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Durability of Concrete Materials in High-Magnesium Brine

Lillian D. Wakeley, Toy S. Poole, J. Pete Burkes
Structures Laboratory
U.S. Army Corps of Engineers
Waterways Experiment Station
Vicksburg, MS 39180-6199

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ABSTRACT

Cement pastes and mortars representing 11 combinations of candidate concrete materials were cast in the laboratory and monitored for susceptibility to chemical deterioration in high-magnesium brine. Mixtures were selected to include materials included in the current leading candidate concrete for seals at the Waste Isolation Pilot Plant (WIPP). Some materials were included in the experimental matrix to answer questions that had arisen during study of the concrete used for construction of the liner of the WIPP waste-handling shaft. Mixture combinations compared Class C and Class F fly ashes, presence or absence of an expansive component, and presence or absence of salt as a mixture component.

Experimental conditions exposed the pastes and mortars to extreme conditions, those being very high levels of Mg ion and an effectively unlimited supply of brine. All pastes and mortars showed deterioration with brine exposure. In general, mortars deteriorated more extensively than the corresponding pastes. Two-inch cube specimens of mortar were not uniformly deteriorated, but showed obvious zoning even after a year in the brine, with a relatively unreacted zone remaining at the center of each cube. Loss of calcium from the calcium hydroxide of paste/aggregate interfaces caused measurable strength loss in the reacted zone comprising the outer portion of every mortar specimen.

Comparisons of the microstructure of deteriorated specimens and control specimens revealed the destruction of the bond-forming calcium hydroxide in reacted zones, and formation of gypsum at these interfaces. Changes in phase composition as determined by X-ray diffraction, and in total chemical composition as determined by energy-dispersive X-ray analysis, showed that calcium loss

began immediately with initiation of exposure. Calcium hydroxide was destroyed rapidly and calcium silicate hydrate more slowly, with the deterioration product magnesium silicate hydrate appearing only after more than 6 months of brine exposure. Strength loss is related to loss of calcium rather than formation of weak phases.

Of the variables analyzed statistically, presence or absence of salt as a mixture component had the strongest influence on rate of deterioration. Salt mitigated magnesium-related deterioration of mixtures that had an expansive component. Rates of deterioration were estimated from strength loss. The deterioration rate estimated for mixtures formulated with an expansive component but without salt was about 0.04 mm/day. For other mixtures, the rate was estimated to be between 0.015 and 0.03 mm/day.

The current candidate mass concrete for WIPP seals includes salt as an initial component, and has a relatively closed initial microstructure. Both of these features contribute to its suitability for use in large placements within the Salado Formation.

ACKNOWLEDGEMENT

This work was performed for Sandia National Laboratories (SNL) supported by the U.S. Department of Energy under contract DE-AC04-76DP00789. The performing agency was the Concrete Technology Division (CTD), Structures Laboratory (SL), U.S. Army Engineer Waterways Experiment Station (WES), under SNL Document AA 2030. Dr. Lillian D. Wakeley, WES, was Principal Investigator. Dr. Frank Hansen was Project Manager for SNL Department 6121, of which Dr. Joe Tillerson is Manager.

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1.0 BACKGROUND

The Waste Isolation Pilot Plant (WIPP) near Carlsbad, New Mexico, is a research and development facility of the U.S. Department of Energy. It is planned as a mined geologic repository for transuranic wastes generated by U.S. defense activities. The U.S. Army Engineer Waterways Experiment Station (WES) provides research support to Sandia National Laboratories (SNL) involving concretes and concrete technology for the WIPP facility.

In 1990, 6-yr-old concrete in the liner of the WIPP waste-handling shaft was found to have deteriorated in some locations by reactions with magnesium ions in the groundwater (Wakeley et al., 1992). This concrete was designed for hydrostatic conditions with a minimum compressive strength of 5000 psi and an expected service life of 25 years (Westinghouse Electric Corporation, 1992). Its potential chemical stability was not studied before placement. Although it would have been considered a high-quality concrete in many service environments, it had not been proportioned for specific resistance to chemical attack. The functional requirements of a shaft liner are less demanding than are those of seal components, for several reasons. The shaft liner has a shorter service life, does not function as a gas barrier, and does not support the lithostatic loads of the repository level. In addition, a shaft liner is accessible for remediation, which is not true of seal components. The concrete shaft liner, with appropriate maintenance and repair, is expected to perform satisfactorily for its intended period of service (Westinghouse Electric Corporation, 1992).

Groundwaters at the WIPP facility horizon (656 m underground) and close above it include high concentrations of a variety of salts (Krumhansl et al., 1991). Some of these salts are known to cause deterioration and loss of strength to hardened concrete in many environments. The most notable of these are salts of magnesium and sulfate (Mather, 1966; Oberste-Padtberg, 1985; Bonen and Cohen, 1992). A summary of the most widely proposed mechanism of deterioration of cements in high-Mg brine is given by Poole et al. (1994).

1.1 Four Lines of Research

Since the recognition of magnesium-related deterioration in part of the WIPP system in 1990, research at the WES has considered the mechanism of this deterioration and ways to make concrete more resistant to it. Our research has approached the issue of concrete durability in the WIPP environment through four studies, which are outlined in the following paragraphs.

The first study was analysis of concrete and grout cores recovered from the repository floor after 6 years *in situ* (Wakeley et al., 1993). In that study, we determined that a concrete proportioned by the WES and placed at the WIPP in 1985 had performed as intended, and had experienced no chemical deterioration even though it had been exposed to brine. It had gained strength after placement, achieving over 8000 psi compressive strength when tested in 1991, relative to its strength of 3500 psi within months after placement.

The second line of research began in 1991, with initiation of a major effort to develop concrete for use in large-scale placements at the proposed repository horizon. This involved selection of component materials with enhanced resistance to chemical degradation, and proportioning a concrete with appropriate mixing and handling characteristics for mass placements underground. The material developed during this study is Salado Mass Concrete (SMC). Because SMC is intended for mass-concrete seals, its thermal and mechanical properties were carefully controlled and verified in the laboratory study (Wakeley et al., 1993). Work on SMC has continued at WES, with determination of mixture modifications and guidelines appropriate for large-scale mixing and placing.

The third and fourth studies have been conducted to determine experimentally whether this concrete will be durable for the design life of the seal component, approximately 100 years. One of these approached the mechanism of deterioration of portland-cement paste in brines rich in magnesium, sulfate, and chloride. The purpose was to determine whether the deleterious action of these ions is individual or interdependent. The results indicated a mechanism that involves dissolution of calcium (Ca) phases and transport of Ca ions to the surface of the sample, followed by formation of magnesium (Mg) phases at this reaction surface, rather than directly by substitution within the microstructure of hydrated cement (Poole et al., 1994). The rate of deterioration by this mechanism will depend on Mg-ion concentration, availability of brine, and microstructure and composition of the concrete exposed to that brine.

1.2 Purpose of the Research Reported Here

The fourth study is the subject of this report. The purpose of this work was to determine the extent to which different candidate concrete materials and combinations of materials are susceptible to deterioration in high-Mg brine. Questions about the extent to which certain materials might affect overall concrete performance had arisen from the study of the deteriorated concrete from the liner of the waste-handling shaft. Specifically, use of Class C fly ash and use of a non-salt concrete were thought to have contributed to the susceptibility of that concrete to chemical attack (Wakeley et al., 1992). Thus, the work described here was intended to confirm or refute the chemistry-based arguments for selection of component materials of SMC. A further purpose was to identify techniques by which to quantify physical and chemical affects of these brines on candidate mixtures. Also, we used the results of this study to approximate deterioration rate.

2.0 MATERIALS AND SPECIMENS

To study the susceptibility to chemical attack attributed by various component materials, we cast in the laboratory both cement-paste and mortar[1] specimens representing 11 combinations of materials. These mixtures represent the range and combinations of materials that have been considered for application at the WIPP, guided by the questions that arose from the study of the waste-shaft concrete. Specimens were exposed to a synthetic brine with concentrations of magnesium and sulfate representing the high end of the range of these ions in brines at the WIPP. Changes in compressive strength, chemical composition, and phase composition of pastes and mortars were examined.

The following three sections first describe the materials included in the experimental matrix. Second, the eleven mixtures are defined and procedures used for fabricating and aging specimens are described. The third section gives compositions of brines used in various sets of experiments.

2.1 Materials

Of the eleven mixtures, nine were proportioned with a Class H oilwell cement (Smith, 1990). This oilwell cement has been used extensively in grouts and concretes investigated for underground applications (Buck, 1985a,b; Stormont, 1985; Gulick and Wakeley, 1989; Wakeley, 1990; Wakeley and Ernzen, 1991). Two of the mixtures included a Type K cement (ASTM C 845). It has been considered for use underground at the WIPP because of its shrinkage-compensating properties, and because it has been used extensively in grouts at the Nevada Test Site.

Type K cement is not a component of the currently recommended formulation for Salado Mass Concrete, principally because of its early temperature rise and difficulty with extending its working time for several hours. Still, its application at the WIPP is plausible, given that it is commercially available and its use would decrease the number of components of the concrete. Type K could be used only if mixing and placing procedures do not require a long working time. It, too, has been

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[1] "Cement paste" is a mixture of hydraulic cement and water, in this case with other finely divided cementitious materials and water soluble chemical admixtures added. Pastes include no aggregate. "Mortar" is a mixture of cement paste and fine aggregate. It corresponds to the material occupying the interstices among particles of coarse aggregates in concrete.

used successfully for many years at the Nevada Test Site (Houston, 1970; Hoff, 1973); and it is available near the WIPP. Physical and chemical properties of these cements are summarized in Appendix A.

One Class C and one Class F fly ash (ASTM C 618) were used in mixtures containing Class H cement. Class C fly ash was used in much of the early developmental work because of its contribution the expansive properties of concrete (Wakeley and Walley, 1986). Use of Class F fly ash has been recommended to reduce early heat evolution and to contribute resistance to chemical deterioration. It is currently the preferred material for Salado Mass Concrete (SMC) (Wakeley et al., 1993). We anticipated that this comparison between fly ash classes would support the decision to use Class F ash in SMC. Physical and chemical properties of the fly ashes are summarized in Appendix A.

Plaster of paris (Cal Seal, Halliburton; see Appendix A) was used to develop expansive properties in some of the mixtures containing Class H cement. The SMC has an expansive component, to enhance bonding between concrete and host rock. Therefore an expansive component was included in the matrix for the current work, to determine if it had an independent affect on susceptibility to deterioration, and secondarily to assess differences in performance attributable to chemical interaction between an expansive component and salt.

A high-range water-reducing admixture (Sikament-10 ESL) was used to get necessary workability at a fixed water-cement ratio. It was selected because it permitted us to achieve equivalent short-term workability for all mixtures without causing foaming in the presence of salt (Grutzeck, 1985). We were not concerned with long working times or rapid slump loss in this study, so these were not considered in selection of an admixture. Sikament-10 ESL had not been considered in previous development of concrete for the WIPP, because it has been available in the US only since 1991.

Commercial grade sodium chloride was used to saturate mixing water in salt-containing mixtures. The reasons for using salt-saturated concrete at the WIPP are discussed elsewhere (Wakeley et al., 1993). In this study, we compared the deterioration of salt-containing mixtures in the test brines to that of non-salt equivalents.

Deionized water (ASTM C 1193) was used to make all solutions and specimens. Reagent grade $MgCl_2 \bullet 6H_2O$, NaCl, $MgSO_4 \bullet 7H_2O$, and KCl were used to make brines. The mortars included a graded standard sand (ASTM C 778, from U.S. Silica).

2.2 Mixtures and Fabrication of Specimens

Compositions of the eleven mortar mixtures are outlined in Table 2-1. Water to cementitious materials ratio was 0.35 (by mass). Sand-to-cement ratio was 2.75, by mass. High-range water reducer was used to adjust flow to $110\% \pm 5\%$ (ASTM C 109). This required 0.8% admixture (vol/mass of cementitious material) for mixtures 1 through 9, and 1.5% for mixtures 10 and 11. Mortars were mixed and specimens fabricated according to ASTM C 305 and C 109, respectively.

Non-salt cubes were demolded after 24 hrs. Salt-containing cubes were demolded after 48 hrs. They needed additional time in the molds because they gained strength more slowly, and additional time gave them adequate strength to be demolded without breaking. After demolding, each group of samples was cured for about 2 weeks, to allow strength gain and development of the microstructure of hydrated cement before exposure to the test brines. We did not try to recreate a specific in situ condition. However, we considered it unlikely that a concrete placement will be exposed to a large quantity of brine immediately after placement. Thus it was reasonable to allow the samples to gain strength for a few weeks before initiating brine-immersion tests.

Non-salt cubes were stored 17 days in water saturated with calcium hydrated at 73 °F, then immersed in brine at 80 °F until age for compressive-strength testing. Control cubes were immersed in saturated lime water at 80 °F. Salt-containing cubes could not be immersed in the standard lime water solution for the first two weeks, because this solution removes salts from the concrete. Instead, they were cured for 16 days in water-saturated air at 73 °F, then immersed in brine. Control cubes were stored in water-saturated air at 80 °F. Each group of 12 cubes was stored in a plastic cylinder (6- by 12-in. concrete-cylinder mold) with 3800 mL of brine.

A second set of specimens was prepared of eleven cement-paste mixtures, equivalent to the paste fraction of the 11 mortar mixtures. These were used to evaluate chemical effects of the brine on the hydrated cement paste, without the complicating factor of interfaces with aggregates. Pastes

Table 2-1. Composition of Mortars

Mixture No.	Cement	Fly Ash	Plaster	NaCl
M-1	Class H	none	none	none
M-2	Class H, 65% by vol.	Class C, 35% by vol.	10% by mass cementitious materials	SMW*
M-3	Class H, 65% by vol.	Class C, 35% by vol.	10% by mass cementitious materials	none
M-4	Class H, 65% by vol.	Class C, 35% by vol.	none	SMW*
M-5	Class H, 65% by vol.	Class C, 35% by vol.	none	none
M-6	Class H, 65% by vol.	Class F, 35% by vol.	10% by mass cementitious materials	SMW*
M-7	Class H, 65% by vol.	Class F, 35% by vol.	10% by mass cementitious materials	none
M-8	Class H, 65% by vol.	Class F, 35% by vol.	none	SMW*
M-9	Class H, 65% by vol.	Class F, 35% by vol.	none	none
M-10	Type K	none	none	SMW*
M-11	Type K	none	none	none

* Saturated mixing water.

specimens were cast and cured in 20- by 70-mm Nalgene vials with snap-on caps. They were cured in molds at 73 °F for 18 days, stripped from molds and exposed to either brine H-1RSCM or limewater or water-saturated air at 80 °F, as described for the mortar cubes, except specimens were stored in glass jars. Each jar held 4 cylinders and 240 mL of liquid (brine or saturated lime water). Some specimens were cured for 155 days before exposure to brine. These are represented by and "A" suffix to the mixture number, e.g. M-1A.

A third set of samples was prepared for additional experiments with pastes of M-6 and M-8. These two mixtures were chosen because the component materials were the most similar to materials used in SMC. Additional experimentation with these pastes was coupled with the work on SMC development. Paste samples were cured for about three weeks, and then exposed to each of four brines with different magnesium concentrations: 4.2, 20.0, 42.4 (H-1RSCM), and 60.0 g/L Mg^{2+}. The purpose of these conditions was to evaluate the effect of Mg concentration on rate of deterioration, and to identify a threshold level of magnesium if one exists.

2.3 Brines

The principal brine solution used in this work is designated H-1RSCM. Its composition was recommended by S. Lambert (SNL) from analysis of brines sampled in the vicinity of the WIPP. Both the Mg and SO_4 ion concentrations are very high relative to what has been analyzed from brine seeps at the repository horizon. The Mg ion concentration is 42.4 g/L. The SO_4 ion concentration is 18.2 g/L. The mass of salts used (Krumhansl et al., 1991) to make this brine are summarized in Table 2-2.

Table 2-2. Composition of Brine H-1RSCM

Salt	Concentration (g/L)
NaCl	104.8
$MgCl_2.6H_2O$	319.9
$MgSO_4.7H_2O$	46.8
KCl	32.1

Pastes representing two mixtures (M-6 and M-8) were exposed to three other brines the Mg ion concentrations of which had been modified from this composition (4.2, 20.0, and 60.0 g/L). These changes were made by varying the amount of $MgCl_2.6H_2O$, consequently, the concentration of Cl ion varied somewhat also. The concentration of NaCl in the 60.0 g/L solution was reduced relative to H-1RSCM to maintain complete solubility of the brine salts.

3.0 METHODS OF ANALYSIS

At test ages, mortar cubes were removed from storage, surface dried, and their mass determined after precipitates were scraped off. We had intended to use loss of mass as an indicator of concrete deterioration. Also to indicate deterioration, compressive strength of each cube was tested according to ASTM C 109, for comparison with strength of control cubes at ages of 30, 90, 180, and 365 days.

Mortar cubes of mixture M-6, the mortar most similar to SMC, were analyzed in detail for changes in chemical and phase composition and microstructure after one year in H-1RSCM brine. For these analyses, samples were prepared to present a cross-section from the outer deteriorated zone of the cube to the center, where the mortar appeared to be in a less deteriorated condition. This made it possible to follow the ingress of brine and document its progressive effects, and to contrast the microstructure and composition of the brine-exposed specimens to these characteristics of the controls. It also exposed paste-aggregate interfaces in these gradational reaction zones.

Analytical techniques applied to the year-old mortar cubes included: visual examination; X-ray diffraction (XRD), for determination of phase composition; scanning-electron microscopy (SEM) coupled with energy-dispersive X-ray analysis (EDX), for determination of microstructure and relationships between microstructure and chemical composition; and petrographic thin sections, for study of hydration products and interfaces. Details of samples preparation methods and analytical techniques are in Appendix B.

Disc-shaped specimens were cut from the brine-exposed or control paste cylinders for visual examination, XRD, SEM, and EDX. Again, the subsampling scheme was designed to expose the gradation of reaction zones. For EDX analysis, USGS rock standard GSP-1 was used as the standard (Flanagan, 1967). Data were not normalized to 100%, therefore, the formation or removal of any one chemical compound did not affect the reported composition of other compounds. Sample preparation and analytical methods are described in Appendix B.

4.0 RESULTS

4.1 Effect of Brine on Paste Specimens

4.1.1 Effect of H-1RSCM Brine on Eleven Paste Mixtures

The cylinders that were stored in brine were coated with a patchy layer of white precipitate. This material had been collecting on the specimens since soon after they were placed in the brine. It was thicker and covered more completely the surfaces of cylinders kept in the brines with higher magnesium contents. There was some apparent deterioration at top and bottom rims of the cylinders, where the paste was obviously softened.

Determination of chemical composition by EDX revealed chemical alteration of paste specimens as a result of exposure to brine. Some chemical species were lost and others accumulated, as described below for each constituent. Effects were particularly apparent in the data from EDX analyses of samples at 1 mm from exposed surfaces. At a depth of 3 mm, these changes were less obvious or not apparent. These patterns are illustrated in Figures 4-1 through 4-6 for mixtures M-6 and M-7. Data from the other mixtures revealed similar trends. Complete EDX data are in Appendix C.

