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Interactions Between Concrete and Brine at the Waste Isolation Pilot Plant (WIPP) Site, New Mexico

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INTERACTIONS BETWEEN CONCRETE AND BRINE AT THE WASTE ISOLATION PILOT PLANT (WIPP) SITE, NEW MEXICO[1]

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ABSTRACT

A concrete liner emplaced in 1984 in a shaft at the Waste Isolation Pilot Plant has served as a natural laboratory for observing interactions among concrete, evaporite rocks, and brine. During a routine inspection of the liner in the spring of 1990, discoloration, deposition of secondary salts, wet areas with exposed aggregate grains, softening of paste, surficial spalling, and cracking were observed locally on the concrete surface of the liner. Some construction joints showed apparent leakage of brine from behind the liner, which was nominally 50 cm thick. Seepage brines were nearly saturated relative to $CaCl2$ and contained lesser amounts of $MgCl2$ and KCl, and minor $NaCl$. The liner surface was locally altered to a 1-2 cm friable hygroscopic layer containing little cement paste; concrete cores (7 or 10 cm diameter) through the liner at depths of 248, 254, 255, and 271 m showed similar degrees of alteration at the liner/rock interface. The most profound alteration of concrete was developed in a ~7 cm zone subparallel to and straddling the construction joint cored at a depth of ~254.5 m. This zone was extensively microfractured, transected aggregate grains, and contained brucite, gypsum, magnesium hydroxychloride hydrate, and locally calcium chloroaluminate instead of the usual phases of hydrated portland cement. Several mechanisms of chemical degradation have been proposed, the most likely being attack by magnesium ions.

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[1] This report has been accepted for publication in the Proceedings of the Materials Research Society. The present version differs in format only.

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INTRODUCTION

Background

The Waste Isolation Pilot Plant (WIPP) was authorized by Public Law 96-164 "as a defense activity of the DOE [Department of Energy] for the express purpose of providing a research and development facility to demonstrate the safe disposal of radioactive wastes from the defense activities and programs of the United States." The WIPP facility has been excavated at a depth of 660 meters in bedded rock salt of the Salado Formation (Ochoan Epoch, Permian Period) in the Delaware Basin of southeastern New Mexico.

The waste-handling shaft was designed to function throughout the operational period of the WIPP (approximately 30 years). The concrete liner of the Waste shaft was placed between November, 1983, and April, 1984. The liner was constructed from the surface downward to a depth of approximately 250 meters (nominally 50 cm thick). Then a key (nominally 1 meter thick) to support the liner was constructed upward from a depth of approximately 275 meters to approximately 250 meters in 2.5 to 3 meter lifts; more than two weeks elapsed between placing the concrete at the bottom of the liner and placing the concrete at the top of the key. In addition, the concrete at the top of the key was placed underneath the previously placed liner. Figure 1 shows the approximate configuration of the shaft liner (not to scale).

During inspections of the Waste shaft in May, 1990, patchy surface layers of deteriorated concrete were observed between approximately 247 and 274 meters below the surface. Highly-developed deterioration and possible seepage of groundwater were noted at joints in the liner key, particularly at what appeared to be a joint located 254.5 meters (834 joint; Figure 1) below the surface.

Objectives

The objectives of this work were to analyze samples of apparently deteriorated concrete to determine its condition and possible mechanisms of its deterioration. This work is part of an ongoing program to investigate the long-term behavior of cementitious materials in evaporite environments.

Although previous studies have documented the interactions between cementitious concretes and solutions of high ionic strengths (such as sea water), concretes are not routinely exposed to environments containing groundwater with such high concentrations of solutes as in the evaporites of the northern Delaware Basin. Consequently, this environment provides a unique natural laboratory for investigating the behavior of concrete under unusually harsh geochemical conditions.

Materials in the Shaft Liner

The aggregate was dolomite, required to conform to ASTM C 33 and was supplied from local commercial quarry operations in the Carlsbad, New Mexico area. The cement was required to conform to ASTM C 150, Type V. This concrete formulation can be described generally as a mixture of low-alumina cement, a pozzolan (Class C fly ash substituting for 18%

