PRE-REMEDIAL DESIGN STUDIES FOR IDAHO NATIONAL LABORATORY BURIED WASTE

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ABSTRACT

The Idaho Closure Project (ICP) at the U. S. Department of Energy's Idaho National Laboratory (INL) is investigating potential treatment methods for the waste buried at the Subsurface Disposal Area, a radioactive landfill that is part of the Radioactive Waste Management Complex (RWMC). The wastes include a variety of sludges, debris, and nitrate salts, contaminated with hazardous chemicals and transuranic (TRU) and non-TRU radionuclides. Alternatives under consideration include in situ thermal desorption (ISTD), in situ grouting (ISG), and ex- situ grouting (ESG). The investigations involve literature searches and bench- and engineering-scale tests to determine the feasibility and applicability of the candidate treatment methods. Researchers have used both surrogate and retrieved waste in recent testing. The retrieved wastes were nitrate salts from Pad A and TRU sludge from composite samples collected during the Glovebox Extractor Method Project at Pit 9.

Project personnel have collected information from previously performed ISV testing, but did not perform any additional ISV testing. Reactivity and thermal desorption tests have filled in gaps in support of in situ thermal desorption. At the same time, compressive strength, porosity, hydraulic conductivity, and leachability tests have generated data to assess the feasibility of grouting the SDA waste. Additional tests will be performed in fiscal year 2005. This paper summarizes the fiscal year 2004 testing and results.

INTRODUCTION

One of the major obstacles to closing out the Environmental Management (EM) work at the INL is determining how to address the waste buried at the SDA, a radioactive landfill that is part of the RWMC. The SDA has been designated Waste Area Group (WAG) 7 Operable Unit (OU) 7-13/14 under the Comprehensive Environmental Response, Compensation, and Liability Act remediation of the facility. Negotiations are underway among the Department of Energy, the State of Idaho and the Environmental Protection Agency to establish which wastes need to be retrieved to reduce the risk of contamination spread, and which wastes can safely be left in place, possibly following in situ treatment. The ICP is conducting a Remedial Investigation/ Feasibility Study (RI/FS) to document the efficacy and implementability of alternatives for both in situ and ex-situ treatment of the SDA waste. The treatment alternatives under investigation include in situ and ex-situ grouting, in situ and ex-situ thermal desorption, and in situ vitrification. This paper presents the results of a wide range of tests conducted to investigate the treatment options under consideration.

The testing performed in support of the RI/FS was limited to those technologies that were deemed most promising for eventual application. Remedial technologies and process options that failed to adequately meet the requirements of CERCLA criteria during initial screening have been eliminated from further analysis and consideration [1]. The screening process in Zitnik et al. streamlined the list of available remedial technologies and process options, retaining for subsequent development and screening only those that met the criteria. Several considerations were weighed in determining the treatment technologies to be investigated. The considerations included both technical and programmatic aspects of handling the SDA remediation. Technically, the makeup of the waste and the time that the waste has been buried add to the complexity of the waste treatment problem. Waste containers have been breached releasing contaminants to the surrounding soil and mingling constituents that may be reactive under some conditions. Certain technologies, if applied inappropriately, could spread mobile contaminants, adding to the overall problem. Thermal treatment systems often come under intense scrutiny because of their potential to produce hazardous gas-phase compounds. The remediation effort will retrieve some or all of certain waste types depending on the results of negotiations with stakeholders. Based on these considerations, the following treatment technologies were examined in the studies reported here: in situ and ex-situ thermal desorption, in situ grouting, and ex situ grouting. In situ vitrification (ISV) information was gathered from previous studies, but because the application of ISV appeared to be unlikely, investigation of the technology was cut short.

ISTD focuses on remediating chlorinated organics in sludge and debris in the SDA, possibly mingled with nitrate salts. ISG and ESG would physically stabilize waste, and slow the release and migration of most hazardous inorganics and radionuclides. In addition, ISG-treated areas of the SDA would minimize subsidence and support a future surface barrier to reduce water infiltration into the waste. Tests to evaluate each of the alternatives were guided by the *Test Plan for the Evaluation of In Situ Thermal Desorption and Grouting Technologies for Operable Unit 7-13/14* [2]. The tests reported in this paper address the CERCLA criteria of effectiveness, both short- and long-term; reduction in mobility of contaminants through stabilization; and implementability. Additional tests are being performed in fiscal year 2005 (FY05), some of which were identified as needed because of testing performed in FY04 and others that resulted from data gaps identified during early application of in situ grouting in the SDA

SITE BACKGROUND

Information regarding the waste buried at the SDA and efforts to develop remediation pathways for that waste is provided in the following sections of the report.

