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Characterization of a Low-Level Radioactive Waste Grout: Sampling and Test Results

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CHARACTERIZATION OF A LOW-LEVEL RADIOACTIVE WASTE GROUT: SAMPLING AND TEST RESULTS

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Richland, Washington 99352

SUMMARY

Westinghouse Hanford Company (WHC) manages and operates the Grout Treatment Facility (GTF), at the Hanford site, as part of a U.S. Department of Energy (DOE) program to clean up wastes stored at federal nuclear production sites. Pacific Northwest Laboratory (PNL) provides support to the Grout Disposal Program through pilot-scale tests, performance assessments, and formulation verification activities. In 1988 and 1989, over one million gallons of a low-level radioactive liquid waste was processed through the facility to produce a grout waste form that was then deposited in an underground vault. For this campaign, the liquid waste was Phosphate/Sulfate Waste (PSW) generated during decontamination operations on the N Reactor. PNL sampled and tested the grout produced during the second half of the PSW campaign to support quality verification activities prior to grout vault closure.

Samples of grout were obtained by inserting nested-tube samplers into the grout slurry in the vault. After the grout had cured, the inner tube of the sampler was removed and the grout samples extracted. Tests for compressive strength, sonic velocity, and leach testing were used to assess grout quality. Results of these tests were compared to results obtained from pilot-scale test grouts made with a simulated PSW.

The grout produced during the second half of the PSW campaign exceeded compressive strength and leachability formulation criteria. The nested tube samplers were effective in collecting samples of grout although their use introduced greater variability into the compressive strength data.

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1.0 INTRODUCTION

As part of efforts to clean up federal production sites, the U.S. Department of Energy (DOE) is treating selected low-level liquid wastes by incorporating them into grout. At the Hanford Site, low-level radioactive liquid wastes will be mixed with a blend of Portland cement, fly ash, clays, and other ingredients in a continuous process at the Grout Treatment Facility (GTF). The resulting slurry will be pumped to lined, underground concrete vaults where the grout will harden, thereby immobilizing contaminants. Westinghouse Hanford Company operates the GTF as part of the Grout Disposal Program at Hanford. Pacific Northwest Laboratory (PNL)[a] provides technical support to the program through pilot-scale tests, performance assessments, and formulation verification activities.

In July 1989, a program milestone was achieved with the production of over one million gallons of Phosphate/Sulfate Waste (PSW) grout. The phosphate originated from a commercial phosphoric-acid-based cleaning solution that was used in decontamination operations at the N Reactor. The remainder of the waste was generated during maintenance of the fuel storage basin, and consisted of a waste from the regeneration of ion-exchange resins with sulfuric acid, and sandfilter backwash.

1.1 SCOPE

Pacific Northwest Laboratory sampled the grout produced during the second half of the PSW campaign to obtain representative samples of grout for use in demonstrating product quality. As part of this effort, the resulting data were compared with data from samples obtained during a previous pilot-scale test of PSW grout production. Comparisons of grouts made in the laboratory to grouts made using the pilot-scale system are presented in an earlier report (Lokken et al. 1988).

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2.0 PROCEDURES

Product quality was evaluated using standard grout characterization tests. Tests used to evaluate the grout included bulk density, sonic velocity, compressive strength, and leachability.

2.1 SAMPLING

A nested-tube sampler, previously used to sample grout during pilot-scale tests, was chosen for this sampling effort because it offered an easy, inexpensive means of obtaining sufficient quantities of samples for testing. This sampling method has several advantages: 1) by leaving the sampler in place, the sampled grout cures under representative conditions; 2) mixing of the grout with free liquid, as may occur with samples obtained by dipping, is avoided; and 3) the sampled grout remains uncontaminated by core drilling fluids.

Obtaining samples using this sampler-tube method has one disadvantage, compared to core drilling; semi-solid grout may be disturbed during insertion. Data presented in this report indicate that, as the sampler penetrated deeper into "older" grout, the grout structure was affected such that increased variability was found in compressive strength data.

2.1.1 Sampler Tube Description

The samplers were constructed of thin wall tubing with a narrow gap between tubes to minimize disturbance of grout (see Figure 1). The outer tube was a 2-in. schedule five, galvanized steel pipe. A small hole was drilled near the top of the outer tube so that a loop of cable could be inserted to lower the assembled sampler through the grout vault riser. The inner tube was a 1.5-in. class 125, PVC irrigation pipe. The upper end of the PVC tube was reinforced to accept a pin for pulling out the inner tube with a cable. The pin was also used to twist the inner tube to break the core sample free of the underlying grout. The inner and outer tubes were separated by two o-rings at the bottom to prevent infiltration of grout into the annulus.

