_3	16.8	% retained	
Fe_2O_3	5.5	Fineness variation,	Negligible
Total[1]	51.0		
CaO	27.9	Water Requirement,	91%
R Factor	-		
MgO	9.4	Density, Mg/m^3	2.71 Mg/m^3
SO_3	4.1	Density variation,	4%
Moisture Content	0.2		
Loss on Ignition	0.7	Autoclave Expansion,	0.05%
Available Alkalies	1.7		
Total Alkalies		Strength Activity Index	149%
Na_2O	2.24	w/cement, 7-day	
K_2O	0.20		
Total as Na_2O	2.37		

Table A-2. Sources of Materials Used in Candidate Mixtures

Material	Identification	Source
Portland Cement	API Class H Oilwell (HSR)	Lone Star Industries, Inc. P.O. Box 1639 Sweetwater, TX 79556 (915) 288-4221
Fly Ash	Class F Centralia	Pozzolanic Ross Island Sand and Gravel Dry Mix Division 4315 S.E. McLoughlin Blvd. Portland, OR 97202
Shrinkage Compensating Cement	Chem Comp III	CTS Cement Manufacturing Co. 2337 Roscomare, Suite 10 Los Angeles, CA 90077 (310) 472-8898
Fine Aggregate	Natural Sand	Mississippi Materials Co. Grange Hall Rd. Vicksburg, MS 39180 (601) 634-8787
Coarse Aggregate	3/8-in. NMS natural gravel	Western Sand and Gravel Co. Jena, LA (318) 992-2398
Sodium Citrate	Sodium Citrate	Any Chemical Supply Company
Salt	Non-iodized table salt	Any Industrial Supply Company
Air-detraining admixture	D-Air-1	Halliburton Services P.O. Box 1308 14 Moran Rd. Natchez, MS 39120 (601) 422-5484

Table A-3. Other Materials Used in Trial Batches of SMC

Material	Identification	Source
Fly ash	Class C Centralia	Pozzolanic Ross Island Sand and Gravel Dry Mix Division 4315 S.E. McLoughlin Blvd. Portland, OR 97202
Plaster	Cal-Seal	Halliburton Services P.O. Box 1308 14 Moran Rd. Natchez, MS 39120 (601) 422-5484

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US Department of Energy (3)
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Mail Stop 4-H-3
Washington, DC 20555

Boards

Defense Nuclear Facilities Safety Board
Attn: D. Winters
625 Indiana Ave. NW, Suite 700
Washington, DC 20004

Nuclear Waste Technical Review Board (2)
Attn: Chairman
S.J.S. Parry
1100 Wilson Blvd., Suite 910
Arlington, VA 22209-2297

Advisory Committee on Nuclear Waste
Nuclear Regulatory Commission
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Santa Fe, NM 87503-0968

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Socorro, NM 87801

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