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Operable Unit 7-13/14 In Situ Grouting Treatability Studies Bench-ScaleTesting

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Idaho National Engineering and Environmental Laboratory
Bechtel BWXT Idaho, LLC

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Approved by

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ABSTRACT

This engineering design file describes the method and rationale for conducting a complex series of laboratory tests on six grouts applicable to the in situ grouting technology. The six grouts were chosen based on either actual past performance in jet grouting applications, or similarities to jet groutable materials for application for supporting disposal of buried waste sites. Bench-scale testing was performed by University of Akron personnel at the university.

Data from the bench-scale testing will be used to support full-scale field testing of the selected grout at the Idaho National Engineering and Environmental Laboratory. The full-scale field test has been conducted to determine whether the in situ grouting technology is suitable for the stabilization or treatment of wastes buried at the Subsurface Disposal Area at the Radioactive Waste Management Complex.

Data from the treatability studies will be used to support the Operable Unit 7-13/14 remedial investigation/feasibility study and ultimately the Operable Unit 7-13/14 record of decision.

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1. INTRODUCTION

1.1 Purpose

This engineering design file describes the method and rationale for conducting a complex series of laboratory tests on six grouts applicable to the in situ grouting technology. The six grouts were chosen based on either actual past performance in jet grouting applications, or similarities to jet groutable materials for application for supporting disposal of buried waste sites. Bench-scale testing was performed by University of Akron personnel at the university.

Data from the bench-scale testing will be used to support full-scale field testing of the selected grout at the Idaho National Engineering and Environmental Laboratory (INEEL). The full-scale field test has been conducted to determine whether the in situ grouting technology is suitable for the stabilization or treatment of wastes buried at the Subsurface Disposal Area at the Radioactive Waste Management Complex (RWMC) at the INEEL.

Data from the treatability studies will be used to support the Operable Unit (OU) 7-13/14 remedial investigation/feasibility study (RI/FS) and ultimately the OU 7-13/14 record of decision. Operable Unit 7-13/14 is the designation recognized under the Federal Agreement and Consent Order (FFA/CO) (DOE ID 1991) and the Comprensive Environmental Response, Compensation and Liability Act (CERCLA) (42 USC § 9601 et seq., 1980for the comprehensive RI/FS for the RWMC. Waste Area Group 7 is the designation recognized under the FFA/CO and CERCLA for remediation of the RWMC. For remediation management purposes, the FFA/CO divided the INEEL into 10 WAGs. Waste Area Group 7, comprising the RWMC, is located in the southwest quadrant of the INEEL.

1.2 Background

The INEEL is seeking new technologies to reduce costs for waste management to meet regulatory requirements and the goals of the Department of Energy (DOE) Idaho Operations Office (DOE-ID). Wastes disposed of in the Subsurface Disposal Area (SDA) at the Radioactive Waste Management Complex were originally dumped in trenches. The SDA was established on 113 acres in July of 1952. The site was designated as the Nuclear Reactor Test Site (NRTS) Burial Ground. The wastes originally disposed of in the SDA was materials generated as part of the nuclear power research conducted at the NRTS, currently designated as the INEEL. The disposed waste typically consisted of debris type material including paper, laboratory ware, filters, metal pipe fittings, and other items contaminated by the mixed fission products generated at the INEEL. The waste was typically packaged in cardboard boxes. The boxes were then taped shut and collected in the dumpsters. The dumpsters were then emptied into the trenches and covered with native soil.

Rocky Flats plant transuranic (TRU) waste was also disposed of in the SDA. The Rocky Flats wastes were containerized in metal drums and wooden crates and stacked horizontally in the pits and trenches among the mixed/fission- product wastes from the INEEL. The waste forms were then covered with native soils. Approximately 2-million cubic feet of waste were placed in the SDA using these landfill techniques between the approximate dates 1952-1970. An in-situ grout (ISG) treatability study is needed to focus on the encapsulation and stabilization of radioactive and radiologically contaminated waste and intermixed soils buried at OU 7-13/14.

2. EXPERIMENTAL DESCRIPTION

Six grout materials were subjected to a series of tests to obtain quantitative information regarding their strengths and weaknesses relative to in-situ grouting for stabilization and solidification of low-level radioactive waste materials. The following grouts were tested for this project:

- TECT-HG—A hematite-pozzolanic, cementitious mixture from Carter Technologies, Houston, Texas
- PREMIUM US GROUT—Proprietary pumice-based grout from US GROUT Malad City, Idaho
- ENVIROBLEND—Proprietary phosphate-based grout from American Minerals
- WAXFIX—Molten paraffin-based grout from Carter Technologies Houston Texas
- SAVANNAH RIVER SITE SALT STONE—Blast furnace slag, fly ash, and minor amounts of Portland Cement
- SAVANNAH RIVER TANK CLOSURE GROUT—ASTM type V Portland Cement, blast furnace slag, and silica fume. This grout was modified during the course of the project and will hereafter be referred to as C-75.