Waste and Contaminants of Concern

Contaminants—disposed of in shallow subsurface disposal units consisting of pits, trenches, and soil vaults—include hazardous chemicals, remote-handled fission and activation products, and TRU radionuclides. Disposal of TRU and mixed waste—mostly from the Rocky Flats Plant (RFP) in Colorado—was allowed through 1970. Radioactive waste from off-site sources originated from a variety of facilities, including defense agencies, universities, commercial operations, and the Atomic Energy Commission [1]. The waste with its associated contaminants can either be concentrated at the location where it was dumped, or it can be dispersed through the surrounding soil matrix. Waste retrieved recently in the Glovebox Extractor Method Project still had some plastic bags intact, but the drums in which the waste had been disposed of, had corroded away. Wastes at Pad A, a retrievable waste storage site, are mainly nitrate salts with radioactive and hazardous contaminants.

Examples of waste types at the SDA are nitrate salts similar to those in Pad A, combustibles, soil, and three types of sludges (inorganic, organic, and nitrate salt). Depending on the trench, the wastes also contain TRU and non-TRU radionuclides. Based on recent mapping and burial records, nitrate or organic sludge drums are found in 20% or less of four pits [3,4]. Where organic sludge drums are located, the density is usually less than 4.5 drums per square meter. Where nitrate salt drums are located; the density is less than 1.6 drums per square meter. High-density areas greater than this occur in less than 10% of the total drum area; thus areas of high density are rare.

Organic sludge found in the SDA contains chlorinated volatile organic contaminants of concern, such as trichloroethylene (TCE), trichloroethane (TCA), perchloroethylene (PCE), methylene chloride, and carbon tetrachloride. Table I provides an estimated composition of the organic sludge of greatest concern. The actual composition is still being evaluated.

Table I. SDA organic sludge (Rocky Flats Series 743).

		Historic Estimated Concentration				
		Vol%		Wt%		
Material	Specific Gravity [a]	1981 [b]	1998 [d]	1981	1998 [d]	Selected Wt%
Texaco oil	0.87	22	16	20	14	29
Misc oil [c]	0.90	11 [c]	8	13	10	
CCl_4	1.59	17	19	27	30	27
TCE [c]	1.46	4	5	6	6	7
TCA	1.44	5	6	9	8	9
PCE [c]	1.59	4	5	6	6	7
$CaSiO_3$	2.5	8.4	12	21	29	13.5
Oil Dri	2.3					7.3

a. The organic chlorinated solvents are over 60% denser than the oils (Sp. Gr. [7} 1.5 vs 0.87).
b. The year of records and estimation.
c. 43% misc. oil (mineral oil used for the surrogate) and solvents listed in shipping records, of which 20% has been divided amongst TCE and PCE. TCE and PCE put here from miscellaneous oil category.
d. E. C. Miller and J.D. Navratil (INL/EXT-98-00112) [5].

The primary halogenated hydrocarbon COC in the organic sludge is carbon tetrachloride. Calcium silicate, Oil-Dri, Microcel®, or other sorbent material was added as an absorbent for the organic liquids (Texaco Regal Oil [R&O 32 or 68]) [6].

Previous Testing

Previous research, particularly extensive bench and field grout tests, set the stage for much of the FY04 testing. ISTD has been determined to be robust enough for operation in metals, debris, and containerized waste, as well as soil [7]. Depending on the temperature at which the system is run, the system can either volatize and collect or destroy many organic materials, including polyethylene, polyvinyl chloride, latex, paper, ion exchange resins, solvents, and oils. Basic physical data for various candidate grouts for ISG application were previously gathered during a number of studies [8]. Parameters studied include hydraulic conductivity, compressive strength, tensile strength, heat of hydration, initial and final gel times, pressure filtration, viscosity, and leachate Eh and pH. ISG has been demonstrated on mixed-waste contaminated soil sites [9]. Grout stabilization of nitrate salts was investigated by several researchers [10,11,12].

SCOPE OF FY04 TESTING

The *Test Plan for the Evaluation of In Situ Thermal Desorption and Grouting Technologies for Operable Unit 7-13/14* [2] outlines the testing that was originally planned for FY04 completion. Of the tests described, project personnel completed 15 in FY04. Four of the tests: macroencapsulation, microencapsulation, plutonium aerosolization, and fracture propagation were postponed or eliminated. Plutonium aerosolization testing may be performed in FY05 depending on decisions that will be made regarding the potential for implementation of ISG in portions of SDA where plutonium waste is expected to be present. One study in addition to those described by the test plan, an investigation of the durability of WAXFIX, was performed. Tests for ISTD and ISG also used surrogate and actual TRU waste from the SDA. ISG tests used surrogate non-TRU waste. ESG bench tests used waste from Pad A. A series of non-radioactive (cold) tests established the approach for radioactive (hot) testing. Engineers conducted hot tests using appropriately spiked surrogate wastes, material retrieved from Pit 9 by the OU 7-10 Glovebox Excavator Method Project, and material from Pad A. All preparations for hot testing, including safety documentation, were completed before accepting material from Pit 9 or Pad A.