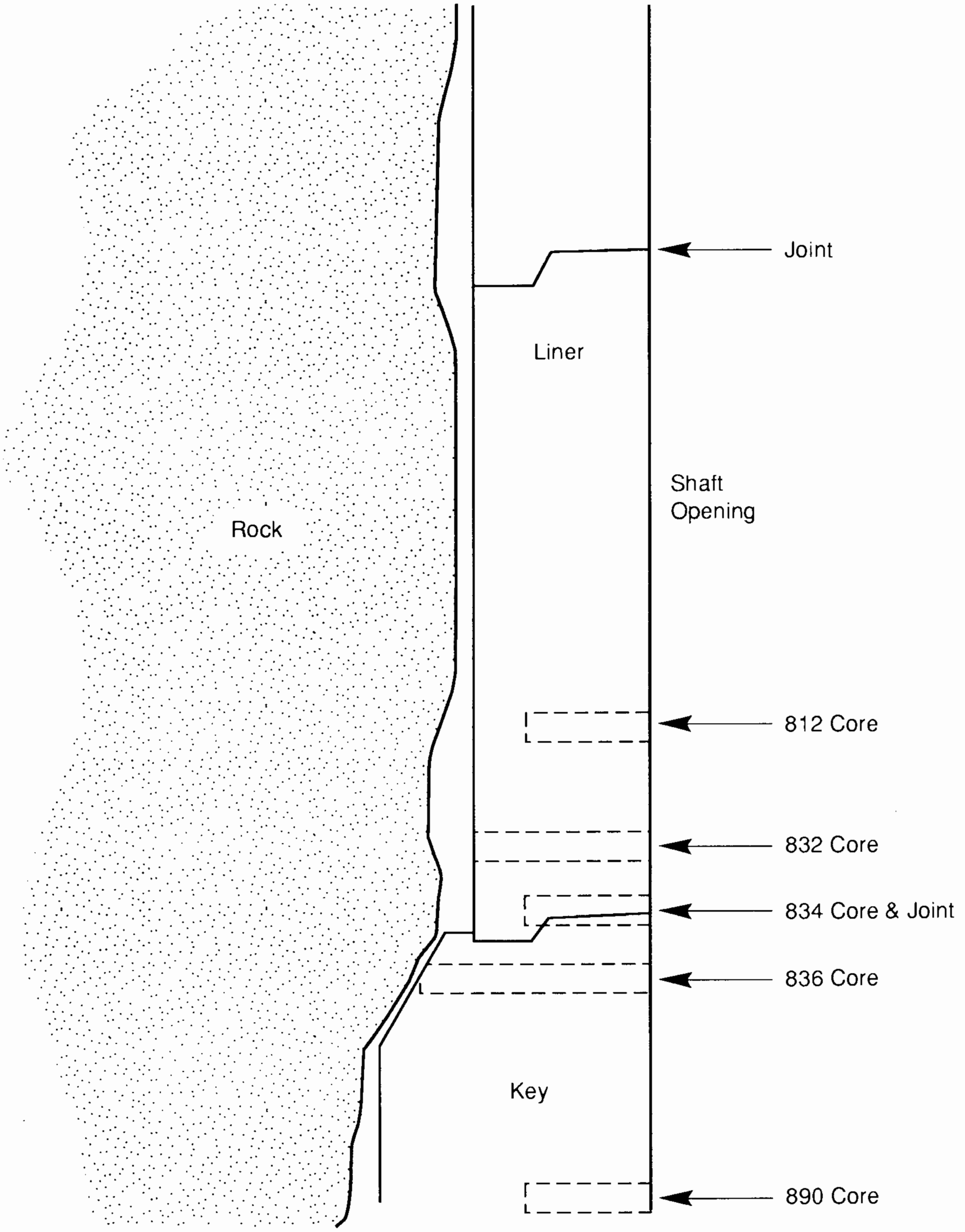

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Figure 1. Schematic configuration of WIPP waste shaft and concrete liner, showing relative locations of cores in this study, designated by depths in feet. Not to scale.

cement), and an aggregate, with a low ratio of water to cementitious materials.

Both cementitious and chemical grouts were injected into the space behind the liner to control water inflow, on two separate occasions. The first grouting was done following the completion of the liner and key; the second took place two years later, after water leakage into the shaft was observed.

SAMPLING AND ANALYSIS

Concrete Samples

In June, 1990, soon after the inspection of the shaft liner that revealed surficial deterioration of concrete, grab-samples of friable surficial material (possibly representing topically applied mortar) were collected at depths of 248 and 271 meters, together with cores 11-16 cm long, which did not completely penetrate the shaft liner. Because brine was seeping from the joint between the key and the bottom of the liner, resulting in dampening and discoloration, the "cold joint" was the suspected pathway for brine movement through the liner. A horizontal core, 15 cm long, was obtained from the cold joint (254.5 meters depth). Finally, cores through the liner concrete were obtained above and below the cold joint, at 253 and 255 meters depth (cores 832 and 836; Figure 1).