The bulk of the testing program involved five of these grouts. The WAXFIX grout was only involved in a special investigation concerning the distribution of boron in the WAXFIX grout. The other five grout materials were first subjected to a series of implementability tests to insure that each grout had the necessary physical characteristics to allow its eventual use under field conditions. The grout properties of primary interest at this stage were viscosity, stability, rate of stiffening, maximum temperature during setting, and the amount of bleed water or settlement of the grout prior to setting. The grout implementability evaluation tests and performance requirements are listed in Table 1.

Table 1. Implementability tests and criteria for all grouts except WAXFIX.

Measurement	Performance Requirement	Procedure
Viscosity	≤ 7 minutes	API Procedure RP-13B-1
Initial gelation	(100 Pa) ≥ to 2 hours	Vane Shear Test
Final gelation	(1000 Pa) ≥ to 2 hours	Vane Shear Test
Pressure filtration	0.1 to 0.6 $min^{-0.5}$	API Procedure RP-13B
Maximum set temp.	< 100° C	In situ thermocouple
Minimum free water at curing	Qualitative judgment	Direct observation

Upon completion of the implementability testing phase of the project, each grout was mixed with three different interferences that simulated materials being considered for treatment by in-situ grouting at the INEEL site. The interferences were (1) a mixture of salts referred to as the nitrate salt mixture, (2) a combination of oil, organic solvents, and absorbents referred to as the organic sludge, and (3) soil from the INEEL site. Each of the grouts was mixed with various amounts of each of the interferences to evaluate the reasonable maximum loading for each grout-interference combination. Compressive strength testing at 14 days of age was used as the evaluation parameter. Based on these test results, interference loading levels were established for the remaining portions of the testing program.

More extensive physical and chemical evaluations were performed on the neat grouts and mixtures of each grout-interference combination prepared using the interference dosage established based on the interference tolerance testing. Testing was also performed to evaluate the encapsulation capabilities of the three most promising grout materials. This testing involved special microencapsulation testing and macroencapsulation testing. This testing was performed for the C75, T, and U grouts.

The final two project tasks did not involve the five primary grouts evaluated in the project. A special investigation was conducted to evaluate different approaches to distributing boron in the WAXFIX grout, and a study of the possible use of powdered activated carbon as a VOC absorbent was performed.

2.1 Procedures

At the beginning of the testing program, a specimen labeling system was developed to identify the various grouts and grout-interference combinations involved in the study. The labeling of neat grout specimens involved the use of a single letter to identify the type of grout. These were C for the C75 grout, E for the Enviro Blend grout, S for the Savannah River Salt Stone grout, T for the Tect grout, U for the US Premium grout, and W for the WAXFIX grout. For grout and interference mixtures, the letter indicating the grout type was followed by a two digit number indicating the interference loading percentage, which was followed by a letter indicating the interference type. The interference type designations were N for the nitrate salt mixture, O for the organic sludge mixture, and S for the INEEL soil. As an example, the grout-interference specimen containing the Savannah River Salt Stone grout with the nitrate salt mixture at 25 weight percent would be labeled S25N. This allowed accurate identification of the specimen type. When testing of multiple specimens was required for a particular test, the appropriate number of specimens were randomly selected from the batch of specimens, and the specimen name was appended with a specimen number or letter (1, 2, 3 or A, B, C) to distinguish the specimens from each another.

Table 2 is a summary of the testing methods and procedures for this project. Where applicable standardized tests were available, these test methods were used. In cases where there were no directly applicable standardized test methods, a suitable standardized test method was adapted to meet the needs of the project, or special test procedures were developed.

The subsections that follow include specific details and/or test modifications for test procedures utilized for this study.

2.1.1 ANS 16.1 Leach Test

Leach testing was performed for a minimum duration of 90 days in accordance with the procedures in ANS 16.1 - Measurement of the Leachability of Solidified Low-Level Radioactive Wastes by a Short-Term Procedure. The test was designed to provide a standardized laboratory method for characterizing the leaching behavior of low-level waste forms. Although the procedure does not necessarily simulate leaching behavior under actual burial conditions, the test allows a comparison of relative leachability of various combinations of waste and grout.