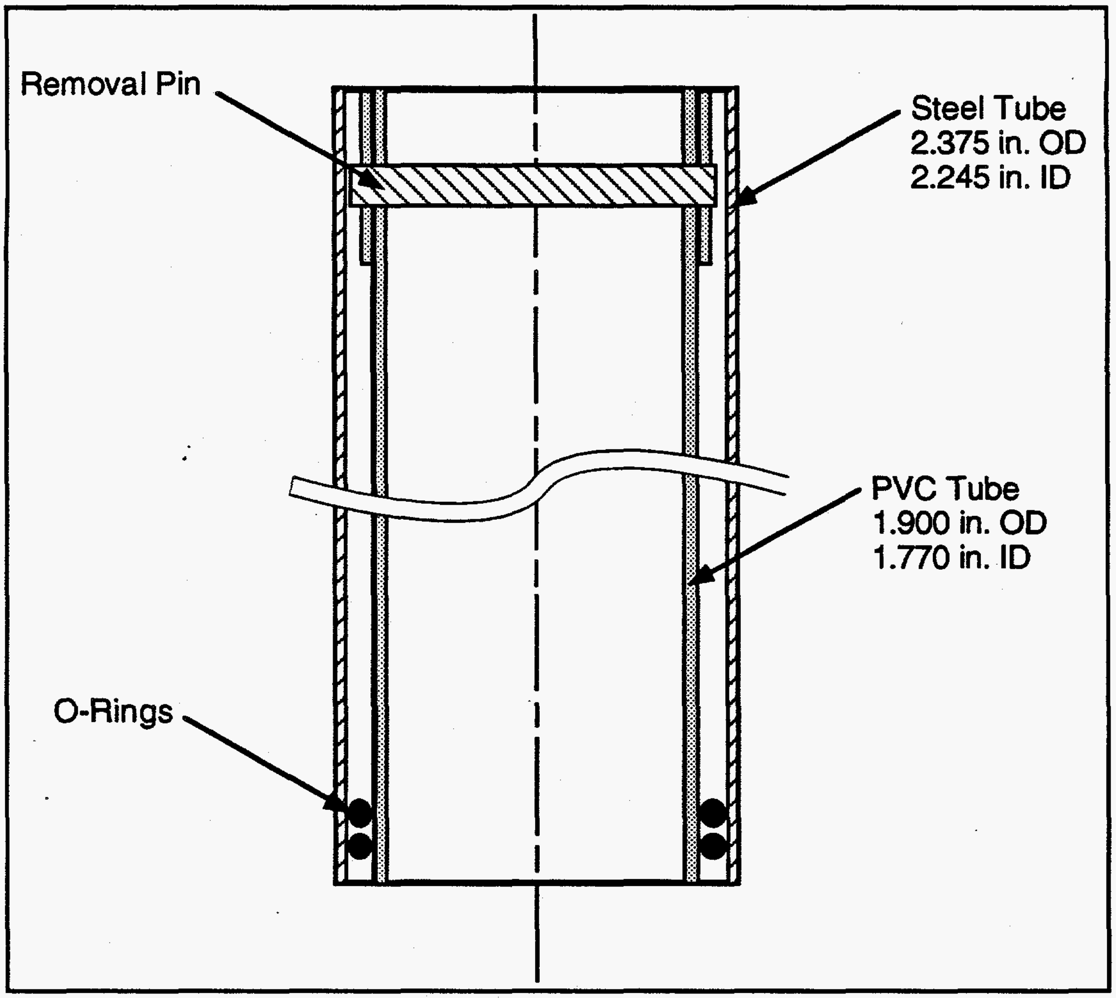

FIGURE 1. Schematic of Nested-Tube Sampler

2.1.2 Sampler Tube Placement and Removal

The distance from the top of the riser to the grout surface was measured using a continuity tester. A length of electrical cord with exposed conductors was lowered until contact with free liquid completed the electrical circuit. The cord was then lowered through the free liquid until slack was observed in the cord, indicating contact with semi-solid grout. Depth measurements were recorded and the sampler tubes cut to the length required to sample a sufficient amount of grout and eliminate penetrations through the cold-cap due to leaving the outer tube behind.

Sampler tubes were installed by lowering the assembly through a sealable "guide tube" to approximately 6 inches above the measured grout surface. The tubes then sank through the slurry until they made contact with semi-hardened grout at a depth of approximately 1-foot, effectively sealing the sampler from further filling with grout slurry. The 1-foot depth was approximately equal to the amount of grout produced in 12 hours. The retrieval cable was pre-marked to later verify that the sampler had sunk to the appropriate depth. No further force was required to sink the samplers to the required depth. Figure 2 shows an inserted sampler, containing sampled grout, imbedded in grout placed during continued processing.

When the grout had cured, sampler tubes were twisted free of the underlying grout then pulled out using the attached retrieval cable. A backup removal method consisted of a length of PVC pipe that could be inserted and glued into the sampler tube. This backup removal tube projected above the vault cover to allow twisting and lifting of the sampler tube.

2.1.3 Grout Sample Removal

After the sampler had been pulled to the surface, the top of the grout in the sampler was located by placing a large "hair pin" over the edge of the tube. Because both legs of the hair pin were of equal length, the inside leg touched the top of the sample and the outside leg showed where the tube could be cut. A small hole was drilled immediately above the grout sample and the free liquid was allowed to drain back into the vault. The sampler tube was shortened and double bagged for shipment. In the lab the tube was cut from the core sample using an abrasive blade with a depth stop so that the samples would not be damaged. Samples were removed from the sampler tube and immediately covered in plastic to prevent drying.