Grouts under consideration for ESG of nitrate salts were tested with samples of actual waste from Pad A. Uranium is the major contaminant of concern associated with the nitrate salts. The mobility of the uranium in the salts was examined along with some physical attributes of the waste forms produced.

Grouts under consideration for ISG—neat and with various admixtures—were tested for durability, for characteristics that would effect leaching or binding of contaminants, and for attributes that would impact contaminant transport in treated waste forms.

Tests in support of ISTD quantified major emissions as wastes and soils were slowly heated, and determined the degree of hazardous organic contaminant and nitrate removal and/or destruction from soil and waste. Potential mixtures of organics and nitrates were tested for reactivity with temperature variation.

The tests conducted or initiated in FY04 were the following:

- Compressive strength
- Porosity
- Hydraulic conductivity
- Department of Transportation (DOT) oxidizer
- Reactivity: differential scanning calorimetry
- Drum-scale reactivity at Energetic Materials Research and Testing Center (EMRTC)
- Drum-scale thermal desorption at MSE Technologies

- Thermal desorption of TRU waste
- Boron retention and distribution
- Hydrogen generation
- Grouting and leaching of non-radioactive surrogate
- Grouting and leaching of Pad A waste
- Grouting and leaching of non-TRU waste
- Grouting and leaching TRU waste
- Grouting and leaching of thermally desorbed TRU waste

Because of space limitations the results of the grouting and leaching studies are not reported in detail here. The INL will issue a report that will summarize the results of all of the FY04 Pre-Remedial Design Studies that will eventually be revised to include FY05 test data. Status of the report can be obtained by contacting the authors of this paper at the INL.

PHYSICAL PROPERTY TESTING

Project personnel conducted testing to determine the physical properties of neat grouts and grouts mixed with surrogate waste. The results of the tests will be used in the design of a cap for the SDA and to model transport of water and contaminants through areas of the SDA that had been treated with ISG or ISTD followed by ISG.

Compressive Strength

The unconfined compressive strength was evaluated for a paraffin-based grout and three cementitious grouts ASTM procedure C-39, “Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens.” Project personnel used an Instron 4505 screw-driven load frame to perform the tests. The resulting data will determine which of the grouts have sufficient unconfined compressive strength for serving as a monolithic support for a cap on the SDA.

The test matrix included evaluating the grouts with several levels of waste loading. The grouts were the least tolerant of the organic sludge surrogate and the most tolerant of the soil. ISTD treatment significantly increases the amount of organic sludge surrogate that can be mixed with the grout. The ISTD treated material is much drier than the other materials and required additional water to be added for the cementitious grouts. Table II contains a summary of the compressive strength results.

Table II. Compressive strength test average results.

Compressive Strength of Grout (psi)					
Waste Type	Waste Loading (wt%)	Paraffin	Cementitious 1	Cementitious 2	Cementitious 3[a]
INL soil	12		5,884	4,150	3,896
INL soil	25		6,048	3,654	3,098
INL soil	40	366			
INL soil	50	212	2,529	1,924	1,278
INL soil	60	346			
INL soil	70	671			
INL soil	75				805
INL soil	80	328			
Organic sludge	3		7349	4295	3276
Organic sludge	5	123	6,100	3,706	2,878
Organic sludge	7	124	6,215	2,820	2,644
Organic sludge	9		6083	2618	3136
Organic sludge	10	112			
Organic sludge	12	114		2,347	
Organic sludge	25			204	
Organic sludge	30	45			
Organic sludge	50			6.5	
Nitrate salts	12		3171	3239	4802
Nitrate salts	25		2883	1193	1383
Nitrate salts	40	214.4			
Nitrate salts	50	185.0	2.7		1813.7
Nitrate salts	60	192.6			
Nitrate salts	75	<100	104		869
Nitrate salts	12		3171	3239	4802
Nitrate salts	25		2883	1193	1383
Nitrate salts	40	214.4			

[a]The results for the cementitious grouts were taken from Loomis et al. (2002)[8].

Porosity

The porosity test measured connected and surface-accessible pores. Project personnel used ASTM Procedure C 642-97, "Standard Test Method for Density, Absorption, and Voids in Hardened Concrete," the porosity test for aggregate concrete, to determine the porosity of cured candidate grouts. The grouts were tested both as neat grout and at 50 % waste loading, as specified by the test plan except for the paraffin-based grout, which was tested as neat grout and at 70 % waste loading.

The porosity of the paraffin-based grout was the lower than that of the cementitiuos grouts. This is consistent with the macro-encapsulation properties of the paraffin-based grout. Paraffin is a solid long-chain, hydrophobic hydrocarbon solution; this material contains pores, but they are not connected. Figure 1 summarizes the results of the porosity tests.