Cylinder specimens (5.08 cm-diameter by 10.16 cm-height) were leached in de-mineralized water, suspended by Teflon string (see Figure 1). The volume of leachant employed was 2200 ml, as specified by the ratio of 10 ± 0.2 of leachant volume to external geometric surface are of the specimen. After rinsing the specimens for an initial period of 30 seconds, the leachant was replenished at the following time intervals (2 and 7 hours, and 1, 2, 3, 4, 5, 19, 47, and 90 days), for ten leachate samples. Aliquots of the leachants were analyzed for Sr, Al, Ca, and $N0_3^{-2}$ using ICP. The leaching data is presented in terms of

Table 2. Testing plan for physical and chemical testing of neat grouts.

Property	Test Method
Viscosity	API Procedure RP-13B-1
Density	ASTM D 4380-84
Initial/Final Gel Time	Vane Shear Test
Maximum Set Temperature [(a)]	In situ thermocouple
Tensile Strength	ASTM C 496-96
Comp. Strength	ASTM C 39-96
Hydraulic Conductivity	ASTM D 5084-90
Shrinkage	Direct measurement
Pressure Filtration	API-RP-13B
Time to Set	ASTM C191-82 (vicant needle)
Eh [(b)]	ASTM D 1498-93
pH [(b)]	ASTM D 1293-95
Leach Test [(c)]	ANS 16.1

a. Temperature were recorded every 20 minutes using a thermocouple embedded into the sample until the temperature completes a cycle from room temperature to heat of hydration temperature back to room temperature.
b. Grout samples used for the ANS 16.1 test are the same samples used for Eh and pH measurements. Additionally, Eh and pH measurements will be recorded for the leachate at each step in the ANS 16.1 leach test process.
c. The leachate from the ANS 16.1 Leach Test will be analyzed for Al, Ca, Si, Sr and NO_3. Metals determinations will be performed using EPA method 200.8, *metals by ICP-MS*. Nitrate determinations will be performed using either EPA method 300.0 or SW-846 9056. Reporting will include raw data (g of element per area per time), leach indexes, and diffusion coefficients.

Figure 1. ANS 16.1 leach test apparatus - plastic tub with de-ionized water and sample suspended by string.

Diffusivity Coefficient and Leachability Index. Average leachability indices (LI) and diffusivity coefficients (D_e) were calculated for each of the replicate sets.

2.1.2 Macroencapsulation and Microencapsulation of Organic Sludge

This test is designed to simulate diffusion of untreated volatile organic compounds (VOCs) through competent regions of the grouted monolith such as the perimeter region (macroencapsulation) and from organic sludge-containing grout (microencapsulation). The organic sludge composition is shown in Table 3 (note that this same recipe was used in the grout interference testing).

For the macroencapsulation test, neat grout cylinders (7.62 cm-diameter by 6.35 cm-height) with a 3.81 cm-diameter by 2.54 cm-height cavity (formed with a plastic plug) were prepared and allowed to cure for 14 days. After the curing period, 12.52 g-carbon tetrachloride, 3.61 g-tetrachloroethylene, 2.92 g-trichloroethylene, and 3.41 g-trichloroethane was placed in the cavity and immediately covered with fresh grout (note that the cavity dimensions are such that the shortest distance VOC must travel through the neat grout is a uniform distance of 1.91 cm). The cylinder was then placed in a Teflon-sealed jar (total volume of 305 ml and air volume of 15.42 ml). Gas phase samples (20 µl) were taken every 10 days for 90 days.

Table 3. Material quantities for 1 kg organic sludge interference mixture.

Ingredient	Quantity	
Calcium Silicate	135.26	grams
Oil Dri	72.84	grams
Carbon Tetrachloride	171.4	milliliters
Tetrachloroethylene (PCE)	47.32	milliliters
Trichloroethylene (TCE)	47.32	milliliters
Trichloroethane (TCA)	65.86	milliliters
Texaco Regal Oil, R&O 68	328.05	milliliters

For the microencapsulation test, neat grout was mixed with approximately 9 wt.% organic sludge (Table 4 shows the mass of each compound in the sample), poured into cylinder molds of 7.62 cm-diameter by 6.35 cm-height, immediately placed inside of a Teflon-sealed jar (total volume of 305 ml and air volume of 15.42 ml), and allowed to cure for 14 days. A gas phase sample (20 µl) was taken from the jar after 14 days, then the jar was opened, the mold was cut from the sample, the sample was placed back into the jar, and then gas phase samples (20 µl) were taken every 10 days for 90 days.

Table 4. Microencapsulation test individual VOC mass for each grout. Weights correspond to 9 wt.% sludge in each grout.