2.2 TESTING

The purpose of the sampling and testing effort was to assess the quality of the solidified grout produced during the second half of the PSW campaign. The properties of concern for this investigation were the compressive

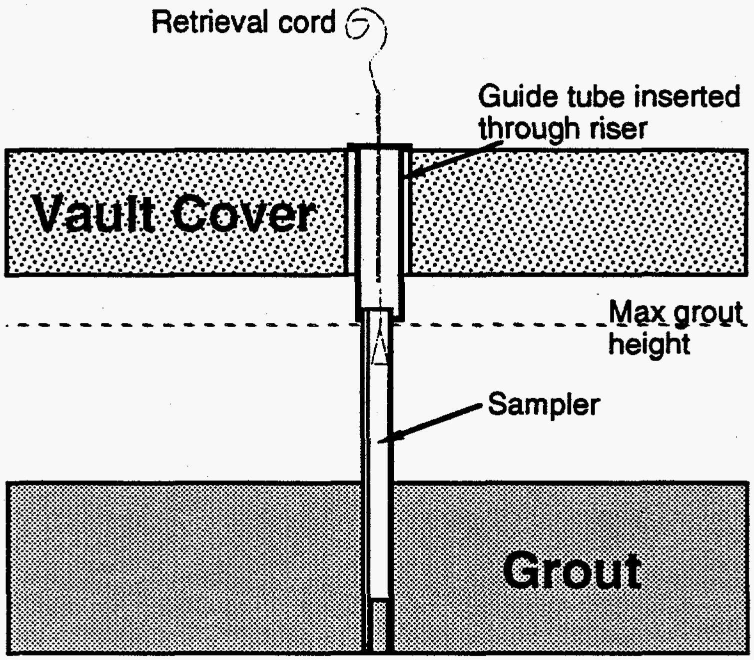

FIGURE 2. Schematic of Inserted Nested-Tube Grout Sampler

strength and leach resistance. Chemical and radiochemical compositions, densities, moisture contents, and sonic velocities were also determined.

The physical strength of grout samples was determined by performing unconfined compressive strength tests. Density and sonic velocity (the speed at which a compressional wave can be conducted through a sample) were determined because the data generated correlate well with compressive strengths (Neville 1981 and Lokken 1988). Leach testing was conducted to determine how well the grout inhibits diffusion of contaminants. The leach tests were conducted following the American Nuclear Society (ANS) 16.1 procedure (ANS 1986).

2.2.1 Sample Preparation

The cores were sectioned using a saw and miter box. After cutting, a jig was used to hold the samples at right angles so that the faces of the specimen could be made parallel. Each sample was placed in the jig with one end exposed and then rubbed over a stiff flat screen until the desired flatness and overall length was achieved. The same cylindrical sample was used to determine bulk density, sonic velocity, compressive strength, and evaporable water content. The first three tests were conducted on two samples from each core, while evaporable water was determined on only one sample from each core.

2.2.2 Density

Bulk density of the samples was determined by dividing sample weight by sample volume, as determined from dimensions of samples used for compressive strength testing.

2.2.3 Compressive Strength

Compressive strength tests were performed on right circular cylinders with a length-to-diameter ratio of 2, using ASTM method C39-84 (ASTM 1984). An Instron[a] test machine was used to conduct the tests. The cross-head rate was 0.05 in./min. The load at failure was determined by using the maximum load reading from the test curve. The compressive strength was calculated by dividing the load at failure by the cross-sectional area of the sample.

2.2.4 Sonic Velocity

Sonic velocity was determined using a James[b] Sonic Velocity Meter (V-Meter) Model 4902, according to the test method outlined in ASTM C597-83 (ASTM 1985). This procedure was modified by omitting the use of grease as an acoustic coupling agent. All samples were tested with water as the coupling agent. The accuracy of this method was verified by retesting half of the samples using grease as a coupling agent; identical results were obtained.

2.2.5 Moisture Content

Following compressive strength testing, those samples not contaminated with grease from sonic velocity testing were dried to determine the amount of

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[a] Instron Corporation, Canton, Massachusetts.

[b] James Instruments Incorporated, Chicago, Illinois.

evaporable water. Evaporable water was determined by heating the samples at 105°C until a constant weight was reached.

2.2.6 ANS 16.1 Leach Test

The final characteristic used to evaluate the grouts was leach resistance. A modified ANS 16.1 leach procedure was used to gauge the leachability of nine samples. The procedure was altered by omitting the 30-second rinse and using cumulative leaching times of 7 hours; 1, 2, and 4 days; and 1, 2, 4, 8, and 13 weeks. Two cores were tested in triplicate to determine the reproducibility of experimental results.

2.2.7 Chemical Analysis

X-ray fluorescence (XRF) was used to determine the initial inventory of chemical species (A_0) in the grouts. The compressive strength samples, one from each core, that were dried to determine moisture content (see 2.2.5 above) were then ground to a fine consistency with an analytical mill prior to analysis. The average of these five analyses was used for subsequent leach test calculations.

Leachates were passed through 0.45-µm filters and then acidified with 1 vol% nitric acid for cation analysis by inductively coupled argon plasma atomic emission spectroscopy (ICP). Untreated leachates were submitted for anion analysis using an ion chromatograph (IC).

Leachate uranium analysis was performed using a Scintrex[a] UA-3 uranium analyzer. This technique employs an ultra-violet light/uranyl salt fluorescence to determine the concentration of uranium in solution. This test was performed using acidified leachate samples. Spike additions were made to the uranium test solutions to make sure that quenching of peaks was not occurring.

A Nuclear Data[b] Model 6700 analyzer with peak search software was used for radionuclide analysis. Leachates were passed through 0.45-µm filters and acidified prior to assay. Twenty milliliter aliquots were counted from 2 to 24 hours in germanium detectors to determine gamma radiation spectra.

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[a] Scintrex, Concord, Ontario, Canada.
[b] Nuclear Data Inc, Schaumberg, Illinois.