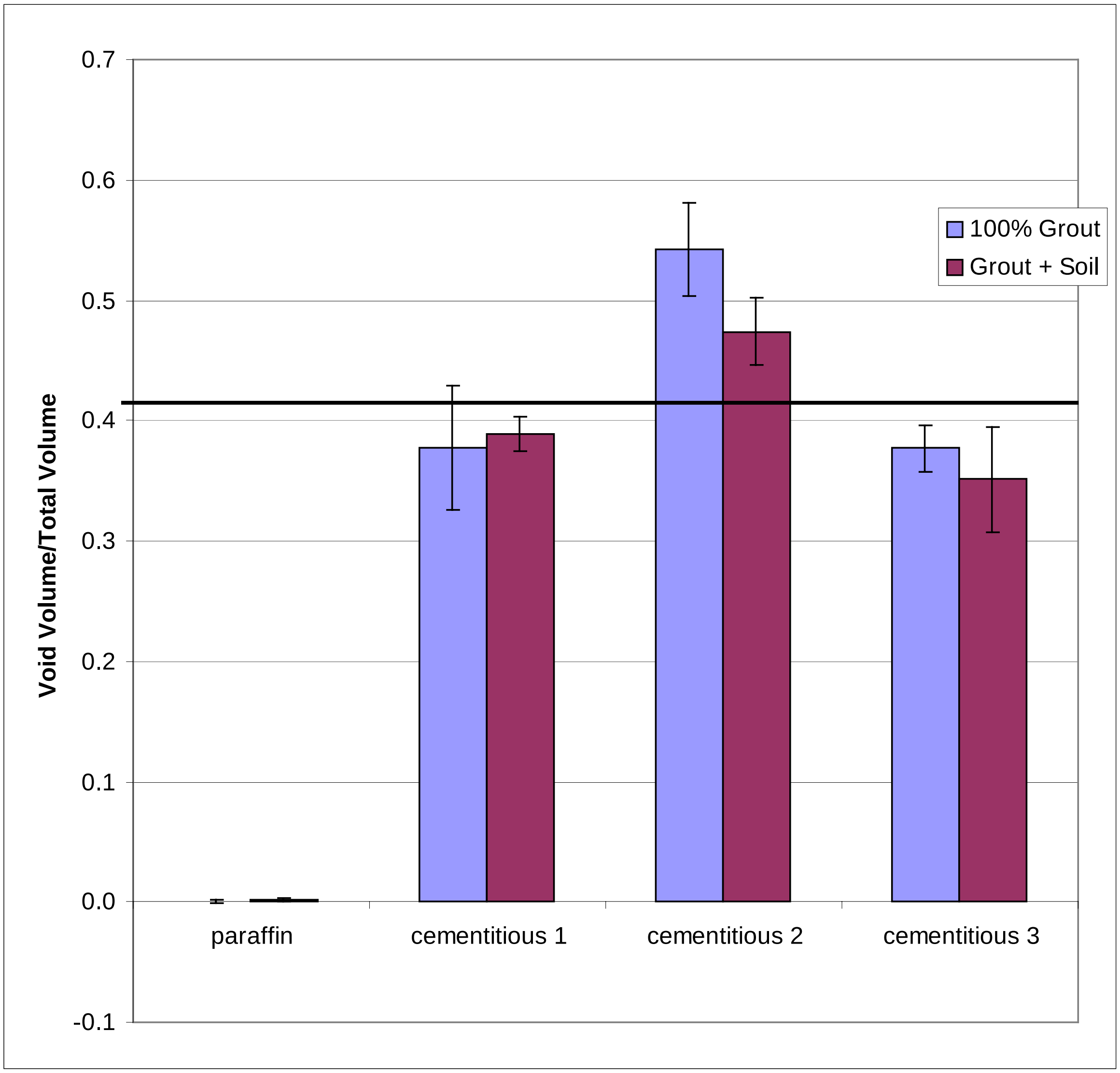

Fig. 1. Porosity of grouted waste forms.

Hydraulic Conductivity

The hydraulic conductivity of grouted waste was needed to support modeling for ISG of waste areas in the SDA that would be otherwise left intact with a cover. The performance of grouts that are candidates for use in the SDA was measured to see if one was considerably better than the others. Hydraulic conductivity was measured using ASTM 5084 "Standard Test Method for Measurement of Hydraulic Conductivity of Saturated Porous Material Using a Flexible Wall Permeameter." A paraffin grout as a neat material had the lowest hydraulic conductivity of all of the grouts tested; this is consistent with the porosity data collected on these materials. The addition of waste materials generally decreased the compressive strength and increased the hydraulic conductivity and porosity of the grouts relative to neat grout samples. These measurements suggest that the ability of the grouts to immobilize contaminants decreased with the presence of waste. Figures 2 and 3 show the results of the hydraulic conductivity testing.