In visual examination of pastes stored in brines, there no notable difference in appearance or depth of reaction zones between 28 and 220 days. Data from chemical analyses over this age range were subjected to linear regression, which revealed no significant time dependence. The chemical changes that were to occur in the outer 1 mm of the sample had occurred by 28 days, and after this time the composition remained nearly constant. That is, chemical changes at the 1-mm analysis point were not time dependent. Given that there was no significant difference between these data sets, they were averaged for comparisons of brine-exposed and control specimens. These comparisons are summarized in Tables 4-2a through 4-2h.

The comparisons in Tables 4-2a through 4-2h were made by student's t-Test. This test compares the difference between two means relative to the variation in the test method. The t statistic is the ratio of the difference between the two means and the averaged standard deviation of the

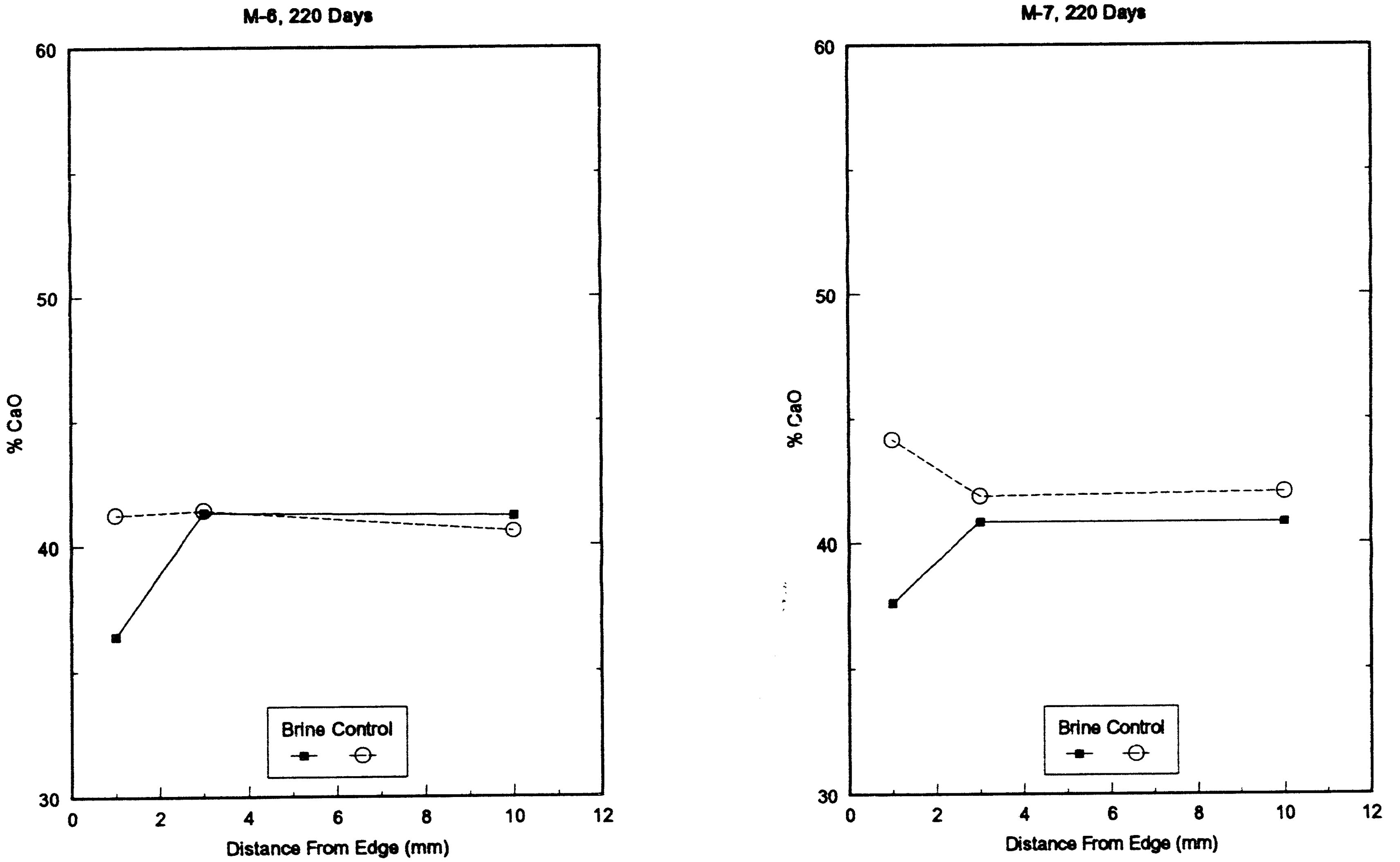

Figure 4-1. Percentage of calcium oxide in M-6 and M-7 pastes.

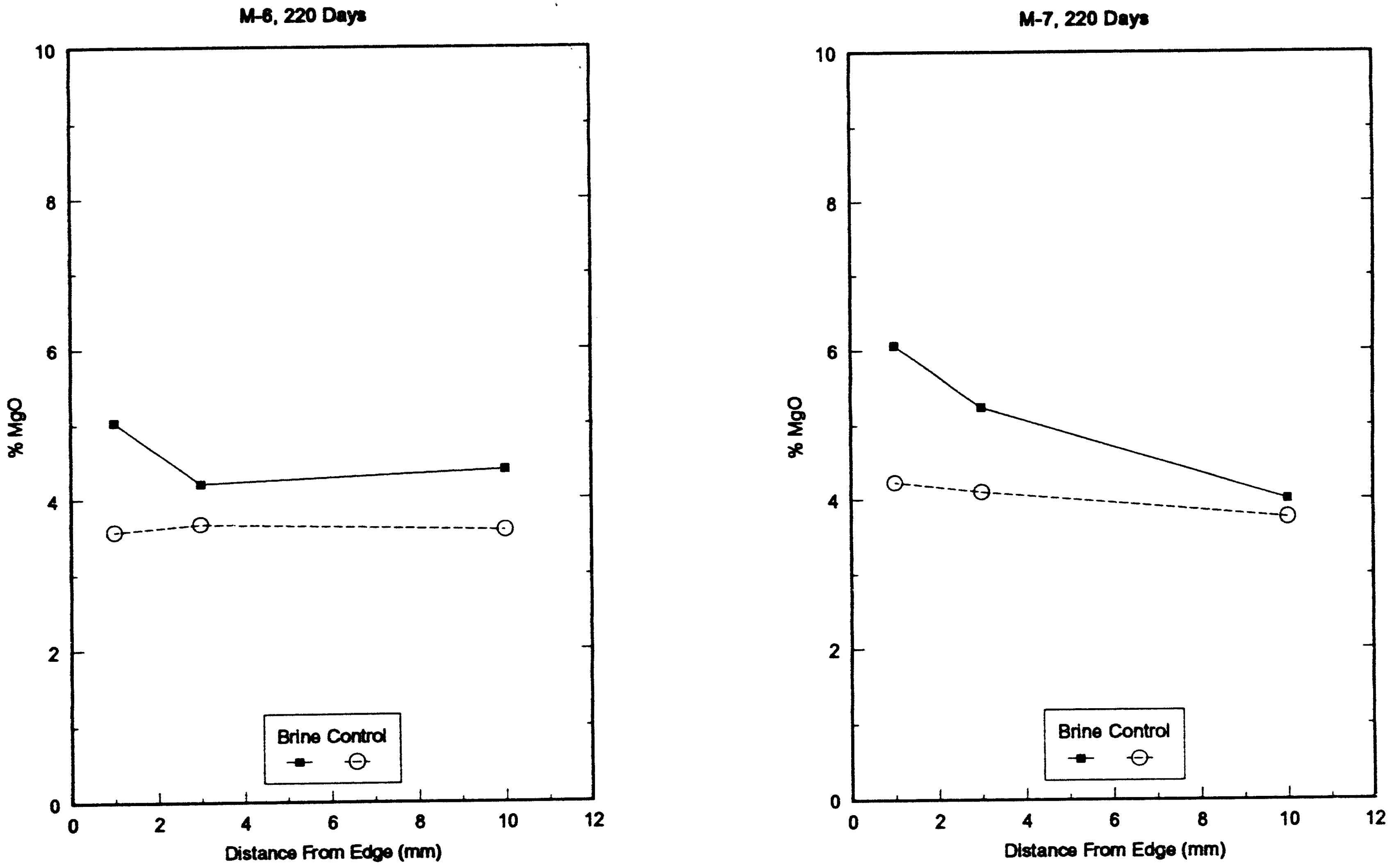

Figure 4-2. Percentage of magnesium oxide in M-6 and M-7 pastes.

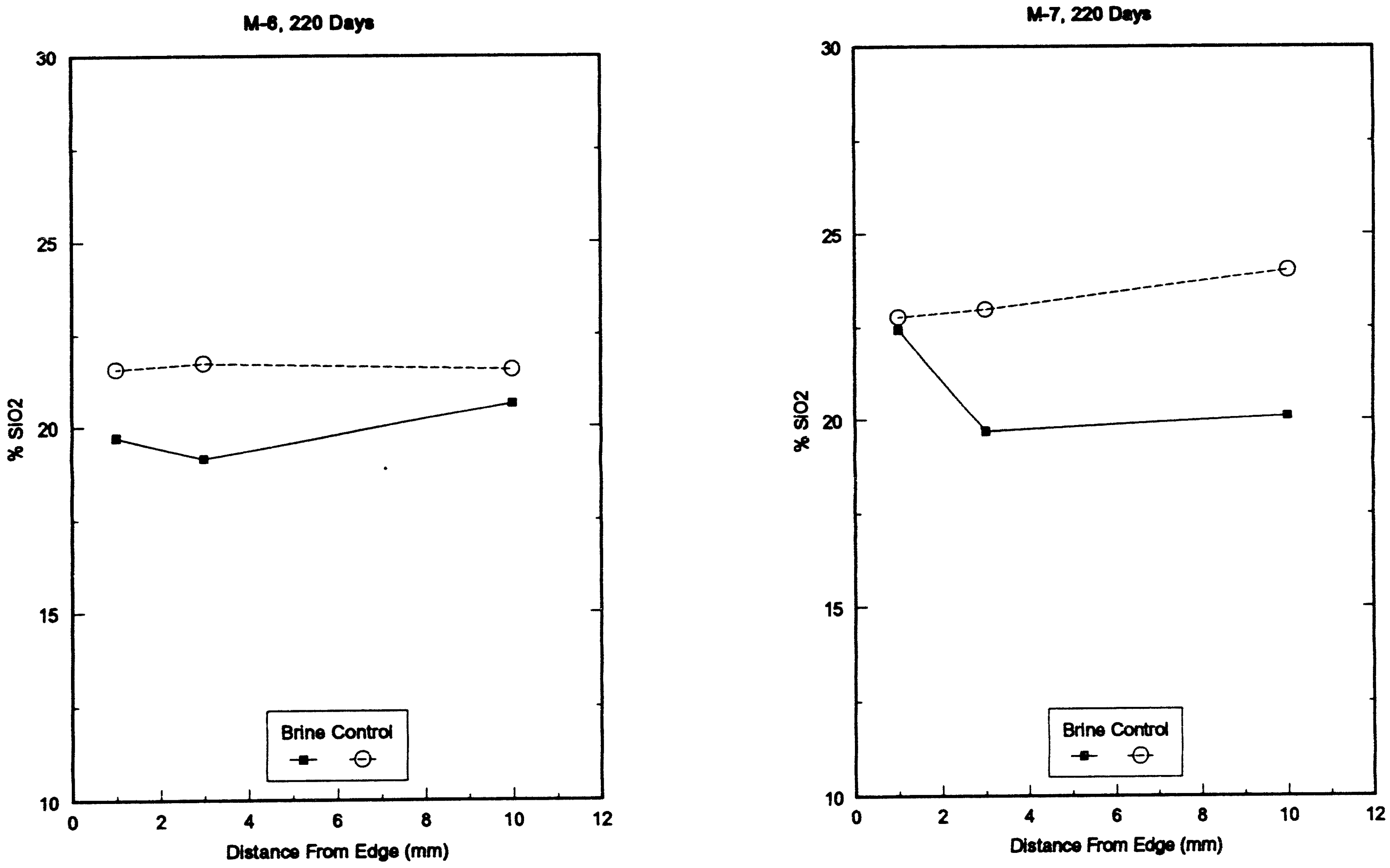

Figure 4-3. Percentage of silica in M-6 and M-7 pastes.

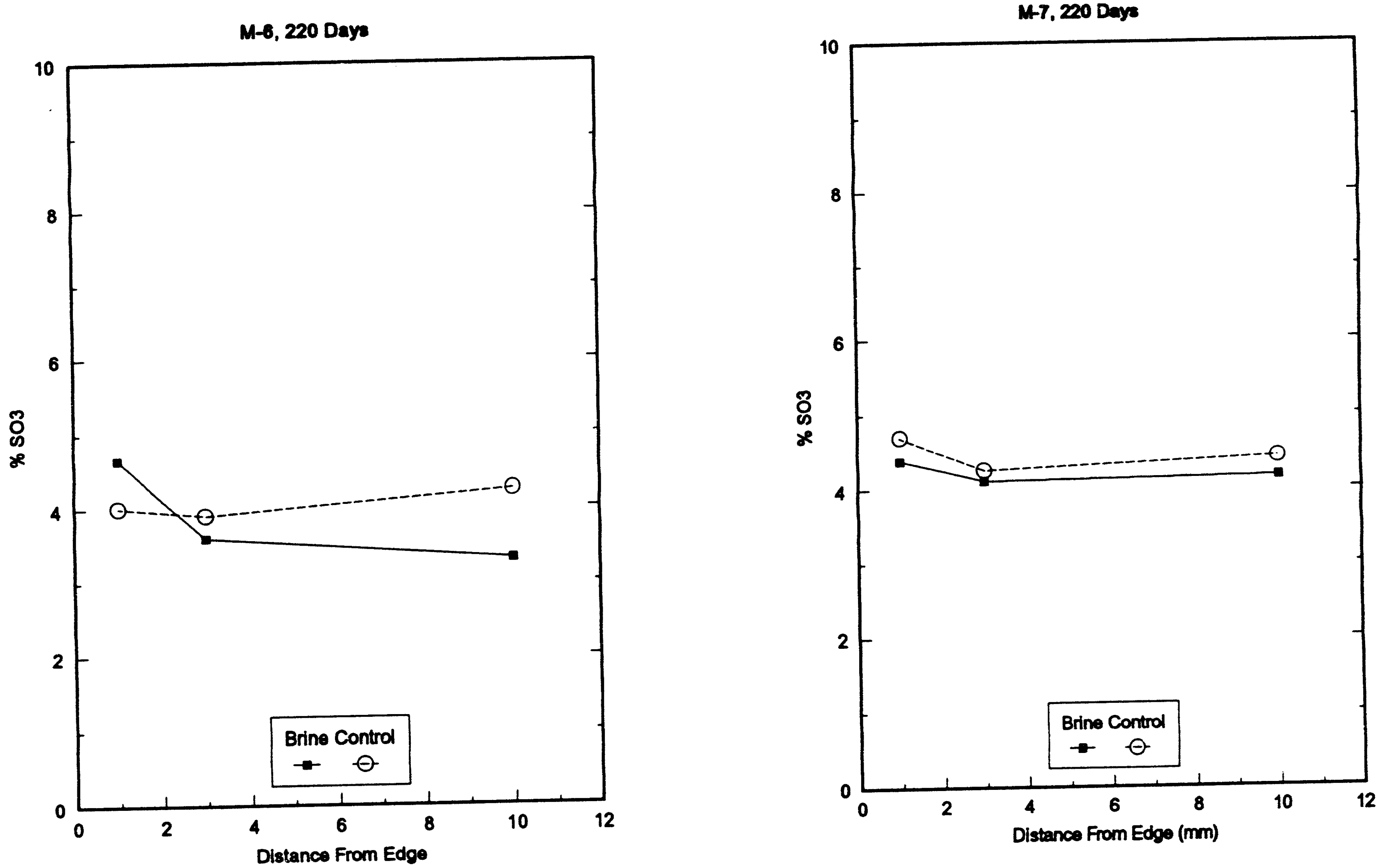

Figure 4-4. Percentage of sulfur trioxide in M-6 and M-7 pastes.

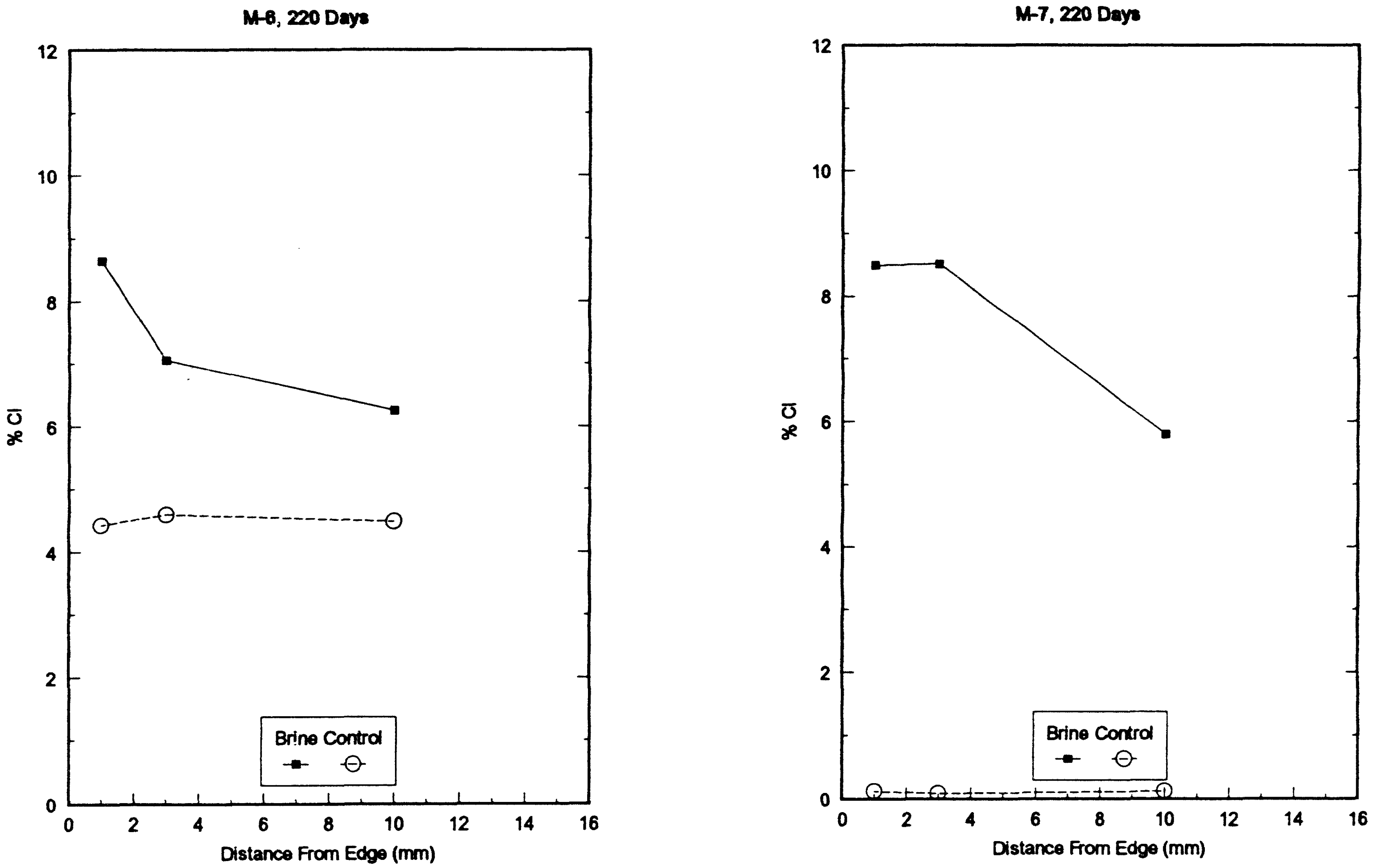

Figure 4-5. Percentage of chloride in M-6 and M-7 pastes.