Macroscopic evidence of concrete deterioration included discoloration, apparent deposits of secondary salts, spalling, cracking, and wet areas showing grains of aggregate exposed in relief. Petrographic evidence included etching of aggregate grains and softening of aggregate and paste due to the formation of secondary phases. The thickness of the damaged zone varied from about 3 mm (248 m depth) to about 4.5 cm (271 m depth). Damage was apparent both at the concrete/rock interface and at the inner exposed end of the full-thickness cores. Except for these damaged zones at interfaces and along the cold joint at 254.5 m, the concrete appeared intact. The nature of the alteration on either side of the cold joint, which apparently was a flow-path for brine, was studied in more detail.

Phase changes accompanying the deterioration were determined by X-ray diffraction (XRD), scanning electron microscopy (SEM), and energy-dispersive X-ray spectrometry (EDX). Table I gives the secondary phases observed in the deteriorated layers and secondary deposits.

Brine Samples

Two samples of brine were obtained for analysis, one from a seep issuing from the cold joint and one from a collector ring installed at the bottom of the shaft liner. Solute analyses were provided by W. B. Chambers of Sandia National Laboratories, as determined by inductively-coupled plasma-atomic emission spectroscopy, ion chromatography, and titrimetry. The solute data, together with those of nearby groundwaters, are given in Table II.

Table I. Crystalline Phases Identified by XRD in Grab Samples

Field Sample[a]	Depth, m	Phase Composition (determined by XRD)
812-145	248	gypsum, calcium sulfate hemihydrate, brucite, sylvite, halite, $Ca_3Al_2O_6 \cdot CaCl_2 \cdot 10H_2O$
812-265	248	$Ca_3Al_2O_6 \cdot CaCl_2 \cdot 10H_2O$, halite, brucite, $Mg_2(OH)_3Cl \cdot 4H_2O$
890-085	271	gypsum, calcium sulfate hemihydrate, brucite, halite

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[a] Field sample identification is derived from the depth, in feet, followed by the azimuth relative to magnetic north.

Table II. Comparison of Solutes in Groundwaters (mg/liter)

Brine	Source	Ca	Mg	K	Na	Cl	SO_4
AEC-8 Bell Canyon Fm.	[1]	10000	2500	860	55000	120000	240
H-1 Rustler/Salado	[2]	13000	30000	17000	56000	210000	520
H-2C Rustler/Salado	[2]	9200	25000	9100	66000	200000	1300
H-3 Rustler/Salado	[2]	18000	25000	14000	59000	210000	370
seep, cold joint	(this	185000	20600	12300	1370	485000	959
collector ring	work)	192000	20600	15100	4110	426000	411

RESULTS AND DISCUSSION

Source of Seepage Brine

A comparison of the solute data in Table II shows that the solutes in the cold-joint seep and collector-ring brine samples are not derived directly from local groundwaters. Although brines high in Ca and Cl are found locally in the Bell Canyon Formation [1], these occur at about 1200 meters depth; they are not likely candidates for the source of waste-shaft seepage. The seepage brines are anomalous in that they are nearly saturated solutions of $CaCl_2$, with relatively little NaCl. Thus, the seepage brines are not directly derived from the contact zone between the Rustler and Salado Formations (~250 m depth; represented by samples from boreholes H-1, H-2C, and H-3 in Table II), although this zone is the nearest source of natural fluid. The similarities in K/Mg ratios between the seepage brines and those from the Rustler/Salado contact suggest that the former were derived from the latter, with modification due to the leaching of Ca from the shaft-liner concrete. Such a mechanism, however, requires a large decrease in Na; although ionic exchange of Na for Ca is in principle possible, we are not presently able to propose a detailed description of such a process.

Nature of Concrete Degradation

The core through the cold joint at 254.5 m exhibited the most readily observable deterioration of concrete, and represents the interactions between concrete and brine in the WIPP waste shaft. Within a zone extending about 7 cm on either side of the joint the concrete was visibly discolored (reddish-brown) and contained extensive fracturing and microfracturing roughly parallel to the joint. Figure 2 depicts this zonation, resulting from both the joint-filling grout and the subsequent deterioration. The zonation is further described in Table III.