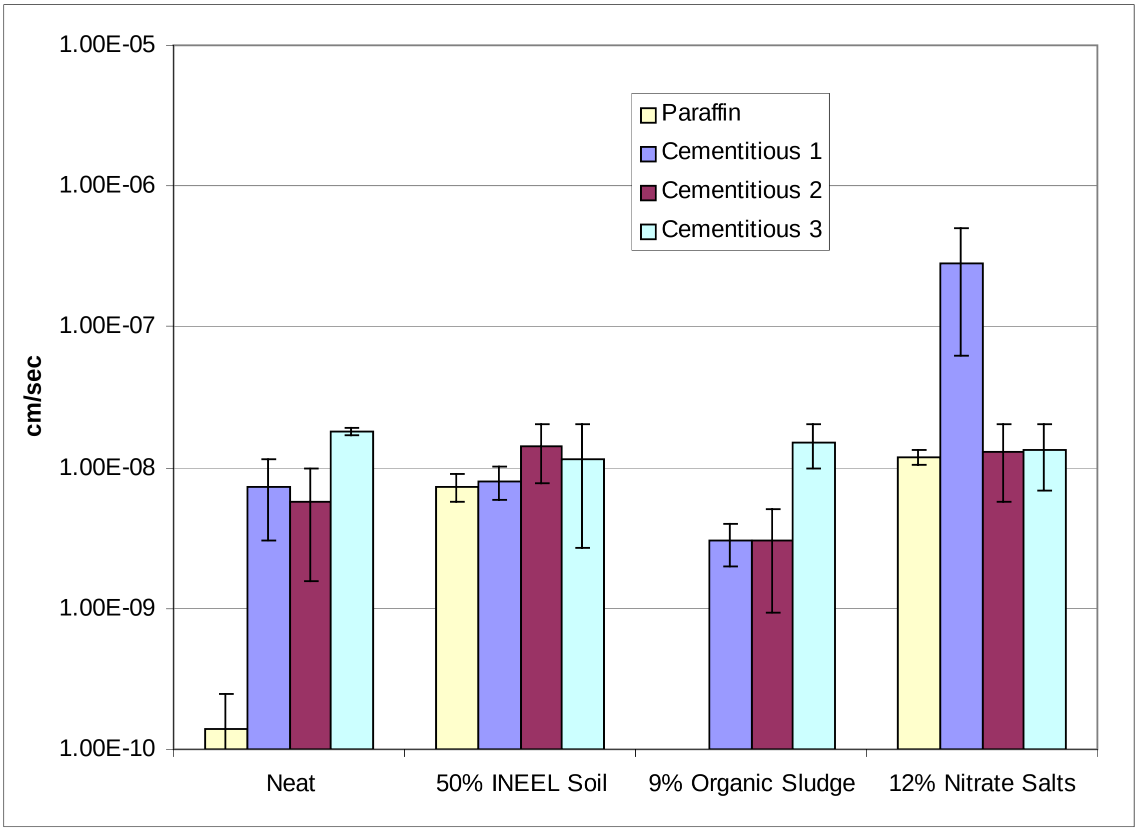

Fig. 2. Results of hydraulic conductivity testing of grouts and surrogate waste. (Paraffin was tested with 70% soil and 60 % nitrate salts.)