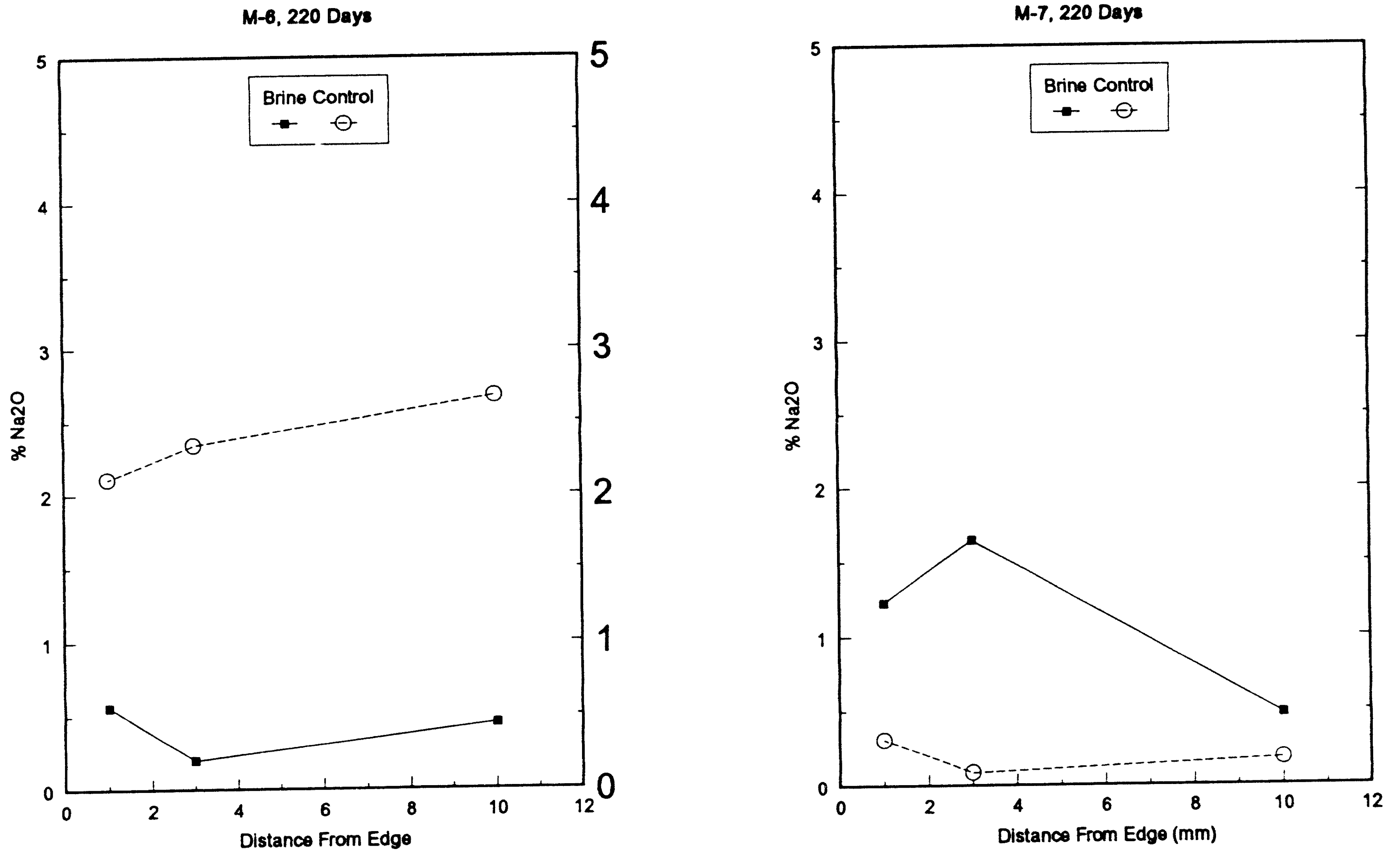

Figure 4-6. Percentage of sodium oxide in M-6 and M-7 pastes.

Table 4-2. Comparison of Percentages of Several Ions at 1 mm Depth in Brine-Exposed vs. Control Pastes

Table 4-2a. Comparison of CaO

Mixture	Brine Exposed	Control	Difference (Br.-Contr.)	Variance**
M-1	44.76	53.79	-9.03*	3.05
M-2	41.41	44.33	-2.92*	27.95
M-3	40.95	46.38	-5.43*	2.11
M-4	38.25	43.14	-4.89	17.81
M-5	39.99	45.16	-5.17*	10.50
M-6	37.50	41.95	-4.45*	4.40
M-7	44.16	35.53	-8.63*	22.47
M-8	36.45	40.10	-3.65*	12.22
M-9	38.12	42.03	-3.96*	6.91
M-10	42.76	47.17	-4.41*	3.26
M-11	42.93	49.35	-6.42*	3.08

Table 4-2b. Comparison of MgO Levels

Mixture	Brine Exposed	Control	Difference (Br.-Contr.)	Variance**
M-1	7.04	4.35	2.69	8.31
M-2	5.92	2.80	3.12*	4.00
M-3	6.45	3.89	2.56*	2.59
M-4	6.17	3.62	2.55*	1.95
M-5	5.91	4.15	1.76	2.00
M-6	5.03	3.15	1.88*	0.74
M-7	10.02	4.22	5.80	39.92
M-8	8.85	3.00	5.85*	31.72
M-9	6.59	3.71	2.88*	7.64
M-10	7.58	5.52	2.06	3.41
M-11	7.48	6.59	0.89	13.05

* Different from 0.00 at a probability level of 5%, as analyzed by Student's t-Test.

** Variance in oxide analyses, each analysis taken as an average from 3 spectra.

Table 4-2c. Comparison of SiO_2 Levels

Mixture	Brine Exposed	Control	Difference (Br.-Contr.)	Variance**
M-1	16.14	18.68	-2.54*	4.36
M-2	13.03	12.30	0.73	0.98
M-3	16.73	18.23	-1.50*	0.68
M-4	17.16	18.63	-1.47	2.60
M-5	18.25	19.66	-1.47	1.99
M-6	21.01	22.30	-1.29	1.71
M-7	21.33	22.78	-1.45	2.75
M-8	23.15	24.63	-1.48*	1.78
M-9	23.34	26.30	-2.96	3.79
M-10	14.05	13.78	0.27	1.60
M-11	13.42	14.53	-1.11	1.42

Table 4-2d. Comparison of Al_2O_3 Levels

Mixture	Brine Exposed	Control	Difference (Br.-Contr.)	Variance
M-1	2.61	3.56	-0.95*	0.25
M-2	2.96	2.90	0.06	0.18
M-3	5.41	5.43	-0.02	0.21
M-4	5.45	6.07	-0.62	0.75
M-5	6.02	6.26	-0.24	0.34
M-6	5.64	6.05	-0.41	0.28
M-7	5.65	6.24	-0.59	0.26
M-8	6.26	7.06	-0.80*	0.47
M-9	6.75	7.56	-0.81	0.59
M-10	4.16	3.76	0.40	0.14
M-11	4.10	4.29	-0.19	0.12

* Different from 0.00 at a probability level of 5%, as analyzed by Student's t-Test.

** Variance in oxide analysis, each analysis taken as an average from 3 spectra.

Table 4-2e. Comparison of Fe_2O_3 Levels

Mixture	Brine Exposed	Control	Difference (Br.-Contr.)	Variance**
M-1	2.85	2.85	0.00	0.20
M-2	2.07	2.17	-0.10	0.18
M-3	2.57	2.49	0.08	0.03
M-4	2.65	2.68	-0.03	0.06
M-5	2.84	2.88	-0.04	0.06
M-6	2.66	2.54	0.12	0.08
M-7	2.68	2.94	-0.26	0.10
M-8	2.81	2.89	-0.08	0.11
M-9	2.82	3.24	-0.42	0.19
M-10	2.03	1.90	0.13	0.06
M-11	2.03	2.02	0.01	0.05

Table 4-2f. Comparison of SO_3 Levels

Mixture	Brine Exposed	Control	Difference (Br.-Contr.)	Variance**
M-1	1.61	1.25	0.36*	0.04
M-2	9.68	7.63	2.05*	1.10
M-3	4.56	4.59	-0.03	0.14
M-4	1.99	1.15	0.84*	0.29
M-5	1.66	1.32	0.34	0.10
M-6	4.55	4.10	0.45*	0.17
M-7	4.20	4.70	0.50	0.18
M-8	1.78	0.94	0.84*	0.28
M-9	1.67	1.19	0.48*	0.11
M-10	3.91	3.10	0.81*	0.34
M-11	3.33	3.16	0.17	0.15

* Different from 0.00 at a probability level of 5%, as analyzed by Student's t-Test.

** Variance in oxide analysis, each analysis taken as an average from 3 spectra.

Table 4-2g. Comparison of Cl Levels

Mixture	Brine Exposed	Control	Difference (Br.-Contr.)	Variance**
M-1	10.31	0.02	10.29*	8.68
M-2	8.67	5.44	3.23*	5.53
M-3	7.07	0.26	6.81*	1.68
M-4	10.70	4.89	5.81	35.02
M-5	9.51	0.25	9.26*	16.75
M-6	7.59	5.38	2.21*	1.31
M-7	8.00	0.13	7.87*	0.88
M-8	8.84	6.29	2.55*	0.55
M-9	8.31	0.27	8.04*	2.51
M-10	10.24	5.25	4.99*	8.16
M-11	9.94	0.22	9.72*	7.67

Table 4-2h. Comparison of Na_2O Levels

Mixture	Brine Exposed	Control	Difference (Br.-Contr.)	Variance**
M-1	0.69	0.63	0.06	0.74
M-2	0.68	3.54	-2.86*	1.67
M-3	0.59	0.12	0.47	0.41
M-4	0.49	1.08	-0.59*	0.12
M-5	0.15	0.04	0.11	0.01
M-6	1.06	3.10	-2.07*	1.10
M-7	0.86	0.31	0.55	0.22
M-8	0.80	3.42	-2.62*	0.33
M-9	0.85	0.64	0.21	0.50
M-10	0.86	3.06	-2.20*	1.73
M-11	0.75	0.16	0.59	1.52

* Different from 0.00 at a probability level of 5%, as analyzed by Student's t-Test.

** Variance in oxide analysis, each analysis taken as an average from 3 spectra.

means. The size of the t statistic that represents a significant difference between the means depends on the sample size and the probability of drawing an incorrect conclusion from the analysis. The sample size is usually expressed as degrees of freedom (d.f.), which is the sample size minus 1. The probability (P) is the probability that the difference in the means could be gotten by chance. The smaller the level of P, the stronger is a conclusion that two means differ.

The most conspicuous effect of exposure of paste specimens to brine was loss of calcium, expressed as CaO. CaO levels in the outer millimetre of brine-exposed specimens, averaged over the 11 mixtures, were 5.4% less than those of controls, expressed as a simple difference between CaO levels in brine-exposed and control specimens. This represents a loss of 0.096 moles of CaO per 100 grams of paste. There was some variation among mixtures, as summarized in Table 4-2a.

Mixtures containing salt (2, 4, 6, 8, and 10) lost on average 4.06% CaO, compared to a 6.44% loss of CaO from salt-free mixtures. This difference was significant when compared by Student's t-Test ($t=2.47$, $P=0.035$, 9 d.f.). The presence or absence of calcium sulfate, the type of fly ash, and the type of cement were all statistically insignificant variables when compared by Student's t-Test at $P \leq 0.05$.

Magnesium (expressed as MgO) levels were consistently elevated in the outer millimetre relative to controls, by an average of 2.9% (Table 4-2b). This represents a gain of 0.073 moles of MgO per 100 g of paste. Variation in MgO levels among analyses appeared to be higher than would be expected from normal analytical error. For example, the coefficient of variation (CV) among analyses of the first millimetre of brine-exposed specimens of M-1 was about 35%, while the CV of controls was about 8%. This pattern suggests that the accumulated Mg was not uniformly distributed in the paste. This high variation would also explain why some apparently large accumulations of MgO were not found to be statistically significant when compared to controls by Student's t-Test, e.g. M-1, Table 4-2b. The presence or absence of salt in the mixing water, the presence or absence of calcium sulfate, the type of fly ash, and the type of cement were all statistically insignificant variables when compared by Student's t-Test at $P \leq 0.05$.

There was a weak pattern of loss of silicon from paste, with silicon expressed as SiO_2: 9 of 11 brine-exposed specimens contained less SiO_2 than controls (see Table 4-2c). The difference, averaged over all mixtures, was 1.34%, or 0.022 moles per 100 grams of paste. In only three, however, was the loss statistically different from zero (M-1, M-3, and M-8). The comparison between salt-containing and non-salt-containing mixtures indicated a weak pattern. Non-salt mixtures lost more SiO_2 (mean=1.84%) than salt-containing mixtures (mean=0.65%), $t=2.19$, $P=0.056$, 9 d.f. The presence or absence of calcium sulfate, the type of fly ash, and the type of cement all were statistically insignificant variables when compared by Student's t-Test at $P \leq 0.05$.

Losses of aluminum, as Al_2O_3, were similar to those experienced by SiO_2 in that small losses were observed in all but mixtures M-2 and M-10 (see Table 4-2d). The average taken over all mixtures was 0.38%. Losses in M-1 and M-8 were the only ones statistically distinguishable from zero at $P=0.05\%$. The presence or absence of salt in the mixing water, the presence or absence of calcium sulfate, the type of fly ash, and the type of cement all were statistically insignificant variables when compared by Student's t-Test at $P \leq 0.05$.

Changes in iron, expressed as Fe_2O_3, were determined to be insignificant relative to all variables (see Table 4-2e).

There were small but statistically distinct differences among mixtures in changes in levels of sulfur, expressed as SO_3, with exposure to brine (Table 4-2f). The salt-containing mixtures all showed significant increases in SO_3. The mean increase for these mixtures was 1.00% over controls. The mixtures containing no salt gained an average of 0.30% SO_3. The difference between these two means was significant ($t=3.38$, $P<0.01$, 9 d.f.). The presence or absence of calcium sulfate, fly ash type, and cement type were not statistically significant variables when compared by Student's t-Test at $P \leq 0.05$.

Changes in chloride (Cl) levels also showed a distinct effect relative to presence or absence of salt in the mixture (see Table 4-2g). Salt-containing mixtures showed less Cl accumulation than did non-salt mixtures (8.67% vs. 3.76%, $t=5.66$, $P=0.0003$, 9 d.f.). All brine-exposed mixtures appeared to converge on a Cl content of between 7% and 10%, regardless of the amount of Cl included in the mixing water. The other variables all were statistically insignificant when compared by Student's t-Test at $P \leq 0.05$.

Sodium, expressed as Na_2O, changes were also strongly dependent on the salt content of the mixture, as summarized in Table 4-2h. Mixtures made with salt tended to lose Na_2O (mean=2.06%), while mixtures made without salt tended to accumulate a small amount (mean=0.33%). This difference was significant ($t=5.89$, $P<0.003$, 9 d.f.). As with Cl, there appeared to be a tendency for all mixtures to converge on a common level of Na_2O after exposure to brine. This level ranged from about 0.2% to 1.0%. The other variables all were statistically insignificant when compared by Student's t-Test at $P \leq 0.05$.

Major crystalline phases present in the pastes after 180 days exposure to brine are summarized in Table 4-3. Comparisons are made between relative peak intensities of each phase as found in the outer millimetre and at the center of the specimen. Calcium hydroxide (CH) is consistently less in the outer millimetre than at the center. Calcium sulfoaluminate (CSA) and calcium chloroaluminate (CCA) each formed, but rarely did so in the same mixture. CSA formed in mixtures without salt and containing either Cal Seal or Type K cement (M-3, M-7, and M-11). There was no strong rim-center gradient. This phase was largely absent from the other mixtures, where CCA was consistently present. In the three mixtures that were both expansive and salt-containing (M-2, M-6, M-10), calcium sulfate dihydrate was present and apparently persisted without converting to CSA to any perceptible extent. In summary, the presence of salt in expansive mixtures appears to retard the reaction between the calcium aluminates and the calcium sulfates to form CSA.

4.1.2 Effect of Varying Mg^{2+} Levels on Chemical Composition of Pastes

Loss of CaO and accumulation of MgO were studied in detail for mixtures M-6 and M-8 aged in brines with various concentrations of Mg. These mixtures were chosen because they most closely approximated the paste fraction of concrete mixtures then under consideration. Chemical composition was determined for disk samples of these pastes along a series of points at increasing distance from the brine-exposed surface, specifically at 1, 2, 4, 6, 8, and 10 mm from the perimeter.

Measurable effects were limited almost exclusively to the 1- and 2-mm depths. After 180 days of exposure, CaO was lost in approximate proportion to the Mg concentration in the brine, as illustrated in Figures 4-7 and 4-8.

MgO gain also was proportional to the Mg concentration in the brine, as illustrated in Figures 4-9 and 4-10. There was some indication of a threshold effect between 40 and 60 g/L Mg, with less MgO accumulation at the latter concentration, but as in the analysis of CaO patterns, there are insufficient data to be conclusive.

Table 4-3. Relative Abundance of Phases Detected by XRD in Rim[1] and Center of Paste Disks

Mixture	Location	Phase				
		$CaSO_4 \bullet 2\,H_2O$	CCA[2]	CSA2[3]	$Ca(OH)_2$	NaCl
M-1	rim		Minor[4]		Minor	
	center		Minor		Major[5]	
M-2	rim	Major	Minor			Minor
	center	Minor	Minor		Major	Minor
M-3	rim	Minor		Minor	Minor	
	center			Major	Major	
M-4	rim	Minor	Major			Major
	center		Major		Major	Major
M-5	rim		Major		Minor	
	center		Minor		Major	
M-6	rim	Major	Minor		Minor	Minor
	center	Major	Major		Major	Minor
M-7	rim			Minor		
	center			Minor	Minor	
M-8	rim	Minor	Minor			Minor
	center		Minor			Minor
M-9	rim		Minor		Minor	Minor
	center		Major		Major	Minor
M-10	rim	Major	Major		Minor	Minor
	center	Minor	Major	Minor	Major	Major
M-11	rim			Major	Minor	
	center			Minor	Major	

[1] Rim is defined as within 1 mm of edge of specimen
[2] CCA = calcium chloroaluminate
[3] CSA = calcium sulfoaluminate
[4] Minor - detectable but not predominant
[5] Major - a predominant phase in XRD pattern

Blank entries indicate phase not detected.