3.0 RESULTS AND DISCUSSION

Product quality can be evaluated in terms of whether the grout meets or exceeds design criteria and whether the grout produced is as uniform or homogeneous as desired. An estimate of homogeneity could be inferred from the experimental data, however no benchmark for this parameter has been established for PSW grout. This report addresses two performance criteria: compressive strength and leachability. Formulation criteria specify a minimum unconfined compressive strength of 60 psi. In the section that follows, compressive strength is related to other parameters that are also potentially useful in evaluating grout quality.

A discussion of the leach indices for these grout samples, the second design criterion considered in this report, is made difficult because of the absence of any regulated quantities of hazardous components and the low concentrations of radionuclides. Therefore, attention was focused on sodium leach behavior for assessing the performance of the grout with respect to leach resistance criteria. Sodium was chosen because it is a highly soluble ion and is present in sufficiently large quantities to yield concentrations well above detection limits in the leachates. As a highly soluble ion, sodium is present in the interstitial fluids of the grout and therefore represents those chemical species in the grout which are most readily leached. Using sodium to determine the leach index for the grout, therefore, represents a conservative approach.

3.1 FIELD RADIATION MEASUREMENTS

Measurements of dose rates and total beta-gamma radiation were made by WHC in the field. The maximum uncorrected dose rate through a 60-mL container filled with newly sampled grout was 0.3 mrem/hour. Direct beta-gamma, for the same sample, was measured at 20,000 dpm.

3.2 SAMPLER PERFORMANCE

Four of the six sampler tubes were removed using the attached retrieval cable. The retrieval pin in two samplers snapped or pulled through the PVC tubing during removal efforts. It is believed that removal problems were caused by grout that had leaked past the o-rings into the space between the inner and outer tubes. One additional tube was removed using the backup method; the final core was not retrieved because the tube was weakened by the heat generated within the vault and broke off above the core when twisted for removal.

3.3 VISUAL OBSERVATION

Two of the five cores had three breaks spread out along their lengths, one core had two breaks, and the two shortest cores each had one break (see Figure 3). It is believed that these breaks occurred when the PVC tube was twisted to break it free. The torsional stresses imparted during removal were transferred, in part, to the grout core and resulted in radial fractures.

The core samples were uniform in appearance with no large voids or inclusions. Each core sample had a thin soft layer on top due to settling of suspended solids from the free liquid. The bottom portion of the core from sampler 1-riser 15 appeared to be highly cracked. The cracking is thought to be an artifact of sampling grout that had become fairly hard because the grout pour was sampled more deeply at this location. This sampler was dropped from a height greater than 6 inches from the top of the grout surface, and thereby penetrated into a layer of grout that had cured for a longer time. Grout in the last stages of gelling cannot easily flow back together and "heal" when disturbed and would result in a region where cracking would be evident. Samples for grout characterization were taken from the middle of the core samples to avoid these two "edge effects" (see Figure 4).

Because the sampler displaced grout as it was inserted, the length of the core samples was expected to be different than the insertion depth. If all of the displaced grout was directed up into the inner tube, and the insertion depth was 12 inches, the core length would be 21.6 inches. However, no provision was made for holding the samplers at depth; they were merely

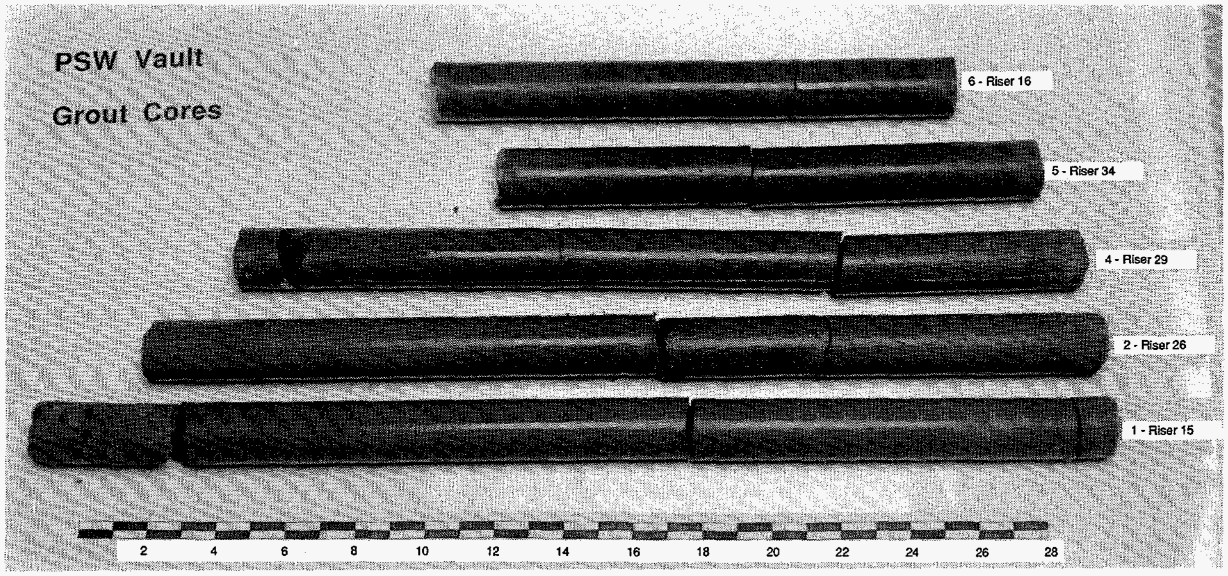
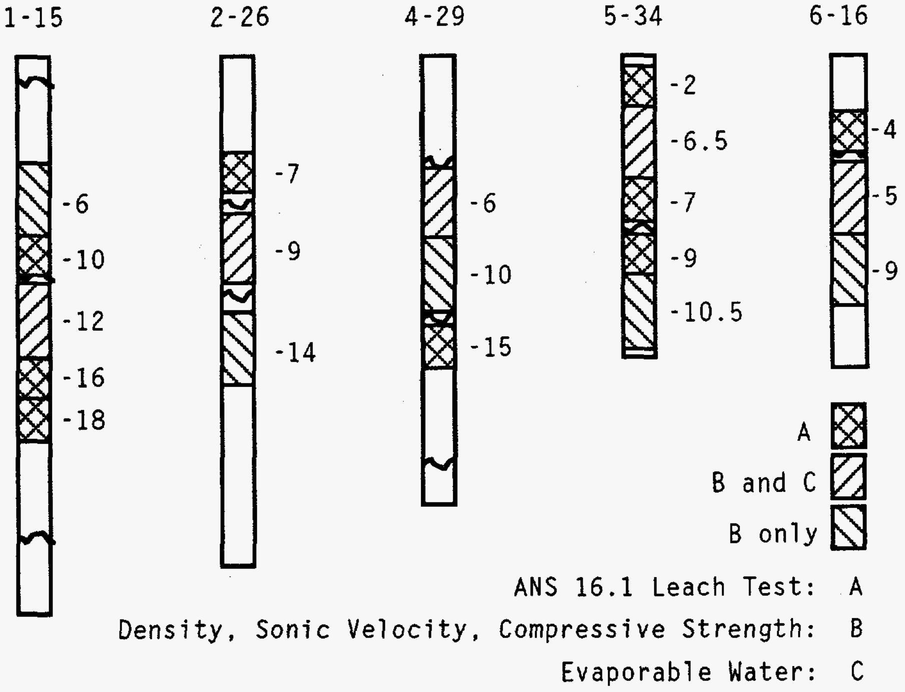