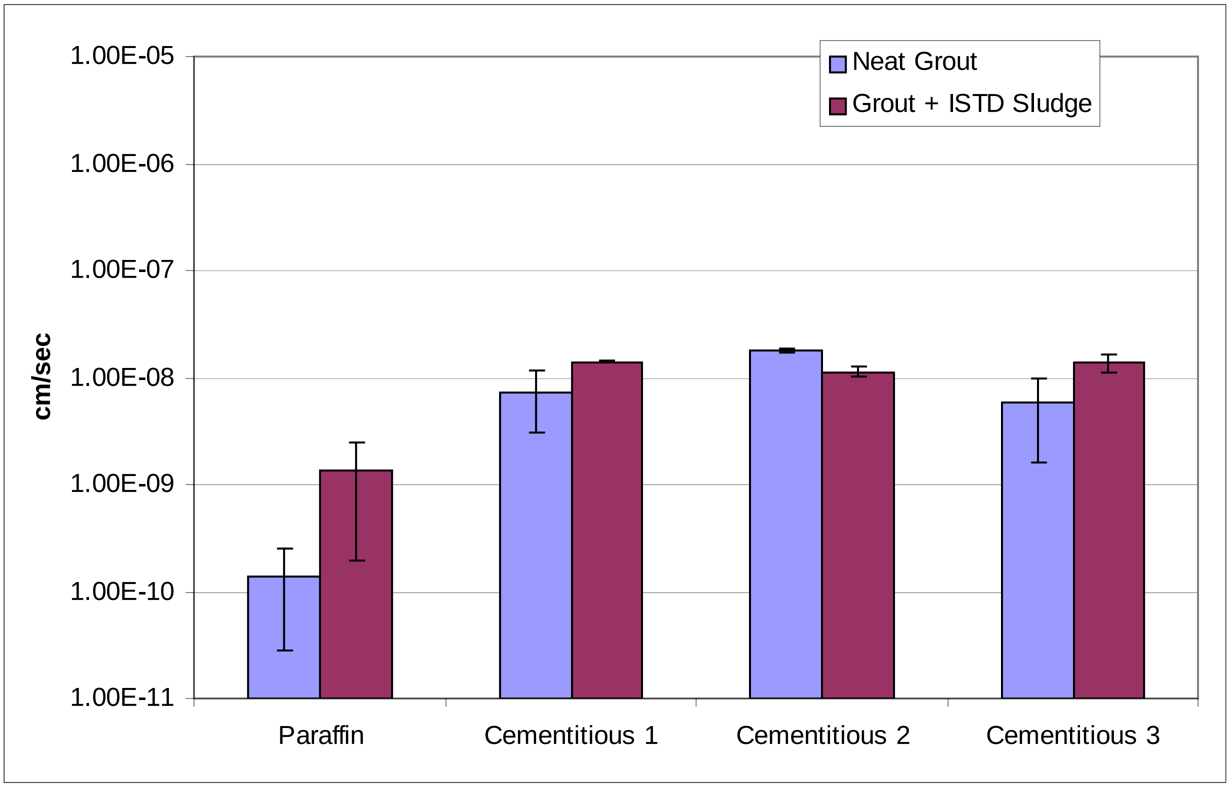

Fig. 3. Hydraulic conductivity of grouted thermally desorbed organic sludge.

REACTIVITY TESTING

Four forms of reactivity testing were performed or researched in the FY04 activities: DOT oxidizer tests, thermal gravimetric analysis, bench-scale heating tests, and drum-scale thermal-desorption reactivity tests. The DOT oxidizer data were needed to support the application of paraffin-based grout for stabilization of the Pad A nitrate salts. The other three activities were aimed at determining the potential for exothermic reactions caused by the ISTD heaters operation in waste mixtures containing organics and nitrate salts.

The DOT oxidizer tests were included in the Pre-Remedial Design Studies to evaluate whether the presence of a paraffin-based grout increases the potential for nitrate salts (the primary ingredient of the Pad A nitrate salts) to act as an oxidizer. Because the possibility for a rapid reaction is present if nitrates are involved in an accidental fire during storage or shipping, the objective of these tests was to determine whether sodium nitrate encapsulated in paraffin would be classified as a DOT oxidizer. The oxidizer tests were " . . .designed to measure the potential for a solid substance to increase the burning intensity of a combustible substance when the two are thoroughly mixed . . .." [13]

Project personnel decided that, because the base material of the candidate grout is paraffin, the grout should react similarly with an oxidizer. Research into previous tests with paraffin uncovered data that show that paraffin retards the oxidizing potential of nitrate salts. These data were taken as sufficient to demonstrate the potential impact of a paraffin grout coming in contact with SDA nitrate salts without additional testing. Table III provides a summary of the data derived from the literature.

Table III. Results from the U.S. Department of Transportation oxidizer tests

Combustion Time for Test Materials		
	1 to 1 Mass Ratio Test Mixture	4 to 1 Mass Ratio Test Mixture
Material Composition (wt%)	Burn Time (seconds)	Burn Time (seconds)
50% chopped wax (less than 2 mm)/50% $NaNO_3$	161[a]	188[a]
Encapsulation: 50% Wax/50% $NaNO_3$	131[a]	819[a]
Encapsulation: 50% Wax/50% $NaNO_3$ and sieve to less than 9.5 mm	628[b]	525[b]
100% $NaNO_3$	37[b]	25[b]

a. Mean of two replicates.
b. Based on one replicate.

The ISTD process is designed to destroy or remove organic material underground, particularly chlorinated solvents [14]. Most of the previous remediations using ISTD have been on contaminated soil sites, although one demonstration did include some debris [7]. To use ISTD in the subsurface at the RWMC would involve heating potential mixtures of previously containerized waste, including graphite powder, organic debris, chlorinated solvents, oils, and nitrate salts. During heating, these nitrate salts may melt and become potential oxidizers. No testing has addressed potential reactive interactions when slowly heating together containers of debris (Rocky Flats Series 743, 744), organic sludge, and (Rocky Flats Series 745) nitrate salts. To address these potential issues, the reactivity was tested of selected mixtures of simulated wastes using both bench-scale and drum-scale methods.

Thermal gravimetric analyses were performed on three carbon-containing waste surrogates: organic sludge, wax, and carbon powder mixed with the nitrate salt surrogate. Reactions at high heat rates were observed and were significantly reduced as ISTD heating rates were approached.