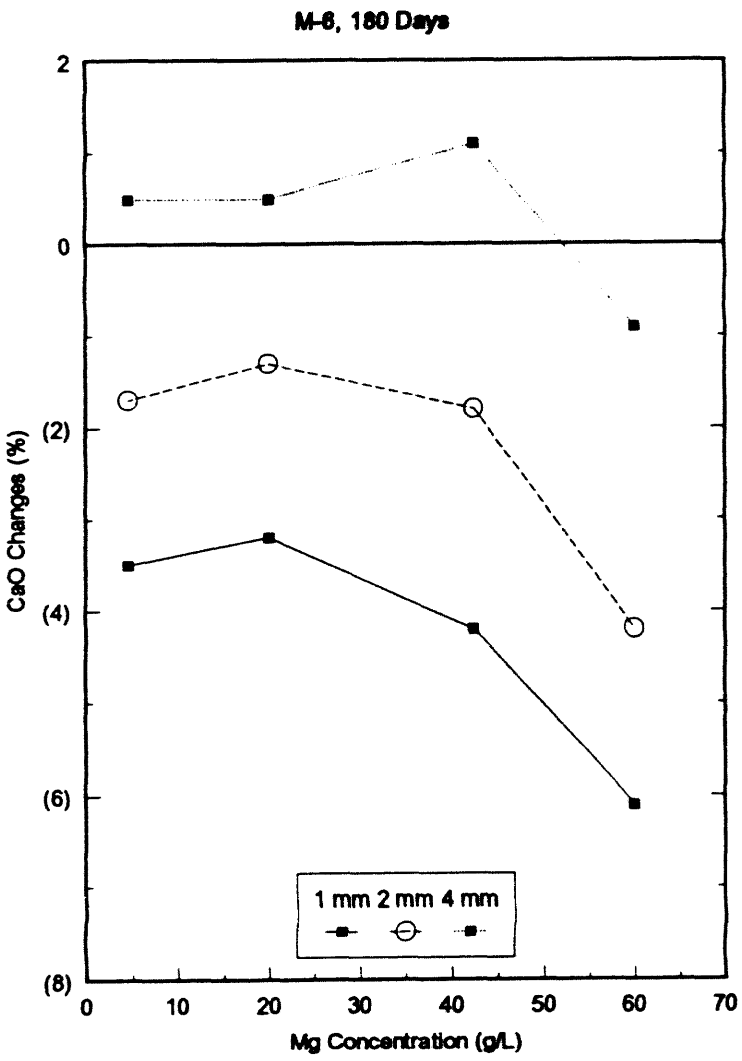

Figure 4-7. Change in CaO in M-6 as a function of Mg in brine.

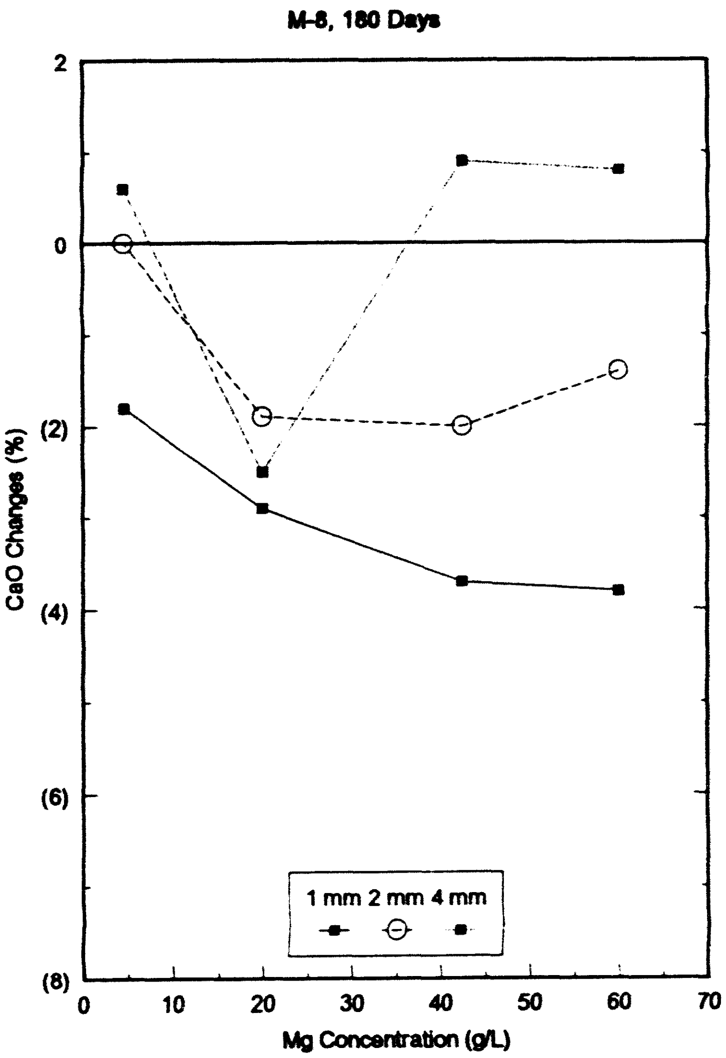

Figure 4-8. Change in CaO in M-8 as a function of Mg in brine.

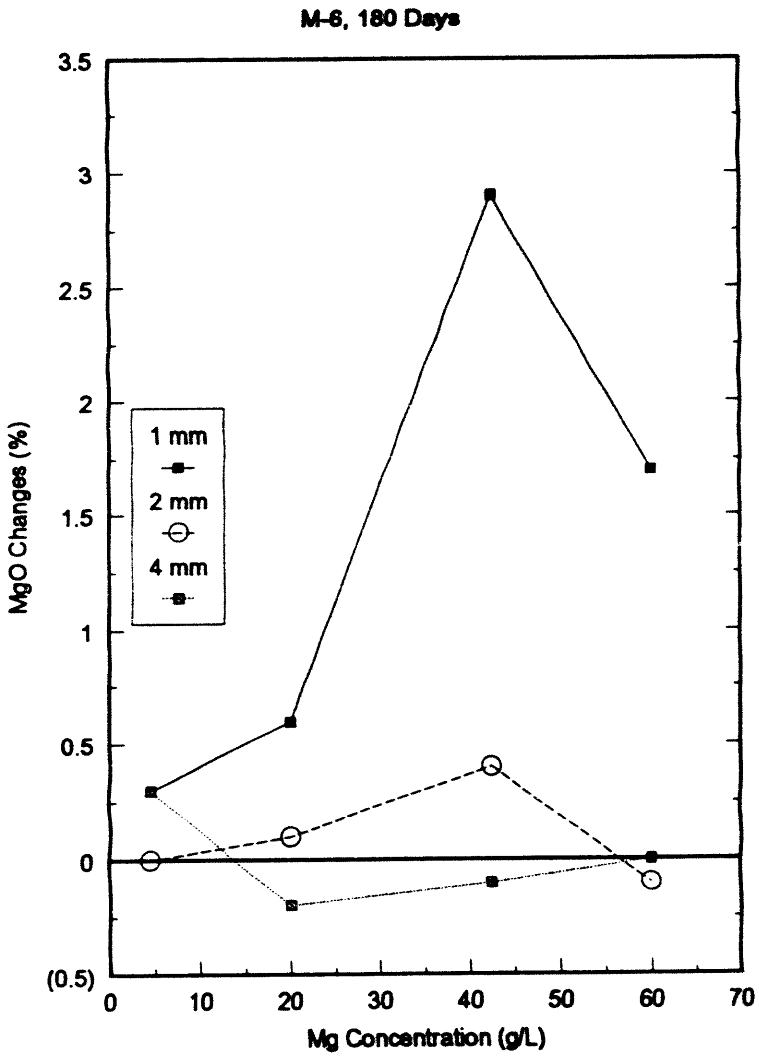

Figure 4-9. Change in MgO in M-6 as a function of Mg in brine.

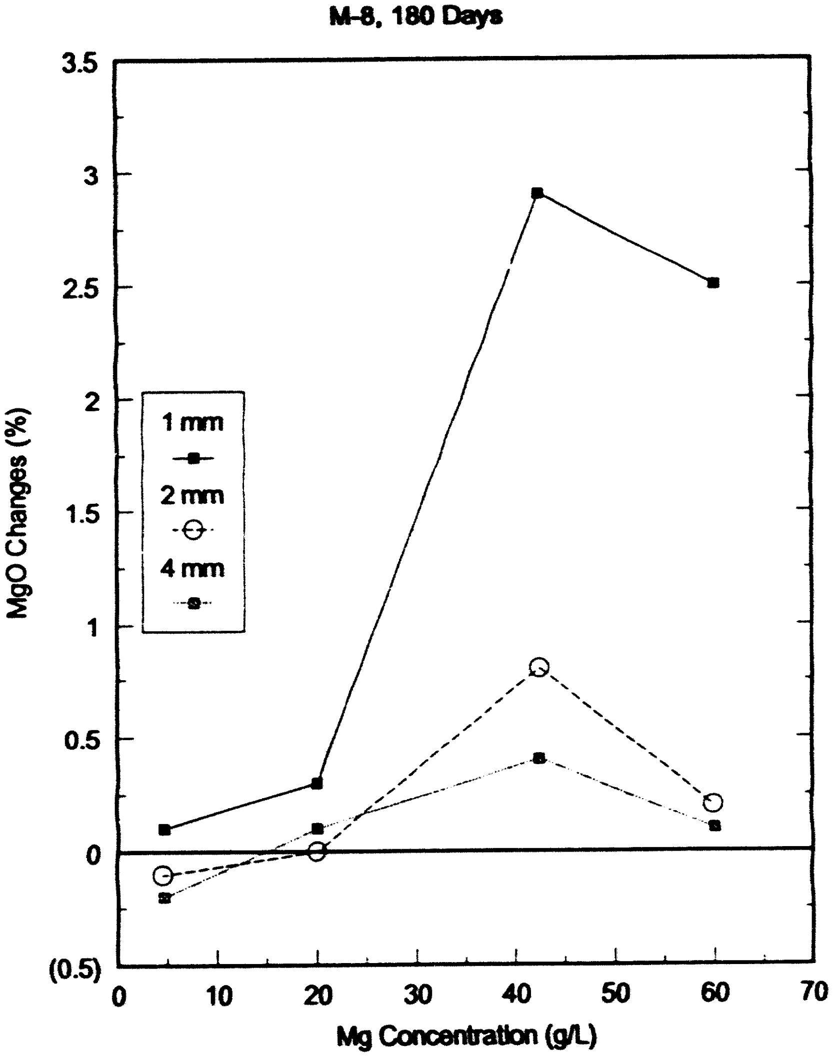

Figure 4-10. Change in MgO in M-8 as a function of Mg in brine.

4.1.3 Analysis of M-6 Paste after One Year

Samples of the M-6 paste were examined in detail after aging for about a year in H-1RSCM brine. There was special interest in this paste, because of its similarity to the paste portion of SMC. Each disk cut from a paste cylinder was vacuum dried for analysis. This drying revealed a zone or reaction rim, distinguishable by its lighter color, occupying the outer 4 to 5 mm of the disk. Observed at low magnification, these rims were found to include numerous irregular voids of a form not noted in control specimens.

Total phase assemblages for samples aged in brine and for control specimens were similar. Both data sets include calcium chloroaluminate, calcium sulfate dihydrate, and halite. The differences are in amounts of certain phases. Unhydrated portland cement and the normal hydration products calcium silicate hydrate (CSH) and calcium hydroxide (CH) are far more abundant in the control specimens. For brine-aged specimens, calcium hydroxide was barely detectable in the first 3 to 4 mm in from the perimeter, but easily detected in the center. These observations are consistent with the lower level of Ca in brine-aged samples revealed by chemical analyses.

Magnesium silicate hydrate (MSH) was detected in the outer rims of paste specimens aged in brine for one year. The presence of this elusive phase was confirmed by comparison of XRD data from these experiments with those from laboratory simulation of MSH at WES. It was not detectable in any samples at earlier times. XRD peaks for MSH were much more prominent in paste near the surface of the tested cylinder than were CSH peaks. MSH was not detected at the center of the disk.

Semiquantitative chemical data were collected by EDX at three different distances from the perimeter of brine-aged and control disks. Data for the control disk showed no gradient for any element from the edge to the center (same elements as shown in Table 4-2). The disk stored in brine, however, showed several notable differences. They are: 1) sodium is depleted throughout the disk; 2) magnesium is high at the edge, but drops off from the edge to the center; 3) sulfur is higher at the edge but at the control level at the center; 4) chlorine does not change much from the edge to the center, although it is a little higher at the edge; and 5) calcium is roughly 10% lower at the edge than it is at the center.

4.2 Effect of Brine on Mortar Specimens

The mortar cubes stored in brine were coated heavily with white salts. They also showed damage at edges and corners, where the paste had become soft. Some small areas of paste at the edges and corners were missing.

4.2.1 Strength Changes

Strength-change data were analyzed in two ways. First, strengths of brine-exposed cubes were compared with strengths of control cubes at the same ages. These results are expressed as "percent of control." A second type of analysis was performed because the control and brine-storage conditions of salt-containing mixtures differed in a way that might confound the data analysis when expressed as percentage of control. This difference was that control specimens for salt-containing mixtures were stored in water-saturated air, to avoid dissolution and removal of water-soluble phases, while control specimens for non-salt mixtures were stored in saturated-lime water, which is the standard condition for curing mortar or concrete specimens.

This difference would particularly confound comparisons of salt- and non-salt-containing mixtures. If the differences in availability of liquid water between these conditions affects hydration, then the salt-containing control specimens would develop relatively lower strength than the non-salt specimens. This factor would be independent of the presence or absence of salt. Therefore, salt-containing cubes exposed to brine would have a different basis of comparison than non-salt-containing cubes, and could appear to perform relatively better, when in fact the differences were not in the strength of the brine-exposed cubes, but rather in differences in the controls.

In this second analysis, the strength of brine-exposed cubes at 365 days was compared to maximum strength developed by the brine-exposed cubes at any age. These maxima generally occurred at the 30- or 90-day test age. This comparison standardized the availability of liquid water. In this analysis, the results are expressed at "percent of maximum strength."

Table 4-4 compares strength at 365 days expressed as a percentage of maximum strergth to strength expressed as a percentage of control. The two representations of the strength data are highly correlated (correlation coefficient = 0.87, 10 d.f.), indicating that the differences in curing conditions of the salt and non-salt controls was not large enough to affect results significantly.

Table 4-4. Strength of Mortars at 1 yr, Expressed as Percentage of Maximum Strength Attained and as Percentage of Strength of Control

Mix	Salt Y=yes N=no	Strength, % Max	Strength, % Control
M-1	N	72.3	53.3
M-2	Y	84.8	44.3
M-3	N	26.3	13.6
M-4	Y	60.1	43.7
M-5	N	46.2	34.4
M-6	Y	41.0	29.5
M-7	N	34.6	21.0
M-8	Y	60.1	41.2
M-9	N	50.3	39.9
M-10	Y	53.0	41.7
M-11	N	57.9	46.3

In general, all mixtures lose strength when aged in brine. Strengths ranged from 14 to 53 percent of control at 365 days (Table 4-5). Time-dependent data for mixtures M-6 and M-7 are illustrated in Figures 4-11 and 4-12. Differences among most mixtures was small, but two mixtures appeared to be exceptional. These are mixtures M-3 and M-7. Both of these mixtures contain plaster as an expansive admixture but do not contain salt. They differ only in the class of fly ash used. Not only was the 365-day strength reduction greater than the other mixtures, but the reduction appeared at earlier ages, particularly with M-3. This mixture contained Class C fly ash.

Data from calculations of strength reduction imply a uniform loss of strength throughout each mortar cube. Examination of pieces of some of the cubes after they were broken showed a markedly zoned pattern of deterioration. Conspicuous deterioration was restricted to the first few millimetres inward from the surface of the cube. The remaining inner core was not obviously deteriorated. Figure 4-13 shows this zoning.

Table 4-5. Summary of Strength and Mass Changes of Mortars

			Strength				Mass Change	
Mix	Salt Y=yes N=no	Nominal Age (days)	Control	Brine	% Control	Std. Dev. % Control	Control	Brine
M-1	N	30	7106	6480	91.2	9.1	0.23	0.96
		90	7455	6090	81.7	8.3	0.52	1.42
		180	8301	6064	73.0	7.1	0.74	2.31
		365	8785	4682	53.3	6.1	2.02	9.35
M-2	Y	30	2782	2315	83.2	22.3	0.27	1.10
		90	3039	2381	78.4	19.9	0.70	1.61
		180	4125	1888	45.8	12.7	1.37	3.21
		365	4561	2019	44.3	11.4	2.59	9.63
M-3	N	30	5087	5311	104.4	33.8	1.19	1.84
		90	7405	3159	42.7	17.5	1.64	3.85
		180	9374	892	9.5	12.8	1.86	9.69
		365	10226	1397	13.6	11.8	10.19	14.52
M-4	Y	30	5216	4594	88.1	12.2	0.67	2.21
		90	6005	5428	90.4	10.7	1.12	0.77
		180	6830	4922	72.1	8.6	1.17	-0.07
		365	7462	3262	43.7	7.0	2.16	7.38
M-5	N	30	7638	6652	87.1	12.8	0.43	1.23
		90	7259	5930	81.7	13.1	0.79	0.48
		180	8922	5416	60.7	9.6	0.98	0.02
		365	8929	3072	34.4	8.7	2.16	7.06
M-6	Y	30	3245	2712	83.6	19.2	0.02	0.31
		90	4122	2799	67.9	14.0	0.03	0.75
		180	4241	3235	76.3	14.1	0.54	0.64
		365	5495	1623	29.5	9.1	2.90	10.64
M-7	N	30	6415	5779	90.1	10.0	0.30	0.46
		90	8683	7640	88.0	7.3	0.70	1.28
		180	9938	3808	38.3	5.1	1.09	5.11
		365	12598	2647	21.0	3.9	2.61	12.85
M-8	Y	30	4252	4859	114.3	26.3	0.13	0.41
		90	6618	5463	82.6	14.4	0.53	1.16
		180	6628	3842	58.0	12.8	1.10	0.97
		365	7977	3287	41.2	1.0	2.30	10.33
M-9	N	30	5942	6426	108.1	18.2	0.19	-0.03
		90	7178	5774	80.4	13.2	0.40	0.75
		180	8528	5062	59.4	10.0	0.43	2.49
		365	8093	3232	39.9	9.8	1.64	12.64
M-10	Y	30	7056	5559	78.8	13.3	-0.02	0.35
		90	8252	6701	81.2	11.5	-0.07	1.30
		180	8290	5410	65.3	10.6	0.48	1.12
		365	8512	3548	41.7	9.4	2.11	9.79
M-11	N	30	8697	7404	85.1	11.1	0.29	1.89
		90	8318	7748	93.1	13.9	0.59	1.58
		180	10297	6790	65.9	8.6	1.01	0.58
		365	10415	4820	46.3	7.8	2.08	9.49

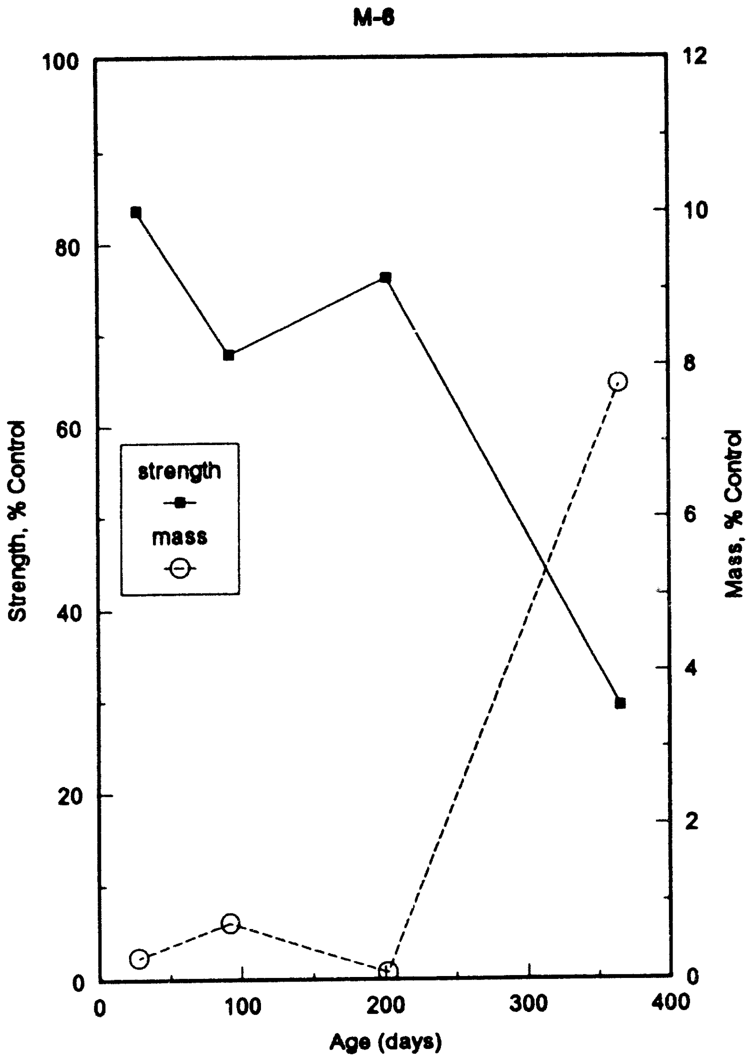

Figure 4-11. Changes in mass and strength of M-6 with age.