FIGURE 3. PSW Vault Grout Cores, (Lengths in inches)



ANS 16.1 Leach Test: A

Density, Sonic Velocity, Compressive Strength: B

Evaporable Water: C

FIGURE 4. Core Sectioning Diagram

dropped and then the depth of penetration determined. (The samplers were held in place by the surrounding semi-solid grout, and were kept from tilting by the guide tube.) Furthermore, the first samplers placed were longer and therefore heavier than the samplers placed at the end of the campaign. The heavier samplers would tend to penetrate deeper into the grout slurry than the lighter samplers, other factors being equal (see Table 1).

3.4 PHYSICAL PROPERTIES

Results from tests to determine density, sonic velocity, compressive strength, and evaporable water are listed in Table 2. Also included in the table are several other attributes potentially important to consider when evaluating test results. The distance from the grout pipe outlet (vault inlet) to the sampling riser was determined for each of the sampling locations. The age of the grout on the day of testing (i.e. elapsed time from placement to testing), and the distance from the top of the core sample to the mid-point of the test specimen, are also included in the table.

TABLE 1. Sampler Lengths and Installation Measurements

	Distance from Top of Riser				
Riser Number	Free Liquid	Grout Slurry	Sampler Number	As-built Sampler Length	Date Installed
15	21' 7"	22' 7"	1	15' 7"	6/21
26	18' 5"	19'10"	2	12' 4"	6/23
37	14' 3"	16' 4"	3	8'10"	7/6
29	14'	14' 6"	4	7'	7/7
34	11' 5"	12' 8"	5	5' 2"	7/10
16	10'	11' 7"	6	4' 1"	7/11

TABLE 2. Summary of Physical Properties for PSW Grout and Property Correlations

Sample	Density (g/cc)	Sonic Velocity (m/s)	Comp. Strength (psi)	Evap. Water (wt%)	Outlet to Sampler Distance (ft.)	Age (days)	Distance from Top of Core (in.)
1-15-12	1.354	1887	239	55.3	23	92	14
1-15-06	1.370	1928	315	ND	23	92	8
2-26-14	1.395	1975	288	51.6	43	90	16
2-26-09	1.384	1969	355	ND	43	90	11
4-29-10	1.407	1958	280	49.0	45	76	12
4-29-06	1.415	2050	506	ND	45	76	8
5-34-06	1.499	2250	679	43.0	23	73	4
5-34-10	1.444	2054	410	ND	23	73	12
6-16-05	1.451	2115	560	46.4	35	72	6
6-16-09	1.423	2025	439	ND	35	72	10
Density		+ 0.97	+ 0.89	- 0.99	- 0.15	- 0.83	- 0.65
S.Veloc.	+ 0.97		+ 0.96	- 0.94	- 0.16	- 0.72	- 0.74
C. Str.	+ 0.89	+ 0.96		- 0.91	- 0.14	- 0.71	- 0.85
E. Water	- 0.99	- 0.94	- 0.91		+ 0.09	+ 0.90	+ 0.89

ND = Not determined

The bottom portion of Table 2 lists the linear correlation coefficients, r, between pairs of measured properties. From the correlations several conclusions can be drawn:

- The strong correlation between sonic velocity and compressive strength indicates that a reliable model for predicting compressive strength using nondestructive sonic velocity measurements could be developed for a particular formulation.