MSE Technologies (MSE) performed bench-scale tests with mixtures of surrogate organic waste and nitrates to determine whether larger-scale tests would be safe to conduct in their facility. The mixtures were observed while being heated to determine if highly energetic reactions occurred in the temperature regime specified for the large-scale runs. The mixtures proved to be highly reactive, so large-scale testing of the organic-with-nitrates mixtures was canceled at MSE.

Project personnel modified drum-scale reactivity testing scheduled for performance at EMRTC to incorporate portions of the testing that could not be safely performed at MSE. EMRTC conducted three drum-scale tests with mixtures of surrogate waste and nitrates. The mixtures were powdered graphite, organic sludge, and organic debris mixed separately with nitrate salts. The tests were performed at a remote site where an explosion-proof bunker could protect personnel and instruments in the event of a highly energetic reaction. An in situ thermal desorption heating element was inserted into a drum packed with one of the mixtures and heating was initiated. Temperatures and, to some extent, offgases were tracked to follow what was occurring in the drum. In each case, after some heating, an exothermic reaction occurred that rapidly burned the remaining contents of the drum or forced the contents out of the drum as a deflagration. Though the probability is small that waste mixtures in the SDA would achieve the bounding conditions of these tests, an ISTD system would have to address the safety concerns raised by these tests before it could be deployed.

ISTD

MSE conducted thermal desorption tests on four surrogate sludges at a drum scale to assist in assessing the capability of a Terratherm, Inc. ISTD heating assembly in treating SDA waste. The results of these tests were reported previously [15] and are compiled in a formal report available from MSE [16]. The MSE tests demonstrated that heating coupled with sufficient airflow through the waste matrix will not only drive off the volatile organics, but also, at least partially oxidize them. Increasing the airflow can dramatically increase the heat-up rate after ignition temperatures are reached. Prior to ignition, the airflow can retard heating to a point that heat-up is stopped, so the airflow rate is a critical parameter that would have to be controlled in field applications. Based on the tests, the offgas treatment system for an ISTD system would have to be robust to be able to handle VOCs coming from an area of organic-rich sludge.

Additional testing was performed to determine the effects of thermally desorption on SDA waste. Both surrogates and actual waste were heated in a furnace, cooled, and analyzed to determine if the desorption process affected the leachability of the radionuclides in the resulting waste matrix. The surrogates were spiked with rare earth elements, while the actual wastes were contaminated with a variety of radionuclides including transuranics. Each surrogate waste changed in color and texture upon heating. All surrogate wastes and soil lost progressively more weight as heating temperature increased. Chemical changes were apparent. The residue after heating appeared different in both color and texture from the original material suggesting that the weight loss was due to more than losing water. Leach testing of sludges and soils containing spiked rare earth tracers or radionuclides and actual contaminated waste and soil verified no increased mobility and indicated in many cases some amount of fixation of metals on solids, especially at higher temperatures. This retention increased with temperature being most pronounced beyond the 450°C target temperature. In summary the data from these tests indicate that in situ thermal desorption would not increase the mobility of the radionuclides in the waste matrix and in some cases improve their retention.

BORON RETENTION TESTS

One concern with using a paraffin-based grout in the SDA was the potential for the main ingredient of WAXFIX, paraffin, to act as a moderator when combined with radionuclides and raise the potential for a criticality occurring in the subsurface. The boron retention tests evaluated the potential for adding boron, a moderator poison, to the paraffin to eliminate the concerns of the grout acting as a moderator. Boron retention and distribution in wax were tested using cold surrogates. A soil test was conducted to assess the potential for B-10 to be filtered from the wax as the wax migrates from the original placement site during cooling. In addition project personnel conducted a settling test to determine if the boron would stratify before the paraffin hardened.

Preliminary data based on density changes indicated that boron concentration was reduced when molten wax passed through soil. These data were not verified with chemical analyses. Separation test results indicated that, over a 5-day cooling period, a small amount of separation occurs. In August of 2003, a report was published [17], which stated that the presence of paraffin grout within the SDA would not lead to the formation of a critical mass. That information eliminated the need for the retention testing, so the tests were terminated.

HYDROGEN GENERATION TESTING

The Pre-Remedial Design Studies also addressed the potential problem of hydrogen generation resulting from the radiolysis of a paraffin grout injected into a subsurface waste zone heavily contaminated with transuranics. Uranium-233 in a solid dioxide form was selected to serve as the alpha source in the testing. The U-233 and paraffin were placed in a 60-ml reactor and allowed to sit. Gas samples were taken periodically and examined with a gas chromatograph.

A special sample was drawn from two reactors for higher organic gases (methane to propane were not detectable). Alkanes and some complex organics were detected, but the concentrations were below 1.0 ppm(v).

The test data are within the expected range and are a good estimate for the paraffin alpha radiolysis. The net G_{H2} values for reasonable particle size do appear to be within the expected range of 1.0 to 3.0. The rates of net hydrogen production are shown in Figure 4.