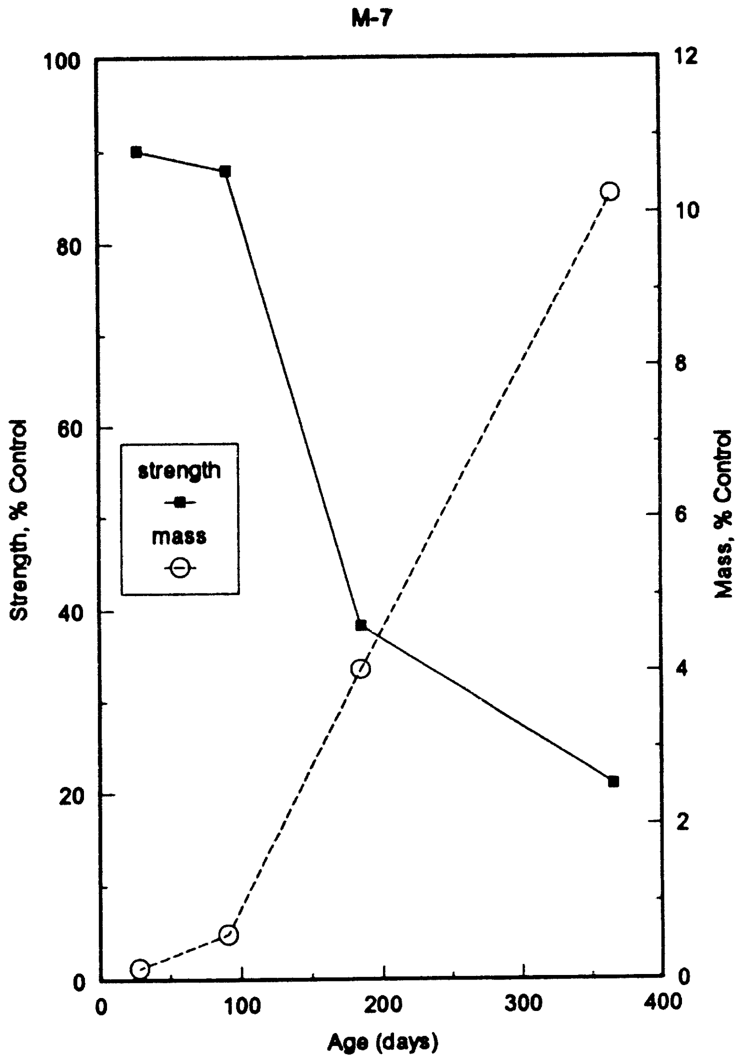

Figure 4-12. Changes in mass and strength of M-7 with age.

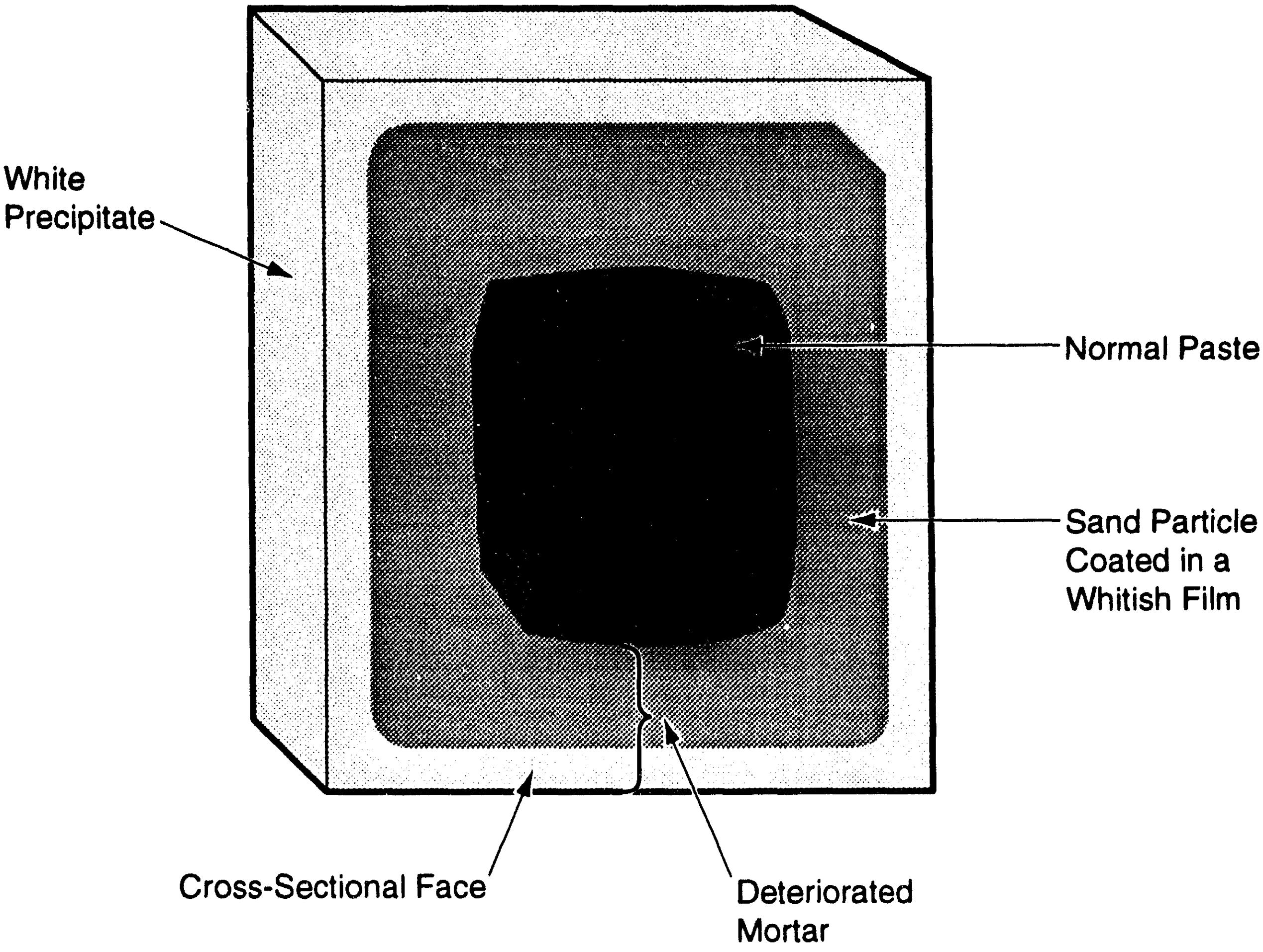

Figure 4-13. Schematic of zoned deterioration of mortar cubes.

As described by ASTM C 109, the calculation of compressive strength uses the total end area of the cube, assuming a uniform strength for the entire sample. In this case, brine exposure caused differences in strength from very low values at the perimeter to higher values in the interior, corresponding to the zoning pattern in Figure 4-13. We were not able to measure this range of strength directly. Consequently, the measured strength of brine-exposed cubes probably reflected more a reduction in cross section of the higher-strength portion of the specimen than a homogeneous loss of strength.

This can be expressed as:

$$S_{\%} = \frac{S_{brine}}{S_{control}} \times 100 = \frac{(L - 2D)^2}{L^2}$$

Where

$S_{\%}$ = strength expressed as percentage of strength of a control specimen
S_{brine} = strength of sample exposed to brine
$S_{control}$ = strength of sample from control condition
L = length of cube edge
D = depth of deterioration

Assuming this model to be correct, the depth of deterioration can be estimated at a given time by calculating the loss of cross sectional area required to give the measured strength reduction. By rearranging the equation above, the depth of deterioration of a cube was calculated from:

$$D = \frac{L}{2}\left(1 - \sqrt{\frac{S_{\%}}{100}}\right)$$

Rates of deterioration are taken from the slopes of linear regression equations relating estimates of depth of deterioration to time of brine exposure. These are summarized in Table 4-6. These estimates of rate reflect a 1-year exposure and are in units of millimetres per day. Figure 4-14 shows this trend for mixtures 6 and 7. A linear model appears to fit most of the data well for 9 of the 11 mixtures. Results for two mixtures (M-2 and M-3) suggest a non-linear pattern. These mixtures both showed higher rates of deterioration at early ages followed by a slowing trend at later ages. Given the small size of data sets and the indirect way in which depths of deterioration were calculated, it seems appropriate to apply only a simple linear model to these data, with no specific assumptions about mechanism. Non-salt mixtures M-3 and M-7 showed higher estimated rates of

Table 4-6. Estimates of Rate of Deterioration Calculated from Strength-Change Data

Mixture	Deterioration Rate (mm/day)	Standard Error (2 df)	Linear Correlation Coefficient (r)
M-1	0.017	0.001	0.99
M-2	0.020	0.008	0.87
M-3	0.043	0.025	0.78
M-4	0.023	0.003	0.98
M-5	0.027	0.002	0.99
M-6	0.027	0.008	0.96
M-7	0.040	0.009	0.95
M-8	0.027	0.007	0.97
M-9	0.027	0.005	0.98
M-10	0.020	0.003	0.96
M-11	0.021	0.005	0.95

deterioration than did other mixtures ($\bar{X}$=0.041 mm/day for M-3 and M-7 compared to $\bar{X}$ =0.023 mm/day for other mixtures). This difference was significant (t=6.25, P=0.002, 9 d.f.). No other variables were significant in analysis by Student's t-Test, $P \leq 0.05$.

4.2.2 Mass Changes

Data from measurements of changes in mass are summarized in Table 4-5. Time-dependent changes for mixtures M-6 and M-7 are illustrated in Figures 4-11 and 4-12. These data show mass increasing and strength decreasing for these mortars as a function of time.

We had elected to document changes in mass, since we expected mass loss to accompany deterioration as the paste softened and portions of the sample were detached. Yet all mixtures showed measurable mass gain when exposed to brine for 365 days. Mass gain for most brine-exposed mixtures was less than about 2% for the first 180 days of exposure, with some mixtures showing a slight mass loss to this age (≤1%). Two of the three mixtures that contained expansive components but not salt, M-3 and M-7, deviated from this pattern in that mass gain was obvious by 180 days. All mixtures showed much larger mass gains between 180 days and 365 days, ranging from 7% to 14%.

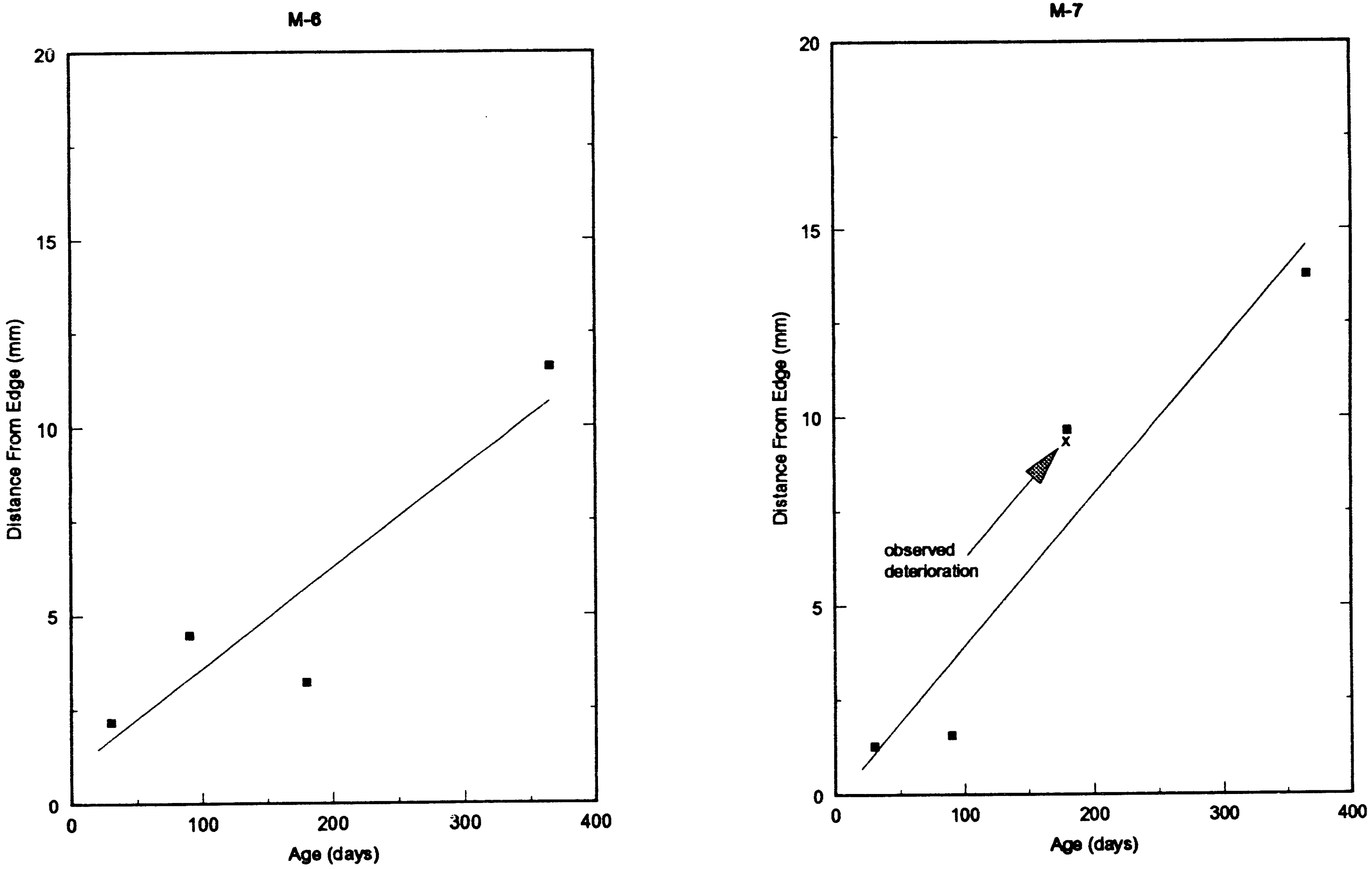

Figure 4-14. Regression curves for rate of deterioration calculated for M-6 and M-7.

Controls also gained mass. This was largely negligible through 180 days, but increased to 2 to 3% by 365 days, except for mixture M-3, which gained 10% after 365 days exposure.

4.2.3 Analysis of M-6 Mortar after a Year in Brine

Crystalline phases were identified by XRD on the surface and from within a brine-exposed and a control cube of M-6 after about a year. The results show that the paste near the surface and paste from the center of the control cube have similar total phase assemblages, but differ largely in abundance of key phases. For example, calcium hydroxide (CH) is more abundant near the center of the brine-aged cube than it is at the edge, were there is only a trace. MSH was present within the first 3 or 4 mm of the cube, apparently having formed by alteration of previously existing CSH. The XRD pattern from the paste portion of mortar near the center showed that the peaks of CSH were present and were not accompanied by MSH. A trace of $Mg(OH)_2$ (MH or brucite) was present near the surface of the cube, but absent in the center.

The white precipitate on M-6 cubes consisted of gypsum, halite, and two different magnesium-hydroxy-chloride hydrate phases -- $Mg_2(OH)_3Cl.4H_2O$ and $Mg_3(OH)_5Cl.4H_2O$. The insoluble material that was left after removal of water-soluble phases was brucite.

Figure 4-15 is an SEM micrograph showing the surface growth that began forming soon after the cubes were placed in brine. The micrograph shows rod-like crystals of magnesium hydroxychloride hydrate and crystals of gypsum. The identification of these crystals was confirmed by XRD. Halite was also identified by XRD. The abundance of precipitate increased on the surface as the amount of Mg/L of brine increased.

Figures 4-16 and 4-17 are SEM micrographs showing the paste within the first millimetre of the exposed surface of a cube aged in brine, and a control cube stored in air, respectively. Figure 4-16 shows paste with numerous microcracks, an open microstructure without massive crystals, and small gypsum crystals scattered throughout the paste and on surfaces exposed by removal of aggregate when the specimen was broken for analysis. EDX analysis of the crystals circled in Figure 4-16 showed that calcium and sulfur are the only elements present, confirming the identification of gypsum (the technique does not detect oxygen or hydrogen). CH was not visually identified. Figure 4-17, at the same magnification, shows a more dense paste with virtually no microcracks in the outer rim of the control cube after a year. The paste includes large (100 μm) CH crystals and no detecta' le gypsum.

Figure 4-15. SEM micrograph of precipitate on cube surface.

Figure 4-16. SEM micrograph of deteriorated mortar from near edge of M-6 after 1 yr in brine.