- There were no significant correlations between the distance the grout flows from the outlet and other measured physical properties. If air is incorporated into the grout as it falls into the vault, changes in density, sonic velocity, and compressive strength might be expected. However, the closest riser was 23 feet from the outlet, which probably provided a sufficient flow length for significant loss of entrained air to occur.

- The amount of evaporable water increases with increasing grout age. This is consistent with the observed absorption of free liquid during grout curing.

- As distance from the top of the core increases, grout compressive strength generally decreases. Simultaneously, as distance from the top of the core increases, the amount of evaporable water tends to increase, resulting in lower bulk densities. These changes are conceivably due to sampler insertion, as explained below.

The use of a nested-tube sampler disturbs the gelled grout and may cause changes in physical properties. Changes may be brought about by competing processes that may become more or less important given the distance from the surface and the degree of set achieved by the grout. These processes include densification and breakup of the grout structure.

As the sampler is inserted, grout is disturbed because the sampler can only hold approximately 56 vol% of the grout it displaces. The displaced grout is pushed further up the inner sample tube or to the outside of the sampler. This disturbance may result in the the displacement of interstitial fluid and/or initiate the loss of entrained air. Loss of fluid or air from the slurry would result in the densification of the grout within the tube. This effect would likely be most pronounced at the top of the sample, where released air bubbles and/or displaced liquid would have only a short path to travel to escape the slurry. Air bubbles and/or liquid further down the sampled grout column are likely to be trapped by the grout as the gel structure rebuilds. Therefore, gradients for density, compressive strength, and evaporable water content might be expected.

For samples near the bottom of the core, densification cannot occur as easily because the displaced air or liquid has too great a distance to travel and therefore simply becomes reincorporated. But, the damage imparted to the structure by inserting the sampler cannot heal as easily because set has proceeded further in the deeper, older grout. Because densification may not take place and the structure may be damaged, a net decrease in compressive strength would be expected.

Physical data obtained for the PSW grout (Vault Tubes) are compared with data from earlier tests of simulated PSW grout in Table 3 (Pilot-Scale Tubes and Cores). Data included are from samples produced during pilot-scale testing (Lokken et al. 1988). These data consist of test results from samples obtained using the nested-tube technique (tubes) and from samples obtained by core drilling (cores) in the pilot-scale trench.

This sampling effort was not designed to provide a rigorous comparison to pilot-scale data because there are too many variables in production, materials, and sampling. However, an analysis of variance (ANOVA) was run on this body of data to determine if the data sets were significantly different. The "p" values from the ANOVA, calculated using the data for Table 3, are presented for information only as follows: density = 0.0572, compressive strength = 0.0075, sonic velocity = 0.2706.

In addition, a "Duncan's Multiple Range Test" was used to illustrate whether the data sets shown in Table 3 were significantly different. The results of this test are shown in Figure 5 where the presence of a bar indicates that the included sets of data were not found to be significantly different. No significant differences were found between data sets when comparing sonic velocity data. When comparing density data, no significant differences were found between the PSW vault tube data and the other two data sets considered individually, however, the analysis did show a significant difference between pilot-scale core and pilot-scale tube sample density data. Finally, compressive strength data for the pilot-scale tubes was found to be significantly different from the other two data sets.

TABLE 3. Average Physical Properties of Pilot-Scale and Actual PSW Grout

Sample Type (No. of Tests)	Density (g/cc)	Sonic Velocity (m/s)	Compressive Strength (psi)
Pilot-Scale Tubes (31)	1.422 ± 0.021*	1990 ± 75	333 ± 67
PSW Vault Tubes (10)	1.414 ± 0.043	2021 ± 105	407 ± 141
Pilot-Scale Cores (16)	1.402 ± 0.022	2030 ± 131	402 ± 52

* ± values are 1 standard deviation from mean

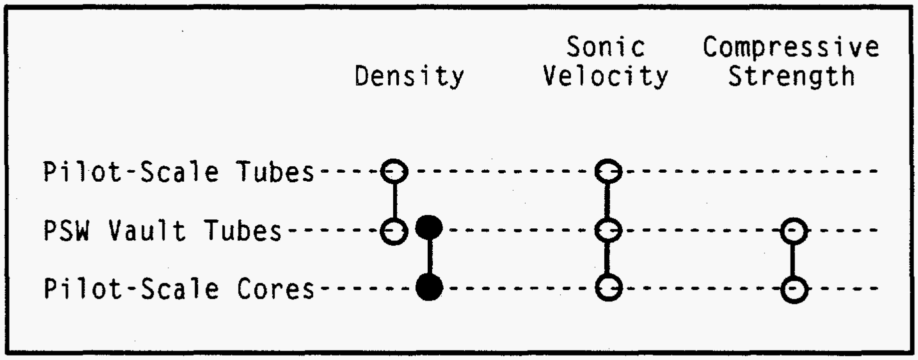


FIGURE 5. Duncan's Multiple Range Test Results

3.5 COMPOSITION

Table 4 lists the weight percent composition of each core sample for the leached species examined in this set of tests. The data were obtained by XRF and radiochemical counting analyses of compressive strength samples and were used to establish initial inventories for chemical species. The XRF analyses yielded very good mass balance results, typically accounting for 98-99 wt% of the grout composition.