	GROUT		
Ingredient	T	U	C75
Carbon Tetrachloride	15.74 g	12.54 g	13.87 g
Tetrachloroethylene (PCE)	4.45 g	3.55 g	3.93 g
Trichloroethylene (TCE)	3.99 g	3.18 g	3.52 g
Trichloroethane (TCA)	5.09 g	4.05 g	4.49 g

2.1.3 Controlled Temperature Curing

Curing of all of the grout and grouted interference specimens was done under controlled temperature and at 100 percent relative humidity. Except for the curing of the neat grout specimens used for the physical and chemical testing part of the study, all specimens were cured at 23±1.7 degrees Celsius. The neat grout specimens for the physical and chemical testing part of the study were cured using a special temperature-matching water bath. The reference temperature was taken from a mixture of the grout and INEEL soil prepared using equal weights of grout and soil. This mixture was placed into an insulated container that was then placed into the controlled temperature water bath along with the neat grout specimens. A thermocouple inserted into the soil-grout specimen provided the command signal for the temperature controller that controlled the temperature of the water bath. The temperature of the water bath was maintained at about 1 degree Celsius less than the temperature of the reference specimen in the insulated container for the full 14-day curing period. Each set of neat grout specimens was cured in its own controlled temperature water bath using its own reference specimen to control the temperature. The system consisted of an insulated container, a circulating pump, a water heater, temperature sensors, control valves, a heat exchanger for cooling, and a chilled water loop. One of the controlled temperature water baths is shown in Figure 2.

Figure 2. Temperature controlled water bath and temperature controller used to do temperature matched curing of the neat grout specimens for physical and chemical evaluations.

2.1.4 Maximum Set Temperature

To determine the maximum set temperature, specimens were placed into insulated containers. The containers were plastic lined and insulated by a glass vacuum bottle. A thermocouple was inserted through a hole drilled into the screw-on cover of the container. The thermocouple was positioned near the center of the specimen. The temperature of the each test specimen was recorded at 5-minute intervals using a Keithley 2700 Series data acquisition system. Using the recorded data, the peak temperature for each specimen was determined. The insulated containers and the data acquisition system are shown in Figure 3.

Figure 3. Insulated containers and data acquisition system used to recording set temperatures.

2.1.5 Initial and Final Gelation

The shear strengths corresponding to initial gelation and final gelation of the grout were specified as 100 Pa and 1000 Pa, respectively, in the scope of work for this project. A laboratory vane shear apparatus was used to measure the shear strength of each gelation time test specimen at various times after mixing of the grout. The shear strength values were then plotted as a function of time, and the times corresponding to shear strengths of 100 Pa and 1000 Pa were read from the graph. The test specimens were approximately 22 centimeters in diameter and about 8.5 centimeters deep. The shear vane measured 25.4 millimeters across the width of the vanes, and the vanes were 25.4 millimeters tall. The specimen size allowed numerous tests to be performed while maintaining a clear distance of at least 12 millimeters between test locations. The shear vane was placed approximately at mid-depth in the test specimen for each test. The laboratory vane shear apparatus is shown in Figure 4.

Figure 4. Laboratory vane shear apparatus used to determine the initial and final gelation times.

2.1.6 Shrinkage

The term shrinkage is used to indicate the change in height of the hardened grout specimen relative to its height immediately after being cast. The primary mechanism associated with this change in height is settlement of the solids portion of the grout, which is accompanied by the accumulation of bleed water on the surface of the specimen. The term settlement more accurately reflects the mechanism causing the height change of the specimen. However, since shrinkage was the term used for this measurement in the scope of work for the project, shrinkage is the term used in the report as well.

Shrinkage of each grout was determined by measuring the height of a test specimen relative to the height of the specimen container. At the time of casting the specimens, great care was exercised to insure that the initial height of the specimen coincided with the height of the specimen mold. After the grout had hardened, the height of the specimen mold was measured at three locations, and the height of the grout specimen was measured at three locations. The difference between the averages of these two groups of measurements, expressed as a percentage of the initial height of the specimen, is the shrinkage.