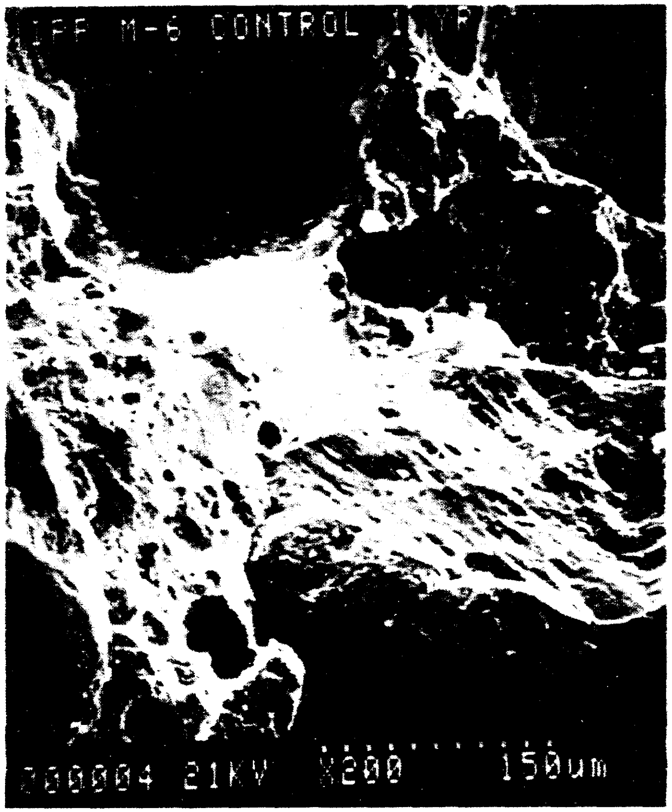

Figure 4-17. SEM micrograph of dense mortar from near edge of M-6 control.

SEM micrographs of the mortar at 4 to 5 mm from the edges of cubes from the same two conditions again showed a much more open microstructure for the brine-stored sample, although it was not fractured as extensively as the rim. Large platy CH crystals are abundant in the control sample. CH is present at this depth in the cube stored in brine, but it is far less abundant than it is at the same depth in the control cube. Figure 4-18 shows the interface between the cement paste and an aggregate particle in this region. Gypsum crystals are visible at this interface, where it is apparent that the CH has been removed, leaving a space partly filled with gypsum and weakening the paste-aggregate bond.

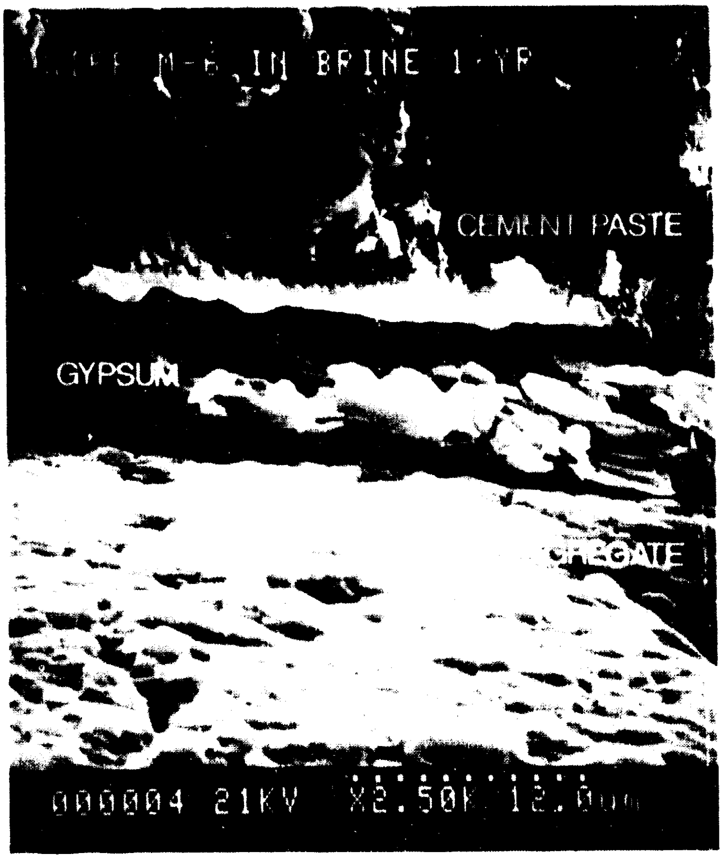

Figure 4-18. SEM micrograph showing partial removal of CH at paste-aggregate interface.

Figures 4-19 and 4-20 are SEM micrographs of the center area of a cube stored in brine and a control cube, respectively. Here, the cementitious matrix of the mortar aged in brine is much denser than was the matrix of the same cube nearer to its surface. In contrast to the outer few millimetres of the cube, calcium hydroxide is visible. There is minimal difference in observed features between the brine-aged mortar and the control at the centers of cubes.

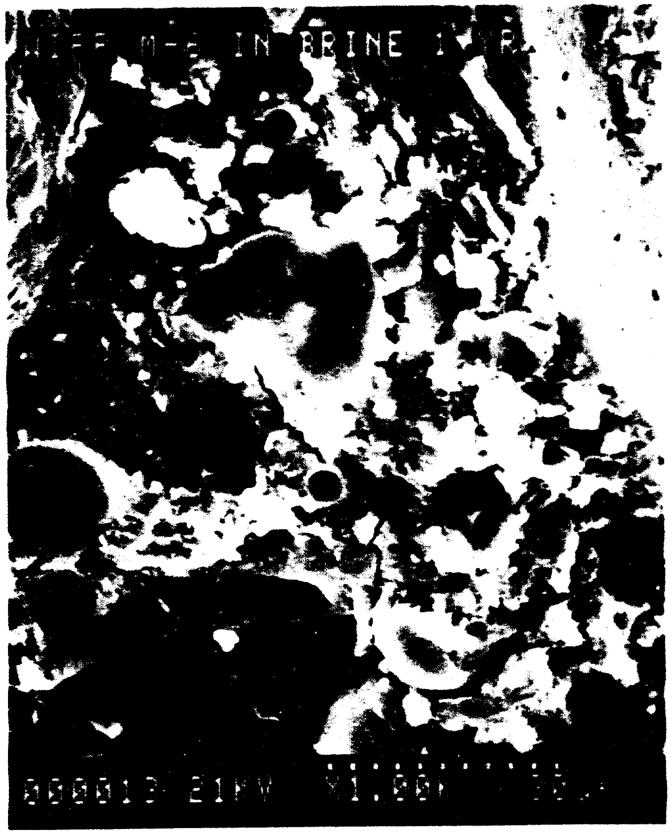

Figure 4-19. SEM micrograph of non-deteriorated mortar from center of M-6 after 1yr in brine.

Figure 4-20. SEM micrograph of mortar from center of M-6 control.

Figure 4-21 shows the white film that separated paste from aggregate in the area 5 to 15 mm from the outer surface of a brine-exposed cube, as seen by SEM. EDX analysis in the area circled showed that the film consisted mainly of gypsum, and also included a $CaCl_2$ phase.

Figure 4-21. SEM micrograph of gypsum coating on quartz particle in deteriorated mortar.

We also prepared petrographic thin sections from the cubes aged in both conditions. Within 3 mm of the surface of the brine-aged cube, there is no calcium hydroxide (CH) at aggregate-paste interfaces. Interfaces near the center of the brine-aged cube showed abundant CH, as did interfaces throughout the control. CH is the usual phase at such interfaces in normal portland cement mortar or concrete.

5.0 DISCUSSION

Exposure to high-magnesium brine caused deterioration in all mixtures. Mortars deteriorated more rapidly than did pastes. The one variable of paste composition that appeared to exert the strongest control on rate of deterioration of expansive mortars was presence or absence of salt as a mixture component. The two mixtures containing expansive admixture but no salt deteriorated more rapidly than did the companion mixtures formulated with salt. Estimated rates of deterioration for these non-salt mixtures were about 0.04 mm/day.

The slower rate of deterioration in salt-containing mortars, between 0.015 and 0.03 mm/day, may be attributable to the tendency of chloride ions to predominate over sulfate ions in reactions with aluminates in the paste. In the case of a mixture containing an expansive component but without salt, the composition is dominated by sulfate with minimal chloride. A plausible explanation for this observation is that expansive calcium sulfoaluminates continue forming after the matrix becomes rigid. Abundant calcium sulfoaluminate gives an open microstructure to the matrix. As it continues to form in the rigid matrix of set cement, this phase causes microfracturing. Thus the set mortars have an open microstructure which is likely to be microfractured. Such a matrix is at risk for deterioration reactions, such as Mg attack on hydrated paste.

When high concentrations of Cl are present, the calcium aluminates in the paste tend to form calcium chloroaluminate (CCA) preferentially, instead of calcium sulfoaluminates (Mather, 1966). CCA tends to persist even when high concentrations of sulfate are encountered subsequently. Calcium chloroaluminate is only slightly expansive and the resulting microstructure is less open and less likely to be extensively fractured. Mixtures that contain salt initially also form CCA early in hydration. This is even less likely to disrupt the microstructure than would be the case if the CCA formed later by reaction with chloride from an external source. Thus the presence of salt changes the microstructure of the paste, apparently making it less susceptible to diffusion reactions. These findings support the selection of a salt-containing concrete for increased durability in panel seals.

The study showed that pastes deteriorate at a slower rate than mortars because of changes that occur in mortars at paste-aggregate interfaces. Loss of calcium from brine-aged mortars is the chemical change with the worst implications for structural performance. One of the two most important calcium-bearing phases, calcium hydroxide (CH), was completely removed from areas in contact with brine. This occurred relatively soon after exposure to brine. CH hydroxide contributes significant strength properties to mortar, by forming a bonding layer between paste and aggregates (Bentz et al., 1992; Diamond, 1987).

Deterioration of calcium silicate hydrate, the principal strength-giving phase of normal portland cement paste, became evident much later, after months of brine exposure. In our previous work (Poole et al., 1994), evidence for destruction of CSH was found in loss of more calcium from specimens than could be accounted for by total destruction of calcium hydroxide, and loss of strength occurred without formation of MSH. In the present study, additional evidence for loss of CSH was the appearance of magnesium silicate hydrate (MSH), a non-cementitious phase that forms at the expense of CSH.

MSH is not easily identified in data from common analytical techniques. XRD patterns of MSH do not exhibit the well-defined peaks of well-crystallized phases. Sometime between 6 months and a year of brine exposure, MSH formed in mortars in an amount that was detectable by XRD. This relatively late appearance of MSH is consistent with work reported by Bonen and Cohen (1992). As in our previous work (Poole et al., 1994) measurable strength loss occurred well before the detectable appearance of MSH, and deterioration is attributed to loss of calcium from hydrated cement phases, rather than to the formation of MSH.

Even after a year of exposure to brine, the deterioration of pastes as measured by calcium loss was limited to the outer few millimetres of each cylinder. Mortars had deteriorated to a depth of about 15 mm in the same period. The pastes and mortars were proportioned with the same ratio of water to cementitious materials, so different initial densities of microstructure should not have been a factor.

The principal difference between the mortar specimens and paste specimens is the presence of aggregates in the former. with the accompanying paste-aggregate interface zones. The paste-aggregate interface is chemically and microstructurally distinct from the paste farther from aggregate particles. In most concretes, this interface is very rich in calcium hydroxide. Given that the initial deterioration mechanism appears to be the removal of CH, interfaces would deteriorate much faster than would other regions of the sample.

In a concrete, loss of CH at paste-aggregate interfaces would cause loss of strength, as the connection between paste and aggregate is destroyed. It also opens the material to brine movement and thus exposes even more of the total mass to reaction with brine. Paste alone is not a practical structural material. It cannot be placed in a large volume because of its heat output and resultant thermal stresses and cracking on cooling. Aggregate interfaces will be present in a concrete, and will be susceptible to attack if brine is present. Because the required durability of concrete in this service environment largely is resistance to chemical attack, formulating concrete for enhanced chemical resistance is strongly recommended. This can be accomplished by using salt-saturated concrete, to minimize chemical gradients between the host rock and the seal components; and by using aggregates that are less susceptible to chemical interaction with magnesium-bearing brine. Other steps to improve the chemical resistance of seal-component concrete are discussed elsewhere (Wakeley et al., 1993).

The EDX data show evidence of S and Cl moving from brine into the pastes (Table 4-2). The film of gypsum crystals and $CaCl_2$ at the interface with aggregate particles in M-6 mortar after a year in brine is a secondary deposit. These phases are not present as aggregate coatings in the relatively unaltered mortar in the center of the cube, even though the mixture included plaster (10% by mass). It appears that these phases formed in the interface voids left by removal of CH. They give no strength to the system, and appeared to be accompanied by enough void space that they are unlikely to restrict further movement of brine into mortar. While mass loss by deterioration of cubes was expected, measurements showed apparent mass gain, because of increased movement of brine into the samples as calcium was removed.

The cementitious mixtures used in this work were complex in that they included both cement and fly ash, and several additional components. The principal mechanism of deterioration appears to involve the products of hydrated portland cement. It is not obvious how the other components affected the nature or rate of reactions. Thus the study does not reveal whether deterioration was due simply to the effect of Mg ion on the hydrated calcium silicate and calcium hydroxide phases, or whether there were more complex interactions involved. For example, the presence of high concentrations of sulfates and chlorides in the brine, and aluminates in the pastes created a scenario in which calcium sulfoaluminates or calcium chloroaluminates or both were formed. The chloroaluminates appear to favor resistance to chemical attack, even in systems that include an expansive (sulfoaluminate) component. The study does reveal that there are both independent and interactive effects of the major constituents of the brine on hydrated cement paste and paste/aggregate interfaces, and that the presence of these interfaces makes the system more susceptible to deterioration.

6.0 CONCLUSIONS

High-magnesium brine caused deterioration in all mixtures studied. Mortars deteriorated more rapidly than pastes. The initial mechanism of strength loss is loss of calcium from calcium hydroxide, which forms the interfacial layer between paste and aggregate in mortars or concretes. Loss of calcium is a self-sustaining reaction, in that it leaves a more open microstructure and makes the system continuously more susceptible to brine ingress and further reaction.

Deterioration is exacerbated by higher magnesium content of the brine. The principal deterioration mechanism is not one of formation of weak magnesium-bearing phases at the expense of calcium-bearing phases, as has been asserted in other studies. In this system, the loss of calcium and loss of strength occurs first, and formation of magnesium silicate hydrate occurs much later, after the paste is weak and open.

The presence of salt in mortars or pastes with an expansive component slows the rate of deterioration. The large percentages of strength loss measured for cube specimens in this study do not indicate such extreme loss of strength for a mass of concrete. Strength loss occurred in an outer reaction zone of each specimen, leaving a core of relatively strong and unaltered material in the center, even after a year immersed in very aggressive brine. This indicates that a large concrete member may experience deterioration only in a relatively thin outside layer, leaving the bulk of a mass placement intact. Also, the experiments in this study were carried out in extreme conditions, with very high concentrations of magnesium and a large volume of brine relative to the volume of mortar or paste. A mass concrete seal component at the WIPP probably would encounter only a limited supply of brine. Even though magnesium concentration could be very high, total deterioration might be small relative to the large mass of the concrete.

The current candidate concrete for WIPP seals includes salt as an initial component, and has a relatively closed initial microstructure. Both of these features contribute to its suitability for use in large placements within the Salado Formation.

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APPENDIX A: COMPOSITION AND PROPERTIES OF COMPONENT MATERIALS

Table A-1. Class H Oilwell Cement, HAN-1 C-1, Tested 1/25/91

Chemical Properties		Physical Properties	
Property	Analysis (%)	Property	Analysis
SiO_2	22.3	Surface Area (Air Permeability)	264 m^2/kg
Al_2O_3	4.4	Autoclave Expansion	0.05 %
Fe_2O_3	3.6	Initial Set (Gillmore)	245 min
CaO	61.6	Final Set (Gillmore)	360 min
MgO	3.9	Air Content	8 %
SO_3	2.2	3-day Strength	1760 psi
Loss on Ignition	0.7	7-day Strength	2460 psi
Insoluble Residue	0.33	3-day Heat of Hydration	56 cal/g
Na_2O	0.21	7-day Heat of Hydration	65 cal/g
K_2O	0.54	Density	3.22 Mg/m^3
C_3A	6		
C_3S	38		
C_2S	35		
C_4AF	11		

Table A-2. Type K Expansive Cement, WIPP-1, RC-1, Tested 6 Aug 90

Chemical Properties		Physical Properties	
Property	Analysis (%)	Property	Analysis
SiO_2	17.6	Surface Area (Blaine)	557
Al_2O_3	4.9	Air Content	4%
Fe_2O_3	2.6	Time of Setting (Vicat)	120 min
CaO	59.6	Density	3.03
MgO	4.3	7-day Strength	2954 psi
SO_3	2.4	28-day Strength	5190 psi
LOI	3.2	7-day Length Change	0.050 %
ISR	0.25	28-day, % 7 day	112.7 %
Na_2O	0.16	3-day Heat of Hydration	72 cal/g
K_2O	0.75	7-day Heat of Hydration	75 cal/g

Table A-3. Class C Fly Ash, WIPP-4 FA-1, Tested 3/28/91

Chemical Properties		Physical Properties	
Property	Analysis (%)	Property	Analysis
SiO_2	29.9	Fineness, 45-μm Sieve	17 % ret.
Al_2O_3	18.3	Water Requirement	96 %
Fe_2O_3	5.2	Strength Activity Index	112 %
CaO	16.7	Density	2.66 Mg/m^3
MgO	6.2		
SO_3	2.3		
LOI	0.3		

Table A-4. Class F Fly Ash, WIPP-4 FA-2, Tested 3/26/91

Chemical Properties

Property	Analysis (%)
SiO_2	55.0
Al_2O_3	20.3
Fe_2O_3	6.2
CaO	8.4
MgO	2.0
SO_3	0.6
LOI	0.8

Table A-5. Cal Seal, Halliburton

Property	Analysis
Density	2.74 mg/m^3
CaO	29.4 %
SO_3	34.0 %
Free Water	0.1 %
Combined Water	19.5 %

APPENDIX B: METHODS OF SAMPLE PREPARATION AND PETROGRAPHIC EXAMINATION OF MORTARS AND PASTES AFTER ONE YEAR IN BRINE

APPENDIX B: METHODS OF SAMPLE PREPARATION AND PETROGRAPHIC EXAMINATION OF MORTARS AND PASTES AFTER ONE YEAR IN BRINE

For X-ray diffraction analysis, paste concentrates were prepared by gently crushing the material in a mortar and pestle. The crushed material was then kneaded to remove the hydrated paste from fine aggregate grains. The pastes were sieved through a 45-μm (No. 325) sieve and then examined by XRD as a tightly packed powder, using nickel-filtered copper radiation, with a Philips APD 3600 diffractometer. Six XRD samples were examined; four samples were from the cube stored in brine, and two from the control cube. The four samples taken from the cube stored in brine came from the white precipitate on the surface before it was washed with water, the white precipitant after it was washed in water, paste material from the outer 4-mm of the cube and paste material from the center of the cube. The two samples from the control cube came from paste in the outer 4-mm, and from paste material in the center.

For scanning electron microscopy (SEM) and its attached energy dispersive X-ray (EDX) analysis, fresh fracture surfaces were prepared from a cube that was stored in brine, and from a control cube. Each was broken so that a surface plane that extending from the center of a cube face through the center of the cube could be examined. This plane made it possible to follow the effect of brine penetration on the microstructure of the paste, and the paste-aggregate interface could be studied. Specific areas examined were: (1) The white precipitate on the outer edge of cubes stored in brine. (2) The outer surface (within the first millimetre of a cube) that was stored in brine. (3) The paste and paste-aggregate interface within the first 4-mm of a cube stored in brine and a cube stored in air (control). (4) The paste within the center of a cube stored in brine and a cube stored in air (control).