3.6 ANS 16.1 LEACH TEST

Table 5 presents leach test data for seven species from the PSW grout cores. The first line in each data set gives the average leach index and standard deviation obtained from nine leach intervals during 91 days of testing. The second row shows the 99.9% confidence range (ANS 16.1) based on a statistical analysis of the leach indexes calculated for each sample. The third row lists the correlation coefficient, r, (ANS 16.1). The sign of r

TABLE 4. Initial Composition of Selected Species in PSW Grout (wt%)

Core	Al	Ca	Na	Si	SO_4	Co-60 (x 10^{-9}	Cs-137 Ci/g)
1	5.84	21.30	2.40	15.01	0.65	2.3	0.22
2	6.06	20.60	2.77	14.82	1.26	3.3	0.25
4	6.37	21.50	2.44	15.43	0.90	2.5	0.26
5	6.30	21.40	2.26	15.19	0.98	2.6	0.26
6	6.04	21.80	2.35	14.91	1.14	2.7	0.24
Average	6.12	21.32	2.44	15.07	0.99	2.7	0.24
S.D.	0.21	0.44	0.19	0.24	0.23	0.4	0.02

indicates whether the leach index tends to increase (+r) or decrease (-r) during the test. A large positive r indicates a significant overall decrease in leach rate from the start to the finish of the test. A small r value for this measure is desirable, particularly if the confidence range is relatively small, because this implies little deviation from diffusion controlled leaching. (Diffusion control yields a horizontal line when "leach index" is plotted versus time.)

Attention was focused on sodium leach behavior for assessing the performance of the grout with respect to leach resistance criteria. Sodium was chosen because it is a highly soluble ion and is present in sufficiently large quantities to yield concentrations well above detection limits in the leachates. As a highly soluble ion, sodium is present in the interstitial fluids of the grout and therefore represents those chemical species in the grout which are most readily leached. Using sodium to determine the leach index for the grout therefore represents a conservative approach. Figure 5 shows the average cumulative fraction of sodium leached during ANS 16.1 testing over the 13-week test. The last error bar in Figure 5 shows a relative standard deviation (RSD) of only 9.7%. Bias in the calculations was

TABLE 5. Average Leach Index, Standard Deviation, and Statistics for PSW Grout Cores

Sample	Aluminum	Calcium	Sodium	Silicon	Sulfate	Co-60	Cs-137
1–15–18	11.7 ± 0.3	10.9 ± 0.4	8.5 ± 0.3	11.8 ± 0.3	9.6 ± 0.9	10.2 ± 1.1	7.7 ± 0.3
Confidence Range [a]	12.2-11.2	11.5-10.3	9.0-8.0	12.3-11.4	11.1-8.1	11.9-8.6	8.2-7.3
Correlation, r [b]	0.89	0.75	0.29	-0.19	0.66	0.78	0.65
1–15–16	11.6 ± 0.3	10.9 ± 0.4	8.5 ± 0.3	11.8 ± 0.3	9.5 ± 0.9	11.0 ± 1.1	8.9 ± 1.0
	12.1-11.1	11.6-10.2	9.0-8.0	12.3-11.4	10.9-8.0	12.8-9.1	10.5-7.3
	0.88	0.71	0.24	-0.19	0.60	0.82	0.80
1–15–10	11.5 ± 0.3	11.0 ± 0.4	8.4 ± 0.3	11.8 ± 0.3	9.2 ± 0.8	10.4 ± 0.8	8.1 ± 0.4
	12.0-11.0	11.5-10.4	8.8-8.0	12.2-11.4	10.5-7.9	11.6-9.1	8.7-7.5
	0.88	0.70	0.32	-0.17	0.54	0.85	0.15
2–26–07	11.7 ± 0.3	11.1 ± 0.4	8.5 ± 0.3	11.7 ± 0.2	10.1 ± 0.9	10.8 ± 1.4	8.9 ± 1.0
	12.2-11.2	11.7-10.6	9.0-8.0	12.0-11.4	11.6-8.6	13.1-8.6	10.4-7.3
	0.86	0.59	0.35	0.32	0.59	0.96	0.92
4–29–15	11.9 ± 0.2	10.9 ± 0.3	8.6 ± 0.3	12.0 ± 0.3	9.8 ± 0.8	10.9 ± 1.5	9.1 ± 1.1
	12.3-11.6	11.4-10.4	9.0-8.1	12.5-11.4	11.1-8.5	13.4-8.5	10.8-7.3
	0.85	0.83	0.56	-0.16	0.66	0.80	0.87
5–34–02	12.0 ± 0.3	11.0 ± 0.4	8.7 ± 0.4	12.1 ± 0.3	10.1 ± 1.0	10.6 ± 1.3	8.2 ± 0.8
	12.5-11.6	11.5-10.4	9.3-8.1	12.6-11.6	11.7-8.6	12.7-8.5	9.5-6.8
	0.86	0.86	0.67	-0.11	0.54	0.84	0.16
5–34–07	12.1 ± 0.3	10.9 ± 0.4	8.7 ± 0.4	12.2 ± 0.4	9.9 ± 0.9	11.1 ± 1.1	9.4 ± 0.8
	12.5-11.6	11.6-10.3	9.4-8.0	12.7-11.6	11.3-8.6	12.9-9.2	10.7-8.1
	0.87	0.87	0.66	-0.14	0.64	0.78	0.78
5–34–09	12.1 ± 0.3	10.9 ± 0.3	8.6 ± 0.4	12.1 ± 0.4	9.9 ± 0.8	10.5 ± 1.0	8.8 ± 0.8
	12.5-11.6	11.4-10.4	9.2-8.1	12.7-11.5	11.2-8.6	12.1-8.9	10.1-7.6
	0.86	0.82	0.64	-0.13	0.67	0.80	0.68
6–16–04	11.9 ± 0.3	10.9 ± 0.4	8.6 ± 0.4	12.0 ± 0.3	10.2 ± 0.9	10.6 ± 1.0	8.8 ± 0.9
	12.3-11.4	11.5-10.3	9.2-8.1	12.5-11.6	11.6-8.7	12.3-9.0	10.3-7.3
	0.89	0.80	0.63	-0.11	0.63	0.83	0.75

---

[a] 99.9% confidence range, (ANS 16.1).