2.1.7 Hydraulic Conductivity

The hydraulic conductivity testing of the neat grout specimens and the grouted interference specimens was conducted in general accordance with ASTM D 5084 Standard Test Method for Measurement of Hydraulic Conductivity of Saturated Porous Materials Using a Flexible Wall Permeameter. According to the test method, the test is terminated after four successive values of the measured hydraulic conductivity are within 25 percent of the mean value or within 50% of the mean value, depending on the magnitude of the mean value. Several of the tests in this study did not satisfy this criterion. This is primarily because the specimens containing the interferences are well below saturation at the start of the test, and that the specimens are relatively low permeability. These two factors contribute to the variability of the individual measurements of a particular test. The termination criteria of the test method are most applicable for specimens that are fully saturated, or nearly so, at the start of the test, as suggested by the title of the test method. Despite the fact that the specified termination criterion was not satisfied in all cases, the test results still provide valuable information on the relative hydraulic conductivity of the grouts and the various grout-interference combinations. In many cases the termination criterion of the test method were satisfied. In the cases that did not satisfy the termination criterion, they generally came close to meeting it, and the test duration was usually 4 to 7 days. As a result, the recorded values provide a good indication of the hydraulic conductivity of the specimens tested. The custom-made set of equipment used for the hydraulic conductivity testing is shown in Figure 5. The other set of equipment utilized for the testing uses the same test arrangement, but is produced by a different manufacturer.

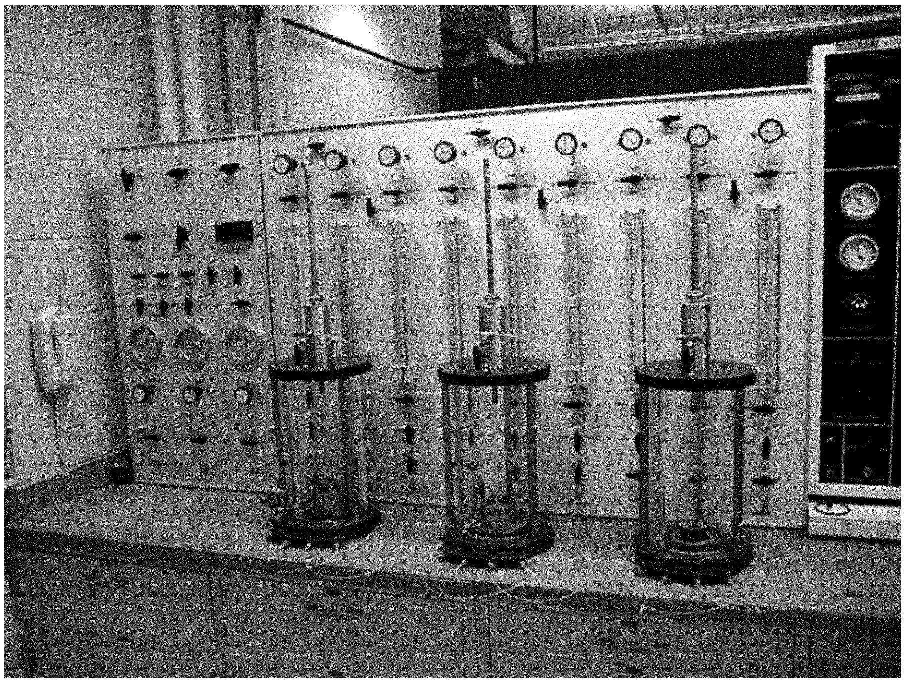

Figure 5. Hydraulic conductivity testing apparatus.

2.2 Quality Assurance Procedures

All testing was accomplished according to the test plan and recorded in a logbook. The appropriate controls for individual tests were prepared and measured and data was reported according to the ASTM or with other accepted statistical measures.

2.3 Equipment List

The following equipment was used in the testing program:

Water—Nanopure system

Drill with stainless steel mixing blade

pH meter—Cole Parmer Model 05669-20

Inductively Coupled Plasma (ICP) Analyzer

Water bath—Fisher Model 127

Drying oven—Grieve Model LW-201C

Scale—Ohaus Analytical

Plastic syringe

Glass and plastic beakers

Vacuum filtration unit

MTS 300 HV-1005 loading apparatus

Geological soil sieves (Standard)

Electronic Balance – Mettler

Marsh Funnel Test Set – Fann Instrument Co.

Mud Balance – Fann Instrument Co.

Mixer – Hobart N50

Mixer – Silverson RBXL Abramixer

Compression Machine, 2 kip – Geotest Instrument Corp.

Compression Machine, 100 kip – MTS servo-hydraulic

Compression Machine, 300 kip – Warner & Swasey

Pressure Filtration Apparatus – Fan Instrument Co.

Vane Shear Apparatus –

Hydraulic Conductivity Apparatus – Geotest Instrument Corp.

Hydraulic Conductivity Apparatus – custom made

Digital Multimeter / Datalogger – Keithley Instruments

Thermocouples – Omega Engineering

Fume Hoods

Microwave Oven – Panasonic

Controlled Temperature Curing Baths – custom made

Concrete Curing Room – custom built

Vicat Test Apparatus – Humboldt Manufacturing

Assorted hand tools