Two thin sections were made. One came from a cube stored in brine, and the other from a control cube stored in air. The sections were made from freshly broken surfaces. The sections came from a plane as near as possible to a surface plane starting at the center of a cube face, passing through the center of the cube and exiting through the center of the opposite cube face. The sections were examined with a polarizing microscope.

Disks were cut from paste cylinders using an Isomet saw lubricated with light oil. First a 5 mm-thick disc was cut to remove any deteriorated material from the end of the cylinder, then a 3 mm-thick disc was cut for XRD analysis. Disks were ground to pass a 45-μm sieve and examined as tightly packed powders, as described for mortars, above.

For EDX analysis, a second disk was cut from the cylinder and broken so that the fracture plane crossed the center of the disk. One side was ground smooth using a 5-μm grit and methanol as a lubricant. The grit was washed from the surface with methanol. Three EDX spectra were collected at each specified distance from the exposed edge of the specimen (these were usually 1, 3, and 10 mm). A sample of USGS rock standard GSP-1 (Flanagan, 1967) was used as an EDX standard. Data were not normalized to 100%, therefore, the appearance or disappearance of a particular species did not affect the reported composition of any other species. Elemental analysis is reported by the oxide convention.

APPENDIX C: MEAN OXIDE ANALYSES FOR PASTES AT THREE DEPTHS FROM EXPOSED SURFACE AT SEVERAL AGES

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                      MEAN OXIDE ANALYSIS AT 1, 3, AND 10 MM
                                           10:04 Friday, April 23, 1993

------------------------- MIX=M-1 AGE=91 SOLN=BRINE -------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2    SO3     CL   K2O    CAO  TIO2  FE2O3

  1     1   2.39  12.27  2.61   16.79   2.11  15.63  0.93  45.33  0.04  2.56
  2     3   2.43   7.29  3.02   17.37   1.81   7.73  0.37  57.74  0.36  2.84
  3    10   2.42   6.40  3.07   18.42   1.82   5.65  0.53  58.03  0.38  2.91

------------------------ MIX=M-1 AGE=91 SOLN=CONTROL ------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2    SO3     CL   K2O    CAO  TIO2  FE2O3

  4     1   1.24   5.35  4.23   19.91   1.40      .  0.15  54.41  0.82  3.12
  5     3   1.72   5.23  4.18   19.94   1.42      .  0.24  54.82  0.81  3.22
  6    10   1.63   5.12  4.32   19.72   1.35      .  0.34  54.56  0.75  3.16

------------------------- MIX=M-1 AGE=220 SOLN=BRINE ------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2    SO3     CL   K2O    CAO  TIO2  FE2O3

  7     1   0.80   4.37  2.80   18.20   1.69  11.12  0.38  44.85  0.11  3.06
  8     3   0.00   2.95  2.07   15.19   1.15   7.03  0.22  48.97  0.86  3.20
  9    10   0.11   3.18  2.39   16.09   1.12   4.89  0.20  51.54  0.04  2.52

------------------------ MIX=M-1 AGE=220 SOLN=CONTROL -----------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2    SO3     CL   K2O    CAO  TIO2  FE2O3

 10     1   0.02   3.36  2.89   17.45   1.10   0.02  0.03  53.14  0.19  2.58
 11     3   0.08   3.64  2.99   16.96   1.12   0.02  0.08  52.81  0.12  2.69
 12    10   0.16   3.26  2.88   17.29   1.08   0.02  0.10  52.76  0.28  2.60

------------------------- MIX=M-1A AGE=28 SOLN=BRINE ------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2    SO3     CL   K2O    CAO  TIO2  FE2O3

 49     1   0.79   4.88  2.33   14.40   1.47   8.15  0.22  41.93  0.27  2.23
 50     3   0.25   4.68  2.51   15.19   1.36   3.95  0.23  45.43  0.41  2.33
 51    10   0.00   4.75  2.34   14.63   1.22   1.72  0.09  44.08  0.30  2.11

------------------------ MIX=M-1A AGE=118 SOLN=BRINE ------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2    SO3     CL   K2O    CAO  TIO2  FE2O3

 52     1   0.00   8.44  3.07   17.53   1.53  10.84  0.42  45.09  0.20  2.81
 53     3   0.00   4.81  2.85   16.19   1.12   5.05  0.24  52.26  0.27  2.70
 54    10   0.04   5.31  2.99   16.87   1.15   3.65  0.31  52.60  0.34  2.67

------------------------ MIX=M-1A AGE=180 SOLN=BRINE ------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2    SO3     CL   K2O    CAO  TIO2  FE2O3

 55     1   0.00   7.51  2.87   17.64   1.69  10.15  0.35  46.31  0.17  2.80
 56     3   0.10   4.33  2.57   15.57   1.23   5.77  0.22  52.50  0.38  2.48
 57    10   0.00   5.37  2.82   16.46   1.24   4.51  0.29  52.37  0.32  2.47
```

```
------------------------ MIX=M-1AR AGE=28 SOLN=BRINE ------------------------

OBS  DEPTH  NA2O    MGO   AL2O3   SIO2     SO3     CL    K2O    CAO   TIO2   FE2O3

 58     1   0.14   4.76   1.99   12.25    1.16   5.98   0.13   45.11   0.34   3.63
 59     3   0.15   3.65   2.18   13.31    0.95   3.08   0.17   50.91   0.37   3.75
 60    10   0.07   4.08   2.31   14.44    1.05   1.40   0.09   54.05   0.59   3.95

------------------------- MIX=M-2 AGE=91 SOLN=BRINE -------------------------

OBS  DEPTH  NA2O    MGO   AL2O3   SIO2     SO3     CL    K2O    CAO   TIO2   FE2O3

 61     1   2.19   9.23   3.24   13.09   10.15  13.50   0.92   39.47   0.18   1.79
 62     3   2.31   4.92   3.44   12.09    8.34  11.55   0.50   49.45   0.42   1.97
 63    10   2.22   4.68   3.61   13.06    8.19  10.52   0.49   50.05   0.44   2.02

------------------------ MIX=M-2 AGE=91 SOLN=CONTROL ------------------------

OBS  DEPTH  NA2O    MGO   AL2O3   SIO2     SO3     CL    K2O    CAO   TIO2   FE2O3

 64     1   5.59   3.10   2.87   12.56    7.74   7.23   0.53   44.69   0.57   2.39
 65     3   4.81   3.02   2.98   13.13    8.42   6.60   0.40   46.82   0.65   2.35
 66    10   4.23   3.10   3.75   14.85    8.72   5.68   0.47   48.05   0.59   2.37

------------------------- MIX=M-2 AGE=220 SOLN=BRINE ------------------------

OBS  DEPTH  NA2O    MGO   AL2O3   SIO2     SO3     CL    K2O    CAO   TIO2   FE2O3

 67     1   0.11   3.86   2.45   12.38    9.25   7.68   0.28   38.99   0.21   1.95
 68     3   0.06   2.91   2.59   11.76    7.82   7.93   0.16   41.91   0.12   2.09
 69    10   0.21   2.48   2.46   11.10    7.44   6.95   0.16   43.93   0.18   1.96

------------------------ MIX=M-2 AGE=220 SOLN=CONTROL -----------------------

OBS  DEPTH  NA2O    MGO   AL2O3   SIO2     SO3     CL    K2O    CAO   TIO2   FE2O3

 70     1   1.50   2.50   2.93   12.04    7.51   3.65   0.15   43.97   0.30   1.94
 71     3   1.35   2.38   2.78   11.94    7.69   2.84   0.13   44.06   0.25   1.94
 72    10   1.29   1.98   2.52   12.12    7.27   2.86   0.10   43.62   0.19   1.72

------------------------- MIX=M-2A AGE=28 SOLN=BRINE ------------------------

OBS  DEPTH  NA2O    MGO   AL2O3   SIO2     SO3     CL    K2O    CAO   TIO2   FE2O3

 73     1   0.60   5.37   2.39   11.75    9.26   7.00   0.30   34.74   0.21   1.68
 74     3   0.95   3.62   2.51   11.74    7.93   6.06   0.27   39.33   0.39   1.59
 75    10   1.30   3.93   2.53   11.50    7.66   5.02   0.31   38.33   0.25   1.68

------------------------ MIX=M-2A AGE=118 SOLN=BRINE ------------------------

OBS  DEPTH  NA2O    MGO   AL2O3   SIO2     SO3     CL    K2O    CAO   TIO2   FE2O3

 76     1   0.37   4.95   3.59   13.74    8.89   6.78   0.40   41.33   0.36   2.01
 77     3   0.52   3.24   2.67   11.43    7.04   5.87   0.30   45.86   0.36   1.91
 78    10   0.77   3.89   2.78   11.97    6.97   5.97   0.34   45.36   0.41   2.06
```

```
------------------------- MIX=M-2A AGE=180 SOLN=BRINE -------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2    SO3      CL   K2O    CAO  TIO2  FE2O3

 79      1  0.01   5.76   3.06  13.02   9.07    8.04  0.39  40.99  0.32   2.03
 80      3  0.02   3.52   2.98  12.88   7.33    7.67  0.39  45.34  0.40   1.95
 81     10  0.32   3.99   3.20  12.83   7.02    7.36  0.38  46.15  0.38   2.11

------------------------- MIX=M-2AR AGE=28 SOLN=BRINE -------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2    SO3      CL   K2O    CAO  TIO2  FE2O3

 82      1  0.85   6.38   3.02  14.21  11.44    9.00  0.45  52.91  0.45   2.94
 83      3  1.06   3.95   3.61  14.85  10.84    6.77  0.37  64.63  0.60   3.74
 84     10  1.52   3.45   2.97  13.60   9.10    5.44  0.40  57.60  0.98   3.34

-------------------------- MIX=M-3 AGE=91 SOLN=BRINE --------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2    SO3      CL   K2O    CAO  TIO2  FE2O3

 85      1  1.65   5.66   5.02  16.34   4.83    7.46  0.25  40.12  0.72   2.73
 86      3  1.59   5.30   4.91  15.75   4.50    5.56  0.22  43.34  0.74   2.49
 87     10  1.98   5.36   5.40  16.56   4.71    4.91  0.29  43.17  0.77   2.68

-------------------------- MIX=M-3 AGE=220 SOLN=BRINE -------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2    SO3      CL   K2O    CAO  TIO2  FE2O3

 88      1  0.98   4.82   5.40  16.90   4.51    8.21  0.26  41.42  0.54   2.63
 89      3  1.08   3.28   5.13  14.90   3.79    7.46  0.26  42.92  0.41   2.60
 90     10  0.83   3.63   5.21  16.79   3.88    6.04  0.27  43.91  0.37   2.48

------------------------- MIX=M-3 AGE=220 SOLN=CONTROL ------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2    SO3      CL   K2O    CAO  TIO2  FE2O3

 91      1  0.12   3.89   5.43  18.23   4.59    0.26  0.10  46.38  0.58   2.49
 92      3  0.13   4.02   6.21  17.99   4.80    0.29  0.10  46.02  0.51   2.57
 93     10  0.18   3.96   5.67  18.22   4.46    0.24  0.13  46.39  0.64   2.35

-------------------------- MIX=M-3A AGE=28 SOLN=BRINE -------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2    SO3      CL   K2O    CAO  TIO2  FE2O3

 94      1  0.11   7.08   5.53  16.09   4.73    4.63  0.14  38.72  0.44   2.33
 95      3  0.54   5.45   5.76  16.54   4.41    2.83  0.18  40.07  0.54   2.40
 96     10  0.30   5.87   5.85  17.40   4.33    0.16  0.08  41.64  0.64   2.41

------------------------- MIX=M-3A AGE=118 SOLN=BRINE -------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2    SO3      CL   K2O    CAO  TIO2  FE2O3

 97      1  0.18   6.71   5.88  17.16   3.96    7.17  0.20  42.61  0.65   2.64
 98      3  0.41   5.89   6.09  17.32   3.83    3.74  0.27  46.07  0.80   2.70
 99     10  0.45   5.68   6.25  17.23   3.83    2.28  0.23  45.15  0.99   2.66
```

```
------------------------ MIX=M-3A AGE=180 SOLN=BRINE ------------------------

OBS   DEPTH   NA2O    MGO   AL2O3    SIO2     SO3      CL    K2O     CAO   TIO2   FE2O3

100     1     0.04   7.97   5.20    17.18    4.80    7.85   0.26   41.90   0.68   2.52
101     3     0.36   5.22   9.52    13.01    4.38    3.31   0.24   45.39   0.71   2.37
102    10     0.37   5.41   5.67    17.62    4.01    3.55   0.31   45.60   0.69   2.45

------------------------- MIX=M-4 AGE=91 SOLN=BRINE -------------------------

OBS   DEPTH   NA2O    MGO   AL2O3    SIO2     SO3      CL    K2O     CAO   TIO2   FE2O3

103     1     15.9   7.43   3.86    14.30    1.53   22.29   0.28   30.32   0.20   2.41
104     3     11.5   5.04   4.52    15.18    0.82   16.04   0.37   38.62   0.49   2.65
105    10     10.9   5.94   4.82    16.52    0.82   14.47   0.32   37.60   0.51   2.50

------------------------- MIX=M-4 AGE=220 SOLN=BRINE ------------------------

OBS   DEPTH   NA2O    MGO   AL2O3    SIO2     SO3      CL    K2O     CAO   TIO2   FE2O3

106     1     0.34   4.83   6.43    17.67    2.66    8.03   0.29   40.78   0.60   2.88
107     3     0.17   3.51   6.07    17.63    0.99    7.15   0.26   43.32   0.46   2.58
108    10     0.30   3.57   6.16    18.18    1.08    6.28   0.31   42.86   0.53   2.73

------------------------ MIX=M-4 AGE=220 SOLN=CONTROL -----------------------

OBS   DEPTH   NA2O    MGO   AL2O3    SIO2     SO3      CL    K2O     CAO   TIO2   FE2O3

109     1     1.09   3.62   6.07    18.63    1.15    4.89   0.18   43.14   0.65   2.68
110     3     1.09   3.58   6.17    18.60    1.36    4.95   0.22   43.92   0.51   2.67
111    10     1.05   3.33   5.66    18.80    1.38    4.90   0.24   42.95   0.48   2.76

------------------------- MIX=M-4A AGE=28 SOLN=BRINE ------------------------

OBS   DEPTH   NA2O    MGO   AL2O3    SIO2     SO3      CL    K2O     CAO   TIO2   FE2O3

112     1     0.98   5.47   5.40    16.91    1.33    5.77   0.36   37.08   0.58   2.50
113     3     2.27   5.39   5.21    16.52    1.15    6.33   0.26   37.53   0.57   2.45
114    10     2.71   5.34   5.43    17.19    1.47    6.49   0.22   37.35   0.66   2.47

------------------------ MIX=M-4A AGE=118 SOLN=BRINE ------------------------

OBS   DEPTH   NA2O    MGO   AL2O3    SIO2     SO3      CL    K2O     CAO   TIO2   FE2O3

115     1     0.49   7.59   5.69    18.05    2.16    8.25   0.44   41.35   0.57   2.82
116     3     1.55   5.36   5.85    17.63    0.95    7.27   0.48   43.21   0.70   2.75
117    10     3.71   5.10   5.91    17.53    1.25    7.07   0.37   42.31   0.56   2.70

------------------------ MIX=M-4A AGE=180 SOLN=BRINE ------------------------

OBS   DEPTH   NA2O    MGO   AL2O3    SIO2     SO3      CL    K2O     CAO   TIO2   FE2O3

118     1     0.16   5.53   5.87    18.85    2.26    9.19   0.51   41.70   0.66   2.62
119     3     0.80   5.65   6.07    18.21    1.13    8.06   0.52   44.05   0.71   2.57
120    10     2.34   5.91   6.18    18.35    1.30    6.61   0.45   44.28   0.71   2.45
```

```
------------------------- MIX=M-5 AGE=91 SOLN=BRINE -------------------------

OBS  DEPTH  NA2O   MGO  AL2O3   SIO2    SO3     CL   K2O    CAO  TIO2  FE2O3

121     1   11.4  7.92  6.08   18.45   1.19  16.83  0.40  36.15  0.72  3.04
122     3   3.61  6.68  5.69   18.92   0.94   9.61  0.51  45.92  0.88  3.15
123    10   3.89  7.22  6.14   19.53   1.06   7.76  0.86  44.84  0.90  3.38

------------------------- MIX=M-5 AGE=220 SOLN=BRINE ------------------------

OBS  DEPTH  NA2O   MGO  AL2O3   SIO2    SO3     CL   K2O    CAO  TIO2  FE2O3

124     1   0.26  4.46  6.19   18.48   1.87   5.88  0.25  39.14  0.50  2.79
125     3   0.28  3.93  5.84   17.92   1.32   8.40  0.25  43.83  0.40  2.77
126    10   0.19  3.97  5.77   18.64   1.29   7.16  0.28  44.16  0.51  2.73

------------------------ MIX=M-5 AGE=220 SOLN=CONTROL -----------------------

OBS  DEPTH  NA2O   MGO  AL2O3   SIO2    SO3     CL   K2O    CAO  TIO2  FE2O3

127     1   0.04  4.15  6.26   19.66   1.32   0.25  0.07  45.16  0.81  2.88
128     3   0.07  4.46  6.77   20.16   1.30   0.28  0.07  45.98  0.81  2.78
129    10   0.07  4.34  6.25   20.20   1.33   0.25  0.06  45.85  0.85  3.05

------------------------- MIX=M-5A AGE=28 SOLN=BRINE ------------------------

OBS  DEPTH  NA2O   MGO  AL2O3   SIO2    SO3     CL   K2O    CAO  TIO2  FE2O3

130     1   0.16  5.26  5.23   15.81   1.46   7.89  0.27  37.02  0.52  2.50
131     3   0.59  5.27  5.26   15.98   1.29   6.24  0.39  37.72  0.63  2.39
132    10   0.13  5.69  6.02   18.15   1.33   2.30  0.15  40.27  0.67  2.67

------------------------ MIX=M-5A AGE=118 SOLN=BRINE ------------------------

OBS  DEPTH  NA2O   MGO  AL2O3   SIO2    SO3     CL   K2O    CAO  TIO2  FE2O3

133     1   0.08  5.97  5.78   19.22   1.98   8.34  0.28  44.30  0.60  2.98
134     3   0.58  6.07  6.08   18.82   1.11   6.44  0.32  45.81  0.78  2.89
135    10   0.54  6.14  6.34   19.88   1.27   4.83  0.44  45.91  0.74  2.82

------------------------ MIX=M-5A AGE=180 SOLN=BRINE ------------------------

OBS  DEPTH  NA2O   MGO  AL2O3   SIO2    SO3     CL   K2O    CAO  TIO2  FE2O3

136     1   0.10  5.96  6.82   19.30   1.78   8.59  0.28  43.35  0.63  2.91
137     3   0.33  6.07  6.25   18.60   1.24   7.19  0.32  45.78  0.62  2.67
138    10   0.63  5.74  6.41   19.40   1.30   5.91  0.54  44.88  0.67  2.67