Intact paste in zones T and Z (Table III) contains the expected $Ca(OH)_2$ and calcium silicate hydrates; evidence for deterioration in zones U, V, and Y includes absence of these phases, and the presence of magnesium chloride hydroxide hydrate ($Mg_2(OH)_3Cl \cdot 4H_2O$), brucite, and gypsum [3]. The scarcity of calcium chloroaluminate ($Ca_3Al_2O_6 \cdot CaCl_2 \cdot 10H_2O$) in both intact and deteriorated concrete above the joint, its presence below the joint, and a difference in Ca/Si ratio (determined by EDX) suggest a difference in chemical composition of the cementitious materials, which may make comparisons of deterioration across the joint more difficult.

It was inferred from construction records that zone X was originally building paper, and that the chemical grout (zone W) was calcium acrylate.

The dolomite aggregate grains (a few cm across) in deteriorated concrete were coated with white crystalline material, consisting largely of magnesium chloride hydroxide hydrate, accompanied by brucite and gypsum below the joint. The extensive fracturing parallel to the joint in Zone U below the joint transects paste and aggregate without differentiation; aggregate grains straddling the Zone T/U boundary break along the boundary. The portion of each aggregate grain within intact concrete remained intact (without alteration) and bonded to the paste. Therefore, we interpret this boundary as a

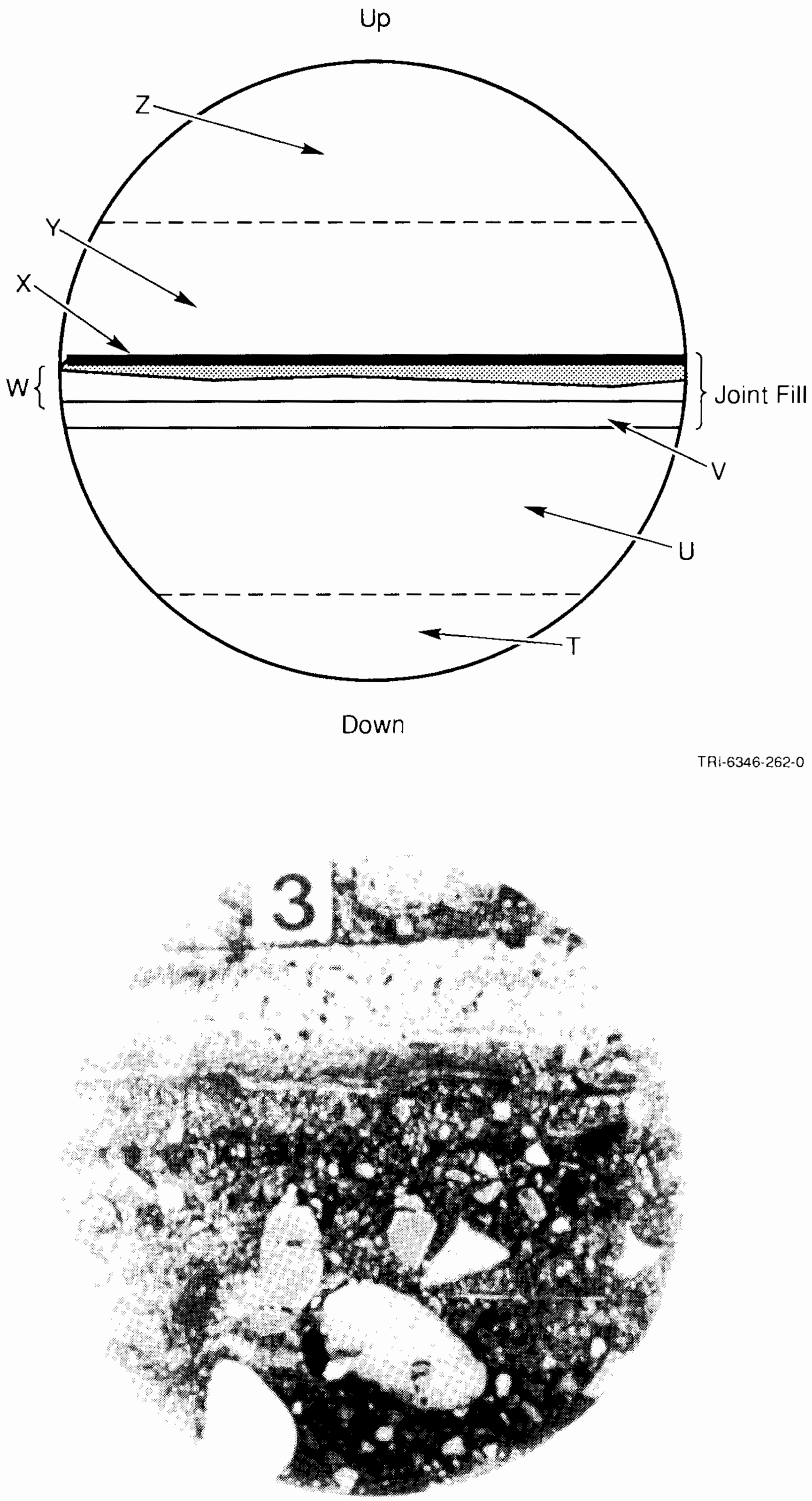

Figure 2. Cross section of core through cold joint (254.5 m depth); core cross-section is 7.5 cm. Zonation is described in Table III.