[b] Correlation coefficient, r, (ANS 16.1).

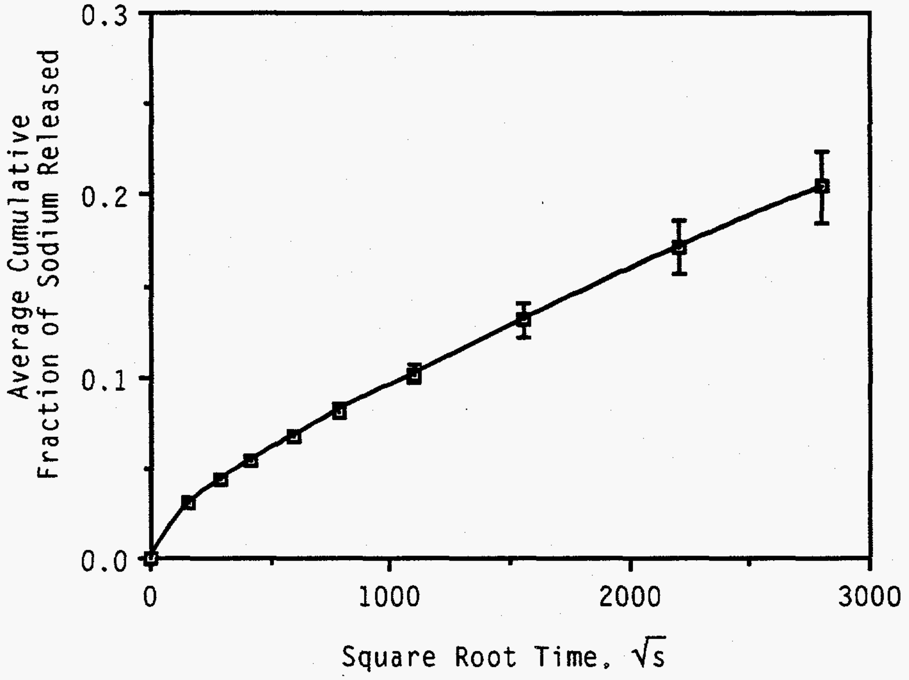


FIGURE 6. Average Cumulative Fraction of Sodium Leached from Nine PSW Core Samples

eliminated by substituting a single average value obtained from the cores tested in triplicate. The RSD was calculated by dividing the standard deviation of the data by average total sodium released. This error value is close to the nominal accuracy for the analytical methods used, generally quoted as 10%. A similar error analysis conducted for each of the cores tested in triplicate yielded 6.5% and 5.6% error for cores one and five, respectively.

In terms of grout formulation leachability criteria, the data show that the grout exceeds the minimum leach index of 7 for the other analyzed species. One apparent exception is the lower range for cesium in sample 5-34-02. The confidence range dips to 6.8, but because the leach index calculations used detection limit values, this value is conservatively low. The low initial inventory of nuclides in the grout resulted in very low concentrations in the leachates, often indistinguishable from background activity. As a consequence, many of the values used to calculate the leach index for the tested samples represent minimum detectable activity (detection limits) as calculated for each detector run. When a nuclide was detected, its calculated concentration was generally associated with a large error (>20%), making the leach indexes variable and lower than anticipated.

Sulfate, cobalt-60, and cesium-137 were present in relatively low concentrations in the grout. The results show that there were some difficulties in analyzing for these three species as indicated by the wide confidence ranges. The width of the confidence ranges is affected by a number of factors: the sensitivity and precision of the analytical techniques and the effect of changing leachate conditions on the leaching behavior of certain species. For these three materials, the wide confidence ranges are believed to be due to low initial concentrations (requiring increased analytical sensitivity) rather than shortcomings in analytical techniques.

No uranium was detected in any of the leachate solutions. The detection limit for uranium was approximately 1.2 parts per billion.

4.0 CONCLUSIONS

Based on the results presented in this report, the following conclusions can be made:

- Grout samples from the PSW vault exceeded the minimum compressive strength criterion of 60 psi.
- Using the nested-tube sampling technique affected the partially cured grout and resulted in greater scatter for compressive strength measurements than observed using other sampling techniques. Disturbance of grout by the sampler resulted in minor property variations in density and sonic velocity.
- Sonic velocity, density, compressive strength, and evaporable water showed strong correlations. Sonic velocity is a more precise predictor of compressive strength than is density.
- No physical property changes resulted from grout flow in the vault between tested distances of 23 and 45 feet from outlet.
- Uranium concentrations in leachates were below detection limits of 1.2 ppb.
- Samples exceeded the minimum leachability index criterion of 7.
- Leachability index for sodium averaged 8.6, with a relative standard variation of less than 10%.

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