------------------------- MIX=M-6 AGE=91 SOLN=BRINE -------------------------

OBS  DEPTH  NA2O   MGO  AL2O3   SIO2    SO3     CL   K2O    CAO  TIO2  FE2O3

139     1   2.72  5.86  6.17   22.71   4.03   7.71  0.74  35.20  0.54  3.10
140     3   2.79  4.10  6.34   22.94   2.86   6.41  0.61  40.72  0.72  3.08
141    10   3.10  4.02  5.92   22.23   2.78   5.66  0.53  41.31  0.68  2.98
```

```
------------------------ MIX=M-6 AGE=91 SOLN=CONTROL ------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2     SO3     CL   K2O    CAO   TIO2  FE2O3

142    1    4.10   2.72  6.58   23.02    4.21   6.33  0.80  42.64  0.63   2.78
143    3    4.29   2.45  6.13   22.87    4.33   6.32  0.85  43.09  0.73   2.81
144   10    3.67   2.59  6.14   22.15    3.96   5.29  0.66  38.75  0.79   2.79

------------------------- MIX=M-6 AGE=220 SOLN=BRINE ------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2     SO3     CL   K2O    CAO   TIO2  FE2O3

145    1    0.56   5.03  5.53   19.73    4.65   8.64  0.48  36.39  0.35   2.55
146    3    0.20   4.21  5.13   19.16    3.59   7.05  0.36  41.35  0.69   2.72
147   10    0.45   4.39  5.77   20.65    3.30   6.26  0.38  41.21  0.50   2.76

------------------------ MIX=M-6 AGE=220 SOLN=CONTROL -----------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2     SO3     CL   K2O    CAO   TIO2  FE2O3

148    1    2.11   3.57  5.51   21.58    4.01   4.43  0.32  41.26  0.47   2.29
149    3    2.34   3.67  5.59   21.74    3.90   4.60  0.41  41.42  0.38   2.54
150   10    2.67   3.58  5.71   21.57    4.24   4.49  0.36  40.57  0.39   2.35

                               MEANS AT 1, 3, AND 10 MM                     134
                                             10:04 Friday, April 23, 1993

------------------------- MIX=M-6A AGE=28 SOLN=BRINE ------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2     SO3     CL   K2O    CAO   TIO2  FE2O3

151    1    1.20   5.14  5.30   20.06    4.93   8.80  0.45  35.57  0.23   2.41
152    3    2.59   3.25  4.55   18.44    3.65   6.25  0.40  36.57  0.21   2.07
153   10    3.19   2.84  5.17   20.02    3.63   5.39  0.33  35.12  0.21   2.21

------------------------ MIX=M-6A AGE=118 SOLN=BRINE ------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2     SO3     CL   K2O    CAO   TIO2  FE2O3

154    1    0.33   5.32  5.62   21.57    4.94   5.77  0.38  40.14  0.46   2.63
155    3    0.97   3.60  5.70   21.15    3.54   5.00  0.41  41.99  0.46   2.49
156   10    2.54   3.25  5.37   21.15    3.79   4.55  0.49  42.04  0.48   2.45

------------------------ MIX=M-6A AGE=180 SOLN=BRINE ------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2     SO3     CL   K2O    CAO   TIO2  FE2O3

157    1    0.36   3.82  5.43   20.24    4.37   7.78  0.70  39.17  0.50   2.49
158    3    0.79   2.97  5.43   19.47    3.23   6.99  0.68  42.37  0.49   2.51
159   10    2.67   3.41  5.46   19.61    3.88   5.43  0.64  41.16  0.46   2.34

------------------------- MIX=M-7 AGE=91 SOLN=BRINE -------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2     SO3     CL   K2O    CAO   TIO2  FE2O3

160    1    0.95  19.10  5.71   22.92    3.67   9.53  0.79  28.07  0.41   2.88
161    3    1.39   5.15  5.91   23.48    2.69   5.80  0.60  40.87  0.78   3.30
162   10    1.39   2.96  4.93   19.51    2.91   4.19  0.49  41.39  0.68   2.88
```

```
------------------------ MIX=M-7 AGE=220 SOLN=BRINE ------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2     SO3      CL   K2O    CAO   TIO2  FE2O3

163    1    1.23   6.07  6.42   22.43    4.38    8.49  0.47  37.62  0.45  2.56
164    3    1.65   5.24  5.60   19.70    4.11    8.51  0.37  40.86  0.45  2.62
165   10    0.48   4.00  5.92   20.11    4.16    5.80  0.35  40.87  0.79  2.74

----------------------- MIX=M-7 AGE=220 SOLN=CONTROL -----------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2     SO3      CL   K2O    CAO   TIO2  FE2O3

166    1    0.31   4.23  6.25   22.78    4.70    0.13  0.16  44.16  0.41  2.94
167    3    0.09   4.10  6.59   22.98    4.25    0.09  0.18  41.89  0.38  2.70
168   10    0.18   3.75  6.84   24.03    4.42    0.13  0.25  42.05  0.37  2.48

------------------------ MIX=M-7A AGE=28 SOLN=BRINE ------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2     SO3      CL   K2O    CAO   TIO2  FE2O3

169    1    1.27   3.96  4.95   18.55    4.30    7.43  0.37  34.69  0.28  2.35
170    3    0.96   3.85  5.10   19.11    4.14    4.46  0.36  38.05  0.33  2.32
171   10    0.74   3.39  5.13   19.66    4.18    2.65  0.42  37.46  0.36  2.36

----------------------- MIX=M-7A AGE=118 SOLN=BRINE ------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2     SO3      CL   K2O    CAO   TIO2  FE2O3

172    1    0.76   5.45  5.48   21.23    4.39    6.97  0.42  42.02  0.41  2.79
173    3    0.56   3.50  5.15   20.42    3.64    6.31  0.45  45.70  0.55  2.73
174   10    0.68   3.51  5.78   21.53    3.82    4.39  0.62  44.65  0.47  2.78

----------------------- MIX=M-7A AGE=180 SOLN=BRINE ------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2     SO3      CL   K2O    CAO   TIO2  FE2O3

175    1    0.11  15.53  5.69   21.53    4.29    7.60  0.45  35.26  0.51  2.82
176    3    0.61   3.61  5.15   19.35    3.61    6.41  0.36  42.68  0.36  2.34
177   10    0.48   3.50  5.63   20.49    3.54    5.19  0.53  42.06  0.40  2.48

------------------------ MIX=M-8 AGE=91 SOLN=BRINE -------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2     SO3      CL   K2O    CAO   TIO2  FE2O3

178    1    1.77  10.15  6.57   24.44    1.95    9.02  1.04  32.53  0.63  3.34
179    3    4.32   5.69  5.73   22.03    0.86   10.51  0.75  39.56  0.63  2.97
180   10    5.72   3.84  5.84   22.96    0.70   10.24  1.05  37.70  0.56  3.07

----------------------- MIX=M-8 AGE=91 SOLN=CONTROL ------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2     SO3      CL   K2O    CAO   TIO2  FE2O3

181    1    3.49   2.10  7.52   25.23    0.83    6.44  0.74  40.37  0.78  3.06
182    3    3.68   2.23  6.88   23.98    1.04    7.35  0.82  43.27  0.70  3.38
183   10    3.90   2.42  6.99   24.68    1.09    7.28  0.88  42.49  0.72  3.19
```

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------------------------- MIX=M-8 AGE=220 SOLN=BRINE -------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2    SO3     CL   K2O    CAO  TIO2  FE2O3

184     1   0.68   8.56  5.53   22.70   2.70   9.14  0.58  36.82  0.28  2.86
185     3   0.23   3.95  5.53   21.12   1.72   7.80  0.41  40.45  0.37  2.79
186    10   0.63   3.91  6.18   22.94   0.74   6.58  0.52  41.71  0.43  2.78


------------------------ MIX=M-8 AGE=220 SOLN=CONTROL ------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2    SO3     CL   K2O    CAO  TIO2  FE2O3

187     1   3.36   3.89  6.60   24.02   1.05   6.14  0.40  39.83  0.33  2.72
188     3   3.47   3.54  6.59   23.64   1.20   6.29  0.45  40.92  0.41  2.86
189    10   3.29   4.11  6.65   23.83   1.13   5.77  0.48  39.74  0.34  2.74


------------------------- MIX=M-8A AGE=28 SOLN=BRINE -------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2    SO3     CL   K2O    CAO  TIO2  FE2O3

190     1   1.11   4.39  7.15   21.82   1.40   8.35  0.48  36.46  0.41  2.51
191     3   3.05   3.64  7.37   22.76   1.11   5.63  0.39  36.54  0.24  2.50
192    10   3.28   2.94  7.20   22.84   1.16   5.23  0.44  37.18  0.31  2.49


------------------------ MIX=M-8A AGE=118 SOLN=BRINE -------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2    SO3     CL   K2O    CAO  TIO2  FE2O3

193     1   0.34   4.22  5.86   23.10   1.22   7.80  0.51  42.25  0.44  2.78
194     3   1.13   3.90  6.17   24.44   0.95   6.01  0.55  43.03  0.46  2.89
195    10   2.41   3.72  6.40   23.91   1.11   5.26  0.48  42.22  0.53  3.06


------------------------ MIX=M-8A AGE=180 SOLN=BRINE -------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2    SO3     CL   K2O    CAO  TIO2  FE2O3

196     1   0.11  16.91  6.19   23.70   1.65   9.90  0.71  34.19  0.47  2.57
197     3   1.30   3.26  5.59   21.75   0.93   7.15  0.63  41.17  0.51  2.63
198    10   3.28   2.90  6.19   22.47   1.01   6.05  0.57  40.50  0.50  2.67


------------------------- MIX=M-9 AGE=91 SOLN=BRINE --------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2    SO3     CL   K2O    CAO  TIO2  FE2O3

199     1   2.27  11.71  8.03   27.28   2.14   9.10  0.69  35.57  0.18  2.53
200     3   2.31   5.52  7.59   27.30   1.56   6.28  0.55  46.08  0.41  3.24
201    10   2.49   5.33  6.84   25.96   1.69   5.34  0.70  45.79  0.38  2.74


------------------------ MIX=M-9 AGE=91 SOLN=CONTROL -------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2    SO3     CL   K2O    CAO  TIO2  FE2O3

202     1   0.86   2.93  7.93   26.34   1.17     .   0.47  41.55  0.99  3.31
203     3   1.17   3.74  7.58   26.23   1.16     .   0.54  42.55  1.03  3.65
204    10   0.89   3.42  8.63   26.05   1.17     .   0.60  41.26  0.97  3.37
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------------------------- MIX=M-9 AGE=220 SOLN=BRINE -------------------------

OBS  DEPTH  NA2O     MGO  AL2O3   SIO2     SO3     CL    K2O    CAO   TIO2  FE2O3

205     1   0.63    6.58  6.15   22.04    1.61  10.98   0.56  36.70   0.21   2.91
206     3   0.73    6.96  5.78   21.47    1.07  10.27   0.57  41.28   0.29   2.86
207    10   1.24    4.27  6.27   23.21    1.16   6.96   0.61  41.44   0.35   3.00

------------------------ MIX=M-9 AGE=220 SOLN=CONTROL ------------------------

OBS  DEPTH  NA2O     MGO  AL2O3   SIO2     SO3     CL    K2O    CAO   TIO2  FE2O3

208     1   0.30    4.89  7.02   26.23    1.23   0.27   0.25  42.75   0.53   3.14
209     3   0.33    4.59  7.61   26.55    1.18   0.26   0.24  44.13   0.55   3.09
210    10   0.38    4.74  7.23   25.16    1.09   0.17   0.26  43.82   0.55   2.96

------------------------ MIX=M-9A AGE=28 SOLN=BRINE -------------------------

OBS  DEPTH  NA2O     MGO  AL2O3   SIO2     SO3     CL    K2O    CAO   TIO2  FE2O3

211     1   0.73    5.39  6.82   21.93    1.25   7.44   0.47  36.46   0.34   2.63
212     3   0.29    3.87  6.86   22.89    1.10   2.42   0.26  38.63   0.34   2.76
213    10   0.21    3.71  7.35   23.21    1.10   0.41   0.20  38.57   0.34   2.58

------------------------ MIX=M-9A AGE=118 SOLN=BRINE ------------------------

OBS  DEPTH  NA2O     MGO  AL2O3   SIO2     SO3     CL    K2O    CAO   TIO2  FE2O3

214     1   0.36    4.77  6.84   22.71    1.62   7.14   0.41  41.10   0.47   2.85
215     3   0.83    3.85  6.72   22.59    1.00   5.32   0.51  42.70   0.47   3.27
216    10   0.18    4.20  6.81   23.40    0.97   0.71   0.21  46.72   0.49   3.39

------------------------ MIX=M-9A AGE=180 SOLN=BRINE ------------------------

OBS  DEPTH  NA2O     MGO  AL2O3   SIO2     SO3     CL    K2O    CAO   TIO2  FE2O3

217     1   0.27    4.49  5.93   22.74    1.75   6.90   0.40  40.78   0.41   3.22
218     3   0.63    3.74  6.24   23.80    1.02   5.27   0.40  42.83   0.46   2.86
219    10   0.32    4.07  6.55   24.57    1.04   2.61   0.36  43.33   0.61   3.00

------------------------- MIX=M-10 AGE=91 SOLN=BRINE ------------------------

OBS  DEPTH  NA2O     MGO  AL2O3   SIO2     SO3     CL    K2O    CAO   TIO2  FE2O3

 13     1   3.45   10.59  3.89   13.73    4.96  14.97   0.62  41.75   0.15   1.87
 14     3   5.20    7.62  4.10   13.69    3.34  14.03   0.44  53.38   0.31   2.09
 15    10   6.07    6.79  4.13   13.80    3.15  14.40   0.45  51.51   0.23   1.99

------------------------ MIX=M-10 AGE=180 SOLN=BRINE ------------------------

OBS  DEPTH  NA2O     MGO  AL2O3   SIO2     SO3     CL    K2O    CAO   TIO2  FE2O3

 16     1   0.11    7.43  3.90   14.39    3.34  10.08   0.37  44.16   0.42   2.26
 17     3   0.84    5.83  3.52   13.46    2.43   7.38   0.39  49.37   0.43   1.91
 18    10   0.99    5.33  3.73   13.40    2.37   6.35   0.37  46.33   0.36   1.87
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------------------------- MIX=M-10 AGE=220 SOLN=BRINE -------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2     SO3      CL   K2O     CAO  TIO2  FE2O3

 19     1   0.20   7.28  4.31   15.49    3.53   11.14  0.39   43.01  0.20  2.15
 20     3   0.11   5.70  3.72   13.26    2.54    9.12  0.28   47.05  0.19  2.04
 21    10   0.54   5.83  3.80   13.10    2.48    8.31  0.28   47.19  0.19  1.95

------------------------ MIX=M-10 AGE=220 SOLN=CONTROL ------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2     SO3      CL   K2O     CAO  TIO2  FE2O3

 22     1   3.06   5.52  3.76   13.78    3.10    5.25  0.55   47.17  0.19  1.90
 23     3   3.44   5.63  3.79   13.65    3.19    5.13  0.53   46.75  0.13  1.85
 24    10   2.96   5.58  3.69   13.80    3.09    4.89  0.55   47.09  0.13  2.03

------------------------- MIX=M-10A AGE=28 SOLN=BRINE -------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2     SO3      CL   K2O     CAO  TIO2  FE2O3

 25     1   0.47   5.95  3.96   11.83    3.87    7.36  0.29   40.00  0.18  1.67
 26     3   1.25   5.84  3.98   12.31    2.71    5.48  0.31   42.26  0.17  1.71
 27    10   2.15   5.41  3.94   12.84    2.92    5.18  0.30   43.29  0.26  1.69

------------------------- MIX=M-10A AGE=118 SOLN=BRINE ------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2     SO3      CL   K2O     CAO  TIO2  FE2O3

 28     1   0.05   6.67  4.74   14.81    3.87    7.64  0.25   44.86  0.50  2.18
 29     3   0.13   4.81  3.76   12.95    2.81    5.86  0.23   51.09  0.59  2.08
 30    10   0.72   6.20  3.99   13.85    2.60    4.54  0.21   50.88  0.38  2.09

-------------------------- MIX=M-11 AGE=91 SOLN=BRINE -------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2     SO3      CL   K2O     CAO  TIO2  FE2O3

 31     1   3.21  13.73  4.00   13.75    3.89   14.86  1.17   41.13  0.05  1.79
 32     3   2.21   7.11  4.51   15.50    3.91    6.28  0.35   56.33  0.54  2.19
 33    10   2.19   7.75  4.69   15.57    3.88    3.61  0.79   55.19  0.45  2.23

------------------------- MIX=M-11 AGE=180 SOLN=BRINE -------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2     SO3      CL   K2O     CAO  TIO2  FE2O3

 34     1   0.05   7.14  4.06   14.66    3.31    9.81  0.39   44.55  0.43  2.11
 35     3   0.14   6.16  3.89   13.81    3.00    5.26  0.28   50.11  0.42  1.85
 36    10   0.51   5.77  4.12   14.35    3.07    3.67  0.43   50.16  0.40  1.94

------------------------- MIX=M-11 AGE=220 SOLN=BRINE -------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2     SO3      CL   K2O     CAO  TIO2  FE2O3

 37     1   0.39   6.36  4.14   14.62    3.49    9.91  0.30   42.31  0.23  2.17
 38     3   0.30   5.78  3.63   13.24    3.06    7.68  0.31   47.95  0.15  2.05
 39    10   0.54   5.73  3.86   14.02    3.18    5.06  0.40   48.45  0.12  1.95
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----------------------- MIX=M-11 AGE=220 SOLN=CONTROL -----------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2    SO3     CL   K2O    CAO  TIO2  FE2O3

 40      1  0.16   6.59  4.29   14.53   3.16   0.22  0.06  49.35  0.01  2.02
 41      3  0.02   6.11  4.42   15.06   3.41   0.29  0.14  51.29  0.11  2.11
 42     10  0.15   6.61  4.17   14.80   3.44   0.30  0.17  50.67  0.12  1.96

------------------------ MIX=M-11A AGE=28 SOLN=BRINE ------------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2    SO3     CL   K2O    CAO  TIO2  FE2O3

 43      1  0.10   5.88  4.69   12.19   3.22   6.10  0.13  41.95  0.22  1.80
 44      3  0.13   5.70  4.62   12.99   3.21   3.03  0.25  44.55  0.27  1.70
 45     10  0.07   6.21  4.44   12.80   3.11   0.10  0.19  43.77  0.26  1.73

------------------------ MIX=M-11A AGE=118 SOLN=BRINE -----------------------

OBS  DEPTH  NA2O    MGO  AL2O3   SIO2    SO3     CL   K2O    CAO  TIO2  FE2O3

 46      1  0.00   4.27  3.63   11.87   2.74   9.02  0.29  44.73  0.33  2.29
 47      3  0.15   4.70  3.24   11.36   2.43   3.90  0.24  49.44  0.35  2.19
 48     10  0.02   4.60  3.27   12.08   2.65   2.91  0.40  51.00  0.53  2.20
```

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