Table III. Phase Composition of Components of Cores through the Cold Joint, 254.5 Meters Depth

Zone (Fig. 2)	Material	Phase Composition (determined by XRD)
Z	intact concrete, above joint	calcium silicate hydrate, calcium hydroxide, $Ca_4Al_2O_7\cdot\frac{1}{2}CO_2\cdot 12H_2O$, $Ca_3Al_2O_6\cdot CaCl_2\cdot 10H_2O$ (trace)
Y	partially deteriorated concrete, above joint	$Ca_3Al_2O_6\cdot CaCl_2\cdot 10H_2O$, calcite, dolomite
X	top of joint fill	organic
W	joint fill--chemical grout	gypsum, brucite, calcite, $Mg_2(OH)_3Cl\cdot 4H_2O$
V	grout in joint	gypsum, $Mg_2(OH)_3Cl\cdot 4H_2O$, brucite, magnesium silicate hydrate
U	deteriorated concrete below joint	gypsum, $Mg_2(OH)_3Cl\cdot 4H_2O$, ettringite, $Ca_3Al_2O_6\cdot CaCl_2\cdot 10H_2O$, halite (face)
T	concrete below joint	calcium silicate hydrate, calcium hydroxide, $Ca_4Al_2O_7\cdot\frac{1}{2}CO_2\cdot 12H_2O$, ettringite, $Ca_3Al_2O_6\cdot CaCl_2\cdot 10H_2O$

reaction front representing the extent of chemical alteration, where both paste and aggregate have similar degrees of alteration.

MECHANISMS OF CONCRETE DETERIORATION

The first hypothesis for the origin of the friable layer on the surface of the WIPP waste-shaft liner was that the layer was merely a superficial residue from the evaporation of native groundwater, represented by the seepage brines. However, the mismatch between solutes in seepage brine and those in nearby groundwaters, together with the exposure of aggregate grains in relief suggested processes of deterioration entailing chemical interaction between fluid and concrete. We considered acid attack (possibly due to absorption of atmospheric CO_2 by groundwater on the inner surface of the liner), cracking due to cyclic wetting and drying, chemical alteration of cement paste by reaction with brine, and combinations of these mechanisms. Some of these require exposure at the surface to air from which CO_2 could be absorbed and into which water could evaporate, but the degradation on either side of the construction joint and at the concrete-rock interface, where there could be no direct exposure to ventilation air suggested that exposure to atmosphere was not necessary for deterioration.

Chloroaluminate occurs in the intact concrete, but it is more abundant throughout the deteriorated zone and in the intact key-concrete, suggesting a significant degree of interaction between some reactive alumina in the concrete and chloride in seepage brine. Despite the availability of magnesium and sulfate in the seepage brine, only traces of ettringite occur in both deteriorated and intact concrete below the cold joint. Although the concrete mixture was chosen to resist attack by sulfate, interaction with chloride appears to have favored the formation of chloroaluminate [4,5]. However, the most profound changes in the concrete system are the disappearance of calcium silicate hydrate, and the appearance of magnesium phases on exposed surfaces and in cracks, both in paste and aggregate, exemplified by magnesium chloride hydroxide hydrate and brucite; the former is reminiscent of Sorel cement [6]. The only remaining calcium phase in the grouted zone (V), inferred to have been originally cementitious, is gypsum. The destruction of calcium silicates may have released silica gel, which reacted with brucite to form magnesium silicate hydrate, the ultimate product of magnesium attack [7]; this phase is difficult to identify conclusively by XRD and EDX. The paste within the deteriorated concrete contains calcium sulfates and carbonates instead of the calcium silicates typical of cementitious systems, suggesting that attack of the concrete by magnesium in the seepage brine (21000 mg/l; Table II) is an important mechanism of concrete deterioration, analogous to findings in experimental systems [8]. We infer that the secondary growth of magnesium chloride hydroxide hydrate and brucite in the dolomite aggregate grains, representing Mg-addition and/or Ca-loss, has resulted from this same attack.

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