Better and more complete test results can be obtained with controlled particle size of the U-233, a better sampling method, longer time intervals, and a more complete analysis of the gas. The data collected from this series of tests will be used to evaluate the feasibility of injecting paraffin into areas of the SDA and the areas subsequently capped.

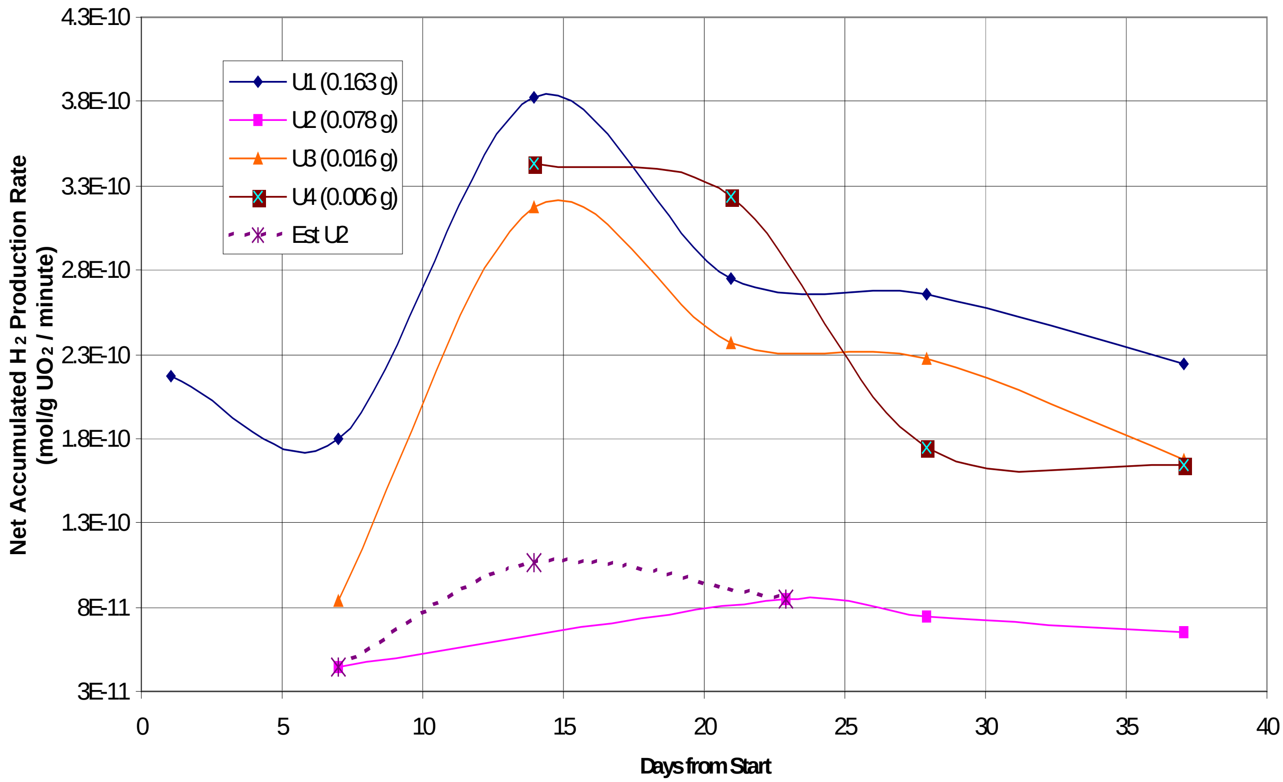

Fig. 4. Uranium-233 radiolysis of paraffin.

CONCLUSION

The FY04 Pre-Remedial Design Studies, performed to support decisions for the remediation of the INL's SDA, were very successful. The project personnel were able to complete the huge variety of testing at a considerably lower cost than expected. Engineers were able to devise tests such as the hydrogen generation procedure that produced meaningful data in the short time available. Each of the teams assigned tests were able to overcome the obstacles they faced and provide draft write-ups in time to support the RI/FS schedule. The data will be extremely valuable in determining the feasibility and applicability of the technologies and grouts tested. The data will also be used in models and designs for the remediation activities at the SDA. Some of the information has already been used to support the selection and subsequent injection of paraffin grout to reduce the corrosion of beryllium shield blocks buried in the SDA. Thermal treatment technologies presently face considerable opposition to deployment at the INL, so analysis of the alternatives must show the thermal treatment to be considerably more effective than others to be adopted. Presently the questions raised by the drum-scale reactivity tests at EMRTC would have to be answered before ISTD would be seriously considered. Additionally, much of the waste that would be appropriate for ISTD treatment will be retrieved and treated ex situ, if treatment is even required before shipment for disposal. ISG may prove to be appropriate for some areas of the SDA, but risk analyses for a capped SDA may show that grouting is not needed. Regardless of the outcome, the data gathered are a valuable resource to those who will have to make tough remediation decisions in the near future.

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